

# Report on Proficiency Test on incurred and spiked pesticides in barley



**EU Reference Laboratory  
on Cereals & Feeding stuff**

**EUPT-C6  
2012**

DTU Food  
National Food Institute

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**EU PROFICIENCY TESTS  
EUPT-C6, 2012**

**Incurred and Spiked Pesticide  
Residues in Barley**

**Final Report**

Version 2

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## PREFACE

Regulation 882/2004/EC [1], defines the general tasks and duties of the European Union Reference Laboratories (EURLs) for Food, Feed and Animal Health including the organisation of comparative tests. These proficiency tests are carried out on an annual basis, and aim to improve the quality, accuracy and comparability of the analytical results generated by EU Member States within the framework of the EU co-ordinated control and national monitoring programmes. Participation in the proficiency test scheme “European Union Proficiency Tests (EUPTs) for pesticide residues” is mandatory according to Article 28 of Regulation 396/2005/EC on maximum residue levels of pesticides in, or on, food and feed of plant and animal origin [2], as long as the analytical scope of the PT and the laboratory overlap.

The present EUPT was the sixth organized within the frame of the EURL activities with cereal matrices as Test Items. The previous PTs were EUPT-C1/SRM2 on wheat (2007), EUPT-C2 on wheat (2008), EUPT-C3/SRM4 on oat (2009), EUPT-C4 on rye (2010) and EUPT-C5/SRM6 on rice (2011). The PTs in 2007, 2009 and 2011 was jointly organised by the EURL-CF and EURL-SRM using and focusing on both MRM and SRM pesticides, where the present EUPT-C6 on barley (2012) are only focusing on MRM-pesticides. The barley flour Test Item used for EUPT-C6 was treated with 18 compounds partly in the field and partly post-harvest in the laboratory

Participation in EUPT-6 was compulsory to all National Reference Laboratories (NRLs) and Official Laboratories (OfLs) within the EU involved in the determination of pesticide residues in cereals and feeds using multiresidue methods for the EU co-ordinated control and/or their national programmes. Official laboratories from EFTA countries (Iceland, Norway and Switzerland), also contributing data to the EU-coordinated control programme as well as official laboratories from EU-candidate states (Croatia, FYROM and Turkey) were also invited to take part in this EUPT. Selected laboratories from Third Countries were also allowed to take part in this exercise, but their results, together with the EU-candidate state laboratories, were not used when establishing the Assigned Values. All NRLs and OfLs that were supposed to participate in this exercise, but decided not to take part, were asked to state the reasons for their non-participation. Laboratories that had registered to participate in this exercise, but then did not submit results, were also asked to provide explanations.

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





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# EUROPEAN COMMISSION EURL PROFICIENCY TEST ON PESTICIDE RESIDUES IN CEREALS EUPT-C6, 2012

## 1. INTRODUCTION

On 28 October 2011 the announcement of the 6<sup>th</sup> European Commission's Proficiency Test on Cereals (EUP-T-C6) was published together with the Calendar. The Specific protocol (**Annex 2**) was published on 14 November together with the Pesticide Target List including all compounds that could potentially be present in the Test Item. The Target Pesticides List included 107 individual compounds requiring the use of multiresidue methods (MRMs), along with a minimum required reporting level (MRRL) stipulated for each compound.

Links to The General Protocol containing information see (**Annex 1**) that is common to all EUPTs, as well as a list of labs that are obliged to take part in the EUPT-C6 was also provided via the homepage. The laboratories were able to register on-line from the 15 December 2011 to 15 January 2012. In total 127 laboratories from EU and EFTA countries agreed to participate in the test as well as 22 laboratories (from Third Countries (including EU-Candidate States)). Several EU-laboratories provided explanations for their non-participation as requested by DG-SANCO.

The present proficiency test was performed using barley flour of Danish origin, which had been partly treated in the field, and partly spiked post-harvest at the facilities of the EURL-CF. The Test Item contained 18 compounds in total. Danish Centre for Food and Agriculture at Aarhus University performed the field treatments. The pesticides employed for field treatment were selected by the EURL-CF and the quality group and the application rates and harvest intervals chosen was based on previous experience and data from supervised residue trials. The harvested grain was treated with nine pesticides post-harvest, and then checked for homogeneity before shipping to participants. Furthermore, the stabilities of the pesticides in the Test Item were checked several times during the period of time allowed for laboratories to undertake the PT exercise.

The participating laboratories were provided with 150 g portions of the treated whole barley flour Test Item and 150 g of the blank whole barley flour. The Test Items were shipped to the participants on 30 January 2012 and the deadline for submission of results to the Organiser was the 26 February 2012. The participants were asked to analyse the treated Test Item as well as the 'blank' material and report the concentrations of any pesticide residues found which were included in the Target Pesticide List (see **Appendix 2**). Submission of results was performed online via the website.



## 1.1 Analytical methods

The QuEChERS method [3], described briefly below, was used by the organiser to test the homogeneity and stability of the Test Items:

QuEChERS method, determination using GC-MS/MS and LC-MS/MS.

- Cold water was added to the milled sample and shaken. The extraction was performed immediately after, shaking with acetonitrile, before a salt and buffer mixture was added and the sample shaken again. The clean-up was performed in two steps. After centrifugation an aliquot of the supernatant was frozen out. After an additional centrifugation of the cold extract the supernatant was transferred to a tube with PSA and MgSO<sub>4</sub>. After shaking and centrifugation the extract was ready for analysis by GC-MS/MS and LC-MS/MS.
- For more details see [www.quechers.com](http://www.quechers.com)

## 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-coordinated control programme, a pesticide priority list ranking the pesticides according to their relevance and risk-potential, as well as a list of pesticides relevant to the specific commodity (barley). The overall capacity and capability of the laboratories within the EU, as assessed from previous PTs and surveys, was also taken into account. The minimum required reporting level (MRRL) for all pesticides in the target list was set at 0.01 mg/kg.

## 1.3 Preparation of the treated Test Item

Before preparing the Test Item, the pesticides and suitable target residue levels for the study were selected. The application rates and harvest intervals for the eight pesticides used for treatment in the field were chosen based on experience from earlier PT and data from supervised residue trials. The field spraying was performed by the Danish Centre for Food and Agriculture at Aarhus University. Approximately, sixty kilograms of the harvested barley grain was delivered for preparation of the Test Item. Following a preliminary analysis of the material it was decided to additionally spike in the laboratory with an additional eight pesticides, which were not included in the field treatments (see **Table 1**). Spiking in the laboratory was performed using pure standards for most of the pesticides. One kilogram of the field treated barley was spiked with all eight pesticide standards or formulations and was subsequently mixed with 59 kg field treated barley and homogenised thoroughly. The 60 kg of mixed barley grain was milled separately as four kilograms portions. To ensure that a well-homogenised bulk, with respect to both incurred and spiked residues, was obtained, the 4 kg portions were initially mixed individually, then doubled and mixed again and finally all mixed together. One hundred fifty gram portions were weighed out into screw-capped polyethylene plastic bottles, sealed, numbered, and stored in a freezer at about -20 °C prior to homogeneity testing and distribution to participants.

#### 1.4 Preparation of the 'blank' Test Item

The barley used to prepare the blank Test Item was also produced by the Danish Centre for Food and Agriculture at Aarhus University under similar growing conditions as the treated crop but without any pesticide treatment in the field or spiking in the laboratory. One hundred and fifty gram portions were weighed out into screw-capped polyethylene plastic bottles, sealed, and stored in a freezer at about -20 °C prior to distribution to participants.

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

Pesticide	Application in the field	Spike in laboratory	Formulation
Azoxystrobin	x		Amistar
Boscalid	x		Cantus
Carbendazim		x	Bavistin
Carboxin		x	Standard
Chlorpropham		x	Standard
Chlorpyrifos		x	Standard
Cypermethrin		x	Standard
Cyprodinil		x	Standard
Diflubenzuron		x	Standard
Epoconazole	x		Rubric
Fenpropidin	x		Tern
Isoprothiolane		x	Standard
Pendimethalin		x	Standard
Pirimicarb		x	Standard
Propiconazole	x		Bumper
Prothioconazole	x		Proline
Pyraclostrobin	x		Comet
Tebuconazole	x		Folicur

## 1.5 Homogeneity test

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

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

The acceptance criteria for the Test Item to be sufficiently homogenous for the proficiency test was that:  $S_s^2 < c$  where  $S_s$  is the between-bottle sampling standard deviation and  $c = F_1 \times \sigma_{all}^2 + F_2 \times s_{an}^2$ :  $F_1$  and  $F_2$  being constants with values of 1.83 and 0.93, respectively, from the 11 samples taken.  $\sigma_{all}^2 = 0.3 \times \text{FFP RSD (25\%)} \times$  the analytical sampling mean for all pesticides, and  $s_{an}$  is the estimate of the analytical standard deviation.

As all pesticides passed the homogeneity test, the Test Item was considered to be sufficiently homogenous and suitable for the PT-C6.

**Table 2** Statistical evaluation of the homogeneity test data (n=22 analyses using a sub-sample of 5 g in each case).

	Azoxystrobin	Boscalid	Carbendazim	Carboxin	Chlorpropham	Chlorpyrifos	Cypermethrin	Cyprodinil	Diflubenzuron
<b>Mean, mg/kg</b>	0.150	0.897	0.198	0.136	0.200	0.153	0.312	0.156	0.168
<b>S<sub>s</sub><sup>2</sup></b>	0.0001	0.0025	0.00004	0	0	0	0.0001	0.00001	0.0001
<b>c</b>	0.0004	0.0092	0.0006	0.0003	0.0007	0.0003	0.0018	0.0003	0.0008
<b>Pass/Fail</b>	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass

	Epoxiconazole	Fenpropidin	Isoprothiolane	Pendimethalin	Pirimicarb	Propiconazole	Prothioconazole -desthio	Pyraclostrobin	Tebuconazole
<b>Mean, mg/kg</b>	0.567	0.768	0.082	0.109	0.260	0.169	0.082	0.391	0.390
<b>S<sub>s</sub><sup>2</sup></b>	0.0011	0.00001	0.00000	0	0.00000	0	0.00002	0.0007	0.0002
<b>c</b>	0.0035	0.0002	0.0001	0.0003	0.0010	0.0006	0.0001	0.0017	0.0022
<b>Pass/Fail</b>	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass

S<sub>s</sub>: Between Sampling Standard Deviation

## 1.6 Stability tests

The analytical methods described briefly above (in section 1.1) were also used for the stability tests. The tests were performed on five occasions. In each case one test was performed before the start of the PT-exercise and one after the completion date.

Two different storage temperatures were used; room temperature and -18 °C. The stability tests were performed on five occasions at a storage temperature of -18 °C and on three occasions at room temperature (Day 1, 2 and 5):

The dates of testing were as follows:

Day 1: 31 January 2012

Day 2: 6 February 2012

Day 3: 13 February 2012

Day 4: 20 February 2012

Day 5: 27 February 2012

The average results from each stability test for the MRM pesticides are given in **Table 3**. The tests did not show any significant decrease<sup>1</sup> in the pesticide levels at -18 °C indicating that at these storage conditions the pesticides present in the Test Item remained stable for the entire duration of the Proficiency Test. Figures of the stability data and slope of the regression line<sup>1</sup> can be seen in **Appendix 4**. The test performed by storage at room temperature, likewise showed no significant decrease, except for carboxin where the decrease was 8%. However, the specific protocol prescribes that the Test Item should be stored at -18 °C and the Test Item was shipped with a freezer block that kept the sample cold at least for a couple of days. The test items arrived to the European laboratories within approx. 1-3 days and three participants that obtained z-scores below -2 received the shipment on 31 January, only one day after the parcel was shipped from Copenhagen. The low result can therefore not be related to possible degradation of carboxin. For the overseas participants the shipment period was 2-19 days. The two participants that obtained z-scores below -2 (-2.8 and -2.6) received the test items after 8 days. However, they obtained low results for many compounds, with 9-10 z-scores below -2.

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<sup>1</sup> If the slope of the regression line of the 3-5 results was above -5% the test material was defined as stable.

**Table 3** Stability test results at -18 °C and room temperature

	Azoxystrobin	Boscalid	Carbendazim	Carboxin	Chlorpropham	Chlorpyrifos	Cypermethrin	Cyprodinil	Diflubenzuron
Day 1	0.151	0.874	0.187	0.125	0.228	0.169	0.244	0.142	0.318
Storage at -18 °C (mean values in mg/kg, n=5)									
Day 2	0.175	0.926	0.198	0.142	0.239	0.179	0.293	0.156	0.351
Day 3	0.173	0.962	0.189	0.144	0.234	0.176	0.275	0.144	0.318
Day 4	0.173	0.891	0.185	0.140	0.234	0.175	0.251	0.146	0.317
Day 5	0.125	0.716	0.180	0.101	0.187	0.140	0.233	0.125	0.285
	<b>pass</b>	<b>pass</b>	<b>pass</b>	<b>pass</b>	<b>pass</b>	<b>pass</b>	<b>pass</b>	<b>pass</b>	<b>pass</b>
Storage at Room temperature (mean values in mg/kg, n=5) – informative purpose only									
Day 2	0.160	0.915	0.186	0.099	0.235	0.155	0.233	0.135	0.307
Day 5	0.146	0.815	0.182	0.082	0.232	0.141	0.218	0.133	0.297

	Epoxiconazole	Fenpropidin	Isoprothiolane	Pendimethalin	Pirimicarb	Propiconazole	Prothioconazole-desthio	Pyraclostrobin	Tebuconazole
Day 1	0.549	0.863	0.078	0.105	0.257	0.162	0.079	0.384	0.374
Storage at -18 °C (mean values in mg/kg, n=5)									
Day 2	0.568	0.901	0.083	0.109	0.266	0.176	0.077	0.419	0.410
Day 3	0.598	1.064	0.080	0.108	0.262	0.185	0.085	0.410	0.414
Day 4	0.542	0.937	0.079	0.110	0.256	0.161	0.071	0.372	0.393
Day 5	0.436	0.737	0.069	0.087	0.243	0.129	0.064	0.318	0.299
	<b>pass</b>	<b>pass</b>	<b>pass</b>	<b>pass</b>	<b>Pass</b>	<b>pass</b>	<b>pass</b>	<b>pass</b>	<b>pass</b>
Storage at Room temperature (mean values in mg/kg, n=5) – informative purpose only									
Day 2	0.566	0.818	0.078	0.103	0.255	0.159	0.080	0.397	0.381
Day 5	0.509	0.833	0.075	0.094	0.245	0.148	0.075	0.357	0.353

## **1.7 Organisational details**

### *1.7.1 Access to documents, registration and confidentiality*

In the invitation letter of 28 October 2012, all NRLs and OfLs were prompted to register at the CIRCA platform of the pesticide EURLs where the online registration link and all documents related to this EUPT (Calendar, Target Pesticides List, Specific Protocol, General Protocol) were uploaded. All relevant laboratories, independent of whether, or not, they were intending to participate at this exercise had to register on the webpage by the 15 January 2012. Laboratories that were intending not to participate were given the opportunity to explain the reasons for their non-participation. Participants from third countries also had access to the online registration link. However, their participation had to be approved by the Commission. After registration, the participants were provided with a username, password, laboratory code and the link for the online result submission website. This ensured confidentiality throughout the entire duration of the PT.

### *1.7.2 Distribution of the Test Item*

On 30 January 2012, one bottle of treated Test Item (150 g) and one bottle of 'blank' material (150 g) were shipped to all participants in boxes containing a freezer block. The laboratories were asked to check the state of the sample on receipt and to enter the website (see above) and communicate whether they accept/not accept the Test Items.

### *1.7.3 Submission of results*

An online submission tool was developed, allowing participants to submit their results via the internet. All participants had access to the result-submission website (<http://thor.dfvf.dk/ptc>) from the day of shipment until the result-submission deadline (27 February 2012). Participants were asked not only to report their analytical results, but also to give information regarding accreditation, reporting limits and details about the methods they used to analyse the Test Items.

## 2. EVALUATION OF THE RESULTS

### 2.1 False positives and negatives

#### 2.1.1 False positives

These are results reported above the MRRLs that suggest the presence of pesticides that were listed in the Target Pesticide List, but which were: (i) not detected by the Organiser, even after repeated analyses, and/or (ii) not detected by the overwhelming majority (e.g. 95%) of the participating laboratories that had targeted the specific pesticide. However, in certain instances, case-by-case decisions by the EUPT-Panel may be necessary.

Any results reported that are 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 used by the Organiser to treat the Test Item and were detected by the Organiser and the majority of the participants that had targeted these specific pesticides, at or above the MRRL. Results reported as <RL (RL= Reporting Limit of the laboratory) will be considered as not detected and will be judged as false negatives. However, in certain instances, case-by-case decisions by the EUPT-Panel may be necessary.

In cases of the assigned value being less than a factor of 4 times the MRRL, false negatives will not be assigned as this is not statistically justifiable.

### 2.2 Estimation of the true concentration ( $\mu$ )

The “true” concentration (assigned value) will be typically estimated using the median of all the results. In special justifiable cases, the EUPT-Panel may decide to use only part of the population of results to establish the median (e.g. only results with z scores > 5.0, or by excluding results generated by a method that demonstrably generates significantly biased results, e.g. due to incomplete extraction).

### 2.3 Standard deviation of the assigned value (target standard deviation)

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

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

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

### 2.4 z-Scores

This parameter was calculated using the following formula:

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

Where:  $x_i$  is the value reported by the laboratory,  $\mu_i$  the assigned value, and  $\delta_i$  the standard deviation at that level for each pesticide (i).

Any z-scores of  $> 5$  will be reported as  $>5$  and where combined z-scores are calculated a value of "5" will be used.

z-scores will be interpreted in the following way:

$|z| \leq 2$  Acceptable

$2 < |z| \leq 3$  Questionable

$|z| > 3$  Unacceptable

For results that are considered to be false negatives, z-scores will be calculated using the MRRL or RL (the laboratory's Reporting Limit) if the RL < MRRL.

The EUPT-Panel will consider whether, or not, these values should appear in the z-score histograms.

z-Scores will not be calculated for any false positive result.

## 2.5 Category A and B classification and combined -Scores ( $AZ^2$ )

The EUPT-Panel will decide whether to classify the laboratories into two groups - A or B. Laboratories that detect a sufficiently high percentage of the pesticides present in the Test Item (e.g. at least 90%) and reported no false positives will have demonstrated 'sufficient scope' and will therefore be classified into Category A. The 90% criterion will be applied following **Table 4**. For evaluation of the overall performance of laboratories within Category A, the Average of the squared z-Score ( $AZ^2$ ), will be used.

Laboratories within Category B will be ranked according to the total number of pesticides present in the sample. The number of acceptable z-scores achieved will be presented too. The EURL-Panel retains the right to calculate combined z-scores (see below) also for Category B labs, e.g. for informative purposes, provided that a minimum number of results (z-scores) is available

## 2.6 Combined z-scores

For evaluation of the overall performance, the Average of the squared z-Score ( $AZ^2$ ) will be used. The  $AZ^2$  is calculated as follows:

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

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".

This formula multiplies each z-score by itself and not by an arbitrary number. Based on the  $AZ^2$  achieved, the laboratories are classified as follows:

This SWZ has the following classification similar to the z-score:

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

The sum of weighted z-scores is considered to be of lesser importance than the individual z-scores. Therefore the organiser, in agreement with the EUPT-Panel, retained the right not to use them if they were considered to be unhelpful.

**Table 4.** No. of pesticides needed to be detected to have sufficient scope.

No. of Pesticides Present in the Sample (N)	90%	No. of Pesticides needed to be detected 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	18.0	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	



### 3. RESULTS

#### 3.1 Participation

In total, 127 EU laboratories, representing 29 countries (27 EU member states), agreed to participate in this proficiency test. All participants submitted data but five had such a limited number of pesticides in their scope that they could not report results for many of the pesticides present in the test item. The participating laboratories are listed in **Appendix 1**.

#### 3.2 Overview of results

An overview of the number of results submitted by laboratories from the EU and EFTA can be seen in **Table 8**. All reported analytical results can be seen in **Table 11a-11h**, and the methods used are published as **Appendix 6** but in a separate electronic file. However, only results submitted by laboratories from EU and EFTA countries are included in **Table 5-10 and 14**. Likewise only EU and EFTA countries are included in the z-scores histograms in **Appendix 5**.

Azoxystrobin, boscalid, chlorpyrifos, cypermethrin, cyprodinil, pendimethalin, pirimicarb, propiconazole and tebuconazole were the most frequently analysed compounds with 80 % or more of the labs submitting results for these compounds.

##### 3.2.1 False positives

Two participants from EU and EFTA countries reported four results above the MRRL for additional pesticides that had not been used to treat the Test Item (see **Table 5**). In all cases the compounds were not detected either by the Organizer, or by the overwhelming majority of the other participating laboratories. The reported results were therefore considered to be false positives.

One laboratory reported two results below the MRRL for additional pesticides (see **Table 6**). These results were not considered to be false positives. However, all three results were above the laboratories reporting limits.

##### 3.2.2 False negatives

Missing 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. Twenty results were judged as false negatives, which represents 1 % of the total number of pesticide results. Around 10 % of the participants (15 laboratories) reported false negative results. This is a significant decrease in the number of false negatives from EUPT-C4, where 61 were reported by 30% of the participants and from EUPT-C5 where 41 false negative results were reported by 20% of the participants. Several of the laboratories stated after the deadline for submitting results that they hadn't actually analysed for the pesticides. Nevertheless, these results, which in this case were due to administrative, rather than, analytical errors, have still been evaluated as false negative results. Some laboratories did not submit any quantitative results, but claimed to have qualitatively identified the compound. These have also been judged to be false negatives, because the PT is a quantitative exercise and numerical figures must be reported. This will be clarified in the next edition of the general protocol. No false negatives results were seen for chlorpyrifos, cyprodinil, epoxiconazole, tebuconazole, carbendazim, diflubenzuron and pendimethalin.

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

Laboratory code	Pesticide	Concentration, mg/kg	Determination Technique	RL, mg/kg	MRRL, mg/kg
75	Methacrifos	0.074	LC-MS/MS	0.01	0.01
131	Pyrimethanil	0.629	Not reported	0.08	0.01

**Table 6** False positive results below 0.01 mg/kg, the concentration level detected in mg/kg, determination technique, reporting level and MRRL in mg/kg.

Laboratory code	Pesticide	Concentration, mg/kg	Determination Technique	RL, mg/kg	MRRL, mg/kg
150	Bifenthrin	0.009	LC-MS/MS	0.008	0.01
150	Quinoxifen	0.005	LC-MS/MS	0.002	0.01

**Table 7** False negative results.

Lab code	Azoxystrobin	Boscalid	Carboxin	Chlorpropham	Fenpropidin	Isoprothiolane	Pendimethalin	Pirimicarb	Propiconazole	Prothioconazole -desthio	Pyraclostrobin
1						FN					
26				FN							
29			FN								
54	FN										FN
58		FN									
63							FN				
69									FN		
76								FN			
109									FN		
130		FN								FN	
131					FN	FN					
136								FN			
141	FN				FN						FN
148			FN		FN				FN		
151									FN		

**Table 8** 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.

Pesticides	No. of results	No. of NA <sup>1)</sup>	No. of FN <sup>2)</sup>	% results <sup>3)</sup>
<b>Azoxystrobin</b>	115	12	2	91
<b>Boscalid</b>	108	19	2	85
<b>Carbendazim</b>	92	35		72
<b>Carboxin</b>	73	54	2	57
<b>Chlorpropham</b>	100	27	1	79
<b>Chlorpyrifos</b>	121	6		95
<b>Cypermethrin</b>	113	14		89
<b>Cyprodinil</b>	111	16		87
<b>Diflubenzuron</b>	74	53		58
<b>Epoxiconazole</b>	99	28		78
<b>Fenpropidin</b>	76	51	3	60
<b>Isoprothiolane</b>	70	57	2	55
<b>Pendimethalin</b>	102	25		80
<b>Pirimicarb</b>	102	25	1	80
<b>Propiconazole</b>	114	13	2	90
<b>Prothioconazole-desthio</b>	64	63	4	50
<b>Pyraclostrobin</b>	91	36	1	72
<b>Tebuconazole</b>	116	11		91

1) NA = not analysed

2) FN = not detected (false negatives)

3) '% results' have been calculated using the number of laboratories that reported results for that particular compounds and the total number of EU laboratories submitting results (n = 133). False negatives are included in reported results.

### 3.3 Assigned values and target standard deviations

To establish the Assigned Values, the medians of the reported results submitted by laboratories from EU and EFTA countries, excluding outliers, were calculated. However, due to significantly biased results from laboratories not adding water to the sample before extraction (or adding water to the extraction solvent), these results were not included in the calculation of the median. Also results from laboratories that did not provide information about their extraction method were excluded from the calculations.

Seventeen results were regarded as outliers (z-score  $\geq 5$ ); carbendazim (0.604 mg/kg), carboxin (0.503, 0.512 and 0.517 mg/kg), chlorpropham (0.550 mg/kg), chlorpyrifos (0.410 and 1.278 mg/kg), cypermethrin (1.252 and 1.433 mg/kg), diflubenzuron (0.299 and 0.526mg/kg), fenpropidin (2.25 mg/kg), pendimethalin (0.270 mg/kg), propiconazole (0.525 mg/kg), prothioconazole-desthio (0.202 mg/kg), pyraclostrobin (1.35 mg/kg) and tebuconazole (1.66 mg/kg).

All Assigned Values for the pesticides can be seen in **Table 9**. The target standard deviation was obtained using a fixed FFP-RSD value of 25 %. In parallel, the robust standard deviation (Qn-RSD) was calculated for informative purposes only. The range of Qn-RSD values was from 16-29 % but on average the Qn-RSD was 22 %, and thus close to the 25 % FFP-RSD used for the calculations.

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

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

Where  $s^*$  is the robust standard deviation estimate and  $n$  is the number of datapoint equal to the number of results used to calculate the assigned value (number of results in **Table 8**)

**Table 9** Assigned values and the Uncertainty in mg/kg and Fit For Purpose Relative Standard Deviation (FFP RSD) and Robust Relative Standard Deviation (Qn RSD) for the pesticides present in the Test Item.

Pesticides	MRRL (mg/kg)	Assigned value (mg/kg)	Uncertainty, mg/kg	FFP RSD (%)	Qn RSD (%)
Azoxystrobin	0.01	0.196	0.006	25	28
Boscalid	0.01	0.910	0.024	25	23
Carbendazim	0.01	0.225	0.008	25	28
Carboxin	0.01	0.144	0.005	25	25
Chlorpropham	0.01	0.239	0.007	25	23
Chlorpyrifos	0.01	0.171	0.005	25	23
Cypermethrin	0.01	0.285	0.009	25	25
Cyprodinil	0.01	0.150	0.003	25	19
Diflubenzuron	0.01	0.128	0.005	25	28
Epoxiconazole	0.01	0.594	0.017	25	23
Fenpropidin	0.01	0.943	0.031	25	23
Isoprothiolane	0.01	0.083	0.002	25	18
Pendimethalin	0.01	0.108	0.002	25	18
Pirimicarb	0.01	0.252	0.006	25	20
Propiconazole	0.01	0.206	0.006	25	24
Prothioconazole-desthio	0.01	0.088	0.002	25	17
Pyraclostrobin	0.01	0.473	0.015	25	25
Tebuconazole	0.01	0.431	0.010	25	21

### 3.4 Assessment of laboratory performance

#### 3.4.1 z-Scores

Z-scores have been calculated for all the quantified pesticides using the FFP RSD of 25 %. **Table 10** shows an overview of the acceptable, questionable and unacceptable z-scores and **Tables 11a-11h** shows the individual results and z-scores for each laboratory and pesticide together with the assigned values. A graphical representation of the z-scores (for EU and EFTA countries) can be seen in **Appendix 5**.

**Table 10** Number of acceptable, questionable, unacceptable z-scores and false negatives. The unacceptable z-scores include the false negatives.

Pesticides	Acceptable z-scores	Questionable z-scores	Unacceptable z-scores	False negatives
Azoxystrobin	110	1	4	2
Boscalid	97	5	6	2
Carbendazim	78	8	6	
Carboxin	62	2	9	2
Chlorpropham	91	6	3	1
Chlorpyrifos	112	4	5	
Cypermethrin	104	6	3	
Cyprodinil	102	5	4	
Diflubenzuron	62	6	6	
Epoxiconazole	89	9	1	
Fenpropidin	64	7	5	3
Isoprothiolane	66	2	2	2
Pendimethalin	99	1	2	
Pirimicarb	93	5	4	1
Propiconazole	102	7	5	2
Prothioconazole-desthio	57	2	5	4
Pyraclostrobin	81	5	5	1
Tebuconazole	110	3	3	

For azoxystrobin, boscalid, chlorpropham, chlorpyrifos, cypermethrin, cyprodinil, epoxiconazole, isoprothiolane, pendimethalin, pirimicarb and tebuconazole acceptable results were obtained for 90-97% of the laboratories. For carbendazim, carboxin, diflubenzuron, fenpropidin, propiconazole, prothioconazole-desthio and pyraclostrobin acceptable results were obtained by 84-89% of the laboratories.

**Table 11a** Results in mg/kg and z-scores for azoxystrobin, boscalid, carbendazim, carboxin, chlorpropham, chlorpyrifos cypermethrin, cyprodinil and diflubenzuron.

Laboratory code	Azoxystrobin	Boscalid		Carbendazim		Carboxin		Chlorpropham		Chlorpyrifos		Cypermethrin		Cyprodinil		Diflubenzuron		
	MRRL	Z-scores (FFP RSD (25%))																
Assigned value	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01		
	0.196	0.910	0.225	0.144	0.239	0.173	0.284	0.150	0.128									
1	0.143	-1.1	0.940	0.1	0.172	-0.9	0.126	-0.5	0.214	-0.4	0.148	-0.6	0.258	-0.4	0.120	-0.8	0.191	2.0
2									0.138	-1.7	0.138	-0.8						
3	0.142	-1.1	0.747	-0.7	0.191	-0.6	0.135	-0.2	0.190	-0.8	0.134	-0.9	0.277	-0.1	0.137	-0.3	0.095	-1.0
4	0.269	1.5	1.17	1.1	0.229	0.1	0.150	0.2	0.294	0.9	0.190	0.4	0.244	-0.6	0.181	0.8	0.124	-0.1
5	0.0852	-2.3	0.828	-0.4	0.220	-0.1	0.0946	-1.4	0.221	-0.3	0.180	0.2	0.318	0.5	0.135	-0.4	0.135	0.2
6	0.224	0.6	0.204	-3.1	0.0968	-2.3					0.171	0.0	0.161	-1.7	0.157	0.2		
7	0.186	-0.2	1.003	0.4	0.214	-0.2	0.517	>5	0.195	-0.7	0.134	-0.9	0.476	2.7	0.113	-1.0		
8	0.172	-0.5	0.703	-0.9	0.233	0.1	0.151	0.2	0.238	0.0	0.185	0.3	0.315	0.4	0.153	0.1	0.137	0.3
9	0.269	1.5	1.14	1.0	0.292	1.2	0.184	1.1	0.314	1.3	0.183	0.2	FN	-3.9	0.196	1.2	FN	-3.7
10	0.143	-1.1	0.751	-0.7	0.203	-0.4	0.082	-1.7	0.232	-0.1	0.161	-0.3	0.253	-0.4	0.139	-0.3	0.106	-0.7
11	0.143	-1.1	0.721	-0.8	0.190	-0.6	0.125	-0.5	0.234	-0.1	0.103	-1.6	0.242	-0.6	0.132	-0.5	0.110	-0.6
12	0.196	0.0	0.989	0.3	0.317	1.6	0.172	0.8	0.280	0.7	0.197	0.6	0.351	0.9	0.174	0.6	0.146	0.6
13	0.218	0.4	1.11	0.9	0.141	-1.5					0.166	-0.2			0.0898	-1.6	0.138	0.3
14	0.221	0.5	1.005	0.4	0.218	-0.1	0.194	1.4	0.253	0.2	0.135	-0.9	0.280	-0.1	0.163	0.3	0.127	0.0
15											0.164	-0.2						
16	0.127	-1.4	0.493	-1.8	0.142	-1.5	0.109	-1.0	0.223	-0.3	0.119	-1.2	0.259	-0.4	0.0745	-2.0	0.0981	-0.9
17	0.197	0.0	0.94	0.1	0.229	0.1	0.181	1.0	0.243	0.1	0.166	-0.2	0.267	-0.2	0.179	0.8	0.124	-0.1
18																		
19	0.065	-2.7	0.401	-2.2	0.164	-1.1	0.049	-2.6	0.104	-2.3	0.107	-1.5	0.303	0.3	0.133	-0.5	0.110	-0.6
20					0.260	0.6			0.250	0.2	0.160	-0.3	0.236	-0.7	0.146	-0.1	0.134	0.2
21	0.131	-1.3	0.815	-0.4	0.296	1.3	0.182	1.1	0.223	-0.3	0.175	0.0	0.242	-0.6	0.136	-0.4	FN	-3.7
22	0.238	0.9	0.997	0.4	0.318	1.7	0.325	5.0	0.266	0.5	0.144	-0.7	0.315	0.4	0.180	0.8	0.130	0.1
23	0.210	0.3	0.822	-0.4	0.221	-0.1			0.275	0.6	0.190	0.4	0.274	-0.1	0.140	-0.3		
24	0.201	0.1									0.150	-0.5	0.336	0.7				
25	0.24	0.9	1.12	0.9	0.24	0.3	0.18	1.0	0.091	-2.5	0.068	-2.4			0.20	1.3	0.12	-0.3
26	0.295	2.0	1.931	4.5	0.364	2.5			FN	-3.8	0.339	3.8	0.402	1.7	0.317	4.5		
27	0.107	-1.8	0.822	-0.4	0.160	-1.2	0.120	-0.7	0.197	-0.7	0.125	-1.1	0.284	0.0	0.118	-0.9	0.094	-1.1
28	0.171	-0.5	0.907	0.0	0.243	0.3	0.094	-1.4	0.230	-0.2	0.170	-0.1	0.263	-0.3	0.152	0.1	0.299	>5
29	0.0588	-2.8	0.372	-2.4	0.0395	-3.3	FN	-3.7	0.139	-1.7	0.193	0.5	0.336	0.7	0.125	-0.7		
30	0.216	0.4							0.270	0.5	0.241	1.6	0.474	2.7				
31	0.196	0.0	0.847	-0.3	0.234	0.2	0.153	0.3	0.207	-0.5	0.151	-0.5	0.254	-0.4	0.136	-0.4	0.125	-0.1
32	0.107	-1.8	0.631	-1.2	0.149	-1.4	0.103	-1.1	0.184	-0.9	0.172	0.0	0.295	0.2	0.115	-0.9	0.125	-0.1
34	0.215	0.4	1.03	0.5	0.209	-0.3	0.149	0.1	0.238	0.0	0.173	0.0	0.309	0.4	0.147	-0.1	0.115	-0.4
35	0.234	0.8	0.570	-1.5	0.314	1.6					0.171	0.0	0.100	-2.6	0.132	-0.5		
36	0.0656	-2.7	0.248	-2.9	FN	-3.8	0.0414	-2.9	0.0878	-2.5	0.0628	-2.5	0.514	3.2	0.0535	-2.6	FN	-3.7
37	0.152	-0.9	0.813	-0.4	0.191	-0.6	0.132	-0.3	0.241	0.0	0.156	-0.4	0.400	1.6	0.146	-0.1	0.126	-0.1
39	0.198	0.0	0.893	-0.1	0.192	-0.6	0.136	-0.2	0.357	2.0	0.117	-1.3	0.144	-2.0	0.114	-1.0	0.116	-0.4
40	0.329	2.7							0.251	0.2	0.312	3.2	0.366	1.2	0.128	-0.6		

**Table 11b** Results in mg/kg and z-scores for epoxiconazole, fenpropidin, isoprothiolane, pendimethalin, pirimicarb, propiconazole, prothioconazole-desthio, pyraclostrobin and tebuconazole.

Laboratory code	Epoxiconazole	Fenpropidin		Isoprothiolane		Pendimethalin		Pirimicarb		Propiconazole		Prothioconazole-desthio		Pyraclostrobin		Tebuconazole		
MRRL	0.01	0.01		0.01		0.01		0.01		0.01		0.01		0.01		0.01		
Assigned value	0.576	0.927		0.082		0.108		0.251		0.202		0.087		0.465		0.423		
		Z-scores (FFP RSD (25%))		Z-scores (FFP RSD (25%))		Z-scores (FFP RSD (25%))		Z-scores (FFP RSD (25%))		Z-scores (FFP RSD (25%))		Z-scores (FFP RSD (25%))		Z-scores (FFP RSD (25%))		Z-scores (FFP RSD (25%))		
1	0.549	-0.3	0.966	0.1	FN	-3.5	0.100	-0.3	0.203	-0.8	0.150	-1.1	0.078	-0.4	0.465	-0.1	0.348	-0.8
2																		
3	0.377	-1.5	0.716	-1.0	0.077	-0.3	0.074	-1.3	0.223	-0.5	0.157	-0.9	0.059	-1.3	0.335	-1.2	0.330	-0.9
4	0.749	1.0	1.032	0.4	0.0831	0.0	0.115	0.3	0.312	1.0	0.254	0.9	0.081	-0.3	0.413	-0.5	0.549	1.1
5	0.434	-1.1	0.320	-2.6	0.0833	0.0	0.132	0.9	0.177	-1.2	0.163	-0.8			0.430	-0.4	0.323	-1.0
6	0.154	-3.0					0.110	0.1	0.219	-0.5	0.259	1.0			0.120	-3.0	0.481	0.5
7	0.432	-1.1	0.855	-0.4			0.0947	-0.5	0.174	-1.2	0.131	-1.5	0.0832	-0.2	0.490	0.1	0.409	-0.2
8	0.604	0.1	0.830	-0.5	0.0873	0.2	0.101	-0.3	0.193	-0.9	0.174	-0.6	0.0799	-0.4	0.520	0.4	0.519	0.8
9	0.690	0.6			0.105	1.1	0.132	0.9	0.512	4.1	0.244	0.7			0.473	0.0	0.635	1.9
10	0.493	-0.7	0.730	-0.9	0.065	-0.9	0.108	0.0	0.252	0.0	0.169	-0.7	0.072	-0.7	0.305	-1.4	0.387	-0.4
11	0.456	-0.9	0.512	-1.8	0.070	-0.6	0.092	-0.6	0.203	-0.8	0.161	-0.9	0.065	-1.0	0.421	-0.4	0.342	-0.8
12	0.642	0.3	1.08	0.6	0.0886	0.3	0.121	0.5	0.278	0.4	0.210	0.1	0.0938	0.3	0.485	0.1	0.410	-0.2
13							0.117	0.3	0.282	0.5	0.228	0.4			0.383	-0.8	0.444	0.1
14	0.642	0.3	1.118	0.7	0.094	0.5	0.090	-0.7	0.273	0.3	0.147	-1.1	0.084	-0.2	0.476	0.0	0.500	0.6
15																		
16	0.310	-1.9					0.103	-0.2	0.222	-0.5	0.163	-0.8			0.195	-2.3	0.305	-1.2
17	0.545	-0.3	0.907	-0.2			0.090	-0.7	0.240	-0.2	0.216	0.2	0.083	-0.2	0.382	-0.8	0.413	-0.2
18																		
19	0.249	-2.3	0.269	-2.9	0.060	-1.1	0.137	1.1	0.055	-3.1	0.097	-2.1	0.035	-2.4	0.279	-1.6	0.220	-2.0
20									0.230	-0.3	0.240	0.7			0.554	0.7	0.410	-0.2
21	0.485	-0.7	0.828	-0.5	0.091	0.4	0.104	-0.1	0.299	0.7	0.176	-0.6			0.370	-0.9	0.320	-1.0
22	0.434	-1.1	1.028	0.4	0.066	-0.8	0.114	0.2	0.252	0.0	0.253	0.9	0.0828	-0.2	0.650	1.5	0.511	0.7
23	0.576	-0.1					0.153	1.7	0.230	-0.3	0.215	0.2			0.472	0.0	0.399	-0.3
24									0.261	0.1	0.259	1.0					0.508	0.7
25	0.68	0.6	1.06	0.5	0.093	0.5	0.16	1.9	0.16	-1.5	0.24	0.7			0.89	3.5	0.60	1.6
26							0.213	3.9	0.482	3.7	0.525	>5			0.448	-0.2	0.212	-2.0
27	0.275	-2.1	0.396	-2.3	0.104	1.0	0.114	0.2	0.210	-0.7	0.125	-1.6			0.265	-1.8	0.236	-1.8
28	0.476	-0.8	0.922	-0.1	0.078	-0.2	0.108	0.0	0.235	-0.3	0.190	-0.3	0.077	-0.5	0.413	-0.5	0.366	-0.6
29	0.172	-2.8	0.208	-3.1			0.107	0.0	0.0655	-3.0	0.0822	-2.4			0.259	-1.8	0.248	-1.7
30							0.146	1.4	0.319	1.1	0.248	0.8					0.512	0.8
31	0.613	0.1	1.24	1.3	0.0767	-0.3	0.0715	-1.4	0.256	0.1	0.198	-0.1	0.0925	0.2	0.536	0.5	0.394	-0.3
32	0.381	-1.4	0.721	-0.9	0.063	-1.0	0.098	-0.4	0.218	-0.5	0.142	-1.2	0.043	-2.0	0.305	-1.4	0.288	-1.3
34	0.644	0.3	1.08	0.6	0.0820	0.0	0.117	0.3	0.249	0.0	0.228	0.4	0.105	0.8	0.536	0.5	0.446	0.1
35							0.116	0.3	0.283	0.5	0.250	0.9			0.408	-0.5	0.349	-0.8
36	0.172	-2.8			0.0294	-2.6	FN	-3.6	0.0837	-2.7	0.148	-1.1			0.160	-2.6	0.129	-2.8
37	0.542	-0.4	0.931	-0.1	0.081	-0.1	0.099	-0.3	0.236	-0.3	0.183	-0.4			0.388	-0.7	0.392	-0.4
39	0.712	0.8	0.887	-0.2	0.070	-0.6	0.072	-1.3	0.256	0.1	0.246	0.8			0.458	-0.1	0.365	-0.6
40									0.0754	-2.8	0.462	5.0					0.423	-0.1

**Table 11c** Results in mg/kg and z-scores for azoxystrobin, boscalid, carbendazim, carboxin, chlorpropham, chlorpyrifos, cypermethrin, cyprodinil and diflubenzuron.

Laboratory code	Azoxystrobin	Boscalid		Carbendazim		Carboxin		Chlorpropham		Chlorpyrifos		Cypermethrin		Cyprodinil		Diflubenzuron		
	MRRL	Z-scores (FFP RSD (25%))																
	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01		
Assigned value	0.196	0.910	0.225	0.144	0.239	0.173	0.284	0.150	0.128									
41	0.213	0.3	1.02	0.5	0.255	0.5	0.118	-0.7	0.257	0.3	0.169	-0.1	0.263	-0.3	0.150	0.0	0.131	0.1
42	0.13	-1.3	0.72	-0.8	0.19	-0.6			0.219	-0.3	0.17	-0.1	0.19	-1.3	0.125	-0.7		
43	0.206	0.2	1.01	0.4	0.215	-0.2	0.160	0.4	0.263	0.4	0.194	0.5	0.316	0.5	0.175	0.7	0.128	0.0
44	0.216	0.4	1.371	2.0	0.136	-1.6	0.2795	3.8			0.232	1.4	0.356	1.0	0.168	0.5	0.144	0.5
45	0.198	0.0	0.891	-0.1	0.262	0.7	0.175	0.9	0.233	-0.1	0.174	0.0	1.433	16.2	0.159	0.2	0.210	2.6
46	0.277	1.7	0.716	-0.9	0.416	3.4	0.212	1.9	0.184	-0.9	0.139	-0.8	0.252	-0.5	0.226	2.0	0.282	4.8
47	0.266	1.4	1.138	1.0	0.268	0.8	0.174	0.8	0.203	-0.6	0.163	-0.2	0.260	-0.3	0.169	0.5	0.202	2.3
49	0.231	0.7	1.14	1.0	0.242	0.3	0.187	1.2	0.221	-0.3	0.196	0.5	0.253	-0.4	0.163	0.3	0.146	0.6
50	0.134	-1.3	0.661	-1.1	0.206	-0.3	0.136	-0.2	0.336	1.6	0.306	3.1	0.227	-0.8	0.140	-0.3	0.111	-0.5
51	0.210	0.3	1.056	0.6	0.327	1.8	0.172	0.8	0.258	0.3	0.179	0.1	0.328	0.6	0.186	1.0	0.148	0.6
52	0.209	0.3	1.15	1.1	0.246	0.4			0.200	-0.7	0.169	-0.1	0.314	0.4	0.127	-0.6	0.121	-0.2
53	0.170	-0.5	0.818	-0.4	0.226	0.0	0.141	-0.1	0.230	-0.2	0.161	-0.3	0.324	0.6	0.150	0.0	0.120	-0.3
54	FN	-3.8	1.84	4.1					0.36	2.0	0.24	1.5	0.26	-0.3	0.24	2.4		
55																		
56	0.201	0.1	0.994	0.4	0.604	>5	0.262	3.3	0.225	-0.2	0.165	-0.2	0.259	-0.4	0.145	-0.1		
57	0.123	-1.5	0.583	-1.4	0.176	-0.9	0.098	-1.3	0.207	-0.5	0.147	-0.6	0.475	2.7	0.122	-0.7	0.132	0.1
58	0.16	-0.7	FN	-4.0	0.31	1.5					0.17	-0.1	0.30	0.2	0.14	-0.3	0.20	2.3
59	0.208	0.2	0.924	0.1	0.219	-0.1	0.165	0.6	0.232	-0.1	0.162	-0.3	0.309	0.4	0.150	0.0	0.127	0.0
60																		
61	0.217	0.4	0.997	0.4	0.449	4.0	0.204	1.7	0.271	0.5	0.199	0.6	0.354	1.0	0.177	0.7	0.160	1.0
62	0.202	0.1	1.18	1.2	0.242	0.3	0.148	0.1	0.267	0.5	0.188	0.3	0.310	0.4	0.146	-0.1	0.137	0.3
63	0.054	-2.9	0.458	-2.0	0.123	-1.8	0.059	-2.4	0.099	-2.3	0.106	-1.5	0.243	-0.6	0.123	-0.7	0.142	0.4
64	0.103	-1.9	0.581	-1.4	0.182	-0.8	0.145	0.0	0.160	-1.3	0.150	-0.5	0.258	-0.4	0.121	-0.8	0.106	-0.7
65	0.0940	-2.1	0.611	-1.3	0.222	-0.1	0.119	-0.7	0.193	-0.8	0.154	-0.4	0.295	0.2	0.137	-0.3	0.0767	-1.6
66	0.166	-0.6	0.855	-0.2	0.350	2.2	0.137	-0.2	0.288	0.8	0.212	0.9	0.296	0.2	0.188	1.0	0.125	-0.1
67	0.0389	-3.2			0.365	2.5			0.0557	-3.1	0.0470	-2.9						
68	0.19	-0.1	0.91	0.0	0.25	0.4	0.14	-0.1	0.27	0.5	0.17	-0.1	0.20	-1.2	0.15	0.0	0.14	0.4
69	0.145	-1.0	0.780	-0.6	0.113	-2.0	0.059	-2.4	0.265	0.4	0.160	-0.3	0.150	-1.9	0.131	-0.5	0.060	-2.1
70	0.26	1.3	0.88	-0.1	0.25	0.4			0.19	-0.8	0.14	-0.8	0.21	-1.0	0.12	-0.8	0.15	0.7
71	0.196	0.0	1.068	0.7	0.214	-0.2	0.166	0.6	0.210	-0.5	0.191	0.4	0.274	-0.1	0.163	0.3	0.147	0.6
72	0.162	-0.7	0.756	-0.7	0.278	0.9	0.151	0.2	0.231	-0.1	0.166	-0.2	0.377	1.3	0.134	-0.4	0.156	0.9
73	0.109	-1.8	0.657	-1.1					0.188	-0.9	0.162	-0.3	0.275	-0.1	0.120	-0.8		
74	0.170	-0.5	0.695	-0.9							0.223	1.2	0.230	-0.8	0.19	1.1		
75	0.164	-0.7	1.01	0.4	0.175	-0.9	0.153	0.3	0.243	0.1	0.133	-0.9	0.176	-1.5	0.092	-1.5	0.077	-1.6
76	0.226	0.6	1.021	0.5	0.230	0.1					0.18	0.2	0.211	-1.0	0.154	0.1		
77									0.138	-1.7	0.145	-0.6						
78											0.152	-0.5						
79							0.199	1.5			0.228	1.3			0.170	0.5		

**Table 11d** Results in mg/kg and z-scores for epoxiconazole, fenpropidin, isoprothiolane, pendimethalin, pirimicarb, propiconazole, prothioconazole-desthio, pyraclostrobin and tebuconazole.

Laboratory code	Epoxiconazole	Fenpropidin		Isoprothiolane		Pendimethalin		Pirimicarb		Propiconazole		Prothioconazole-desthio		Pyraclostrobin		Tebuconazole		
MRRL	0.01	0.01		0.01		0.01		0.01		0.01		0.01		0.01		0.01		
Assigned value	0.576	0.927		0.082		0.108		0.251		0.202		0.087		0.465		0.423		
	Z-scores (FFP RSD (25%))																	
41	0.670	0.5	1.06	0.5	0.087	0.2	0.110	0.1	0.260	0.1	0.240	0.7	0.202	>5	0.510	0.3	0.467	0.3
42	0.52	-0.5					0.11	0.1	0.19	-1.0	0.21	0.1			0.45	-0.2	0.42	-0.1
43	0.565	-0.2	1.028	0.4	0.0992	0.8	0.130	0.8	0.276	0.4	0.199	-0.1	0.0872	0.0	0.528	0.5	0.418	-0.1
44	0.648	0.4	0.629	-1.3	0.084	0.0	0.135	1.0	0.328	1.2	0.105	-2.0			0.328	-1.2	0.451	0.2
45	0.198	-2.7	1.065	0.5	0.091	0.4	0.102	-0.2	0.258	0.1	0.350	2.8	0.098	0.5	0.485	0.1	0.483	0.5
46	0.676	0.6	0.484	-1.9	0.116	1.6	0.132	0.9	0.373	1.9	0.177	-0.6	0.110	1.0	0.495	0.2	0.307	-1.2
47	0.734	0.9	1.40	1.9	0.098	0.7	0.106	-0.1	0.296	0.7	0.262	1.1	0.108	0.9	0.624	1.3	0.542	1.0
49	0.630	0.2	1.10	0.7	0.101	0.9	0.124	0.6	0.302	0.8	0.234	0.6	0.110	1.0	0.551	0.7	0.467	0.3
50	0.504	-0.6	0.915	-0.1	0.067	-0.8	0.105	-0.1	0.219	-0.5	0.196	-0.2	0.077	-0.5	0.355	-1.0	0.371	-0.6
51	0.654	0.4	1.035	0.4	0.0970	0.7	0.138	1.1	0.314	1.0	0.227	0.4	0.101	0.6	0.498	0.2	0.476	0.4
52	0.505	-0.6					0.129	0.8	0.218	-0.5	0.195	-0.2	0.092	0.2	0.465	-0.1	0.450	0.2
53	0.526	-0.5	0.831	-0.5	0.080	-0.1	0.102	-0.2	0.250	0.0	0.180	-0.5	0.078	-0.4	0.388	-0.7	0.377	-0.5
54											0.18	-0.5			1.35	>5	0.23	-1.9
55																		
56	0.638	0.3	2.25	>5	0.0740	-0.4	0.0910	-0.6	0.227	-0.4	0.226	0.4	0.0883	0.0	0.648	1.5	0.460	0.3
57	0.351	-1.6	0.628	-1.3	0.077	-0.3	0.098	-0.4	0.151	-1.6	0.124	-1.6			0.242	-2.0	0.328	-1.0
58	0.39	-1.4					0.11	0.1	0.24	-0.2	0.19	-0.3			0.39	-0.7	0.44	0.1
59	0.584	-0.1	0.951	0.0	0.0818	-0.1	0.0991	-0.3	0.246	-0.1	0.214	0.2	0.0907	0.1	0.500	0.2	0.440	0.1
60																		
61	0.525	-0.5	0.949	0.0			0.093	-0.6	0.320	1.1	0.241	0.7	0.122	1.6	0.473	0.0	0.409	-0.2
62	0.568	-0.2	1.04	0.4	0.0980	0.7	0.127	0.7	0.235	-0.3	0.215	0.2	0.102	0.7	0.728	2.2	0.485	0.5
63	0.216	-2.5	0.309	-2.7	0.063	-1.0	0.068	-1.5	FN	-3.8	0.089	-2.3			0.208	-2.2	0.239	-1.8
64	0.352	-1.6	0.627	-1.3	0.063	-1.0	0.104	-0.1	0.202	-0.8	0.129	-1.5	0.056	-1.4	0.295	-1.5	0.292	-1.3
65	0.354	-1.6	0.706	-1.0	0.0594	-1.1	0.0944	-0.5	0.228	-0.4	0.126	-1.5	0.0509	-1.7	0.263	-1.8	0.340	-0.8
66	0.577	-0.1	0.764	-0.8	0.088	0.2	0.122	0.5	0.269	0.3	0.199	-0.1			0.434	-0.3	0.439	0.1
67	0.634	0.3					0.106	-0.1	0.267	0.2					0.338	-1.1	0.133	-2.8
68	0.56	-0.2	1.06	0.5	0.087	0.2	0.11	0.1	0.27	0.3	0.19	-0.3	0.090	0.1	0.46	-0.1	0.42	-0.1
69	0.473	-0.8	0.699	-1.0	0.036	-2.3	0.098	-0.4	0.218	-0.5	0.168	-0.7	FN	-3.5	0.308	-1.4	0.391	-0.4
70	0.56	-0.2	0.35	-2.5			0.11	0.1	0.22	-0.5	0.23	0.5	0.12	1.5	0.56	0.7	0.38	-0.5
71	0.625	0.2	1.108	0.7	0.086	0.1	0.115	0.3	0.222	-0.5	0.203	0.0	0.108	0.9	0.540	0.6	0.416	-0.1
72	0.459	-0.9	0.669	-1.2	0.099	0.8	0.103	-0.2	0.209	-0.7	0.168	-0.7	0.099	0.5	0.378	-0.8	0.385	-0.4
73	0.373	-1.5							0.170	-1.3	0.139	-1.3					0.291	-1.3
74	0.660	0.4									0.234	0.6					0.430	0.0
75	0.355	-1.6	0.424	-2.2	0.075	-0.4	0.084	-0.9	0.251	0.0	0.161	-0.9	0.102	0.7	0.525	0.4	0.312	-1.1
76	0.691	0.7	1.021	0.3			0.116	0.3	0.270	0.3	FN	-3.8			0.599	1.1	0.534	1.0
77																		
78																		
79					0.0990	0.8			0.660	>5								

**Table 11e** Results in mg/kg and z-scores for azoxystrobin, boscalid, carbendazim, carboxin, chlorpropham, chlorpyrifos cypermethrin, cyprodinil and diflubenzuron.

Laboratory code	Azoxystrobin	Boscalid		Carbendazim		Carboxin		Chlorpropham		Chlorpyrifos		Cypermethrin		Cyprodinil		Diflubenzuron		
	MRRL	Z-scores (FFP RSD (25%))																
	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01		
Assigned value	0.196	0.910	0.225	0.144	0.239	0.173	0.284	0.150	0.128									
80																		
81	0.183	-0.3	1.001	0.4	0.194	-0.6	0.126	-0.5	0.256	0.3	0.185	0.3	0.335	0.7	0.159	0.2	0.145	0.5
82	0.137	-1.2	0.787	-0.5	0.211	-0.2			0.226	-0.2	0.161	-0.3	1.252	13.6	0.147	-0.1	0.086	-1.3
83									0.145	-1.6	0.120	-1.2						
84	0.184	-0.2	0.972	0.3	0.307	1.5	0.150	0.2	0.225	-0.2	0.135	-0.9	0.299	0.2	0.139	-0.3	0.174	1.4
85	0.095	-2.1	0.465	-2.0	0.125	-1.8	0.08	-1.8	0.136	-1.7	0.099	-1.7	0.204	-1.1	0.112	-1.0		
86	0.218	0.4	0.921	0.0	0.316	1.6	0.168	0.7	0.273	0.6	0.181	0.2	0.315	0.4	0.157	0.2	0.234	3.3
87	0.205	0.2	1.01	0.4	0.186	-0.7	0.132	-0.3	0.258	0.3	0.166	-0.2	0.288	0.1	0.142	-0.2	0.135	0.2
88	0.11	-1.8	0.68	-1.0							0.15	-0.5	0.17	-1.6				
89									0.19	-0.8	0.16	-0.3			0.10	-1.3		
90	0.174	-0.4	0.766	-0.6	0.208	-0.3	0.148	0.1	0.310	1.2	0.295	2.8	0.320	0.5	0.185	0.9	0.122	-0.2
91	0.171	-0.5	0.720	-0.8	0.250	0.4	0.126	-0.5	0.240	0.0	0.177	0.1	0.235	-0.7	0.099	-1.4	FN	-3.7
92	0.215	0.4	1.01	0.4	0.213	-0.2	0.139	-0.1	0.269	0.5	0.197	0.6	0.338	0.8	0.161	0.3	0.153	0.8
93	0.180	-0.3	0.845	-0.3	0.210	-0.3	0.143	0.0	0.235	-0.1	0.211	0.9	0.284	0.0	0.150	0.0	0.125	-0.1
94	0.0837	-2.3	0.500	-1.8	0.260	0.6	0.139	-0.1	0.162	-1.3	0.140	-0.8	0.230	-0.8	0.125	-0.7	0.0971	-1.0
95	0.086	-2.2	0.584	-1.4	0.141	-1.5					0.102	-1.6	0.225	-0.8	0.097	-1.4		
96	0.123	-1.5	0.777	-0.6	0.141	-1.5			0.217	-0.4	0.141	-0.7	0.199	-1.2	0.140	-0.3		
97	0.180	-0.3	1.02	0.5	0.212	-0.2			0.225	-0.2	0.163	-0.2	0.268	-0.2	0.146	-0.1		
98	0.224	0.6	1.18	1.2	0.253	0.5	0.193	1.4	0.222	-0.3	0.171	0.0	0.305	0.3	0.137	-0.3	0.161	1.0
99	0.190	-0.1	0.807	-0.5	0.214	-0.2	0.136	-0.2	0.244	0.1	0.167	-0.1	0.270	-0.2	0.129	-0.6	0.112	-0.5
100	0.169	-0.6									0.214	0.9	0.240	-0.6	0.158	0.2		
101	0.171	-0.5	1.08	0.7	0.267	0.7					0.159	-0.3			0.166	0.4		
102	0.207	0.2	1.00	0.4			0.110	-0.9	0.263	0.4	0.181	0.2	0.337	0.7	0.156	0.2		
103	0.181	-0.3	1.05	0.6	0.173	-0.9	0.503	>5	0.246	0.1	0.247	1.7	0.405	1.7	0.166	0.4	0.526	>5
104	0.140	-1.1	0.770	-0.6	0.254	0.5	0.087	-1.6	0.309	1.2	0.156	-0.4	0.265	-0.3	0.174	0.6	0.118	-0.3
105	0.19	-0.1	0.69	-1.0					0.40	2.7	0.27	2.2	0.11	-2.5	0.31	4.3		
106																		
107	0.171	-0.5	0.856	-0.2							0.229	1.3			0.158	0.2		
108	0.171	-0.5	0.834	-0.3	0.064	-2.9	0.130	-0.4	0.323	1.4	0.190	0.4	0.329	0.6	0.138	-0.3	0.101	-0.8
109	0.243	1.0	1.13	1.0	0.323	1.7	0.182	1.1	0.313	1.2	0.192	0.4	0.286	0.0	0.177	0.7	0.229	3.2
110	0.179	-0.3	0.709	-0.9							0.220	1.1	0.216	-1.0	0.165	0.4		
111	0.27	1.5	1.0	0.4	0.21	-0.3			0.22	-0.3	0.21	0.9			0.14	-0.3		
112	0.168	-0.6									0.218	1.0			0.173	0.6		
113	0.163	-0.7	0.818	-0.4							0.245	1.7	0.352	1.0	0.182	0.9		
114	0.236	0.8	0.860	-0.2	0.269	0.8	0.140	-0.1	0.223	-0.3	0.160	-0.3	0.297	0.2	0.142	-0.2		
115	0.226	0.6	1.17	1.1					0.278	0.7	0.111	-1.4	0.255	-0.4	0.243	2.5		
116																		
117	0.223	0.6	1.123	0.9					0.288	0.8	0.189	0.4	0.481	2.8	0.120	-0.8		

**Table 11f** Results in mg/kg and z-scores for epoxiconazole, fenpropidin, isoprothiolane, pendimethalin, pirimicarb, propiconazole, prothioconazole-desthio, pyraclostrobin and tebuconazole.

Laboratory code	Epoxiconazole	Fenpropidin		Isoprothiolane		Pendimethalin		Pirimicarb		Propiconazole		Prothioconazole-desthio		Pyraclostrobin		Tebuconazole		
MRRL	0.01	0.01		0.01		0.01		0.01		0.01		0.01		0.01		0.01		
Assigned value	0.576	0.927		0.082		0.108		0.251		0.202		0.087		0.465		0.423		
		Z-scores (FFP RSD (25%))		Z-scores (FFP RSD (25%))		Z-scores (FFP RSD (25%))		Z-scores (FFP RSD (25%))		Z-scores (FFP RSD (25%))		Z-scores (FFP RSD (25%))		Z-scores (FFP RSD (25%))		Z-scores (FFP RSD (25%))		
80																		
81	0.613	0.1	1.06	0.5	0.0726	-0.5	0.119	0.4	0.259	0.1	0.202	-0.1	0.100	0.6	0.524	0.4	0.456	0.2
82	0.489	-0.7					0.110	0.1	0.193	-0.9	0.185	-0.4			0.244	-1.9	0.383	-0.4
83																	0.210	-2.1
84	0.605	0.1	1.085	0.6	0.058	-1.2	0.116	0.3	0.259	0.1	0.202	-0.1	0.082	-0.3	0.457	-0.1	0.431	0.0
85	0.276	-2.1	0.421	-2.2	0.071	-0.6	0.077	-1.1	0.161	-1.4	0.1	-2.1			0.277	-1.7	0.248	-1.7
86	0.607	0.1	0.947	0.0	0.080	-0.1	0.116	0.3	0.261	0.1	0.221	0.3	0.100	0.6	0.505	0.3	0.482	0.5
87	0.617	0.2	0.908	-0.1	0.083	0.0	0.107	0.0	0.235	-0.3	0.208	0.0	0.093	0.2	0.505	0.3	0.421	-0.1
88	0.37	-1.5					0.10	-0.3	0.17	-1.3	0.15	-1.1					0.3	-1.2
89							0.12	0.4			0.13	-1.5						
90	0.495	-0.7	1.03	0.4	0.075	-0.4	0.115	0.3	0.260	0.1	0.165	-0.8	0.077	-0.5	0.364	-0.9	0.435	0.0
91	0.521	-0.5	0.434	-2.2			0.070	-1.4	0.210	-0.7	0.184	-0.4			0.800	2.8	0.391	-0.4
92	0.613	0.1	0.908	-0.1	0.0754	-0.4	0.113	0.2	0.271	0.3	0.226	0.4	0.0863	-0.1	0.469	0.0	0.430	0.0
93	0.479	-0.8	0.821	-0.5	0.070	-0.6	0.110	0.1	0.270	0.3	0.180	-0.5	0.077	-0.5	0.350	-1.0	0.353	-0.7
94	0.283	-2.1	0.586	-1.5	0.0512	-1.5	0.0815	-1.0	0.243	-0.1	0.111	-1.8	0.0418	-2.1	0.225	-2.1	0.234	-1.8
95							0.061	-1.7	0.128	-2.0	0.095	-2.2					0.360	-0.7
96							0.103	-0.2	0.212	-0.6	0.086	-2.3					0.396	-0.3
97	0.646	0.4					0.0910	-0.6	0.242	-0.2	0.214	0.2	0.0892	0.1	0.438	-0.3	0.381	-0.5
98	0.665	0.5	1.12	0.8	0.0940	0.5	0.115	0.3	0.287	0.6	0.260	1.1	0.0894	0.1	0.491	0.2	0.592	1.5
99	0.557	-0.2					0.102	-0.2	0.208	-0.7	0.192	-0.3			0.513	0.3	0.399	-0.3
100											0.218	0.2					0.431	0.0
101							0.109	0.0							0.405	-0.6	0.462	0.3
102							0.119	0.4	0.258	0.1	0.223	0.3					0.446	0.1
103	0.548	-0.3	0.685	-1.1			0.136	1.0	0.280	0.4	0.263	1.1			0.103	-3.1	0.548	1.1
104	0.550	-0.3	0.704	-1.0	0.063	-1.0	0.096	-0.4	0.254	0.0	0.198	-0.1	0.079	-0.4	0.601	1.1	0.362	-0.6
105	0.61	0.1					0.10	-0.3	0.43	2.8	0.23	0.5			0.48	0.1	0.49	0.5
106																		
107	0.684	0.6									0.257	1.0					0.417	-0.1
108	0.608	0.1	1.02	0.3	0.074	-0.4	0.107	0.0	0.302	0.8	0.231	0.5	0.093	0.2	0.786	2.7	0.541	1.0
109	0.715	0.8	1.12	0.8	0.090	0.3	0.102	-0.2	0.311	0.9	0.227	0.4	FN	-3.5	0.632	1.4	0.485	0.5
110	0.660	0.4									0.205	0.0					0.391	-0.4
111					0.070	-0.6			0.28	0.4							0.53	0.9
112	0.710	0.8									0.221	0.3					0.453	0.2
113	0.717	0.8									0.243	0.7					0.499	0.6
114	0.588	0.0	0.939	0.0	0.101	0.9	0.105	-0.1	0.182	-1.1	0.199	-0.1	0.101	0.6	0.542	0.6	0.396	-0.3
115	0.648	0.4					0.063	-1.7	0.255	0.0	0.262	1.1	0.095	0.3			1.66	>5
116																		
117											0.206	0.0					0.519	0.8

**Table 11g** Results in mg/kg and z-scores for azoxystrobin, boscalid, carbendazim, carboxin, chlorpropham, chlorpyrifos, cypermethrin, cyprodinil and diflubenzuron.

Laboratory code	Azoxystrobin	Boscalid		Carbendazim		Carboxin		Chlorpropham		Chlorpyrifos		Cypermethrin		Cyprodinil		Diflubenzuron		
MRRL	0.01	0.01		0.01		0.01		0.01		0.01		0.01		0.01		0.01		
Assigned value	0.196	0.910		0.225		0.144		0.239		0.173		0.284		0.150		0.128		
	Z-scores (FFP RSD (25%))																	
118										0.233	1.4	0.148	-1.9					
119	0.212	0.3	0.957	0.2	0.228	0.1	0.167	0.6	0.279	0.7	0.158	-0.3	0.339	0.8	0.113	-1.0	0.110	-0.6
120			0.341	-2.5							0.093	-1.8	0.198	-1.2				
122	0.174	-0.4	0.848	-0.3	0.224	0.0	0.125	-0.5	0.239	0.0	0.149	-0.6	0.320	0.5	0.149	0.0	0.114	-0.4
123	0.201	0.1	0.870	-0.2	0.270	0.8	0.512	>5	0.284	0.8	0.181	0.2	0.269	-0.2	0.166	0.4	0.204	2.4
124	0.287	1.9							0.118	-2.0	0.169	-0.1	0.393	1.5	0.073	-2.1		
125	0.183	-0.3	0.917	0.0	0.190	-0.6	0.158	0.4	0.225	-0.2	0.198	0.6	0.203	-1.1	0.147	-0.1	0.106	-0.7
126																		
127											0.1346	-0.9						
128	0.201	0.1	1.07	0.7	0.339	2.0	0.148	0.1	0.287	0.8	0.208	0.8	0.260	-0.3	0.167	0.5	0.139	0.3
129	0.23	0.7	1.33	1.8	0.24	0.3			0.55	>5	0.41	>5	0.61	>5	0.29	3.7		
130	0.221	0.5	FN	-4.0					0.171	-1.1	0.208	0.8	0.234	-0.7	0.114	-1.0	0.0445	-2.6
131	0.321	2.6	0.860	-0.2					0.294	0.9	1.278	25.5	0.308	0.3	0.151	0.0		
132	0.168	-0.6	0.688	-1.0	0.208	-0.3	0.141	-0.1	0.153	-1.4	0.152	-0.5	0.297	0.2	0.138	-0.3	0.148	0.6
133	0.20	0.1	0.75	-0.7							0.20	0.6	0.25	-0.5	0.12	-0.8		
135	0.170	-0.5	1.08	0.7	0.08	-2.6	0.112	-0.9	0.165	-1.2	0.159	-0.3	0.261	-0.3	0.114	-1.0	0.068	-1.9
136	0.249	1.1	1.07	0.7	0.266	0.7	0.177	0.9	0.282	0.7	0.186	0.3	0.304	0.3	0.169	0.5	0.156	0.9
137	0.176	-0.4	0.832	-0.3	0.191	-0.6			0.327	1.5	0.245	1.7	0.367	1.2	0.165	0.4	0.109	-0.6
138	0.0814	-2.3			0.218	-0.1					0.221	1.1	0.970	9.7				
139	0.151	-0.9	0.752	-0.7					0.215	-0.4	0.173	0.0	0.326	0.6	0.137	-0.3		
140	0.0729 2	-2.5									0.2201 6	1.1	0.4526 8	2.4				
141	FN	-3.8	0.832	-0.3	0.206	-0.3			0.234	-0.1	0.17	-0.1	0.182	-1.4	0.17	0.5	0.155	0.8
142																		
143											0.209	0.8	0.356	1.0				
144	0.264	1.4	0.925	0.1	0.335	2.0			0.256	0.3	0.165	-0.2	0.434	2.1	0.198	1.3	0.178	1.6
145	0.174	-0.4	0.831	-0.3	0.126	-1.8	0.125	-0.5	0.229	-0.2	0.131	-1.0	0.215	-1.0	0.131	-0.5		
146	0.197	0.0	1.01	0.4	0.193	-0.6	0.123	-0.6	0.252	0.2	0.150	-0.5	0.301	0.2	0.140	-0.3	0.100	-0.9
147											0.133	-0.9	0.360	1.1				
148	0.0092	-3.8	0.185	-3.2	0.016	-3.7	FN	-3.7	0.073	-2.8	0.132	-0.9	0.145	-2.0	0.061	-2.4		
149											0.213	0.9			0.166	0.4		
150	0.295	2.0	1.04	0.6	0.295	1.2	0.015	-3.6			0.238	1.5	0.194	-1.3	0.312	4.3	0.263	4.2
151	0.226	0.6	1.04	0.6	0.418	3.4			0.301	1.0	0.183	0.2	0.314	0.4	0.165	0.4		
152																		
153	0.231	0.7	0.818	-0.4	0.230	0.1	0.128	-0.4	0.223	-0.3	0.175	0.0	0.290	0.1	0.144	-0.2	0.191	2.0
154	0.0825	-2.3	0.430	-2.1					0.1226	-1.9			0.252	-0.5				

**Table 11h** Results in mg/kg and z-scores for epoxiconazole, fenpropidin, isoprothiolane, pendimethalin, pirimicarb, propiconazole, prothioconazole-desthio, pyraclostrobin and tebuconazole.

Laboratory code	Epoxiconazole	Fenpropidin		Isoprothiolane		Pendimethalin		Pirimicarb		Propiconazole		Prothioconazole-desthio		Pyraclostrobin		Tebuconazole		
MRRL	0.01	0.01		0.01		0.01		0.01		0.01		0.01		0.01		0.01		
Assigned value	0.576	0.927		0.082		0.108		0.251		0.202		0.087		0.465		0.423		
	Z-scores (FFP RSD (25%))																	
118																		
119	0.553	-0.3	0.737	-0.9	0.0830	0.0	0.141	1.2	0.257	0.1	0.187	-0.4	0.0840	-0.2	0.403	-0.6	0.424	-0.1
120											0.088	-2.3					0.196	-2.2
122	0.499	-0.6	1.03	0.4	0.0830	0.0	0.104	-0.1	0.235	-0.3	0.195	-0.2	0.0765	-0.5	0.365	-0.9	0.416	-0.1
123	0.687	0.6	0.718	-1.0	0.082	0.0	0.107	0.0	0.279	0.4	0.191	-0.3	0.088	0.0	0.612	1.2	0.438	0.1
124							0.131	0.9			0.102	-2.0					0.274	-1.5
125	0.726	0.9	1.04	0.4	0.0832	0.0	0.0945	-0.5	0.255	0.0	0.200	-0.1	0.0870	0.0	0.476	0.0	0.509	0.7
126																		
127																		
128	0.725	0.9			0.087	0.2	0.111	0.1	0.304	0.8	0.222	0.3			0.395	-0.7	0.493	0.6
129	0.74	1.0					0.27	6.0	0.44	3.0	0.27	1.3					0.85	3.9
130									0.233	-0.3	0.188	-0.3			FN	-3.9	0.499	0.6
131	0.973	2.6	FN	-4.0	FN	-3.5	0.0539	-2.0	0.0715	-2.9	0.328	2.4					0.338	-0.9
132	0.600	0.0	0.722	-0.9	0.0729	-0.5	0.0713	-1.4	0.0943	-2.5	0.195	-0.2	0.0854	-0.1	0.426	-0.4	0.260	-1.6
133	0.70	0.7									0.24	0.7					0.50	0.6
135	0.784	1.3	1.01	0.3	0.068	-0.7	0.080	-1.0	0.207	-0.7	0.235	0.6			0.410	-0.5	0.340	-0.8
136	0.703	0.7	0.963	0.1	0.094	0.5	0.121	0.5	0.298	0.7	0.262	1.1	0.102	0.7	0.497	0.2	0.440	0.1
137	0.653	0.4					0.142	1.3	0.275	0.4	0.180	-0.5			0.357	-1.0	0.495	0.6
138	0.0858	-3.4							0.242	-0.2					FN	-3.9	0.0703	-3.3
139							0.0977	-0.4	0.252	0.0	FN	-3.8					0.472	0.4
140	0.2788	-2.1			0.0691	-0.7					0.1226	-1.6						
141			FN	-4.0			0.123	0.6	0.271	0.3	0.204	0.0			0.458	-0.1	0.480	0.5
142																		
143							0.126	0.7										
144	0.515	-0.5	1.61	2.8	0.110	1.3	0.157	1.8	0.360	1.7	0.260	1.1	0.098	0.5	0.514	0.4	0.536	1.0
145	0.493	-0.7	0.711	-1.0	0.095	0.6	0.081	-1.0	0.206	-0.7	0.168	-0.7	0.078	-0.4	0.658	1.6	0.469	0.4
146	0.643	0.3	0.850	-0.4	0.075	-0.4	0.117	0.3	0.225	-0.4	0.233	0.5	0.081	-0.3	0.474	0.0	0.400	-0.3
147							0.0993	-0.3										
148	0.080	-3.5	FN	-4.0	0.038	-2.2			0.029	-3.5	0.029	-3.4	FN	-3.5	0.085	-3.3	0.091	-3.2
149											0.218	0.2					0.453	0.2
150	0.520	-0.5	0.616	-1.4	0.115	1.5	0.129	0.8	0.294	0.7	0.242	0.7			0.510	0.3	0.432	0.0
151	0.691	0.7	1.10	0.7			0.112	0.1	0.277	0.4	0.237	0.6	FN	-3.5	0.664	1.6	0.519	0.8
152																		
153	0.450	-1.0	1.03	0.4	0.118	1.7	0.097	-0.4	0.251	0.0	0.213	0.1	0.082	-0.3	0.486	0.1	0.436	0.0
154	0.240	-2.4					0.0660	-1.6										

### 3.4.2 Sum of Weighted z-Scores (AZ<sup>2</sup>) – Category A

To be classified into Category A, the labs had to submit quantitative results for at least 90 % of the pesticides present in the Test Item ( $\geq 14$  pesticide residues, inclusive of false negatives). For the 70 EU and EFTA laboratories in Category A, the results were additionally evaluated by calculating the Average of the squared z-Score (AZ<sup>2</sup>). Of the 70 participants, 59 participants (84 %) obtained AZ<sup>2</sup> at or below 2 (good) and 6 participants (9 %) obtained AZ<sup>2</sup> above 2 but below or at 3 (satisfactory) and 5 participants (7 %) obtained AZ<sup>2</sup> above 3 (unsatisfactory). An additional three laboratories from Third Countries were evaluated in Category A. The AZ<sup>2</sup> scores achieved by the labs can be seen in **Table 12**.

**Table 12** Sum of Weighted z-Scores (SWZ) for laboratories in Category A, number of pesticide analysed by the laboratory, false negatives reported and Classification as good, satisfactory and unsatisfactory.

Lab code	No. of detected pesticides	AZ <sup>2</sup>	False negative	Evaluation	NRL
1	17	1.3	Yes	Good	
3	18	0.8		Good	NRL
4	18	0.6		Good	NRL
5	17	1.2		Good	
7	16	2.5		Satisfactory	
8	18	0.2		Good	NRL
10	18	0.6		Good	
11	18	0.8		Good	NRL
12	18	0.4		Good	
14	18	0.4		Good	NRL
17	17	0.2		Good	
19	18	3.9		Unsatisfactory	
21	16	1.3	Yes	Good	
22	18	2.0		Good	
25	16	2.4		Satisfactory	
27	17	1.7		Good	
28	18	1.6		Good	NRL
31	18	0.3		Good	
32	18	1.3		Good	NRL
34	18	0.1		Good	NRL
37	17	0.3		Good	
39	17	0.9		Good	
41	18	1.5		Good	
43	18	0.2		Good	
44	16	2.1		Satisfactory	
45	18	2.7		Satisfactory	NRL
46	18	3.4		Unsatisfactory	
47	18	1.2		Good	
49	18	0.5		Good	
50	18	1.1		Good	
51	18	0.6		Good	
53	18	0.1		Good	
56	17	3.8		Unsatisfactory	

Lab code	No. of detected pesticides	AZ <sup>2</sup>	False negative	Evaluation	NRL
59	18	0.0		Good	
61	17	1.6		Good	NRL
62	18	0.5		Good	
63	16	4.4	Yes	Unsatisfactory	
64	18	1.3		Good	NRL
65	18	1.4		Good	
66	17	0.5		Good	
68	18	0.1		Good	
69	17	2.3	Yes	Satisfactory	NRL
70	16	0.9		Good	NRL
71	18	0.2		Good	
72	18	0.5		Good	
81	18	0.2		Good	
84	18	0.4		Good	NRL
85	16	2.8		Satisfactory	
86	18	0.9		Good	
87	18	0.1		Good	
90	18	0.8		Good	
92	18	0.2		Good	
93	18	0.3		Good	
94	18	2.1		Satisfactory	NRL
98	18	0.6		Good	NRL
103	16	4.5		Unsatisfactory	
104	18	0.6		Good	
108	18	1.2		Good	
109	17	2.0	Yes	Good	NRL
114	17	0.3		Good	NRL
119	18	0.3		Good	
122	18	0.2		Good	NRL
123	18	2.0		Good	NRL
125	18	0.3		Good	NRL
128	16	0.6		Good	
132	18	0.9		Good	NRL
135	17	1.2		Good	
136	18	0.4		Good	
144	17	1.9		Good	
145	17	0.7		Good	NRL
146	18	0.2		Good	
150	16	4.1		Unsatisfactory	
153	18	0.5		Good	

**Table 13** shows the 57 EU and EFTA laboratories in Category B and the 16 laboratories from Third Countries. The table include information on the number of reported results, the number of acceptable z-scores as well as information on false negative and false positive results.

**Table 13** Number of pesticides analysed, number of acceptable z-scores, false negative and positive for the laboratories in Category B.

Lab code	No. of pesticides detected	No. of acceptable z-scores	False negative	False positive	NRL
2	2	2			
6	12	8			
9	14	13	2		
13	11	11			
15	1	1			
16	15	13			
20	10	10			
23	13	13			NRL
24	6	6			
26	11	2	1		
29	14	7	1		NRL
30	8	7			
35	11	10			
36	13	1	3		
40	8	4			
42	13	13			
52	15	15			
54	8	4	1		
57	17			1	
58	12	11	1		
60	0	0			
67	9	4			
73	10	10			
74	8	8			
75	18	17		1	
76	12	12	1		
77	2	2			
78	1	1			
79	5	4			
82	14	13			NRL
83	3	2			
88	9	9			
89	5	5			
91	15	13	1		
95	10	8			
96	11	10			
97	14	14			
99	15	15			NRL
100	6	6			
101	8	8			

Lab code	No. of pesticides detected	No. of acceptable z-scores	False negative	False positive	NRL
102	11	11			
105	12	7			
106	0	0			
107	7	7			
110	8	8			
111	9	9			
112	6	6			
113	8	8			
115	12	10			
116	0	0			
117	8	7			
118	2	2			
120	5	2			
124	8	5			
126	0	0			
127	1	1		1	
129	12	5			NRL
130	9	8	2		
131	11	5	2	1	
133	8	8			
137	14	14			
138	7	3	1		
139	9	9	1		NRL
140	6	3			
141	12	12	2		
142	0	0			NRL
143	3	3			NRL
147	3	3			
148	13	2	3		
149	4	4			
151	14	13	1		
152	0	0			
154	6	3			NRL

### 3.5 Trends in numbers of participating laboratories and their performance

The number of EU and EFTA laboratories participating in the EUPTs on cereals has increased from 62 in EUPT-C1 (2007), 72 in EUPT-C2 (2008), 102 in EUPT-C3 (2009), 115 in EUPT-C4 (2010), 133 in EUPT-C5 (2011) and then decreased to 127 in EUPT-C6 (see **Table 14**). Over the 6-year period, the Target Pesticide List has been expanded from 34 to 107 individual compounds and the number of spiked or incurred pesticides contained in the Test Items has increased from 7 to 18. The analyte scope of pesticides covered is still rather limited for many laboratories, with 24 % of them submitting results for less than 10 out of the 18 pesticides present in the Test Item.

**Table 14** Overall participation and performance of laboratories in the 6 cereal-based EUPTs focusing on MRM pesticides.

	EUPT-C1	EUPT-C2	EUPT-C3	EUPT-C4	EUPT-C5	EUPT-C6
<b>Type of Test Item</b>	Wheat flour	Wheat flour	Oat flour	Rye flour	Rice flour	Barley flour
<b>Participants submitting results (EU and EFTA)</b>	63	72	102	115	133	127
<b>MRM pesticides in the Target Pesticide List <sup>1)</sup></b>	34	43	51	64	103	107
<b>MRM pesticides in the Test Item</b>	7	13	14	16	16	18
<b>No. of results for MRM pesticides</b>	323	830	981	1624	1521	1741
<b>Range of 'reported results', % <sup>2)</sup></b>	63 – 95	60 - 96	48 - 95	55 - 95	41-95	50-95
<b>Acceptable z-scores, %</b>	87	85	87	87	87	90
<b>Questionable z-scores, %</b>	7	12	8	6	4	5
<b>Unacceptable z-scores, %</b>	6	3	5	7	9	4
<b>False negatives, %</b>	2	3	3	4	3	1
<b>Number of false positives</b>	1	2	3	17	16	2
<b>Category A, % of participating laboratories</b>		60	46	46	44	55
<b>Good SWZ/AZ<sup>2</sup>, %</b>		70	72	77	80	79
<b>Satisfactory SWZ/AZ<sup>2</sup>, %</b>		9	15	8	15	14
<b>Unsatisfactory SWZ/AZ<sup>2</sup>, %</b>		21	13	15	5	7

<sup>1)</sup> Number of pesticides, excluding isomers and degradation product.

<sup>2)</sup> The range of reported results has been calculated using the number of laboratories that reported results from the total number of laboratories submitting results (see Table 8).

Some improvement in the overall analytical performance has been observed when looking at the percentage of acceptable, questionable, unacceptable z-scores and false negative results. Especially, the number of false negatives has dropped significantly from 17 in EUPT-C4 to 2 in EUPT-C6. Also the number of false negatives has decrease from 60 and 40 in EUPT-C4 and C5, respectively to 20 in EUPT-C6. The percentage of Category A laboratories increased from 44 to 55% from EUPT-C5 to EUPT-C6. However, it is difficult to assess any improvement/deterioration in laboratory performance between all the six Proficiency Tests, because the pesticides in the Test Item and the laboratories participating in the PTs

have significantly increased. However, azoxystrobin and carbendazim, which were included in all six Test Items, can be used as good indicators of the developments in laboratory performance over the years. While the analytical performance for azoxystrobin and carbendazim improved from EUPT-C1 to EUPT-C5, it worsened slightly in EUPT-C6.

The number of laboratories participating in the EUPTs with cereal Test Items increased from 62 in EUPT-C1, 72 in EUPT-C2, 102 in EUPT-C3, 115 in EUPT-C4, 133 in EUPT-C5 and 127 in EUPT-C6. The target pesticide list has increased from 34 MRM pesticides to 65 MRM pesticides and the number of pesticides, spiked or incurred in the Test Item has increased from 7 to 16.

The scope is still very limited for many laboratories, as 25% of the laboratories submitted results for less than 10 pesticides. No significant improvement in performance has been seen when looking at the percentage of acceptable, questionable, unacceptable z-scores and false negative results. The percentage of Category A laboratories has fallen from 60 to 46 % from EUPT-C2 to EUPT-C3 and in EUPT-C6 it is still at 46%. Nevertheless, it is difficult to assess any improvement/deterioration in laboratory performance between the different proficiency tests, because the pesticides in the Test Items and the laboratories participating in the PTs have changed. However, azoxystrobin and carbendazim have been included in all three Test Items, and can therefore be used as indicators of trends in accuracy of performance.

The percentage of acceptable z-scores for azoxystrobin has increased from 62% to 88% from EUPT-C1 to EUPT-C6. This is probably due to the recommendation to add water to the sample before extraction. In fact, the percentage of laboratories adding water has also increased from 65% to 86%. For carbendazim, the percentage of acceptable z-scores has not increased, but decreased slightly from 79% to 74%. However, many of the participating laboratories in EUPT-C6 have not participated before, and it is not fully correct to include their results in a trend analysis. Focussing on the results only from laboratories that have participated in all the PTs, the laboratory performance has improved for both compounds. For azoxystrobin the percentage of acceptable results has increased from 62% to 95% and for carbendazim from 79% to 88%. It is therefore reasonable to conclude that the PTs have been valuable for in improving analytical accuracy.

### **3.6 Summary, conclusions and prospect for the EUPTs on pesticide residues in cereals**

EUPT-C6 was the sixth EUPT focusing on cereal-based Test Items. A homogenous Test Item of barley flour, including both incurred and spiked pesticides, was successfully prepared. The barley was sprayed in the field with commercially available pesticide formulations, and also spiked in the laboratory following harvest with pesticide standards of the following pesticides: azoxystrobin, boscalid, carbendazim, carboxin, chlorpropham, chlorpyrifos, cypermethrin, cyprodinil, diflubenzuron, epoxiconazole, fenpropidin, isoprothiolane, pendimethalin, pirimicarb, propiconazole, prothioconazole, pyraclostrobin and tebuconazole. One hundred and twenty seven laboratories, representing 28 EU and EFTA countries, agreed to participate in the Proficiency Test. All laboratories submitted results, but five laboratories did not have any of the 18 pesticide in their scope and consequently were not able to submit results. All Member States and NRLs, except Italy, participated in the PT. However, Malta was represented in the PT by the NRL for the UK. An additional 22 laboratories from Third Countries registered for the PT and 19 submitted results. The Target Pesticide List distributed to the laboratories prior to the test contained 107 individual compounds.

Both the number of false positives and false negatives decreased in comparison to EUPT-C5. The 2 false positive results concerned methacrifos and pyrimethanil. The 20 false negative results concerned azoxystrobin (2), boscalid (2), carboxin (2), chlorpropham (1), fenpropidin (3), isoprothiolane (2), pirimicarb (1), propiconazole (2), prothioconazole-desthio (4) and pyraclostrobin (1). The average Qn-RSD (robust RSD) was at 23 %, close to the FFP-RSD of 25 % with a range from 17 to 28 % for the individual compounds.

For azoxystrobin, boscalid, chlorpropham, chlorpyrifos, cypermethrin, cyprodinil, epoxiconazole, isoprothiolane, pendimethalin, pirimicarb and tebuconazole acceptable results were obtained for 90-97 % of the laboratories. For carbendazim, carboxin, diflubenzuron, fenpropidin, propiconazole, prothioconazole-desthio and pyraclostrobin acceptable results were obtained by 84-89 % of the laboratories. Overall, the performance of the laboratories appears to have shown a small improvement compared to the previous PTs on cereals.

The next PT will focus on a compound animal feeding stuff. However, the selection of pesticides will continue to focus on pesticides included in the scope of the EU coordinated control programme as well as additional pesticides of relevance to animal feed production in Europe and in other parts of the world from where significant quantities of raw materials are imported. The organochlorine pesticides included in Directive 2002/32/EC on undesirable substances in animal feed will be added to the pesticides target list with only few exceptions. The pesticide residues will be incurred wherever practical. In 2013 the Test Item will be feed for laying hens.

Online registration and data submission will continue and be expanded to cover all EUPTs for pesticide residues, if possible.

#### **4. ACKNOWLEDGEMENTS**

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

#### **5. REFERENCES**

[1] Regulation (EC) N° 882 /2004 of the European Parliament and of the Council on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules. Published at OJ of the EU L191 of 28.05.2004

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

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

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

[5] ISO 13528:2005 – Statistical methods for use in proficiency testing by interlaboratory comparison

## APPENDICES

### Appendix 1 List of Laboratories registered to participate in the EUPT-C6

#### Participating labs of EU and EFTA member states EU and EFTA

Country	Institution	City	NRL- CF	Report data
Austria	Austrian Agency for Health and Food Safety, Competence Center for Residues of Plant Protection Products	Innsbruck	NRL	Yes
Belgium	LOVAP (Laboratorium voor Onderzoek Van levensmiddelen en Aanverwante Produkten) NV	Geel		Yes
Belgium	Scientific Institute of Public Health	Brussels	NRL	Yes
Belgium	Fytolab	Gent - Zwijnaarde		Yes
Belgium	Federal Laboratory for Safety of Food Chain, Tervuren	Tervuren		Yes
Bulgaria	Central Laboratory for Chemical Testing and Control, Sofia	Sofia		Yes
Cyprus	Laboratory of Pesticide Residues Analysis, State General Laboratory	Nicosia	NRL	Yes
Cyprus	Animal Feeds and Feed Additives Laboratory of the Analytical Laboratories of the Department of Agric	Nicosia	NRL	Yes
Czech Republic	Institute of Chemical Technology, Dept. of Food Chemistry and Analysis - Prague	Praha		Yes
Czech Republic	Czech Agriculture and Food Inspection Authority	Praha	NRL	Yes
Czech Republic	UKZUZ - NRL Central Institute for Supervising and Testing in Agriculture	Brno	NRL	Yes
Denmark	Danish Veterinary and Food Administration, Region East	Ringsted		Yes
Denmark	Danish Plant Directorate, Laboratory for Feed and Fertilizers	Lyngby	NRL	Yes
Estonia	Agricultural Research Centre, Saku, Lab for Residues and Contaminants	Saku	NRL	Yes
Finland	Customs Laboratory	Espoo	NRL	Yes
Finland	Finnish Food Safety Authority	Helsinki		Yes
France	CERECO SUD	GARONS		Yes
France	Service Commun des Laboratoires / Laboratoire Ile de France - Massy	Massy	NRL	Yes
France	Analysis Center Mediterranean Pyrenees	perpignan		Yes
France	Laboratoire Départemental d'Analyses de la Sarthe, Département de Chimie	Le Mans		Yes
France	Laboratoire Départemental d'Analyses des LANDES	Mont de Marsan		Yes
France	Service Commun des Laboratoires / Laboratoire de Rennes	Rennes		Yes
France	Service Commun des Laboratoires / Laboratoire de Montpellier	Montpellier		Yes
Germany	LUFA-ITL GmbH	Kiel		Yes
Germany	Landesuntersuchungsamt Institut für Lebensmittelchemie Speyer	Speyer		Yes
Germany	Eurofins - Dr. Specht Laboratorien GmbH	Hamburg		Yes
Germany	Bavarian Health and Food Safety Authority Office Erlangen	Erlangen		Yes
Germany	Landwirtschaftliches Technologiezentrum Augustenberg, Karlsruhe	Karlsruhe		Yes

Country	Institution	City	NRL- CF	Report data
Germany	Chemical and Veterinary Analytical Institute Rhine-Ruhr-Wupper	Krefeld		Yes
Germany	Food and Veterinary Institute Oldenburg	Oldenburg		Yes
Germany	State Department of Environmental and Agricultural Operations in Saxony	Leipzig		Yes
Germany	Chemical and Veterinary Analytical Institute Muensterland-Emscher Lippe	Münster		Yes
Germany	State Laboratory Schleswig-Holstein	Neumünster		Yes
Germany	State Institute for Chemical and Veterinary Analysis of Food, Stuttgart (Residues)	Fellbach		Yes
Germany	Landesamt für Verbraucherschutz - Sachsen-Anhalt	Halle/Saale		Yes
Germany	Thuringian Institute of Agriculture	Jena		Yes
Germany	Berlin-Brandenburg State Laboratory, Potsdam	Potsdam		Yes
Germany	Landwirtschaftliche Untersuchungs- und Forschungsanstalt Speyer	Speyer		Yes
Germany	Landesanstalt für Landwirtschaft, Forsten und Gartenbau, Halle	Halle/Saale		Yes
Germany	State Investigation Institute of Health and Veterinary Saxony	Dresden		Yes
Germany	Landesamt für Landwirtschaft, Lebensmittelsicherheit und Fischerei Mecklenburg-Vorpommern	Rostock		Yes
Germany	Chemisches und Lebensmitteluntersuchungsamt der Stadt Dortmund	Dortmund		Yes
Germany	Chemisches und Veterinäruntersuchungsamt Rheinland, Standort Bonn	Bonn		Yes
Germany	Institut für Hygiene und Umwelt Hamburg	Hamburg		Yes
Germany	Federal Office of Consumer Protection and Food Safety, NRL for Pesticide Residues	Berlin	NRL	Yes
Germany	Bavarian Health and Food Safety Authority Office Oberschleißheim Feed Analytics (Feedingstuff)	Oberschleißheim		Yes
Greece	General Chemical State Laboratory, D Division, Pesticide Residues Laboratory	Athens	NRL	Yes
Greece	Benaki Phytopathological Institute, Pesticide Residues Laboratory	Kifissia	NRL	Yes
Greece	Regional Center of Plant Protection and Quality Control of Ioannina, Pesticide Residues Laboratory	Ioannina		Yes
Hungary	Agricultural Office, Directorate of Plant Protection, Soil Conservation and Agri-environment - Pesti	Velence	NRL	Yes
Hungary	Agricultural Office, Directorate of Plant Protection, Soil Conservation and Agri-Environment, Pestic	Miskolc		Yes
Hungary	Agricultural Office, Directorate of Plant Protection, Soil Conservation and Agri-Environment, Pestic	Hódmezovásárhely		Yes
Hungary	Agricultural Office, Directorate of Plant Protection, Soil Conservation and Agri-Environment, Pestic	Szolnok		Yes
Ireland	Pesticide Control Laboratory, Department of Agriculture, Fisheries and Food	Co. Kildare	NRL	Yes
Italy	Istituto Zooprofilattico Sperimentale Umbria e Marche, PERUGIA	Perugia		Yes
Italy	ARPA Ferrara Eccellenza Fitofarmaci	Ferrara		Yes
Italy	ARPA Puglia - Dipartimento di Brindisi	Brindisi		Yes
Italy	ARPA Puglia - Dipartimento di Bari	Bari		Yes

Country	Institution	City	NRL- CF	Report data
Italy	APPA Bolzano	Bolzano		Yes
Italy	Istituto Zooprofilattico Sperimentale Lombardia ed Emilia Romagna	Brescia		Yes
Italy	ARPAL Sez. di La Spezia	La Spezia		Yes
Italy	ARPAM Dipartimento di Macerata	Macerata		Yes
Italy	Laboratorio di Sanità Pubblica ASL BERGAMO	Beragmo		Yes
Italy	ARPA Piemonte POLO ALIMENTI	La Loggia (Torino)		Yes
Italy	Istituto Zooprofilattico Sperimentale Lazio e Toscana	Roma		Yes
Italy	Environmental Regional Protection Agency - Laboratory of Pordenone	Pordenone		Yes
Italy	APPA Trento Settore Laboratorio e Controlli	Trento		Yes
Italy	Istituto Zooprofilattico Sperimentale Abruzzo e Molise	Teramo		Yes
Italy	ARPA VENETO DIP.REG.LAB. S.L. VERONA	Verona		Yes
Italy	Centro di referenza nazionale per la sorveglianza ed il controllo degli alimenti per animali	Genova	NRL	Yes
Italy	Istituto Superiore di Sanità, Pesticide Section	Roma		Yes
Latvia	Institute of Food Safety, Animal Health and Environment (BIOR) - Riga	Riga	NRL	Yes
Lithuania	National Food and Veterinary Risk Assessment Institute (Lithuania, Vilnius)	Vilnius	NRL	Yes
Luxembourg	National Health Laboratory Luxembourg (Food Laboratory)	Luxembourg	NRL	Yes
Netherlands	RIKILT Institute of Food Safety (Natural Toxins & Pesticides)	Wageningen		Yes
Netherlands	Netherlands Food and Consumer Products Safety Authority	Wageningen	NRL	Yes
Netherlands	Handelslaboratorium Dr. Verwey	Rotterdam		Yes
Netherlands	Grond-, Gewas- en Milieulaboratorium Zeeuws-Vlaanderen b.v.	Graauw		Yes
Norway	Norwegian Institute for Agricultural and Environmental Research, Plant Health and Plant Protection D	Aas	NRL	Yes
Poland	Institute of Horticulture, Food Safety Laboratory (Skierniewice)	Skierniewice		Yes
Poland	Voievodship Sanitary - Epidemiological Station in Warszaw	Warszaw		Yes
Poland	Institute of Plant Protection Pesticide Residue Laboratory, Bialystok	Bialystok		Yes
Poland	Institute of Plant Protection - National Research Institute, Regional Experimental Station in Rzeszo	Rzeszow		Yes
Poland	Zaklad Higieny Weterynaryjnej w Gdansku (Kartuska)	Gdansk		Yes
Poland	National Research Institute Regional Experimental Station in Trzebnica	Trzebnica		Yes
Poland	Institute of Plant Protection - National Research Institute, Branch Sosnicowice	Sosnicowice		Yes
Poland	Voievodship Sanitary - Epidemiological Station in Opole	Opole		Yes
Poland	Institute of Plant Protection, Department of Pesticide Residue Research - Poznan	Poznan	NRL	Yes
Poland	Zaklad Higieny Weterynaryjnej w Opolu	Opole		Yes
Poland	Regional Veterinary Laboratory Wroclaw	Wroclaw		Yes

Country	Institution	City	NRL- CF	Report data
Poland	Zakład Higieny Weterynaryjnej w Szczecinie, Pracownia Analityki Chemicznej	Szczecin		Yes
Poland	Zakład Higieny Weterynaryjnej w Katowicach, Pracownia Badania Pasz	Katowice		Yes
Poland	Zakład Higieny Weterynaryjnej w Białymstoku	Białystok		Yes
Poland	Wojewódzki Inspektorat Weterynarii z/s w Siedlcach, Zakład Higieny Weterynaryjnej w Warszawie	Warszawa		Yes
Poland	Provincial Veterinary Inspectorate Establishment of Veterinary Hygiene	Poznan		Yes
Portugal	L-DRAPN, Direcção Regional de Agricultura e Pescas do Norte-DEQAL	Matosinhos		Yes
Portugal	Regional Laboratory of Veterinary and Food Safety - Madeira Island	Funchal - Madeira Island		Yes
Portugal	INIA - Pesticides Residues Laboratory	Oeiras	NRL	Yes
Romania	Central Laboratory for Pesticides Residues Control in Plants and Vegetable Products - Bucharest	Bucharest	NRL	Yes
Romania	Sanitary Veterinary and Food Safety Directorate, Bucharest	Bucharest		Yes
Romania	Sanitary Veterinary and Food Safety Laboratory - IASI	Iasi		Yes
Romania	Sanitary Veterinary and Food Safety Directorate Cluj, Gas-Chromatography Laboratory	Cluj Napoca		Yes
Romania	Institute for Hygiene and Veterinary Public Health - Bucharest	Bucharest	NRL	Yes
Slovakia	State Veterinary and Food Institute Bratislava	Bratislava	NRL	Yes
Slovakia	Public Health Authority of Slovak Republic	Bratislava		Yes
Slovenia	Agricultural Institute of Slovenia, Central Laboratories	Ljubljana	NRL	Yes
Slovenia	Institute of Public Health, Maribor	Maribor		Yes
Slovenia	Institute of Public Health, Ljubljana	Ljubljana	NRL	Yes
Spain	Agrofood Laboratory of the Comunidad Valenciana	Burjassot-Valencia		Yes
Spain	Laboratorio de Producción y Sanidad Vegetal de Huelva	Cartaya (Huelva)		Yes
Spain	Laboratorio Arbitral Agroalimentario, Madrid	Madrid	NRL	Yes
Spain	Laboratorio Regional CCAA La Rioja	Logroño		Yes
Spain	Laboratorio Agrario Regional - Junta de Castilla y Leon	Burgos		Yes
Spain	Agricultural and Phytopathological Laboratory of Galicia	Abegondo. A Coruña		Yes
Spain	NASERTIC Navarra de Servicios y Tecnologías, S.A.	Villava		Yes
Spain	Laboratorio Agroalimentario de Zaragoza	Zaragoza		Yes
Spain	Laboratori Agroalimentari de la Generalitat de Catalunya	Cabrils		Yes
Spain	Laboratorio Agrario Regional de Castilla La Mancha	Albacete		Yes
Spain	National Centre for Food - Spain, Majadahonda	Majadahonda	NRL	Yes
Spain	Servicio de Laboratorio y Control de Santander	Santander		Yes
Sweden	Eurofins - Food&Agro Sweden, Lidköping	Lidköping		Yes

Country	Institution	City	NRL- CF	Report data
Sweden	Chemistry Division 1, National Food Administration	Uppsala	NRL	Yes
Switzerland	Kantonales Laboratorium Zürich	Zürich		Yes
Switzerland	Food authority control and veterinary affairs of Geneva	GENEVE		Yes
UK	Eurofins - United Kingdom, Wolverhampton	Wolverhampton		Yes
UK	Agri-Food and Biosciences Institute	Belfast		Yes
UK	The Food and Environment Research Agency - York	York	NRL	Yes

### Participating labs from EU candidate state and the 3rd countries

Country	Institution	City	Report data
Argentina	INTI -LACTEOS	San Martin - Buenos Aires	Yes
Australia	National Measurement Institute	Port Melbourne	Yes
Australia	Symbio Alliance	Eight Mile Plains, QLD	Yes
Brazil	Bioensaios Análises e Consultoria Ambiental	Viamão	Yes
Brazil	Laboratório Nacional Agropecuário - LANAGRO/MG	Pedro Leopoldo	Yes
Burkina Faso	Laboratoire National de Santé Publique	Ouagadougou	Yes
China	ARD department of JiangSu Rotam Chemistry Co.,Ltd	Suzhou Kunshan	Yes
China	Shangqiu Testing Center of Quality and Technical Supervision and Inspection	Shangqiu City	Yes
China	Shanxi Institute For Food and Drug Control	Taiyuan	Yes
China	Guangzhou Quality Supervision and Testing Institute	Guangzhou	Yes
China	Dongguan Supervision and Test Istitution of Agricultral Product Quality and Safety	dongguan	Yes
Costa Rica	Centro de Investigacion en Contaminacion Ambiental (CICA)/ Universidad de Costa Rica	San Jose	Yes
Croatia	Institute of public health Split	Split	Yes
Croatia	Institute of public health Rijeka	Rijeka	Yes
Egypt	Central Lab of Residue Analysis of Pesticides and Heavy Metals in Foods	Dokki, Giza,	Yes
Kenya	Kenya Bureau of Standards (KEBS)	Nairobi	No
Russian Federation	FSFI «Centre for Grain Quality Assurance» Chemical Toxicology Research Department	Ramenskoe, Moscow Region	Yes
SERBIA	SP LABORATORIJA	BECEJ	Yes
Singapore	Veterinary Public Health Laboratory	Singapore	Yes
Turkey	MSM Food Control Laboratory	Mersin	Yes
UAE	dr malik alamin	alain	No
Zambia	Biochemistry and Toxicology Laboratory	Lusaka	No



## Appendix 2 Target Pesticide List

Pesticides	MRRL (mg/kg)
<b>2-phenyl phenol</b>	0.01
3-hydroxycarbofuran	0.01
Acephate	0.01
Azinphos-methyl	0.01
Azoxystrobin	0.01
Bifenthrin	0.01
<b>Boscalid</b>	0.01
Captan	0.01
Carbaryl	0.01
Carbendazim	0.01
Carbofuran	0.01
Carboxin	0.01
Cis-deltamethrin	0.01
Chlorfenvinphos	0.01
Chlorothanlonil	0.01
Chlorpropham	0.01
Chlorpyrifos	0.01
Chlorpyrifos-methyl	0.01
Clothianidin	0.01
Cyfluthrin	0.01
Cypermethrin	0.01
Cyproconazole	0.01
Cyprodinil	0.01
p,p'-DDT	0.01
o,p'-DDT	0.01
p,p'-DDE	0.01
p,p'-TDE	0.01
Demeton-S-methylsulfone	0.01
Diazinon	0.01
Dichlorvos	0.01
Difenoconazole	0.01
Diflubenzuron	0.01
Dimethoate	0.01
Endosulfan sulfate	0.01
Endosulfan $\alpha$	0.01
Endosulfan $\beta$	0.01

Pesticides	MRRL (mg/kg)
Epoxiconazole	0.01
Ethion	0.01
Fenbuconazole	0.01
Fenhexamid	0.01
Fenitrothion	0.01
<b>Fenpropidin</b>	0.01
Fenpropimorph	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 and Esfenvalerate (Sum of RR/SS and RS/SR isomers)	0.01
Fipronil (parent compound)	0.01
Fludioxonil	0.01
Fluquinconazole	0.01
Flusilazole	0.01
Flutriafol	0.01
HCH-alpha	0.01
HCH-beta	0.01
Hexaconazole	0.01
Imazalil	0.01
Imidacloprid	0.01
Iprodione	0.01
Isoprothiolane	0.01
Isoproturon	0.01
Kresoxim-methyl	0.01
Lambda-cyhalothrin	0.01
Lindane (gamma- isomer of hexachlorocyclohexane (HCH))	0.01
Linuron	0.01
Malaoxon	0.01
Malathion	0.01
Metconazole	0.01
Methacrifos	0.01
Methomyl	0.01
Metribuzin	0.01
Omethoate	0.01
Oxydemeton-methyl	0.01

Pesticides	MRRL (mg/kg)
Paclobutrazol	0.01
Parathion	0.01
Penconazole	0.01
Pendimethalin	0.01
Permethrin (sum of isomers)	0.01
Phosphamidon	0.01
Pirimicarb	0.01
Pirimicarb-desmethyl	
Pirimiphos-methyl	0.01
Prochloraz	0.01
Procymidone	0.01
Propiconazole	0.01
Prothioconazole-desthio	0.01
Pyraclostrobin	0.01
Pyrimethanil	0.01
Quinoxyfen	0.01
Spiroxamine	0.01
Tebuconazole	0.01
Tebufenozide	0.01
Thiabendazole	0.01
Thiacloprid	0.01
Thiamethoxam	0.01
Thiodicarb	0.01
Thiophanate-methyl	0.01
Triadimefon	0.01
Triadimenol	0.01
Triazophos	0.01
Tricyclazole	0.01
Trifloxystrobin	0.01
Trifluralin	0.01
Triticonazole	0.01
Vinclozolin	0.01

Three new MRM pesticides added to the Target Pesticide List from EUPT-C5 (2011) are marked in bold. Only individual compounds are included in the pesticide target list, except for pyrethroids where the sum of isomers should be reported, unless other is specified in the list.



**Appendix 3 Homogeneity data**

Sample no.	Azoxystrobin, mg/kg		Boscalid, mg/kg		Carbendazim, mg/kg	
	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
001	0.151	0.138	0.868	0.912	0.180	0.211
035	0.166	0.149	0.990	0.918	0.223	0.210
056	0.172	0.185	0.966	0.947	0.178	0.198
071	0.148	0.179	0.962	0.944	0.197	0.191
106	0.139	0.142	0.983	0.930	0.212	0.227
134	0.132	0.132	0.818	0.766	0.198	0.174
154	0.169	0.147	0.873	0.827	0.204	0.186
168	0.137	0.150	0.872	0.834	0.181	0.200
204	0.151	0.126	0.885	0.837	0.201	0.173
219	0.167	0.132	0.895	0.883	0.181	0.198
256	0.136	0.158	0.930	0.891	0.226	0.198

Sample no.	Carboxin, mg/kg		Chlorpropham, mg/kg		Chlorpyrifos, mg/kg	
	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
001	0.134	0.127	0.282	0.250	0.208	0.179
035	0.134	0.149	0.305	0.294	0.229	0.207
056	0.133	0.133	0.272	0.238	0.205	0.210
071	0.138	0.134	0.278	0.224	0.192	0.211
106	0.130	0.151	0.239	0.275	0.176	0.192
134	0.145	0.134	0.286	0.277	0.206	0.203
154	0.145	0.134	0.244	0.267	0.218	0.201
168	0.132	0.149	0.256	0.282	0.190	0.213
204	0.136	0.115	0.282	0.214	0.203	0.166
219	0.129	0.129	0.279	0.243	0.220	0.173
256	0.140	0.147	0.269	0.284	0.191	0.207

Sample no.	Cypermethrin, mg/kg		Cyprodinil, mg/kg		Diflubenzuron, mg/kg	
	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
001	0.305	0.316	0.145	0.171	0.189	0.170
035	0.349	0.337	0.157	0.155	0.207	0.169
056	0.305	0.276	0.132	0.142	0.139	0.183
071	0.275	0.284	0.138	0.147	0.136	0.164
106	0.269	0.330	0.157	0.166	0.217	0.179
134	0.338	0.301	0.142	0.128	0.192	0.137
154	0.351	0.340	0.160	0.144	0.155	0.136
168	0.328	0.336	0.152	0.143	0.137	0.142
204	0.335	0.261	0.150	0.141	0.181	0.151
219	0.325	0.264	0.137	0.150	0.171	0.189
256	0.298	0.343	0.162	0.140	0.154	0.195

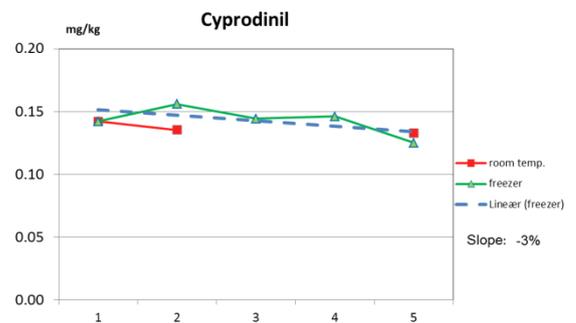
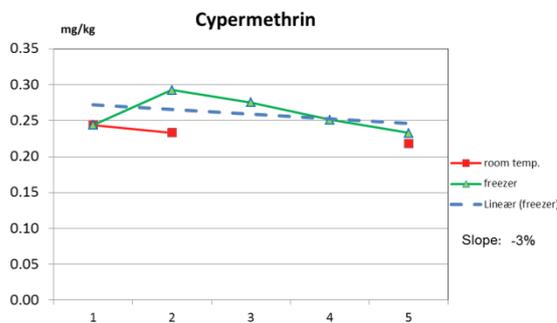
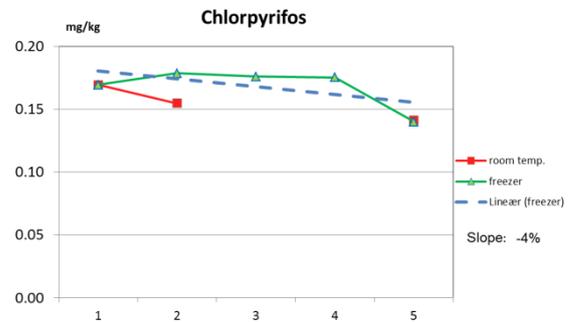
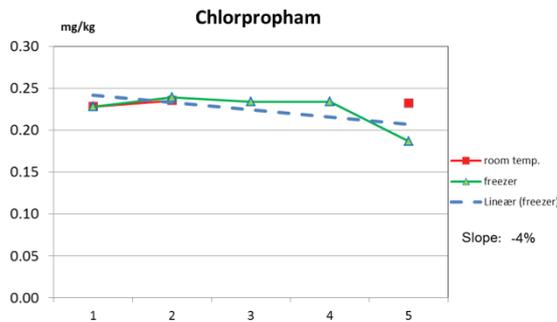
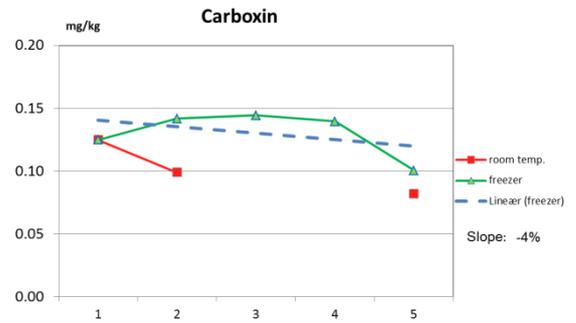
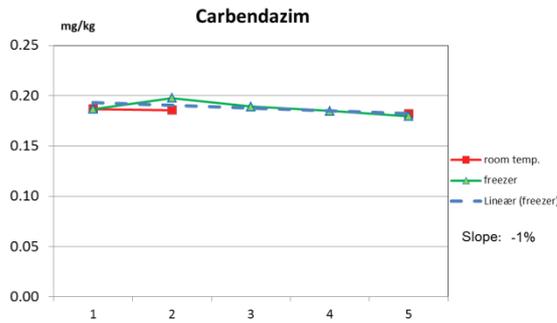
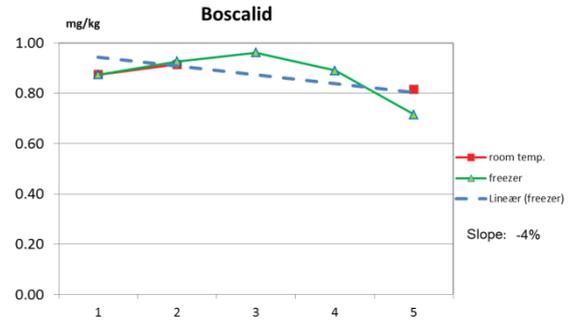
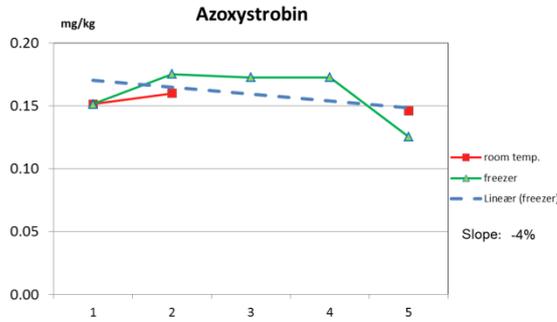
Sample no.	Epoxiconazole, mg/kg		Fenpropidin, mg/kg		Isoprothiolane, mg/kg	
	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
001	0.540	0.589	0.701	0.722	0.078	0.092
035	0.607	0.602	0.782	0.877	0.087	0.089
056	0.622	0.586	0.769	0.999	0.075	0.078
071	0.585	0.582	0.825	0.921	0.084	0.081
106	0.585	0.587	0.684	0.576	0.085	0.093
134	0.480	0.484	0.621	0.686	0.079	0.073
154	0.560	0.536	1.006	0.644	0.088	0.079
168	0.552	0.558	0.786	0.757	0.080	0.082
204	0.547	0.554	0.694	0.711	0.079	0.074
219	0.568	0.588	0.779	0.785	0.077	0.079
256	0.583	0.583	0.834	0.730	0.091	0.076

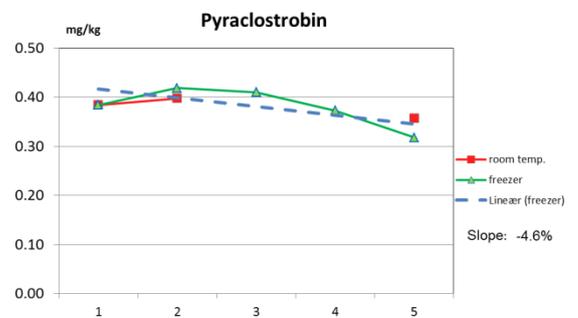
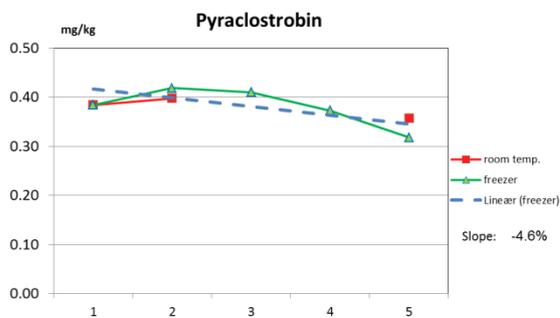
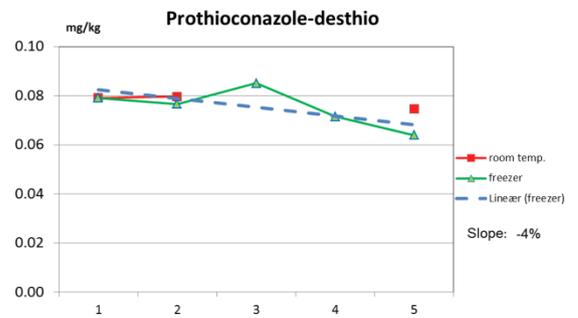
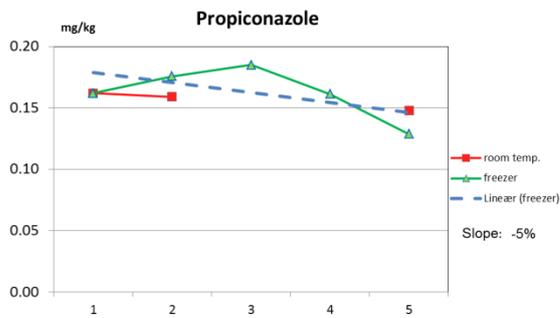
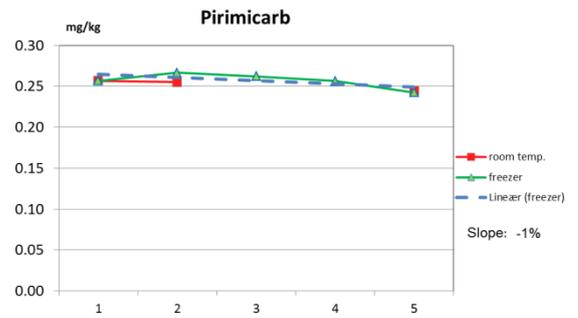
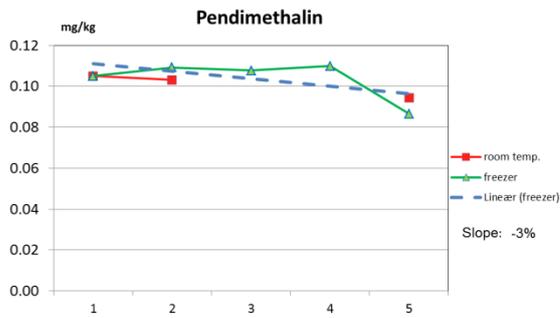
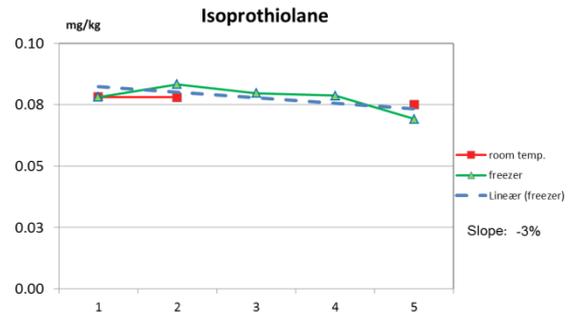
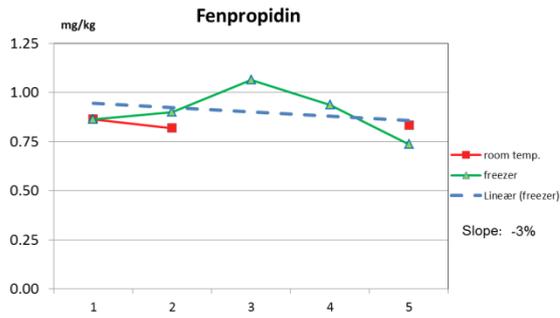
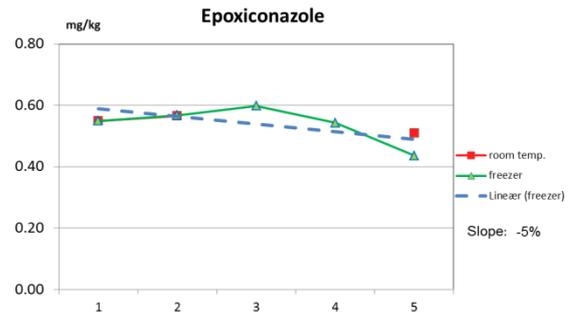
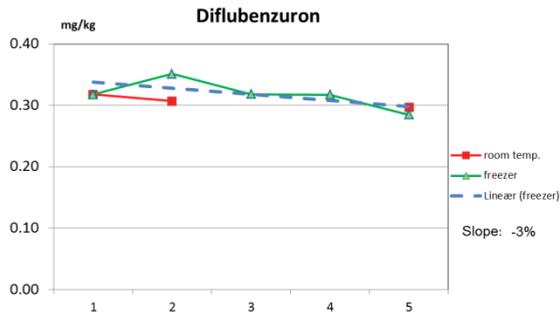
Sample no.	Pendimethalin, mg/kg		Pirimicarb, mg/kg		Propiconazole, mg/kg	
	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
001	0.096	0.139	0.239	0.289	0.175	0.146
035	0.101	0.134	0.271	0.276	0.192	0.182
056	0.098	0.098	0.241	0.253	0.197	0.182
071	0.113	0.120	0.265	0.256	0.175	0.173
106	0.114	0.122	0.275	0.294	0.155	0.164
134	0.101	0.091	0.251	0.232	0.157	0.155
154	0.124	0.095	0.280	0.248	0.156	0.172
168	0.109	0.092	0.249	0.266	0.160	0.177
204	0.105	0.099	0.260	0.236	0.173	0.142
219	0.099	0.104	0.237	0.262	0.191	0.151
256	0.129	0.107	0.290	0.252	0.148	0.190

Sample no.	Prothioconazole- desthio, mg/kg		Pyraclostrobin, mg/kg		Tebuconazole, mg/kg	
	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
001	0.075	0.081	0.371	0.382	0.189	0.207
035	0.083	0.082	0.444	0.433	0.169	0.170
056	0.091	0.081	0.404	0.405	0.139	0.183
071	0.087	0.087	0.423	0.392	0.164	0.137
106	0.085	0.083	0.401	0.394	0.155	0.142
134	0.072	0.072	0.329	0.334	0.181	0.151
154	0.081	0.080	0.366	0.363	0.171	0.189
168	0.078	0.069	0.379	0.402	0.154	0.195
204	0.084	0.086	0.394	0.395	0.034	0.217
219	0.088	0.084	0.397	0.388	0.179	0.192
256	0.085	0.087	0.408	0.390	0.136	0.137

## Appendix 4 Stability figures

The figures below are a graphical presentation of the stability data for the test item stored at -18 °C and room temperature. The regression line for the data derived from storage at -18 °C is shown with the dotted line (in the figures called lineær (freezer)). The slope of the regression line is added to the figure. The stability is accepted if the slope is above -5%.



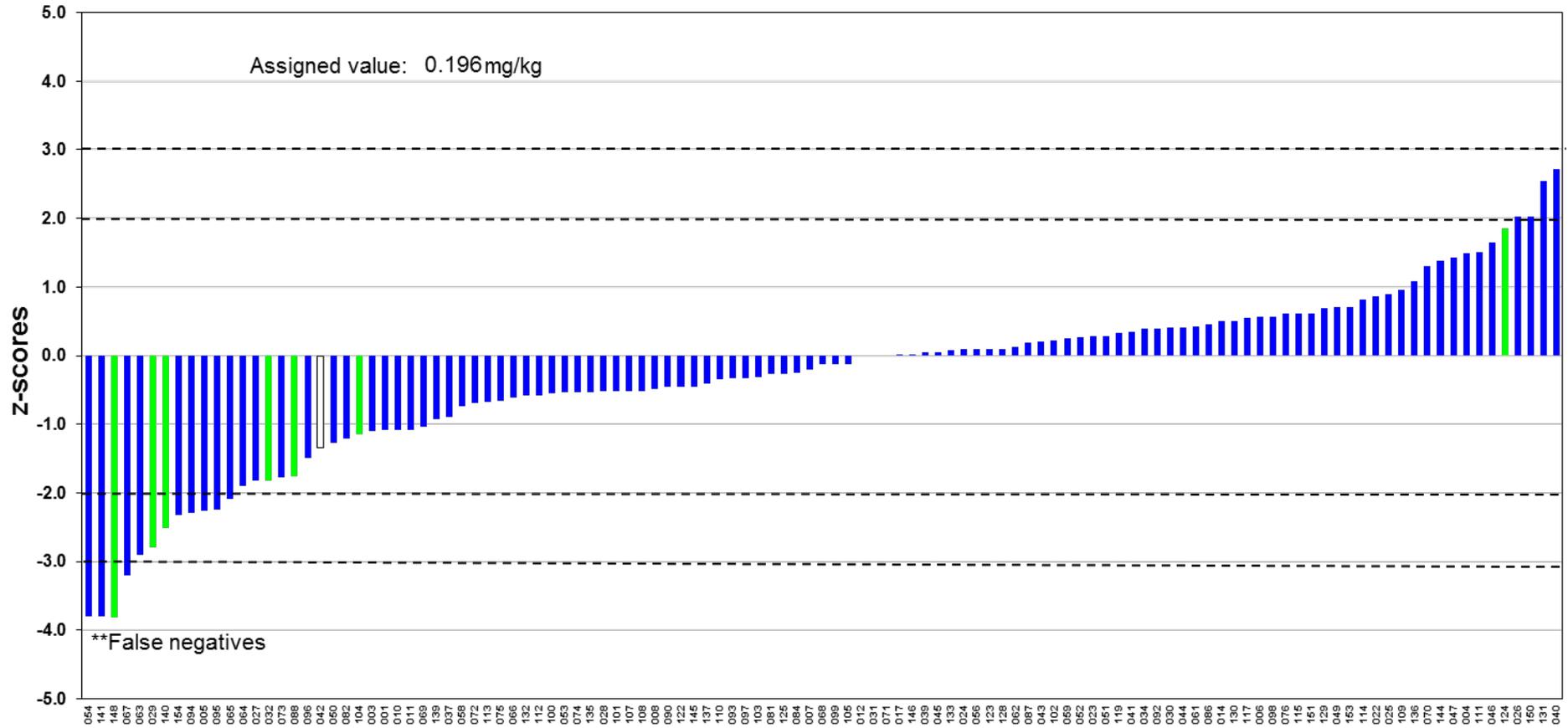


Appendix 6 Graphical presentation of z-scores

**Azoxystrobin**

Acceptable 101  
 Questionable 10  
 Unacceptable 4  
 Number of labs 115

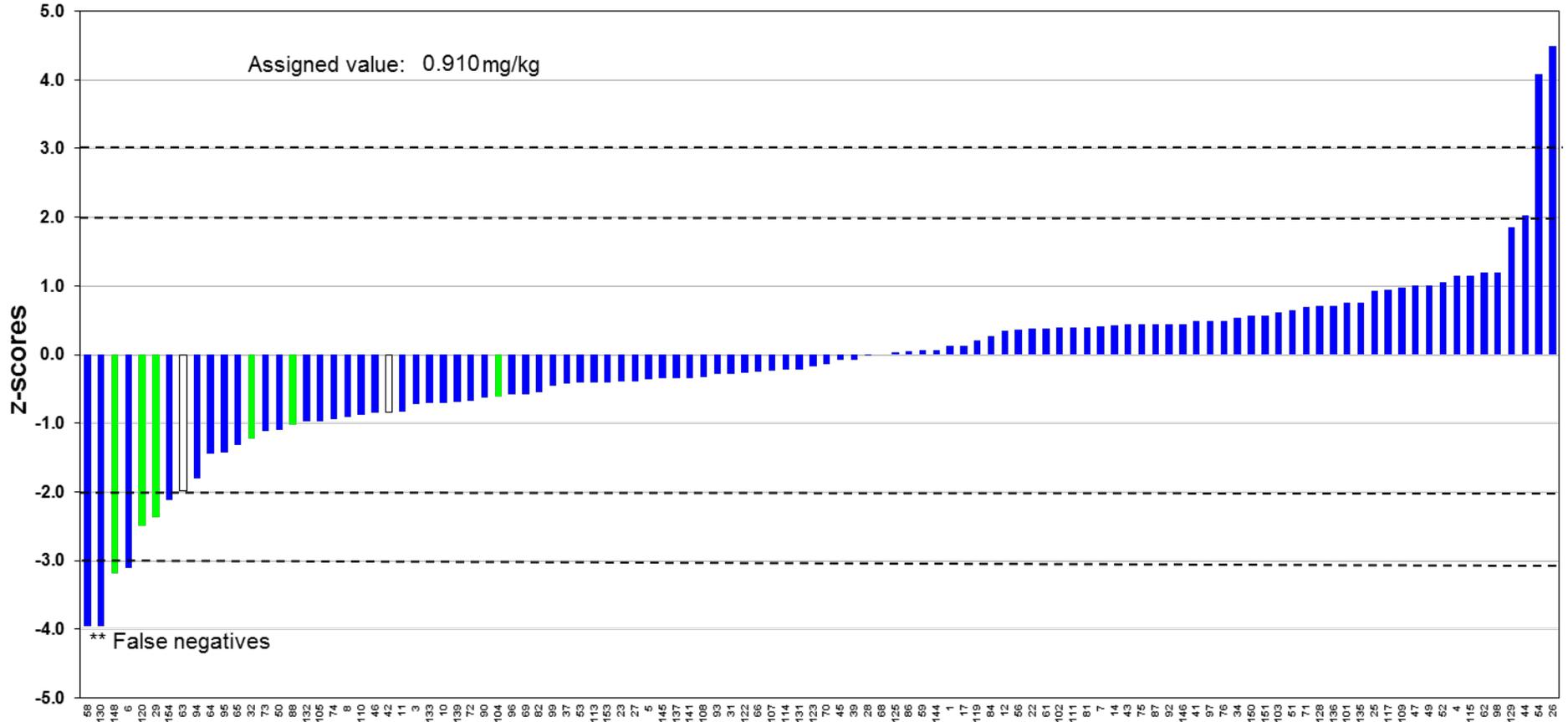
- Water added
- No water added
- Not specified



- Water added
- No water added
- Not specified

### Boscalid

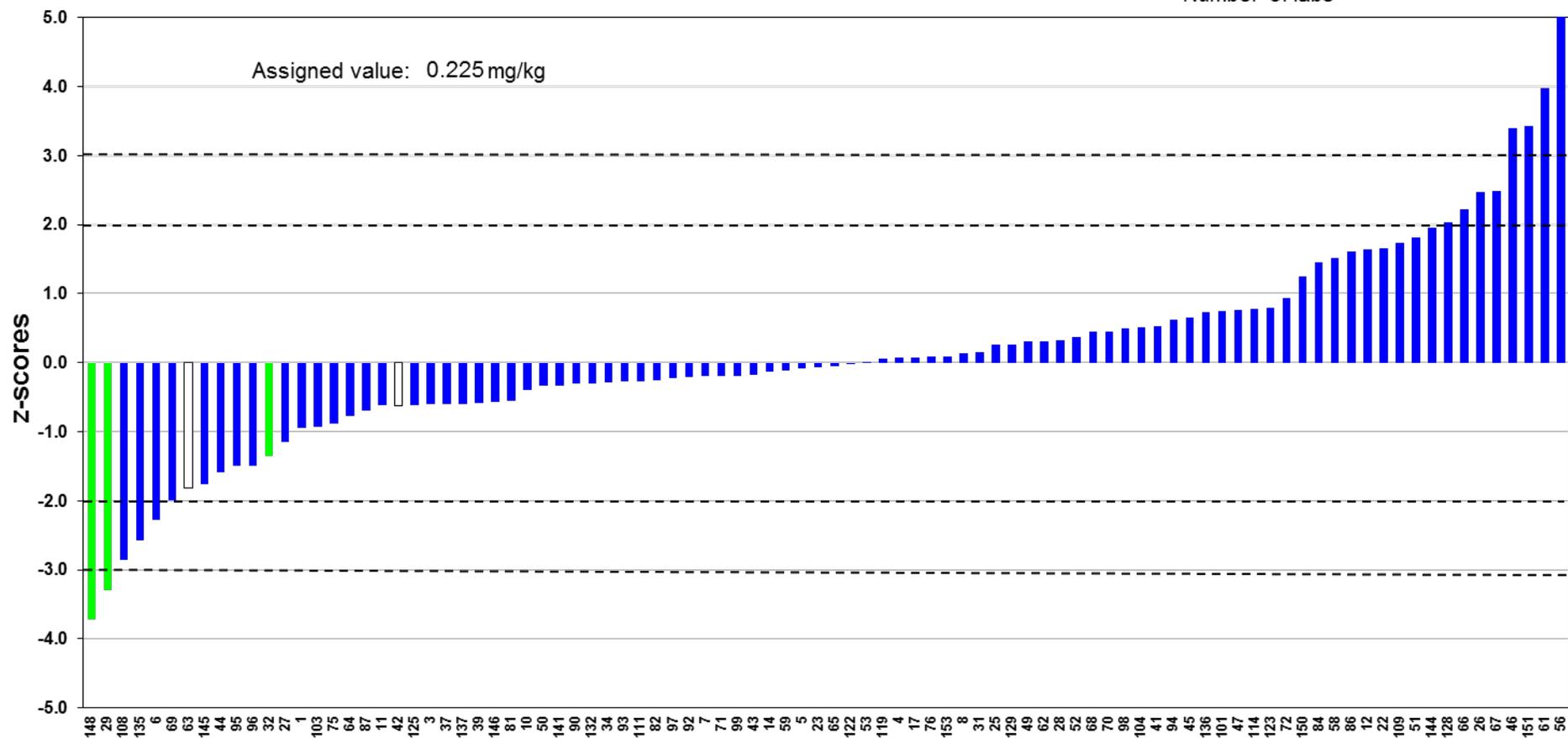
Acceptable 98  
Questionable 4  
Unacceptable 6  
Number of labs 108



# Carbendazim

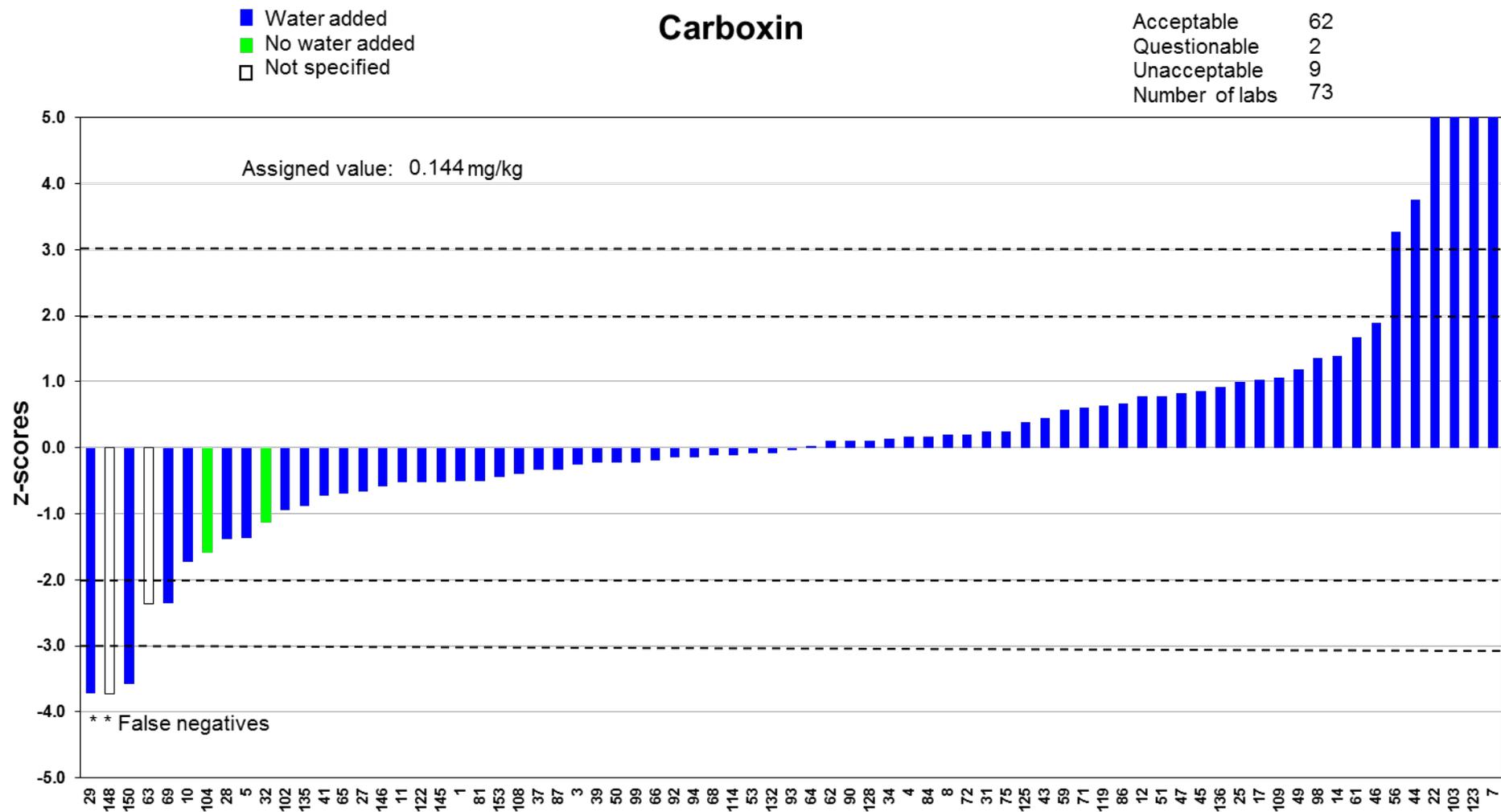
Acceptable	79
Questionable	7
Unacceptable	6
Number of labs	92

- Water added
- No water added
- Not specified



# Carboxin

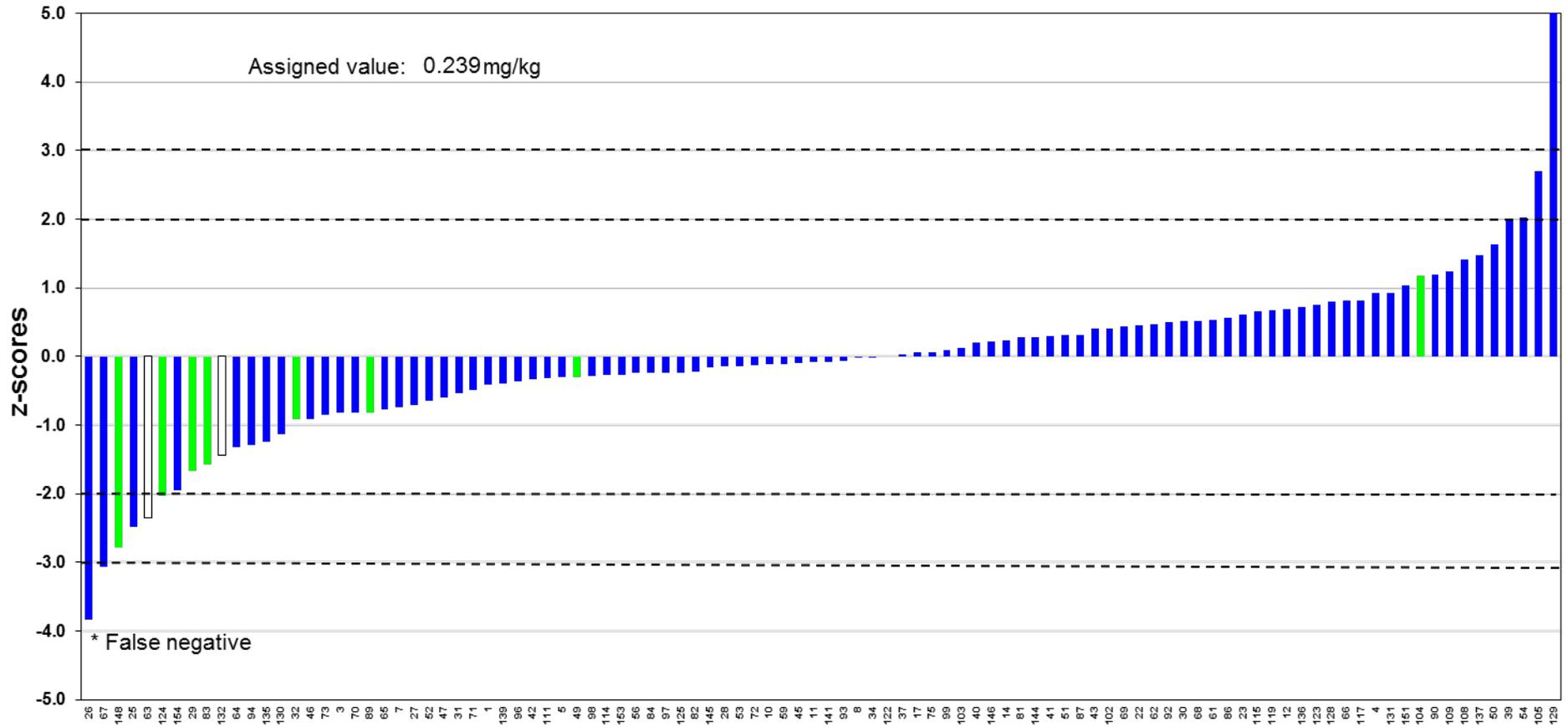
Acceptable	62
Questionable	2
Unacceptable	9
Number of labs	73



# Chlorpropham

Acceptable 91  
 Questionable 6  
 Unacceptable 3  
 Number of labs 100

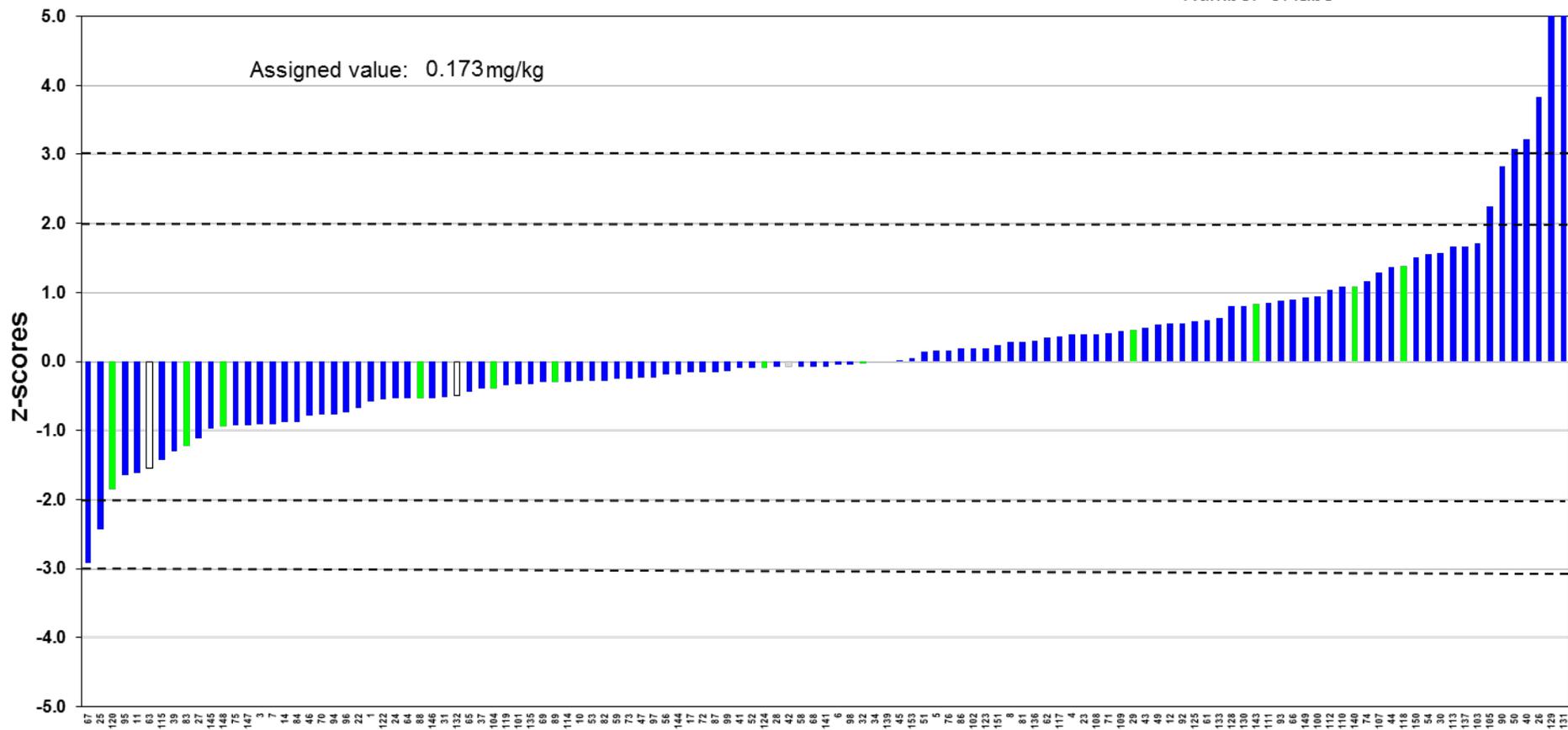
- Water added
- No water added
- Not specified



# Chlorpyrifos

- Water added
- No water added
- Not specified

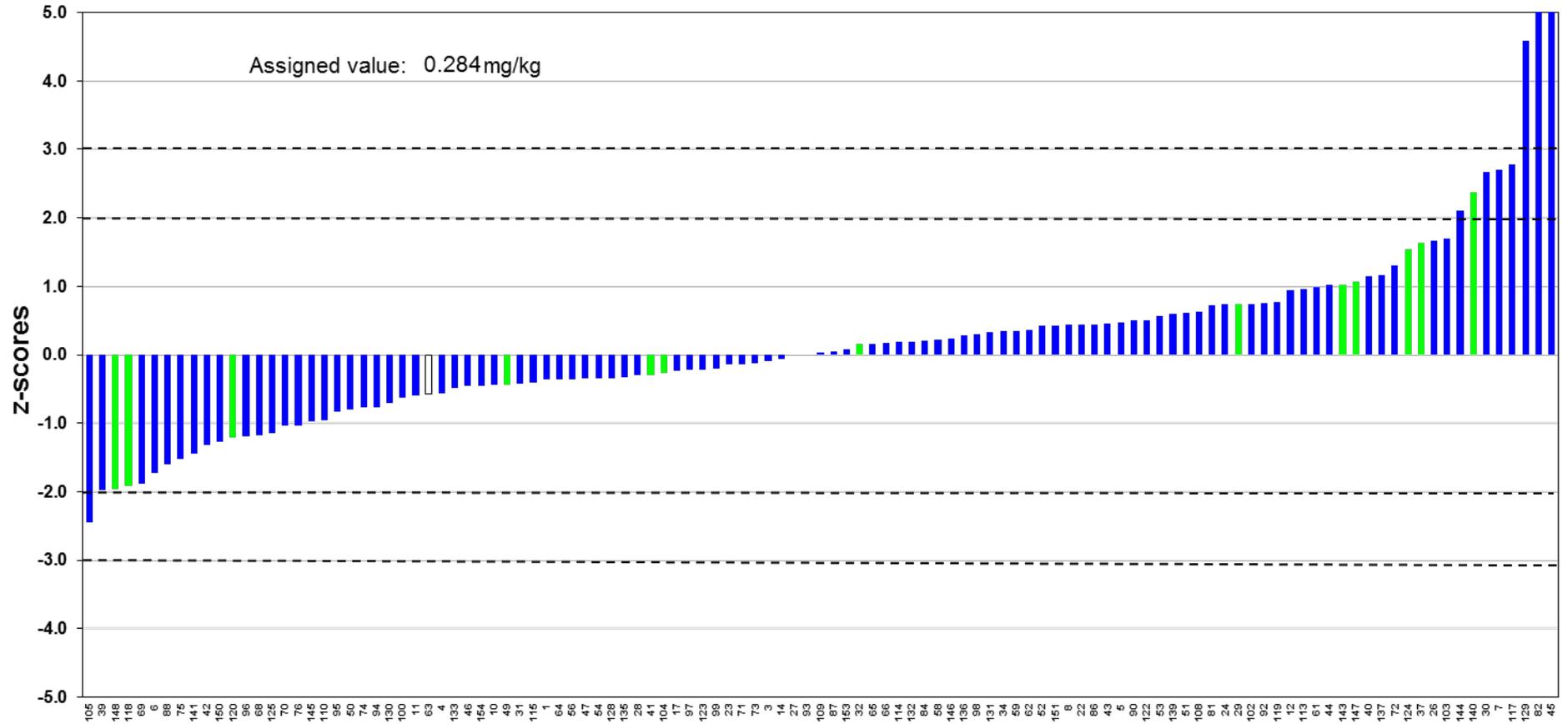
Acceptable	112
Questionable	4
Unacceptable	5
Number of labs	121



- Water added
- No water added
- Not specified

## Cypermethrin (sum of isomers)

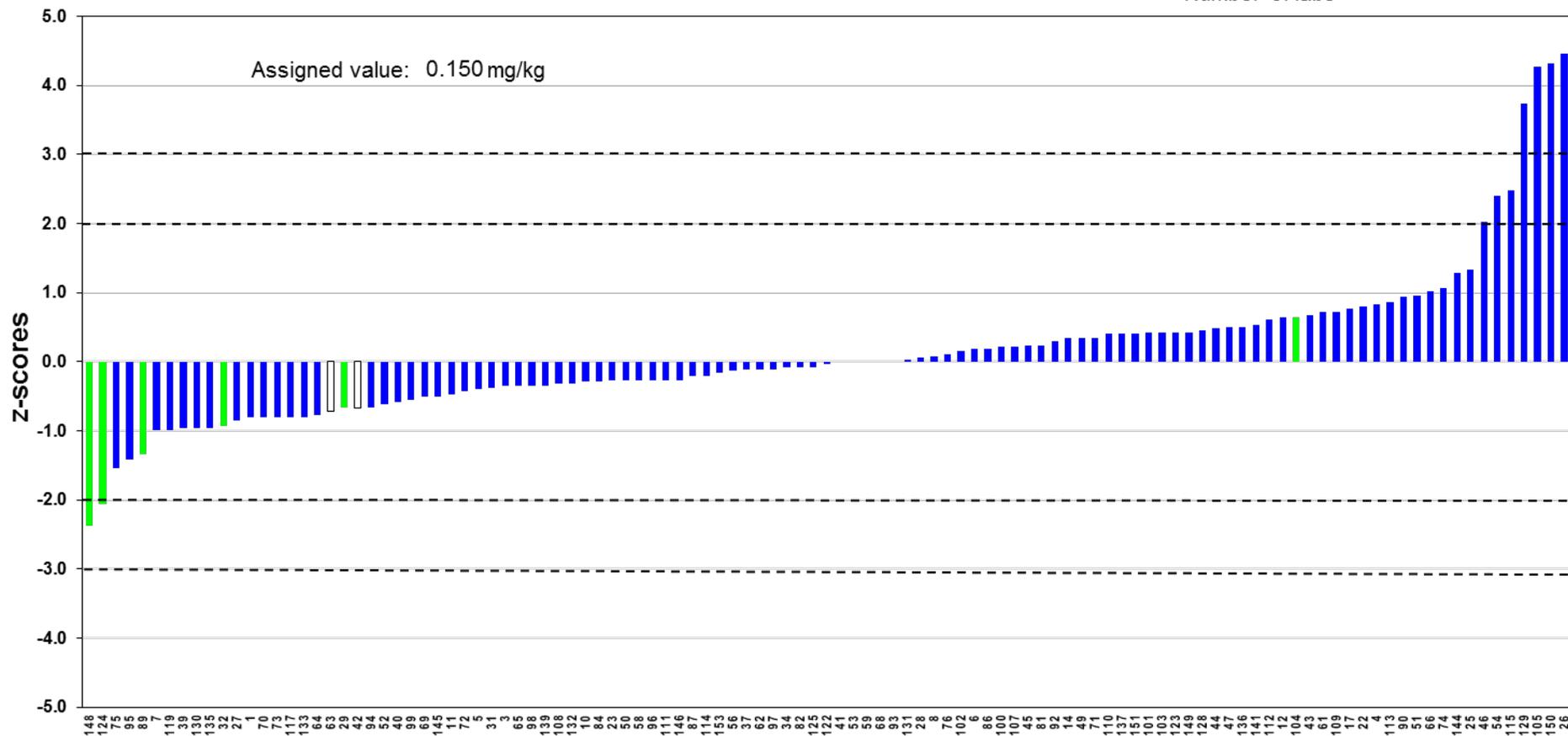
Acceptable	104
Questionable	6
Unacceptable	3
Number of labs	113



# Cyprodinil

- Water added
- No water added
- Not specified

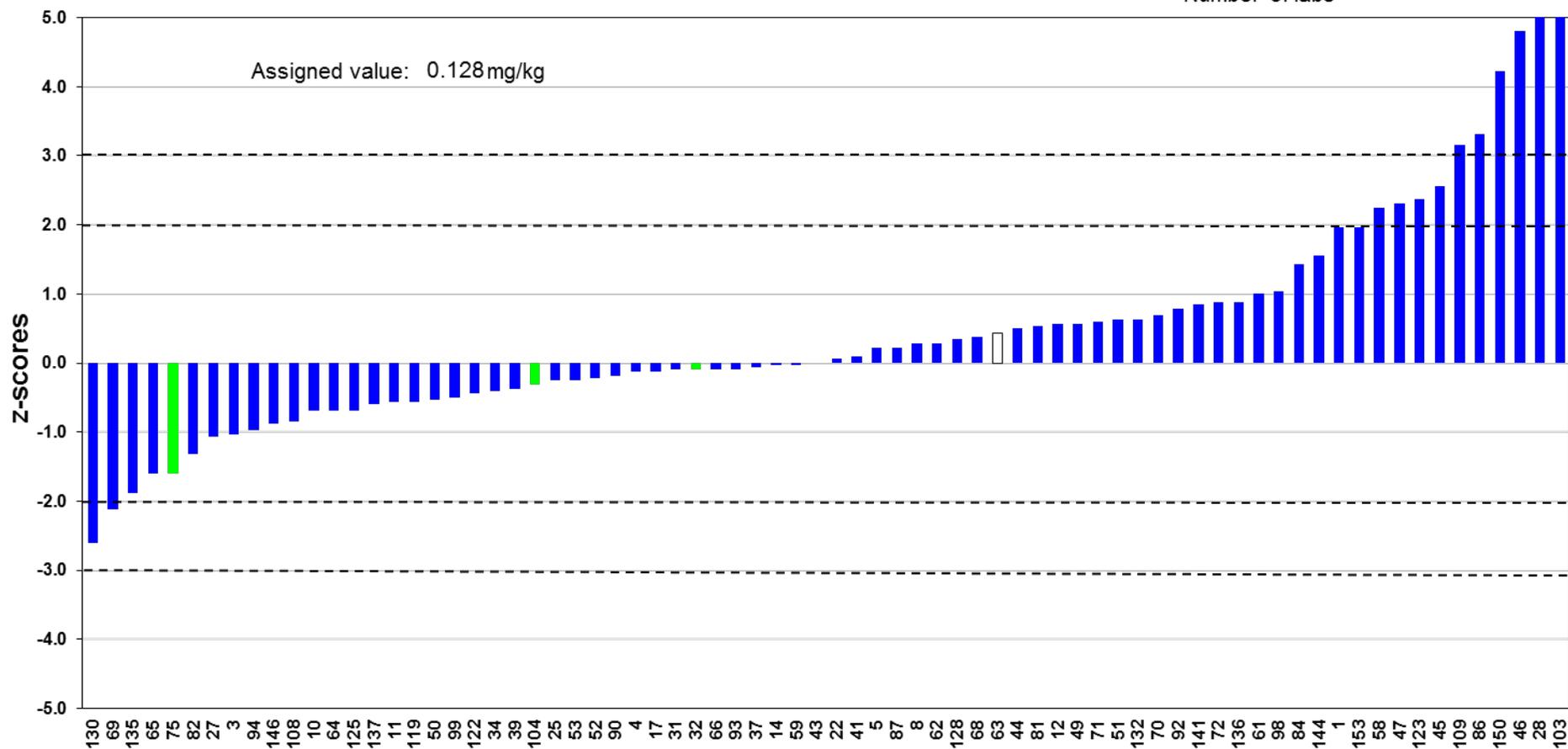
Acceptable	102
Questionable	5
Unacceptable	4
Number of labs	111



# Diflubenzuron

- Water added
- No water added
- Not specified

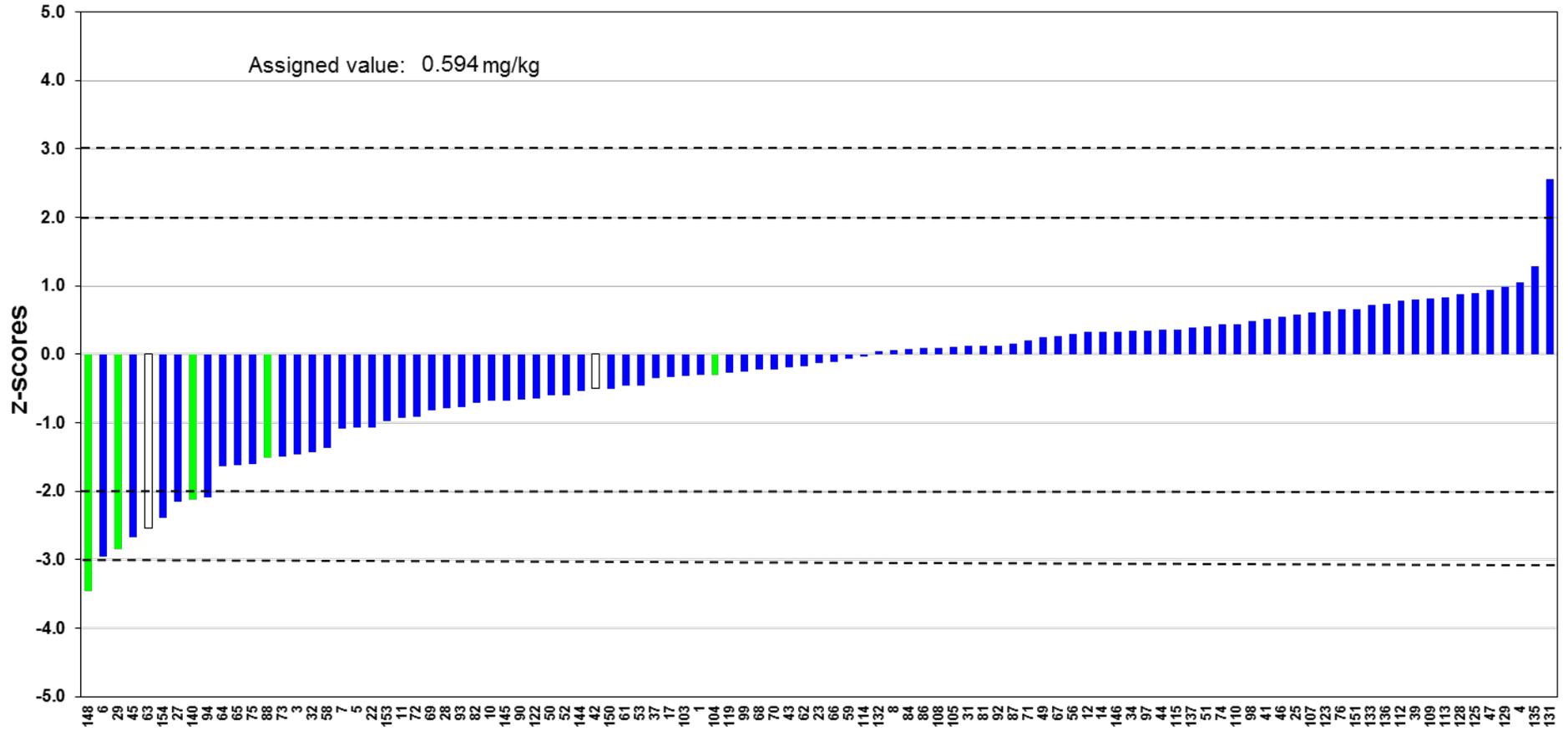
Acceptable	62
Questionable	6
Unacceptable	6
Number of labs	74



# Epoxiconazole

- Water added
- No water added
- Not specified

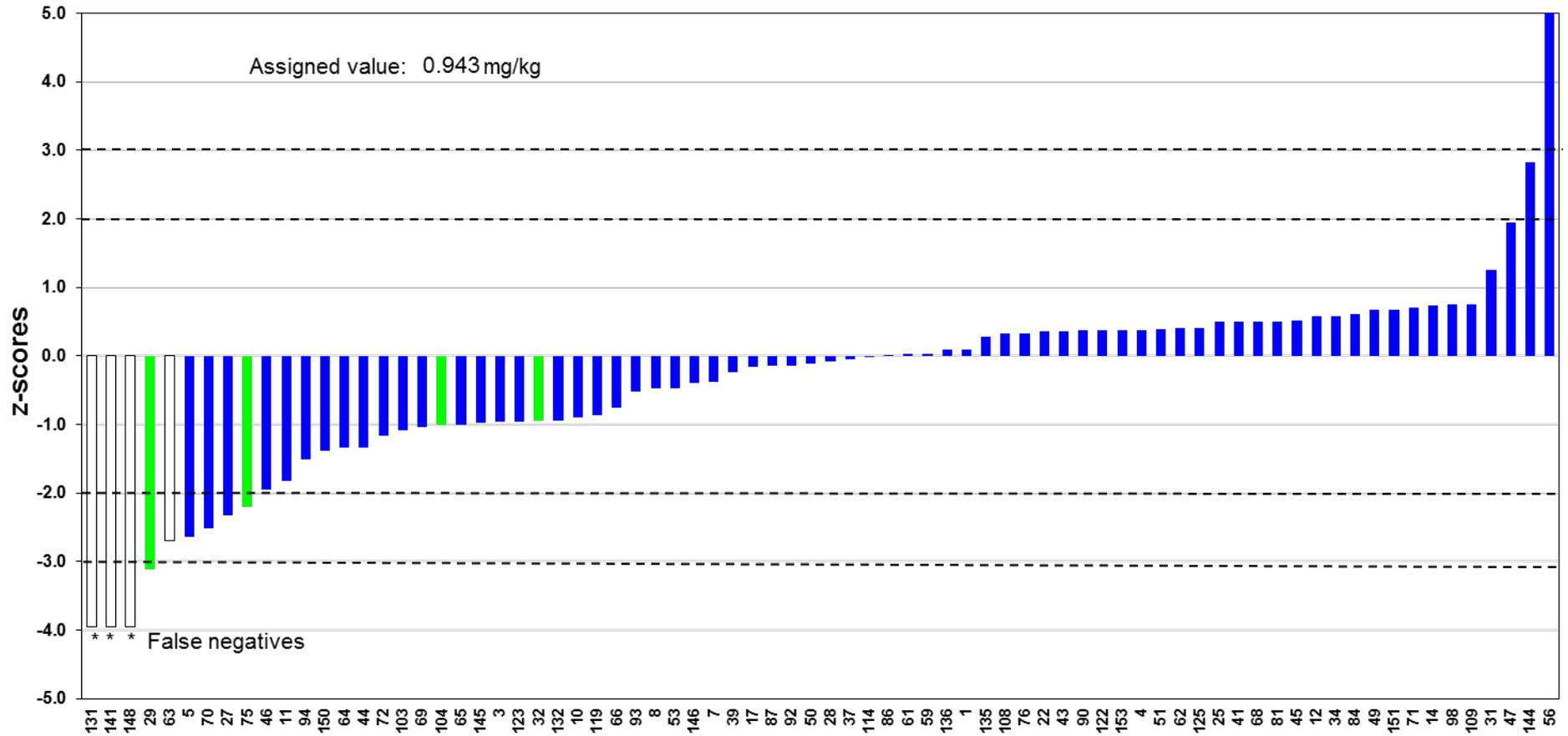
Acceptable 89  
Questionable 9  
Unacceptable 1  
Number of labs 99



# Fenpropidin

- Water added
- No water added
- Not specified

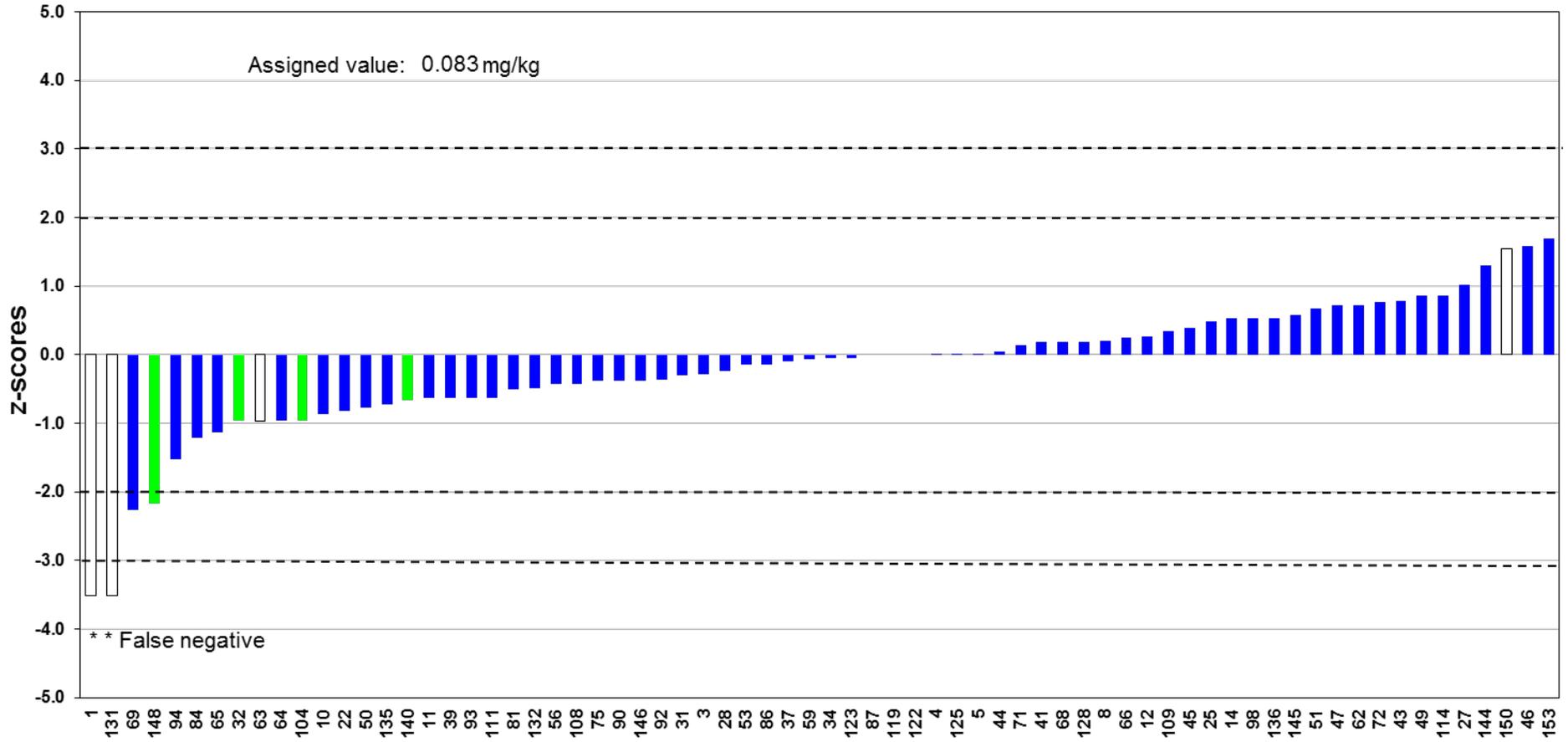
Acceptable	65
Questionable	6
Unacceptable	5
Number of labs	76



# Isoprothiolane

- Water added
- No water added
- Not specified

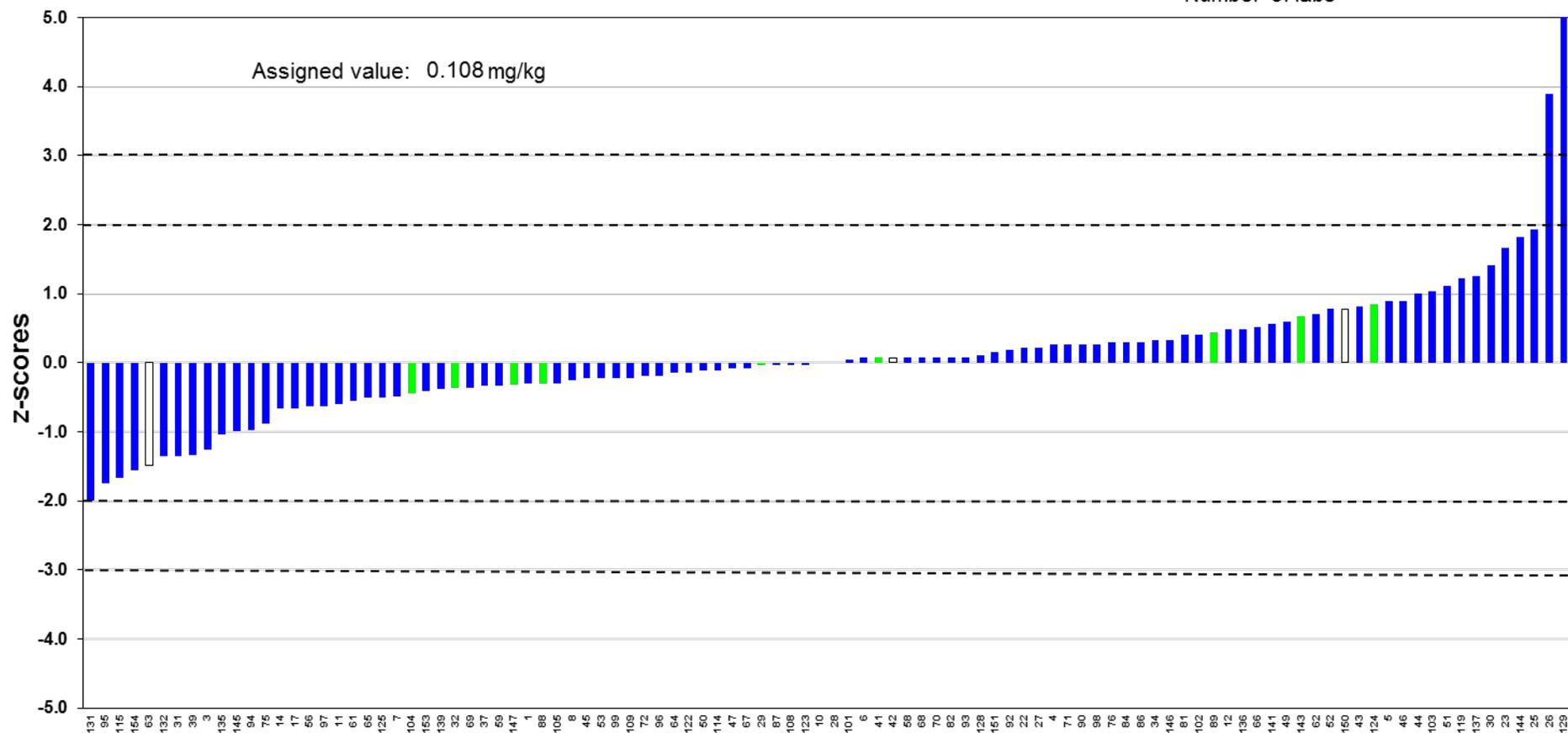
Acceptable	66
Questionable	2
Unacceptable	2
Number of labs	70



# Pendimethalin

- Water added
- No water added
- Not specified

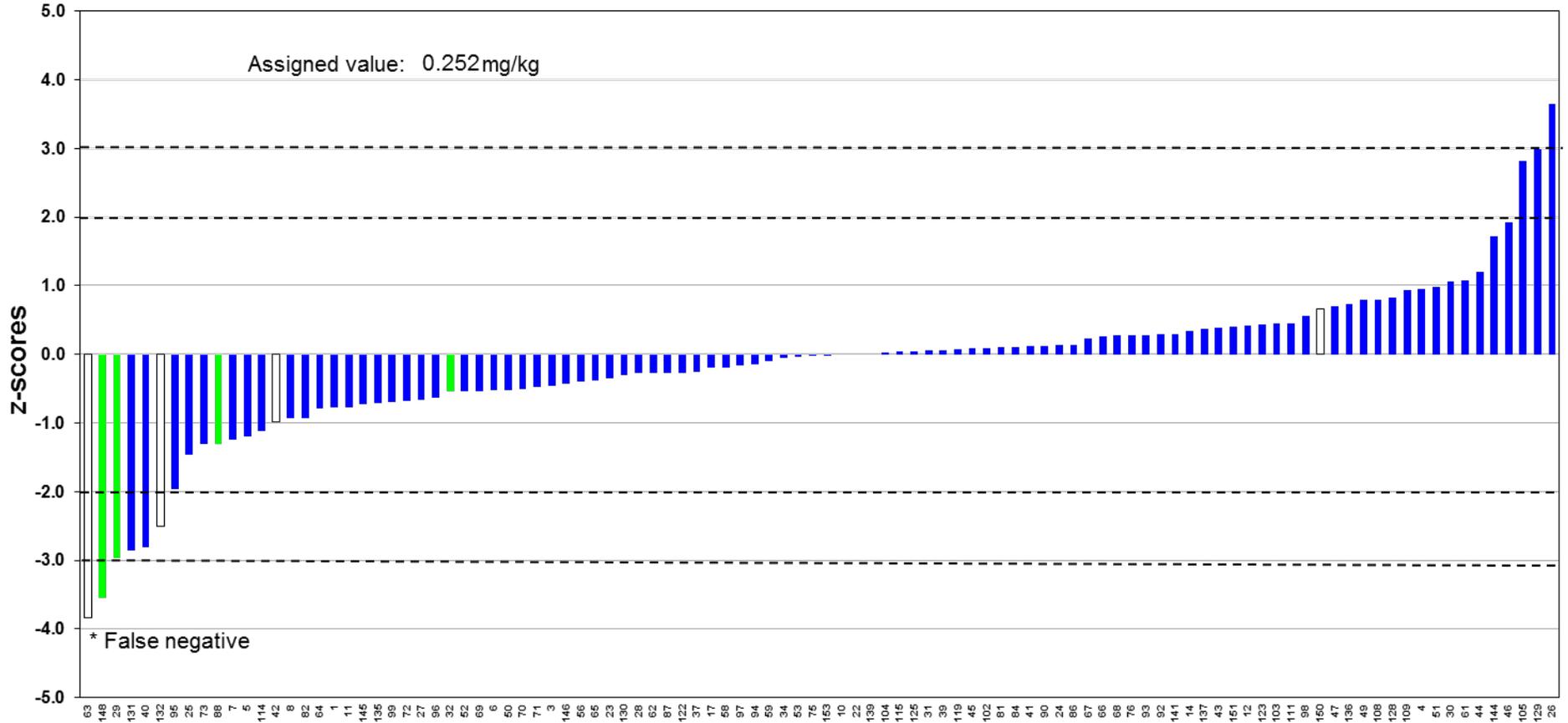
Acceptable	99
Questionable	1
Unacceptable	2
Number of labs	102



Water added  
No water added  
Not specified

### Pirimicarb

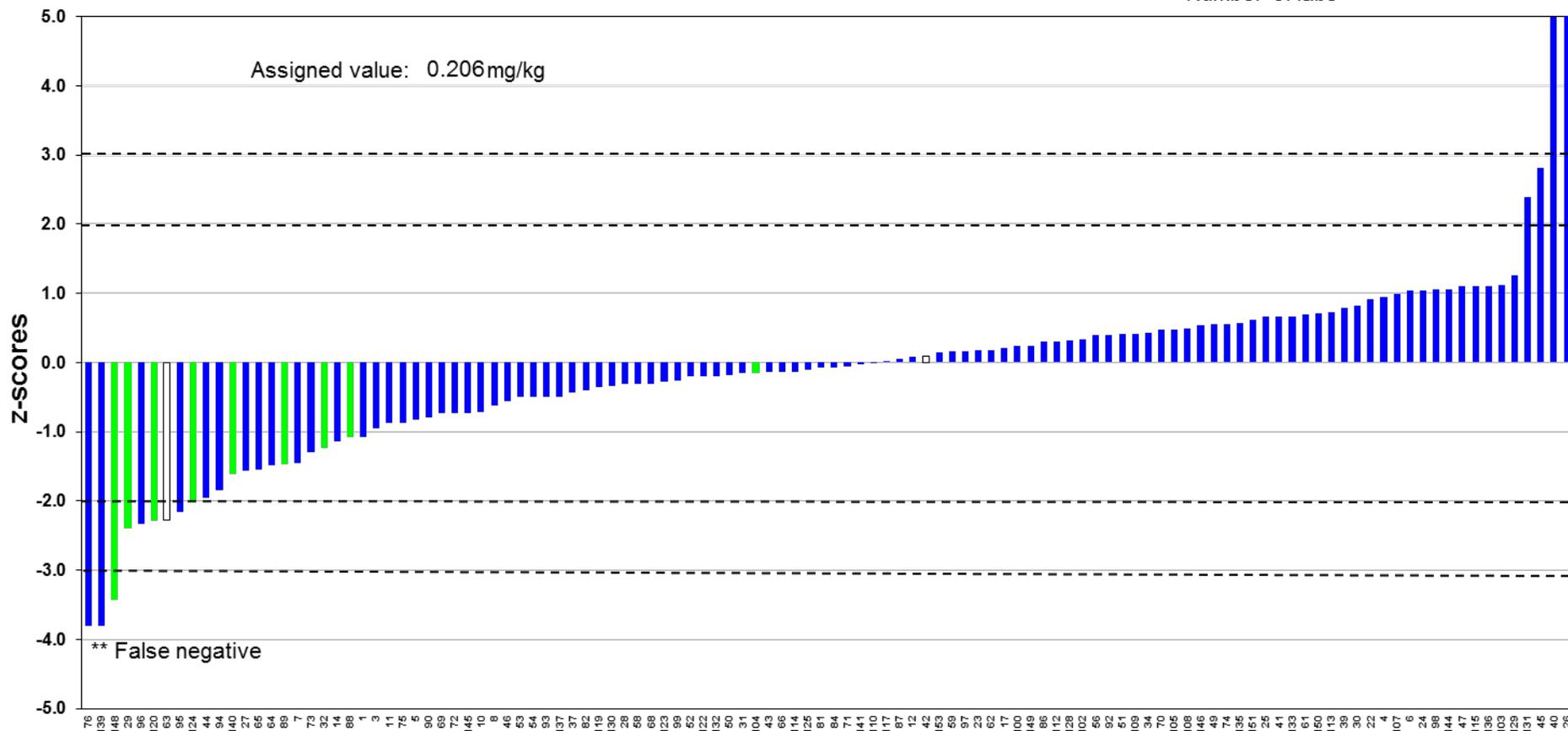
Acceptable 93  
Questionable 6  
Unacceptable 3  
Number of labs 102



# Propiconazole

- Water added
- No water added
- Not specified

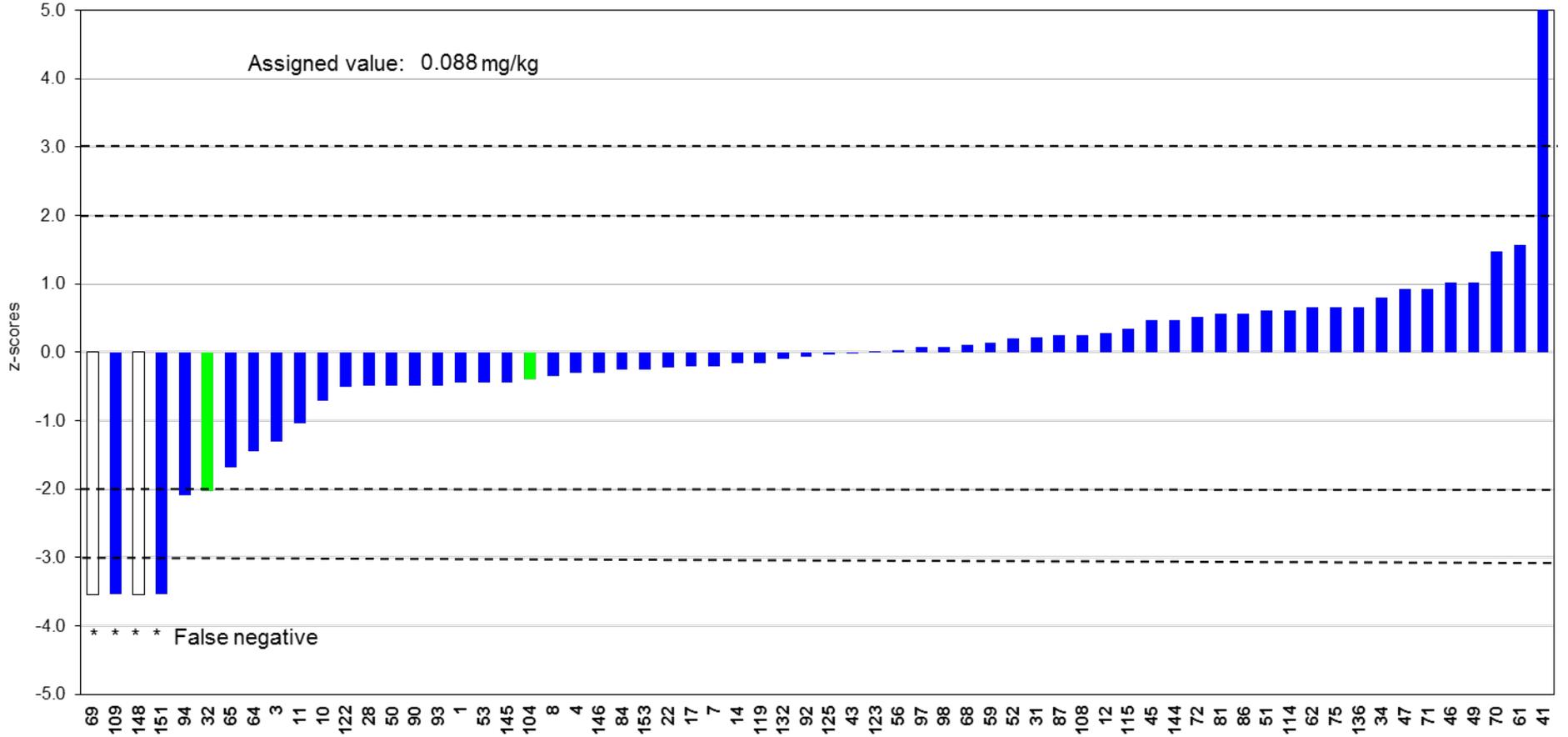
Acceptable	101
Questionable	8
Unacceptable	5
Number of labs	114



### Prothioconazole-desthio

Acceptable	57
Questionable	2
Unacceptable	5
Number of labs	64

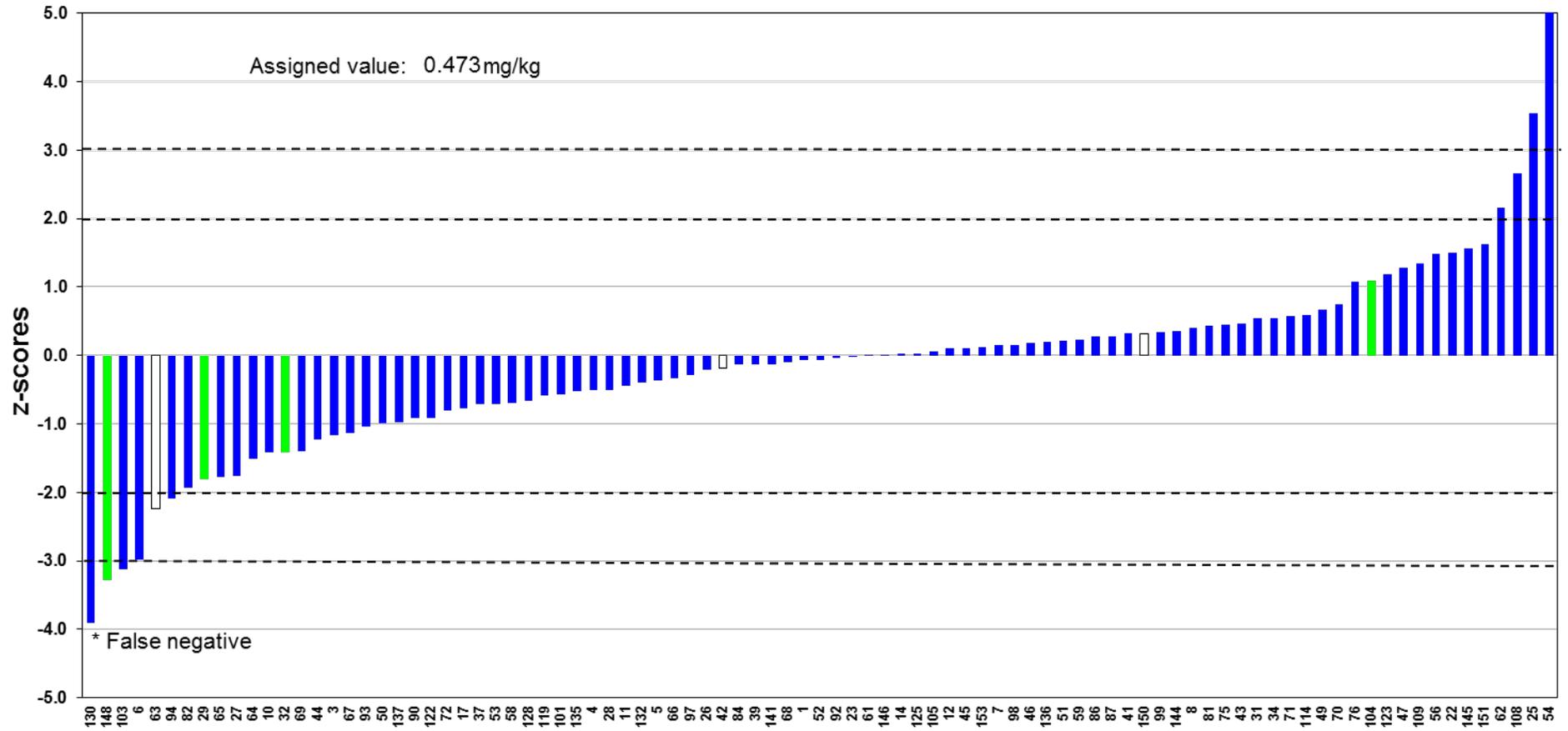
■ Water added  
■ No water added  
□ Not specified



# Pyraclostrobin

- Water added
- No water added
- Not specified

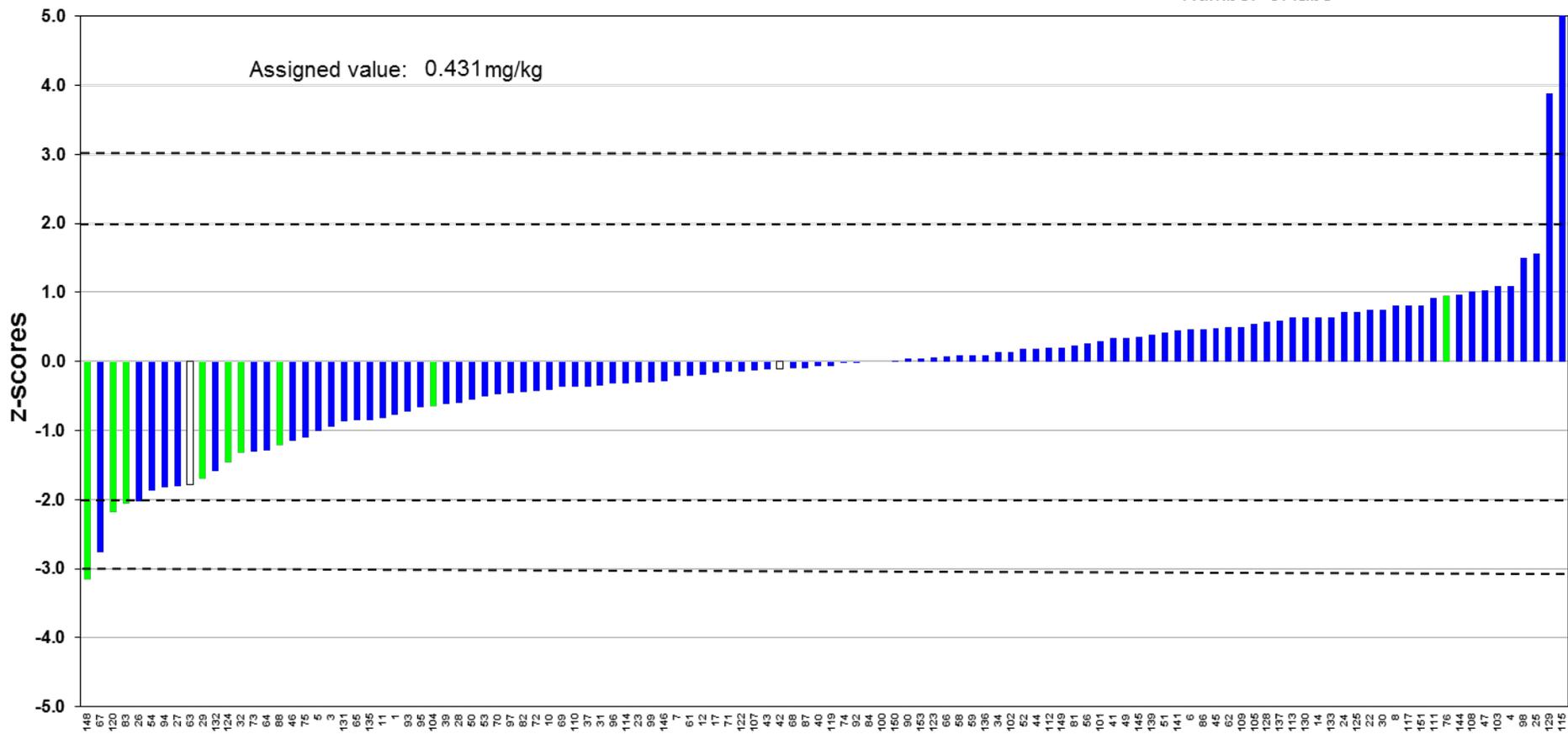
Acceptable	81
Questionable	5
Unacceptable	5
Number of labs	91



# Tebuconazole

Acceptable	109
Questionable	4
Unacceptable	3
Number of labs	116

- Water added
- No water added
- Not specified



## ANNEXES



# GENERAL PROTOCOL

## for EU Proficiency Tests for Pesticide Residues in Food and Feed

### Introduction

This protocol contains general procedures valid for all European Union Proficiency Tests (EUPTs) organised on behalf of the European Commission, DG-SANCO<sup>1</sup> by the four European Union Reference Laboratories (EURLs) for pesticide residues in food and feed. These EUPTs are directed at all National Reference Laboratories (NRLs) and Official Laboratories (OfLs) within the EU Member States. Laboratories outside of this EURL/NRL/OfL-Network<sup>2</sup> may be permitted to participate on a case-by-case basis after consultation with DG-SANCO.

The following four EURLs for pesticide residues were appointed by DG-SANCO based on regulation 882/2004/EC<sup>3</sup>:

- 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 Single Residue Methods (EURL-SRM)

NRLs are appointed by Member State based on the provisions of Regulation 882/2004/EC, whereas OfLs are laboratories that are actively involved in official controls following Article 26 of Regulation 396/2004/EC (e.g. by conducting pesticide residue analyses within the framework of national and/or EU-controlled programmes).

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<sup>1</sup> DG-SANCO = European Commission, Health and Consumer Protection Directorate-General

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

<sup>3</sup> Regulation (EC) No 882/2004 of the European Parliament and of the Council on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules. Published at OJ of the EU L191 of 28.05.2004

3rd Edition

Approved: January 2012

According to Article 28 (3) of Regulation 396/2005/EC<sup>4</sup>, all laboratories analysing samples for the official control of pesticide residues shall participate in the European Union Proficiency Test(s) organised by the European Union. The aim of these EUPTs is to obtain information regarding the quality, accuracy and comparability of the pesticide residue data in food and feed sent to the European Union within the framework of the national control programmes and the co-ordinated multiannual community control programme<sup>5</sup>. Participating laboratories will be provided with an assessment of their analytical performance and the reliability of their data – compared to the other participating laboratories.

### EUPT-Panel

EUPTs are organised by individual EURLs or by more than one EURL in joint cooperation.

An **Organising Team** is appointed from the EURL(s) in charge. This team is responsible for all administrative and technical matters concerning the organisation of the PT, e.g. PT-announcement; Test Item production; undertaking the homogeneity and stability tests; packing and shipment of Test Item, as well as the handling and first assessment of participants' results.

Approved by DG SANCO, expert scientists with long-term experience in pesticide residue analysis will be chosen as members of a joint **EUPT-Scientific Committee** (SC). This Committee is made up of the following two subgroups:

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

The SC's role is to help the organisers make decisions regarding the EUPT design: the selection of pesticides to be included in the Target Pesticide List (see below); the establishment of the Minimum Required Reporting Levels (MRRLs); the evaluation and statistical treatment of the results and the drafting of the protocol and final report. The

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<sup>4</sup> 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.

<sup>5</sup> European Commission Proficiency Tests for Pesticide Residues in Fruits and Vegetables, Trends in Analytical Chemistry, 2010, 29 (1), 70-83.

3rd Edition

Approved: January 2012

QCG has the additional function of supervising the quality of the EUPT and to assist the EURL in confidential aspects such as the choice of the pesticides to be present in the Test Item and the concentration levels at which they should be present in the Test Item.

The EUPT-Organising Team and the EUPT-Scientific Committee (the AG and the QCG) together form the **EUPT-Panel**.

The present EUPT General Protocol was drafted by the EUPT-Panel and was approved by DG-SANCO.

### **EUPT Participants**

All NRLs operating in the same area as the organising EURL are legally obliged to participate in EUPTs - as well as all OfLs whose scope overlaps with that of the EUPT. The four EURLs will be annually issuing and distributing via the EURL website, a joint list of all OfLs that shall participate in all EUPTs to be conducted within a given year. The “list of obliged labs” is to be considered as tentative as it will be only based on information submitted by OfLs concerning their commodity scope and status. The legal obligation of NRLs and OfLs to participate in EUPTs arises from:

- Art. 28 of Reg. 396/2005/EC (for all OfLs analyzing for pesticide residues within the framework of official controls in food or feed)
- Art. 33 of Reg. 882/2004/EC (for all NRLs)

If necessary the “list of obliged labs” will be updated within the same year to take account of any changes in the lab profiles.

NRLs are responsible for checking whether all relevant OfLs within their network are included in the list of obliged laboratories and whether the contact information is correct.

The NRLs should further make arrangements to urge all relevant OfLs within their network to participate in all EUPT relevant to them.

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

Any OfL not intending to participate in a given EUPT will have to explain to the EURL its reasons for non-participation without prejudice of any legal action taken against it for not

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participating. This also applies to initially participating laboratories that do not deliver results.

Official labs from EFTA countries and EU-candidate countries are also welcome to participate in the EUPTs. In special cases, the Organisers, upon consultation with DG-SANCO, will also allow laboratories outside of the EURL/NRL/OfL-Network to participate in EUPTs.

### **Confidentiality**

The proprietor of all EUPT data is DG-SANCO and thus has access to all information.

In each EUPT, the laboratories are given a unique code, initially only known to themselves and the Organisers. In the final EUPT-Report, the list of participating laboratories will not be linked to their laboratory codes. It should be noted that the organisers, at the request of DG-SANCO, may present the EUPT-results to the Standing Committee on the Food Chain and Animal Health on a country-by-country basis. It is therefore possible that a link between codes and laboratories could be made, especially for those countries where only one laboratory has participated.

As laid down in Regulation 882/2004, NRLs are responsible for evaluating and improving their own OfL network. For this reason, the EURLs will provide the OfL laboratory codes to their NRLs together with the final report. This will allow NRLs to correlate the laboratories within their network and their performance. Furthermore, the EURLs reserve the right to share EUPT results and codes among themselves: for example, for the purpose of evaluating overall lab performance as requested by DG-SANCO.

### **Communication**

The official language used in all EUPTs is English.

Communication between participating laboratories during the test on matters concerning this PT exercise is not permitted.

## **Announcement / Invitation Letter**

The announcement of the individual EUPT will be issued at least 3 months before the Test Item is distributed to the laboratories. The announcement will be published on the EURL portal and additionally distributed via e-mail to the NRL/OfL mailing list available to the EURLs. The announcement will contain an invitation letter, details on how to register and where to find additionally-related documents, as well as some preliminary information on the specific protocol such as the tentative calendar, the name of the commodity expected to be used, and the tentative Target Pesticide List.

## **Target Pesticide List**

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

In some cases, that will be clearly marked, results calculated according to the pesticide residue definition may be requested with those residue definitions differing from the legal ones in certain cases.

## **Specific Protocol**

For each EUPT a Specific Protocol will be published at least 2 weeks before the Test Item is distributed to the laboratories. This protocol will contain all the information previously included in the Invitation Letter but in its final version, in addition to information on payment for delivery service and/or participation. It will furthermore include instructions on how to handle the Test Item upon receipt, on how to submit results, and any other relevant information.

## **General procedures for reporting results**

Laboratories are responsible for reporting their results to the Organiser within the stipulated deadlines. Any pesticide that was targeted by a participating laboratory should be reported as “analysed”. Each laboratory must report only one result for each of the analytes detected in the Test Items, using the analytical procedure(s) that they

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would routinely use for each compound for monitoring purposes. The residue levels of the pesticides detected should be expressed in mg/kg and in some cases for products of animal origin in µg/kg fat.

One Test Item is intentionally treated with pesticides and one is not. Both Test Items have to be analysed by the laboratories and any pesticide detected in them shall be reported.

### **Correction of results for recovery**

According to the Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed, (Document SANCO), it is common practice that pesticide analysis results are not corrected for recovery, but may be corrected if the average recovery is significantly different from 100% (typically if outside the 70-120% range with good precision), therefore, if residue data are adjusted for recovery, then this must be indicated on the specific field of the 'reporting result form'. Laboratories are required to report whether their results were adjusted for recovery and, if this was the case, the recovery (as percentage) used should be also reported. No recovery data are required where correction for recovery results automatically from using the 'standard addition(s)' approach, or isotopically-labelled internal standards (in both cases with spiking of the Test Item at the beginning of the extraction procedures). In these cases, the laboratories should report the calculation technique used for the results instead of the recovery data.

### **Methodology information**

All laboratories are requested to provide information on the analytical method(s) they have used. If no sufficient information on the methodology used is provided, the Organiser reserves the right not to accept the analytical results reported by the participants concerned.

### **Results evaluation**

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

– ***False Positives***

These are results reported above the MRRLs that suggest the presence of pesticides that were listed in the Target Pesticide List, but which were: (i) not detected by the Organiser, even after repeated analyses, and/or (ii) not detected by the overwhelming majority (e.g. 95%) of the participating laboratories that had targeted the specific pesticide. However, in certain instances, case-by-case decisions by the EUPT-Panel may be necessary.

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

– ***False Negatives***

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

In cases of the assigned value being less than a factor of 4 times the MRRL, false negatives will not be assigned as this is not statistically justifiable.

– ***Estimation of the true concentration ( $\mu$ )***

The “true” concentration (assigned value) will be typically estimated using the median of all the results. In special justifiable cases, the EUPT-Panel may decide to use only part of the population of results to establish the median (e.g. only results with z-scores  $\leq 5.0$ , or by excluding results generated by a method that demonstrably generates significantly biased results, e.g. due to incomplete extraction).

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

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

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

The percentage FFP-RSD is set at 25% based on experience from previous EUPTs<sup>6</sup>. 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.

– **z-scores**

This parameter is calculated using the following formula:

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

Where:  $x_i$  is the value reported by the laboratory,  $\mu_i$  the assigned value, and  $\delta_i$  the standard deviation at that level for each pesticide (i).

Any z-scores of > 5 will be reported as >5 and where combined z-scores are calculated a value of “5” will be used.

z-Scores will be interpreted in the following way:

$ z  \leq 2$	Acceptable
$2 <  z  \leq 3$	Questionable
$ z  > 3$	Unacceptable

For results that are considered to be false negatives, z-scores will be calculated using the MRRL or RL (the laboratory’s Reporting Limit) if the RL < MRRL.

<sup>6</sup> 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.

The EUPT-Panel will consider whether, or not, these values should appear in the z-score histograms.

z-Scores will not be calculated for any false positive result.

– **Category A and B classification**

The EUPT-Panel will decide whether to classify the laboratories into two groups - A or B. Laboratories that detect a sufficiently high percentage of the pesticides present in the Test Item (e.g. at least 90%) and reported no false positives will have demonstrated ‘sufficient scope’ and will therefore be classified into Category A. The 90% criterion will be applied following Table 1.

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

No. of Pesticides Present in the Sample (N)	90%	No. of Pesticides needed to be detected to have sufficient scope (n)	n
3	2.7	3	N
4	3.6	4	
5	4.5	4	
6	5.4	5	N - 1
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	
16	14.4	14	
17	15.3	15	
18	16.2	16	
19	17.1	17	
20	18.0	18	
21	18.9	19	
22	19.8	20	
23	20.7	21	
24	21.6	22	N - 3
25	22.5	22	
26	23.4	23	

For evaluation of the overall performance of laboratories within Category A, the Average of the Squared z-Score ( $AZ^2$ )<sup>7,8</sup> will be used.

Laboratories within Category B will be ranked according to the total number of pesticides present in the sample. The number of acceptable z-scores achieved will be presented too. The EURL-Panel retains the right to calculate combined z-scores (see below) also for Category B labs, e.g. for informative purposes, provided that a minimum number of results (z-scores) is available.

#### – Combined z-scores

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

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

This formula multiplies each z-score by itself and not by an arbitrary number. Based on the  $AZ^2$  achieved, the laboratories are classified as follows:

Formula	Good	Satisfactory	Unsatisfactory
$AZ^2$	$\leq 2$	$2 < AZ^2 \leq 3$	$AZ^2 > 3$

Combined z-scores are considered to be of lesser importance than the individual z-scores. The EUPT-Panel retains the right not to calculate  $AZ^2$  if it is considered as not being useful. In the case of EUPT-SRMs, where only few results per lab are available, the Average of the Absolute z-scores (AAZ) will be calculated for informative purposes, but only for labs within Category A and as long as 5 or more z-scores are available.

<sup>7</sup> Formerly named “Sum of squared z-scores ( $SZ^2$ )”

<sup>8</sup> Laboratory assessment by combined z-score values in proficiency tests: experience gained through the EUPT for pesticide residues in fruits and vegetables. Anal. Bioanal. Chem., 2010, 397, 3061–3070.

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### **Publication of results**

The EURLs will publish a preliminary report, containing tentative medians and z-score values for all pesticides present in the test sample, within 2 months from the deadline for result submission.

The Final Report will be published after the EUPT-Panel has discussed the results. Taking into account that the EUPT-Panel meets normally only once a year to discuss the results of all EUPTs organised annually by the EURLs in the running year, the final report may be published up to 8 months after the deadline for results submission.

### **Certificates of participation**

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

### **Feedback**

After the distribution of the final report of an EUPT, participating laboratories will be given the opportunity to give their feedback to the Organiser and make suggestions for future improvements.

### **Follow-up activities**

Laboratories are expected to undertake follow-up activities to trace back to the source of any erroneous or (strongly) deviating results - including all false positives and false negatives, along with results with  $|z| > 2$ .

Upon request, the laboratory's corresponding NRL, or EURL, are to be informed of the outcome of these traceability activities.

According to instructions by DG-SANCO, the "Protocol for management of underperformance in comparative testing and/or lack of collaboration of National

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Reference Laboratories (NRLs) with EU Reference Laboratories (EURLs) activities” will be followed for NRLs.

**Disclaimer**

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

**Laboratory Rights**

After the Final Report has been sent, the laboratories will have the right to communicate the nonconformity of their result evaluation in written form. Any detected errors in the preliminary report should also be reported to the Organiser. The Organiser, assisted by the Scientific Committee, will decide upon any re-evaluation and will give a corresponding explanation.

# SPECIFIC PROTOCOL

## for the EU Proficiency Test for Pesticide Residues in Cereals using Multi-Residue Methods, EUPT-C6 (2012)

(last updated: 18.01.2012 – new email address for contact about invoice)

### Introduction

This protocol is complementary to the [General Protocol for EU Proficiency Tests for Pesticide Residues in Food and Feed](#). The current proficiency test covers pesticides that are determined by Multi-Residue Methods. This EUPT is to be performed by all National Reference Laboratories for Cereals and Feeding stuffs (NRL-CFs) as well as by all official EU laboratories (OfLs) responsible for official pesticide residue controls on cereals and feeding stuff, as far as their scope overlaps with that of the EUPT-C6. The commodity barley is to be considered as representative for commodities with “high starch and/or protein content and low water and fat content” (see [SANCO document 12495/2011](#)).

### Test Item (Test Material)

This proficiency test concerns the analysis of pesticide residues in barley. The barley was grown in Denmark in 2011 and pesticides were applied in the field. Following harvest, the rye was also spiked with some additional pesticides.

In addition, a blank Test Item is also provided, that can be used for recovery experiments as well as for the preparation of matrix-matched calibration standards for both MRM and SRM-pesticides. However, the blank Test Item must also be analysed and possible detected pesticides reported.

The Organizers will check the Test Items for sufficient homogeneity and for stability at conditions reproducing sample shipment and storage during the duration of the test. The blank Test Item will be also checked to prove that the target analytes are not contained at any relevant levels. All these tests will be conducted by the EURL-CF that is ISO 17025 accredited.

### **Analytical Parameters**

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

Laboratories should carefully read the Target Pesticides List, where important information about reporting of results, as well as the Minimum Required Reporting Levels (MRRLs) is given. The Target Pesticides List contains only individual compounds and results should only be reported for individual compounds, no matter how the residue definitions are set.

The MRRL values will be used to help identify false positive and false negative results and for the calculation of z-scores for false negatives.

It should not be assumed that only pesticides registered for use on barley are present in the Test Item.

### **Amount of Test Item**

The participants will receive:

- approximately 100 g of barley Test Item with incurred and spiked pesticides and
- approximately 100 g of blank barley Test Item.

### **Shipment of Test Items**

The Test Items are planned to be shipped on 30 January, 2012.

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

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

### **Instructions on Test Item Handling**

Once received, the Test Items should be stored deep frozen (at  $-18^{\circ}\text{C}$  or less) before analysis to avoid any possible deterioration/spoilage and to minimize pesticide losses. The Test Items should be mixed thoroughly, before taking the analytical portion(s).

All participants should use their own routine standard operating procedures for extraction, clean-up and analytical measurement and their own reference standards for identification and quantification purposes. Considering the available amount of Test Item, laboratories employing methods requiring large analytical portions are advised to scale them down. As the test material is already milled and sufficiently homogeneous, method sensitivity is the only major factor to consider when deciding about the size of the analytical portion.

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

### **Results Submission Website and Deadlines**

Sample receipt acknowledgement, analytical results and method information are to be submitted via the [EUPT-C6 Result Submission Website](http://thor.dfvf.dk/eupt-c6) (<http://thor.dfvf.dk/eupt-c6>).

This website will be accessible from 30 January 2012 onwards and also contains a link to specific instructions on how to enter the data in the result submission website.

To access the data submission forms participants must use their unique login data (username and password) given in the confirmation e-mails sent to the laboratories upon registration.

The labs can fill-in the sub-pages at different stages/sessions. **Remember to save the data of each page before leaving it.**

**The deadline for result submission is 27 February at 14.00 CET**

### Test Item Receipt and Acceptance - Subpage 0

Once the laboratory has received the Test Items it must report to the organiser, via the [EUPT-C6 Result Submission Website](#) the date of receipt, the condition of the Test Item, and its acceptance. The deadline for acceptance is the 3 February 2012. If the laboratory does not respond by this deadline the Organisers will assume that the Test Items have been received and accepted. **If any participants have not received the Test Items by the 2 February at noon, they must inform the Organiser immediately by e-mail** ([crlicereal@food.dtu.dk](mailto:crlicereal@food.dtu.dk)).

## Reporting Qualitative and Quantitative Results - Subpages 1 and 2

To report their results, laboratories must access the [EUPT-C6 Result Submission Website](#).

**Deadline: All results must be reported on the online result submission website by 27 February at 14:00 p.m., at the latest.** The website will not be accessible for result submission after this date, and any results reported after the deadline will not be included in the statistical treatment, or in the final report.

Results should not be reported where a pesticide was not detected, or was detected below the RL (Reporting Limit) of the laboratory, or below the MRRL. Results reported as <RL will be considered as „Not Detected“.

The results (residue levels of the pesticides detected) must be expressed in mg/kg.

Significant Figures:

Residue levels <0.010 mg/kg;

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

Residue levels  $\geq$  0.010 mg/kg;

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

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

- **“Concentration in mg/kg”**: here the results should be filled-in, that you would report in your routine work. That means, the recovery-corrected result should be reported, if it reflects the normal procedure in your lab otherwise the non-recovery-corrected result should be reported.
- **“Conc. in blank in mg/kg”**: any concentration values of pesticides from the Target Pesticides List you will determine in the blank (even at levels below the MRRL) you can enter here.
- **“Experience with this compound”**. Use the dropdown-menu to indicate how many years you have analysed for this compound using the method applied in this EUPT.
- **“Is your result recovery-corrected?”**: Please specify whether the result was recovery-corrected and the kind of recovery-correction via the dropdown-menu.

- **“Recovery figure (in %)”**: Here labs can report any recovery figures (in %) obtained for the analyte in question. If a recovery factor was used to correct for recovery, the recovery figure (in %) used for the calculation MUST be reported.

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

#### Reporting Information on Analytical Methodology - Subpage 3

All laboratories are requested to provide information on the analytical method(s) they have used via the [EUPT-C6 Result Submission Website](#). The laboratories are asked to thoroughly fill-in this important information in order to minimize the administrative burden of collecting this information a posteriori.

#### Reporting missing information after result submission deadline – Subpage 4

In case of false negative results the affected laboratories will be asked to provide details on the methodology used after the deadline for result submission. This can be done by accessing subpage 4 within the [EUPT-C6 Result Submission Website](#). The dates this subpage will be accessible will be announced in due time. If the page is empty when you access subpage 4, no further information is needed from you and you can leave the page without any further actions.

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

#### **Follow-up actions**

According to instructions by DG-SANCO, 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](#)” will be followed for NRLs.

#### **Documents**

All documents relating to EUPT–C6 can be found in the EURL-Documents Repository ([CIRCA/FIS-VL](#)). Links to the documents can be found in the [EUPT–C6 Website](#).

**Calendar** (see also [http://www.crl-pesticides.eu/library/docs/cf/EUPT\\_C5\\_SRM6\\_Target.pdf](http://www.crl-pesticides.eu/library/docs/cf/EUPT_C5_SRM6_Target.pdf))

<b>Activity</b>	<i>Who ?</i>	<i>Dates</i>
Access to " <b>EUPT-Registration Website</b> "	EURL-CF	15 December 2011
Deadline for registration	Invited Labs	15 January 2012
Release of Specific Protocol	EURL-CF	January 2012
Preparation of Test Material	EURL-CF	January 2012 (final preparation)
Homogeneity tests	EURL-CF	January 2012
Stability tests	EURL-CF	February 2012
<ul style="list-style-type: none"> <li>▪ <b>Distribution of Test materials</b></li> <li>▪ Information to the laboratories regarding shipment</li> </ul>	EURL-CF	<b>30 January 2012</b>
Activation of " <b>EUPT-C6 Result Submission Website</b> "	EURL-CF	30 January 2012
<b>Deadline for Receipt and Acceptance of Test Materials:</b> Online Submission of Form 0 (sub-page 0)	Participating Labs	within 24 hr of receipt and not later than 3 February 2012
<b>Deadline for Result Submission</b> <b>Pesticide scope, Results, Method Information</b> Submission of Form 1 – 3 (sub-pages 1 – 3)	Participating Labs	<b>27 February 2012 at 14.00 CET</b>
EUPT Evaluation Meeting	EURL-FV, EURL-SRM, Commission, EUPT- Scientific Committee	September 2012
Preliminary Report (only compilation of results)	EURL-CF	May 2012
Final Report	EURL-CF	December 2012

### **Participation Fees**

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

- 175 €

The participation fees for laboratories from third countries:

- 350 €

For further information visit the website [www.eurl-pesticides.eu](http://www.eurl-pesticides.eu)

### **Delays in Payment**

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

Any question concerning invoices must be directed to Carina Hillingsoe Groelsted at the financial department [cah@adm.dtu.dk](mailto:cah@adm.dtu.dk)

## Contact information

### **EURL-CF**

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