

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



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

**EUPT-CF7  
2013**



**EU PROFICIENCY TESTS  
EUPT-CF7, 2013**

**Incurred and Spiked Pesticide  
Residues in a Feed for Laying Hens**

**Final Report**

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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 multi-annual co-ordinated control and national monitoring programmes. Participation in the proficiency test scheme “European Union Proficiency Tests (EUPTs) for pesticide residues” is mandatory according to Article 28 of Regulation 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 first organized within the frame of the EURL activities with feed matrix as Test Items. However, six previous PTs with cereals as Test Items were organised and these were; EUPT-C1/SRM2 on wheat (2007), EUPT-C2 on wheat (2008), EUPT-C3/SRM4 on oats (2009), EUPT-C4 on rye (2010), EUPT-C5/SRM6 on rice (2011) and EUPT-C6 on barley. The PTs in 2007, 2009 and 2011 were jointly organised by the EURL-CF and EURL-SRM using and focusing on both MRM and SRM pesticides, whereas the present EUPT-CF7 on feed for laying hens (2013) was only focused on MRM-pesticides. The feed Test Item used for EUPT-CF7 was treated with 23 compounds partly in the field and partly post-harvest in the laboratory

Participation in EUPT-CF7 was compulsory for all National Reference Laboratories (NRLs) and Official Laboratories (OfLs) within the EU involved in the determination of pesticide residues in feeds using multiresidue methods for their national programmes. Official laboratories from EFTA countries (Iceland, Norway and Switzerland), as well as official laboratories from EU-candidate states (Croatia, FYROM, Turkey and Serbia) were invited to take part in this EUPT. Selected laboratories from Third Countries were also allowed to take part in this exercise, but their results, together with the EU-candidate state laboratories, were not used when establishing the Assigned Values. 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.

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





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# EUROPEAN COMMISSION EURL PROFICIENCY TEST ON PESTICIDE RESIDUES IN CEREALS EUPT-CF7, 2013

## 1. INTRODUCTION

On 10 January 2013 the announcement of the 1<sup>th</sup> European Commission's Proficiency Test on an animal feedstuff (EUPT-CF7) was published on the EURL homepage, together with the Calendar and the Pesticide Target List including all compounds that could potentially be present in the Test Item. The Target Pesticides List included 116 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, the Specific protocol (**Annex 2**) as well as a list of labs that are obliged to take part in the EUPT-CF7 was provided via the homepage. Laboratories were able to register on-line from the 20 March to 15 April 2013. In total 106 laboratories from EU and EFTA countries agreed to participate in the test as well as 14 laboratories from EU-Candidate States and Third Countries.

The present proficiency test was performed using a feed for laying hens. The feed was based on cereals, which had been treated with pesticide formulations in the field. However, the Test Item was spiked with additional pesticides at the facilities of the EURL-CF. The Test Item contained 23 compounds in total. The Danish Centre for Food and Agriculture at Aarhus University (Denmark) performed the field treatments and Research Institute of Feed Technology of the International Research Association of Feed Technology (Germany) produced the feed Test Item and Blank Test Item from raw materials provided from EURL-CF. The field treated cereals and the pesticides used for additional spiking were selected by the EURL-CF and the EURL Quality Control Group. Furthermore, the stabilities of the pesticides in the treated feed 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 100 g portions of the treated feed Test Item and 100 g of untreated Blank Test Item. Both Test Items were shipped to participants on 13 May 2013 and the deadline for submission of results to the Organiser was the 10 June 2013. The participants were asked to analyse the treated Test Item as well as the Blank Test Item and report the concentrations of any pesticide residues found 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 treated Test Item:

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

- Cold water was added to a milled portion of the treated test item 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.

## 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 EURL Quality Control Group, taking into account the present and upcoming scope of the EU multi-annual coordinated control programme, a pesticide priority list ranking the pesticides according to their relevance and risk-potential, as well as a list of pesticides particularly relevant to the feed. Consequently, 9 pesticides from Directive 2002/32 on undesirable substance in animal feed were included in the list. 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

A feed was produced at Research Institute of Feed Technology of the International Research Association of Feed Technology (Germany), IFF Braunschweig, by milling, mixing and pelleting from 100 kg raw materials shipped from EURL-CF. Field treated cereals that were left over from earlier EUPTs were selected due to the suitability as ingredients in feed for laying hens as well as the incurred pesticide levels. The field spraying was originally performed by the Danish Centre for Food and Agriculture at Aarhus University. Other ingredients were provided from the feed producer DLG (Denmark). The composition of the feed can be seen in **Table 1**. The pesticides that were used for treatment either in the field or in the lab can be seen in **Table 2**. However, it should be noted that additional pesticides were used for field treatment but due to dilution when producing the feed, the resulting pesticide levels were too low to assess in this PT. The pesticide used for spiked either to increase the incurred pesticides were spiked into the soya bean oil.

After receiving the feed from IFF Braunschweig, 35 kg of treated feed pellets were thoroughly mixed to ensure a high degree of homogeneity. Subsequently, the feed pellets were milled separately as four 4 kg 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 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

A feed was produced at Research Institute of Feed Technology of the International Research Association of Feed Technology (Germany), IFF Braunschweig, by milling, mixing and pelleting from 80 kg raw materials shipped from EURL-CF. The raw materials used to prepare the feed for the blank Test Item was based on untreated cereals 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 and organically produced ingredients from DLG (Denmark). As it is not possible to obtain organically produced soybean meal, soybean cake was used instead and the fat content re-adjusted by adding the relevant amount of soybean oil. After receiving the feed from IFF Braunschweig, 25 kg of the feed was milled and 100 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.** Composition of the feed Test Items.

Ingredients	Test Item	Blank Test Item
Wheat	25%	25%
Barley	13%	12%
Rye	13%	12%
Maize	13%	12%
Soybean meal	25%	
Soybean cake		27%
Lime	9%	9%
Soybean oil	3%	1%
Mineral and vitamin premix	1%	1%
<b>Total amount</b>	<b>100%</b>	<b>100%</b>

**Table 2** Pesticides used for application in the field and/or spiked into the soybean oil in the laboratory

Pesticide	Application on cereals in the field				Spiked in lab into soybean oil
	Wheat from EUPT-C1	Wheat from EUPT-C2	Rye from EUPT-C4	Barley from EUPT-C6	
Aldrin					x
Azoxystrobin	x	x	x		
Boscalid				x	
Carbendazim		x	x		
Chlorpyrifos-methyl		x	x		x
Cypermethrin		x			x
Endosulfan-alpha					x
Endosulfan-sulfate					x
Epoxiconazole		x		x	
Fenpropidin				x	
Fenpropimorph			x		
Fluquinconazole			x		
Flutriafol			x		
Iprodione		x			x
Kresoxim-methyl			x		
Lindane					x
Malathion			x		x
Propiconazole				x	x
Pyraclostrobin				x	
Spiroxamine		x	x		
Tebuconazole				x	
Triadimenol		x			
Trifloxystrobin		x			

Residues of eight additional pesticides were present in the Test Item below the 0.04 mg/kg. The pesticide residues were bifenthrin, deltamethrin, diazinon, difenoconazole, lambda-cyhalothrin, pirimicarb, pirimiphos-methyl and prothioconazole-dethio. These pesticides were not included in the evaluation because the levels were below 0.04 mg/kg. The results, median of the results as well as a pseudo z-score are listed in **Appendix 6**.

### 1.5 Homogeneity test

Eleven bottles of pesticide treated Test Items 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 3**. 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.  $\sigma_{all}^2 = 0.3 \times \text{FFP RSD (25\%)} \times \text{the analytical sampling mean for all pesticides}$ , 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 EUPT-CF7.

### 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: 14 May 2013

Day 2: 21 May 2013

Day 3: 28 May 2013

Day 4: 2 June 2013

Day 5: 9 June 2013

Along with the stability test samples the Test Item no 213 was analysed. This sample was originally shipped to a laboratory in Brazil but rejected at the border and was shipped back to EUURL-CF.

The average results from each stability test for the MRM pesticides are given in **Table 4** (page 16). 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**. Likewise, neither the stability test performed by storage at room temperature nor the Test Item 213 showed any significant decrease.

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

**Table 3** Statistical evaluation of the homogeneity test data (n=22 analyses using a sub-sample of 5 g in each case).  $S_s$ : Between Sampling Standard Deviation

Pesticide	Mean, mg/kg	$S_s^2$	c	$S_s^2 < c$ Pass/Fail
Aldrin	0.329	0.00011	0.0026	Pass
Azoxystrobin	0.106	0.00014	0.0001	Pass
Boscalid	0.132	0.00000	0.0004	Pass
Carbendazim	0.228	0	0.0008	Pass
Chlorpyrifos-methyl	0.244	0.00003	0.0010	Pass
Cypermethrin	0.236	0	0.0017	Pass
Endosulfan-alpha	0.232	0.00004	0.0007	Pass
Endosulfan-sulfate	0.312	0	0.0019	Pass
Epoxiconazole	0.129	0	0.0003	Pass
Fenpropidin	0.101	0.00005	0.0002	Pass
Fenpropimorph	0.352	0	0.0020	Pass
Fluquinconazole	0.099	0	0.0002	Pass
Flutriafol	0.317	0.00003	0.0019	Pass
Iprodione	0.322	0	0.0021	Pass
Kresoxim- methyl	0.070	0.00000	0.0001	Pass
Lindane	0.286	0.00002	0.0017	Pass
Malathion	0.259	0	0.0014	Pass
Propiconazole	0.213	0.00001	0.0006	Pass
Pyraclostrobin	0.084	0	0.0001	Pass
Spiroxamin	0.181	0.00001	0.0007	Pass
Tebuconazole	0.070	0	0.0001	Pass
Triadimenol	0.236	0.00001	0.0009	Pass
Trifloxystrobin	0.053	0.00001	0.0000	Pass

## 1.7 Organisational details

### 1.7.1 Access to documents, registration and confidentiality

In the invitation letter of 10 January 2013, all NRLs and OfLs were prompted to register using the online registration link from 18 March to 15 April 2013. All documents related to this EUPT (Calendar, Target Pesticides List, Specific Protocol, General Protocol) were uploaded to the EURL website and the CIRCA platform. Laboratories that were intending not to participate were given the opportunity to explain the reasons for their non-participation. Participants from third countries also had access to another online registration link after contacting the EURL. After registration, the participants were provided with 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 13 May 2013, one bottle of treated Test Item (100 g) and one bottle of Blank Test Item (100 g) were shipped to all participants in insulated polystyrene boxes containing a freezer block. The laboratories were asked to check the state of the sample on receipt and to enter the website (see above) and communicate

whether they accept/not accept the Test Items. Test Items for Third Countries were shipped one week earlier due the often very time-consuming custom procedures at the borders.

### 1.7.3 Submission of results

An online submission tool was developed that allowed participants to submit their results via the internet. All participants had access to the result-submission website (<http://thor.dvfv.dk/ptc>) from a few days after shipment until the result-submission deadline (10 June 2013). Participants were asked not only to report their analytical results, but also to give information regarding accreditation, reporting limits and details regarding the methods they used to analyse the Test Items.

**Table 4** Stability test results at -18 °C and at room temperature

Pesticide	-18 °C (mg/kg, n=3)						Room temperature (mg/kg, n=3)		
	Day 1	Day 2	Day 3	Day 4	Day 5		Day 1	Day 2	Day 5
Aldrin	0.317	0.324	0.329	0.316	0.343	pass	0.317	0.332	0.357
Azoxystrobin	0.126	0.120	0.137	0.129	0.117	pass	0.126	0.127	0.111
Boscalid	0.162	0.157	0.173	0.172	0.155	pass	0.162	0.160	0.137
Carbendazim	0.233	0.211	0.227	0.245	0.212	pass	0.233	0.226	0.217
Chlorpyrifos-methyl	0.237	0.237	0.265	0.251	0.227	pass	0.237	0.239	0.223
Cypermethrin	0.300	0.302	0.302	0.307	0.290	pass	0.300	0.308	0.294
Endosulfan-alpha	0.248	0.239	0.287	0.263	0.241	pass	0.248	0.248	0.231
Endosulfan-sulfate	0.324	0.313	0.330	0.351	0.326	pass	0.324	0.330	0.288
Epoxiconazole	0.135	0.132	0.126	0.137	0.133	pass	0.135	0.134	0.137
Fenpropidin	0.077	0.063	0.075	0.075	0.059	pass	0.077	0.073	0.070
Fenpropimorph	0.380	0.359	0.445	0.415	0.380	pass	0.380	0.394	0.368
Fluquinconazole	0.110	0.110	0.120	0.114	0.105	pass	0.110	0.110	0.101
Flutriafol	0.293	0.311	0.337	0.321	0.280	pass	0.293	0.349	0.318
Iprodione	0.348	0.347	0.385	0.380	0.329	pass	0.348	0.345	0.320
Kresoxim- methyl	0.074	0.061	0.065	0.068	0.066	pass	0.074	0.068	0.085
Lindane	0.268	0.266	0.327	0.282	0.264	pass	0.268	0.279	0.296
Malathion	0.298	0.288	0.344	0.314	0.281	pass	0.298	0.297	0.292
Propiconazole	0.232	0.232	0.245	0.245	0.234	pass	0.232	0.230	0.217
Pyraclostrobin	0.098	0.087	0.094	0.102	0.093	pass	0.098	0.095	0.094
Spiroxamin	0.201	0.183	0.190	0.199	0.185	pass	0.201	0.191	0.199
Tebuconazole	0.081	0.075	0.093	0.077	0.076	pass	0.081	0.079	0.082
Triadimenol	0.280	0.291	0.300	0.307	0.237	pass	0.280	0.280	0.246
Trifloxystrobin	0.053	0.066	0.059	0.050	0.058	pass	0.053	0.070	0.051



## 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 assigned values for each pesticide are based on the median level of the results reported by EU and EFTA countries laboratories, excluding outliers. Individual results without any numerical values reported, such as detected (D), were not considered. The spread of results for each pesticide was tested for multimodality. Taking into account the normative for robust analysis in ISO 13528 [3], the uncertainty was accompanying the assigned value for each pesticide, which was calculated according to the following equation:

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

Where:

- u is the uncertainty in mg/kg.
- s\* is the robust standard deviation estimate
- n is the total number of laboratories giving a result for each pesticide, excluding outliers.

### 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

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

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

Where:

- $x_i$  is the value reported by the laboratory
- $\mu_i$  the assigned value
- $\delta_i$  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.

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

The EUPT-Panel will 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. 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 also be presented. 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) are 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:

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

where "n" is the number of each laboratory's z-scores that were considered in this formula.

For the calculation, any z-score  $> 5$  was set at "5".

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  $AZ^2$  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  $AZ^2$  is considered to be of lesser importance than the individual z-scores. Therefore the organiser, in agreement with the EUPT-Panel, retains the right not to use them if they are considered to be unhelpful.

### 3. RESULTS

#### 3.1 Summary of reported results

In total, 106 EU and EFTA laboratories, representing 29 countries (27 EU member states), agreed to participate in this proficiency test. Malta was represented by UK NRL. Three NRLs did not register and three participants (one NRL) did not submit data. Additionally, 14 Third Countries registered for the PT. Two samples did not reach the laboratories due to difficulties with custom clearance and two laboratories did not submit results. The participating laboratories are listed in **Appendix 1**.

An overview of results submitted by laboratories from the EU and EFTA can be seen in **Table 5**. All reported analytical results can be seen in **Table 11-13**, and the methods used are published as **Appendix 7** but in a separate electronic file. However, only results submitted by laboratories from EU and EFTA countries are included in **Table 5-10** and in the z-scores histograms in **Appendix 5**.

**Table 5** Overview of number of results, number of not analysed (NA), number of not detected (ND=false negatives) and the percentage of laboratories that reported results for the pesticides in the treated Test Item.

Pesticide	No. of reported results	No. of NA <sup>1)</sup>	False negatives <sup>2)</sup>	% results <sup>3)</sup>
Aldrin	95	8	3	92
Azoxystrobin	87	16	3	84
Boscalid	86	17		83
Carbendazim	69	34		67
Chlorpyrifos-methyl	94	9		91
Cypermethrin	92	11	2	89
Endosulfan-alpha	100	3	5	97
Endosulfan-sulfate	95	8	2	92
Epoxiconazole	81	22	1	79
Fenpropidin	62	41	4	60
Fenpropimorph	78	25		76
Fluquinconazole	78	25	1	76
Flutriafol	79	24	5	77
Iprodione	82	21		80
Kresoxim-methyl	88	15	4	85
Lindane	97	6	3	94
Malathion	94	9	1	91
Propiconazole	88	15		85
Pyraclostrobin	69	34	5	67
Spiroxamine	69	34	2	67
Tebuconazole	87	16	2	84
Triadimenol	78	25	4	76
Trifloxystrobin	84	19	3	82

1) NA = not analysed

2) False negatives = not detected

3) '% results' have been calculated using the number of laboratories that reported results for each particular compound and the total number of EU laboratories that submitted results (n = 103). False negatives are included in reported results.

Aldrin, chlorpyrifos-methyl, endosulfan-alpha, endosulfan-sulfate and lindane were the most frequently analysed compounds with 90 % or more of the labs submitting results for these compounds. Azoxystrobin,

boscalid, cypermethrin, iprodione, kresoxim-methyl, propiconazole, tebuconazole and trifloxystrobin were analysed by more than 80% of the participants and carbendazim, epoxiconazole, fenpropidin, fenpropimorph, fluquinconazole, flutriafol, pyraclostrobin, spiroxamine and triadimenol only reported by 60-79% of participants.

### 3.1.1 *False positives*

Eleven participants from EU and EFTA countries reported 13 results above the MRRL for additional pesticides that had not been used to treat the Test Item (see **Table 6**). 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.

Four laboratories reported 9 results below the MRRL for additional pesticides (see **Table 7**). These results were not considered to be false positives. However, all the results were also below the laboratories reporting limits. Four of the results were for HCH-beta which may co-elute with lindane on the GC. Two of these four labs did not report results for lindane. Additionally, one lab reported 2 results below the MRRL but did not report the laboratory's reporting limits. Furthermore, one lab reported to have detected methacrifos in the Blank Test Item. This was not judged as a false positive result.

As mentioned in section 1.4, residues of eight additional pesticides were present in the treated Test Item below the 0.04 mg/kg. The pesticide residues were bifenthrin, deltamethrin, diazinon, difenoconazole, lambda-cyhalothrin, pirimicarb, pirimiphos-methyl and prothioconazole-desthio. These pesticides were not included in the evaluation because the levels were below 0.04 mg/kg. The results, median of the results as well as a pseudo z-score are listed in Appendix 6.

### 3.1.2 *False negatives*

Missing results for pesticides actually present in the treated Test Item were judged as false negatives. **Table 8** summarizes the number of reported false negatives for each pesticide. Fifty results were judged as false negatives, which represents 3 % of the total number of results. Around 20 % of the participants (22 laboratories) reported false negative results. This is equivalent to the first EUPT on cereals (wheat) where 20-30% of the labs reported false negative results. No false negatives results were reported for boscalid, carbendazim, chlorpyrifos-methyl, fenpropimorph, iprodione and propiconazole.

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

Lab code	Pesticide	Concentration mg/kg	Determination technique	RL, mg/kg	MRRL, mg/kg
6	Trifluralin	0.072	LC-MS/MS QQQ	0.01	0.01
31	p'p'-DDT	0.063	GC- (μ) ECD	0.01	0.01
31	Quinoxifen	0.046	GC-Ion Trap	0.01	0.01
34	HCH-beta	0.239	GC-MS/MS (QQQ)	0.01	0.01
35	Hexaconazole	0.018	LC-MS/MS QQQ	0.01	0.01
68	HCH-beta	0.270	GC-MS/MS (QQQ)	0.005	0.01
91	Chlorpyrifos	0.021	GC-MS/MS (QQQ)	0.01	0.01
91	Pendimethalin	0.017	LC-MS	0.01	0.01
122	p'p'-DDT	0.015	GC-Ion Trap	0.015	0.01
125	Permethrin	0.020	<sup>1</sup>	<sup>1</sup>	0.01
126 <sup>2</sup>	HCH-beta	0.092	GC-MSD	0.01	0.01
129	HCH-beta	0.150	GC-MSD	0.005	0.01

<sup>1</sup> No information received.

<sup>2</sup> This laboratory also reported that they found 0.05 mg/kg methacrifos in the Blank Test Item.

**Table 7** False positive results below 0.01 mg/kg, the concentration detected in mg/kg, the determination technique used, the reporting level and the MRRL in mg/kg.

Lab code	Pesticide	Concentration mg/kg	Determination technique	RL, mg/kg
27	Pirimicarb-desmethyl	0.006	LC-MS/MS QQQ	0.01
61	Chlorpyrifos	0.0011	GC-MS/MS (QQQ)	0.005
61	Pirimicarb-desmethyl	0.0041	LC-MS/MS QQQ	0.005
61	Prochloraz	0.0014	LC-MS/MS QQQ	0.005
61	Triadimefon	0.0014	LC-MS/MS QQQ	0.005
67	HCH-alpha	0.0024	GC-MS/MS (QQQ)	0.01
67	Pirimicarb-desmethyl	0.0056	LC-MS/MS QQQ	0.01
113	Malaoxon	0.0008	<sup>1</sup>	0.01
113	Pirimicarb-desmethyl	0.0075	LC-MS/MS QQQ	0.01
125	Chlorpyrifos	0.001	<sup>1</sup>	<sup>1</sup>
125	Pendimethalin	0.001	<sup>1</sup>	<sup>1</sup>

<sup>1</sup> No information received.

**Table 8** False negative results (FN).

Lab code	Aldrin	Azoxystrobin	Cypermethrin	Endosulfan-alpha	Endosulfan-sulfate	Epoxiconazole	Fenpropidin	Fluquinconazole	Flutriafol	Kresoxim-methyl	Lindane	Malathion	Pyraclostrobin	Spiroxamine	Tebuconazole	Triadimenol	Trifloxystrobin
12	FN			FN												FN	
13																	FN
19									FN								
28									FN								
30	FN		FN	FN													FN
34											FN						
42															FN	FN	
43										FN							
44	FN			FN	FN						FN						FN
52		FN											FN			FN	
55							FN										
56								FN		FN			FN				
68							FN			FN	FN			FN			
87				FN													
102		FN	FN									FN					
106									FN						FN		
112							FN						FN				
113						FN				FN						FN	
117													FN				
124									FN								
126		FN					FN		FN					FN			
129				FN	FN								FN				

### 3.2 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 using a mixture of water and 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.

Thirty results were regarded as outliers ( $z$ -score  $\geq 5$ ); Aldrin (1.09 and 1.11 mg/kg) carbendazim (0.582, 0.739, 0.848, 0.975 and 1.015 mg/kg), chlorpyrifos-methyl (0.585 and 1.215 mg/kg), cypermethrin (0.675 mg/kg), endosulfan-alpha (0.604, 0.65 and 0.656mg/kg), endosulfan-sulfate (0.7 and 0.802mg/kg), epoxiconazole (0.35 mg/kg), fenpropimorph (0.766 mg/kg), fluquinconazole (0.422mg/kg), iprodione (0.8 and 0.921mg/kg), kresoxim-methyl (0.149 and 0.305 mg/kg), lindane (0.665 mg/kg), malathion (1.28 mg/kg), propiconazole (0.525 mg/kg), pyraclostrobin (0.519 mg/kg), spiroxamine (0.45 mg/kg) and tebuconazole (0.201, 0.221 and 0.363 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 15-29 % but on average the Qn-RSD was 20 %, 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 Treated Test Item.

Pesticides	MRRL (mg/kg)	Assigned value (mg/kg)	Uncertainty, mg/kg	FFP RSD (%)	Qn RSD (%)
Aldrin	0.01	0.405	0.010	25	20
Azoxystrobin	0.01	0.115	0.003	25	19
Boscalid	0.01	0.149	0.004	25	19
Carbendazim	0.01	0.252	0.011	25	29
Chlorpyrifos-methyl	0.01	0.252	0.007	25	22
Cypermethrin	0.01	0.283	0.008	25	21
Endosulfan-alpha	0.01	0.220	0.005	25	20
Endosulfan-sulfate	0.01	0.307	0.008	25	19
Epoxiconazole	0.01	0.121	0.003	25	20
Fenpropidin	0.01	0.171	0.008	25	29
Fenpropimorph	0.01	0.333	0.011	25	24
Fluquinconazole	0.01	0.107	0.003	25	18
Flutriafol	0.01	0.308	0.008	25	18
Iprodione	0.01	0.325	0.009	25	21
Kresoxim-methyl	0.01	0.060	0.001	25	16
Lindane	0.01	0.279	0.006	25	17
Malathion	0.01	0.316	0.007	25	18
Propiconazole	0.01	0.217	0.007	25	15
Pyraclostrobin	0.01	0.096	0.003	25	21
Spiroxamine	0.01	0.176	0.006	25	23
Tebuconazole	0.01	0.076	0.002	25	20
Triadimenol	0.01	0.230	0.006	25	17
Trifloxystrobin	0.01	0.057	0.001	25	15

### 3.3 Assessment of laboratory performance

#### 3.3.1 Z-scores

Z-scores have been calculated for all the quantified pesticides using the FFP RSD of 25 %. **Table 10** shows an overview of the acceptable, questionable and unacceptable z-scores and **Tables 11-13** show the individual results and z-scores for each laboratory and pesticide together with the assigned values. A graphical representation of the z-scores (for EU and EFTA countries) can be seen in **Appendix 5**. Results for the additional six pesticide residues at low levels can be seen in **Appendix 6**.

**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
Aldrin	85	4	6	3
Azoxystrobin	77	6	4	3
Boscalid	82	4	0	
Carbendazim	59	4	6	
Chlorpyrifos-methyl	89	2	3	
Cypermethrin	85	3	4	2
Endosulfan-alpha	87	3	10	5
Endosulfan-sulfate	87	4	4	2
Epoxiconazole	70	8	3	1
Fenpropidin	52	4	6	4
Fenpropimorph	71	4	3	
Fluquinconazole	72	3	3	1
Flutriafol	71	1	7	5
Iprodione	76	4	2	
Kresoxim-methyl	82	0	6	4
Lindane	88	4	5	3
Malathion	90	1	3	1
Propiconazole	84	2	2	
Pyraclostrobin	58	2	9	5
Spiroxamine	62	4	3	2
Tebuconazole	77	3	7	2
Triadimenol	70	4	4	4
Trifloxystrobin	73	5	6	3

For boscalid, chlorpyrifos-methyl, cypermethrin, endosulfan-sulfate, fenpropimorph, fluquinconazole, flutriafol, iprodione, kresoxim-methyl, lindane, malathion, propiconazole, spiroxamine and triadimenol acceptable results were obtained by 90-96% of the laboratories. For aldrin, azoxystrobin, carbendazim, endosulfan-alpha, epoxiconazole, fenpropidin, pyraclostrobin, tebuconazole and trifloxystrobin acceptable results were obtained by 84-89% of the laboratories.

**Table 11** Results for aldrin, azoxystrobin, boscalid, carbendazim, chlorpyrifos-methyl, cypermethrin endosulfan-alpha and endosulfan-sulfate in mg/kg, the corresponding z-scores, the MRRLs and the assigned values.

Laboratory code	Aldrin	Azoxystrobin		Boscalid		Carbendazim		Chlorpyrifos-methyl		Cypermethrin		Endosulfan-alpha		Endosulfan-sulfate		
MRRL	0.01	0.01		0.01		0.01		0.01		0.01		0.01		0.01		
Assigned value	0.405	0.115		0.149		0.252		0.252		0.283		0.220		0.307		
		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	0.27	-1.3	0.1	-0.7	0.14	-0.2	0.25	0.0	0.16	-1.5	0.23	-0.7	0.16	-1.1	0.26	-0.6
2			0.0	-3.1	0.0572	-2.5	0.114	-2.2	0.175	-1.2	0.675	>5	0.332	2.0		
3	0.423	0.2	0.1	-0.2	0.126	-0.6	0.221	-0.5	0.172	-1.3	0.228	-0.8	0.197	-0.4	0.233	-1.0
5	0.508	1.0	0.1	0.2	0.161	0.3	0.278	0.4	0.278	0.4	0.278	-0.1	0.267	0.9	0.347	0.5
6	0.419	0.1	0.2	1.3	0.168	0.5	0.848	>5	0.271	0.3	0.325	0.6	0.181	-0.7	0.245	-0.8
7	0.326	-0.8	0.1	0.0	0.147	-0.1	0.204	-0.8	0.238	-0.2	0.243	-0.6	0.207	-0.2	0.324	0.2
9	0.411	0.1	0.1	-0.7	0.127	-0.6	0.211	-0.7	0.204	-0.8	0.219	-0.9	0.223	0.1	0.275	-0.4
11	0.24	-1.6							0.08	-2.7	0.05	-3.3				
12	FN	-3.9											FN	-3.8	0.245	-0.8
13	0.328	-0.8	0.1	0.2					0.226	-0.4	0.26	-0.3	0.202	-0.3	0.298	-0.1
14	0.455	0.5	0.1	-0.3	0.136	-0.3	0.193	-0.9	0.282	0.5	0.319	0.5	0.255	0.6	0.326	0.2
15	0.483	0.8	0.1	-0.4	0.129	-0.5	0.397	2.3	0.275	0.4	0.292	0.1	0.243	0.4	0.291	-0.2
16	0.448	0.4	0.1	0.1	0.141	-0.2	0.259	0.1	0.253	0.0	0.22	-0.9	0.19	-0.5	0.31	0.0
17	0.311	-0.9	0.1	0.1	0.179	0.8	0.275	0.4	0.174	-1.2	0.259	-0.3	0.176	-0.8	0.306	0.0
18	0.388	-0.2	0.1	-0.1	0.177	0.8			0.313	1.0	0.364	1.2	0.21	-0.2	0.334	0.4
19	0.377	-0.3	0.1	0.2	0.17	0.6	0.582	>5			0.275	-0.1	0.211	-0.2		
20	0.359	-0.4	0.1	0.0	0.17	0.6	0.202	-0.8	0.293	0.7	0.345	0.9	0.23	0.2	0.348	0.5
21	0.49	0.9	0.1	0.3	0.166	0.5	0.358	1.7	0.35	1.6	0.29	0.1	0.259	0.7	0.52	2.8
22	0.476	0.7							0.235	-0.3	0.379	1.4	0.218	0.0	0.334	0.4
23	0.418	0.1	0.1	-0.2	0.152	0.1	0.294	0.7	0.216	-0.6	0.282	0.0	0.233	0.2	0.423	1.5
24	0.388	-0.2	0.1	-0.3	0.154	0.1			0.326	1.2	0.286	0.0	0.201	-0.3	0.33	0.3
25	0.476	0.7	0.1	0.4	0.162	0.3	0.266	0.2	0.221	-0.5	0.323	0.6	0.225	0.1	0.272	-0.5
26	0.423	0.2	0.1	-0.2	0.145	-0.1	0.393	2.2	0.27	0.3	0.241	-0.6	0.224	0.1	0.303	-0.1
27	0.34	-0.6	0.1	-0.8	0.156	0.2	0.301	0.8	0.26	0.1	0.198	-1.2	0.215	-0.1	0.277	-0.4
28	0.366	-0.4	0.1	-0.3	0.14	-0.2	0.202	-0.8	0.252	0.0	0.278	-0.1	0.23	0.2	0.372	0.8
29	0.361	-0.4	0.1	-0.5	0.145	-0.1	0.254	0.0	0.248	-0.1	0.233	-0.7	0.241	0.4	0.331	0.3
30	FN	-3.9	0.1	0.3	0.149	0.0	0.335	1.3	0.267	0.2	FN	-3.9	FN	-3.8	0.245	-0.8
31	0.33	-0.7	0.1	0.2	0.14	-0.2			0.31	0.9	0.34	0.8	0.22	0.0	0.31	0.0
32	0.37	-0.3	0.1	-0.8	0.109	-1.1	0.17	-1.3	0.225	-0.4	0.238	-0.6	0.435	3.9	0.32	0.2
33	0.496	0.9	0.1	-0.1	0.148	0.0	0.308	0.9	0.271	0.3	0.35	1.0	0.239	0.3	0.231	-1.0
34	0.349	-0.5	0.1	-0.5	0.127	-0.6	0.197	-0.9	0.215	-0.6	0.218	-0.9	0.164	-1.0	0.307	0.0
35	0.424	0.2	0.1	0.0	0.138	-0.3	0.341	1.4	0.304	0.8	0.332	0.7	0.242	0.4	0.347	0.5
36	0.249	-1.5	0.1	1.1	0.199	1.3	0.197	-0.9	0.112	-2.2	0.275	-0.1	0.246	0.5	0.218	-1.2
37	0.415	0.1	0.1	-0.5	0.127	-0.6	0.18	-1.1	0.215	-0.6	0.187	-1.4	0.189	-0.6	0.336	0.4
38	0.539	1.3									0.251	-0.4	0.257	0.7	0.324	0.2
39	0.425	0.2	0.1	0.7	0.188	1.0	0.288	0.6	0.218	-0.5	0.272	-0.1	0.22	0.0	0.251	-0.7
40	0.327	-0.8							0.206	-0.7	0.204	-1.1	0.082	-2.5	0.19	-1.5
41	0.425	0.2	0.1	-0.6	0.163	0.4			0.334	1.3	0.336	0.8	0.196	-0.4	0.297	-0.1
42			0.2	1.9					0.16	-1.5			0.168	-0.9	0.25	-0.7
43	0.342	-0.6	0.1	0.8	0.165	0.4	0.241	-0.2	0.268	0.3	0.28	0.0	0.27	0.9	0.307	0.0
44	FN	-3.9											FN	-3.8	FN	-3.9

Laboratory code	Aldrin	z-scores (FFP RSD (25%))	Azoxystrobin	z-scores (FFP RSD (25%))	Boscalid	z-scores (FFP RSD (25%))	Carbendazim	z-scores (FFP RSD (25%))	Chlorpyrifos-methyl	z-scores (FFP RSD (25%))	Cypermethrin	z-scores (FFP RSD (25%))	Endosulfan-alpha	z-scores (FFP RSD (25%))	Endosulfan-sulfate	z-scores (FFP RSD (25%))
MRRL	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	0.405	0.115	0.149	0.252	0.252	0.283	0.220	0.307								
45	0.455	0.5							0.276	0.4			0.239	0.3	0.251	-0.7
47																
48	0.365	-0.4							0.371	1.9	0.339	0.8	0.288	1.2	0.385	1.0
49																
50	0.474	0.7	0.1	0.0	0.148	0.0	0.228	-0.4	0.228	-0.4	0.263	-0.3	0.247	0.5	0.335	0.4
51	0.331	-0.7	0.1	0.1	0.16	0.3	0.258	0.1	0.251	0.0	0.234	-0.7	0.277	1.0	0.274	-0.4
52	0.564	1.6	FN	-3.7	0.139	-0.3			0.421	2.7	0.343	0.9	0.301	1.5	0.318	0.1
54							0.0989	-2.4								
55	0.623	2.2	0.1	-0.2	0.0831	-1.8	1.015	>5	0.23	-0.3	0.212	-1.0	0.604	>5	0.271	-0.5
56	0.57	1.6	0.0	-2.9	0.108	-1.1	0.164	-1.4	0.333	1.3	0.246	-0.5	0.196	-0.4	0.339	0.4
57	0.379	-0.2	0.1	-2.3	0.171	0.6			0.14	-1.8	0.195	-1.2	0.195	-0.5	0.213	-1.2
58					0.146	-0.1			0.184	-1.1						
59	0.477	0.7	0.1	-0.6	0.116	-0.9	0.246	-0.1	0.241	-0.2	0.254	-0.4	0.266	0.8	0.346	0.5
60	0.5	1.0	0.1	0.9			0.198	-0.9	0.26	0.1	0.399	1.6	0.25	0.5	0.4	1.2
61	0.68	2.7	0.1	-1.3	0.109	-1.1	0.247	-0.1	0.244	-0.1	0.283	0.0	0.224	0.1	0.299	-0.1
62	0.69	2.8	0.2	1.3	0.176	0.7	0.197	-0.9	0.423	2.7	0.3	0.2	0.354	2.4	0.263	-0.6
63	0.371	-0.3	0.1	0.2	0.14	-0.2	0.223	-0.5	0.229	-0.4	0.274	-0.1	0.233	0.2	0.354	0.6
64	0.44	0.4	0.1	0.1	0.147	-0.1	0.234	-0.3	0.264	0.2	0.227	-0.8	0.184	-0.7	0.3	-0.1
65	0.399	0.0	0.2	1.3	0.19	1.1	0.273	0.3	0.213	-0.6	0.292	0.1	0.261	0.7	0.385	1.0
66	0.387	-0.2											0.198	-0.4	0.369	0.8
67	0.405	0.0	0.1	-0.6	0.142	-0.2	0.296	0.7	0.242	-0.2	0.263	-0.3	0.196	-0.4	0.278	-0.4
68	0.418	0.1	0.0	-2.5	0.0618	-2.3	0.179	-1.2	0.184	-1.1	0.206	-1.1	0.147	-1.3	0.113	-2.5
69	0.454	0.5	0.1	1.1	0.163	0.4	0.318	1.0	0.334	1.3	0.25	-0.5	0.193	-0.5	0.356	0.6
70	0.444	0.4	0.1	1.0	0.174	0.7	0.349	1.5	0.208	-0.7	0.296	0.2	0.285	1.2	0.258	-0.6
71	0.416	0.1	0.1	0.9	0.177	0.8	0.231	-0.3	0.283	0.5	0.226	-0.8	0.201	-0.3	0.273	-0.4
72	0.374	-0.3	0.1	0.1	0.187	1.0			0.305	0.9			0.219	0.0	0.321	0.2
73	0.32	-0.8	0.1	-2.3	0.107	-1.1	0.55	4.7	0.285	0.5	0.203	-1.1	0.065	-2.8	0.22	-1.1
74	0.411	0.1	0.1	0.0	0.156	0.2	0.201	-0.8	0.248	-0.1	0.281	0.0	0.242	0.4	0.323	0.2
75	0.41	0.1	0.1	0.5	0.154	0.1	0.267	0.2	0.261	0.2	0.357	1.1	0.26	0.7	0.35	0.6
76	0.378	-0.3	0.1	-0.4	0.124	-0.7	0.255	0.0	0.222	-0.5			0.176	-0.8	0.339	0.4
77	0.512	1.1	0.1	-0.7	0.171	0.6			0.309	0.9	0.315	0.5	0.212	-0.1	0.286	-0.3
79	0.443	0.4	0.1	-1.2	0.14	-0.2	0.263	0.2	0.259	0.1	0.234	-0.7	0.161	-1.1	0.272	-0.5
80	0.561	1.6	0.1	0.8	0.149	0.0			0.289	0.6	0.333	0.7	0.213	-0.1	0.384	1.0
82	0.303	-1.0	0.1	-0.9	0.153	0.1	0.262	0.2	0.207	-0.7	0.244	-0.5	0.207	-0.2	0.243	-0.8
83	0.374	-0.3	0.1	0.8	0.153	0.1	0.27	0.3	0.24	-0.2	0.287	0.1	0.172	-0.9	0.302	-0.1
84	0.408	0.0	0.1	0.8	0.18	0.8			0.323	1.1	0.36	1.1	0.217	-0.1	0.37	0.8
85	0.398	-0.1	0.1	0.0	0.142	-0.2	0.739	>5	0.211	-0.6	0.292	0.1	0.213	-0.1	0.345	0.5
86	0.361	-0.4	0.1	-0.3	0.118	-0.8	0.259	0.1	0.241	-0.2	0.296	0.2	0.199	-0.4	0.291	-0.2
87	0.308	-1.0	0.1	-0.1	0.149	0.0	0.214	-0.6	0.222	-0.5	0.263	-0.3	FN	-3.8	0.307	0.0
88	0.386	-0.2	0.1	-1.4	0.086	-1.7			0.206	-0.7	0.239	-0.6	0.195	-0.5	0.322	0.2
89	0.522	1.2											0.26	0.7	0.33	0.3
90			0.1	-0.2	0.131	-0.5										
91	0.32	-0.8			0.216	1.8			0.239	-0.2	0.274	-0.1	0.21	-0.2	0.27	-0.5

Laboratory code	Aldrin	z-scores (FFP RSD (25%))	Azoxystrobin	z-scores (FFP RSD (25%))	Boscalid	z-scores (FFP RSD (25%))	Carbendazim	z-scores (FFP RSD (25%))	Chlorpyrifos-methyl	z-scores (FFP RSD (25%))	Cypermethrin	z-scores (FFP RSD (25%))	Endosulfan-alpha	z-scores (FFP RSD (25%))	Endosulfan-sulfate	z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01		0.01		0.01		0.01		0.01	
Assigned value	0.405		0.115		0.149		0.252		0.252		0.283		0.220		0.307	
92	0.356	-0.5	0.1	-0.2	0.139	-0.3	0.237	-0.2	0.159	-1.5	0.442	2.3	0.202	-0.3	0.235	-0.9
93	0.404	0.0	0.1	-0.3	0.148	0.0	0.269	0.3	0.264	0.2	0.321	0.5	0.24	0.4	0.296	-0.1
94			0.1	-0.1	0.134	-0.4	0.25	0.0	0.187	-1.0	0.266	-0.2	0.65	>5	0.802	>5
95																
96	0.324	-0.8	0.1	0.5	0.16	0.3	0.283	0.5	0.29	0.6	0.272	-0.1	0.223	0.1	0.359	0.7
98																
99																
100																
101	0.351	-0.5	0.2	2.4	0.149	0.0	0.233	-0.3	0.219	-0.5	0.344	0.9	0.22	0.0	0.365	0.8
102	0.414	0.1	FN	-3.7					0.32	1.1	FN	-3.9	0.411	3.5		
103																
105	0.418	0.1	0.1	0.5	0.142	-0.2	0.205	-0.7	0.191	-1.0	0.302	0.3	0.241	0.4	0.317	0.1
106	0.31	-0.9	0.1	-0.2	0.168	0.5			0.291	0.6	0.308	0.4	0.196	-0.4	0.4	1.2
107	0.496	0.9			0.121	-0.8	0.189	-1.0	0.231	-0.3	0.283	0.0	0.247	0.5		
108	0.487	0.8							0.222	-0.5	0.324	0.6	0.229	0.2	0.335	0.4
109																
110	0.384	-0.2	0.1	0.0	0.157	0.2			0.32	1.1	0.335	0.7	0.207	-0.2		
111			0.1	0.7	0.173	0.6			0.301	0.8	0.298	0.2				
112	0.418	0.1	0.1	0.2	0.122	-0.7			0.255	0.1	0.305	0.3	0.25	0.5	0.33	0.3
113	1.09	>5	0.1	-0.1	0.134	-0.4	0.364	1.8	0.185	-1.1	0.216	-0.9	0.656	>5	0.179	-1.7
115	0.425	0.2							0.585	>5	0.345	0.9	0.172	-0.9	0.272	-0.5
117	0.338	-0.7	0.1	-1.3	0.18	0.8			0.247	-0.1	0.241	-0.6	0.204	-0.3	0.28	-0.4
118	0.4	0.0	0.1	-0.9	0.12	-0.8			0.21	-0.7	0.21	-1.0	0.2	-0.4	0.25	-0.7
120	0.45	0.5	0.0	-2.9	0.0556	-2.5	0.16	-1.5	0.222	-0.5	0.433	2.1	0.198	-0.4	0.521	2.8
121			0.1	0.4	0.143	-0.2	0.243	-0.1	0.256	0.1	0.237	-0.6	0.22	0.0	0.263	-0.6
122	0.642	2.4	0.1	-0.3	0.159	0.3	0.975	>5	1.215	>5	0.386	1.5	0.327	1.9	0.151	-2.0
123			0.1	0.3	0.125	-0.6	0.32	1.1					0.188	-0.6	0.254	-0.7
124	0.755	3.5	0.1	-0.5	0.161	0.3	0.308	0.9	0.276	0.4	0.45	2.4	0.158	-1.1	0.306	0.0
125	0.54	1.3	0.1	-0.9	0.12	-0.8	0.23	-0.3	0.2	-0.8	0.58	4.2	0.25	0.5	0.7	>5
126	0.138	-2.6	FN	-3.7	0.234	2.3	0.177	-1.2	0.278	0.4	0.255	-0.4	0.134	-1.6	0.2	-1.4
127	0.461	0.6	0.1	-0.5	0.122	-0.7	0.231	-0.3	0.236	-0.2	0.393	1.6	0.181	-0.7	0.336	0.4
128	0.425	0.2	0.1	-1.4	0.182	0.9	0.177	-1.2	0.137	-1.8	0.21	-1.0	0.166	-1.0	0.304	0.0
129	1.11	>5							0.54	4.6			FN	-3.8	FN	-3.9
130			0.0	-2.8			0.266	0.2							0.311	0.1
131	0.217	-1.9									0.274	-0.1	0.11	-2.0	0.075	-3.0
132	0.374	-0.3	0.1	0.2	0.112	-1.0	0.25	0.0	0.217	-0.5	0.265	-0.2	0.206	-0.3	0.274	-0.4

**Table 12** Results for epoxiconazole, fenpropidin, fenpropimorph, fluquinconazole, flutriafol, iprodione, kresoxim-methyl and lindane in mg/kg, the corresponding z-scores, the MRRs and the assigned values.

Laboratory code	Epoxiconazole		Fenpropidin		Fenpropimorph		Fluquinconazole		Flutriafol		Iprodione		Kresoxim-methyl		Lindane	
MRRL	0.01	z-scores (FFP RSD (25%))	0.01	z-scores (FFP RSD (25%))	0.01	z-scores (FFP RSD (25%))	0.01	z-scores (FFP RSD (25%))	0.01	z-scores (FFP RSD (25%))	0.01	z-scores (FFP RSD (25%))	0.01	z-scores (FFP RSD (25%))	0.01	z-scores (FFP RSD (25%))
Assigned value	0.121		0.171		0.333		0.107		0.308		0.325		0.060		0.279	
1	0.1	-0.7	0.085	-2.0	0.25	-1.0	0.1	-0.3	0.29	-0.2	0.29	-0.4	0.05	-0.7	0.21	-1.0
2	0.0337	-2.9	0.0163	-3.6	0.194	-1.7	0.0609	-1.7	0.186	-1.6			0.0354	-1.6	0.244	-0.5
3	0.107	-0.5	0.136	-0.8	0.352	0.2	0.078	-1.1	0.205	-1.3	0.307	-0.2	0.039	-1.4	FN	-3.9
5	0.125	0.1	0.199	0.7	0.455	1.5	0.124	0.6	0.329	0.3	0.35	0.3	0.053	-0.5	0.307	0.4
6	0.135	0.5	0.215	1.0	0.445	1.3	0.122	0.6	0.409	1.4	0.921	>5	0.065	0.3	0.181	-1.4
7	0.129	0.3	0.159	-0.3	0.282	-0.6	0.1	-0.3	0.3	-0.1	0.327	0.0	0.059	-0.1	0.286	0.1
9	0.105	-0.5	0.252	1.9	0.258	-0.9	0.0815	-1.0	0.278	-0.3	0.182	-1.8	0.0544	-0.4	0.25	-0.4
11																
12															0.142	-2.0
13	0.097	-0.8			0.368	0.4					0.243	-1.0	0.071	0.7	0.255	-0.3
14	0.127	0.2	0.116	-1.3	0.209	-1.5	0.103	-0.1	0.289	-0.2	0.231	-1.2	0.064	0.3	0.254	-0.4
15	0.108	-0.4	0.169	0.0	0.115	-2.6	0.147	1.5	0.344	0.5	0.313	-0.2	0.0606	0.0	0.286	0.1
16	0.128	0.2	0.182	0.3	0.354	0.3	0.1	-0.3	0.346	0.6	0.326	0.0	0.0697	0.6	0.261	-0.3
17	0.118	-0.1	0.203	0.8	0.323	-0.1	0.102	-0.2	0.225	-1.0	0.204	-1.5	0.0605	0.0	0.234	-0.6
18	0.166	1.5			0.243	-1.1	0.122	0.6	0.236	-0.9	0.443	1.4	0.062	0.1	0.253	-0.4
19	0.12	0.0	0.108	-1.5	0.664	4.0	0.086	-0.8	FN	-3.9	0.422	1.2	0.067	0.5	0.265	-0.2
20	0.121	0.0	0.097	-1.7	0.375	0.5	0.124	0.6	0.298	-0.1	0.327	0.0	0.078	1.2	0.349	1.0
21	0.194	2.4	0.216	1.1	0.393	0.7	0.128	0.8	0.34	0.5	0.35	0.3	0.067	0.5	0.27	-0.1
22															0.263	-0.2
23	0.113	-0.3	0.18	0.2	0.345	0.1	0.123	0.6	0.345	0.5	0.42	1.2	0.062	0.1	0.304	0.4
24	0.157	1.2			0.255	-0.9	0.121	0.5	0.228	-1.0	0.426	1.2	0.062	0.1	0.268	-0.2
25	0.131	0.3			0.368	0.4	0.119	0.4	0.308	0.1	0.307	-0.2	0.052	-0.5	0.273	-0.1
26	0.127	0.2	0.179	0.2	0.32	-0.2	0.1	-0.3	0.303	0.0	0.336	0.1	0.0585	-0.1	0.23	-0.7
27	0.116	-0.2	0.215	1.0	0.381	0.6	0.113	0.2	0.33	0.3	0.23	-1.2	0.068	0.5	0.13	-2.1
28	0.105	-0.5	0.138	-0.8	0.325	-0.1	0.104	-0.1	FN	-3.9	0.34	0.2	0.053	-0.5	0.281	0.0
29	0.116	-0.2	0.184	0.3	0.294	-0.5	0.098	-0.3	0.321	0.2	0.332	0.1	0.052	-0.5	0.267	-0.2
30	0.127	0.2	0.157	-0.3	0.334	0.0	0.106	0.0	0.32	0.2	0.144	-2.2	0.05	-0.7	0.306	0.4
31	0.13	0.3	0.15	-0.5	0.45	1.4	0.12	0.5	0.29	-0.2			0.046	-0.9	0.27	-0.1
32	0.096	-0.8									0.313	-0.2	0.058	-0.1	0.278	0.0
33	0.142	0.7	0.15	-0.5	0.344	0.1	0.124	0.6	0.313	0.1	0.265	-0.7	0.0604	0.0	0.345	0.9
34	0.0975	-0.8	0.152	-0.4	0.347	0.2	0.0648	-1.6	0.272	-0.4	0.316	-0.1	0.0529	-0.5	FN	-3.9
35	0.112	-0.3	0.0984	-1.7	0.256	-0.9	0.124	0.6	0.29	-0.2	0.358	0.4	0.0585	-0.1	0.326	0.7
36							0.091	-0.6	0.276	-0.4			0.069	0.6	0.3	0.3
37	0.11	-0.4	0.12	-1.2	0.292	-0.5	0.08	-1.0	0.258	-0.6	0.335	0.1	0.05	-0.7	0.27	-0.1
38															0.351	1.0
39	0.138	0.6	0.207	0.9	0.256	-0.9	0.107	0.0	0.365	0.8	0.295	-0.4	0.062	0.1	0.289	0.1
40															0.189	-1.3
41	0.185	2.1			0.248	-1.0	0.113	0.2	0.268	-0.5	0.43	1.3	0.071	0.7	0.261	-0.3
42																
43	0.104	-0.6	0.188	0.4	0.35	0.2	0.111	0.1	0.344	0.5	0.342	0.2	FN	-3.3	0.317	0.5
44															FN	-3.9



Laboratory code	Epoxiconazole		Fenpropidin		Fenpropimorph		Fluquinconazole		Flutriafof		Iprodione		Kresoxim-methyl		Lindane	
MRRL	0.01	z-scores (FFP RSD (25%))	0.01	z-scores (FFP RSD (25%))	0.01	z-scores (FFP RSD (25%))	0.01	z-scores (FFP RSD (25%))	0.01	z-scores (FFP RSD (25%))	0.01	z-scores (FFP RSD (25%))	0.01	z-scores (FFP RSD (25%))	0.01	z-scores (FFP RSD (25%))
Assigned value	0.121		0.171		0.333		0.107		0.308		0.325		0.060		0.279	
45															0.285	0.1
47																
48															0.282	0.0
49																
50	0.118	-0.1	0.136	-0.8	0.38	0.6	0.102	-0.2	0.306	0.0	0.302	-0.3	0.063	0.2	0.27	-0.1
51	0.115	-0.2			0.368	0.4	0.092	-0.6	0.305	0.0	0.288	-0.5	0.055	-0.3	0.287	0.1
52	0.116	-0.2			0.702	4.4	0.11	0.1	0.246	-0.8	0.365	0.5	0.067	0.5	0.118	-2.3
54	0.027	-3.1	0.0125	-3.7	0.126	-2.5			0.0516	-3.3						
55	0.0803	-1.3	FN	-3.8	0.371	0.5	0.0633	-1.6	0.228	-1.0	0.25	-0.9	0.0408	-1.3	0.598	4.6
56	0.138	0.6	0.0719	-2.3	0.155	-2.1	FN	-3.6	0.0883	-2.8	0.218	-1.3	FN	-3.3	0.356	1.1
57							0.05	-2.1			0.147	-2.2	0.05	-0.7	0.254	-0.4
58											0.346	0.2	0.0477	-0.8	0.161	-1.7
59	0.12	0.0	0.17	0.0	0.332	0.0	0.091	-0.6	0.291	-0.2	0.29	-0.4	0.06	0.0	0.249	-0.4
60	0.15	1.0	0.199	0.7	0.36	0.3	0.115	0.3	0.305	0.0	0.38	0.7	0.058	-0.1	0.302	0.3
61	0.0905	-1.0	0.139	-0.7	0.271	-0.7	0.0695	-1.4	0.283	-0.3	0.319	-0.1	0.0469	-0.9	0.293	0.2
62	0.196	2.5	0.243	1.7	0.588	3.1	0.25	>5	FN	-3.9	0.53	2.5	0.075	1.0	0.45	2.5
63	0.112	-0.3	0.203	0.8	0.347	0.2	0.0989	-0.3	0.319	0.2	0.335	0.1	0.0558	-0.3	0.295	0.2
64	0.123	0.1	0.184	0.3	0.334	0.0	0.0969	-0.4	0.31	0.1	0.345	0.2	0.0735	0.9	0.275	-0.1
65	0.151	1.0	0.205	0.8	0.496	2.0	0.096	-0.4			0.314	-0.1	0.061	0.1	0.328	0.7
66															0.217	-0.9
67	0.11	-0.4	0.128	-1.0	0.282	-0.6	0.106	0.0	0.32	0.2	0.323	0.0	0.0555	-0.3	0.242	-0.5
68	0.0454	-2.5	FN	-3.8	0.242	-1.1	0.0283	-2.9	0.173	-1.7	0.237	-1.1	FN	-3.3	FN	-3.9
69	0.138	0.6	0.185	0.3	0.432	1.2	0.181	2.8	0.394	1.2	0.355	0.4	0.0576	-0.2	0.303	0.3
70	0.114	-0.2	0.205	0.8	0.38	0.6	0.104	-0.1	0.284	-0.3	0.168	-1.9	0.062	0.1	0.238	-0.6
71	0.143	0.7	0.205	0.8	0.383	0.6	0.121	0.5	0.358	0.7	0.365	0.5	0.069	0.6	0.251	-0.4
72	0.187	2.2			0.275	-0.7	0.105	-0.1	0.286	-0.2	0.509	2.2	0.061	0.1	0.236	-0.6
73			0.18	0.2	0.22	-1.4			0.275	-0.4	0.192	-1.6	0.052	-0.5	0.25	-0.4
74	0.119	-0.1	0.196	0.6	0.349	0.2	0.113	0.2	0.333	0.4	0.347	0.3	0.0541	-0.4	0.279	0.0
75	0.133	0.4	0.171	0.0	0.32	-0.2	0.1	-0.3	0.325	0.3	0.339	0.2	0.0602	0.0	0.3	0.3
76	0.108	-0.4			0.34	0.1	0.0871	-0.7	0.312	0.1	0.328	0.0	0.052	-0.5		
77	0.156	1.2			0.235	-1.2	0.116	0.3	0.248	-0.7	0.413	1.1	0.055	-0.3	0.286	0.1
79	FN	-3.7			FN	-3.9	0.071	-1.3	FN	-3.9	0.205	-1.5	0.0728	0.9	0.113	-2.4
80			0.157	-0.3			0.11	0.1					0.0705	0.7	0.318	0.6
82	0.121	0.0	0.177	0.2	0.329	0.0			0.344	0.5	0.322	0.0	0.0408	-1.3	0.216	-0.9
83	0.115	-0.2	0.149	-0.5	0.316	-0.2	0.12	0.5	0.31	0.1	0.261	-0.8	0.074	0.9	0.294	0.2
84	0.166	1.5			0.26	-0.9	0.129	0.8	0.222	-1.1	0.48	1.9	0.062	0.1	0.308	0.4
85	0.109	-0.4			0.373	0.5	0.099	-0.3	0.31	0.1	0.312	-0.2	0.059	-0.1	0.23	-0.7
86	0.118	-0.1	0.13	-1.0	0.311	-0.3	0.088	-0.7	0.239	-0.9	0.292	-0.4	0.083	1.5	0.217	-0.9
87	0.134	0.4	0.127	-1.0	0.376	0.5	0.109	0.1	0.278	-0.3	0.342	0.2	0.062	0.1	0.246	-0.5
88	0.085	-1.2					0.079	-1.0	0.231	-1.0	0.265	-0.7	0.052	-0.5	0.253	-0.4
89															0.259	-0.3
90																
91											0.32	-0.1	0.077	1.1	0.31	0.4

Laboratory code	Epoxiconazole	Fenpropidin		Fenpropimorph		Fluquinconazole		Flutriafof		Iprodione		Kresoxim-methyl		Lindane		
MRRL	0.01	0.01		0.01		0.01		0.01		0.01		0.01		0.01		
Assigned value	0.121	0.171		0.333		0.107		0.308		0.325		0.060		0.279		
		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%))		
92	0.122	0.0	0.178	0.2	0.362	0.3			0.266	-0.5	0.286	-0.5	0.049	-0.7	0.211	-1.0
93	0.111	-0.3	0.18	0.2	0.39	0.7	0.11	0.1	0.318	0.2	0.322	0.0	0.065	0.3	0.281	0.0
94	0.109	-0.4					0.083	-0.9	0.264	-0.5	0.29	-0.4	0.048	-0.8		
95																
96	0.132	0.4	0.189	0.4	0.322	-0.1	0.119	0.4	0.337	0.4	0.397	0.9	0.065	0.3	0.347	1.0
98																
99																
100																
101	0.116	-0.2	0.044	-3.0	0.126	-2.5	0.107	0.0	0.359	0.7	0.333	0.1	0.061	0.1	0.279	0.0
102											0.359	0.4			0.31	0.4
103																
105	0.108	-0.4	0.106	-1.5	0.378	0.5	0.115	0.3	0.314	0.1	0.362	0.4	0.0498	-0.7	0.28	0.0
106	0.184	2.1			0.23	-1.2	0.123	0.6	FN	-3.9			0.064	0.3	0.24	-0.6
107											0.302	-0.3	0.056	-0.3	0.242	-0.5
108															0.255	-0.3
109																
110	0.168	1.6			0.225	-1.3	0.106	0.0	0.218	-1.1			0.059	-0.1	0.235	-0.6
111			0.18	0.2									0.058	-0.1		
112	0.124	0.1	FN	-3.8	0.332	0.0			0.308	0.1	0.335	0.1	0.06	0.0	0.315	0.5
113	FN	-3.7	0.219	1.1	0.321	-0.1	0.14	1.2	0.358	0.7	0.258	-0.8	FN	-3.3	0.38	1.4
115											0.403	0.9	0.043	-1.1	0.267	-0.2
117	0.13	0.3			0.274	-0.7	0.097	-0.4	0.373	0.9	0.291	-0.4	0.07	0.7	0.248	-0.4
118	0.0905	-1.0			0.25	-1.0			0.25	-0.7	0.28	-0.6	0.0459	-0.9	0.22	-0.8
120	0.0345	-2.9	0.0406	-3.0	0.244	-1.1	0.0544	-2.0							0.315	0.5
121	0.119	-0.1	0.134	-0.9	0.192	-1.7	0.0994	-0.3	0.299	-0.1	0.14	-2.3	0.0471	-0.9		
122	0.202	2.7			0.766	>5	0.113	0.2	0.588	3.7	0.356	0.4	0.066	0.4	0.334	0.8
123	0.0799	-1.4					0.0991	-0.3	0.407	1.4	0.233	-1.1	0.04	-1.3	0.242	-0.5
124	0.138	0.6			0.255	-0.9	0.136	1.1	FN	-3.9	0.399	0.9	0.059	-0.1	0.291	0.2
125	0.09	-1.0	0.1	-1.7	0.36	0.3	0.1	-0.3	0.2	-1.4	0.8	>5	0.08	1.3	0.35	1.0
126	0.35	>5	FN	-3.8	0.199	-1.6	0.238	4.9	FN	-3.9	0.264	-0.8	0.305	>5	0.128	-2.2
127	0.123	0.1	0.297	3.0	0.365	0.4	0.422	>5	0.355	0.7	0.295	-0.4	0.149	>5	0.236	-0.6
128	0.132	0.4	0.104	-1.6	0.319	-0.2	0.116	0.3	0.256	-0.6	0.303	-0.3	0.058	-0.1	0.275	-0.1
129															0.665	>5
130													FN	-3.3		
131															0.106	-2.5
132	0.115	-0.2	0.124	-1.1	0.322	-0.1	0.1	-0.3	0.322	0.2	0.273	-0.7	0.06	0.0	0.23	-0.7

**Table 13** Results for malathion, propiconazole, pyraclostrobin, spiroxamin, tebuconazole, triadimenol and trifloxystrobin in mg/kg, the corresponding z-scores, the MRRLs and the assigned values.

Laboratory code	Malathion	Propiconazole	Pyraclostrobin	Spiroxamine	Tebuconazole	Triadimenol	Trifloxystrobin							
MRRL	0.01	0.01	0.01	0.01	0.01	0.01	0.01							
Assigned value	0.316	0.217	0.096	0.176	0.076	0.230	0.057							
	z-scores (FPP RSD (25%))	z-scores (FPP RSD (25%))	z-scores (FPP RSD (25%))	z-scores (FPP RSD (25%))	z-scores (FPP RSD (25%))	z-scores (FPP RSD (25%))	z-scores (FPP RSD (25%))							
1	0.29	-0.3	0.2	-0.3	0.06	-1.5	0.08	-2.2	FN	-3.5	0.17	-1.0	0.055	-0.1
2	0.286	-0.4	0.188	-0.5	0.0545	-1.7	0.112	-1.5	0.0256	-2.7	0.174	-1.0	0.0478	-0.6
3	0.244	-0.9	0.134	-1.5	0.08	-0.7	0.15	-0.6	0.069	-0.4	0.177	-0.9	0.051	-0.4
5	0.318	0.0	0.215	0.0	0.105	0.4	0.208	0.7	0.084	0.4	0.228	0.0	0.059	0.1
6	0.451	1.7	0.216	0.0	0.098	0.1	0.136	-0.9	0.083	0.4	0.204	-0.4	0.138	>5
7	0.305	-0.1	0.218	0.0	0.096	0.0	0.164	-0.3	0.074	-0.1	0.23	0.0	0.053	-0.3
9	0.274	-0.5	0.18	-0.7	0.0878	-0.3	0.291	2.5	0.0608	-0.8	0.207	-0.4	0.0504	-0.5
11	0.49	2.2												
12											FN	-3.8		
13	0.32	0.1	0.238	0.4					0.08	0.2	0.245	0.3	FN	-3.3
14	0.27	-0.6	0.217	0.0	0.076	-0.8	0.129	-1.1	0.074	-0.1			0.063	0.4
15	0.359	0.5	0.195	-0.4	0.0804	-0.7	0.208	0.7	0.0632	-0.7	0.212	-0.3	0.0565	0.0
16	0.287	-0.4	0.211	-0.1	0.0964	0.0	0.199	0.5	0.075	-0.1	0.239	0.2	0.0574	0.0
17	0.249	-0.8	0.224	0.1	0.103	0.3	0.17	-0.2	0.0804	0.2	0.209	-0.4	0.053	-0.3
18	0.297	-0.2	0.254	0.7					0.108	1.7			0.084	1.9
19	0.359	0.5	0.106	-2.0	0.089	-0.3	0.103	-1.7	0.078	0.1			0.056	-0.1
20	0.332	0.2	0.204	-0.2	0.09	-0.3	0.09	-2.0	0.091	0.8	0.28	0.9	0.054	-0.2
21	0.345	0.4	0.267	0.9	0.11	0.6	0.262	1.9	0.201	>5	0.295	1.1	0.0616	0.3
22	0.291	-0.3												
23	0.276	-0.5	0.213	-0.1	0.092	-0.2	0.194	0.4	0.073	-0.2	0.312	1.4	0.052	-0.4
24	0.287	-0.4	0.265	0.9					0.108	1.7			0.065	0.6
25	0.335	0.2	0.225	0.1	0.101	0.2	0.191	0.3	0.071	-0.3	0.215	-0.3	0.049	-0.6
26	0.215	-1.3	0.205	-0.2	0.1	0.2	0.165	-0.3	0.0907	0.8	0.202	-0.5	0.0565	0.0
27	0.345	0.4	0.243	0.5	0.096	0.0	0.215	0.8	0.065	-0.6	0.23	0.0	0.057	0.0
28	0.326	0.1	0.25	0.6	0.098	0.1	0.176	0.0	0.059	-0.9	0.225	-0.1	0.056	-0.1
29	0.34	0.3	0.223	0.1	0.092	-0.2	0.21	0.7	0.077	0.1	0.248	0.3	0.053	-0.3
30	0.305	-0.1	0.244	0.5	0.0892	-0.3	0.152	-0.6	0.0758	0.0	0.184	-0.8	FN	-3.3
31	0.38	0.8	0.23	0.2			0.45	>5	0.069	-0.4	0.26	0.5	0.061	0.3
32			0.207	-0.2	0.062	-1.4			0.075	-0.1	0.23	0.0	0.063	0.4
33	0.379	0.8	0.221	0.1	0.0752	-0.9	0.173	-0.1	0.0721	-0.2	0.244	0.3	0.0546	-0.2
34	0.323	0.1	0.175	-0.8	0.0807	-0.6	0.158	-0.4	0.0613	-0.8	0.211	-0.3	0.0493	-0.5
35	0.372	0.7	0.233	0.3	0.187	3.8	0.145	-0.7	0.0707	-0.3	0.206	-0.4	0.097	2.8
36	0.194	-1.5	0.378	3.0	0.076	-0.8			0.221	>5	0.263	0.6		
37	0.26	-0.7	0.211	-0.1	0.105	0.4	0.13	-1.1	0.076	0.0	0.248	0.3	0.052	-0.4
38														
39	0.285	-0.4	0.241	0.4	0.1	0.2	0.241	1.4	0.088	0.6	0.253	0.4	0.059	0.1
40	0.268	-0.6												
41	0.262	-0.7	0.292	1.4					0.085	0.5			0.09	2.3
42	0.153	-2.1							FN	-3.5	FN	-3.8	0.0803	1.6
43	0.367	0.6	0.234	0.3	0.103	0.3	0.194	0.4	0.071	-0.3	0.229	0.0	0.051	-0.4
44													FN	-3.3

Laboratory code	Malathion	Propiconazole	Pyraclostrobin	Spiroxamine	Tebuconazole	Triadimenol	Trifloxystrobin							
MRRL	0.01	0.01	0.01	0.01	0.01	0.01	0.01							
Assigned value	0.316	0.217	0.096	0.176	0.076	0.230	0.057							
	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%))							
45	0.165	-1.9												
47														
48	0.43	1.4												
49														
50	0.296	-0.3	0.232	0.3	0.108	0.5	0.162	-0.4	0.072	-0.2	0.236	0.1	0.058	0.1
51	0.333	0.2	0.213	-0.1	0.12	1.0	0.153	-0.6	0.073	-0.2	0.235	0.1	0.052	-0.4
52	0.375	0.7	0.206	-0.2	FN	-3.6	0.271	2.1			FN	-3.8	0.057	0.0
54			0.152	-1.2	0.0329	-2.6								
55	0.223	-1.2	0.169	-0.9	0.0623	-1.4	0.163	-0.3	0.0603	-0.8	0.156	-1.3	0.0384	-1.3
56	0.39	0.9	0.148	-1.3	FN	-3.6	0.216	0.9	0.0512	-1.3	0.184	-0.8		
57									0.068	-0.4			0.095	2.7
58	0.327	0.1	0.238	0.4					0.0895	0.7	0.394	2.9	0.0762	1.3
59	0.278	-0.5	0.21	-0.1	0.087	-0.4	0.202	0.5	0.071	-0.3	0.224	-0.1	0.052	-0.4
60	0.321	0.1	0.208	-0.2			0.213	0.8			0.29	1.1		
61	0.339	0.3	0.202	-0.3	0.0622	-1.4	0.154	-0.5	0.063	-0.7	0.206	-0.4	0.0484	-0.6
62	0.52	2.6	0.398	3.3	FN	-3.6	0.262	1.9	0.11	1.8	FN	-3.8	0.06	0.2
63	0.311	-0.1	0.202	-0.3	0.0924	-0.2	0.199	0.5	0.0704	-0.3	0.236	0.1	0.0574	0.0
64	0.291	-0.3	0.209	-0.1	0.0988	0.1			0.0759	0.0	0.243	0.2	0.0562	-0.1
65	0.365	0.6	0.24	0.4	0.119	0.9	0.158	-0.4	0.087	0.6	0.198	-0.5	0.061	0.3
66														
67	0.229	-1.1	0.216	0.0	0.0626	-1.4	0.149	-0.7	0.0725	-0.2	0.191	-0.7	0.0486	-0.6
68	0.363	0.6	0.021	-3.6	0.0189	-3.2	FN	-3.8	0.0129	-3.3	0.0852	-2.5	0.0486	-0.6
69	0.326	0.1	0.217	0.0	0.106	0.4	0.213	0.8	0.084	0.4	0.267	0.7	0.058	0.1
70	0.381	0.8	0.197	-0.4	0.107	0.4	0.203	0.6	0.082	0.3	0.185	-0.8	0.06	0.2
71	0.355	0.5	0.207	-0.2	0.112	0.7	0.198	0.4	0.087	0.6	0.261	0.5	0.063	0.4
72	0.274	-0.5	0.263	0.8					0.084	0.4			0.08	1.6
73	0.31	-0.1	0.19	-0.5			0.2	0.5	0.11	1.8			0.055	-0.1
74	0.305	-0.1	0.231	0.3	0.104	0.3	0.189	0.2	0.0724	-0.2	0.233	0.1	0.0588	0.1
75	0.316	0.0	0.234	0.3	0.0888	-0.3	0.185	0.2	0.0802	0.2	0.271	0.7	0.0534	-0.3
76	0.288	-0.4	0.213	-0.1	0.089	-0.3	0.17	-0.2	0.0711	-0.3	0.227	0.0	0.0632	0.4
77	0.266	-0.6	0.239	0.4					0.098	1.2			0.085	2.0
79	0.306	-0.1	0.143	-1.4	0.108	0.5			0.0413	-1.8	0.114	-2.0	FN	-3.3
80	0.33	0.2	0.191	-0.5			0.166	-0.3	0.0821	0.3	0.2	-0.5	0.0565	0.0
82	0.323	0.1	0.228	0.2	0.0995	0.1	0.117	-1.4	0.0793	0.2	0.251	0.4	0.0632	0.4
83	0.36	0.6	0.225	0.1	0.085	-0.5	0.119	-1.3	0.091	0.8	0.241	0.2	0.052	-0.4
84	0.274	-0.5	0.253	0.7					0.113	1.9			0.065	0.6
85	0.297	-0.2	0.2	-0.3	0.08	-0.7	0.205	0.6	0.054	-1.2	0.22	-0.2	0.055	-0.1
86	0.257	-0.7	0.151	-1.2	0.099	0.1	0.197	0.4	0.052	-1.3	0.181	-0.8	0.044	-0.9
87	0.278	-0.5	0.218	0.0	0.097	0.0	0.154	-0.5	0.084	0.4	0.231	0.0	0.065	0.6
88	0.273	-0.5	0.187	-0.6					0.063	-0.7	0.174	-1.0	0.046	-0.8
89														
90			0.224	0.1					0.072	-0.2	0.287	1.0		
91	0.318	0.0			0.159	2.6	0.192	0.3			0.378	2.6		

Laboratory code	Malathion	Propiconazole	Pyraclostrobin	Spiroxamine	Tebuconazole	Triadimenol	Trifloxystrobin	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%))
MRRL	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	0.316	0.217	0.096	0.176	0.076	0.230	0.057							
92	0.253	-0.8	0.203	-0.3	0.09	-0.3	0.182	0.1	0.071	-0.3	0.188	-0.7	0.053	-0.3
93	0.384	0.9	0.248	0.6	0.097	0.0	0.266	2.0	0.081	0.3	0.247	0.3	0.056	-0.1
94	0.332	0.2	0.2	-0.3	0.078	-0.8	0.175	-0.1	0.078	0.1	0.18	-0.9	0.053	-0.3
95														
96	0.326	0.1	0.218	0.0	0.0825	-0.6	0.166	-0.3	0.0825	0.3	0.198	-0.5	0.0575	0.0
98														
99														
100	0.79	>5												
101	0.292	-0.3	0.212	-0.1	0.208	4.6	0.067	-2.5	0.075	-0.1	0.268	0.7	0.057	0.0
102	FN	-3.9	0.239	0.4							0.273	0.8		
103														
105	0.287	-0.4	0.222	0.1	0.059	-1.5	0.247	1.6	0.0778	0.1	0.224	-0.1	0.0562	-0.1
106	0.294	-0.3	0.262	0.8					FN	-3.5			0.062	0.4
107	0.32	0.1	0.288	1.3					0.076	0.0	0.168	-1.1	0.048	-0.6
108	0.286	-0.4												
109														
110	0.283	-0.4	0.255	0.7					0.099	1.2			0.065	0.6
111	0.28	-0.5	0.251	0.6	0.105	0.4			0.068	-0.4	0.301	1.2	0.065	0.6
112	0.297	-0.2	0.211	-0.1	FN	-3.6	0.18	0.0	0.08	0.2	0.26	0.5	0.0578	0.1
113	0.243	-0.9	0.2	-0.3	0.0748	-0.9	0.217	0.9	0.0728	-0.2	FN	-3.8	0.0938	2.6
115	0.287	-0.4	0.188	-0.5					0.078	0.1				
117	0.246	-0.9	0.209	-0.1	FN	-3.6	0.13	-1.1	0.132	2.9	0.225	-0.1	0.048	-0.6
118	0.3	-0.2	0.19	-0.5					0.0652	-0.6	0.17	-1.0	0.0504	-0.5
120	0.255	-0.8	0.164	-1.0					0.0336	-2.2				
121	0.232	-1.1	0.213	-0.1	0.0802	-0.7	0.145	-0.7	0.0804	0.2	0.222	-0.1	0.0553	-0.1
122	0.666	4.4	0.458	4.4			0.211	0.7	0.142	3.5	0.225	-0.1	0.067	0.7
123	0.322	0.1					0.147	-0.7	0.0554	-1.1	0.234	0.1		
124	0.46	1.8	0.278	1.1	0.113	0.7	0.117	-1.4	0.077	0.1	0.082	-2.6	0.087	2.1
125	0.18	-1.7	0.25	0.6	0.09	-0.3	0.18	0.0	0.065	-0.6	0.18	-0.9	0.065	0.6
126	0.425	1.4	0.259	0.8	0.519	>5	FN	-3.8	0.363	>5	0.276	0.8	0.299	>5
127	0.2	-1.5	0.3	1.5	0.102	0.2	0.302	2.8	0.04	-1.9	0.223	-0.1	0.13	>5
128	0.327	0.1	0.197	-0.4	0.065	-1.3	0.154	-0.5	0.094	0.9	0.215	-0.3	0.062	0.4
129	1.28	>5			FN	-3.6								
130											0.14	-1.6		
131														
132	0.24	-1.0	0.169	-0.9	0.122	1.1	0.153	-0.6	0.066	-0.5	0.218	-0.2	0.056	-0.1

### 3.3.2 Sum of Weighted Z-Scores ( $AZ^2$ ) – 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 ( $\geq 21$  pesticide residues, inclusive of false negatives). For the 60 EU and EFTA laboratories in Category A, the results were additionally evaluated by calculating the Average of the Squared -Score ( $AZ^2$ ). Of the 60 participants, 46 participants (77 %) obtained  $AZ^2$  values at or below 2 (good) and 4 participants (7 %) obtained  $AZ^2$  values above 2 but below or at 3 (satisfactory) and 10 participants (17 %) obtained  $AZ^2$  values above 3 (unsatisfactory). An additional five laboratories from Third Countries were evaluated and classified into Category A. The  $AZ^2$  scores achieved by the labs can be seen in **Table 14**.

**Table 14** Sum of Weighted z-Scores (SWZ) for laboratories in Category A, the number of pesticide analysed by the laboratory, the number of false negatives reported and the Classification as good, satisfactory and unsatisfactory.

Lab code	No. of detected pesticides	$AZ^2$	No. of false negative	Evaluation	NRL
1	23	1.5	1	Good	
2	21	4.9		Unsatisfactory	
3	23	1.3	1	Good	
5	23	0.3		Good	NRL
6	23	3.9		Unsatisfactory	
7	23	0.1		Good	
9	23	0.9		Good	
14	22	0.5		Good	NRL
15	23	0.8		Good	NRL
16	23	0.1		Good	
17	23	0.4		Good	
20	23	0.6		Good	
21	23	2.4		Satisfactory	NRL
23	23	0.4		Good	
25	22	0.1		Good	NRL
26	23	0.4		Good	NRL
27	23	0.5		Good	NRL
28	23	0.8	1	Good	NRL
29	23	0.1		Good	NRL
30	23	2.8	1	Satisfactory	NRL
33	23	0.3		Good	
34	23	1.1	1	Good	NRL
35	23	1.4		Good	NRL
37	23	0.4		Good	
39	23	0.4		Good	
43	23	0.7		Good	NRL
50	23	0.1		Good	
51	22	0.2		Good	NRL
55	23	4.8	1	Unsatisfactory	
56	22	3.7	1	Unsatisfactory	
59	23	0.2		Good	
61	23	0.8		Good	

Lab code	No. of detected pesticides	AZ <sup>2</sup>	No. of false negative	Evaluation	NRL
62	23	>5	1	Unsatisfactory	
63	23	0.1		Good	
64	22	0.1		Good	
65	22	0.6		Good	
67	23	0.3		Good	
68	23	>5	4	Unsatisfactory	
69	23	0.8		Good	NRL
70	23	0.6		Good	
71	23	0.3		Good	
74	23	0.1		Good	NRL
75	23	0.2		Good	
79	21	3.7	4	Unsatisfactory	
82	22	0.4		Good	
83	23	0.3		Good	
85	22	1.3		Good	
86	23	0.5		Good	NRL
87	23	0.8	1	Good	
92	22	0.6		Good	NRL
93	23	0.3		Good	
96	23	0.2		Good	NRL
101	23	2.2		Satisfactory	
105	23	0.5		Good	
112	21	1.4	2	Good	
113	22	4.9	3	Unsatisfactory	
117	21	1.4	1	Good	
121	21	0.6		Good	
122	21	>5		Unsatisfactory	
124	22	2.6	1	Satisfactory	
125	23	3.7		Unsatisfactory	
126	23	>5	4	Unsatisfactory	
127	23	4.6		Unsatisfactory	
128	23	0.7		Good	
132	23	0.3		Good	NRL

**Table 15** shows the 46 EU and EFTA laboratories in Category B and the 9 laboratories from Third Countries. The table includes 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 15** 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	No. of false negative	No. of false positive	NRL
11	4	1		1	
12	5	3	3		
13	16	15	1		
18	18	17			
19	20	15	1		
22	7	7			NRL
24	18	17			
31	20	18		2	
32	17	14			
36	17	14			
38	5	5			
40	7	6			
41	18	16			
42	8	4	2		
44	5	0	1		
45	6	6	5		
47	0	0			
48	7	7			
49	0	0		1	
52	20	12	1		
54	7	1			NRL
57	13	9			
58	10	8			
60	19	19			NRL
66	4	4		1	
72	17	14			
73	19	15			
76	20	19			
77	18	17			
80	17	15			
84	18	17			
88	18	17			
89	4	4			
90	5	5			
91	13	11		2	
94	19	15			
95	0	0			
98	0	0			



Lab code	No. of pesticides detected	No. of acceptable z-scores	No. of false negative	No. of false positive	NRL
99	0	0			
100	1	0			
102	10	6	3		
103	0	0			
106	17	13	2		
107	14	12			NRL
108	7	7			
109	0	0			NRL
110	16	14			
111	12	11			
115	11	10			
118	18	17			
120	16	9			NRL
123	15	15			NRL
129	7	0	3	1	
130	5	3	1		
131	5	3			

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

EUPT-CF7 was the first EUPT that focused on a cereal-based animal feed Test Item. A Test Item of mixed feed for laying hens, including both incurred and spiked pesticides, was successfully prepared. The cereal used to prepare the feed was sprayed in the field with commercially available pesticide formulations, and the soya oil that was added was spiked in the laboratory. The treated Test Item contained the following pesticides: aldrin, azoxystrobin, boscalid, carbendazim, chlorpyrifos-methyl, cypermethrin, endosulfan-alpha, endosulfan-sulfate, epoxiconazole, fenpropidin, fenpropimorph, fluquinconazole, flutriafol, iprodione, kresoxim-methyl, lindane, malathion, propiconazole, pyraclostrobin, spiroxamine, tebuconazole, triadimenol and trifloxystrobin. One hundred and six laboratories, representing 29 EU and EFTA countries, agreed to participate in this Proficiency Test. Three laboratories did not submit results, and among these was the Italian NRL. All NRLs, except Bulgaria, Luxembourg and Portugal, participated in the PT. Malta was represented in the PT by the NRL for the UK. An additional 14 laboratories from Third Countries registered for the PT and 10 submitted results. The Target Pesticide List distributed to the laboratories prior to the test contained 116 individual compounds.

Both the number of false positives and false negatives increased considerably in comparison to the last EUPT on cereals EUPT-C6. The 15 false positive results were: chlorpyrifos (1), DDT- p'p' (2), HCH-beta (5), hexaconazole (1), pendimethalin (1), permethrin (2), quinoxifen (1), tebufenozide (1) and trifluralin (1). The 50 false negative results concerned 17 different pesticides: aldrin (3), azoxystrobin (3), cypermethrin (2), endosulfan-alpha (5), endosulfan-sulfate (2), epoxiconazole (1), fenpropidin (4), fluquinconazole (1), flutriafol (5), kresoxim-methyl (4), lindane (3), malathion (1), pyraclostrobin (5), spiroxamine (2), tebuconazole (2), triadimenol (4) and trifloxystrobin (3). The average Qn-RSD (robust RSD) was at 20 %, close to the FFP-RSD of 25 % with a range from 15 to 29 % for the individual compounds.

For boscalid, chlorpyrifos-methyl, cypermethrin, endosulfan-sulfate, fenpropimorph, fluquinconazole, flutriafol, iprodione, kresoxim-methyl, lindane, malathion, propiconazole, spiromaxime and triadimenol acceptable results were obtained by 90-96% of the laboratories. For aldrin, azoxystrobin, carbendazim, endosulfan-alpha, epoxiconazole, fenpropidin, pyraclostrobin, tebuconazole and trifloxystrobin acceptable results were obtained by 84-89% of the laboratories.

The next EUPT will be on wheat and the selection of pesticides will continue to focus on pesticides included in the scope of the EU multi-annual coordinated control programme as well as additional pesticides of relevance to cereal production in Europe and in other parts of the world from where significant quantities of cereals materials are imported.

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-CF7

#### 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, Institute for Food Safety Innsbruck - Department for Pes	Innsbruck	NRL	Yes
Belgium	Fytolab	Gent - Zwijnaarde		Yes
Belgium	Federal Laboratory for Safety of Food Chain, Tervuren	Tervuren		Yes
Belgium	Scientific Institute of Public Health	Brussels	NRL	Yes
Bulgaria	Fytolab Bulgaria	Plovdiv		Yes
Bulgaria	Laboratory of SGS Bulgaria LTD	Varna		Yes
Cyprus	Animal Feeds and Feed Additives Laboratory of the Analytical Laboratories of the Department of Agric	Nicosia	NRL	Yes
Czech Republic	Central Institute for Supervising and Testing in Agriculture	Brno	NRL	Yes
Czech Republic	Czech Agriculture and Food Inspection Authority	Praha	NRL	Yes
Denmark	Danish Veterinary and Food Administration, Department of Residues, Ringsted	Ringsted	NRL	Yes
Estonia	Agricultural Research Centre, Saku, Lab for Residues and Contaminants	Saku	NRL	Yes
Finland	Finnish Food Safety Authority	Helsinki	NRL	Yes
France	Laboratoire Départemental d'Analyses des Cotes d'Armor	Ploufragan		Yes
France	Service Commun des Laboratoires / Laboratoire de Rennes	Rennes		Yes
France	Laboratoire Départemental d'Analyses de la Sarthe, Département de Chimie	Le Mans		Yes
France	Analysis Center Mediterranean Pyrenees	Perpignan		No
France	Laboratoire Départemental d'Analyses des LANDES	Mont de Marsan		Yes
France	Service Commun des Laboratoires / Laboratoire Ile de France - Massy	Massy Cedex	NRL	Yes
Germany	Landwirtschaftliche Untersuchungs- und Forschungsanstalt	Speyer		Yes
Germany	Institut für Hygiene und Umwelt Hamburg	Hamburg		No
Germany	LUFA-ITL GmbH	Kiel		Yes
Germany	Niedersächsisches Landesamt für Verbraucherschutz und Lebensmittelsicherheit, Stade	Stade		Yes
Germany	Chemical and Veterinary Analytical Institute Rhine-Ruhr-Wupper	Krefeld		Yes
Germany	State Department of Environmental and Agricultural Operations in Saxony	Nossen		Yes
Germany	Berlin-Brandenburg State Laboratory, Potsdam	Potsdam		Yes
Germany	Federal Office of Consumer Protection and Food Safety, NRL for Pesticide Residues	Berlin	NRL	Yes
Germany	Landwirtschaftliches Technologiezentrum Augustenberg, Karlsruhe	Karlsruhe		Yes
Germany	Landesbetrieb Hessisches Landeslabor, Wiesbaden	Wiesbaden		Yes

Country	Institution	City	NRL- CF	Report data
Germany	Landesanstalt für Landwirtschaft, Forsten und Gartenbau, Halle			Yes
Germany	Bavarian Health and Food Safety Authority Office Oberschleißheim Feed Analytics (Feedingstuff)	Oberschleißheim		Yes
Germany	Eurofins - Dr. Specht Laboratorien GmbH	Hamburg		Yes
Germany	Thuringian Institute of Agriculture	Jena		Yes
Germany	Food and Veterinary Institute Oldenburg	Oldenburg		Yes
Germany	Landwirtschaftliche Untersuchungs- und Forschungsanstalt Speyer	Speyer		Yes
Germany	Landesamt für Landwirtschaft, Lebensmittelsicherheit und Fischerei Mecklenburg-Vorpommern	Rostock		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	Central Agricultural Office, Feed Investigation National Reference Laboratory	Budapest		Yes
Hungary	Central Agricultural Office, Feed Investigation National Reference Laboratory	Hódmezővásárhely		Yes
Hungary	National Food Chain Safety Office, Directorate of Plant Protec- tion, Soil Conservation and Agri-environment, Pesticide Residue Analytical Laboratory, Szolnok	Szolnok		Yes
Hungary	National Food Chain Safety Office, Directorate of Plant Protection, Soil Conservation and Agri-envir	Velence	NRL	Yes
Hungary	National Food Chain Safety Office, Directorate of Plant Protec- tion, Soil Conservation and Agri-Envir	Miskolc		Yes
Ireland	Pesticide Control Laboratory, Department of Agriculture, Fisheries and Food	Co. Kildare	NRL	Yes
Italy	Istituto Superiore di Sanità, Pesticide Section	Roma		Yes
Italy	ARPAM Dipartimento di Macerata	Macerata		Yes
Italy	Public Health Laboratory - Florence	FLORENCE		Yes
Italy	Centro di referenza nazionale per la sorveglianza ed il controllo degli alimenti per animali	Genova	NRL	No
Italy	ARPA Veneto Dip.Reg.Lab. S.L. Verona	Verona		Yes
Italy	Istituto Zooprofilattico Sperimentale Abruzzo e Molise	Teramo		Yes
Italy	Istituto Zooprofilattico Sperimentale Lombardia ed Emilia Romagna	Brescia		Yes
Italy	Istituto Zooprofilattico Sperimentale Puglia e Basilicata	Foggia		Yes
Italy	ARPA Ferrara Eccellenza Fitofarmaci	Ferrara		Yes
Italy	APPA Bolzano	Bolzano		Yes
Italy	Environmental Regional Protection Agency - Laboratory of Pordenone	Pordenone		Yes
Italy	APPA Trento Settore Laboratorio e Controlli	Trento		Yes
Italy	Arpalazio Sezione P.Le di Latina - Servizio Laboratorio Ambiente Salute, Unita' di Chimica Inorgan	Latina		Yes

Country	Institution	City	NRL- CF	Report data
Italy	Istituto Zooprofilattico Sperimentale Lazio e Toscana	Roma		Yes
Italy	ARPA Puglia - Dipartimento di Bari	Bari		Yes
Italy	Istituto Zooprofilattico Sperimentale Umbria e Marche, PerugiaA	Perugia		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		Yes
Netherlands	Handelslaboratorium Dr. Verwey	Rotterdam		Yes
Netherlands	Grond-, Gewas- en Milieulaboratorium Zeeuws-Vlaanderen b.v.	Graauw		Yes
Netherlands	Netherlands Food and Consumer Products Safety Authority	Wageningen	NRL	Yes
Netherlands	Grond-, Gewas- en Milieulaboratorium Zeeuws-Vlaanderen b.v.	Graauw		Yes
Netherlands	RIKILT Institute of Food Safety (Natural Toxins & Pesticides)	Wageningen		Yes
Norway	Norwegian Institute for Agricultural and Environmental Research, Plant Health and Plant Protection D	Aas	NRL	Yes
Poland	Institute of Plant Protection Pesticide Residue Laboratory, Bialystok	Bialystok		Yes
Poland	Institute of Plant Protection - National Research Institute, Branch Sosnicowice	Sosnicowice		Yes
Poland	Regional Veterinary Laboratory in Katowice	Katowice		Yes
Poland	Provincial Veterinary Inspectorate Establishment of Veterinary Hygiene, Poznan	Poznan	NRL	Yes
Poland	Institute of Plant Protection - National Research Institute, Regional Experimental Station in Rzeszo	Rzeszow		Yes
Poland	Regional Veterinary Laboratory in Szczecin	Szczecin		Yes
Poland	Regional Veterinary Laboratory in Warsaw	Warszawa		Yes
Poland	Regional Veterinary Laboratory Wroclaw	Wroclaw		Yes
Poland	Institute of Plant Protection, Department of Pesticide Residue Research - Poznan	Poznan		Yes
Poland	Regional Veterinary Laboratory in Bialystok	Bialystok		Yes
Poland	Regional Veterinary Laboratory in Opole	Opole		Yes
Poland	Regional Veterinary Laboratory in Gdansk (Kartuska)	Gdansk		Yes
Portugal	Regional Laboratory of Veterinary and Food Safety - Madeira Island	Funchal - Madeira Island		Yes
Portugal	Direção Regional de Agricultura e Pescas do Norte	Senhora da Hora (Matosinhos)		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	Sanitary Veterinary and Food Safety Directorate, Bucharest	Bucharest		Yes
Romania	Central Laboratory for Pesticides Residues Control in Plants and Vegetable Products - Bucharest	Bucharest	NRL	Yes
Romania	Institute for Hygiene and Veterinary Public Health - Bucharest	Bucharest	NRL	Yes
Slovakia	Public Health Authority of the Slovak Republic	Bratislava		Yes

Country	Institution	City	NRL- CF	Report data
Slovakia	State Veterinary and Food Institute Bratislava	Bratislava	NRL	Yes
Slovenia	Institute of Public Health, Maribor	Maribor		Yes
Slovenia	Institute of Public Health, Maribor	Maribor		Yes
Slovenia	Agricultural Institute of Slovenia, Central Laboratories	Ljubljana	NRL	Yes
Slovenia	Institute of Public Health, Maribor	Maribor		Yes
Spain	Laboratorio de Salud Pública de Badajoz	Badajoz		Yes
Spain	Laboratory of Barcelona Public Health Agency	Barcelona		Yes
Spain	Agricultural and Phytopathological Laboratory of Galicia	Abegondo. A Coruña		Yes
Spain	Laboratori Agroalimentari de la Generalitat de Catalunya	Cabrils		Yes
Spain	National Centre for Food - Spain, Majadahonda	Majadahonda	NRL	Yes
Spain	Servicio de Laboratorio y Control de Santander	Santander		Yes
Spain	Laboratorio Regional CCAA La Rioja	Logroño		Yes
Spain	Laboratorio Agroalimentario de Zaragoza	Zaragoza		Yes
Spain	Agrofood Laboratory of the Comunidad Valenciana	Burjassot- Valencia		Yes
Spain	Laboratorio Agrario Regional - Junta de Castilla y Leon	Burgos		Yes
Sweden	National Food Agency, Science Department, Chemistry Division 1	Uppsala	NRL	Yes
Sweden	Eurofins - Food&Agro Sweden, Lidköping	Lidköping		Yes
Switzerland	Kantonaes Laboratorium Zürich	Zürich		Yes
United King- dom	The Food and Environment Research Agency - York	York	NRL	Yes
United King- dom	Agri-Food and Biosciences Institute	Belfast		Yes

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

Country	Institution	City	Report data
Argentina	INTI - LACTEOS	San Martin - PCIA Buenos Aires	Yes
Brasil	AgroSafety Monitoramento Agrícola Ltda - EPP	Piracicaba/SP	Yes
Brazil	Bioensaios Analises e Consultoria Ambiental	Viamao	No
Chile	Instituto de Salud Pública de Chile, Sección metrología ambiental y de Alimentos	Santiago	Yes
Costa Rica	Centro de Investigación en Contaminación Ambiental, Universidad de Costa Rica	San José	Yes
Costa Rica	Laboratorio de Análisis de Residuos de Agroquímicos	San José	Yes
Egypt	Central Lab of Residue Analysis of Pesticides and Heavy Metals in Foods	Giza	Yes
Ghana	Food and Drugs Authority Laboratory	Cantonments-Accra	No
Kenya	Kenya Bureau of Standards (KEBS)	Nairobi	No
Kenya	KEPHIS-Analytical Chemistry Laboratory	Nairobi	Yes
Peru	Senasa-Unidad del Centro de ControlL de Insumos y Residuos Toxicos	Lima	No
Serbia	SP Laboratorija	Becej	Yes
Singapore	Veterinary Public Health Laboratory	Singapore	Yes
Turkey	SGS Mersin Food Control Laboratory	Mersin	Yes





## Appendix 2 Target Pesticide List

Pesticides	MRRL (mg/kg)
2-phenyl phenol	0.01
3-hydroxy-carbofuran	0.01
Acephate	0.01
<b>Aldrin</b>	0.01
Azinphos-methyl	0.01
Azoxystrobin	0.01
Bifenthrin	0.01
Boscalid	0.01
Captan	0.01
Carbaryl	0.01
Carbendazim	0.01
Carbofuran	0.01
Carboxin	0.01
<b>Chlordane, cis-</b>	0.02
<b>Chlordane, oxy</b>	0.02
<b>Chlordane, trans-</b>	0.02
Chlorfenvinphos	0.01
Chlorothanlonil	0.01
Chlorpropham (only parent compound)	0.01
Chlorpyrifos	0.01
Chlorpyrifos-methyl	0.01
Cis-deltamethrin (cis-deltamethrin)	0.01
Clothianidin	0.01
Cyfluthrin (cyfluthrin incl. other mixtures of constituent isomers (sum of isomers))	0.01
Cypermethrin (cypermethrin incl. other mixtures of constituent isomers (sum of isomers))	0.01
Cyproconazole	0.01
Cyprodinil	0.01
Demeton-S-methylsulfone	0.01
Diazinon	0.01
Dichlorvos	0.01
<b>Dieldrin</b>	0.01
Difenoconazole	0.01
Diflubenzuron	0.01
Dimethoate	0.01
Endosulfan-alpha	0.01
Endosulfan-beta	0.01
Endosulfan-sulfate	0.01
<b>Endrin</b>	0.01
Epoxiconazole	0.01
Ethion	0.01
Fenbuconazole	0.01

Pesticides	MRRL (mg/kg)
Fenhexamid	0.01
Fenitrothion	0.01
Fenpropidin	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
<b>Heptachlor</b>	0.01
<b>Heptachlorepoxyd-cis</b>	0.01
<b>Heptachlorepoxyd-trans</b>	0.01
Hexaconazole	0.01
Imazalil	0.01
Imidacloprid	0.01
Iprodione	0.01
Isoporthiolane	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
o,p'-DDT	0.01
Omethoate	0.01
Oxydemeton-methyl	0.01
p,p'-DDE	0.01
p,p'-DDT	0.01

Pesticides	MRRL (mg/kg)
p,p'-TDE	0.01
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	0.01
Pirimiphos-methyl	0.01
Prochloraz (parent compound only)	0.01
Procymidone	0.01
Propiconazole	0.01
Prothioconazole (Prothioconazole-desthio)	0.01
Pyraclostrobin	0.01
Pyrimethanil	0.01
Quinoxifen	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 (only parent compound)	0.01

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.	Aldrin, mg/kg		Azoxystrobin, mg/kg		Boscalid, mg/kg	
	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
5	0.394	0.365	0.087	0.088	0.144	0.106
30	0.320	0.278	0.082	0.088	0.148	0.132
76	0.262	0.378	0.093	0.102	0.139	0.129
126	0.350	0.353	0.111	0.105	0.137	0.143
162	0.308	0.334	0.112	0.106	0.133	0.130
188	0.286	0.342	0.109	0.111	0.140	0.109
208	0.375	0.357	0.120	0.118	0.112	0.143
226	0.300	0.282	0.115	0.117	0.114	0.128
240	0.300	0.340	0.117	0.116	0.137	0.144
310	0.363	0.288	0.111	0.113	0.129	0.144

Sample no.	Carbendazim, mg/kg		Chlorpyrifos-methyl, mg/kg		Cypermethrin, mg/kg	
	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
5	0.218	0.241	0.280	0.251	0.297	0.236
30	0.258	0.222	0.235	0.203	0.250	0.192
76	0.229	0.225	0.210	0.265	0.207	0.295
126	0.214	0.222	0.266	0.257	0.267	0.255
162	0.226	0.230	0.237	0.241	0.254	0.230
188	0.204	0.246	0.229	0.248	0.188	0.212
208	0.215	0.233	0.265	0.255	0.269	0.229
226	0.210	0.224	0.246	0.226	0.198	0.242
240	0.246	0.232	0.234	0.262	0.196	0.238
310	0.225	0.238	0.248	0.223	0.250	0.219

Sample no.	Endosulfan-alpha, mg/kg		Endosulfan-sulfate, mg/kg		Epoiconazole, mg/kg	
	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
5	0.258	0.238	0.323	0.308	0.120	0.142
30	0.222	0.211	0.300	0.287	0.144	0.139
76	0.202	0.240	0.291	0.312	0.136	0.138
126	0.248	0.238	0.302	0.324	0.113	0.122
162	0.224	0.231	0.301	0.293	0.125	0.134
188	0.235	0.239	0.355	0.340	0.110	0.142
208	0.244	0.237	0.281	0.370	0.124	0.130
226	0.228	0.215	0.338	0.254	0.111	0.132
240	0.228	0.247	0.349	0.329	0.134	0.125
310	0.237	0.217	0.287	0.298	0.128	0.141

Sample no.	Fenpropidin, mg/kg		Fenpropimorph, mg/kg		Fluquinconazole, mg/kg	
	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
5	0.110	0.117	0.392	0.367	0.108	0.081
30	0.114	0.101	0.344	0.299	0.109	0.099
76	0.104	0.096	0.305	0.375	0.105	0.096
126	0.099	0.106	0.372	0.361	0.099	0.108
162	0.097	0.100	0.355	0.344	0.097	0.098
188	0.108	0.078	0.332	0.372	0.105	0.083
208	0.099	0.111	0.379	0.374	0.085	0.110
226	0.082	0.082	0.364	0.316	0.085	0.096
240	0.113	0.109	0.339	0.372	0.102	0.109
310	0.093	0.104	0.353	0.331	0.093	0.104

Sample no.	Flutriafol, mg/kg		Iprodione, mg/kg		Kresoxim- methyl, mg/kg	
	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
5	0.374	0.318	0.343	0.263	0.075	0.072
30	0.310	0.284	0.353	0.307	0.072	0.076
76	0.287	0.355	0.335	0.319	0.070	0.062
126	0.351	0.341	0.328	0.346	0.083	0.066
162	0.323	0.310	0.326	0.318	0.072	0.072
188	0.263	0.304	0.354	0.287	0.069	0.066
208	0.358	0.308	0.276	0.349	0.066	0.068
226	0.290	0.312	0.300	0.306	0.069	0.070
240	0.290	0.331	0.336	0.347	0.064	0.066
310	0.324	0.300	0.305	0.344	0.066	0.068

Sample no.	Lindane, mg/kg		Malathion, mg/kg		Propiconazole, mg/kg	
	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
5	0.329	0.304	0.314	0.250	0.236	0.212
30	0.274	0.231	0.277	0.220	0.210	0.190
76	0.229	0.316	0.241	0.304	0.199	0.233
126	0.322	0.304	0.286	0.283	0.233	0.223
162	0.278	0.285	0.271	0.245	0.214	0.209
188	0.266	0.296	0.218	0.240	0.193	0.206
208	0.308	0.310	0.270	0.261	0.228	0.212
226	0.288	0.248	0.246	0.255	0.205	0.210
240	0.277	0.308	0.230	0.270	0.200	0.223
310	0.304	0.246	0.260	0.247	0.216	0.211

Sample no.	Pyraclostrobin, mg/kg		Spiroxamin, mg/kg		Tebuconazole, mg/kg	
	Portion 1	Portion 2	Portion 1	Portion 2	Portion 1	Portion 2
5	0.081	0.093	0.166	0.188	0.080	0.068
30	0.088	0.084	0.202	0.175	0.071	0.061
76	0.084	0.072	0.182	0.191	0.065	0.078
126	0.086	0.078	0.159	0.182	0.080	0.073
162	0.082	0.086	0.170	0.184	0.071	0.066
188	0.086	0.084	0.157	0.200	0.060	0.068
208	0.076	0.084	0.163	0.197	0.079	0.068
226	0.082	0.088	0.156	0.192	0.064	0.069
240	0.084	0.084	0.182	0.179	0.062	0.072
310	0.086	0.086	0.176	0.214	0.071	0.068

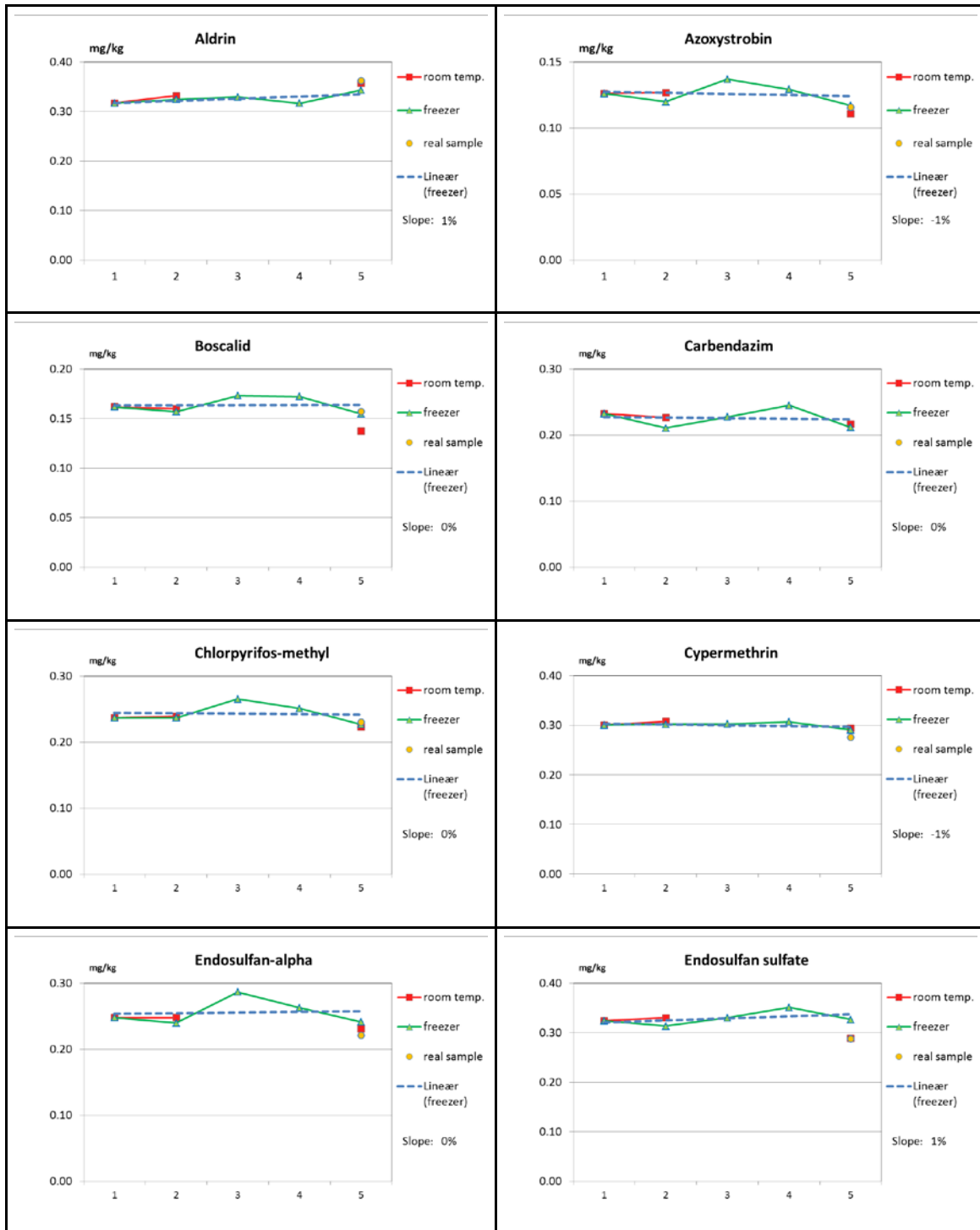
Sample no.	Triadimenol, mg/kg		Trifloxystrobin, mg/kg	
	Portion 1	Portion 2	Portion 1	Portion 2
5	0.263	0.221	0.056	0.052
30	0.238	0.215	0.052	0.052
76	0.222	0.264	0.049	0.056
126	0.244	0.246	0.057	0.053
162	0.251	0.226	0.055	0.051
188	0.216	0.223	0.049	0.052
208	0.240	0.239	0.056	0.051
226	0.234	0.233	0.053	0.048
240	0.220	0.244	0.053	0.055
310	0.233	0.243	0.052	0.056

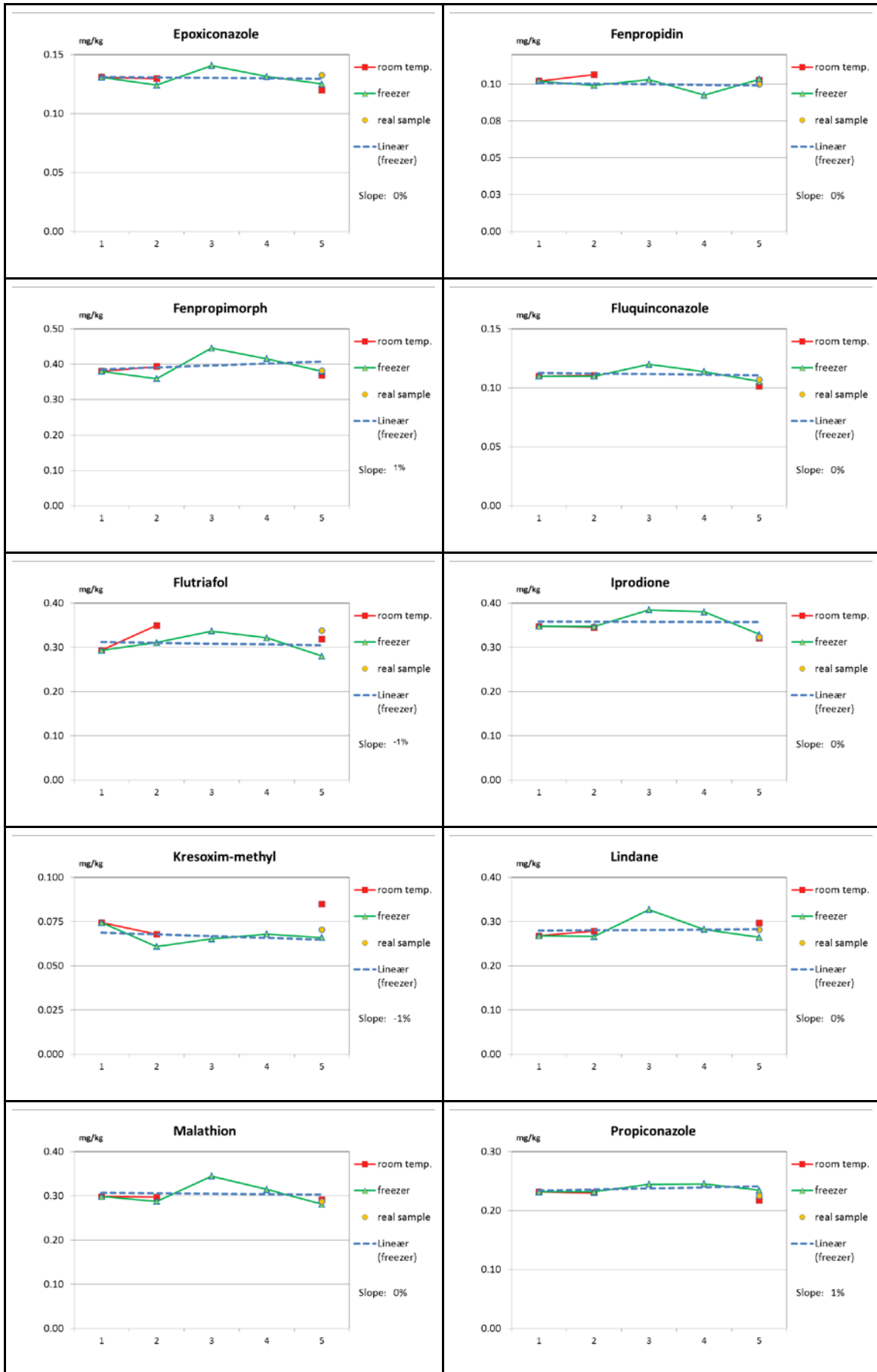


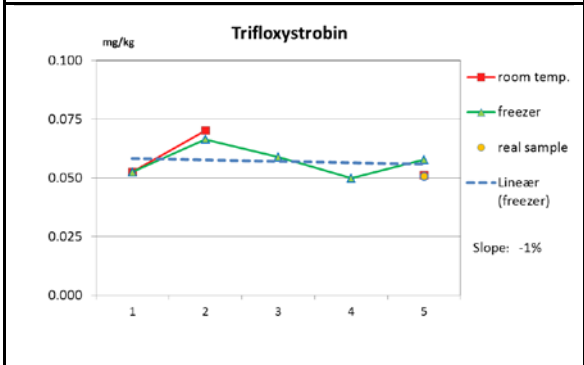
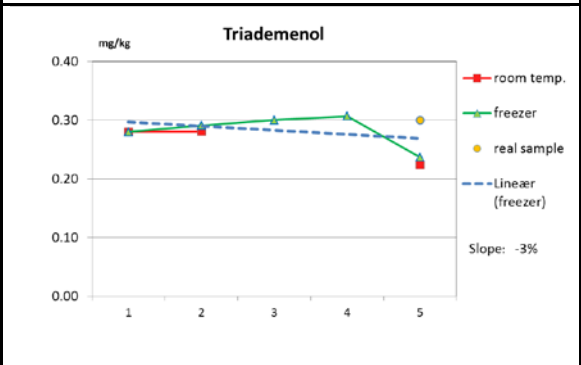
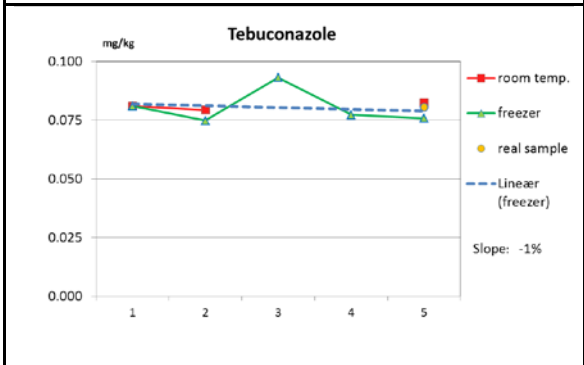
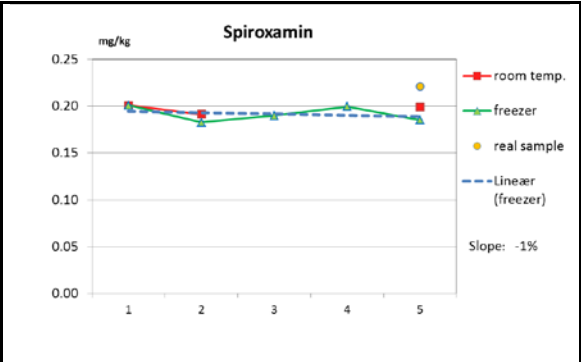
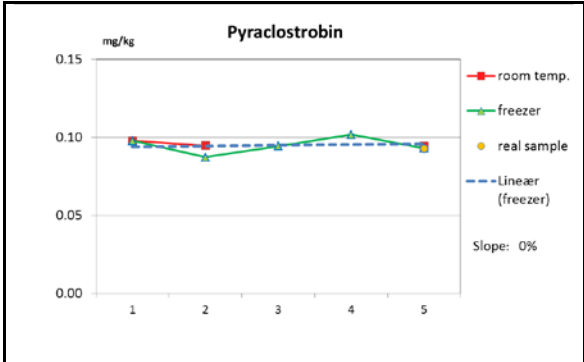


## Appendix 4 Stability figures

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%. The 'real sample' is Test Item no. 213 that originally was shipped to Brazil but rejected at the border and shipped back to EURL-CF

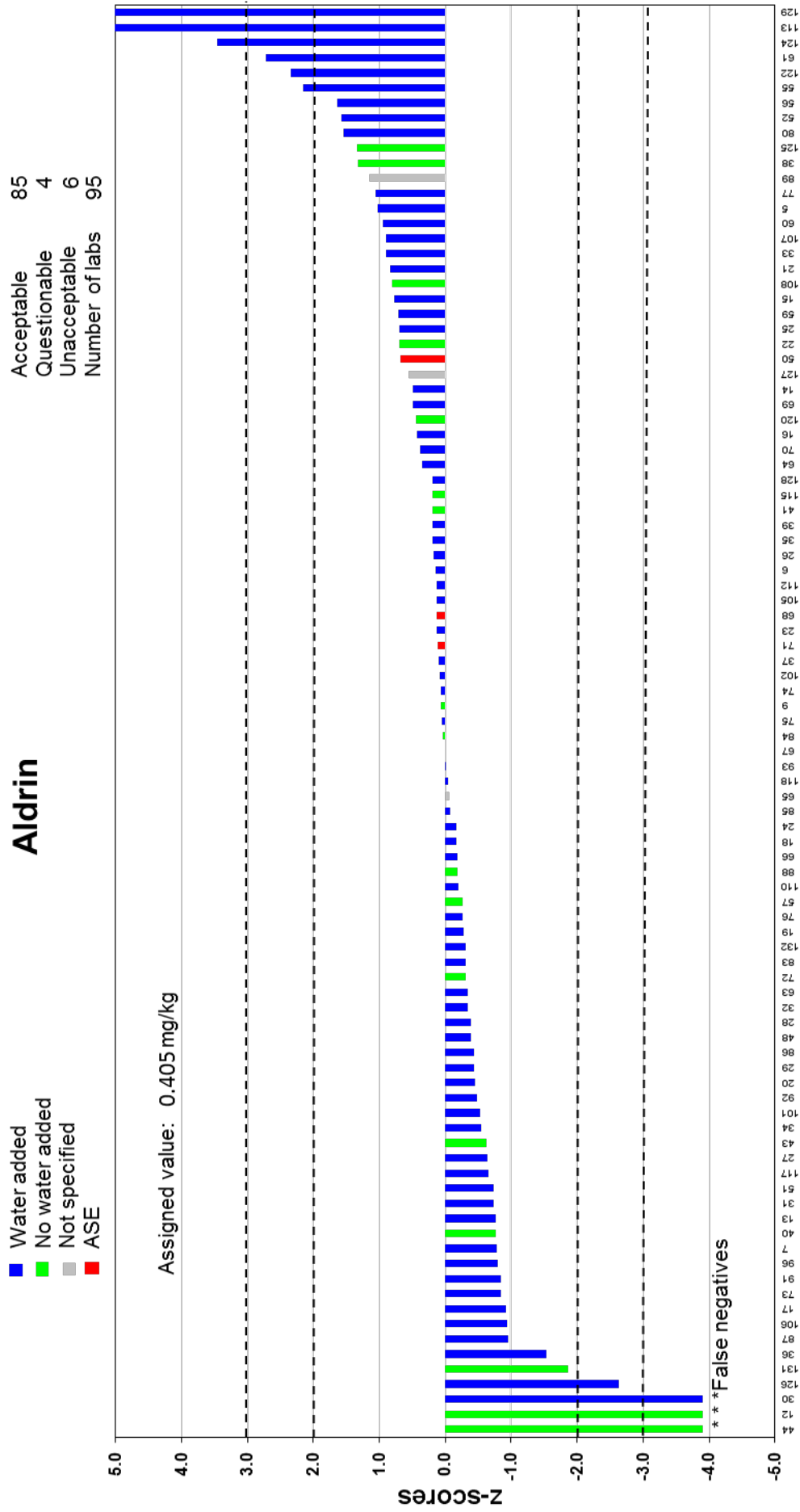








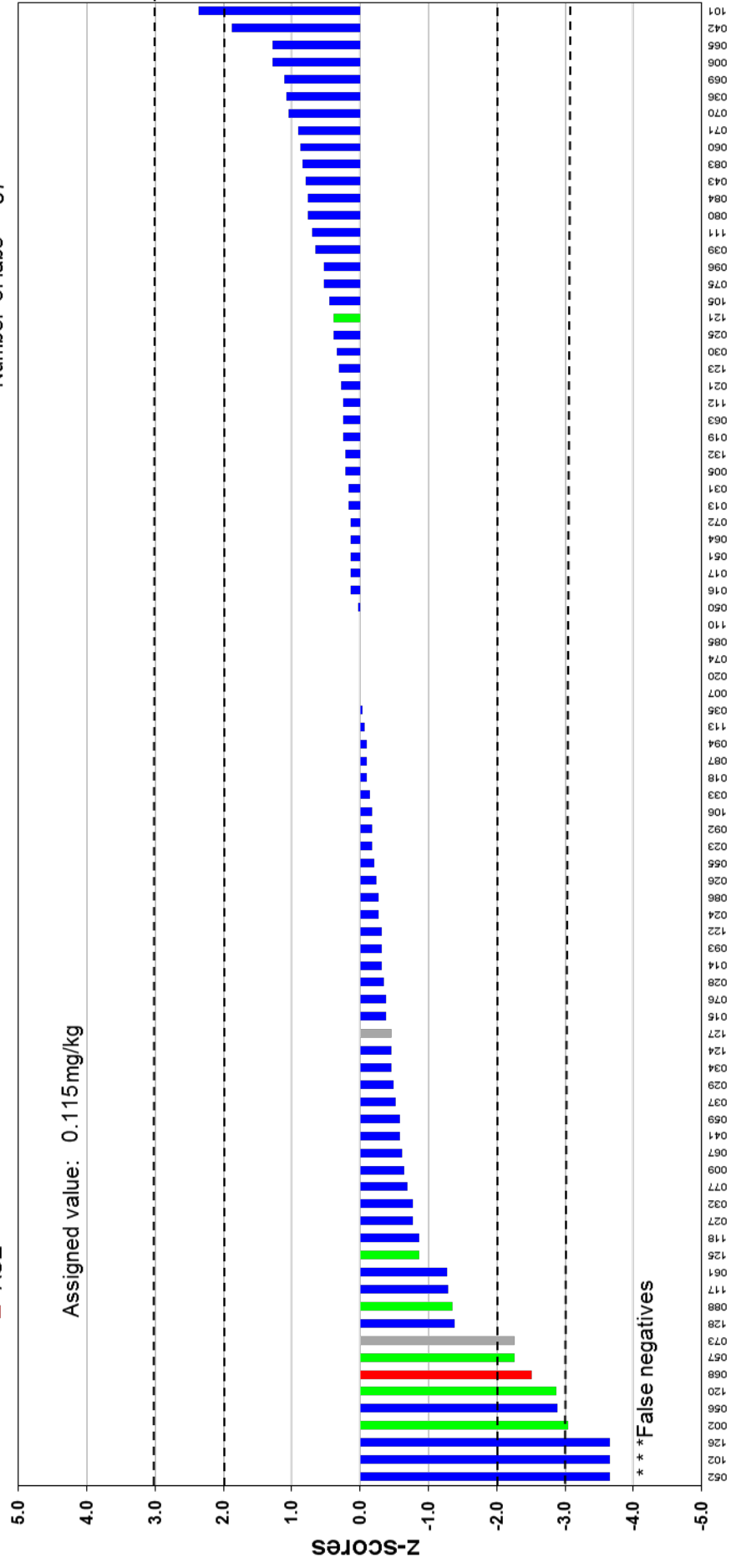
Appendix 5 Graphical presentation of z-scores



# Azoxystrobin

Acceptable 77  
 Questionable 6  
 Unacceptable 4  
 Number of labs 87

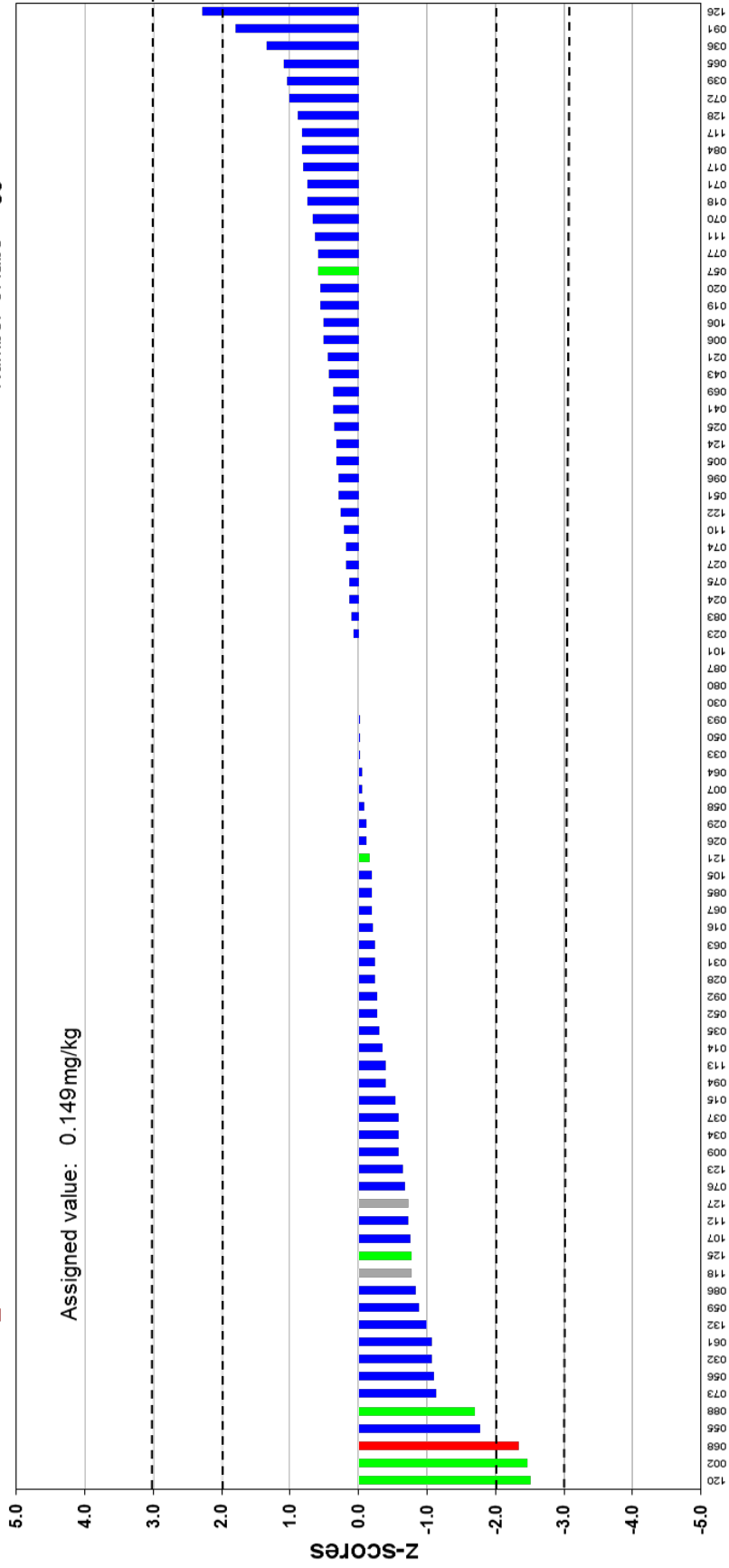
Water added  
 No water added  
 Not specified  
 ASE

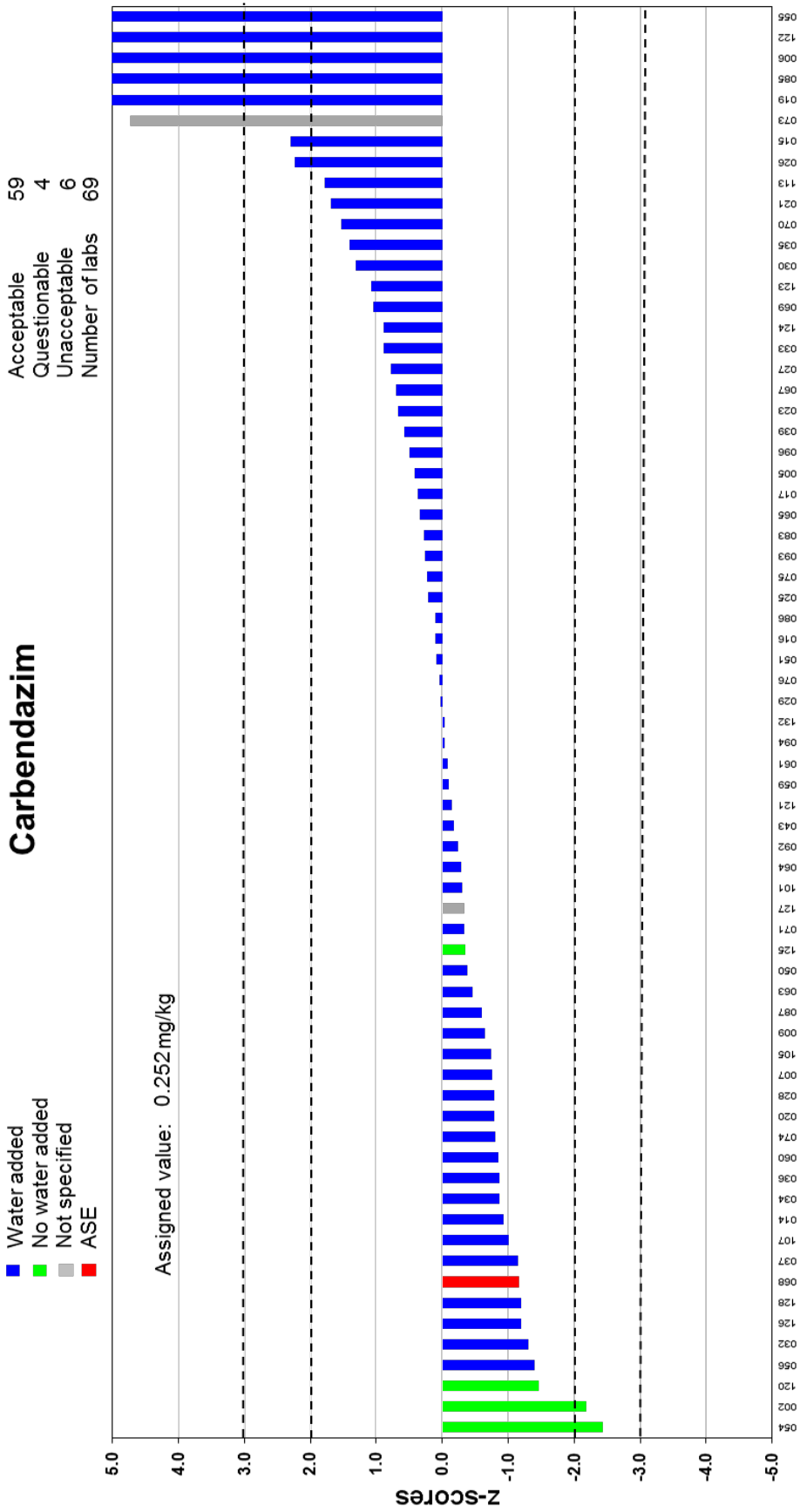


# Boscalid

- Water added
- No water added
- Not specified
- ASE

Acceptable 82  
 Questionable 4  
 Unacceptable 0  
 Number of labs 86



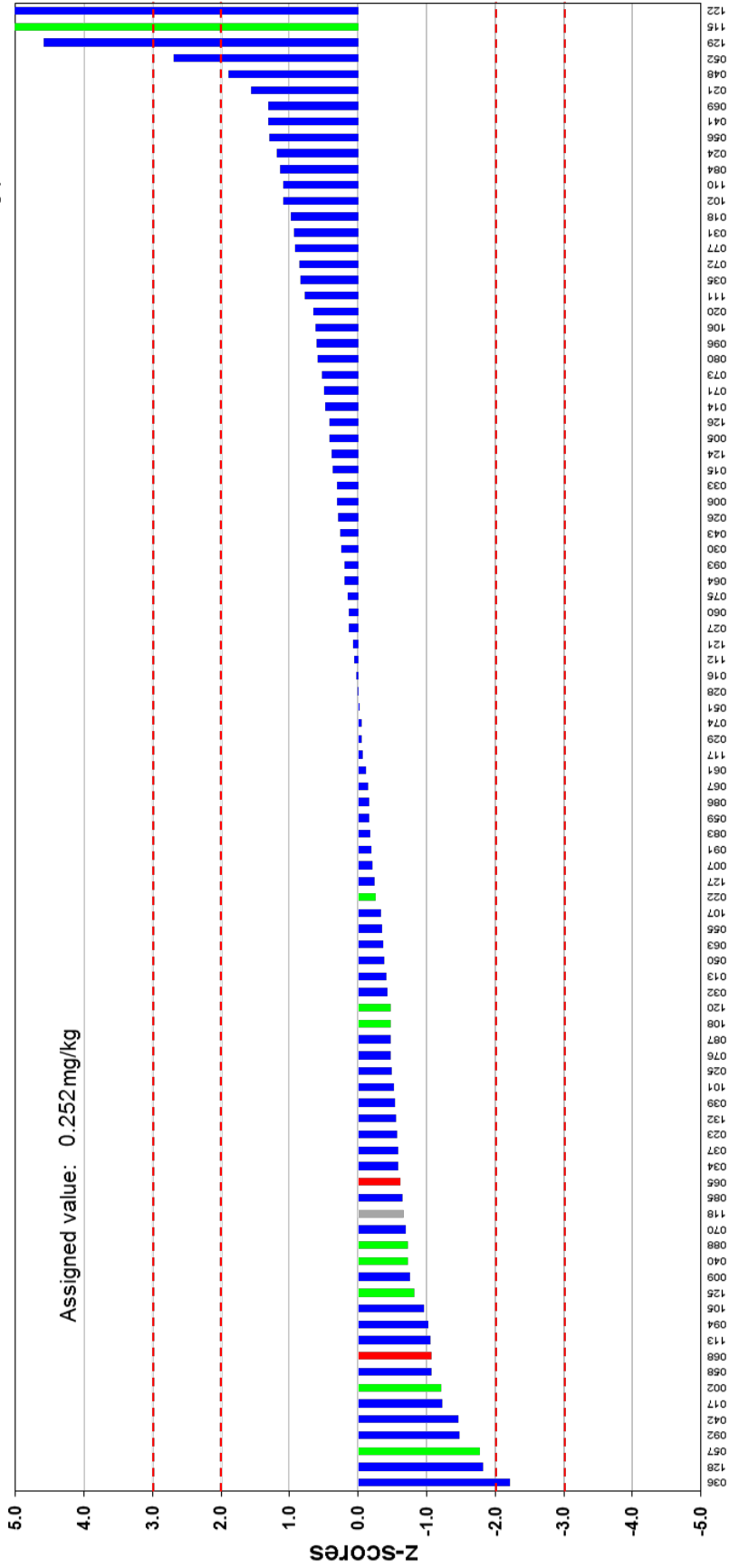




# Chlorpyrifos-methyl

- Water added
- No water added
- Not specified
- ASE

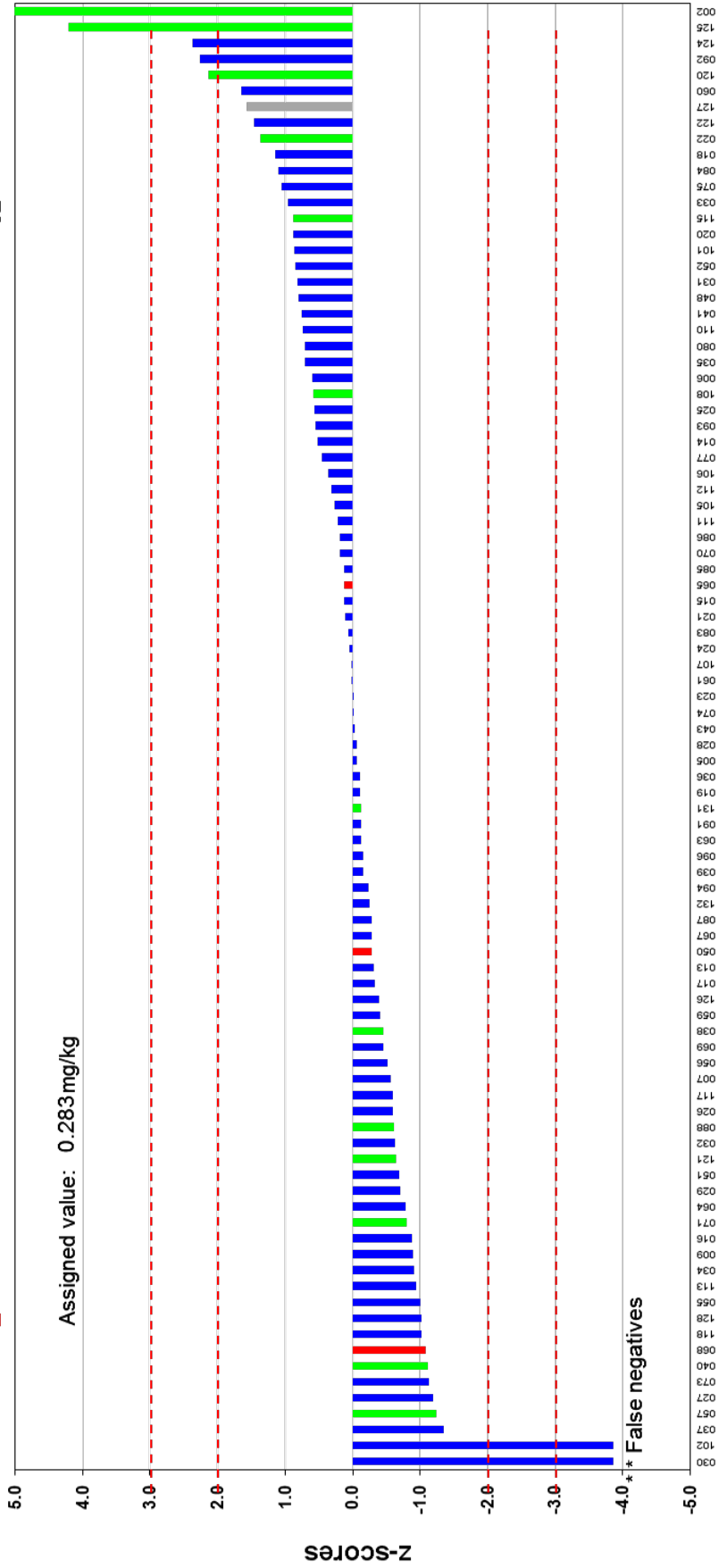
Acceptable 89  
 Questionable 2  
 Unacceptable 3  
 Number of labs 94



# Cypermethrin

Acceptable 85  
 Questionable 3  
 Unacceptable 4  
 Number of labs 92

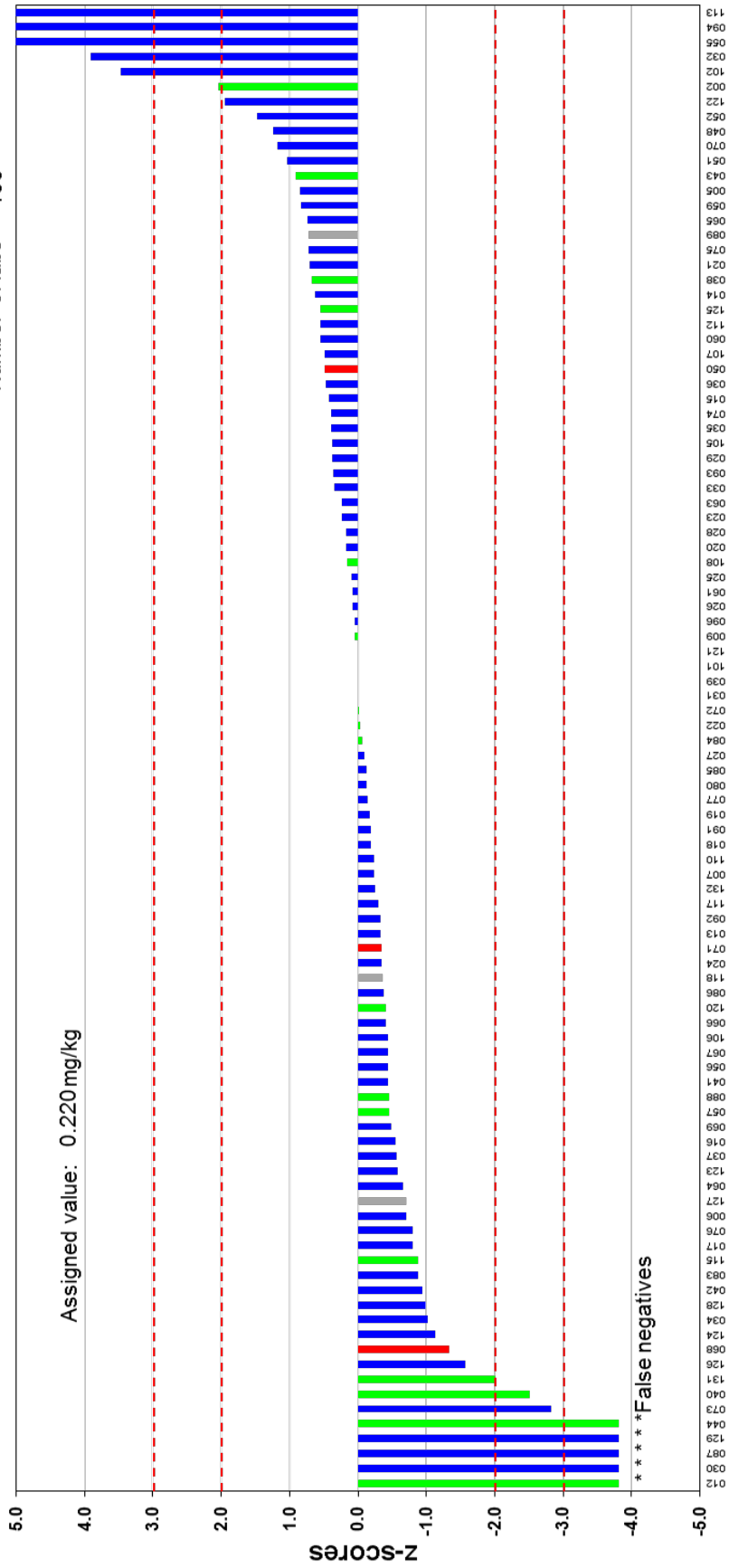
Water added  
 No water added  
 Not specified  
 ASE



# Endosulfan-alpha

Acceptable 88  
 Questionable 2  
 Unacceptable 10  
 Number of labs 100

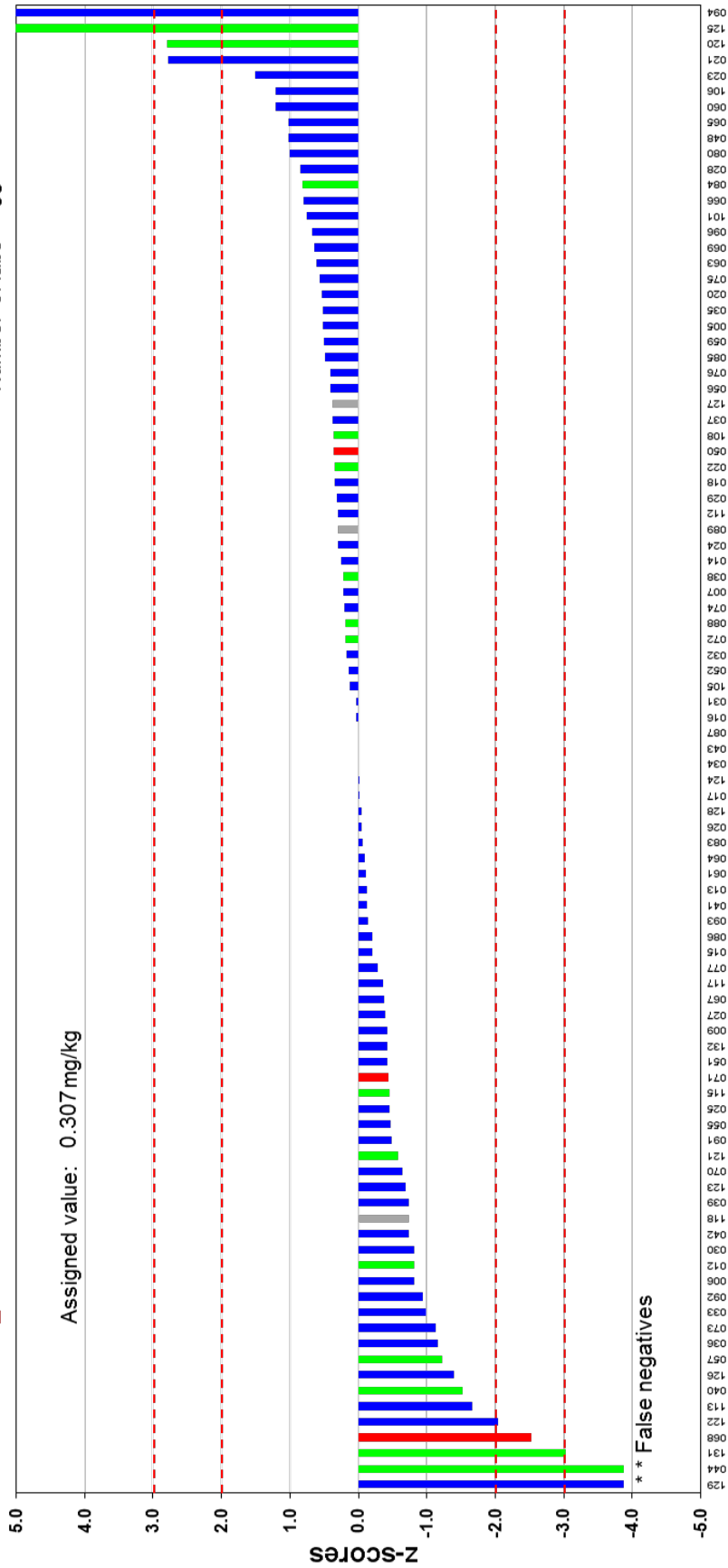
Water added  
 No water added  
 Not specified  
 ASE



# Endosulfan-sulfate

Acceptable 87  
 Questionable 4  
 Unacceptable 4  
 Number of labs 95

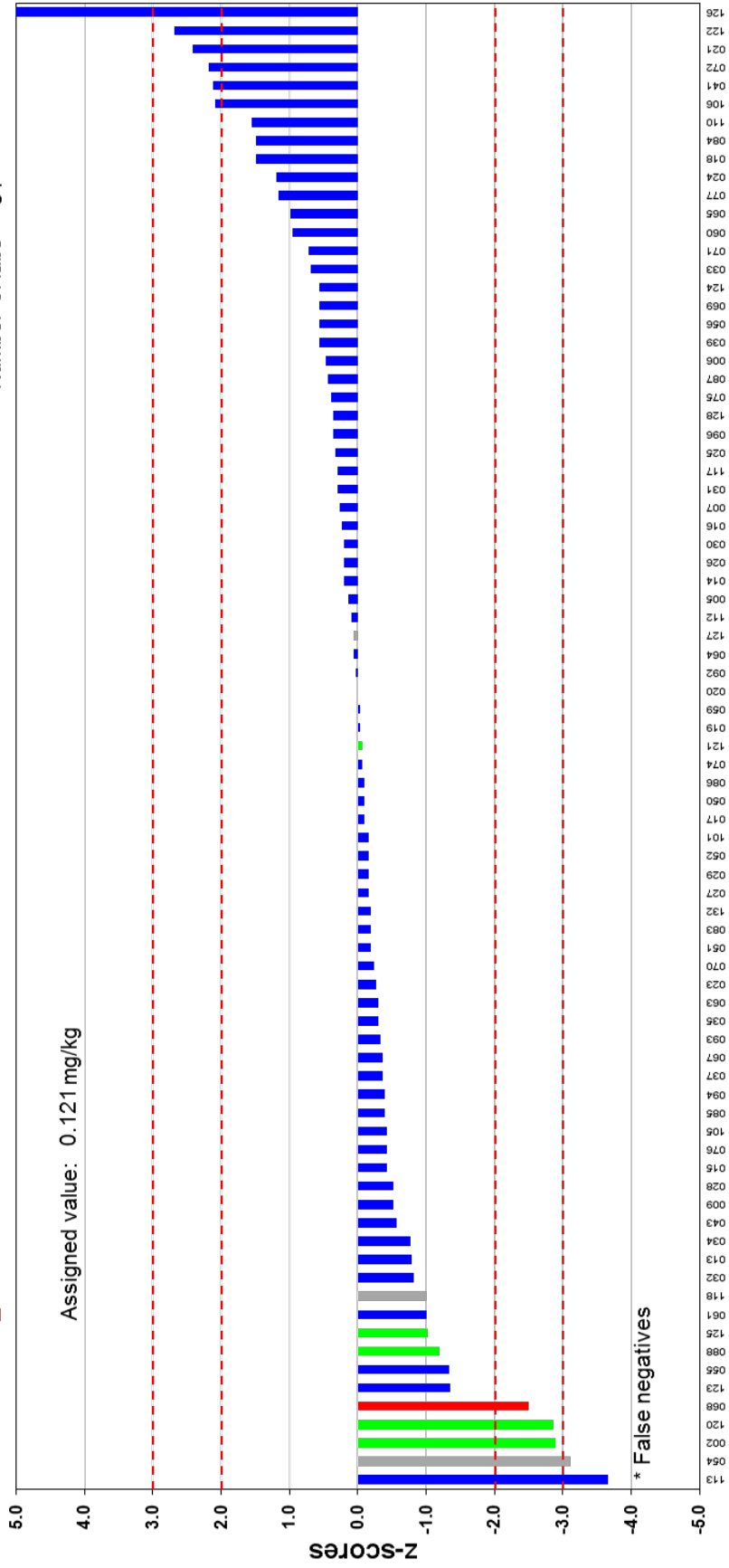
Water added  
 No water added  
 Not specified  
 ASE

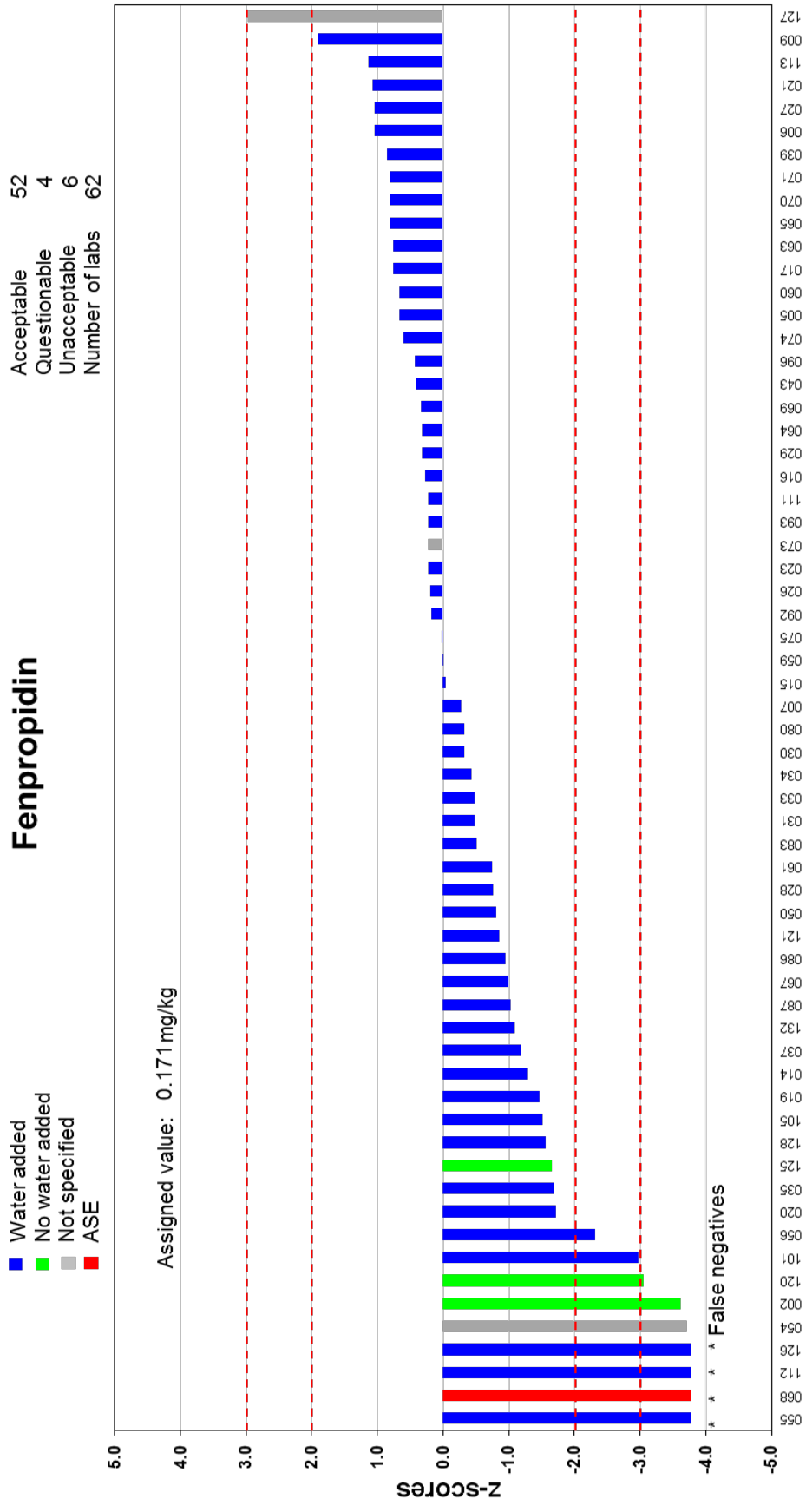


# Epoxiconazole

Acceptable 70  
 Questionable 8  
 Unacceptable 3  
 Number of labs 81

Water added  
 No water added  
 Not specified  
 ASE

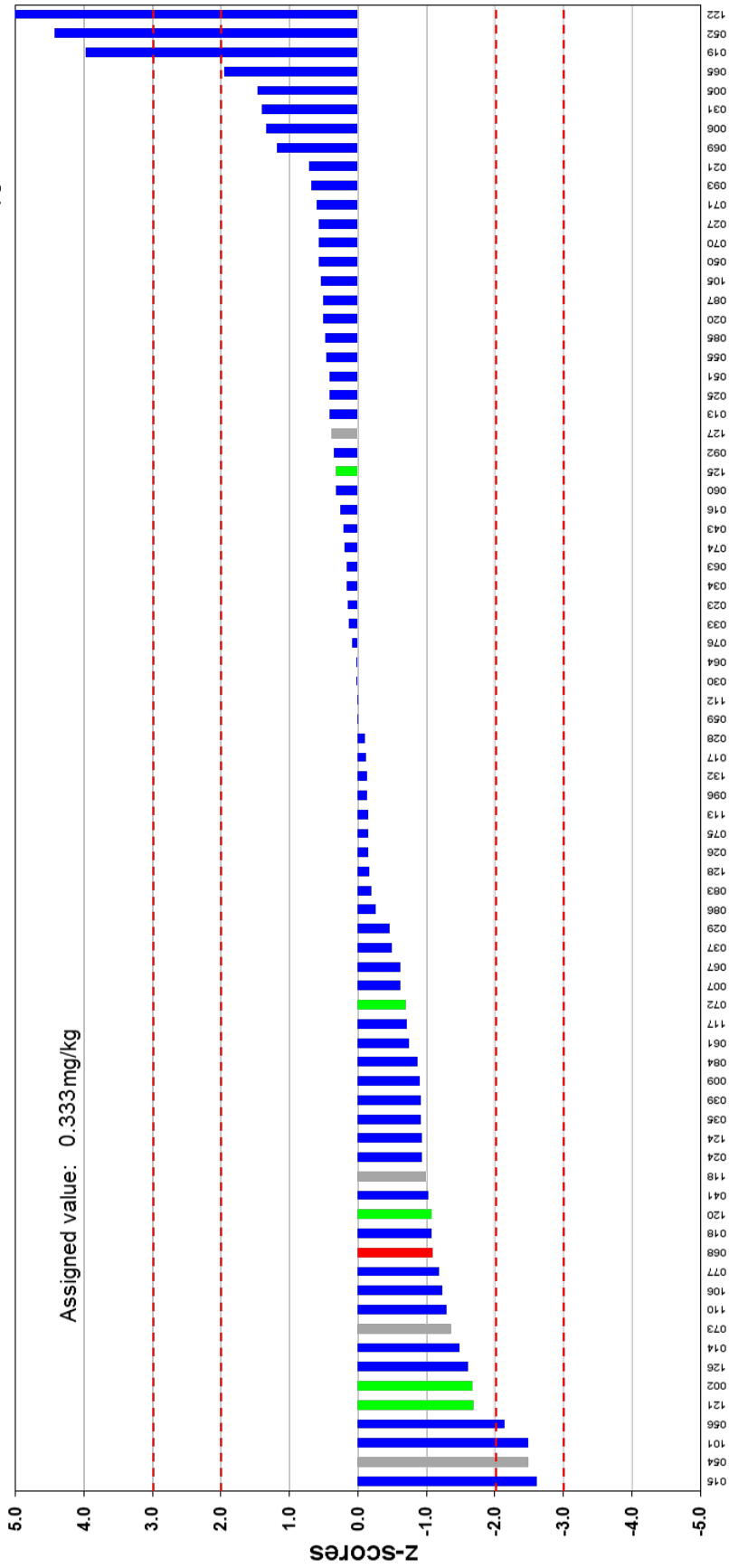


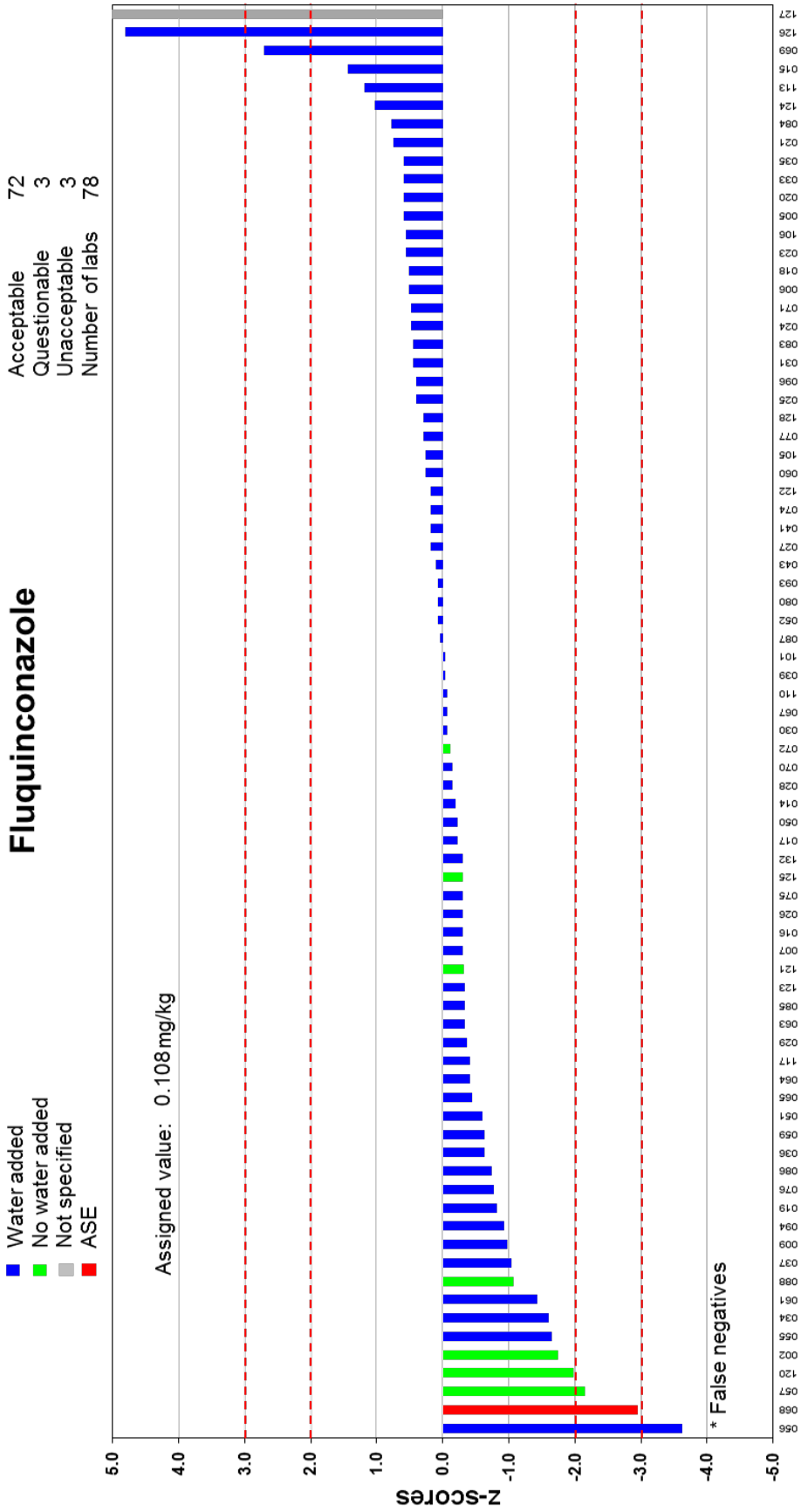


# Fenpropimorph

Acceptable 71  
 Questionable 4  
 Unacceptable 3  
 Number of labs 78

Water added  
 No water added  
 Not specified  
 ASE



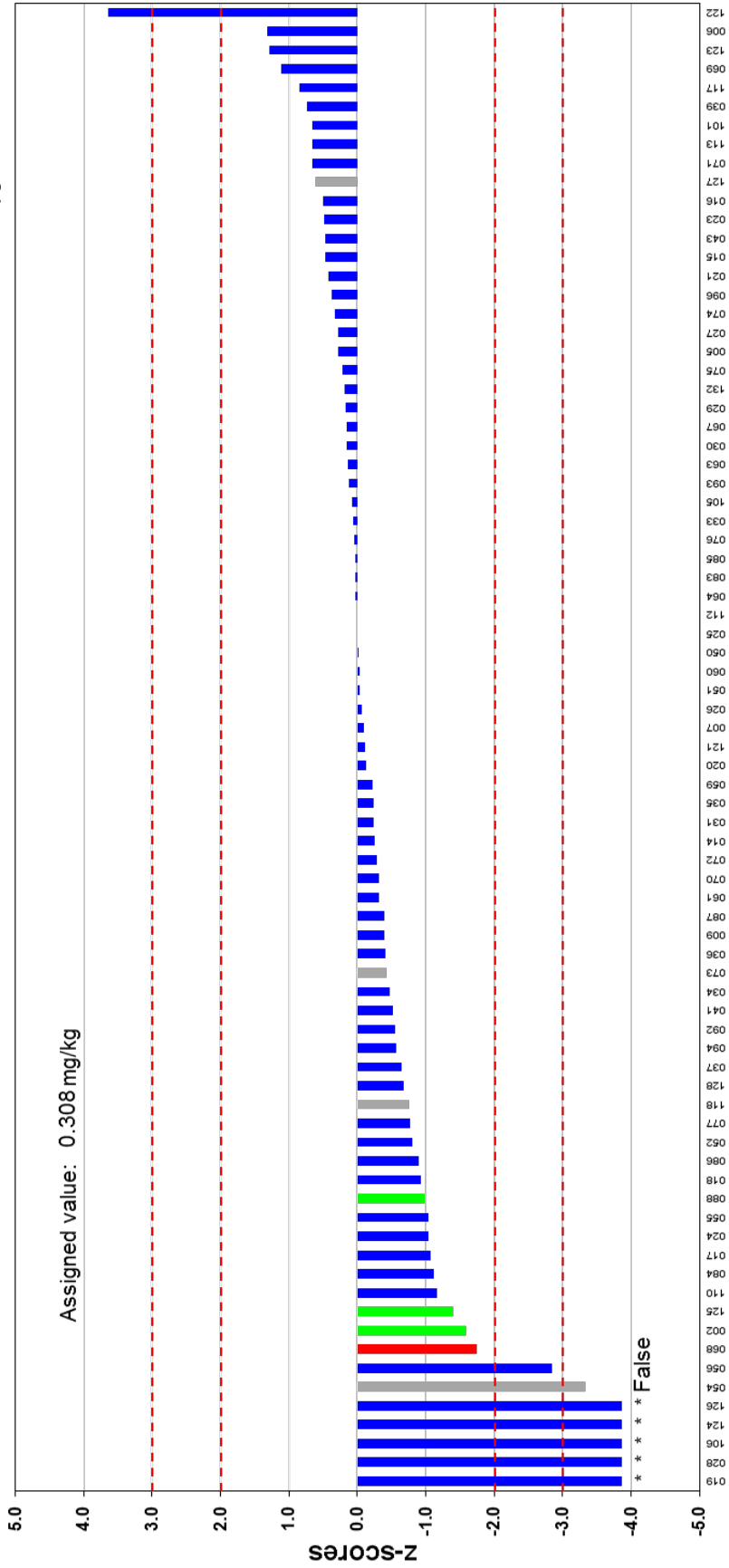




# Flutriafol

- Water added
- No water added
- Not specified
- ASE

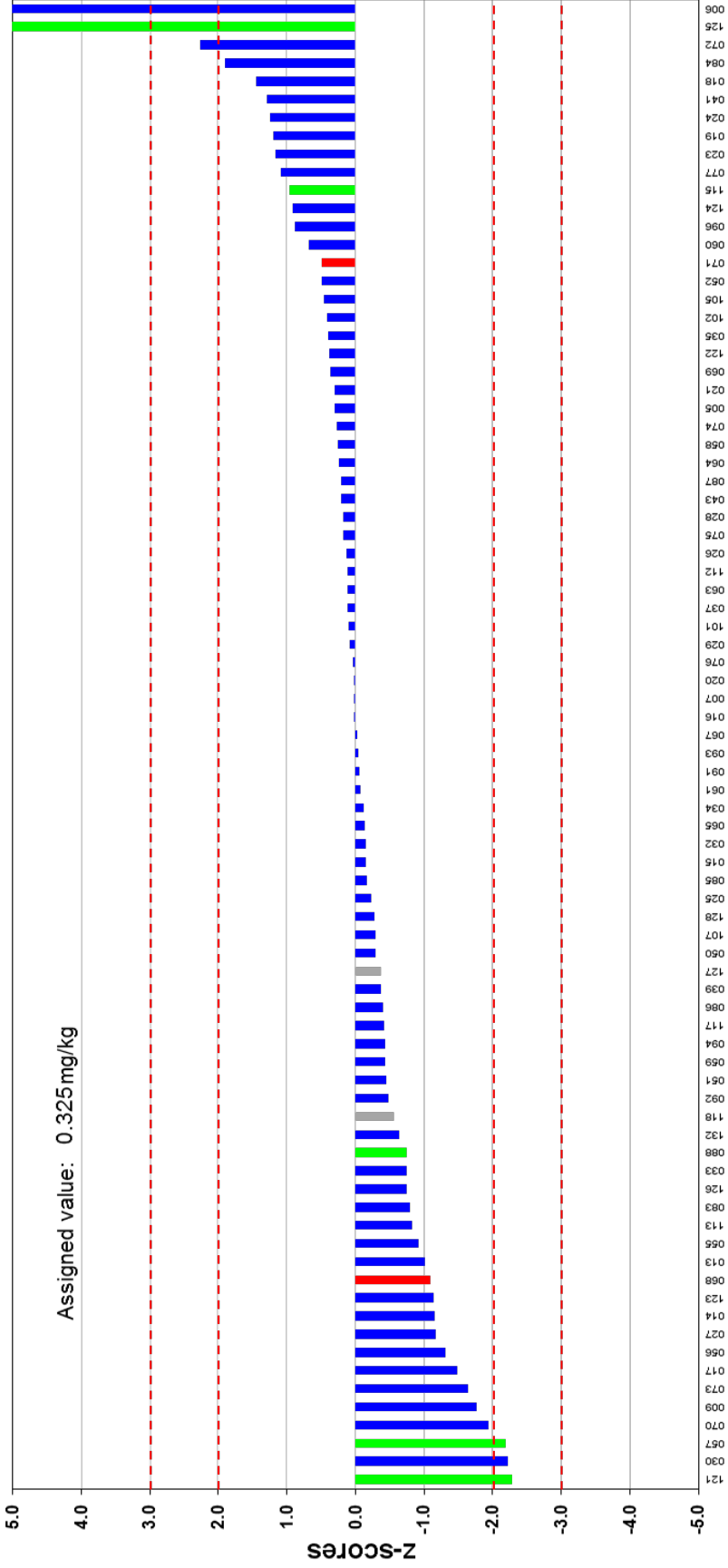
- Acceptable 71
- Questionable 1
- Unacceptable 7
- Number of labs 79



# Iprodione

- Water added
- No water added
- Not specified
- ASE

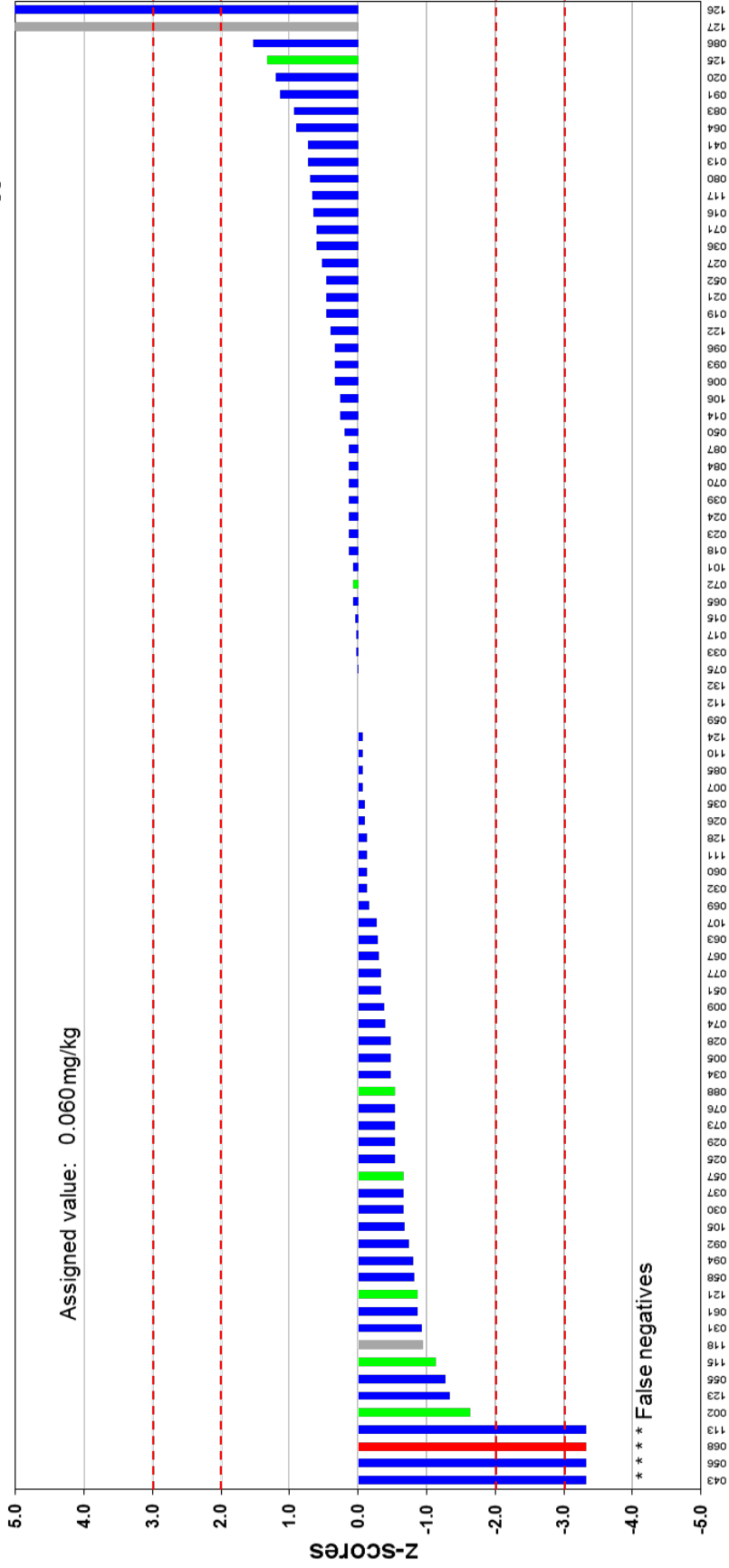
- Acceptable 76
- Questionable 4
- Unacceptable 2
- Number of labs 82



# Kresoxim-methyl

Acceptable 82  
 Questionable 0  
 Unacceptable 6  
 Number of labs 88

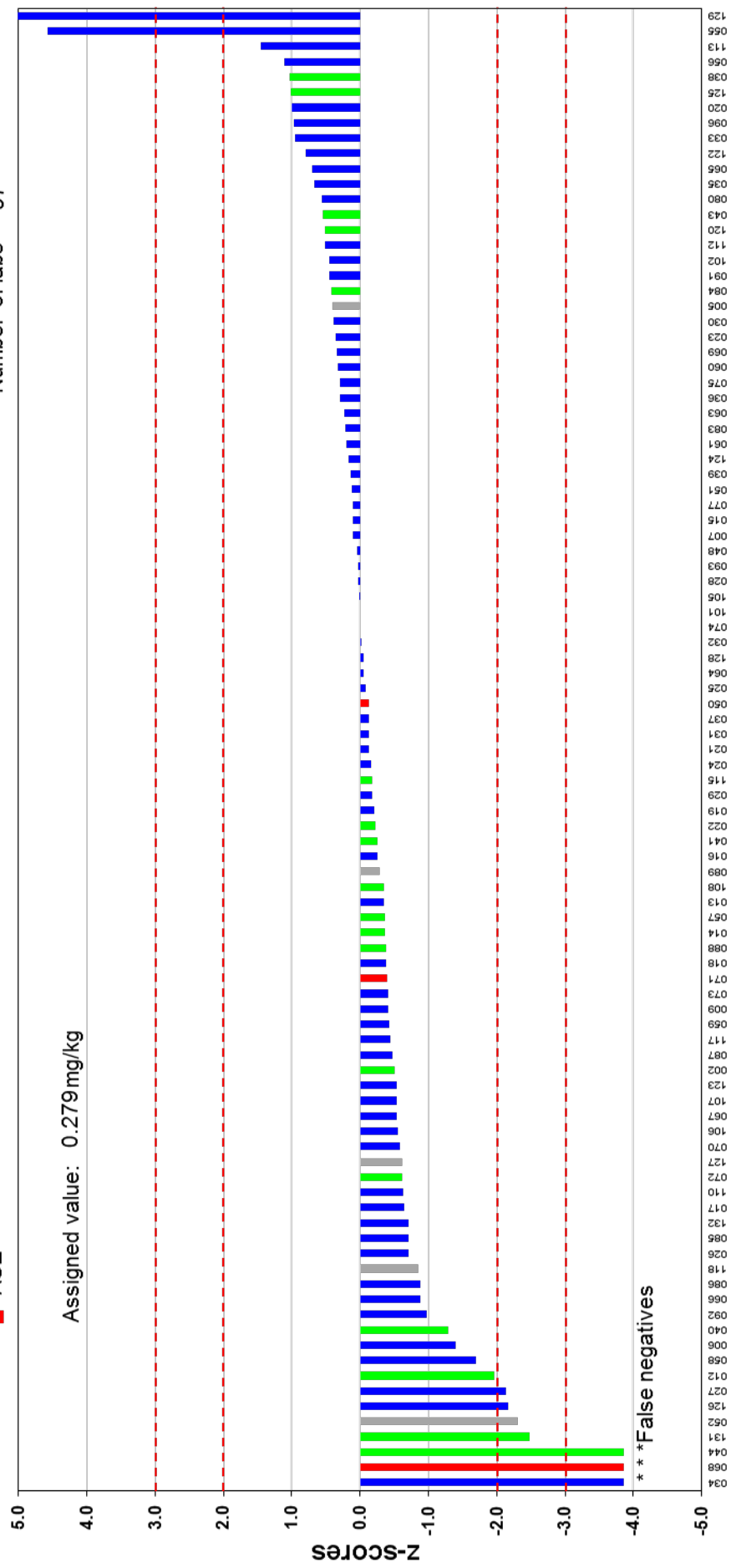
Water added  
 No water added  
 Not specified  
 ASE

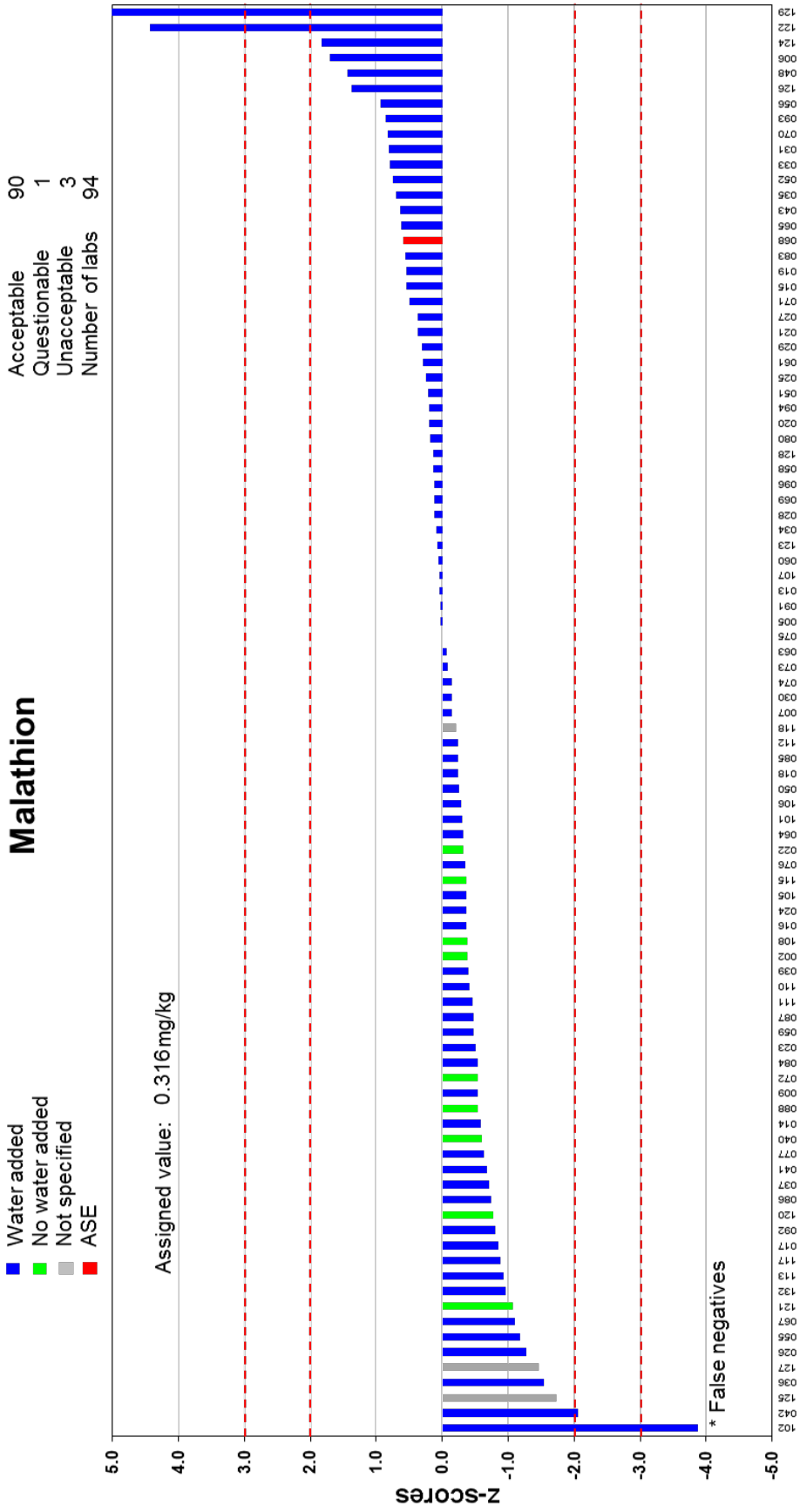


# Lindane

Acceptable 88  
 Questionable 4  
 Unacceptable 5  
 Number of labs 97

Water added  
 No water added  
 Not specified  
 ASE

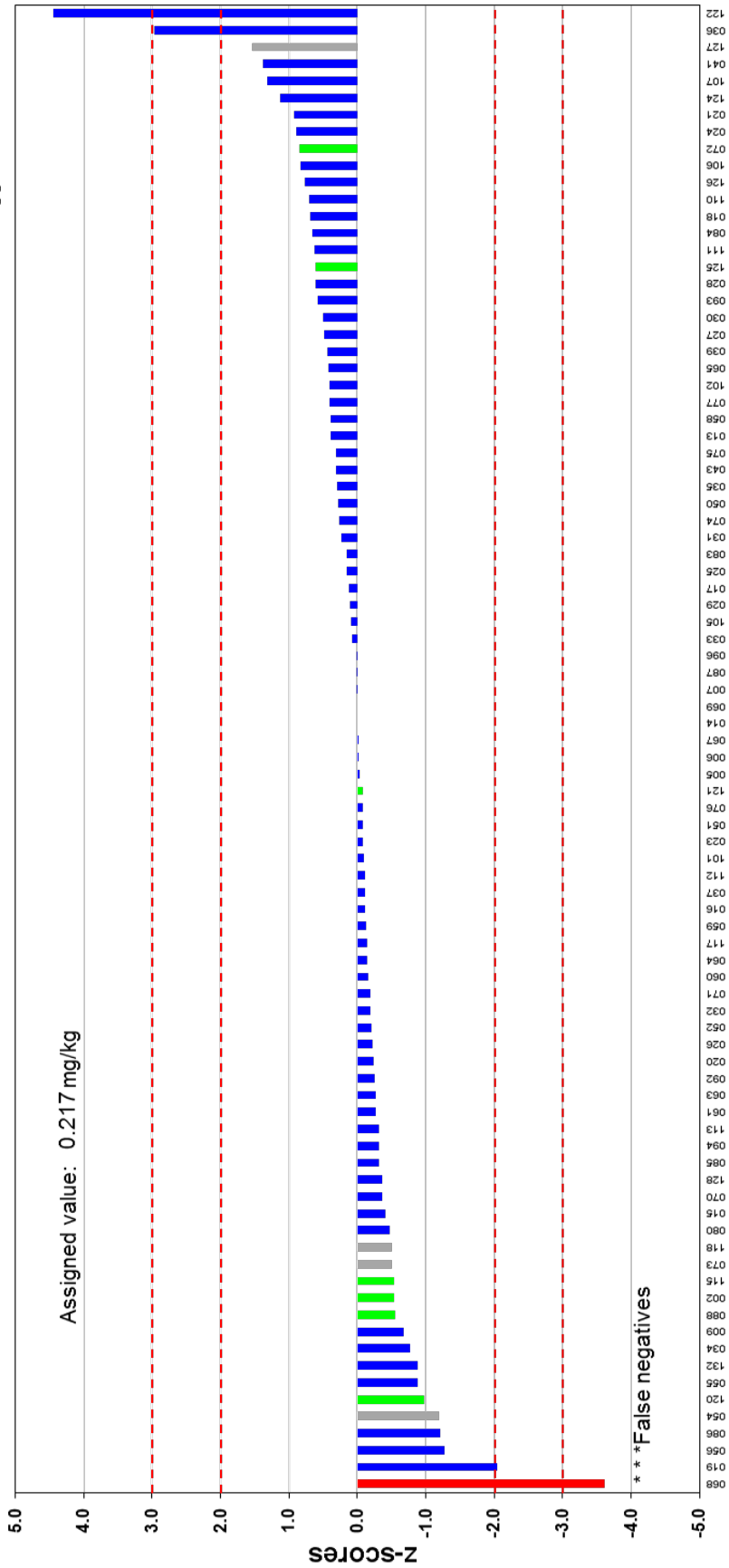




# Propiconazole

- Water added
- No water added
- Not specified
- ASE

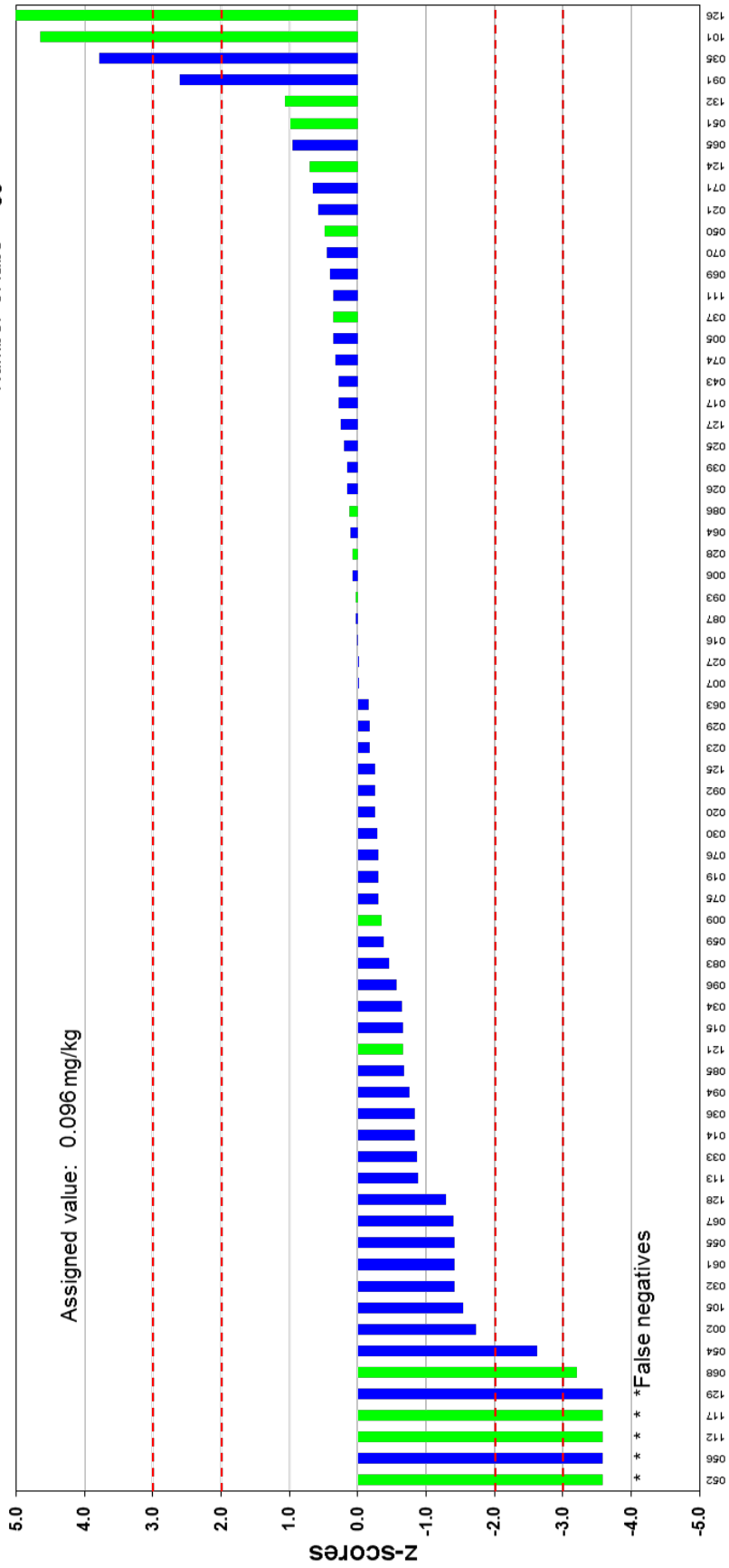
Acceptable 85  
 Questionable 1  
 Unacceptable 2  
 Number of labs 88



# Pyraclostrobin

■ LC  
■ GC

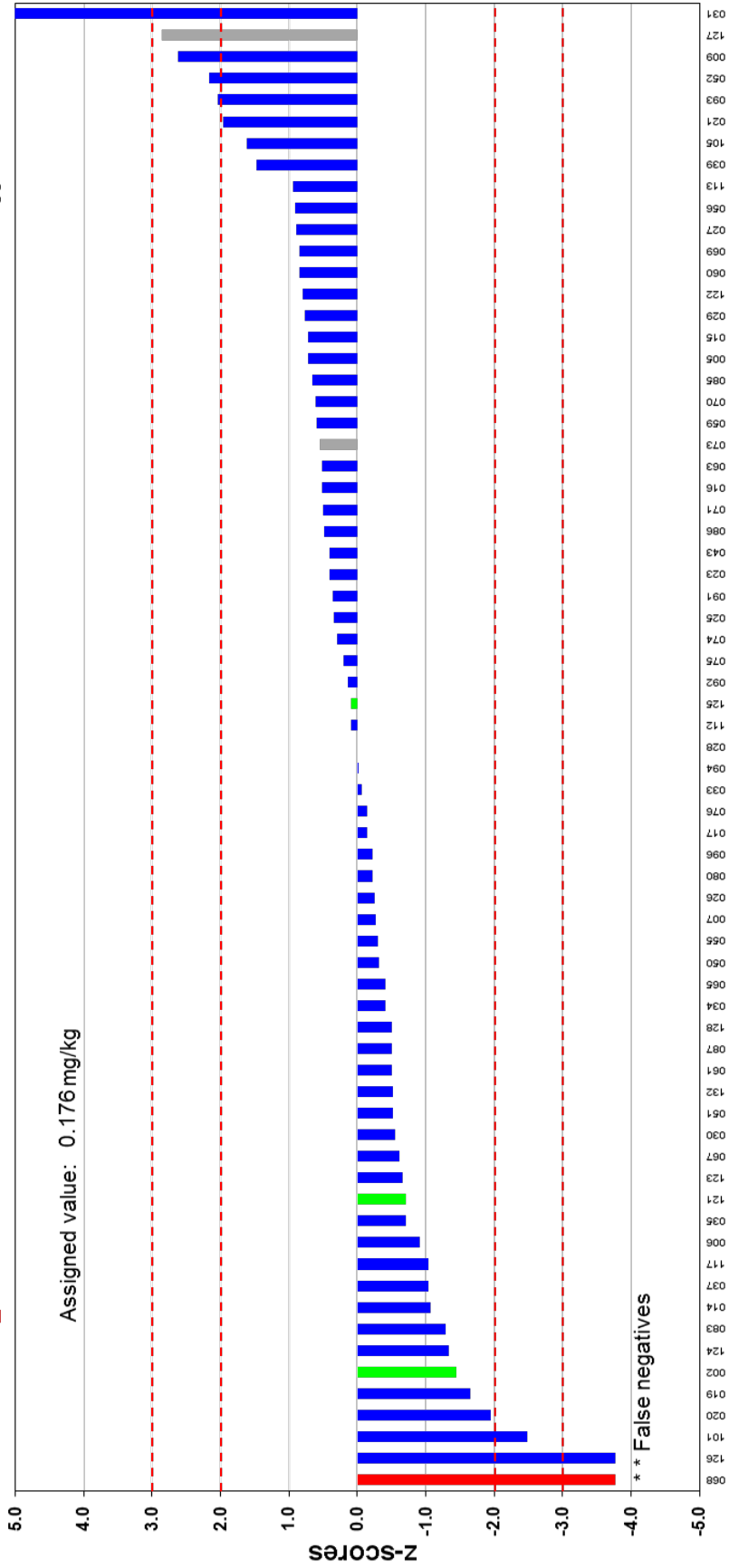
Acceptable 58  
 Questionable 2  
 Unacceptable 9  
 Number of labs 69



# Spiroxamine

- Water added
- No water added
- Not specified
- ASE

Acceptable 62  
 Questionable 4  
 Unacceptable 3  
 Number of labs 69

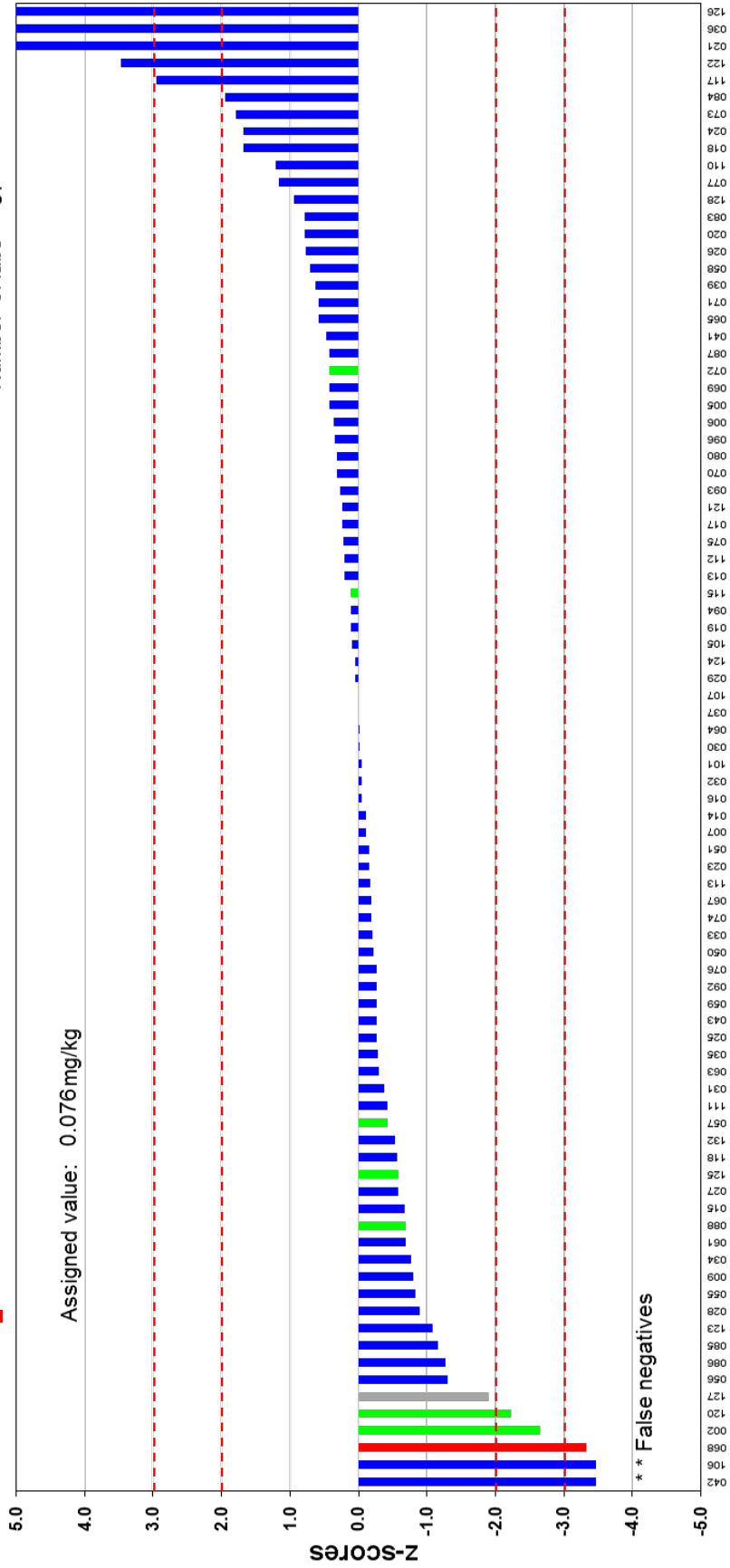




# Tebuconazole

- Water added
- No water added
- Not specified
- ASE

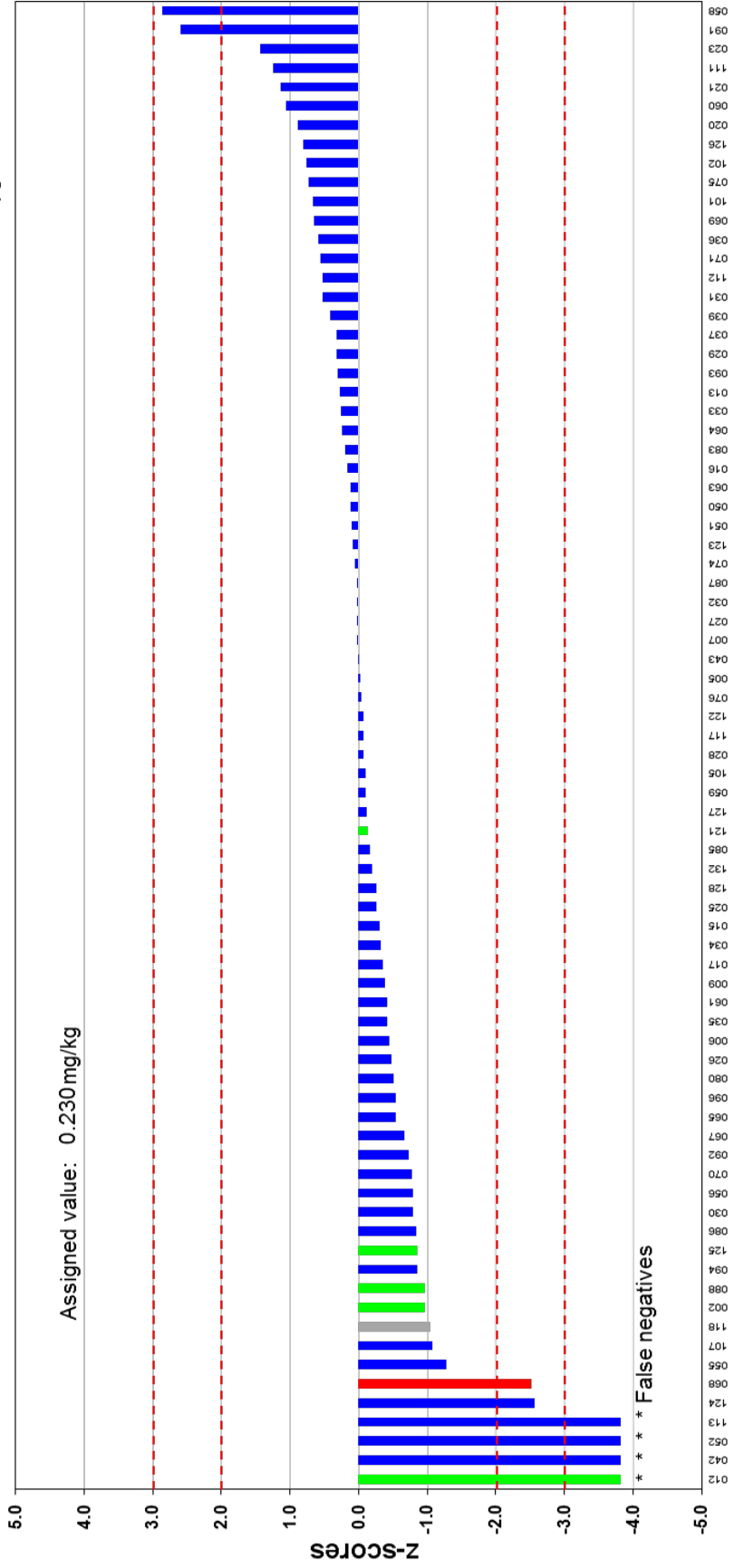
Acceptable 77  
 Questionable 3  
 Unacceptable 7  
 Number of labs 87



# Triadimenol

Acceptable 70  
 Questionable 4  
 Unacceptable 4  
 Number of labs 78

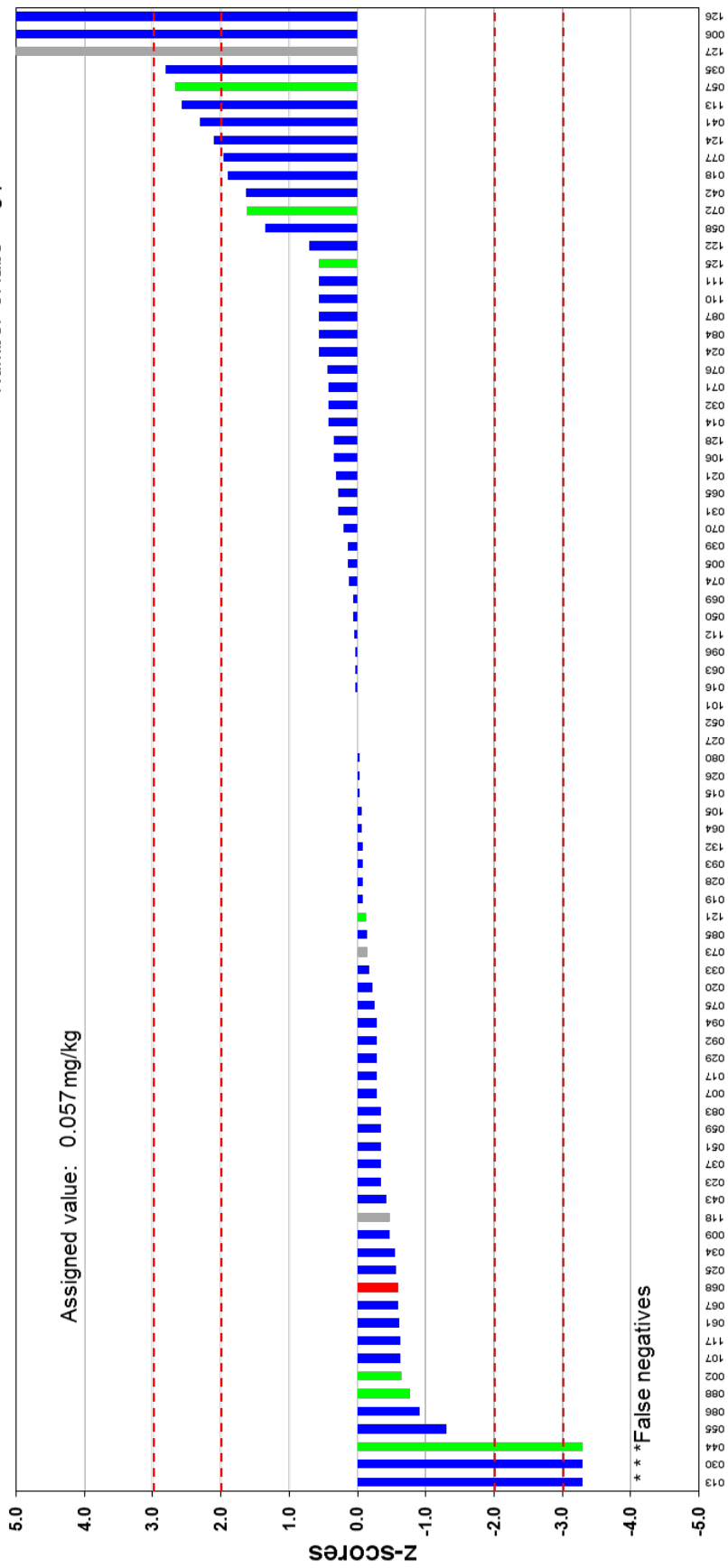
Water added  
 No water added  
 Not specified  
 ASE



# Trifloxystrobin

- Water added
- No water added
- Not specified
- ASE

- Acceptable 73
- Questionable 5
- Unacceptable 6
- Number of labs 84





## Appendix 6

Results for bifenthrin, deltamethrin, diazinon, difenoconazole, lambda-cyhalothrin, pirimicarb, pirimiphos-methyl and prothioconazol-desthio in mg/kg, the pseudo z-scores, the MRRLs and the mean values of all reported results.

Laboratory code	Bifenthrin	pseudo Z-scores	Deltamethrin	pseudo Z-scores	Diazinon	pseudo Z-scores	Difenoconazole	pseudo Z-scores	Lambda-cyhalothrin	pseudo Z-scores	Pirimicarb	pseudo Z-scores	Pirimiphos-methyl	pseudo Z-scores	Prothioconazole-desthio	pseudo Z-scores
MRRL	0.01		0.01		0.01		0.01		0.01		0.01		0.01		0.01	
Median value	0.013		0.012		0.009		0.007		0.012		0.006		0.012		0.015	
1	0.014	0.3					0.009	1.1			0.009	2.5	0.013	0.3		
2	0.0211	2.4			0.0105	0.7							0.014	0.7		
3									0.024	4.2						
5	0.014	0.3	0.011	-0.5					0.011	-0.2					0.016	0.3
6	0.2	>5							0.094	>5						
7	0.0108	-0.7											0.013	0.3	0.0128	-0.5
9															0.013	-0.5
11																
12																
13									0.011	-0.2			0.011	-0.3		
14	0.015	0.6														
15			0.0124	0.0									0.012	0.0	0.0118	-0.8
16	0.0125	-0.2	0.0129	0.2	0.008	-0.4			0.0101	-0.5					0.0143	-0.1
17	0.0101	-0.9	0.0119	-0.2									0.012	0.0	0.0165	0.5
18													0.012	0.0		
19															0.012	-0.7
20													0.014	0.7		
21	0.014	0.3											0.01	-0.7	0.0174	0.7
22	0.0168	1.1							0.0111	-0.2						
23	0.014	0.3	0.012	-0.1	0.006	-1.3			0.01	-0.6	0.006	0.4	0.0107	-0.4	0.017	0.6
24													0.0102	-0.6		
25	0.011	-0.7													0.014	-0.2
26													0.013	0.3		
27					0.009	0.0	0.007	0.0			0.006	0.4			0.016	0.3
28	0.015	0.6							0.013	0.4					0.015	0.1
29	0.013	0.0											0.01	-0.7		
30	0.01	-3.9			0.012	1.3	0.0011	-3.4							0.0108	-1.1
31	0.012	-0.3			0.01	0.4			0.011	-0.2					0.014	-0.2
32													0.0185	2.2		
33	0.0165	1.0											0.011	-0.3	0.0148	0.0
34	0.0124	-0.2	0.0132	0.3					0.0129	0.4					0.0119	-0.8
35	0.0187	1.7	0.0101	-0.7					0.0123	0.2					0.0257	3.0
36	0.033	>5														
37																
38	0.0127	-0.1	0.0119	-0.2									0.0155	1.2		
39	0.014	0.3							0.011	-0.2						
40	0.018	1.5			0.009	0.0			0.014	0.8						
41													0.013	0.3		
42					0.017	3.6							0.012	0.0		

Laboratory code	Bifenthrin	pseudo Z-scores	Deltamethrin	pseudo Z-scores	Diazinon	pseudo Z-scores	Difenoconazole	pseudo Z-scores	Lambda-cyhalothrin	pseudo Z-scores	Pirimicarb	pseudo Z-scores	Pirimiphos-methyl	pseudo Z-scores	Prothioconazole-desthio	pseudo Z-scores
MRRL	0.01		0.01		0.01		0.01		0.01		0.01		0.01		0.01	
Median value	0.013		0.012		0.009		0.007		0.012		0.006		0.012		0.015	
43	0.014	0.3			0.007	-0.9	0.008	0.6					0.013	0.3		
44																
45																
47													0.001	-3.7		
48	0.0121	-0.3	0.0185	2.0	0.01	0.4			0.0138	0.7						
49													0.0858	>5		
50	0.012	-0.3											0.009	-1.0	0.015	0.1
51																
52	0.016	0.9											0.0096	-0.8		
54													0.026	4.7		
55	0.0207	2.3							0.0097	-0.7			0.026	4.7		
56	0.0096	-1.1			0.009	0.0			0.0081	-1.2						
57													0.0123	0.1		
58	0.0171	1.2							0.0203	2.9			0.012	0.0		
59	0.012	-0.3			0.006	-1.3			0.008	-1.3						
60													0.0101	-0.6		
61	0.0124	-0.2	0.0168	1.4	0.007	-0.9	0.0056	-0.8	0.0166	1.7	0.0047	-0.6	0.0047	-2.4	0.0121	-0.7
62	0.025	3.6			0.015	2.7										
63													0.013	0.3		
64	0.0139	0.2	0.013	0.2					0.0114	-0.1			0.013	0.3	0.0143	-0.1
65	0.012	-0.3	0.011	-0.5	0.021	>5			0.011	-0.2					0.019	1.2
66																
67	0.0134	0.1			0.0051	-1.7	0.0053	-1.0	0.01	-0.6	0.005	-0.4				
68	0.0063	-2.1			0.0053	-1.6	0.001	-3.4	0.0025	-3.1			0.0105	-0.5		
69	0.0102	-0.9														
70	0.012	-0.3	0.015	0.8					0.012	0.1					0.016	0.3
71	0.01	-1.0											0.0089	-1.0	0.018	0.9
72													0.0089	-1.0		
73			0.109	>5												
74													0.01	-0.7		
75													0.01	-0.7	0.0121	-0.7
76													0.012	0.0		
77																
79					0.0053	-1.6							0.011	-0.3		
80																
82	0.0085	-1.4			0.0064	-1.2	0.0071	0.1	0.0085	-1.1	0.0044	-0.8				
83	0.0137	0.2			0.0091	0.0	0.0096	1.5							0.0155	0.2
84																
85	0.01	-1.0	0.011	-0.5					0.012	0.1					0.015	0.1
86	0.011	-0.7													0.01	-1.3
87													0.013	0.3		

Laboratory code	Bifenthrin	pseudo Z-scores	Deltamethrin	pseudo Z-scores	Diazinon	pseudo Z-scores	Difenoconazole	pseudo Z-scores	Lambda-cyhalothrin	pseudo Z-scores	Pirimicarb	pseudo Z-scores	Pirimiphos-methyl	pseudo Z-scores	Prothioconazole-desthio	pseudo Z-scores
MRRL	0.01		0.01		0.01		0.01		0.01		0.01		0.01		0.01	
Median value	0.013		0.012		0.009		0.007		0.012		0.006		0.012		0.015	
88																
89													0.012	0.0		
90											0.003	-1.8	0.012	0.0		
91	0.014	0.3							0.013	0.4						
92	0.012	-0.3														
93					0.01	0.4							0.0104	-0.5	0.019	1.2
94	0.011	-0.7	0.012	-0.1	0.008	-0.4	0.006	-0.6								
95																
96	0.0105	-0.8			0.0082	-0.4	0.0071	0.1	0.01	-0.6	0.0057	0.1	0.03	>5	0.0135	-0.3
98													0.03	>5		
99													0.011	-0.3		
100																
101	0.014	0.3	0.013	0.2												
102																
103																
105																
106																
107																
108	0.0134	0.1							0.0118	0.0						
109																
110																
111													0.012	0.0		
112	0.0133	0.0											0.0134	0.5		
113	0.058	>5			0.0106	0.7	0.007	0.0			0.0055	0.0			0.0208	1.6
115													0.013	0.3		
117	0.01	-1.0														
118													0.0137	0.6		
120	0.0122	-0.3	0.0209	2.7					0.0168	1.7			0.011	-0.3		
121	0.0105	-0.8											0.015	1.0	0.0147	0.0
122	0.016	0.9														
123													0.014	0.7	0.011	-1.0
124													0.01	-0.7		
125	0.02	2.1	0.03	>5	0.005	-1.8	0.005	-1.1	0.02	2.8	0.005	-0.4				
126	0.195	>5											0.013	0.3		
127	0.022	2.7			0.02	4.9										
128																
129																
130	0.027	4.2														
131																
132	0.013	0.0	0.012	-0.1					0.014	0.8					0.012	-0.7





## 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 (OLs) within the EU Member States. Laboratories outside of this EURL/NRL/OL-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 OLs are laboratories that are actively involved in official controls following Article 28 of Regulation 396/2004/EC (e.g. by conducting pesticide residue analyses within the framework of national and/or EU-controlled programmes).

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

<sup>2</sup> For more information about the EURL/NRL/OL-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

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

<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.

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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.

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### 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

will 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.



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– **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)**

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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:

$$\delta = b_1 \cdot \mu \quad \text{with } b_1 = 0.25 \text{ (25\% FFP-RSD)}$$

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:

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

Where:  $x_i$  is the value reported by the laboratory,  $\mu$  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.

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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	
7	6.3	6	N - 1
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	N - 2
15	13.5	14	
16	14.4	15	
17	15.3	16	
18	16.2	17	
19	17.1	18	
20	18.0	19	
21	18.9	20	
22	19.8	21	
23	20.7	22	
24	21.6	22	N - 3
25	22.5	22	
26	23.4	23	

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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:

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

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.

#### 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

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/Feeding stuff using Multi-Residue Methods, EUPT-CF7 (2013) (last updated: 1 May 2013)

### 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 feeding stuff, as far as their scope overlaps with that of the EUPT-CF7.

### Test Item (Test Material)

This proficiency test concerns the analysis of pesticide residues in feed for laying hens. The feed has been produced by IFF-Braunschweig<sup>1</sup> of raw ingredients provided by EURL-CF. Some of the cereals used have been grown in Denmark and contain incurred pesticides. Furthermore, some of the ingredients were spiked with additional pesticides before all ingredients were mixed into the feeds. Ingredients in the two Test Items, marked "sample" and "blank" are listed in Table 1 below.

Table 1. Test item composition:

Ingredients	Test item with pesticides (sample)	Test item (blank sample)
Cereals (including maize)	62%	62%
Soya meal	25%	
Soya cake		27%
Soya oil	3%	1%
Lime	9%	9%
Mineral and vitamin premix	1%	1%

The blank Test Item provided, can be used for recovery experiments as well as for the preparation of matrix-matched calibration standards. However, the blank Test Item must also be analysed and

<sup>1</sup> Forschungsinstitut Futtermitteltechnik der IFF, Research Institute of Feed Technology, Frickentmühle 1A, 38110 Braunschweig-Thune, Germany

detected pesticides reported. It should be noted that the composition of the blank Test Item is slightly different from the Sample Test Item, because soya cake has been used instead of soya meal. It is not possible to obtain organically produced soya meal. To equalize the fat content to the same level as in the sample, less soya oil has been added. Consequently, the fat content in both Test Items are the same.

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 also be checked to prove that the target analytes are not contained at any relevant levels. All these tests will be conducted by the EURL-CF 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 have been set.

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

### Amount of Test Item

The participants will receive:

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

### Shipment of Test Items

The Test Items are planned to be shipped on 13 May, 2013.

Test Items will be shipped frozen and packed in thermo-boxes together with a freezer block. 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°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 portions.



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 Items, 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 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-CF7 Result Submission Website](http://hor.divf.dk/EUPT-CF7) (<http://hor.divf.dk/EUPT-CF7>).

Subpage 0 (Test Item recipe) will be accessible from 14 May 2013 and subpage 1-3 from 20 May 2013 and onwards. The webpage contains a link to specific instructions on how to enter the data in the result submission website.

To access the data submission forms, participants must use their unique login data (username and password) 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 7 June 2013**

### Test Item Receipt and Acceptance - Subpage 0

Once the laboratory has received the Test Items it must report to the organiser, via the [EUPT-CF7 Result Submission Website](#) the date of receipt, the condition of the Test Item, and its acceptance. The deadline for acceptance is the 17 May 2013. If the laboratory does not respond by this deadline, the Organisers will assume that the Test Items have been received and accepted.

**If participants have not received the Test Items by the 16 May 2013 at noon, they must inform the Organiser immediately by e-mail ([eu-ri-ctf@food.dtu.dk](mailto:eu-ri-ctf@food.dtu.dk)).**

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

To report their results, laboratories must access the [EUPT-CF7 Result Submission Website](#).

**Deadline:** All results must be reported on the online result submission website by 7 June 2013. The website will not be accessible for result submission after this date and time, and any results reported after the deadline will not be included in the statistical treatment, or in the final report.

**The results should be reported in mg/kg test item with no re-calculation regarding water or fat content.**

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 by two significant figures (e.g. 0.0058 mg/kg).

Residue levels ≥ 0.010 mg/kg;

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

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

- **“Concentration in mg/kg”**: here you should fill in the results 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 what kind of recovery-correction via the dropdown-menu.
- **“Recovery figure (in %)”**: Here, labs can report any recovery figures (in %) obtained for the analyze in question. If a recovery factor was used to correct the result, 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-CF7 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-CF7 Result Submission Website](#). This subpage will be accessible from 11-16 June 2013.

**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-CF7 can be found in the EURL-Document Repository ([CIRCA/FIS-VL](#)). Links to the documents can be found in the [EUPT-CF7 Website](#).

Calendar (see also [http://www.eurl-pesticides.eu/library/docs/cf/EUPT\\_CF7\\_Calendar130304.pdf](http://www.eurl-pesticides.eu/library/docs/cf/EUPT_CF7_Calendar130304.pdf))

Activity	Dates
Announcement Calendar Target Pesticide List	January 2013
EUPT-Registration Website	18 March 2013
Deadline for registration	15 April 2013
Release of Specific Protocol	08 April 2013
Distribution of Test items	13 May 2013
Deadline for Receipt and Acceptance of Test Materials	within 24 hr on receipt
Deadline for Result Submission	7 June 2013
EUPT Evaluation Meeting	Ultimo June 2013
Preliminary Report (only compilation of results)	July 2013
Final Report	December 2013

#### Participation Fees

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

- 175 €
- 350 €

The participation fees for laboratories from third countries:

- 350 €

For further information, visit this 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 questions concerning invoices must be directed to Elena Soerensen at the financial department [elso@adm.dtu.dk](mailto:elso@adm.dtu.dk)

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**European Union Reference Laboratory  
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