Commission Reference Laboratory on Cereals & Feedingstuff and Single Residue Methods

Report on Proficiency Test on incurred and spiked pesticides in oat

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Final report
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Pesticide Residues in Cereals using Multi- and Single Residue Methods

Final Report

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PREFACE

Regulation 882/2004/EC [1], lays down the general tasks and duties of Community Reference Laboratories (CRLs) for Food, Feed and Animal 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 frame of the EU co-ordinated control and national monitoring programmes. At the same time laboratories are provided with an assessment, both of their analytical performance and the reliability of their data - by comparison with the other participating laboratories. This will hopefully result in improvements in the analytical quality of the residue data and increase the scope of the methods used by laboratories.

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 [1], all laboratories analysing samples for the official controls of pesticide residues shall participate in the European Union Proficiency Tests (EUPTs) for pesticide residues facilitated by the Commission as long as the scope of the PT and the laboratory overlap.

The present EUPT was the third one to be organized within the frame of the CRL activities with cereal-matrces as test materials. The first (EUPT-C1/SRM2) was conducted in 2007 using wheat flour containing incurred and laboratory-spiked pesticides. This test was, as was the present one, co-organised by the CRL for Cereals and Feedingstuff (CRL-CF) and the CRL for Single Residue Methods (CRL-SRM), and the pesticides in the test material required the application of both, multi residue methods (MRMs) and single residue methods (MRMs). The second PT (EUPT-C2) was performed in 2008 by the CRL-CF using a wheat flour test material containing mostly MRM-pesticides as well as two SRM pesticides (chlormequat and glyphosate). Another two EUPTs focusing on single residue methods were jointly organised by the CRL-SRM and CRL-FV in 2006 and 2008 using apple juice (EUPT-SRM1) and carrots (EUPT-SRM3) as the test materials. The present EUPT-C3/SRM4 was jointly organised by the CRL-CF and CRL-SRM using oat flour previously treated with 20 compounds partly in the field and partly post-harvest in the laboratory.

Participation in EUPT-C3/SRM4 was open to all National Reference Laboratories (NRLs) and Official Laboratories (OfLs) involved in the determination of pesticide residues in cereals or feeding stuff using multi- or single residue methods within the EU. Additionally, laboratories from the EFTA countries (Iceland, Norway and Switzerland) were invited to take part in the PT as they also contribute data to the EU-coordinated community control programme. NRLs and OfLs that did not take part in this test were asked to explain the reasons for their non-participation. Laboratories that registered to participate in this exercise but finally did not take part, either in the SRM or the MRM part, or both, were also asked to provide explanations.

This report will be presented to the European Commission Standing Committee for Animal Health and the Food Chain. Furthermore, DG-SANCO has full access to all data of EUPTs, including the lab-code/lab-name key.

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INTRODUCTION

On 31 October 2008, all relevant National Reference Laboratories (NRLs) of the 27 EU-Member States (MS) as well as all relevant EU-Official Laboratories (OfLs), as far as their contact data were communicated by the NRLs, were sent an invitation to participate in this 3rd European Commission's Proficiency Test on Cereals using multi- and single residue methods (EUPT-C3/SRM4). To make sure that all relevant official laboratories become aware of the PT, the NRLs were asked to additionally forward the invitation to the relevant laboratories within their countries. Also included in the invitation was a Specific Protocol, a Calendar, as well as a Target Pesticides List including compounds that could potentially be present in the test material (see **Appendix 2** and **Appendix 8**). The Target Pesticides List included 60 compounds (pesticides, metabolites etc.) requiring the use of multiresidue methods (MRMs) and an additional 13 compounds requiring single residue methods (SRMs), along with minimum required reporting level (MRRL) stipulated for each compound. For 8 acidic pesticides included in the SRM list the laboratories had the opportunity to report two results: one derived following alkaline hydrolysis to release bound residues, and a second without the inclusion of this step. A "General Protocol" (see **Annex 1**) containing information that is common to all EUPTs- was also distributed to the laboratories. The laboratories were able to register online by 14 January. In total 111 laboratories from 27 countries agreed to participate. An additional 31 laboratories provided explanations for their non-participation.

The present proficiency test was performed using oat flour of Danish origin, which had been partly treated in the field, and partly spiked post-harvest at the facilities of the CRL-CF. The test material contained 20 compounds in total. The Faculty of Agricultural Sciences, University of Aarhus performed the field treatments. The pesticides employed for field treatment were selected by the CRL-CF and the CRL-SRM with the application rates and harvest intervals chosen based on previous experience and data from supervised residue trials. The harvested grain was treated with two pesticides post-harvest, and then checked for homogeneity before shipping to participants. Furthermore, the stability of the pesticide in the test material was checked several times during the period of time allowed for laboratories to undertake the PT exercise. The participating laboratories were provided with 150 g portions of treated whole oat flour test material and 150 g of blank whole oat flour. The test materials were shipped to the participants on 2 February 2009 and the deadline for submission of results to the Organiser was the 3rd of March 2009. The participants were asked to analyse the treated test material as well as the 'blank' material and report the concentrations of any pesticide residues found which were included in the Target Pesticide list (see **Appendix 2** and **Appendix 8**). Additionally, the 'blank' material could be used for recovery experiments for the pesticides found in the test material, and if necessary, for the preparation of matrix-matched calibration standards. Submission of results was performed online via a website. Eight laboratories did not submit any results.

The medians of the analytical data submitted were used to obtain the assigned values for each of the pesticide residues present. A fit-for-purpose target relative standard deviation (FFP RSD) of 25 % was chosen to calculate the target standard deviations (σ) as well as the z-scores for each of the compounds present. For informative purposes only, the robust standard deviations (Qn-RSD) were also calculated.

1. TEST MATERIALS

1.1 Analytical methods

The following analytical methods, described briefly below, were used by the organisers to test the homogeneity and stability of the test material:

- For **MRM pesticides**: QuEChERS method and determination on GC-MS/MS and LC-MS/MS. For more details see www.crl-pesticides.eu
- For **chlormequat, glyphosate and ethephon**: Extraction following addition of water and methanol containing 1 % formic acid. Centrifugation, filtration and direct analysis by LC-MS/MS of glyphosate and ethephon ESI-neg. mode together and chlormequat separately ESI-pos. mode. For more details see www.crl-pesticides.eu
- For the acidic pesticides (**2,4-D, dicamba**): QuEChERS-method involving water addition and extraction with acetonitrile followed by phase partitioning induced by addition of salts, and a direct determination by LC-MS/MS in the ESI-neg. mode. Where required alkaline hydrolysis was performed directly after the water addition step. For this sodium hydroxide solution was added and th mixture was allowed to react for 30 minutes at room temperature before neutralization with sulfuric acid. The procedure was then continued as described above. For more details see www.crl-pesticides.eu

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 Scientific Committee taking into account the present and upcoming scope of the EU-coordinated monitoring programme, a pesticide priority list ranking the pesticides according to their relevance and risk-potential, as well as a list of pesticides relevant to the specific commodity (oats). The overall capacity and capability of the laboratories within the EU, as assessed from previous PTs and surveys, was also taken into account. In some cases the residue definitions valid for the test were slightly different from those in the legislation to account for analytical difficulties (e.g. in the case of prochloraz and bentazone). In the case of the 8 acidic pesticides the laboratories were given the opportunity to report two results: one derived following alkaline hydrolysis to release bound residues and one without this hydrolysis step (free acid). The minimum required reporting levels (MRRLs) were set at 0.02 mg/kg for the SRM-compounds and at 0.01-0.02 mg/kg, depending on the pesticide, for the MRM-compounds.

1.3 Preparation of the treated test material

Before preparing the test material, the pesticides and suitable target residue levels for the study were selected. The application rates and harvest intervals for the 20 pesticides from the target pesticide list were chosen based on data from supervised residue trials. The field spraying was performed by the Faculty of Agricultural Sciences, University of Aarhus. One hundred kilograms of oats were delivered for preparation of the test material. Following a preliminary analysis of the material it was decided to additionally spike in the laboratory with 2,4-D, which could not be detected in the harvested material in a preliminary test, as well as with ethephon which, was not included in the field treatments, see **Table 1**. Spiking in the laboratory was performed using pesticide formulations. One kilogram of oats was spiked with one of the pesticide

formulations, and a second kilogram was spiked with the other pesticide formulation. The resulting 2 kilograms were mixed with the remaining 58 kg and homogenised thoroughly. The 60 kg mixed oats where milled as four kilograms portions. The portions were stirred thoroughly individually and additionally following two by two additions to ensure that a well-homogenised bulk with respect to both incurred and spiked pesticide residues was attained. 150 g 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 material

The oat flour used for blank test material was produced by the Faculty of Agricultural Sciences, University of Aarhus under similar growing conditions as the treated crop but without any pesticide treatment in the field or spiking in the laboratory. 150 g portions were weighed out into screw-capped polyethylene plastic bottles, sealed, and stored in a freezer at about -20 °C prior to distribution to participants.

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

Pesticide	Application in the field	Spiked in laboratory	Formulation	Company
MRM-Compounds				
Azoxystrobin	X		Amistar	Agros
Carbendazim	X		Bavistin DF	BASF
Chlorpyrifos	X		Dursban WG	Dow
Cyproconazole	X		Alto	Syngenta
Cyprodinil	X		Unix	Syngenta
Esfenvalerate	X		Sumi Alpha	Sumitomo
Fenbuconazole	X		Indar	Dow
Fenpropimorph	X		Corbel	BASF
Fludioxonil	X		Safir	Syngenta
Flusilazole	x		Lyric	DuPont
Lambda-cyhalothrin	x		Karate 2,5 WG	Syngenta
Malathion	x		Maladan 44 EW	Nordisk Alika
Metconazole	x		Juventus	BASF
Procymidone	x		Sumislex	Sumitomo
Pyraclostrobin	x		Comet	BASF
Tebuconazole	x		Folicur	Bayer CropScience
SRM-Compounds				
2,4-D	x	x	Herboxone	AH Marks
Chlormequat	x		Cycocel 750	BASF
Dicamba	x		Inter-Dicamba 480	Inter-Trade
Ethephon		x		
Glyphosate	x		Roundup	Mosanto Crop

1.5 Homogeneity test

Ten bottles of treated test material were randomly chosen and analyses were performed on duplicate portions taken from each bottle. The sequence of analyses was determined using a table of randomly generated numbers. The injection sequence of the 20 extracts was also randomly chosen. The 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 [3,4]. An overview of the statistical analyses of the homogeneity test is shown in **Table 2** and **Table 3** for MRM- and SRM-pesticides, respectively.

The individual residue data from the homogeneity tests, as well as the results of the statistical analyses, are given in **Appendix 3** and **Appendix 9** for MRM- and SRM-pesticides, respectively.

The acceptance criterion for the test material to be sufficiently homogenous for the proficiency test was that $Ss/\sigma < 0.3$, with Ss being the standard deviation of the pesticide concentrations determined for the 10 bottles and σ is the target fit-for-purpose relative standard deviation (25 %) multiplied by the mean concentration of each pesticide.

As all pesticides passed the homogeneity test, the test material was considered to be sufficiently homogenous and suitable for the PT-C3/SRM4.

Table 2 Statistical evaluation of the homogeneity test data of MRM-analytes (n=20 analyses using a sample portion of 5 g in each case).

	Azoxystrobin	Carbendazim	Chlorpyrifos	Cyproconazole	Cyprodinill	Fenbuconazole	Fenpropimorph
Mean [mg/kg]	0.241	0.479	1.269	0.518	0.084	0.563	0.151
S_s/σ	0.010	0.024	0.023	0.008	0.004	0.017	0.004
Pass/Fail	Pass	Pass	Pass	Pass	Pass	Pass	Pass

	Fenvalerate (sum)	Fludioxonil	Flusilazole	Lambda-cyhalothrin	Malathion	Metconazole	Pyraclostrobin	Tebuconazole
Mean [mg/kg]	0.176	0,156	0.908	0.067	0.019	0.478	0.798	1.516
S_s/σ	0.003	0.004	0.029	0.002	0.001	0.016	0.053	0.036
Pass/Fail	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass

S_s: Between Sampling Standard Deviation

Table 3 Statistical evaluation of the homogeneity test data SRM-analytes (n=20 analyses, using a sample portion of 5 g in each case).

	2,4-D (foll. alk. hydr.)	Dicamba (foll. akl. Hydr.)	Chlormequat	Ethephone	Glyphosphate
Mean (mg/kg)	0.491	0.273	0.517	0.549	1.218
Ss/σ	0.140	0.179	0.207	0.143	0.110
Pass/Fail	Pass	Pass	Pass	Pass	Pass

Ss: Between Sampling Standard Deviation

1.6 Stability tests

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

In the case of SRM compounds storage was done only at -18 °C. In the case of MRM compounds two different storage conditions were used; room temperature and -18 °C. In all cases the analyses were performed on 5 randomly chosen samples.

The dates of testing were as follows:

For MRM-compounds:
Day 1: 3 February 2009 (shortly after the first shipment)
Day 2: 9 February 2009
Day 3: 16 February 2009
Day 4: 23 February 2009
Day 5: 3 March 2009

For SRM-compounds:
Day 1: 10 January 2009 (shortly before first shipment)
Day 2: 5 February 2009.
Day 3 10 March 2009

The average results from each stability test for the MRM pesticides are given in **Table 4**. A graphic presentation of the stability test results, including the standard deviaton, are shown in **Appendix 4**. All MRM compounds showed an acceptable stability at both storage conditions (room temperature and freezer) except for cyprodinil and fludioxinil that showed a significant decrease at room temperature over the four weeks storage period. It could not be verified, however, whether this trend was due to a progressive change in the matrix that notably deteriorated the chromatography for these two compounds, or if there was a real degradation. The later hypothesis is supported by the fact that these two compounds belong to

the same chemical class. In any case, the laboratories were asked to store the samples in the freezer until analysis is performed and under these conditions all compounds showed a good stability over a period that was longer than the duration of the Proficiency Test.

Table 4 Stability test results of MRM-pesticides at room temperature and -18 ºC (see also **Appendix 4**)

	Azoxystrobin	Carbendazim	Chlorpyrifos	Cyproconazole	Cyprodinill	Fenbuconazole	Fenpropimorph
Day 1	0.170	0.980	1.10	0.496	0.080	0.525	0.138
Storage at Room temperature (mean values in mg/kg)							
Day 2	0.197	1.40	1.25	0.517	0.084	0.569	0.141
Day 3	0.188	1.55	1.13	0.480	0.075	0.551	0.128
Day 4	0.208	1.47	1.19	0.498	0.067	0.591	0.144
Day 5	0.182		1.07	0.465	0.061	0.557	0.116
	passed	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**
Storage at -18 ºC (mean values in mg/kg)							
Day 2	0.206	1.53	1.17	0.523	0.087	0.559	0.140
Day 3	0.181	1.47	1.07	0.517	0.076	0.531	0.130
Day 4	0.184	1.60	1.16	0.541	0.088	0.542	0.143
Day 5	0.191	0.980	1.26	0.557	0.088	0.548	0.143
	passed	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**

	Fenvalerate (sum)	Fludioxonil	Flusilazole	Lambda-cyhalothrin	Malathion	Metconazole	Tebuconazole
Day 1	0.091	0.133	0.830	0.058	0.011	0.416	1.43
Storage at Room temperature (mean values in mg/kg)							
Day 2	0.099	0.145	0.924	0.066	0.012	0.446	1.46
Day 3	0.094	0.118	0.756	0.062	0.012	0.407	1.40
Day 4	0.092	0.085	0.811	0.068	0.012	0.443	1.55
Day 5	0.084	0.078	0.717	0.063	0.009	0.408	1.47
	passed	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**
Storage at -18 ºC (mean values in mg/kg)							
Day 2 (mean)	0.097	0.133	0.893	0.056	0.011	0.446	1.53
Day 3 (mean)	0.090	0.139	0.807	0.058	0.012	0.438	1.49
Day 4 (mean)	0.095	0.133	0.883	0.055	0.012	0.429	1.47
Day 5 (mean)	0.097	0.148	0.940	0.063	0.012	0.464	1.60
	passed	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**

The average results from each stability test for the MRM pesticides are given in **Table 5**. The individual results of the homogeneity experiments concerning SRM pesticides are shown in **Appendix 10**. The tests did not show any significant decrease in the pesticide levels at -18 °C indicating that at these storage conditions the pesticides present in the test material remained stable for the entire duration of the Proficiency Test.

Table 5 Stability test results of SRM-analytes, storage at -18 ºC, see also **Appendix 10**

	2,4-D (foll. alk. hydr.)	Dicamba (foll. akl. Hydr.)	Chlormequat	Ethephone	Glyphosphate	2,4-D (free acid)	Dicamba (free acid)
Day 1	0.468	0.289	0.503	0.559	1.190	0.440	0.147
	Storage at -18 ºC (mean values in mg/kg)						
Day 2	0.496	0.305	0.508	0.542	1.263		
Day 3	0.439	0.285	0.479	0.530	1.267	0.451	0.148
% deviation Day 3 vs. 1	**-6.1 %**	**-1.5 %**	**-4.8 %**	**-5.2 %**	**6.5 %**	**2.6 %**	**1.0 %**
	passed	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**

1.7 Organisational details

1.7.1 Access to documents, registration and confidentiality

In the invitation letter on 31 October all NRLs and OfLs were prompted to register at the CIRCA platform of the pesticide CRLs where the online registration link and all documents related to this EUPT (Calendar, Target Pesticides List, Specific Protocol, General Protocol) were uploaded. All relevant laboratories, independent of whether or not they were intending to participate at this exercise had to register on the webpage by the 14 January 2009. Laboratories that would not participate were given the opportunity to explain the reasons for their non-participation. After registration, the participants were provided with a laboratory code, a password, 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 material

One bottle of treated test material (150 g) and one bottle of 'blank' material (150 g) were shipped on 2 February 2009 to each participant in boxes containing a freezer pack. 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 material received.

1.7.3 Submission of results

An online submission tool was developed, allowing participants to submit their results via the internet. All participants had access to the result-submission website (http://thor.dfvf.dk/ptc) from the day of the sample shipment until the result-submission deadline (3rd of March 2009). Participants were asked not only to report their analytical results, but also to give information regarding accreditation, reporting limits and details about the methods they used to analyse the test material.

2. EVALUATION OF THE RESULTS

2.1 False positives and negatives

2.1.1 False positives

These are the results that show the apparent presence of pesticides that were listed in the Target Pesticide List, but which were (i) not used in the sample treatment, (ii) and not detected by the organiser, even after a repeat analysis. However, if a number of participants do detect the same additional pesticide, or if the concentration is above the MRRL, then a decision as to whether, or not, this should be considered to be a false positive result will be made on a case-by-case basis. 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 that no numerical values were given, although they were used by the Organiser to treat the test material and were detected by the majority of participants at or above the MRRL.

2.2 Estimation of of the true concentration (μ)

The "true" concentration will be typically estimated using the median of all the results. Therefore a median value for every compound present will be calculated and used as the assigned value. In special justifiable cases, the EUPT Panel may decide to use only part of the population of results to establish the median (e.g. using only results with z-scores ≤ 5.0).

2.3 Establishing the standard deviation of the assigned value (target standard deviation)

The target standard deviation (δ) of the median will be calculated using a Fit-For-Purpose Relative Standard Deviation (FFP-RSD) approach, as follows:

$$\delta = b_i * \mu i \qquad \text{with } b_i \text{ i = FFP-RSD (= 0.25)}$$

The percentage FFP-RSD is typically set at 25 % based on experience from previous EUPTs. The EUPT-Panel reserves the right to also employ other approaches on a case-by-case basis considering analytical difficulties, and experience gained from previous proficiency tests.

2.4 z-Scores

This parameter is calculated using the following formula:

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

Where x_i is the value reported by the laboratory, μ_i the assigned value, and δ_i the standard deviation at that level for each pesticide (i).

Any z-scores of > 5 will be reported as "+5" particularly where summed z-scores of many pesticides are calculated (see SWZ below).

The z-scores will be interpreted in the following way:

$|z| \leq 2$ Acceptable

$2 < |z| \leq 3$ Questionable

$|z| > 3$ Unacceptable

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

The EUPT-Panel will consider whether, or not, these values should appear in the z-score histograms. However, a z-score will not be calculated for any false positive result.

2.5 Category A and B classification and combined z-scores (SWZ)

The EUPT-Panel will decide whether to classify the laboratories in two groups, A and B. Laboratories that detected a sufficiently high percentage of the pesticides present in the test material (e.g. at least 90%), reported no false positives, and sought all the pesticides on the Target Pesticide List marked with an asterisk that were present in the test material, will have demonstrated 'sufficient scope' and will therefore be classified in Category A.

For evaluation of the overall performance of the laboratories within Category A, a ranking according to the sum of weighted z-scores (SWZ) will be calculated.

The sum of weighted z-scores formula uses the z-scores with a fixed maximum value of 5 for individual z-scores, using the following formula:

$$\text{'Sum of weighted z-scores' (Z)} = \frac{\sum_{i=0}^{i\leq 2}|z|\cdot 1+\sum_{i>2}^{i\leq 3}|z|\cdot 3+\sum_{i>3}^{\infty}|z|\cdot 5}{n}$$

n = number of reported results

So for each laboratory:

- The first summation is the sum of all their /z-scores/ between zero to two, multiplied by 1.
- The second summation is the sum of all their /z-scores/ greater than two but less than or equal to, three, multiplied by 3.
- The third summation is the sum of all their z-scores greater than three, multiplied by 5.

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

$Z \leq 2$ Good

$2 < Z \leq 3$ Satisfactory

$Z > 3$ Unsatisfactory

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

SWZ ≤ 2 :	good;
2< SWZ≤ 3:	satisfactory;
SWZ > 3:	unsatisfactory

This categorisation was only applied to laboratories classified in Category A; i.e. laboratories having sufficient scope and including all pesticides marked with an asterisk in the potential pesticides list, and no false positives.

3. RESULTS – MRM PESTICIDES

In total, 111 laboratories representing 27 countries agreed to participate in this proficiency test, and 102 of the laboratories submitted their results for MRM pesticides before 3 March. The participating laboratories are listed in **Appendix 1**. All analytical results reported can be seen in **Table 9**, **Table 10**, **Table 11** and **Table 12**; the methods used are shown in **paragraph 3.4** and in detail in **Appendix 7**

3.1 Results

An overview of the results can be seen in **Table 6**. Chlorpyrifos was the most frequently analysed compound and 95 % of the labs submitted a result for this compound. All other pesticides were targeted by less than 80 % of the laboratories. Results for azoxystrobin and tebuconazole were reported by 70-80 % of the laboratories, while cyproconazole, cyprodinil, fenvalerate (sum), fenbuconazole, fenpropimorph, fludioxonil, flusilazole, lambda-cyhalothrin and pyraclostrobin were only targeted by 50-70 % of the laboratories.

3.1.1 False positives

Two laboratories (Lab code 007 and 153) reported false positive results above MRRL. One laboratory reported findings of procymidone (0.040 mg/kg), procloraz (0.040 mg/kg) and permethrin (0.040 mg/kg). The other laboratory reported finding deltamethrin (0.053 mg/kg). In all cases the compounds were not detected either by the organizer or by the other participating laboratories. Deltamethrin, permethrin and procloraz were not sprayed in the field, or spiked in the laboratory and the reported results are therefore considered to be false positives. Because procymidone had been sprayed in the field this result was not considered as a false positive.

3.1.2 False negatives

Pesticides actually present in the test material but reported as not detected (ND), were considered to be false negatives. **Table 6** summarizes the number of the reported false negatives for each pesticide. In total 32 false negative results were reported, which represents 3 % of the total number of results. The assigned value for malathion was below the MRRL and consequently any NDs reported for this compound were not considered as false negatives.

Table 6 Overview of results for MRM pesticides

Pesticides	No. of results	No. of NA [1)]	No. of ND [2)]	% results [3)]
Azoxystrobin	79	22	1	77
Carbendazim	66	36	0	65
Chlorpyrifos	98	4	0	96
Cyproconazole	62	37	3	61
Cyprodinil	70	30	2	69
Fenvalerate (sum)	71	26	5	70
Fenbuconazole	55	46	1	54
Fenpropimorph	56	44	2	55
Fludioxonil	65	31	6	64
Flusilazole	66	33	3	65
Lambda-cyhalothrin	77	20	5	75
Malathion	11	23	(68)	(11)
Metconazole	49	50	3	48
Pyraclostrobin	55	47	0	54
Tebuconazole	80	21	1	78

1) NA = not analysed
2) ND = not detected, possible false negatives
3) The % results has been calculated using the number of laboratories that reported results from the total number of laboratories submitting results (n = 103). False negatives not included in reported results.

3.2 Assigned values and target standard deviations

To establish the assigned values, the median levels of all the reported results, excluding the outliers, were used. Fifteen results were regarded as outliers; carbendazim (1.15, 1.98, 2.892 and 2.956 mg/kg), cyproconazole (1.22 mg/kg), fenvalerate(sum) (0.28, 0.343, 0.347, 0.361, 0.439, 0.55 mg/kg), fenpropimorph (0.335 mg/kg), fludioxinil (0.84 mg/kg), lambda-cyhalothrin (0.1631mg/kg) and metconazole (1.15 mg/kg).

All assigned values for the MRM pesticides can be seen in **Table 7**. 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 all the reported results for informative purposes only. The range of Qn-RSD values was from 14 to 42 % but on average the Qn-RSD was 26%, and thus very close to the 25% FFP RSD used for the calculations.

It was not possible to perform a reliable statistical evaluation of the results for malathion because the median value was below the MRRL.

The laboratories were asked to report results for 1) fenvalerate and esfenvalerate, RS and SR isomers and 2) fenvalerate and esfenvalerate, RS and SR isomers according to the residue definition, as well the sum of all isomer. Because, isomerisation from one isomer to another occur during analysis it was decided by the Advisory Group to evaluate only the sum of all isomers.

Table 7 Assigned values and RSDs for all MRM pesticides present in the test material.

Pesticides	MRRL	Assigned value (mg/kg)	FFP RSD (%)	Qn RSD all (%)
Azoxystrobin	0.02	0.175	25	27
Carbendazim	0.01	0.472	25	42
Chlorpyrifos	0.02	1.044	25	25
Cyproconazole	0.02	0.453	25	22
Cyprodinil	0.02	0.076	25	23
Fenvalerate (sum)	0.02	0.097	25	33
Fenbuconazole	0.02	0.508	25	25
Fenpropimorph	0.02	0.121	25	29
Fludioxonil	0.02	0.078	25	30
Flusilazole	0.02	0.728	25	14
Lambda-cyhalothrin	0.02	0.050	25	30
Malathion	0.02	0.011		
Metconazole	0.02	0.478	25	28
Pyraclostrobin	0.02	0.746	25	21
Tebuconazole	0.02	1.23	25	24

3.3 Assessment of laboratory performance

3.3.1 z-Scores

The z-scores have been calculated for the quantified pesticides using the FFP RSD of 25 %. **Table 8** shows an overview of the results and **Table 9, Table 10, Table 11** and **Table 12** shows the individual results and z-scores for each laboratory and pesticide together with the assigned value. A graphical representation of the z-scores can be seen in **Appendix 6** and the histograms showing the distribution of the results in **Appendix 5**.

Table 8 Number of acceptable, questionable and unacceptable z-scores for MRM pesticides

Pesticides	Acceptable z-scores	Questionable z-scores	Unacceptable z-scores	False negatives
Azoxystrobin	71	8	1	1
Carbendazim	49	9	8	
Chlorpyrifos	93	3	2	
Cyproconazole	57	4	4	3
Cyprodinil	63	8	1	2
Fenvalerate (sum)	58	4	14	5
Fenbuconazole	50	5	1	1
Fenpropimorph	52	3	3	2
Fludioxonil	58	11	2	6
Flusilazole	62	3	4	3
Lambda-cyhalothrin	69	12	1	5
Malathion				
Metconazole	46	2	4	3
Pyraclostrobin	49	5	1	
Tebuconazole	75	5	1	1

For chlorfyrifos, fenpropimorph, flusilazole, and tebuconazole acceptable results were obtained by 90-94 % of the laboratories. For axoxystrobin, cyproconazole, cyprodinil, fenbuconazole, fludioxonil, lambda-cyhalothrin, metconazole, and pyraclostrobin acceptable results were obtained by 82-89 % of the laboratories. However, for carbendazim and esfenvalerate acceptable z-scores were obtained by only 74-76 % of the laboratories.

Table 9 Results azoxystrobin, carbendazim, chlorpyrifos and cyproconazole in mg/kg and their calculated z-scores using FFP RSD 25 %.

Laboratory code	Azoxystrobin	Z-scores (FFP RSD (25%)	Carbendazim	Z-scores (FFP RSD (25%)	Chlorpyrifos	Z-scores (FFP RSD (25%)	Cyproconazole	Z-scores (FFP RSD (25%)
MRRL	**0.02**		**0.01**		**0.02**		**0.02**	
Assigned value	**0.175**		**0.472**		**1.04**		**0.453**	
001	0.188	0.3	0.41	-0.5	0.924	-0.5	0.453	0.0
002	0.216	0.9	0.63	1.3	1.33	1.1	0.6	1.3
003	0.163	-0.3	0.521	0.4	0.802	-0.9	0.413	-0.4
004	0.193	0.4	0.395	-0.6	1	-0.2	0.465	0.1
005	0.144	-0.7	0.458	-0.1	1.19	0.6	0.404	-0.4
006	0.169	-0.1	0.414	-0.5	0.947	-0.4	0.291	-1.4
007	0.14	-0.8	0.44	-0.3	1.44	1.5	0.42	-0.3
008	0.22	1.0	0.426	-0.4	1.3	1.0		
009	0.275	2.3	0.997	4.5	1.52	1.8	0.548	0.8
012					0.68	-1.4		
013	0.0577	-2.7			2.117	4.1		
014	0.188	0.3	0.55	0.7	0.846	-0.8	0.553	0.9
015	0.192	0.4			0.912	-0.5	0.443	-0.1
016	0.134	-0.9			0.83	-0.8		
017								
018	0.225	1.1	0.532	0.5	1.08	0.1	0.527	0.7
019	0.16	-0.3	0.633	1.4	0.887	-0.6	0.361	-0.8
020								
021	0.17	-0.1	0.37	-0.9	0.77	-1.0		
022	0.207	0.7	0.683	1.8	0.931	-0.4	0.518	0.6
024	0.12	-1.3	0.35	-1.0	1.05	0.0	0.36	-0.8
025			0.35	-1.0	0.499	-2.1		
026	0.185	0.2	0.136	-2.8	0.735	-1.2	0.441	-0.1
028	0.153	-0.5	1.15	5.8	1.05	0.0		
029	0.195	0.5	0.632	1.4	0.893	-0.6	0.553	0.9
030	0.134	-0.9	0.461	-0.1	0.905	-0.5	0.377	-0.7
031	0.175	0.0	0.313	-1.3	0.91	-0.5	0.42	-0.3
032	0.187	0.3	0.563	0.8	1.178	0.5	0.356	-0.9
033	0.0493	-2.9	0.131	-2.9	1.27	0.9	0.221	-2.0
034	0.209	0.8	0.582	0.9	1.19	0.6	0.499	0.4
035	0.167	-0.2	0.255	-1.8	0.851	-0.7		
036	0.16	-0.3			1.1	0.2	0.35	-0.9
037	0.159	-0.4	0.44	-0.3	1.13	0.3	0.527	0.7
038	0.187	0.3	0.723	2.1	1.17	0.5	0.574	1.1
040	0.219	1.0	0.491	0.2	0.887	-0.6	0.462	0.1
041			0.443	-0.2			0.217	-2.1

Laboratory code	Azoxystrobin	Z-scores (FFP RSD (25%)	Carbendazim	Z-scores (FFP RSD (25%)	Chlorpyrifos	Z-scores (FFP RSD (25%)	Cyproconazole	Z-scores (FFP RSD (25%)
MRRL	**0.02**		**0.01**		**0.02**		**0.02**	
Assigned value	**0.175**		**0.472**		**1.04**		**0.453**	
042	0.12	-1.3	0.021	-3.8	1.3	1.0		
043	0.229	1.2	0.51	0.3	0.762	-1.1	0.393	-0.5
044	0.213	0.9	0.589	1.0	0.924	-0.5	0.576	1.1
046	0.172	-0.1	0.404	-0.6	0.929	-0.4	0.408	-0.4
047	0.224	1.1	0.578	0.9	1.126	0.3	0.494	0.4
048	0.152	-0.5	0.852	3.2	1.06	0.1	0.447	-0.1
049	0.202	0.6	0.569	0.8	1.055	0.0	0.488	0.3
050	0.185	0.2	0.51	0.3	1.5	1.7	0.51	0.5
051								
053	0.188	0.3	0.625	1.3	1.135	0.3	0.503	0.4
054	0.219	1.0	0.708	2.0	1.21	0.6	0.527	0.7
055	0.274	2.3	0.457	-0.1	1.26	0.8	0.562	1.0
056	0.042	-3.0	0.169	-2.6	1.05	0.0		
057								
058	0.203	0.6	0.59	1.0	0.998	-0.2	0.453	0.0
059								
060	0.178	0.1	0.589	1.0	1.16	0.4	0.503	0.4
061	0.19	0.3	0.433	-0.3	0.722	-1.2	0.514	0.5
062					1.47	1.6		
063	0.177	0.0	0.482	0.1	1.08	0.1	0.434	-0.2
064					1.45	1.6		
068					1.4	1.4		
069					1.35	1.2		
070	0.147	-0.6	0.432	-0.3	0.81	-0.9	ND	-3.9
071			0.192	-2.4	0.684	-1.4		
073	0.142	-0.8	0.592	1.0	1.119	0.3	0.494	0.4
074					1.34	1.1		
076					1.47	1.6		
077			0.3	-1.5	1.17	0.5		
078	ND	-3.5			0.208	-3.2	ND	-3.9
079					1.48	1.7		
081								
082	0.303	2.9	0.254	-1.8	0.745	-1.1	0.417	-0.3
083	0.111	-1.5	0.118	-3.0	1.103	0.2		
084	0.193	0.4	0.626	1.3	1.08	0.1	0.454	0.0
086	0.184	0.2	0.732	2.2	1.14	0.4	0.435	-0.2
087					0.935	-0.4		
088	0.193	0.4	0.396	-0.6	1.012	-0.1	0.461	0.1
089	0.092	-1.9	0.586	1.0	0.703	-1.3	0.287	-1.5

Laboratory code	Azoxystrobin	Z-scores (FFP RSD (25%)	Carbendazim	Z-scores (FFP RSD (25%)	Chlorpyrifos	Z-scores (FFP RSD (25%)	Cyproconazole	Z-scores (FFP RSD (25%)
MRRL	**0.02**		**0.01**		**0.02**		**0.02**	
Assigned value	**0.175**		**0.472**		**1.04**		**0.453**	
091	0.183	0.2	0.576	0.9	1.123	0.3	0.48	0.2
092	0.128	-1.1			0.907	-0.5	0.122	-2.9
093	0.116	-1.3	2.956	21.1	0.88	-0.6	1.22	6.8
094	0.125	-1.1			0.761	-1.1		
095	0.14	-0.8	0.22	-2.1	1.07	0.1	0.42	-0.3
097					0.76	-1.1		
099	0.152	-0.5	0.64	1.4	1.22	0.7	0.326	-1.1
100	0.112	-1.4	2.892	20.5	0.817	-0.9	0.165	-2.5
101	0.069	-2.4			0.648	-1.5	0.117	-3.0
102	0.145	-0.7			0.92	-0.5	0.475	0.2
103	0.2	0.6			1.02	-0.1	ND	-3.9
106	0.17	-0.1			0.85	-0.7	0.495	0.4
107	0.166	-0.2	0.5	0.2	0.748	-1.1	0.358	-0.8
108	0.25	1.7			0.86	-0.7		
112					0.815	-0.9		
114								
115	0.14	-0.8			1.1	0.2	0.33	-1.1
117								
118	0.076	-2.3	0.042	-3.6	0.327	-2.7		
121	0.163	-0.3			1.16	0.4		
122								
124	0.089	-2.0			1.21	0.6		
125	0.237	1.4			1.033	0.0		
126					1.21	0.6		
127	0.162	-0.3	1.98	12.8	1.08	0.1	0.509	0.5
129	0.18	0.1			1.038	0.0		
130	0.22	1.0	0.27	-1.7	1.44	1.5	0.317	-1.2
133					0.598	-1.7	0.359	-0.8
136								
138	0.209	0.8			0.931	-0.4	0.476	0.2
140	0.178	0.1	0.36	-0.9	1.02	-0.1	0.461	0.1
141					1.42	1.4		
146								
151								
152	0.127	-1.1	0.59	1.0	0.734	-1.2		
153					1.68	2.4		

Table 10 Results for cyprodinil, fenvalerate (sum), fenbuconazole and fenpropimorph in mg/kg and their calculated z-scores using FFP RSD 25 %

Laboratory code	Cyprodinil	Z-scores (FFP RSD (25%)	Fenvalerate (sum)	Z-scores (FFP RSD (25%)	Fenbuconazole	Z-scores (FFP RSD (25%)	Fenpropimorph	Z-scores (FFP RSD (25%)
MRRL	**0.02**		**0.02**		**0.02**		**0.02**	
Assigned value	**0.076**		**0.097**		**0.508**		**0.121**	
001	0.077	0.1	0.132	1.4	0.534	0.2	0.116	-0.2
002	0.099	1.2	0.105	0.3	0.564	0.4	0.134	0.4
003	0.0705	-0.3	0.0865	-0.4	0.494	-0.1	0.0646	-1.9
004	0.084	0.4	0.153	2.3	0.52	0.1	0.109	-0.4
005	0.0678	-0.4	0.129	1.3	0.339	-1.3	0.158	1.2
006	0.0692	-0.4	0.103	0.2	0.369	-1.1	0.102	-0.6
007							0.12	0.0
008	0.11	1.8	0.13	1.4	0.69	1.4	0.12	0.0
009	0.166	4.7	0.19	3.8	0.712	1.6	0.21	2.9
012			0.111	0.6	0.204	-2.4		
013			0.0783	-0.8				
014	0.069	-0.4	0.071	-1.1	0.564	0.4	0.123	0.1
015	0.066	-0.5	0.115	0.7				
016			0.094	-0.1				
017								
018	0.085	0.5	0.096	0.0	0.578	0.6	0.126	0.2
019	0.079	0.2	0.097	0.0	0.425	-0.7	0.083	-1.3
020								
021			0.1	0.1				
022	0.0926	0.9			0.577	0.5	0.121	0.0
024	0.072	-0.2	0.347	10.3	0.4	-0.9	0.13	0.3
025								
026	0.058	-0.9	0.032	-2.7	0.659	1.2	0.183	2.0
028	0.068	-0.4	0.088	-0.4				
029	0.087	0.6	0.28	7.5	0.508	0.0	0.146	0.8
030	0.0578	-1.0	0.0772	-0.8	0.368	-1.1	0.0986	-0.7
031	0.067	-0.5	0.0925	-0.2	0.47	-0.3	0.0585	-2.1
032	0.076	0.0	0.093	-0.2	0.555	0.4	0.122	0.0
033	0.0377	-2.0	0.067	-1.2	0.209	-2.4	0.137	0.5
034	0.086	0.5	0.113	0.7	0.59	0.6	0.134	0.4
035			0.092	-0.2				
036	0.08	0.2						
037	0.055	-1.1	0.077	-0.8	0.527	0.1	0.144	0.8
038	0.0859	0.5	0.1126	0.6	0.626	0.9	0.124	0.1
040	0.084	0.4	0.145	2.0	0.485	-0.2	0.103	-0.6
041					0.205	-2.4	0.066	-1.8
042	0.094	0.9	0.11	0.5				

Laboratory code	Cyprodinil	Z-scores (FFP RSD (25%)	Fenvalerate (sum)	Z-scores (FFP RSD (25%)	Fenbuconazole	Z-scores (FFP RSD (25%)	Fenpropimorph	Z-scores (FFP RSD (25%)
MRRL	**0.02**		**0.02**		**0.02**		**0.02**	
Assigned value	**0.076**		**0.097**		**0.508**		**0.121**	
043	0.079	0.2	0.067	-1.2	0.163	-2.7	0.118	-0.1
044	0.0626	-0.7	0.1	0.1	0.648	1.1	0.125	0.1
046	0.072	-0.2	0.091	-0.2	0.512	0.0	0.162	1.4
047	0.089	0.7	0.115	0.7	0.64	1.0	0.169	1.6
048	0.064	-0.6	0.08	-0.7	0.498	-0.1	0.104	-0.6
049	0.079	0.2	0.123	1.1	0.542	0.3	0.131	0.3
050	0.088	0.6	0.439	14.1	0.47	-0.3	0.145	0.8
051								
053	0.092	0.8	0.107	0.4	0.543	0.3	0.335	7.1
054	0.1	1.3	0.117	0.8	0.743	1.9	0.17	1.6
055	0.089	0.7	0.093	-0.2	0.784	2.2	0.183	2.0
056	0.073	-0.2	0.23	5.5				
057			0.016	-3.3				
058	0.074	-0.1					0.074	-1.6
059								
060	0.09	0.7	0.115	0.7	0.523	0.1	0.119	-0.1
061	0.072	-0.2	0.134	1.5	0.425	-0.7	ND	-3.3
062								
063	0.09	0.7	ND	-3.2			0.148	0.9
064								
068								
069								
070	0.062	-0.7	0.084	-0.5	0.377	-1.0	0.099	-0.7
071			0.07	-1.1				
073	0.097	1.1	0.059	-1.6	0.459	-0.4	0.098	-0.8
074								
076								
077			0.121	1.0				
078	ND	-2.9	ND	-3.2	ND	-3.8	ND	-3.3
079								
081								
082	0.076	0.0	0.167	2.9	0.478	-0.2	0.136	0.5
083			0.105	0.3				
084	0.078	0.1	0.09	-0.3	0.59	0.6	0.116	-0.2
086	0.095	1.0	0.102	0.2	0.667	1.3	0.159	1.3
087								
088	0.059	-0.9	0.07	-1.1	0.437	-0.6	0.123	0.1
089	0.046	-1.6	0.094	-0.1				
091	0.079	0.2	0.17	3.0	0.49	-0.1	0.133	0.4

Laboratory code	Cyprodinil	Z-scores (FFP RSD (25%)	Fenvalerate (sum)	Z-scores (FFP RSD (25%)	Fenbuconazole	Z-scores (FFP RSD (25%)	Fenpropimorph	Z-scores (FFP RSD (25%)
MRRL	**0.02**		**0.02**		**0.02**		**0.02**	
Assigned value	**0.076**		**0.097**		**0.508**		**0.121**	
092			0.142	1.9				
093	0.075	-0.1					0.145	0.8
094	0.049	-1.4	0.07	-1.1				
095	0.08	0.2	0.55	18.7	0.4	-0.9	0.09	-1.0
097	ND	-2.9	0.07	-1.1				
099	0.07	-0.3	0.093	-0.2	0.341	-1.3	0.083	-1.3
100	0.114	2.0	ND	-3.2			0.156	1.2
101	0.035	-2.2			0.268	-1.9	0.068	-1.8
102	0.073	-0.2	0.123	1.1	0.535	0.2		
103	0.12	2.3			0.448	-0.5		
106	0.063	-0.7	0.124	1.1	0.526	0.1	0.112	-0.3
107	0.055	-1.1	0.361	10.9	0.498	-0.1	0.086	-1.2
108	0.08	0.2						
112								
114								
115	0.08	0.2	0.09	-0.3				
117								
118	0.035	-2.2	0.103	0.2			0.052	-2.3
121	0.063	-0.7						
122								
124	0.087	0.6	ND	-3.2				
125								
126			ND	-3.2				
127	0.0806	0.2	0.103	0.2	0.474	-0.3	0.0957	-0.8
129								
130	0.068	-0.4			0.356	-1.2	0.064	-1.9
133	0.044	-1.7	0.078	-0.8				
136								
138			0.091	-0.2	0.534	0.2		
140	0.0716	-0.2	0.0697	-1.1	0.636	1.0	0.0953	-0.8
141								
146								
151								
152	0.0352	-2.1	0.0768	-0.8				
153			0.343	10.1				

Table 11 Results for fludioxonil, flusilazole, lambda-cyhalothrin and malathion in mg/kg and their calculated z-scores using FFP RSD 25%.

Laboratory code	Fludioxonil	Z-scores (FFP RSD (25%)	Flusilazole	Z-scores (FFP RSD (25%)	Lambda-cyhalothrin	Z-scores (FFP RSD (25%)	Malathion	Z-scores (FFP RSD (25%)
MRRL	**0.02**		**0.02**		**0.02**		**0.02**	
Assigned value	**0.078**		**0.728**		**0.050**		**0.011*)**	
001	0.07	-0.4	0.626	-0.6	0.0475	-0.2	ND	
002	0.096	0.9	0.794	0.4	0.058	0.6	ND	
003	0.077	-0.1	0.73	0.0	0.0424	-0.6	ND	
004	0.088	0.5	0.674	-0.3	0.067	1.4	ND	
005	0.102	1.2	0.621	-0.6	0.052	0.2	ND	
006	0.0806	0.1	0.631	-0.5	0.049	-0.1	ND	
007	ND	-3.0			0.05	0.0	ND	
008	0.11	1.6	0.9	0.9	0.068	1.4	0.02	
009	0.12	2.2	0.815	0.5	0.0757	2.1	ND	
012	0.032	-2.4			0.067	1.4		
013					0.0145	-2.8	ND	
014	0.076	-0.1	0.758	0.2	0.056	0.5	ND	
015	0.066	-0.6	0.697	-0.2	0.058	0.6	ND	
016					0.028	-1.8		
017								
018	0.087	0.5	0.831	0.6	0.033	-1.4	ND	
019	0.063	-0.8	0.617	-0.6	0.049	-0.1	ND	
020								
021							ND	
022	0.0987	1.1	0.776	0.3	ND	-2.4	ND	
024	0.07	-0.4	0.62	-0.6	0.038	-1.0	ND	
025					ND	-2.4	ND	
026	0.078	0.0	0.692	-0.2	0.055	0.4	ND	
028					0.034	-1.3	ND	
029	0.079	0.1	0.728	0.0	0.05	0.0	0.01	
030	0.0577	-1.0	0.576	-0.8	0.0396	-0.8		
031	0.0775	0.0	0.76	0.2	0.052	0.2	0.0102	
032	0.081	0.2	0.879	0.8	0.05	0.0	ND	
033	0.0532	-1.3	0.388	-1.9	0.0408	-0.7	ND	
034	0.089	0.6	0.863	0.7	0.054	0.3	ND	
035			0.747	0.1	0.048	-0.2	ND	
036			0.45	-1.5	0.06	0.8		
037	0.068	-0.5	0.732	0.0	0.051	0.1	ND	
038	0.0952	0.9	0.848	0.7	0.0633	1.1	ND	
040	0.078	0.0	0.766	0.2	0.038	-1.0	ND	
041								
042	0.08	0.1	0.65	-0.4	0.053	0.2	ND	

Laboratory code	Fludioxonil	Z-scores (FFP RSD (25%)	Flusilazole	Z-scores (FFP RSD (25%)	Lambda-cyhalothrin	Z-scores (FFP RSD (25%)	Malathion	Z-scores (FFP RSD (25%)
MRRL	**0.02**		**0.02**		**0.02**		**0.02**	
Assigned value	**0.078**		**0.728**		**0.050**		**0.011*)**	
043	0.064	-0.7	0.688	-0.2	0.04	-0.8	ND	
044	0.0641	-0.7	0.928	1.1	0.0506	0.0	0.0077	
046	0.078	0.0	0.593	-0.7	0.049	-0.1	ND	
047	0.112	1.7	0.9	0.9	0.049	-0.1	ND	
048	0.059	-1.0	0.672	-0.3			ND	
049	0.094	0.8	0.819	0.5	0.062	1.0	0.01	
050	0.097	1.0	0.65	-0.4	0.08	2.4	ND	
051								
053	0.067	-0.6	0.82	0.5			ND	
054	0.092	0.7	0.72	0.0	0.0534	0.3	ND	
055	0.094	0.8	0.76	0.2	0.055	0.4	ND	
056	0.43	18.1						
057	0.05	-1.4	ND	-3.9	0.02	-2.4		
058	0.092	0.7	0.756	0.2	0.062	1.0	ND	
059								
060	0.099	1.1	0.835	0.6	0.064	1.1	0.011	
061	0.078	0.0	0.687	-0.2	0.056	0.5	ND	
062								
063	0.098	1.0	0.67	-0.3	0.06	0.8	0.02	
064							ND	
068							ND	
069							ND	
070	0.053	-1.3	0.695	-0.2	0.04	-0.8	ND	
071					0.04	-0.8	ND	
073	0.079	0.1	0.649	-0.4	0.052	0.2	ND	
074								
076								
077					0.044	-0.5		
078	ND	-3.0	ND	-3.9	0.02	-2.4	ND	
079								
081								
082	0.068	-0.5	0.475	-1.4	0.077	2.2	0.02	
083			0.68	-0.3	0.042	-0.6		
084	0.08	0.1	0.714	-0.1	0.048	-0.2	ND	
086	0.094	0.8	0.73	0.0	0.05	0.0	ND	
087							ND	
088	0.069	-0.5	0.696	-0.2	0.032	-1.4	0.009	
089	0.055	-1.2			0.033	-1.4		
091	0.078	0.0	0.793	0.4	0.064	1.1	ND	

Laboratory code	Fludioxonil	Z-scores (FFP RSD (25%)	Flusilazole	Z-scores (FFP RSD (25%)	Lambda-cyhalothrin	Z-scores (FFP RSD (25%)	Malathion	Z-scores (FFP RSD (25%)
MRRL	**0.02**		**0.02**		**0.02**		**0.02**	
Assigned value	**0.078**		**0.728**		**0.050**		**0.011*)**	
092			0.163	-3.1	0.057	0.6	ND	
093			0.816	0.5	0.045	-0.4		
094	0.047	-1.6	0.755	0.2	0.03	-1.6	ND	
095	0.07	-0.4	0.65	-0.4	0.07	1.6	ND	
097	ND	-3.0			0.04	-0.8	ND	
099	0.077	-0.1	0.766	0.2	0.038	-1.0	ND	
100	ND	-3.0	0.798	0.4	0.052	0.2	ND	
101	0.045	-1.7	0.238	-2.7	0.026	-1.9	ND	
102	0.84	39.1	0.8	0.4				
103	ND	-3.0	ND	-3.9	0.06	0.8		
106	0.13	2.7	0.724	0.0	0.041	-0.7	ND	
107	0.021	-2.9	0.754	0.1	0.049	-0.1	ND	
108	0.11	1.6			0.05	0.0	ND	
112								
114								
115	0.08	0.1	0.73	0.0	0.07	1.6	ND	
117								
118	0.046	-1.6	0.214	-2.8	0.042	-0.6	ND	
121	0.077	-0.1	0.692	-0.2	0.163	9.0		
122								
124	ND	-3.0			ND	-2.4		
125					0.034	-1.3	0.021	
126					ND	-2.4	ND	
127	0.118	2.1	0.727	0.0	0.0542	0.3	ND	
129					0.068	1.4		
130	0.089	0.6	0.245	-2.7	0.029	-1.7		
133					ND	-2.4	ND	
136								
138			0.769	0.2				
140	0.0931	0.8	0.776	0.3	0.0754	2.0	0.01	
141							ND	
146								
151								
152	0.0421	-1.8			0.0687	1.5	ND	
153							0.015	

Table 12 Results for metconazole, pyraclostrobin and tebuconazole in mg/kg and their calculated z-scores using FFP RSD 25%.

Laboratory code	Metconazole	Z-scores (FFP RSD (25%)	Pyraclostrobin	Z-scores (FFP RSD (25%)	Tebuconazole	Z-scores (FFP RSD (25%)
MRRL	**0.02**		**0.02**		**0.02**	
Assigned value	**0.478**		**0.746**		**1.23**	
001	0.453	-0.2	0.866	0.6	1.478	0.8
002	0.613	1.1	0.722	-0.1	1.43	0.7
003	0.473	0.0	0.724	-0.1	1.36	0.4
004	0.644	1.4	1.09	1.8	1.23	0.0
005	0.296	-1.5	0.644	-0.5	1.03	-0.6
006	0.432	-0.4	0.691	-0.3	1.05	-0.6
007					0.57	-2.1
008	0.476	0.0	0.92	0.9	1.8	1.9
009	0.642	1.4	1.11	2.0	1.35	0.4
012					0.605	-2.0
013						
014	0.636	1.3	0.69	-0.3	1.214	0.0
015					1.274	0.2
016						
017						
018	0.582	0.9	0.817	0.4	1.484	0.8
019	0.33	-1.2	0.567	-1.0	0.912	-1.0
020						
021			0.37	-2.0	1.03	-0.6
022	0.611	1.1	0.81	0.3	1.21	0.0
024	0.39	-0.7	0.47	-1.5	1.22	0.0
025						
026	0.465	-0.1	1.28	2.9	1.44	0.7
028					1.73	1.6
029	0.426	-0.4	0.687	-0.3	1.349	0.4
030	0.428	-0.4	0.634	-0.6	1.1	-0.4
031	0.455	-0.2	0.852	0.6	1.14	-0.3
032	0.596	1.0	0.843	0.5	1.45	0.7
033	0.251	-1.9	0.477	-1.4	0.661	-1.8
034	0.538	0.5	0.932	1.0	1.54	1.0
035					1.087	-0.5
036					0.9	-1.1
037	0.48	0.0	0.745	0.0	1.3	0.2
038	ND	-3.8			1.51	0.9
040	0.506	0.2	0.733	-0.1	1.29	0.2
041			0.349	-2.1		
042	0.37	-0.9			1.2	-0.1

Laboratory code	Metconazole	Z-scores (FFP RSD (25%)	Pyraclostrobin	Z-scores (FFP RSD (25%)	Tebuconazole	Z-scores (FFP RSD (25%)
MRRL	**0.02**		**0.02**		**0.02**	
Assigned value	**0.478**		**0.746**		**1.23**	
043	0.412	-0.6	0.43	-1.7	0.962	-0.9
044	0.631	1.3	0.897	0.8	1.53	1.0
046	0.398	-0.7	0.784	0.2	1.152	-0.2
047	0.788	2.6	0.827	0.4	1.62	1.3
048	0.537	0.5	0.772	0.1	1.05	-0.6
049	0.549	0.6	0.793	0.3	1.37	0.5
050	0.39	-0.7	0.74	0.0	1.41	0.6
051						
053	0.581	0.9	0.859	0.6	1.435	0.7
054	0.669	1.6	0.82	0.4	1.37	0.5
055	0.638	1.3	0.842	0.5	1.41	0.6
056	0.286	-1.6	0.3	-2.4	1.55	1.1
057					0.758	-1.5
058	0.51	0.3	0.62	-0.7	1.25	0.1
059						
060	0.475	0.0	0.699	-0.3	1.31	0.3
061	0.392	-0.7	0.793	0.3	1.416	0.6
062						
063			0.761	0.1	0.951	-0.9
064						
068						
069						
070	ND	-3.8	0.665	-0.4	1.13	-0.3
071						
073	0.486	0.1	0.893	0.8	1.182	-0.1
074						
076						
077						
078	ND	-3.8			ND	-3.9
079						
081						
082	0.293	-1.5	0.75	0.0	0.99	-0.8
083					1.34	0.4
084	0.634	1.3	0.903	0.8	1.269	0.1
086	0.686	1.7	0.77	0.1	1.48	0.8
087						
088	0.442	-0.3	0.612	-0.7	1.348	0.4
089					0.647	-1.9
091	0.509	0.3	0.758	0.1	1.216	0.0

Laboratory code	Metconazole	Z-scores (FFP RSD (25%)	Pyraclostrobin	Z-scores (FFP RSD (25%)	Tebuconazole	Z-scores (FFP RSD (25%)
MRRL	**0.02**		**0.02**		**0.02**	
Assigned value	**0.478**		**0.746**		**1.23**	
092					0.809	-1.4
093	1.152	5.6	0.575	-0.9	1.494	0.9
094					1.02	-0.7
095	0.4	-0.7	0.09	-3.5	1.21	0.0
097					0.86	-1.2
099	0.35	-1.1	0.834	0.5	1.18	-0.1
100					0.828	-1.3
101					0.558	-2.2
102			0.746	0.0	1.535	1.0
103					1.42	0.6
106	0.495	0.1			1.21	0.0
107			0.648	-0.5	1.115	-0.4
108					1.66	1.4
112					1.01	-0.7
114						
115					1.2	-0.1
117						
118					0.308	-3.0
121					1.16	-0.2
122						
124					1.593	1.2
125						
126					1.75	1.7
127	0.502	0.2	0.657	-0.5	1.38	0.5
129						
130	0.227	-2.1	0.214	-2.9	0.453	-2.5
133						
136						
138			0.81	0.3	1.41	0.6
140			0.698	-0.3	1.28	0.2
141						
146						
151						
152					0.712	-1.7
153						

3.3.2 Sum of Weighted z-Scores (SWZ) – Catagory A

The MRM results were additionally evaluated by calculating the sum of weighted z-scores (SWZ). The SWZ values were calculated for only for the 47 laboratories that reported 12-14 results for MRM pesticides and all * marked pesticides. Malathion was not included in the calculation. The SWZ scores achieved by the labs can be seen in **Table 13** and the corresponding graph in **Figure 1**. In total, 34 participants (72 %) obtained SWZ scores at or below 2 (good) and 7 participants (15 %) obtained SWZ scores above 2 but below or at 3 (satisfactory) and 6 participants (13 %) obtained SWZ scores above 3 (unsatisfactory). No significant differences in the overall performance were seen between National Reference Laboratories (NRL) for cereals and Official Laboratories (OfLs).

Table 13 Sum of Weighted z-Scores (SWZ) for laboratories in Category A., number of pesticide analysed by the laboratory, false negatives reported and status as NRL for cereals.

Lab code	No. of pesticide analysed	SWZ	False negative	NRL	Lab code	No. of pesticide analysed	SWZ	False negative	NRL
001	14	0.4			**046**	14	0.4		
002	14	0.8		x	**047**	14	1.4		
003	14	0.4		x	**049**	14	0.5		
004	14	1.0			**050**	14	2.8		
005	14	0.8		x	**054**	14	1.0		x
006	14	0.5			**055**	14	1.6		
009	14	7.3		x	**058**	12	0.5		x
014	14	0.5			**060**	14	0.5		x
018	14	0.6			**061**	14	1.7	x	
019	14	0.7			**063**	12	1.8	x	x
022	13	1.2	x	x	**070**	14	3.3	x	
024	14	2.4		x	**073**	14	0.6		x
026	14	2.3		x	**082**	14	2.3		
029	14	2.2			**084**	14	0.4		x
030	14	0.7		x	**086**	14	0.6		
031	14	0.8		x	**088**	14	0.5		x
032	14	0.4		x	**091**	14	1.4		
033	14	3.2			**095**	14	8.8		
034	14	0.6			**099**	14	0.7		x
037	14	0.4			**107**	13	5.3		
038	13	2.5	x		**127**	14	2.5		
040	14	0.5		x	**130**	13	3.2		
043	14	1.3		x	**140**	13	0.6		
044	14	0.7							

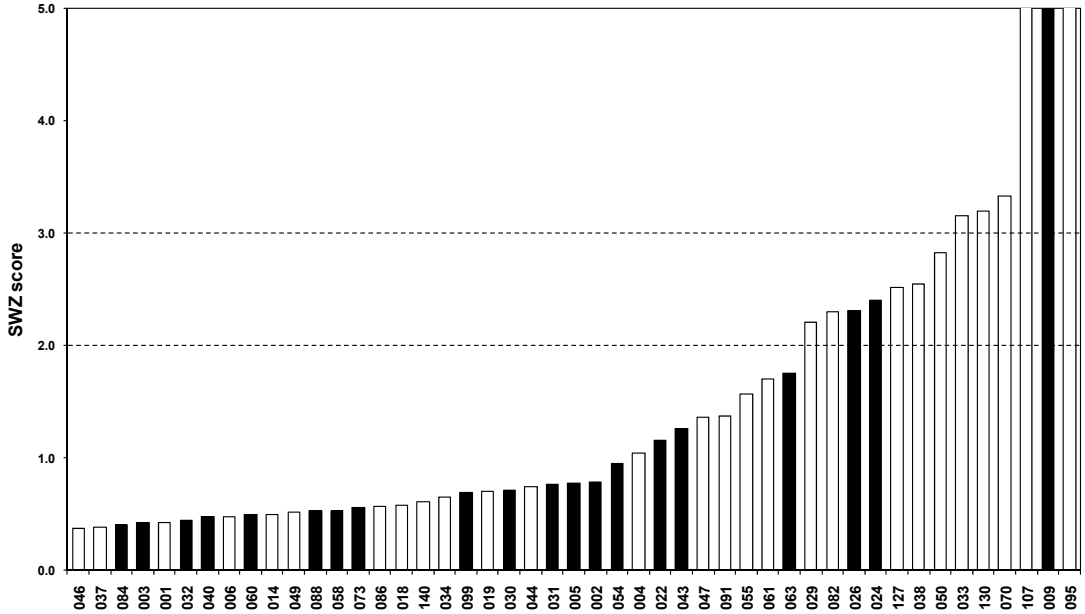


Figure 1: Category A laboratories ranked according to the Sum of Weighted z-Scores, SWZ. Black bars are NRL, white bars are OfL.

3.4 Trends in number of participating laboratories and their performance

The number of laboratories participating in the EUPTs with cereals test material increased from 62 in EUPT-C1, 72 in EUPT-C2 to 102 in EUPT-C3 (see **Table 14**) The target pesticide list has increased from 34 MRM pesticides to 51 MRM pesticides and the number of pesticides, spiked or incurred in the test material has increased from 7 to 14.

In the first two proficiency tests EUPT-C1 and EUPT-C2 60% or more the laboratories submitted results for all the included pesticides. However, in EUPT-C3 only 50 % of the laboratories submitted results for four of the pesticides. No significant improvement in performance has been seen when looking af the percentage of acceptable, questionable, unacceptable z-scores and false negative results. The percentage of Catagory A laboratories has fallen from 60 to 42 % from EUPT-C2 to EUPT-C3. Nevertheless, it is difficult to assess any improvement/deterioration in laboratory performance between the different proficiency tests, because the pesticides in the test materials and the laboratories participating in the PTs have changed. However, azoxystrobin and carbendazim have been included in all three test matrials, and can therefore be used as indicators of accuracy of performance.

The percentage of acceptable z-scores for azoxystrobin has increased from 62 % to 88 % from EUPT-C1 to EUPT-C3. This is probably due to the recommendation to add water to the sample before extraction. In fact, the procentage of laboratories adding water has also increased from 65 % to 86 %. For carbendazim, the percentage of acceptable z-scores has not increased, but decreased slightly from 79 % to 74 %. However, many of the participating laboratories in EUPT-C3 have not participated before, and it is not fully correct to include their results in a trend analysis. Focussing on the results only from laboratories that have participated in all the PTs, the laboratory performance has improved for both compounds. For azoxystrobin

the procentage of acceptable results has increased from 62 % to 93 % and for carbendazim from 79 % to 88 %. It is therefore reasonable to conclude that the PTs have been valuable for in improving analytical accuracy.

Table 14 Number of participants, pesticides, reported results, z-scores, number of Category A laboratories and SWZs for the 3 Cereal MRM PTs .

	EUPT-C1	EUPT-C2	EUPT-C3
Type of test material	Wheat flour	Wheat flour	Oat flour
No. of participants	63	72	102
No. of MRM pesticide included in the Target Pesticide List [1)]	34	43	51
No. of MRM pesticides in the test material	7	13	14
No. of results	323	830	981
Range of 'reported results', % [2)]	63 - 95	60 - 96	48 - 95
Acceptable z-scores, %	87	85	87
Questionable z-scores, %	7	12	8
Unacceptable z-scores, %	6	3	5
False negatives, %	2	3	3
Number of false positives	1	2	3
Catagory A, % of participating laboratories		60	46
Good SWZ, %		70	72
Satisfactory SWZ, %		9	15
Unsatisfactory SWZ, %		21	13

1) Number of pesticides, excluding isomers and degradation products
2) The ranges of reported results have been calculated using the number of laboratories that reported results from the total number of laboratories submitting results (see **Table 6**)

3.5 Analytical methods used for MRM pesticides

Detailed information regarding the methods used by the participants is presented in the following four tables (**Table 15, Table 16,Table 17** and **Table 18**). An overview of the reference methods used is given in **Table 15**. The category ‘other’ refers to other reference methods used or reference methods that were not reported. The QuEChERS method was used by 15-24 participants and was the most commonly used method, although different versions were applied. A few participants used the Dutch method, The CEN method EN 12393, Luke, the German official method. However, the participants used a broad variety of methods and a summary is therefore given below.

Table 15 Overview of the method used.

Pesticide	QuEChERS 1)	Dutch 2)	EN 12393	Luke	German 3)	Other 4)	Total
Azoxystrobin	24	1	3	4	5	42	79
Carbendazim	20		2	1	3	42	66
Chlorpyrifos	24	1	5	5	5	58	98
Cyproconazole	19	1	2	4	3	33	62
Cyprodinil	21	1	3	3	4	38	70
Esfenvalerat	15	1	3	3	5	44	71
Fenbuconazole	19	1	2	2	3	28	55
Fenpropimorph	18	1	1	1	2	33	56
Fludioxonil	20	1	2	2	4	36	65
Flusilazole	21	1	2	4	4	34	66
Lambda-cyhalothrin	19	1	3	4	4	46	77
Malathion	3		2			7	12
Metconazole	18	1	1	1	4	24	49
Pyraclostrobin	19		1	1	2	22	55
Tebuconazole	23	1	3	4	3	46	80

1) QuEChERS: different versions are used.
2) Dutch: Anal.Meth.f.Pest.Res.in Foodst.NL 6th Ed. 1996
3) German: The German official governmental method L 00.00-34 for the determination of pesticides
4) Other: other reference method used or a reference method was not used or reported.

An overview over the detection system used is shown in **Table 16**. The LC systems used were mainly LC/MS/MS. Only a few results were based on LC detectors like DAD, fluorescence and UV. For carbendazim, 13 participants used DAD, fluorescence or UV for detection. For several compounds about half of the results were obtained with GC-systems with selective detectors like NPD, FPD or ECD. The rest were obtained by MS and between 1 and 15 results were obtained by GC-MS/MS. The results for carbendazim (100 %) were solely obtained by LC-methods. Results for fenvalerate (sum) (100 %), lambda-cyhalothrin (99 %), chlorpyrifos (93 %), malathion (72 %) and cyprodinil (71 %) were primarily obtained by GC-methods. For cyproconazole, fenbuconazole, fenpropimorph, fludioxonil, flusilazole, and metconazole about half of the results were obtained by LC-based methods and the other half by GC-based methods.

Between 45-68% of the participants were accredited for the individual pesticides present in the test material (see **Table 17**). Only 45 % of the participants were accredited for fenbuconazole. But in general most of pesticides were analysed by 50-60 % accredited laboratories. Most participants (68 %) were accredited for carbendazim.

Table 16 Overview on the detection systems used by the participants. Numbers of participants thatused GC or LC systems and the respective detectors

Pesticide	GC-Total	GC-NPD	GC-FPD	GC-ECD	GCMS	GC-MS/MS	GC-other	LC- Total	LC-det. [a)]	LC/MS/MS	LC- Other	both GC and LC	not reported
Azoxystrobin	**43**	4		18	16	3	2	**29**	1	27	1	5	2
Carbendazim								**62**	13	48	1	1	3
Chlorpyrifos	**89**	20	9	13	23	15	9	**7**		7		4	2
Cyproconazole	**29**	14		1	10	4		**28**		27	1	4	1
Cyprodinil	**45**	15		1	16	10	3	**18**	1	16	1	5	2
Esfenvalerat	**67**	2		25	22	14	4					3	1
Fenbuconazole	**29**	7		4	13	3	2	**25**		25		3	
Fenpropimorph	**31**	7			15	6	3	**23**		23		3	2
Fludioxonil	**37**	12		1	13	10	1	**26**	1	24	1	3	2
Flusilazole	**39**	17		1	14	5	2	**27**	1	26		4	
Lambda-cyhalothrin	**73**	2		27	26	11	7	**1**		1		3	
Metconazol	**19**	6		1	6	5	1	**27**		26	1	2	1
Pyraclostrobin	**16**	3		2	7	3	1	**36**		36		2	1
Tebuconazol	**51**	19		1	19	8	4	**27**		26	1	4	2

a) DAD, Fluorescence or UV

Table 17 Accredited laboratories, reporting limits (RL) and Sub-Sample Weights (SW) listed for MRM pesticides. The numbers in brackets are the number of participating laboratories that are not accredited. RL are given in mg/kg and SW in g.

Pesticide	ACCREDITED	RL ≤ 0.01	0.01 ≤ LOD ≤ 0.05	0.05 < LOD < 0.1	LOD ≥ 0.1	SW ≤ 5 g	5 < SW ≤ 10 g	10 < SW ≤ 15 g	15 < SW ≤ 20 g	20 < SW ≤ 25 g	SW ≤ 75 g
Azoxystrobin	47 (32)	25	53		1	43	16	6	6	6	2
Carbendazim	45 (21)	49	13	4		42	8	5	1	6	4
Chlorpyrifos	59 (39)	39	58		1	47	22	7	7	8	7
Cyproconazole	34 (28)	15	43	4		37	9	4	6	4	2
Cyprodinil	41 (29)	22	48			41	13	5	4	4	2
Fenvalerate (sum)	41 (29)	16	55			32	13	5	7	8	6
Fenbuconazole	25 (30)	15	37	2		34	9	3	5	2	2
Fenpropimorph	33 (23)	18	37			39	6	5	2	3	1
Fludioxonil	38 (27)	20	45			42	9	4	4	3	3
Flusilazole	38 (28)	21	44	1		36	13	5	6	4	2
Lambda-cyhalothrin	49 (28)	21	57	1		40	15	5	5	7	6
Metconazole	30 (19)	16	31	1		34	6	4	2	2	1
Pyraclostrobin	29 (26)	17	38			40	6	3	2	2	2
Tebuconazole	42 (38)	21	57	1		44	16	5	6	7	2

More than half of the laboratories (63-84 %) used 10 g or less of test material for the analysis of MRM pesticides. Between 45 % and 73 % of the laboratories used 5 g or less of test material for the analysis of MRM pesticides.

Between 77 % and 95 % of the participants added water to the sample prior to extraction which is in accordance with the recommendations in document SANCO/2007/3131 on Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed.

For twelve out of fifhteen compounds cleanup by Dispersive SPE were most widely used (**Table 18)**. The second most used cleanup method was 'no cleanup' and for carbendazim 'no cleanup' was the most widely used. For chlorpyrifos, fenvalerate (sum) and lambda-cyhalthrin DSPE and GPC had been used by about the same number of laboratories.

Table 18 Method information; water addition prior to extraction, clean-up and calibration. The numbers in brackets are participants that have stated that they did not add water to the sample prior to extraction (see **Appendix 14** list of abbreviations)

Pesticide	WATER ADDITION	GPC	DSPE	SPE	None	Freezing out	liq./liq part.	Other	MM-ML	MM-SL	PS-ML	PS-SL	Standard addition
Azoxystrobin	68 (11)	14	20	13	19	4	1	5	50	10	13	3	2
Carbendazim	55 (11)	6	14	7	16	5	8	4	38	4	17	2	2
Chlorpyrifos	77 (21)	23	24	22	15	3	3	5	59	10	4	3	3
Cyproconazole	55 (7)	10	20	6	14	4	1	5	42	8	7	3	2
Cyprodinil	60 (10)	11	20	11	15	3	1	5	56	9	8	2	2
Esfenvalerat	55 (16)	24	17	11	10	2	2	4	57	8	12	3	2
Fenbuconazole	50 (5)	10	17	8	12	4		3	37	6	9	2	1
Fenpropimorph	49 (7)	6	19	6	13	4	1	3	38	6	6	2	2
Fludioxonil	55 (10)	10	18	9	16	4		5	43	8	8	2	2
Flusilazole	57 (9)	12	18	10	13	4	2	5	41	9	11	3	2
Lambda-cyhalothrin	59 (18)	19	22	13	11	4	1	1	57	10	13	3	2
Malathion	11 (1)		3	3	2	1	1	1	7	3	1		
Metconazol	45 (4)	4	17	6	14	2		3	36	5	5		2
Pyraclostrobin	52 (3)	6	14	8	13	6	1	4	40	5	6	1	2
Tebuconazol	66 (14)	15	22	12	19	4	1	5	54	9	11	3	2

3.6 Summary, conclusions and prospect for the MRM part

EUPT-C3/SRM4 was the third PT focussing on cereal-based test materials and the second PT to be organized collaboratively between the CRL for Single Residue Methods and the CRL for Cereals and Feedingstuff.

A homogenous test material of oat flour, including both incurred and spiked samples, was successfully prepared. The oats were sprayed in the field and spiked in the laboratory following harvest with commercially available pesticide formulations. The test material included the following pesticides: azoxystrobin, carbendazim, chlorpyrifos, cyproconazole, cyprodinil, fenvalerate, fenbuconazole, fenpropimorph, fludioxonil, flusilazole, lambda-cyhalothrin, malathion, metconazole, pyraclostrobin, and tebuconazole. One hundred and eleven laboratories, representing 27 countries, agreed to participate in the proficiency test, but eight laboratories failed to submit results. All Member States and NRLs participated, however Malta and Luxembourg have delegated their obligations to laboratories in the UK and Belgium, respectively. Out of the 103 laboratories, 102 submitted at least one result involving MRM-compounds. One laboratory submitted only results for SRM-pesticides, and one laboratory had such a limited scope that it did not include any of the pesticides present in the EUPT-C3 test material.

The target pesticide list distributed to the laboratories prior to the test contained in total 51 MRM-pesticides, excluding isomers and degradation products. The test material contained 14 pesticides namely azoxystrobin, carbendazim, chlorpyrifos, cyproconazole, cyprodinil, fenvalerate, fenbuconazole, fenpropimorph, fludioxonil, flusilazole, lambda-cyhalothrin, metconazole, pyraclostrobin and tebuconazole. Additionally, residues of malathion below the MRRL were present in the test material. All of the pesticides were applied in the field.

A low number of false positives were reported but the number of false negatives washigher. The three false positives were deltamethrin, permethrin and procloraz. The 32 false negatives were azoxystrobin (1) , cyproconazole (3), cyprodinil (2), fenvalerate(sum) (5), fenbuconazole (1), fenpropimorph (2), fludioxonil (6), flusilazole (3), metconazole (3) and tebuconazole (1). The average Qn-RSD (robust RSD) was 26 % close to the FFP-RSD of 25 % and on average ranging from 14 to 42 %.

It was not possible to perform a statistical evaluation of the results for malathion because the median value was below the MRRL and results for fenvalerate and esfenvalerate were evaluated only as the sum of all isomers expressed as fenvalerate. For each laboratory/pesticide combination, z-scores based on the FFP RSD of 25 % were calculated and classified into ‘acceptable’, ‘questionable’ and ‘unacceptable’. For chlorpyrifos, fenpropimorph, flusilazole, and tebuconazole acceptable results were obtained by 90-94 % of the laboratories. For axozystrobin, cyproconazole, cyprodinil, fenbuconazole, fludioxonil, lambda-cyhalothrin, metconazole, and pyraclostrobin acceptable results were obtained by 82-89 % of the laboratories. However, for carbendazim and fenvalerate (sum) acceptable z-scores were obtained by only 74-76 % of the laboratories.

Overall, the performance of the laboratories appears not to have significantly improved compared to the previous PTs on cereals. The percentage of acceptable, z-scores has not increased and the percentage of Category A laboratories has fallen from 60-42 %. However, when focusing on azoxystrobin and carbendazim that has been included in all PTs and only looking at results from laboratories participating in all the PTs a significant improvement in the performance can be seen.

In future PTs, the selection of pesticides will continue to focus on pesticides included in the scope of the EU coordinated control programme as well as additional pesticides of relevance for cereal production in Europe and in other parts of the world from where significant amounts of cereals are imported. The pesticide residues will be incurred wherever practical and the commodites will be alternated between the most important commodities. In 2010 the test material will be rye and in 2011 the test material is planned to be rice. The target pesticide list for 2010 will be increased by the addition of nine pesticides: dichlorvos, flutriafol, hexaconazole, isoproturon, metribuzin, pyrimethanil, tebufenozide, trifluralin and triticonazole.

To encourage the laboratories to lower their LOQs so that they are able to fully enforce EU Regulations, e.g. MRLs for baby foods, the MRRL for almost all pesticides in the next PT will be lowered to 0.01 mg/kg. The aim is that laboratories continue to increase their scope of analytes and to improve their overall performance, both in terms of correctly detecting the pesticides present and determining the residue levels accurately.

The online web submission will continue and be expanded to cover all EUPTs for pesticide residues if possible.

4. RESULTS – SRM PESTICIDES

Out of the 111 laboratories that agreed to participate in this proficiency test, and the 103 that submitted results, only 48 laboratories (representing 23 EU-MS plus Switzerland and Norway) submitted at least one result for an SRM-compound. **Table 19** provides an overview.

Table 19 Overview of the participating laboratories in EUPT-C3/SRM4

Country	Participating labs		Sending at least one SRM-result		Notes
	Labs	NRL-SRM	Labs	NRL-SRM	
Austria	2	1	2	1	
Belgium	2	1	2	1	also representing LU
Bulgaria	6	1	1	1	
Cyprus	1	1	1	1	
Czech Republic	3	1	3	1	
Denmark	2	0	2	0	The NRL-SRM of DK was PT co-organiser
Estonia	2	1	2	1	
Finland	1	1	1	1	
France	4	0	3	0	FR had not yet assigned an NRL-SRM at the time of the PT
Germany	16	1	11	1	
Greece	3	2	2	2	GR has assigned two NRL-SRMs.
Hungary	3	1	3	1	
Ireland	1	1	1	1	
Italy	16	1	3	1	
Latvia	1	1	1	1	
Lithuania	1	1	1	1	
Luxemburg	(1)	(1)	(1)	(1)	Represented by NRL-SRM of BE
Malta	(1)	(1)	(1)	(1)	Represented by NRL-SRM of UK
Netherlands	2	1	1	1	
Poland	18	0	0	0	
Portugal	3	1	0	0	
Romania	3	1	0	0	
Slovakia	2	1	1	1	
Slovenia	3	1	2	1	
Spain	10	0	0	0	ES had not yet assigned an NRL-SRM at the time of the PT
Sweden	2	1 (+ 1)*	2	1 (+ 1)*	* Part of the SRM-compounds were subcontracted by the NRL-SRM to a private laboratory
United Kingdom	1	1	1	1	also representing MT
EU SUM	109	22	46	20	
EU-Countries	25+2	21+2	21+2	19+2	
Norway	1	1	1	1	
Switzerland	1	0	1	0	
OVERALL SUM	**111**	**23**	**48**	**21**	

Spain, Romania, Portugal and Poland have not reported any results for SRM-compounds, whereas Malta and Luxemburg have delegated their obligations to laboratories in UK and Belgium, respectively. The NRL-SRMs not submitting any SRM-results were from the following countries: Denmark, France, Spain, Romania, Portugal and Poland. Spain and France had not appointed any NRL-SRM at the time the PT was undertaken. The NRL-SRM for Denmark, being the co-organiser of this test, did not take part.

All laboratories participating in this PT, including those that have not submitted any results, are listed in **Appendix 1**. All individual results submitted by the laboratories concerning SRM-pesticides are listed in **Table 29**. The analytical methods used by the laboratories and the recoveries achieved are shown in **Chapter 4.4** and **Appendix 13**.

4.1 Overview of results for SRM-pesticides

Table 20 gives an overview of the results reported for each pesticide. Clearly fewer laboratories have analysed SRM-compounds than MRM-compounds, where the majority of the compounds were analysed by more than 60 % of the laboratories (see **Table 6**).

Table 20 Overview of results for SRM pesticides

Pesticides	No. of labs that reported results [1]	% of labs that reported results (based on n=48) [2]	% of labs that reported results (based on n=103) [3]	No. of reported ND [4]
2,4-D (foll. alk. hydr.)	33	69 %	32 %	1
2,4-D (free acid, OPT.) [5]	31	65 %	31 %	0
Dicamba (free acid)	21	44 %	20 %	2
Dicamba (foll. alk. hydr., OPT.) [5]	15	31 %	15 %	2
Chlormequat	38	83 %	37 %	0
Ethephon	7	16 %	7 %	3
Gylphosate	9	19 %	9 %	3

1) Including ND-results
2) 48 laboratories have submitted results for at least one SRM-compound
3) 103 laboratories have submitted results for at least one SRM or MRM-compound
4) ND = not detected, possibly false negatives
5) OPT. = Optional (not considered in overall laboratory ranking)

4.1.1 False positives

In the present test one lab reported mepiquat at < 0.005 mg/kg. As this result is below MRRL of 0.02 mg/kg, it was not regarded as a false positive.

4.1.2 False negatives

One laboratory (Lab code 026) has reported ND for “2,4-D (following alkaline hydrolysis)”. Two other laboratories (Lab codes 034 and 043) have reproted NDs for “dicamba (as free acid)”. As the assigned values (medians) of these analytes were much higher than the MRRL, the NDs reported were considered as false negative results. Furthermore, two laboratories (Lab codes 034 and 118) have reported NDs for the “dicamba (following alkaline hydrolysis)”, but were not considered to be false negative results as this was optional.

False negative results were reported also for ethephon by three labs (Lab codes 054, 055 and 086) and for glyphosate by the same three laboratories.

4.2 Assigned values, target standard deviations and outliers

All assigned values for the SRM pesticides are shown in **Table 21**. To establish the assigned values, the medians of the reported results were used. In the case of "dicamba (free acid)" one value was excluded from the median calculation because it was very distant from the second largest result reported with a z-score of 12.3. This individual result was also an outlier according to the Grubbs test. It should be noted, however, that if this outlier had not been excluded from the calculation the z-scores for "dicamba (free acid)" of the laboratories with the lab codes 022 and 044 would have been lower than 2 and thus classified as "acceptable". Also the WSZ of these two labs would have been lower than 2 and thus categorized as "Good" (see also **Table 23**).
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 (see **Table 21**).

In general, the FFP-RSD matches well with the Qn-RSD with exception of "dicamba (free acid)", which was the parameter with the lowest assigned value of 0.106 mg/kg.

Table 21 Median values and RSDs for all SRM-pesticides present in the test material

Pesticide	MRRL (mg/kg)	Assigned Value[1)] (mg/kg)	FFP-RSD (%)	Qn-RSD (%)
2,4-D (foll. alk. hydr.)	0.02	**0.499**	25	**27.5**
2,4-D (free acid, OPT.)	0.02	**0.471**	25	**22.6**
Dicamba (free acid)	0.02	**0.106**	25	**30.2**
Dicamba (foll. alk. hydr., OPT.)	0.02	**0.264**	25	**26.8**
Chlormequat	0.02	**0.479**	25	**25.8**
Ethephon	0.02	**(0.473)**	25	(54.9) [2)]
Gylphosate	0.02	**(1.218)**	25	(31.9) [2)]
			Average	26.6 [3)]

1) Median
2) Number of results not sufficient for appropriate statistical evaluation
3) Excluding the values for ethephon and Glyphosate

4.3 Assessment of laboratory performance

4.3.1 z-Scores and laboratory ranking

The z-scores for the pesticides were all calculated using the FFP-RSD of 25 %. No z-scores were calculated for ethephon and glyphosate, where too few participants submitted results. For the optional parameters ("2.4-D (free acid)" and "dicamba (following alkaline hydrolysis)") the z-scores were also calculated but only for informative purposes. **Table 22** shows the overall classification of the z-scores achieved by all laboratories. "Acceptable" z-scores (see classification rules in **Chapter 2.4**) were achieved by more than 70 % of the laboratories in all cases, with chlormequat (97 % "Good") at the top and "dicamba as free acid" (72 % Good) at the bottom. Overall 88 % of the results were classified as "Acceptable".

Table 22 Overall classification of z-scores

Pesticide	Acceptable		Questionable		Unacceptable		Number of false negatives
	No	%	No	%	No	%	
2,4-D (foll. alk. hydr.)	29	88	1	3	3	9	1
2,4-D (free acid, OPT.)	29	94	2	6	0	0	0
Dicamba (free acid)	15	72	3	14	3	14	2
Dicamba (foll. alk. hydr., OPT.)	11	74	2	13	2	13	2 (not counted)
Chlormequat	37	97	0	0	1	3	0
Overall	**121**	**88**	**8**	**6**	**9**	**7**	

Although the individual z-scores are considered as more important, the Sum of Weighted z-scores (SWZ), as described in **Chapter 2.5** were also calculated for each laboratory as a measure of overall performance. For this calculation only the results of “2,4-D (foll. alk. hydr.)”, “dicamba (free acid)” and chlormequat were considered. The results of ethephon and glyphosate were not considered due to the small population of results.

Table 23 and **Figure 2** show the laboratories in Category A ranked according to the SWZ scores (Note: The labs with the Lab codes 022 and 044 would have achieved SWZ- scores < 2 (Category “Good”) if the outlier had not been excluded from the median (assigned value) calculation.). **Table 24** shows the same laboratories ranked according to the Average of Absolute z-score (AAZ) values.

Only laboratotries that have a) reported values for 2,4-D (following alkaline hydrolysis), dicamba (free acid) and chlormequat, and b) have targeted MCPA (following alk. hydr.) and mepiquat, that are marked with asterisks, were included in Category A.

Table 25 shows the results achieved by Category B laboratories together with the reasoning for their exclusion from Category A. These labs are listed according to their lab codes.
In **Table 26** all laboratories are ranked according to their absolute z-scores for “2,4-D (foll. alk. hydr.)”, “dicamba (free acid)” and chlormequat. **Table 29** shows all the individual results and z-scores for each laboratory.

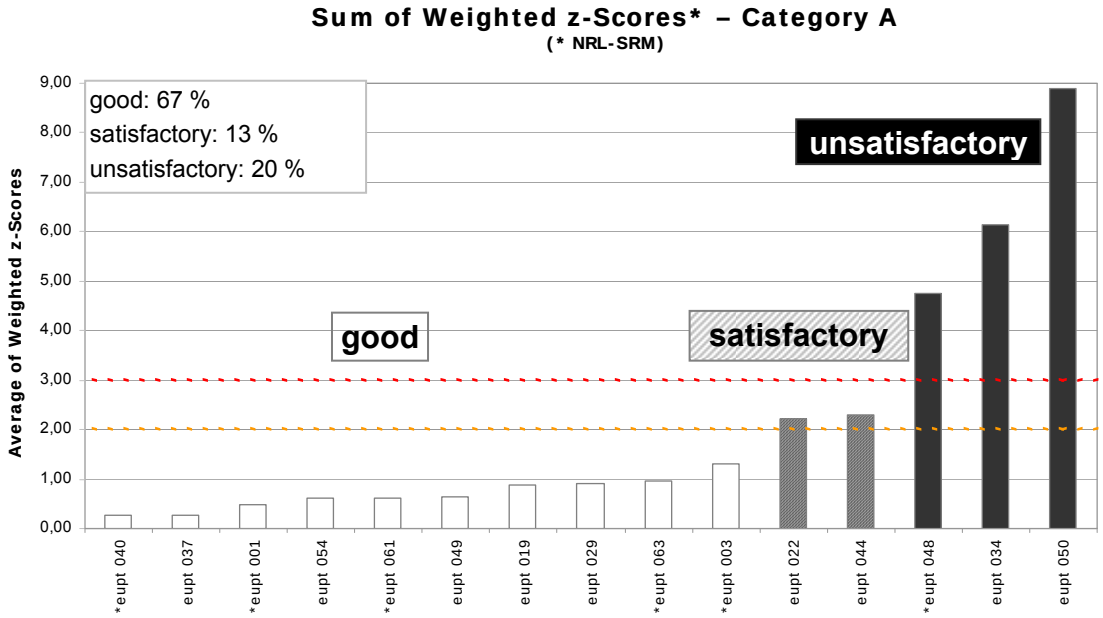
A graphical representation of the z-scores can be seen in **Appendix 11** and the histograms showing the distribution of the results in **Appendix 12**.

Table 23 Category A laboratories ranked according to the Sum of Weighted z-Scores (SWZ) [1)]

Ranking Pos.	Lab-code	NRL-SRM	No of Analytes sought for / correctly found [2)]	z-scores			Result submitted for		Targeted		SWZ	False negatives	Lab. Classification
				2,4-D (foll. alk. hydr.)	Dicamba (free acid)	Chlomequat	Ethephon	Glyphosate	MCPA (foll. alk. hydr.)	Mepiquat			
1	**040**	**Yes**	20 / 6	0.03	-0.44	0.27	**-**	**Yes**	Yes	Yes	**0.25**		**good**
2	**037**		21 / 7	0.14	-0.36	-0.25	**Yes**	**Yes**	Yes	Yes	**0.25**		**good**
3	**001**	**Yes**	14 / 5	-0.57	0.36	-0.52	**-**	**-**	Yes	Yes	**0.48**		**good**
4	**054**		21 / 5	-0.19	1.02	-0.64	**Yes**	**Yes**	Yes	Yes	**0.62**	**ethephon + glyphosate**	**good**
5	**061**	**Yes**	15 / 5	-0.04	-0.55	-1.29	**-**	**-**	Yes	Yes	**0.63**		**good**
6	**049**		19 / 5	-0.03	1.16	0.82	**-**	**-**	Yes	Yes	**0.67**		**good**
7	**019**		19 / 5	-0.79	1.54	-0.35	**-**	**-**	Yes	Yes	**0.89**		**good**
8	**029**		17 / 6	-1.02	-1.31	-0.41	**Yes**	**-**	Yes	Yes	**0.91**		**good**
9	**063**	**Yes**	18 / 6	1.17	-0.74	0.95	**Yes**	**Yes**	Yes	Yes	**0.95**		**good**
10	**003**	**Yes**	15 / 5	-1.42	-0.68	-1.83	**-**	**Yes**	Yes	Yes	**1.31**		**good**
11	**022**		16 / 6	0.33	2.07	0.14	**-**	**Yes**	Yes	Yes	**2.23** [5)]		**satisf.**
12	**044**	**(yes)** [4)]	14 / 4	-0.09	2.14	0.51	**-**	**-**	Yes	Yes	**2.34** [5)]		**satisf**
13	**048**	**Yes**	13 / 4	-2.04	2.67	0.22	**-**	**-**	Yes	Yes	**4.78**		**unsatisf.**
14	**034**		19 / 3	0.9	-3.25 (ND)	1.29	**-**	**-**	Yes	Yes	**6.14**	**dicamba (free acid) + dicamba (foll. Alk. hydr.)**	**unsatisf.**
15	**050**		20 / 6	0.81	5 [3)]	0.85	**Yes**	**-**	Yes	Yes	**8.89**		**unsatisf.**

1) This table includes only laboratories that a) have reported values for 2,4-D (following alkaline hydrolysis), dicamba (free acid) and chlormequat, b) have targeted MCPA (following alk. hydr.) and mepiquat as marked with an asterisk, and c) have not reported any false positive results.
2) This includes also the optional analyses
3) The z-score for dicamba (free acid) of this laboratory was lowered to 5 for the calculation of the AWAZ
4) Subcontracted by an NRL
5) These two labs would have received an WSZ of less than 2 (Good) for “dicamba (free acid)” if the outlier of lab 050 had not been excluded for the median calculation

Figure 2 Category A laboratories ranked according to the Sum of Weightet z-scores



NOTE: The labs with the Lab codes 022 and 044 would have achieved SWZ < 2 (Category "Good") if the outlier had not been excluded in the median (assigned value) calculation.

Table 24 Category A labs ranked according to the Average of Absolute z-Score (AAZ) values [1]

Ranking Position	Lab-code	NRL-SRM	2,4-D (foll. alk. hydr.) z-score	Dicamba (free acid) z-score	Chlormequat z-score	AAZ
1	040	**yes**	0.03	-0.44	0.27	**0.25**
2	037		0.14	-0.36	-0.25	**0.25**
3	001	**yes**	-0.57	0.36	-0.52	**0.48**
4	054		-0.19	1.02	-0.64	**0.62**
5	061	**yes**	-0.04	-0.55	-1.29	**0.63**
6	049		-0.03	1.16	0.82	**0.67**
7	022		0.33	2.07	0.14	**0.85**
8	019		-0.79	1.54	-0.35	**0.89**
9	029		-1.02	-1.31	-0.41	**0.91**
10	044	**(yes)** [3]	-0.09	2.14	0.51	**0.92**
11	063	**yes**	1.17	-0.74	0.95	**0.95**
12	003	**yes**	-1.42	-0.68	-1.83	**1.31**
13	048	**yes**	-2.04	2.67	0.22	**1.65**
14	034		0.9	-3.25 (ND)	1.29	**1.81**
15	050		0.81	5[2]	0.85	**2.22**

1) This table includes only laboratories that a) have reported values for 2,4-D (following alkaline hydrolysis), dicamba (free acid) and chlormequat, and b) have targeted MCPA (following alk. hydr.) and mepiquat, that are marked with an asterisk.
2) The z-score for dicamba (free acid) of this laboratory was lowered to 5 for the calculation of the AAZ
3) Subcontracted by an NRL

Table 25 Category B labs ordered by their Lab Codes

Lab.-Code	NRL-SRM	z-Score			Targeted by lab?		SWZ*	False neg.
		2,4-D (foll. alk. hydr.)	Dicamba (free acid)	Chlormequat	MCPA (foll. alk. hydr.)	Mepiquat		
005				-0.08		Yes	0.08	
006	Yes	0.25	0.94		Yes		0.60	
007	Yes	-2.22		0.01	Yes	Yes	3.34	
008				-0.20		Yes	0.20	
009	Yes	0.69		1.39	Yes	Yes	1.04	
014				0.12		Yes	0.12	
017	Yes	0.20		-3.10	Yes	Yes	7.85	
018		0.17		0.49	Yes	Yes	0.33	
021				-0.41		Yes	0.41	
024	Yes						-***	
026		-3.84 (ND)		0.18	Yes	Yes	9.68	Yes
030	Yes			-0.02		Yes	0.02	
031		-0.03	-0.79		Yes		0.41	
032	Yes	-1.66			Yes	Yes	1.66	
033		0.31		0.26	Yes	Yes	0.28	
038			-0.63	-1.91			1.27	
042				0.18			0.18	
043	Yes		-3.25 (ND)	0.72		Yes	8.47	Yes
046	Yes			0.05		Yes	0.05	
053				0.48		Yes	0.48	
055			-0.38	-1.38		Yes	0.88	Yes
058	Yes	-0.27			Yes		0.27	
060	Yes	1.98		-1.64	Yes		1.81	
082	Yes	0.95			Yes		0.95	
084	Yes	-0.39		-0.85	Yes	Yes	0.62	
086	Yes		0.94	-1.37		Yes	1.16	Yes
088	Yes						_ ***	
091		1.24		-0.30	Yes	Yes	0.77	
095		-0.20		-1.91	Yes	Yes	1.06	
117				0.93		Yes	0.93	
118		-3.80			Yes		18.99	
130		1.72			Yes		1.72	
140		3.52					17.58	

***** Calculated using all the available results of 2,4-D (following alkaline hydrolysis), dicamba (free acid) and chlormequat submitted by each lab.

****** The scope becomes sufficient if counted in combination with the subcontracted private laboratory (Lab code 044). Note: The labs with the Lab codes 006 and 044 had overlapping scope

******* These two laboratories have only reported results for optional SRMs

Table 26 Laboratory ranking according to the absolute z-score achieved for each compound

Parameter				2,4-D (foll. alk. hydr.)	*2,4-D (free acid, OPT.)*	Dicamba (free acid)	*Dicamba (foll. alk. hydr. OPT.)*	Chlor-mequat	Top 10 scores	
Labs Reporting Results				33	*31*	21	*15*	38	all	only compulsory compounds
Lab-Code	NRL-SRM	Cate-gory*	No of Analytes sought for / correctly found	Ranking position	*Ranking position*	Ranking position	*Ranking position*	Ranking position		
001	Yes	A	14 / 5	16	*21*	**4**	***4***	18	2	1
003	Yes	A	15 / 5	25	***2***	**7**		35	2	1
005		B	4 / 1					**3**	1	1
006	Yes	B	12 / 3	**10**	*13*	**10**			2	2
007	Yes	B	4 / 2	30				**1**	1	1
008		B	2 / 1					**7**	1	1
009	Yes	B	8 / 2	17				33	0	0
014		B	3 / 2		*28*			**4**	1	1
017	Yes	B	14 / 3	11	*17*			38	0	0
018		B	9 / 3	**6**			***3***	19	2	1
019		A	19 / 5	18	*24*	15	***9***	12	1	0
021		B	8 / 2		***10***			16	1	0
022		A	16 / 6	13	*20*	16	*11*	**5**	1	1
024	Yes	B	7 / 1		*31*				0	0
026		B	4 / 1	33 (False Neg.)				**9**	1	1
029		A	17 / 6	22	*26*	14	***7***	17	1	0
030	Yes	B	2 / 1					**2**	1	1
031		B	16 / 5	**4**	***6***	**9**	***10***		4	2
032	Yes	B	11 / 1	26					0	0
033		B	10 / 2	14				14	0	0
034		A	19 / 3	20	***3***	19 (False Neg.)	*14 (ND)*	30	1	0
037		A	21 / 7	**7**	*19*	**1**	*12*	**8**	3	3
038		B	5 / 3		***9***	**6**		36	2	1
040	Yes	A	20 / 6	**1**	***4***	**2**	***5***	15	4	2
042		B	1 / 1					**10**	1	1
043	Yes	B	8 / 2		*14*	19 (False Neg.)		23	0	0
044		A	14 / 4	**5**	***7***	17		20	2	1
046	Yes	B	10 / 2		*15*			**6**	1	1
048	Yes	A	13 / 4	29	*30*	18		11	0	0
049		A	19 / 5	**2**	***1***	13	***1***	25	3	1
050		A	20 / 6	19	*25*	21	*13*	26	0	0
053		B	6 / 2		*29*			21	0	0
054		A	21 / 5	**8**	*11*	11	***8***	22	2	1
055		B	13 / 3		*16*	**3**		31	1	1
058	Yes	B	4 / 1	12					0	0
060	Yes	B	11 / 3	28	*23*			34	0	0
061	Yes	B	15 / 5	**3**	*12*	**5**	***6***	29	3	2
063	Yes	A	18 / 6	23	***5***	**8**		27	2	1
082	Yes	B	9 / 2	21			***2***		1	0

Parameter				2,4-D (foll. alk. hydr.)	*2,4-D (free acid, OPT.)*	Dicamba (free acid)	*Dicamba (foll. alk. hydr. OPT.)*	Chlor-mequat	Top 10 scores	
Labs Reporting Results				33	*31*	21	*15*	38	all	only compulsory compounds
Lab-Code	NRL-SRM	Cate-gory*	No of Analytes sought for / correctly found	Ranking position	*Ranking position*	Ranking position	*Ranking position*	Ranking position		
084	Yes	B	17 / 2	15				24	0	0
086	Yes	B	13 / 3		*22*	12		32	0	0
088	Yes	B	2 / 1		*27*				0	0
091		B	14 / 2	24				13	0	0
095		B	5 /2	**9**				37	1	1
117		B	2 / 2					28	0	0
118		B	7 / 1	32			*14 (ND)*		0	0
130		B	14 / 2	27	***8***				1	0
140		B	2 / 2	31	*18*				0	0

***** Laboratories that analysed all the pesticides assigned with an asterisk in the potencial pesticides list (compulsory compounds) and reported positive results for at least of 2,4-D (foll. akl. hydr.), dicamba (free acid) and chlormequat were included in Category A. All other laborytories were clsssified in Category B.

4.3.2 Comparison of NRL-SRMs with other OfLs

Table 27 shows a comparison between the mean number of parameters sought for by NRL-SRMs compared to the parameters submitted by other official laboratories (OfLs). No significant differences were apparent between these two laboratory groups as regards the scope of the laboratories.

Table 28 shows for each of the compounds the absolute z-scores achieved by NRL-SRMs on average compared to those achieved by the other labs. Here also there is no significant trend for one group performing better than the other.

Table 27 Number analytes sought for by labs

	No of Labs (submitted SRM-results)	Mean no of parameters sought for (21 parameters on SRM potential list)
NRL-SRM	21	11.10 (52.8 %)
Not NRL-SRM	27	10.59 (50.4 %)
all labs	**48**	**10.81 (51.5 %)**

Table 28 Comparisson of the medians and mean abs. z-scores achieved by NRL-SRM labs compared to other participating labs*

Parameter	No of results		Mean abs. z-score		
	NRL-SRMs	Not NRL-SRMs	NRL-SRMs	Not NRL-SRMs	Overall
2,4-D (foll. alk. hydr.)	16	17	1.09	**0.89**	0.99
2,4-D (free acid, OPT.)	13	18	0.78	**0.64**	0.70
Dicamba (free acid)	9	12	**0.82**	1.28	1.20
Dicamba (foll. alk. hydr., OPT.)	5	10	**0.38**	1.25	0.88
Chlormequat	15	23	0.94	**0.60**	0.74
Ethephon **	*2*	*5*	-	-	-
Glyphosate **	*3*	*6*	-	-	-

* Results reported as ND (not detected) were not included for the calculation of the mean absolute z-score. z-Scores of > 5 were lowered to 5 (this applied in only one case for a z-score = 12.34).

** Number of results not sufficient for statistical evaluation.

4.4 Analytical methods used for SMR pesticides

Detailed information about the analytical methods used by the laboratories for the various SRM-analytes can be found in **Appendix 13**.

In the case of chlormequat, 33 out of the 38 laboratories that analysed this compound used methanol for extraction. 20 laboratories stated that they have used isotopically labelled chlormequat as internal standard, 10 laboratories stated that they did not use it, and 3 did not give any answer. The determinative step was performed by LC-MS/MS in all cases. 12 of the labs indicated that they were not accredited for this compound.

In the case of 2,4-D (following alkaline hydrolysis) 32 laboratories have submitted method information. One laboratory reporting ND did not submit any information. 26 of the laboratories used acetonitrile for extraction, employing in almost all cases, variations of the QuEChERS-method. Another 4 laboratories reported the use of methanol for extraction and further 3 labs used of other solvents. In the determinative step 29 laboratories employed LC-MS/MS and 3 laboratories GC-MSD. All but four of the laboratories indicated reporting levels equal to, or below, the MRRL.

In the case of the analysis of 2,4-D (as the free acid), which was optional, out of the 31 laboratories that have submitted results, 23 used acetonitrile for extraction, employing in almost every case, variations of the QuEChERS-method. Out of the remaining laboratories, 5 indicated that they used methanol-based methods, 2 laboratories methods employing other solvents and one laboratory did not provide any information in this respect. For the determination step, 27 laboratories employed LC-MS/MS, 3 laboratories GC-MSD or GC-ITD,and one laboratory did not give any details. All but three of these laboratories indicated reporting levels equal to, or below, the MRRL.

In the case of the analysis of dicamba (as the free acid) 21 laboratories submitted method information (including the two laboratories reporting ND). 15 of the laboratories used acetonitrile for extraction, employing in almost every case, variations of the QuEChERS-method. 4 laboratories employed methanol-based methods and 2 laboratories methods employing other solvents. 16 laboratories employed LC-MS/MS, 3 laboratories GC-MSD or GC-ITD, and 1 laboratory GC-MS/MS. 6 out of the 21 laboratories indicated reporting levels above the MRRL, and in 2 cases even above the level of the residue in the test material.

In the case of the analysis of dicamba (following alkaline hydrolysis), which was an optional, 14 laboratories submitted method information (including one of the two laboratories reporting ND). 10 of these labs reported the use of acetonitrile for extraction, employing in almost every case, variations of the QuEChERS-method. Out of the remaining laboratories, 3 indicated the use of methanol and 1 of other solvents. 11 laboratories employed LC-MS/MS, 3 laboratories GC-MSD. 2 out of the 14 laboratories indicated reporting levels above the MRRL.

The amounts of test material employed by the participants for the analysis of 2,4-D, dicamba and chlormequat ranged between 2 g and 50 g, for glyphosate between 3 g and 25 g and for ethephon between 1 g and 5 g. It should be noted that the homogeneity test was performed using 5 g sample portions in all cases. Higher sub-sampling variations are to be expected when smaller sample amounts are used.

Table 29 SRM-Results reported by the laboratories and the respective z-scores calculated using the FFP-RSD of 25 % (excluding ethephon and glyphosate)

Analyte			2,4-D (foll. alk. hydr.)		2,4-D (free acid, OPT.)		Dicamba (free acid)		Dicamba (foll. alk. hydr., OPT.)		Chlormequat		Ethephon		Glyphosate	
Median [mg/kg]			**0.499**		**0.471**		**0.106**		**0.264**		**0.479**		**0.473**		**1.17**	
MRRL [mg/kg]			**0.02**		**0.02**		**0.02**		**0.02**		**0.02**		**0.02**		**0.02**	
Qn RSD (%)			**27.5**		**22.6**		**30.2**		**26.8**		**25.8**		***(54.9)***		***(31.9)***	
Lab.- Code	NRL-SRM	No of Para-meters sought for / correctly found	Result [mg/kg]	z-scores (FFP RSD (25%))	Result [mg/kg]	z-scores (FFP RSD (25%))	Result [mg/kg]	z-scores (FFP RSD (25%))	Result [mg/kg]	z-scores (FFP RSD (25%))	Result [mg/kg]	z-scores (FFP RSD (25%))	Result [mg/kg]	z-scores (FFP RSD (25%))	Result [mg/kg]	z-scores (FFP RSD (25%))
eupt 001	yes	14 / 5	0.427	-0.6	0.373	-0.8	0.115	0.4	0.29	0.4	0.416	-0.5				
eupt 003	yes	15 / 5	0.321	-1.4	0.458	-0.1	0.0876	-0.7			0.26	-1.8			1.1	
eupt 005		4 / 1									0.47	-0.1				
eupt 006	yes	12 / 3	0.526	0.2	0.512	0.3	0.131	1.0								
eupt 007	yes	4 / 2	0.22	-2.2							0.48	0.0				
eupt 008		2 / 1									0.455	-0.2				
eupt 009	yes	8 / 2	0.58	0.7							0.645	1.4				
eupt 014		3 / 2			0.288	-1.6					0.493	0.1				
eupt 017	yes	14 / 3	0.52	0.2	0.407	-0.5					0.108	-3.1				
eupt 018		9 / 3	0.516	0.1					0.245	-0.3	0.538	0.5				
eupt 019		19 / 5	0.4	-0.8	0.346	-1.1	0.146	1.5	0.201	-1.0	0.437	-0.3				
eupt 021		8 / 2			0.5	0.2					0.43	-0.4				
eupt 022		16 / 6	0.54	0.3	0.555	0.7	0.16	2.1	0.35	1.3	0.495	0.1			1.43	
eupt 024	yes	7 / 1			0.13	-2.9										
eupt 026		4 / 1	ND	-3.8							0.5	0.2				
eupt 029		17 / 6	0.372	-1.0	0.309	-1.4	0.071	-1.3	0.219	-0.7	0.43	-0.4	0.79			
eupt 030	yes	2 / 1									0.477	0.0				
eupt 031		16 / 5	0.491	-0.1	0.477	0.1	0.085	-0.8	0.197	-1.0					1.24	
eupt 032	yes	11 / 1	0.29	-1.7												
eupt 033		10 / 2	0.533	0.3							0.51	0.3				

Analyte			2,4-D (foll. alk. hydr.)		2,4-D (free acid, OPT.)		Dicamba (free acid)		Dicamba (foll. alk. hydr., OPT.)		Chlormequat		Ethephon		Glyphosate	
Median [mg/kg]			**0.499**		**0.471**		**0.106**		**0.264**		**0.479**		**0.473**		**1.17**	
MRRL [mg/kg]			**0.02**		**0.02**		**0.02**		**0.02**		**0.02**		**0.02**		**0.02**	
Qn RSD (%)			**27.5**		**22.6**		**30.2**		**26.8**		**25.8**		***(54.9)***		***(31.9)***	
Lab.- Code	NRL-SRM	No of Para-meters sought for / correctly found	Result [mg/kg]	z-scores (FFP RSD (25%))	Result [mg/kg]	z-scores (FFP RSD (25%))	Result [mg/kg]	z-scores (FFP RSD (25%))	Result [mg/kg]	z-scores (FFP RSD (25%))	Result [mg/kg]	z-scores (FFP RSD (25%))	Result [mg/kg]	z-scores (FFP RSD (25%))	Result [mg/kg]	z-scores (FFP RSD (25%))
eupt 034		19 / 3	0.606	0.9	0.458	-0.1	ND	-3.2	ND	-3.7	0.634	1.3				
eupt 037		21 / 7	0.516	0.1	0.406	-0.6	0.096	-0.4	0.098	-2.5	0.449	-0.2	0.396		1.05	
eupt 038		5 / 3			0.449	-0.2	0.0892	-0.6			0.25	-1.9				
eupt 040	yes	20 / 6	0.502	0.0	0.461	-0.1	0.094	-0.4	0.23	-0.5	0.511	0.3			1.51	
eupt 042		1 / 1									0.5	0.2				
eupt 043	yes	8 / 2			0.522	0.4	ND	-3.2			0.565	0.7				
eupt 044		14 / 4	0.487	-0.1	0.486	0.1	0.162	2.1			0.54	0.5				
eupt 046	yes	10 / 2			0.519	0.4					0.485	0.1				
eupt 048	yes	13 / 4	0.244	-2.0	0.195	-2.3	0.176	2.7			0.505	0.2				
eupt 049		19 / 5	0.495	0.0	0.471	0.0	0.136	1.2	0.264	0.0	0.576	0.8				
eupt 050		20 / 6	0.6	0.8	0.6	1.1	0.431	12.3	0.431	2.5	0.58	0.8	0.55			
eupt 053		6 / 2			0.673	1.7					0.536	0.5				
eupt 054		21 / 5	0.471	-0.2	0.496	0.2	0.133	1.0	0.313	0.7	0.402	-0.6	ND		ND	
eupt 055		13 / 3			0.513	0.4	0.096	-0.4			0.314	-1.4	ND		ND	
eupt 058	yes	4 / 1	0.462	-0.3												
eupt 060	yes	11 / 3	0.74	1.9	0.583	1.0					0.283	-1.6				
eupt 061	yes	15 / 5	0.494	0.0	0.436	-0.3	0.091	-0.5	0.3	0.5	0.324	-1.3				
eupt 063	yes	18 / 6	0.644	1.2	0.458	-0.1	0.086	-0.7			0.592	0.9	0.23		0.575	
eupt 082	yes	9 / 2	0.612	0.9					0.27	0.1						
eupt 084	yes	17 / 2	0.447	-0.4							0.377	-0.8				
eupt 086	yes	13 / 3			0.575	0.9	0.131	1.0			0.315	-1.4	ND		ND	

Analyte			2,4-D (foll. alk. hydr.)		2,4-D (free acid, OPT.)		Dicamba (free acid)		Dicamba (foll. alk. hydr., OPT.)		Chlormequat		Ethephon		Glyphosate	
Median [mg/kg]			**0.499**		**0.471**		**0.106**		**0.264**		**0.479**		**0.473**		**1.17**	
MRRL [mg/kg]			**0.02**		**0.02**		**0.02**		**0.02**		**0.02**		**0.02**		**0.02**	
Qn RSD (%)			**27.5**		**22.6**		**30.2**		**26.8**		**25.8**		***(54.9)***		***(31.9)***	
Lab.- Code	NRL-SRM	No of Para-meters sought for / correctly found	Result [mg/kg]	z-scores (FFP RSD (25%))	Result [mg/kg]	z-scores (FFP RSD (25%))	Result [mg/kg]	z-scores (FFP RSD (25%))	Result [mg/kg]	z-scores (FFP RSD (25%))	Result [mg/kg]	z-scores (FFP RSD (25%))	Result [mg/kg]	z-scores (FFP RSD (25%))	Result [mg/kg]	z-scores (FFP RSD (25%))
eupt 088		2 / 1			0.292	-1.5										
eupt 091		14 / 2	0.648	1.2							0.443	-0.3				
eupt 095		5 /2	0.47	-0.2							0.25	-1.9				
eupt 117		2 / 2									0.59	0.9				
eupt 118	yes	7 / 1	0.025	-3.8					ND	-3.7						
eupt 130		14 / 2	0.708	1.7	0.48	0.1										
eupt 140		2 / 2	0.93	3.5	0.526	0.5										

Note: Only laboratories that analysed at least one of the SRM pesticides have been included in this table.

4.5 Summary, Conclusions and Prospects for the SRM pesticides

The EUPT-C3/SRM4 was the fourth PT focusing on single residue compounds and the second to be organized collaboratively between the CRL for Single Residue Methods and the CRL for Cereals and Feedingstuffs.

In total 111 laboratories registered for this PT with 103 of them submitting results. Out of these laboratories 48, representing 25 countries (23 EU-MS plus Switzerland and Norway), submitted at least one result for SRM-compounds. This is a clear increase compared to the EUPT-C1/SRM2 in 2007 (the previous PT for cereals), where only 30 laboratories from 18 countries (16 EU-MS) submitted results. The increased use of LC-MS/MS instrumentation, is probably one of the main reasons for this trend. In the other two other EUPT-SRMs focusing on fruits and vegetables the laboratories submitting results were 24 from 14 countries (13 EU-MS) at the EUPT-SRM1 (in 2006) and 66 from 25 countries (23 EU-MS) at the EUPT-SRM3 (in 2008). In the latter case the high participation was related to the inclusion of dithiocarbamates in the scope. Dithiocarbamates are analysed by a high percentage of laboratories using methods that do not involve the use of LC-MS technology.

Among the EU-MSs Spain, Romania, Portugal, Poland, Malta and Luxemburg did not participate with any of their laboratories in this PT, with the latter two having delegated their obligations to laboratories in the UK and Belgium, respectively. Among the NRL-SRMs not submitting results were those of the above countries as well as those of Denmark and France, with the NRL-SRM for Denmark being a co-organiser of this test and thus not participating. Spain and France had no NRL-SRM appointed at the time the PT was undertaken.

The potential pesticide list distributed to the laboratories prior to the test contained in total 21 SRM-compounds, 8 of them, regarding acidic pesticides, being optional. The test material itself contained 5 pesticides namely; chlormequat, ethephon, glyphosate, 2,4-D and dicamba, with the latter two corresponding to two analytical procedures each, a compulsory and an optional one. All of the pesticides contained in the sample were applied in the field, with 2,4-D being additionally applied in the laboratory after being non-detectable in the harvested crop.

For each laboratory/pesticide combination, z-scores based on the FFP-RSD of 25 % have been calculated and a classification into ‘acceptable’, ‘questionable’ and ‘unacceptable’ was undertaken. Overall the quality of the results was good with 37 out of 38 laboratories (97 %) reporting results within the acceptable range in the case of chlormequat. 25 out of 29 (88 %) in the case of 2,4-D (following alkaline hydrolysis), and 9 out of 16 (72 %) in the case of dicamba (free acid). For 2,4-D (free acid) and dicamba (following alkaline hydrolysis) the numbers of laboratories reporting acceptable results were 27 out of 29 (94 %) in the first case and 13 out of 15 (74 %) in the latter. For ethephon and glyphosate the number of laboratories submitting results was quite low with just 6 laboratories reporting results for glyphosate and just 4 for ethephon.

No false positives were reported but the number of false negatives was quite high: 1 in the case of 2,4-D (following alkaline hydrolysis), 2 in the case of dicamba (free acid), and 3 for both ethephon and glyphosate. 2 laboratories reported a false negative result in the case of “dicamba (following alkaline hydrolysis)”.

The distribution of the results in this EUPT was comparable to previous EUPTs with the robust RSD (Qn-RSD) ranging between 22.6 and 30.2 (26.6 on average) and thus close to the FFP-RSD of 25 %. This justifies once more the use of the generic 25 % value also in the case of SRM-analytes. The Qn-RSD for ethephon and glyphosate were excluded from this calculation due to the small population of results.

Overall, the scope of the laboratories has improved compared to the previous PT on cereals. “2,4-D (following alk. hydr.)” and “dicamba (free acid)” were analysed by 32 and 21 labs, respectively, compared to 19 labs and 18 labs that analysed MCPA, and MCPP in the EUPT-C1/SRM2. A clear improvement can also be observed in the case of chlormequat, where the number of laboratories reporting results has been increasing from 23 for EUPT-SRM1 to 25 for EUPT-C1/SRM2 and to 38 for the present PT. In the case of glyphosate and ethephon, both included in the multiannual EU-coordinated control programme, the participation of the laboratories was very poor and has to be clearly improved in the future.

Laboratories were ranked by based on their overall performance (average of absolute z-scores) as well as their scope, especially considering the pesticides marked with an asterisk in the potential pesticide list. In total 15 laboratories were included in Category A with 7 of them being NRL-SRMs. 10 of the Category A labs were classified as “Good” (SWZ < 2), two laboratories as “Satisfactory” (SWZ 2-3) and another three as “Unsatisfactory” (SWZ > 3). All the other 33 laboratories (including 15 NRL-SRMs) were included in Category B because of insufficient scope. In general there was no clear trend showing that NRL-SRMs performed better than the other laboratories either in terms of scope, nor as regards the accuracy of their results as reflected by the z-scores. It should be noted that the NRLs of certain countries (MT, LU, SE) have delegated all or part of their SRM-analyses to sub-contacted laboratories.

Concerning the release of bound residues by alkaline hydrolysis, the study has continued in this PT with the main aim of supporting any future attempts and discussions to harmonize the residue definitions for acidic pesticides. A ca. 2.3-fold increase of the median value for dicamba (following alkaline hydrolysis) compared to the dicamba free acid was observed, which indicates that (roughly 56 % of the field-sprayed residue was bound. In the case of 2,4-D, which was added post-harvest, the increase of the median following alkaline hydrolysis was around 10 %. In comparison, at the EUPT-C1/SRM2 the increment factor for MCPA was 7.1 indicating that 86 % of the total residue was in a bound form.

In order to encourage laboratories to increase their scope and decrease their reporting levels the Scientific Committee strongly recommends laboratories to be equipped with LC-MS/MS, as most of the SRM-pesticides can only be analysed routinely by liquid chromatography. The aim is that laboratories continue to increase their scope of analytes in their methods in order to be able to fully enforce EU legislation and to improve their overall performance, both in terms of correctly detecting the pesticides present as well as determining the residue levels accurately.

The CRL-SRM will further continue developing, validating and distributing methodologies for compounds not amenable to multiresidue methods that are simple-to-use, fast and cheap.

In future PTs, the selection of pesticides will continue to focus on pesticides included in the scope of the monitoring programmes as well as additional pesticides of relevance.

5. ACKNOWLEDGEMENTS

The Organisers wishes to thank the members of the Scientific Committtee (Quality Control Group and Advisory Group) for their valuable advice.

6. REFERENCES

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[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] Thompson M. and Wood R., Harmonized Guideline for Internal Quality Control in Analytical Chemistry Laboratories. Pure & Appl. Chem., Vol.67, No. 4, pp. 649-666, 1995

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APPENDICES

Appendix 1 List of Laboratories registered to participate in the EUPT-C3/SRM4

Country	Institution	City	NRL-CF	NRL-SRM	Reported results	
					MRM	SRM
Austria	Austrian Agency for Health and Food Safety, Competence Centre for Residue Analysis	Vienna	X		Yes	Yes
Austria	AGES CC PSMR Innsbruck	Innsbruck		X	Yes	Yes
Belgium	Fytolab	Zwijnaarde			Yes	Yes
Belgium	Scientific Institute of Public Health, FOOD	Bruxelles	X	X	Yes	Yes
Bulgaria	Plant Protection Institute, Toxicology		X		No	No
Bulgaria	Central Laboratory for Chemical Testing and Control, Chemical and Biological Contaminants	Sofia		X	Yes	Yes
Bulgaria	Regional Inspectorate for Public Health Protection and Control - Pleven	Pleven			Yes	No
Bulgaria	Regional Inspectorate for Public Health Protection and Control	Veliko Tarnovo			Yes	No
Bulgaria	Regional Inspectorate for Public Healt and Control, Directorate Laboratory Analyses	Sofia			Yes	No
Bulgaria	RIOKOZ - Burgas, Chemical and physical Investigation	Burgas			No	No
Cyprus	State General Laboratory	Nicosia	X	X	Yes	Yes
Czech Republic	Czech Agriculture and Food Inspection Authority, Laboratory Department	Prague	X	X	Yes	Yes
Czech Republic	Central Institute for Supervising and Testing in Agriculture, UKZUZ-NRL	Brno	X		Yes	Yes
Czech Republic	Institute of Chemical Technology Prague, Department of Food Chemistry and Analysis	Prague			Yes	Yes
Denmark	Danish Veterinary and Food Adminstration, Region East	Ringsted			Yes	Yes
Denmark	The Danish Plant Directorate	Lyngby	X		Yes	Yes
Estonia	AGRICULTURAL RESEARCH CENTRE,	Saku	X		Yes	Yes
Estonia	Health Protection Inspectorate	Tartu		X	Yes	Yes
Finland	Finnish Customs Laboratory	Espoo	X	X	Yes	Yes
France	Laboratoire du SCL de Montpellier	Montpellier			Yes	Yes
France	SCL laboratoire d'Ile de France Massy, Residus de Pesticides	Massy Cedex	X		Yes	Yes
France	Laboratoire SCL, Pesticides	Rennes			No	No
France	LDA 56, Chimie	Saint-Ave			Yes	Yes
Germany	LUFA Speyer, Rueckstandsanalytik	Speyer			Yes	No
Germany	LLFG, Agricultural Analysis	Halle			Yes	No
Germany	Betriebsgesellschaft für Umwelt und Landwirtschaft, GB 6 - Labore Landwirtschaft	Leipzig			Yes	Yes
Germany	Landesamt für Verbraucherschutz, Sachsen-Anhalt	Halle/Saale			Yes	Yes
Germany	Federal Office of Consumer Protection and Food Safety (BVL), Unit 501	Berlin	X	X	Yes	Yes
Germany	CVUA Muenster, Dezernat 51	Münster			Yes	Yes
Germany	Landesamt für Landwirtschaft, Lebensmittelsicherheit	Rostock			Yes	Yes

Country	Institution	City	NRL-CF	NRL-SRM	Reported results	
					MRM	SRM
	und Fischerei Mecklenburg-Vorpommern,					
Germany	Niedersaechsisches Landesamt fuer Verbraucher-schutz und Lebensmittelsicherheit	Oldenburg			Yes	Yes
Germany	Landesuntersuchungsamt Speyer	Speyer			Yes	Yes
Germany	Thüringer Landesanstalt für Landwirtschaft (TLL),	Jena			Yes	Yes
Germany	Amt fuer Umwelt, Verbraucherschutz und Lokale Agenda der Stadt Bonn, Amtliche Lebensmittel-untersuchung	Bonn			Yes	No
Germany	Bayerisches Landesamt für Gesundheit und Lebens-mittelsicherheit	Erlangen			Yes	Yes
Germany	Landeslabor Brandenburg, Fachbereich L4	Frankfurt (Oder)			Yes	Yes
Germany	Bayerisches Landesamt für Gesundheit und Lebens-mittelsicherheit, Dienststelle Oberschleissheim	Oberschleiss-heim			Yes	No
Germany	Institut fuer Hygiene und Umwelt, Residues and Contaminants	Hamburg			Yes	Yes
Germany	Landwirtschaftliches Technologiezentrum Augusten-berg , Abteilung 2: Chemische Analysen, Saatgut- und Futtermitteluntersuchungen	Augustenberg			No	No
Greece	Benaki Phytopathological Institute,	Athens	X	X	Yes	Yes
Greece	Ministry of Rural Development and Food, Centre for Crop Protection & Quality Control of Ioannina	Ioannina			Yes	No
Greece	General Chemical State Laboratory, D Division	Athens	X	X	Yes	Yes
Hungary	Agricultural Office of County Fejer, PPSCD	Velence	X		Yes	Yes
Hungary	Agricultural Office of Somogy County, Plant Protection and Soil Conservation Directorate	Kaposvár			Yes	Yes
Hungary	Aggricultural Office of B.-A.-Z. County, Plant Protection and Soil Conservation Directorate	Miskolc		X	Yes	Yes
Ireland	Department of Agriculture, Food and Fisheries	Kildare	X	X	Yes	Yes
Italy	National Institute of Health, Environmental and Primary Prevention - Pesticide section	Rome		X	Yes	Yes
Italy	Istituto Zooprofilattico Sperimentale Lazio e Toscana sede di Roma, Direzione Operativa Chimica	Roma			Yes	No
Italy	ARPA SICILIA, AUSL n.7 Ragusa,	Ragusa Sicilia			Yes	Yes
Italy	APPA TRENTO, Settore Laboratorio e Controlli	Trento			Yes	No
Italy	ARPA MARCHE, MACERATA	Villapotenza-MACERATA			Yes	No
Italy	ARPA FRIULI VENEZIA GIULIA, PORDENONE	Portenone			Yes	No
Italy	ARPA Puglia, Dipartimento di Bari	Bari			Yes	No
Italy	ARPA-VENETO, DIP. REG. LABORATORI	Verona			Yes	No
Italy	ASL PROVINCIA DI BERGAMO, DIPARTIMENTO DI PREVENZIONE	Bergamo			Yes	No
Italy	ARPA Umbria, Perugia	Perugia			Yes	No
Italy	Istituto Zooprofilattico Sperimentale del Piemonte, Liguria e Valle d'Aosta, CreAA	Torino	X		No	No
Italy	ARPA Lombardia, Dipartimento provinciale di Como	Como			Yes	No
Italy	ISTITUTO ZOOPROFILATTICO SPERIMENTALE DELL'ABRUZZO E DEL MOLISE "G. CAPORALE",	Teramo			Yes	No

Country	Institution	City	NRL-CF	NRL-SRM	Reported results	
					MRM	SRM
	FOOD AND FEED HYGIENE					
Italy	ARPA, RAR - Riferimento Analitico Regionale	Chiesuol del Fosso (Ferrara)			Yes	No
Italy	ARPA Bolzano	Bolzano			Yes	Yes
Italy	Agenzia Regionale per la protezione , Dipartimento di Livorno	Livorno			No	No
Latvia	National Diagnostic Centre	Riga	X	X	Yes	Yes
Lithuania	National food and veterinary risk assessent institute, Laboratory department	Vilnius	X	X	Yes	Yes
Malta	Public Health Laboratory, Department for Environmental Health	Portenone	X	X	–*	–*
Norway	Bioforsk, Plant Health and Plant Protection	Aas	X	X	Yes	Yes
Poland	Institute of Plant Protection, Experimental Station in Rzeszow	Rzeszow			Yes	No
Poland	Wojewodzka Stacja Sanitarno-Epidemiologiczna w Opolu	Mickiewicza			Yes	No
Poland	Instytut Ochrony Roślin. Państwowy Instytut Badawczy. Terenowa Stacja Doświadczalna	Trzebnica			Yes	No
Poland	Voivodship Veterinary Inspection, Department of Pesticide Residues	Warszawa			Yes	No
Poland	Wojewodzki Inspektorat Weterynarii, Zakład Higieny Weterynaryjnej	Poznan			Yes	No
Poland	Wojewodzki Inspektorat Weterynarii, Opole	Opole			Yes	No
Poland	Research Institute of Pomology & Floriculture,	Skierniewice			Yes	No
Poland	Wojewódzka Stacja Sanitarno-Epidemiologiczna	Gdaśsk			Yes	No
Poland	The Institute of Plant Protection, Department of Pesticide Residue Research	Poznan	X		Yes	No
Poland	Voivodship Veterinary Inspection, Department of Pesticide Residues				Yes	No
Poland	Wojewodzka Stacja Sanitarno-Epidemiologiczna w Krakowie, Dzial Laboratoryjny WSSE	Krakowie			Yes	No
Poland	Wojewodzki Inspektorat Weterynarii, ZHW Wroclaw				Yes	No
Poland	Wojewodzka Stacja Sanitarno Epidemiologiczna W Rzeszowie	Rzeszowie			Yes	No
Poland	Institute of Plant Protection - National Research Institute	Sosnicowice			Yes	No
Poland	Instytut Ochrony Roslin Panstwowy Instytut Badawczy	Trzebnica			Yes	No
Portugal	Direcção Regional Agrcultura Pescas Norte, Div.Exp.Qualificção e Apoio Laboratorial	Senhora da Hora			Yes	No
Portugal	INRB-L-INIA, Plant Protection Research Unit		X	X	Yes	No
Portugal	Laboratório Regional de Veterinária e Segurança Alimentar - Divisão de Análise de Resíduos	Oeiras			Yes	No
Romania	Central Laboratory for Pesticides Residues Control	Bucharest	X		Yes	No
Romania	Sanitary Veterinary and Food Safety Directorate, Residues Departament	Bucharest			Yes	No
Romania	Sanitary Veterinary and Food Safety Laboratory Bucharest	Bucharest		X	Yes	No
Slovakia	State Veterinary and Food Institute, chromatography	Bratislava	X	X	Yes	Yes

Country	Institution	City	NRL-CF	NRL-SRM	Reported results	
					MRM	SRM
Slovakia	Public health authority of the Slovak republic, Department for Materialization of Living Condition	Bratislava			Yes	No
Slovenia	Institute of Public Health Maribor, Environmental Protection Institute	Maribor		X	Yes	Yes
Slovenia	Institute of public health	Ljubljana	X		Yes	Yes
Slovenia	Kmetijski inštitut Slovenije, Agricultural Institute of Slovenia	Ljubljana	X		Yes	No
Spain	Instituto Tecnologico de Canarias, Analisis Ambiental	Las Palmas			Yes	No
Spain	LABORATORIO AGRARIO REGIONAL, RESIDUOS DE PESTICIDAS	Albacete			Yes	No
Spain	Junta de Castilla y León, Dirección General de Producción Agropecuaria	Burgos			Yes	No
Spain	GOBIERNO DE ARAGÓN, AGRICULTURA Y ALIMENTACIÓN	Zaragoza			Yes	No
Spain	LABORATORI AGROALIMENTARI - DAR, Generalitat de Catalunya	Cabrils (Barcelona)			Yes	No
Spain	Servicio de Laboratorio y Control, Análisis Instrumental	Santander			Yes	No
Spain	A.E.S.A.N. (C.N.A.)	Majadahonda	X		Yes	No
Spain	NASERSA, CROMATOGRAFIA	Villava			Yes	No
Spain	Laboratorio Arbitral Agroalimentario, Pesticides Residues	Madrid	X		Yes	No
Spain	INGACAL, LABORATORIO AGRARIO	La Coruña			No	No
Sweden	National Food Administration, Research and Development	Uppsala	X	X	Yes	Yes
Sweden	Eurofins Food/Agro Sweden AB **	Lidköping		(X)	Yes	Yes
Switzerland	Kantonales Labor Zürich, Abteilung Pestizide	Zurich			Yes	Yes
The Netherlands	RIKILT Institute of Food Safety	Wageningen	X		Yes	No
The Netherlands	VWA - Food and Consumer Product Safety Authority, NRL for Pesticides in Food	Amsterdam	X	X	Yes	Yes
United Kingdom	Central Science Laboratory, Food Science Group	York	X	X	Yes	Yes

* Represented by NRL-SRM of UK

** This lab was subcontracted by the NRL-SRM of Sweden to analyze part of the SRM-pesticides

MRM APPENDICES

Appendix 2 Target- Pesticide List of MRM-pesticides

MRM-Parameter	MRRL [mg/kg]
* Alpha-cypermethrin	0.02
* Azoxystrobin	0.02
* Bifenthrin	0.02
Carbaryl	0.02
* Carbendazim	0.02
Chlormequat (expressed as cation)	0.02
Chlorothanlonil	0.02
* Chlorpyrifos	0.02
* Chlorpyrifos-methyl	0.02
* Cypermethrin	0.02
* Cyproconazole	0.02
* Deltamethrin	0.02
Diazinon	0.02
* Difenoconazole	0.02
*Endosulfan (α + ß + Sulphate expressed as Endosulfan)	0.02
- Endosulfan α	0.02
- Endosulfan β	0.02
- Endosulfan sulphate	0.02
*Epoxiconazole	0.02
* Fenhexamid	0.02
* Fenpropimorph	0.02
* Fluquinconazole	0.02
Glyphosate	0.02
* Imazalil	0.02
* Iprodione	0.02
* Kresoxim-methyl	0.02
* Lambda-cyhalothrin	0.02
* Lindane (gamma-HCH)	0.02
* Malathion (Malathion + Malaoxon,expressed as Malathion)	0.02
* Malathion	0.02
Malaoxon	0.02
Mepiquat (expressed as cation)	0.02
Metconazole	0.02
* Methacrifos	0.02
* Methomyl (Methomyl + Thiodicarb,expressed as Methomyl)	0.02
* Methomyl	0.02
* Thiodicarb	0.02
* Parathion (only parent compound)	0.02
* Penconazole	0.02
* Permethrin	0.02

* Pirimicarb	0.02
* Pirimiphos-methyl	0.02
* Prochloraz (only parent compound)	0.02
* Procymidone	0.02
* Propiconazole	0.02
* Spiroxamine	0.02
* Tebuconazole	0.02
* Thiabendazole	0.02
* Thiophanate-methyl	0.02
Triadimefon (Triadimefon + Triadimenol express. as Triadimefon)	0.02
Triadimefon	0.02
Triadimenol	0.02
Triazophos	0.02
* Trifloxystrobin	0.02
Vinclozolin (only parent compound)	0.02

Appendix 3 Homogeneity data of MRM-pesticides

Azoxystrobin [mg/kg]		
Sample	Portion 1	Portion 2
2	0.209	0.206
4	0.216	0.227
23	0.230	0.211
32	0.210	0.208
56	0.222	0.250
82	0.225	0.226
94	0.203	0.207
121	0.193	0.208
168	0.196	0.199
200	0.218	0.211

Carbendazim [mg/kg]		
Sample	Portion 1	Portion 2
2	0.486	0.483
4	0.532	0.509
23	0.495	0.528
32	0.473	0.492
56	0.462	0.464
82	0.440	0.448
94	0.455	0.471
121	0.500	0.510
168	0.466	0.471
200	0.436	0.459

Chlorpyriphos [mg/kg]		
Sample	Portion 1	Portion 2
2	1.254	1.236
4	1.251	1.351
23	1.344	1.348
32	1.292	1.276
56	1.357	1.240
82	1.237	1.320
94	1.225	1.322
121	1.183	1.230
168	1.241	1.227
200	1.225	1.218

Cyproconazole [mg/kg]		
Sample	Portion 1	Portion 2
2	0.524	0.486
4	0.516	0.554
23	0.540	0.564
32	0.515	0.507
56	0.555	0.521
82	0.530	0.521
94	0.491	0.561
121	0.477	0.508
168	0.489	0.486
200	0.533	0.488

Cyprodinil [mg/kg]		
Sample	Portion 1	Portion 2
2	0.088	0.082
4	0.084	0.089
23	0.092	0.087
32	0.087	0.084
56	0.088	0.086
82	0.092	0.091
94	0.082	0.083
121	0.076	0.084
168	0.075	0.079
200	0.079	0.080

Fenbuconazole [mg/kg]		
Sample	Portion 1	Portion 2
2	0.571	0.544
4	0.565	0.586
23	0.604	0.572
32	0.561	0.565
56	0.582	0.572
82	0.604	0.585
94	0.545	0.561
121	0.528	0.549
168	0.521	0.546
200	0.553	0.544

Fenpropimorph [mg/kg]		
Sample	Portion 1	Portion 2
2	0.149	0.151
4	0.155	0.149
23	0.158	0.151
32	0.154	0.155
56	0.151	0.159
82	0.153	0.157
94	0.148	0.151
121	0.139	0.145
168	0.143	0.149
200	0.141	0.147

Fenvalerate (sum) [mg/kg]		
Sample	Portion 1	Portion 2
2	0.167	0.162
4	0.170	0.192
23	0.186	0.192
32	0.181	0.170
56	0.192	0.164
82	0.179	0.186
94	0.176	0.183
121	0.158	0.175
168	0.169	0.167
200	0.179	0.171

Fludioxonil [mg/kg]		
Sample	Portion 1	Portion 2
2	0.166	0.148
4	0.152	0.176
23	0.156	0.174
32	0.149	0.165
56	0.178	0.152
82	0.150	0.159
94	0.152	0.164
121	0.141	0.155
168	0.140	0.144
200	0.158	0.140

Flusilazole [mg/kg]		
Sample	Portion 1	Portion 2
2	0.909	0.872
4	0.915	0.935
23	0.958	0.945
32	0.903	0.906
56	0.928	0.912
82	0.985	0.946
94	0.884	0.932
121	0.846	0.893
168	0.841	0.862
200	0.905	0.875

Lambda-cyhalothrin [mg/kg]		
Sample	Portion 1	Portion 2
2	0.079	0.071
4	0.075	0.082
23	0.081	0.088
32	0.075	0.075
56	0.082	0.072
82	0.077	0.078
94	0.073	0.078
121	0.069	0.074
168	0.073	0.073
200	0.075	0.073

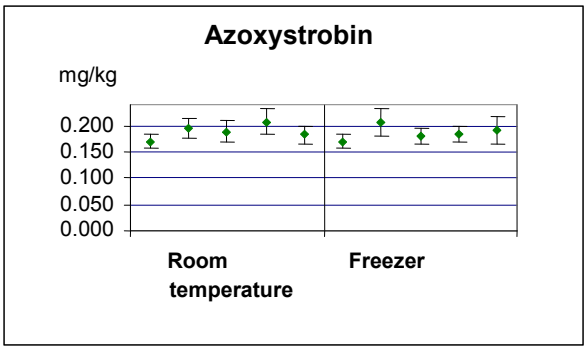
Metconazole [mg/kg]		
Sample	Portion 1	Portion 2
2	0.504	0.450
4	0.481	0.504
23	0.523	0.503
32	0.471	0.469
56	0.519	0.486
82	0.492	0.491
94	0.460	0.506
121	0.438	0.458
168	0.450	0.454
200	0.455	0.452

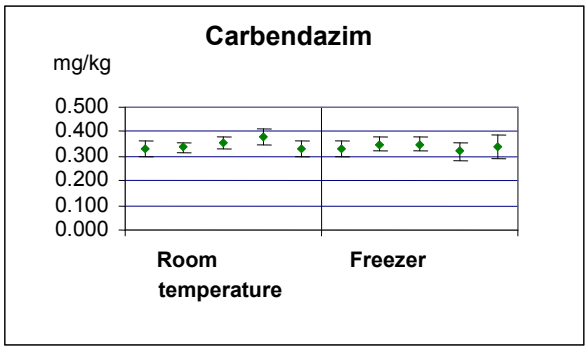
Pyraclostrobin [mg/kg]		
Sample	Portion 1	Portion 2
2	0.850	0.841
4	0.829	0.842
23	0.867	0.921
32	0.788	0.830
56	0.818	0.841
82	0.741	0.744
94	0.828	0.785
121	0.766	0.765
168	0.702	0.702
200	0.733	0.759

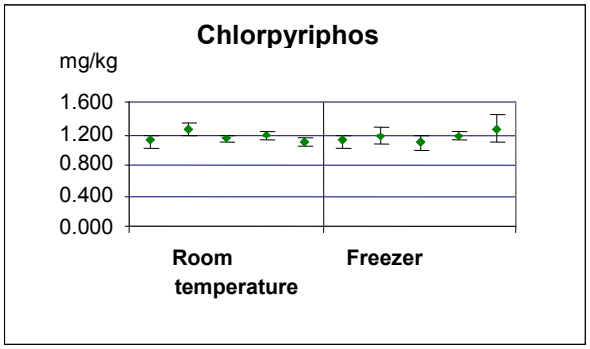
Tebuconazole [mg/kg]		
Sample	Portion 1	Portion 2
2	1.559	1.444
4	1.513	1.603
23	1.594	1.631
32	1.498	1.474
56	1.623	1.523
82	1.575	1.538
94	1.449	1.599
121	1.407	1.473
168	1.424	1.412
200	1.561	1.422

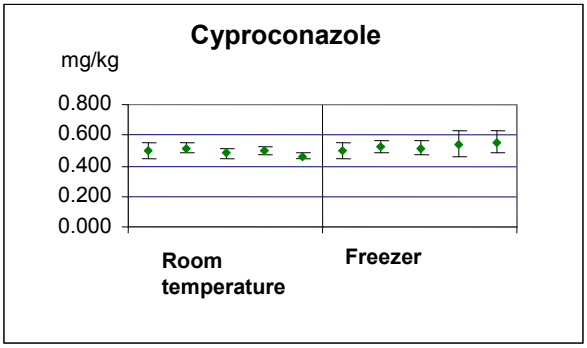
Malathion [mg/kg]		
Sample	Portion 1	Portion 2
2	0.019	0.017
4	0.019	0.020
23	0.021	0.021
32	0.020	0.020
56	0.018	0.018
82	0.021	0.021
94	0.018	0.019
121	0.018	0.018
168	0.019	0.018
200	0.017	0.019

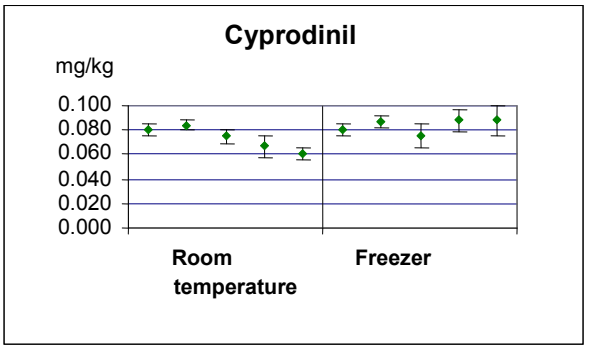
Appendix 4 Stability figures of MRM-pesticides

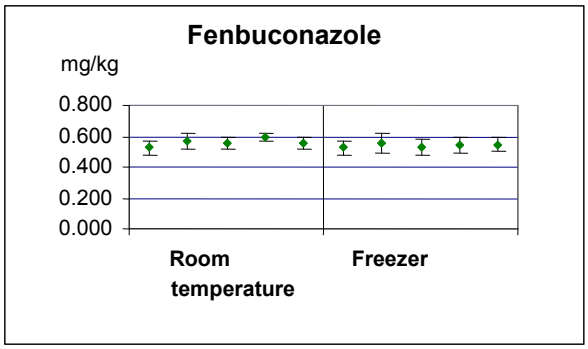


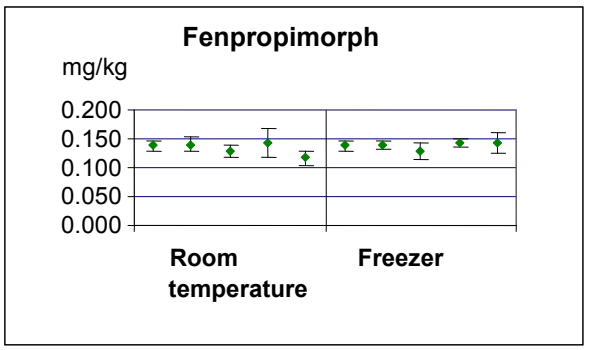


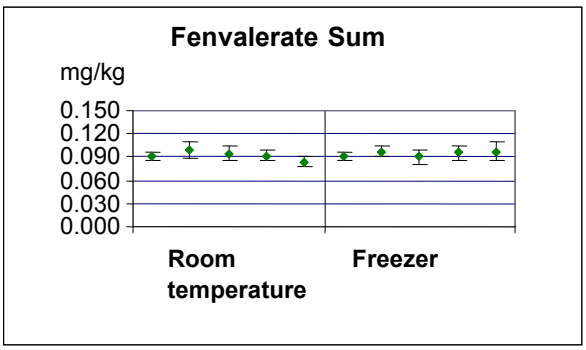


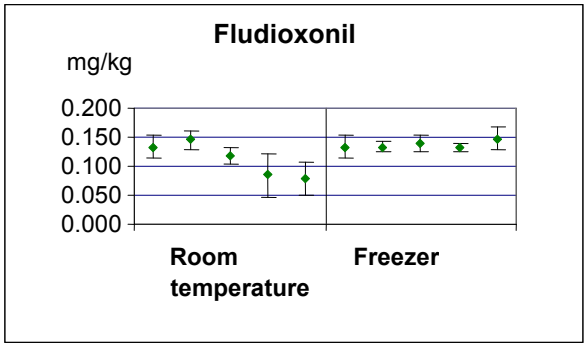


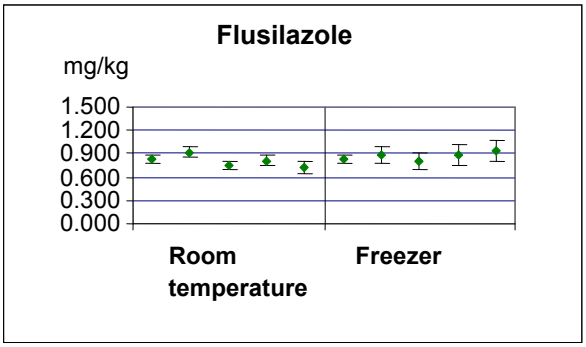


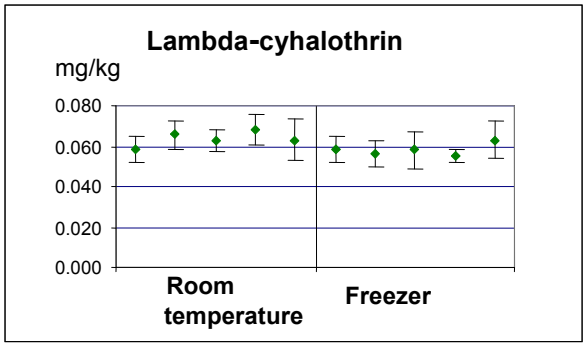


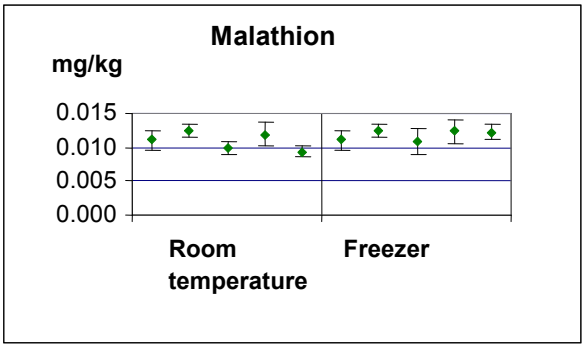


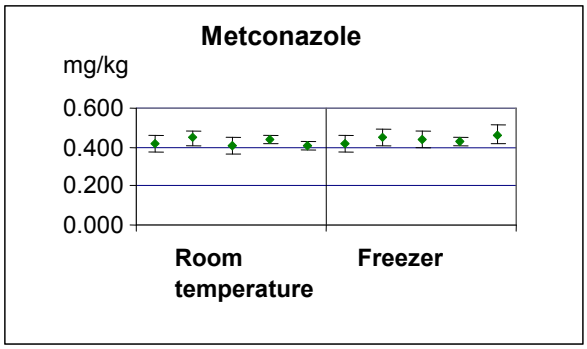


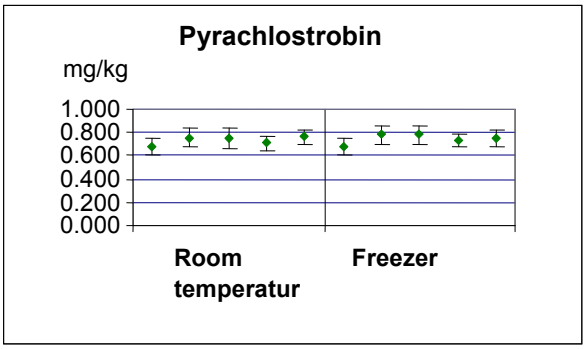

Fludioxonil
mg/kg
0.200
0.150
0.100
0.050
0.000
Room temperature
Freezer

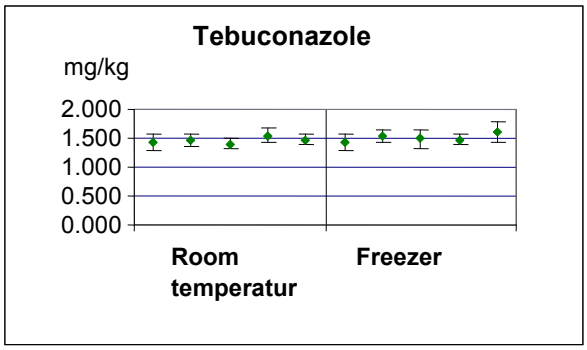

Flusilazole
mg/kg
1.500
1.200
0.900
0.600
0.300
0.000
Room temperature
Freezer


Lambda-cyhalothrin
mg/kg
0.080
0.060
0.040
0.020
0.000
Room temperature
Freezer

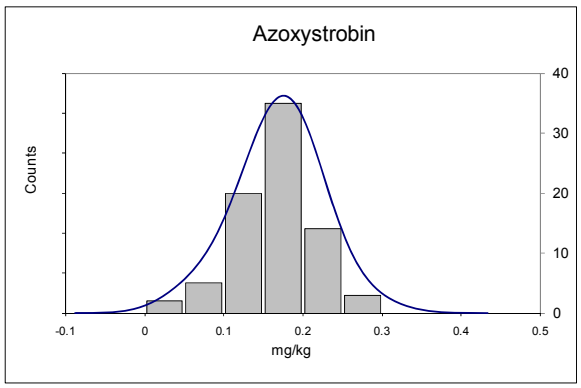

Malathion
mg/kg
0.015
0.010
0.005
0.000
Room temperature
Freezer

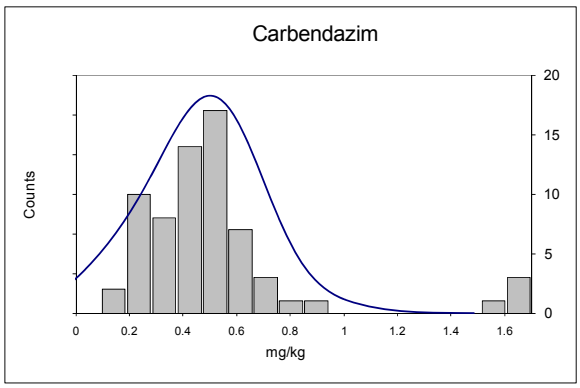

Metconazole
mg/kg
0.600
0.400
0.200
0.000
Room temperature
Freezer

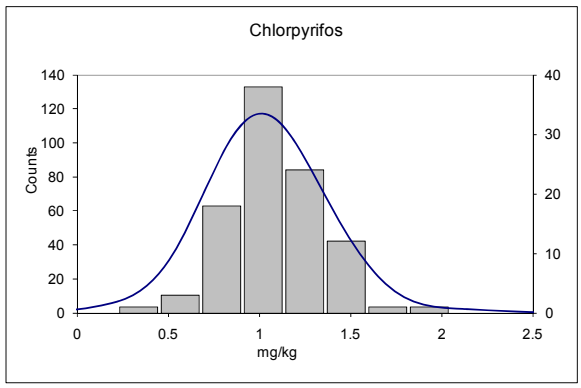

Pyrachlostrobin
mg/kg
1.000
0.800
0.600
0.400
0.200
0.000
Room temperatur
Freezer

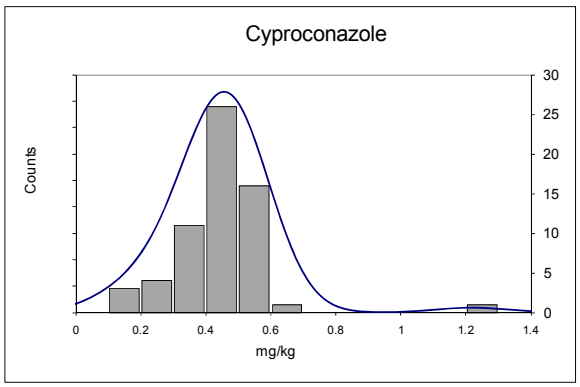

Tebuconazole
mg/kg
2.000
1.500
1.000
0.500
0.000
Room temperatur
Freezer

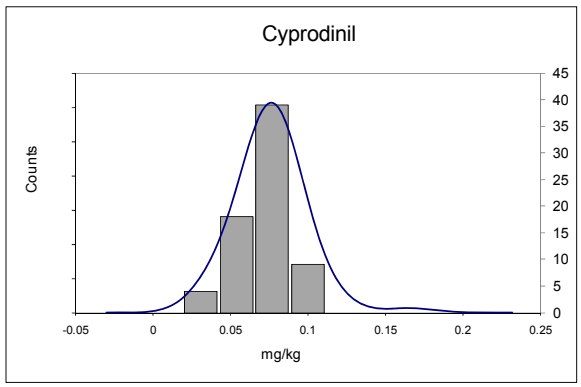
Appendix 5 Histograms of MRM-pesticides

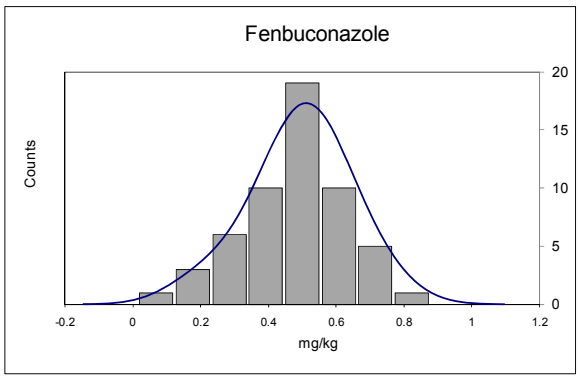


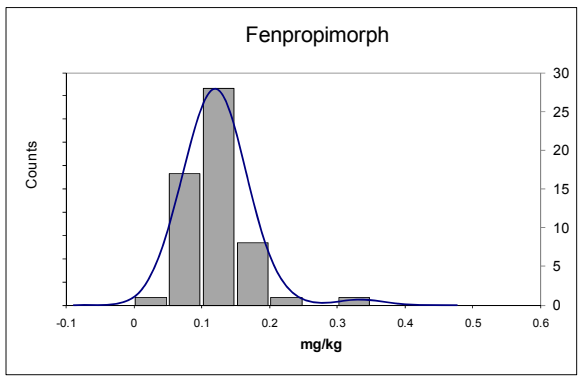


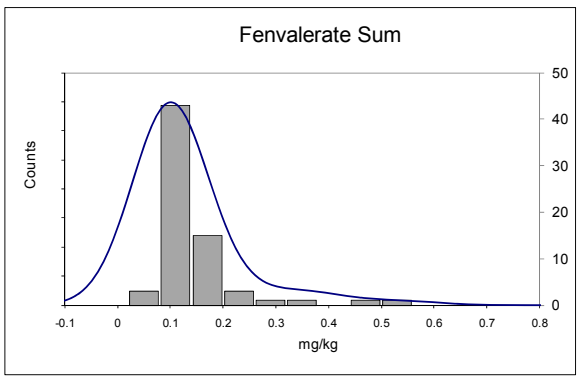


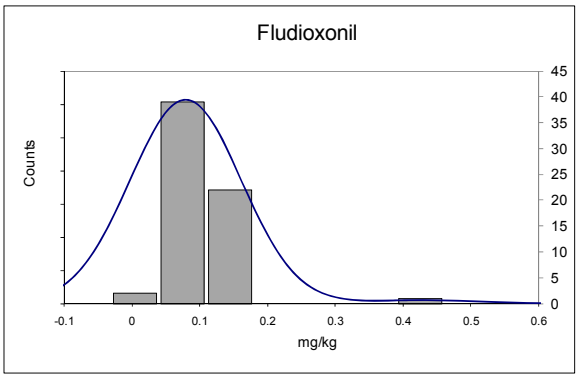


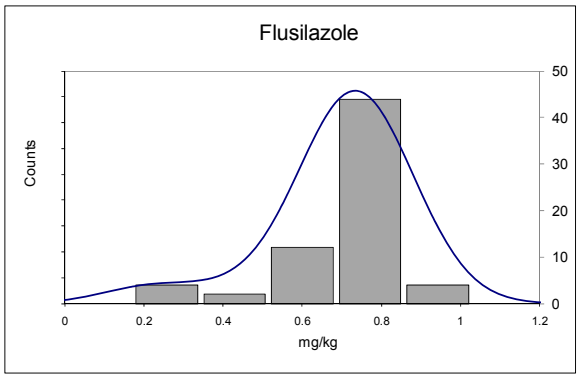


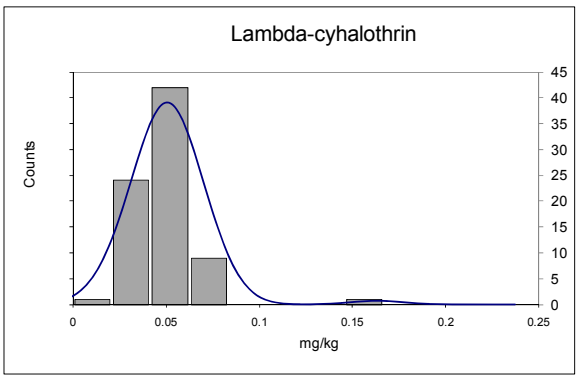


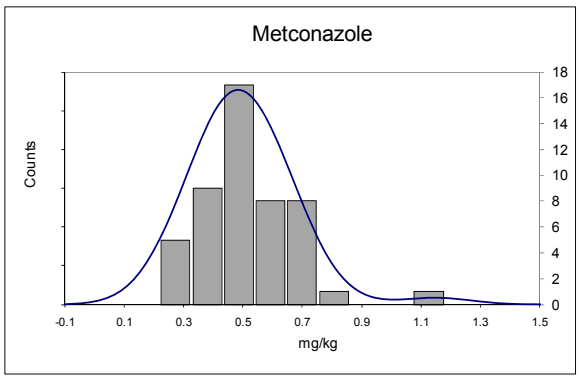


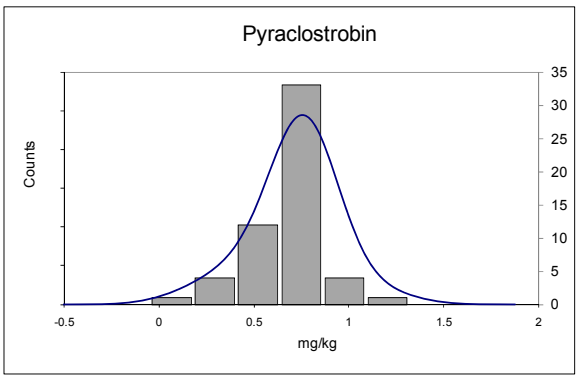


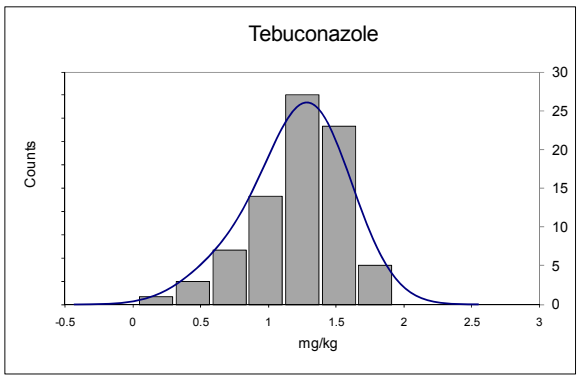

Fludioxonil
Counts
mg/kg
-0.1 0 0.1 0.2 0.3 0.4 0.5 0.6
0 5 10 15 20 25 30 35 40 45


Flusilazole
Counts
mg/kg
0 0.2 0.4 0.6 0.8 1 1.2
0 10 20 30 40 50

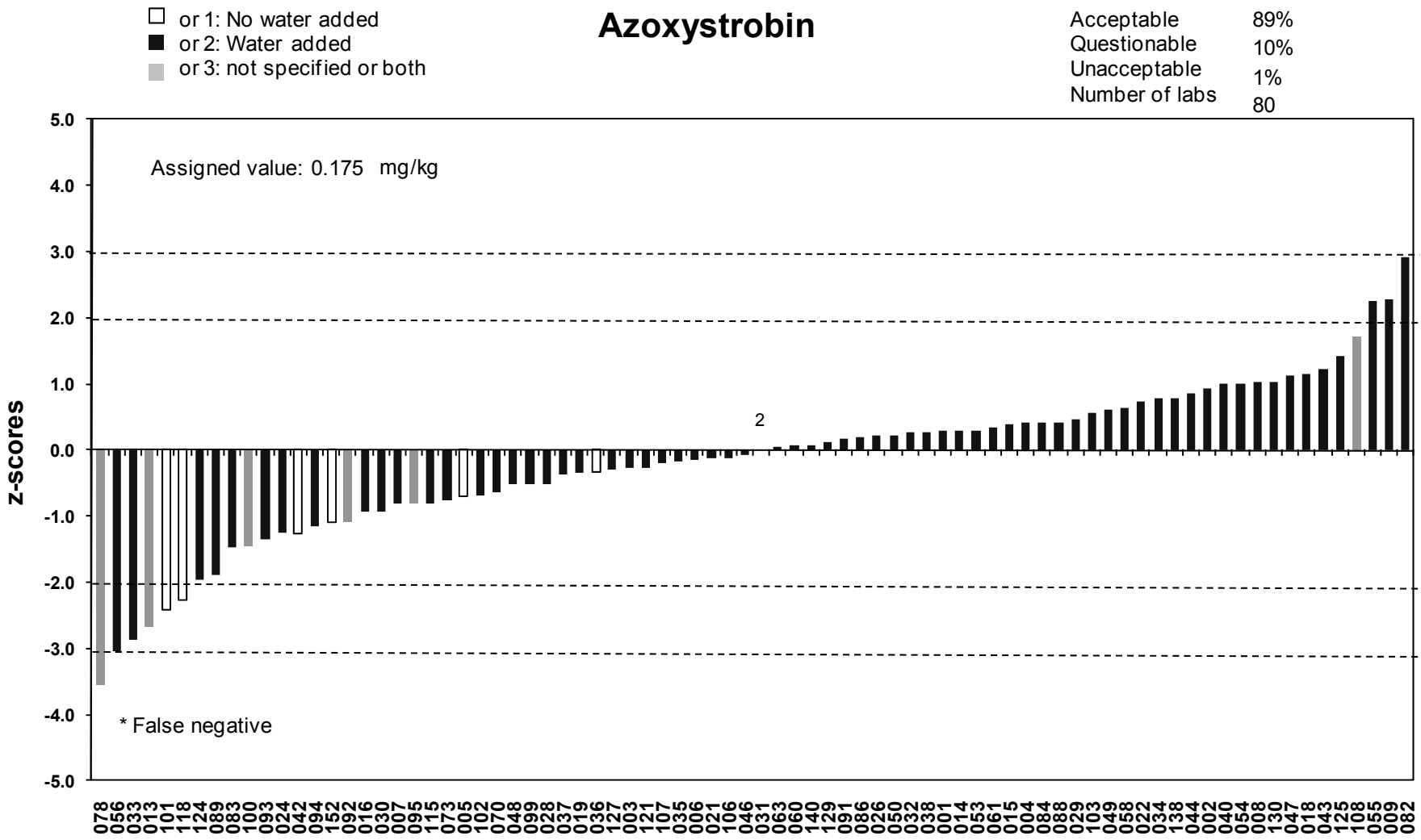

Lambda-cyhalothrin
Counts
mg/kg
0 0.05 0.1 0.15 0.2 0.25
0 5 10 15 20 25 30 35 40 45

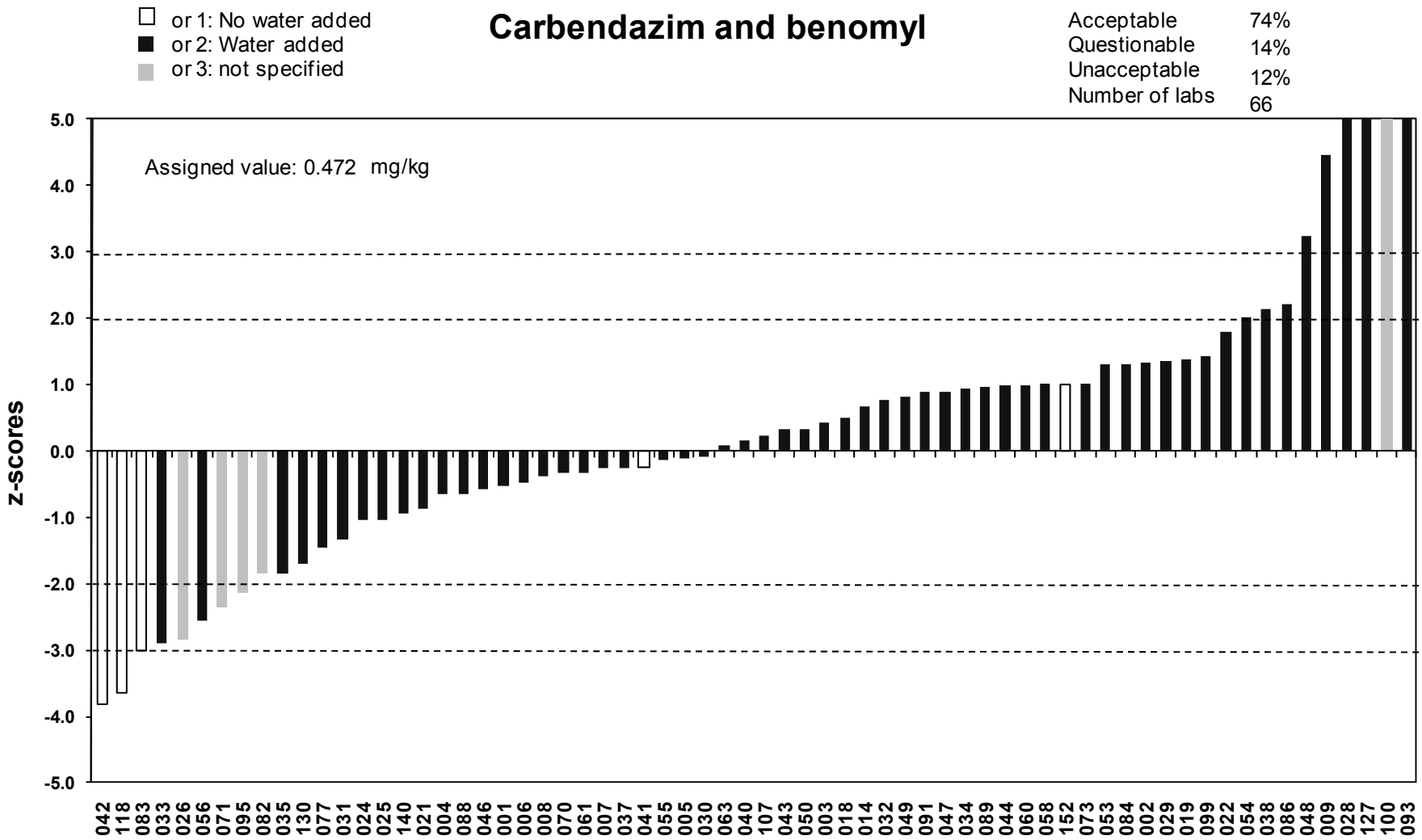

Metconazole
Counts
mg/kg
-0.1 0.1 0.3 0.5 0.7 0.9 1.1 1.3 1.5
0 2 4 6 8 10 12 14 16 18

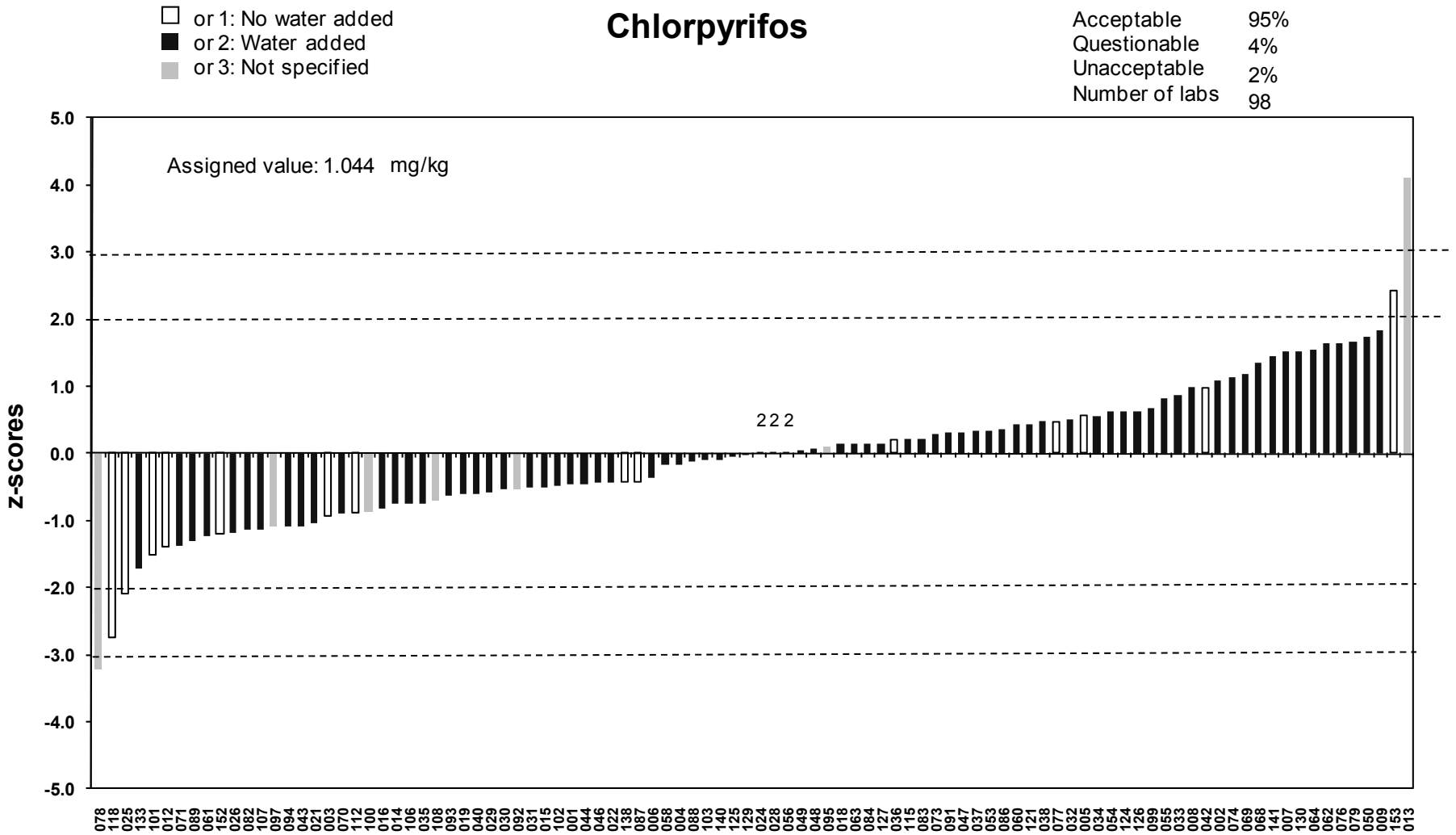

Pyraclostrobin
Counts
mg/kg
-0.5 0 0.5 1 1.5 2
0 5 10 15 20 25 30 35

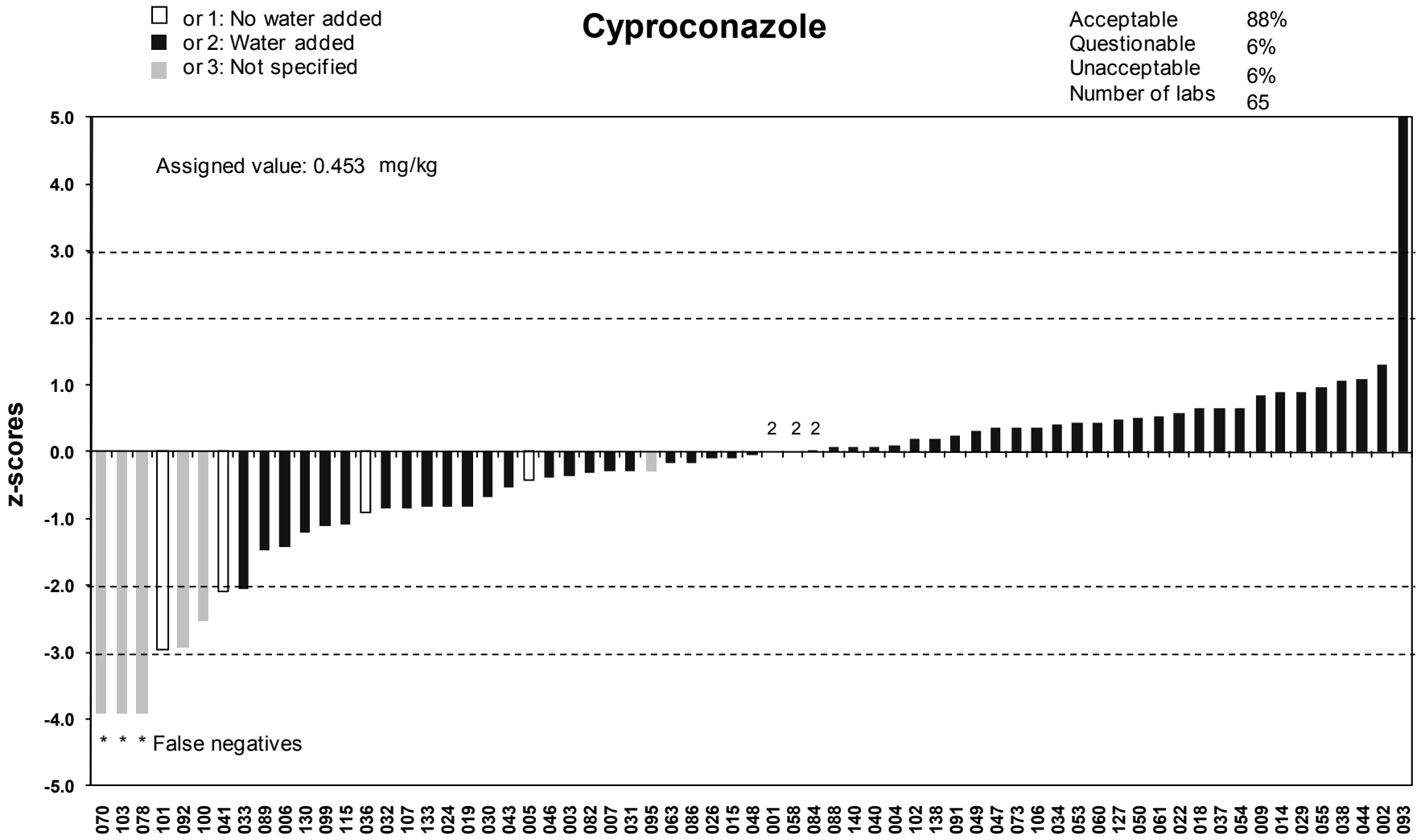

Tebuconazole
Counts
mg/kg
-0.5 0 0.5 1 1.5 2 2.5 3
0 5 10 15 20 25 30

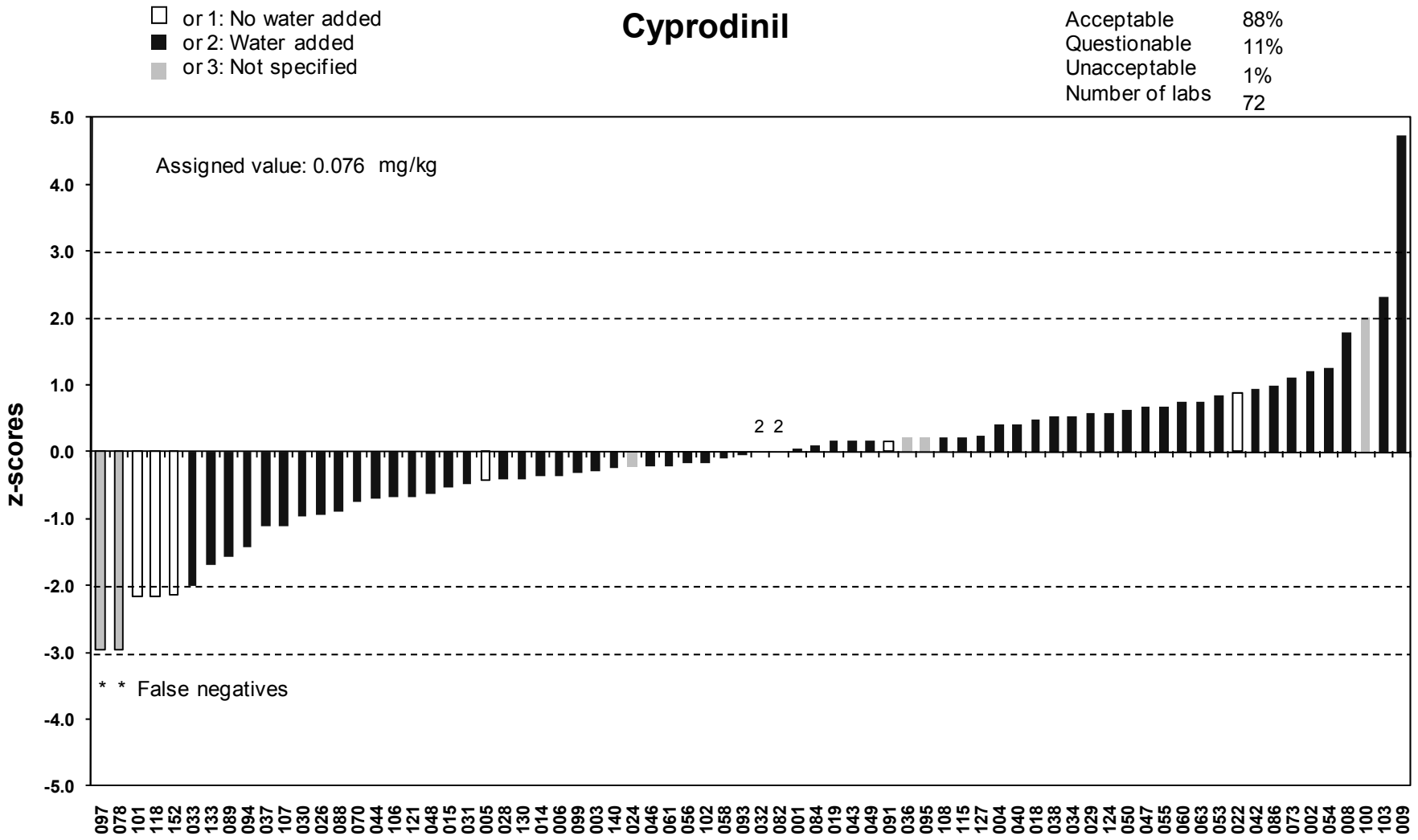
Appendix 6 Graphical presentation of z-scores for each MRM pesticide

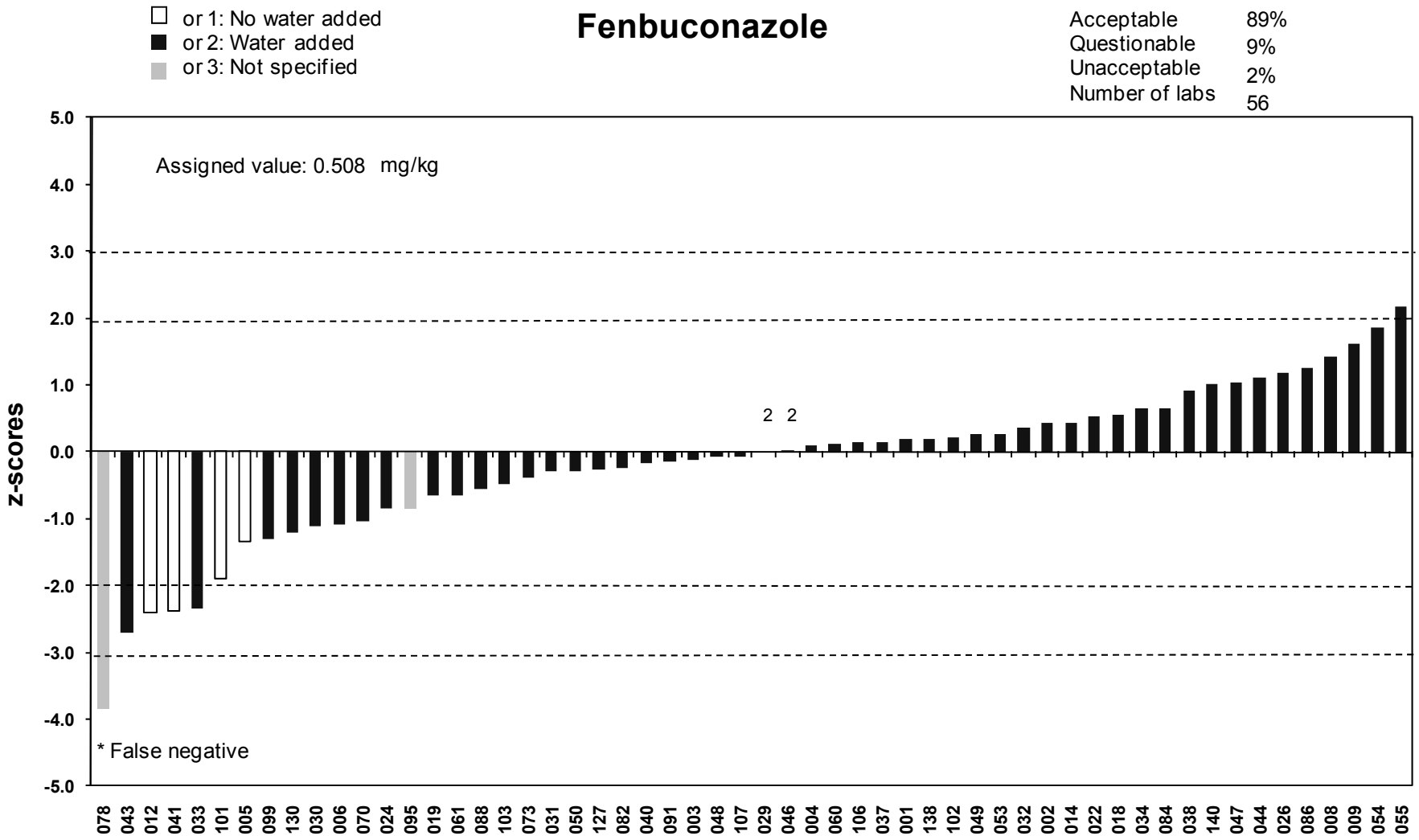


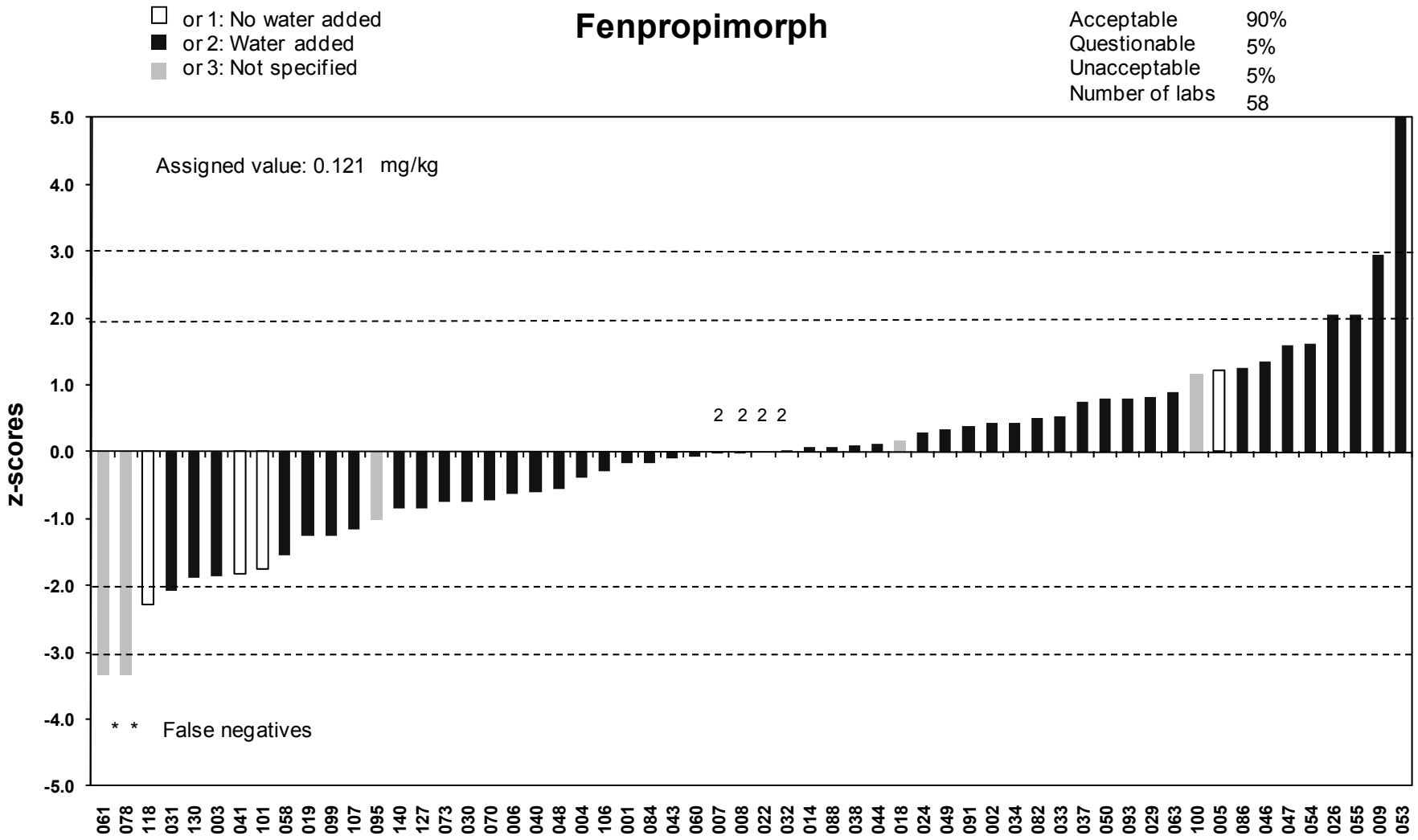

or 1: No water added
or 2: Water added
or 3: not specified
Carbendazim and benomyl
Acceptable 74%
Questionable 14%
Unacceptable 12%
Number of labs 66
Assigned value: 0.472 mg/kg
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
042 118 083 033 026 056 071 095 082 035 130 077 031 024 025 140 021 004 088 046 001 006 008 070 061 007 037 041 055 005 030 063 040 107 043 050 003 018 014 032 049 091 047 034 089 044 060 058 152 073 053 084 002 029 019 099 022 054 038 086 048 009 028 127 100 093


or 1: No water added
or 2: Water added
or 3: Not specified
Chlorpyrifos
Acceptable 95%
Questionable 4%
Unacceptable 2%
Number of labs 98
Assigned value: 1.044 mg/kg
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
222
078 118 025 133 101 012 071 089 061 152 026 082 107 097 094 043 021 003 070 112 100 016 014 106 035 108 093 019 040 029 030 092 031 015 102 001 044 046 022 138 087 006 058 004 088 103 140 125 129 024 028 056 049 048 095 018 063 084 127 036 115 083 073 091 047 037 053 086 060 121 038 077 032 005 034 054 124 126 099 055 033 008 042 002 074 069 068 141 007 130 064 062 076 079 050 009 153 013

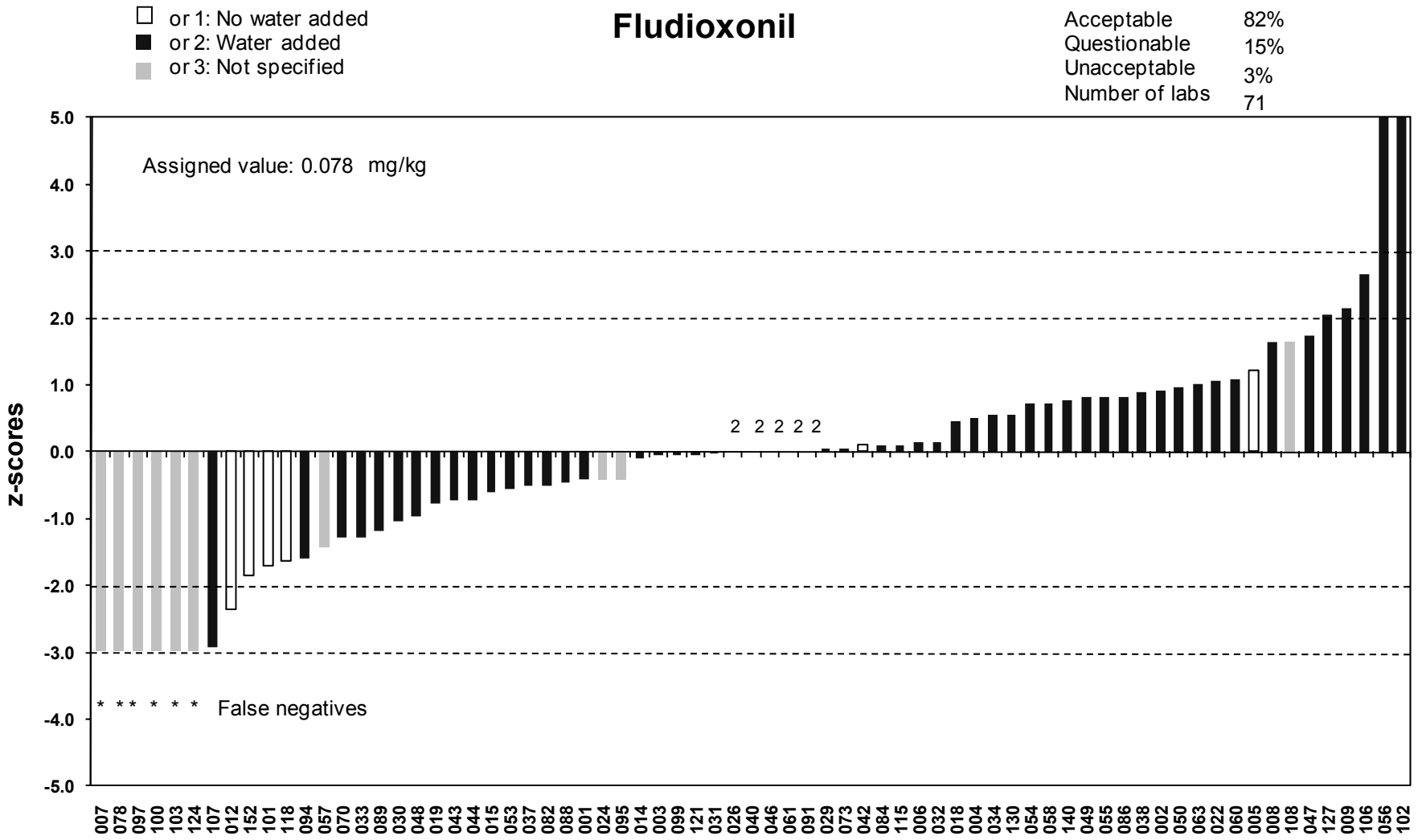

or 1: No water added
or 2: Water added
or 3: Not specified
Cyproconazole
Acceptable 88%
Questionable 6%
Unacceptable 6%
Number of labs 65
Assigned value: 0.453 mg/kg
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
2 2 2
* * * False negatives
070 103 078 101 092 100 041 033 089 006 130 099 115 036 032 107 133 024 019 030 043 005 046 003 082 007 031 095 063 086 026 015 048 001 058 084 088 140 040 004 102 138 091 049 047 073 106 034 053 060 127 050 061 022 018 037 054 009 014 029 055 038 044 002 093

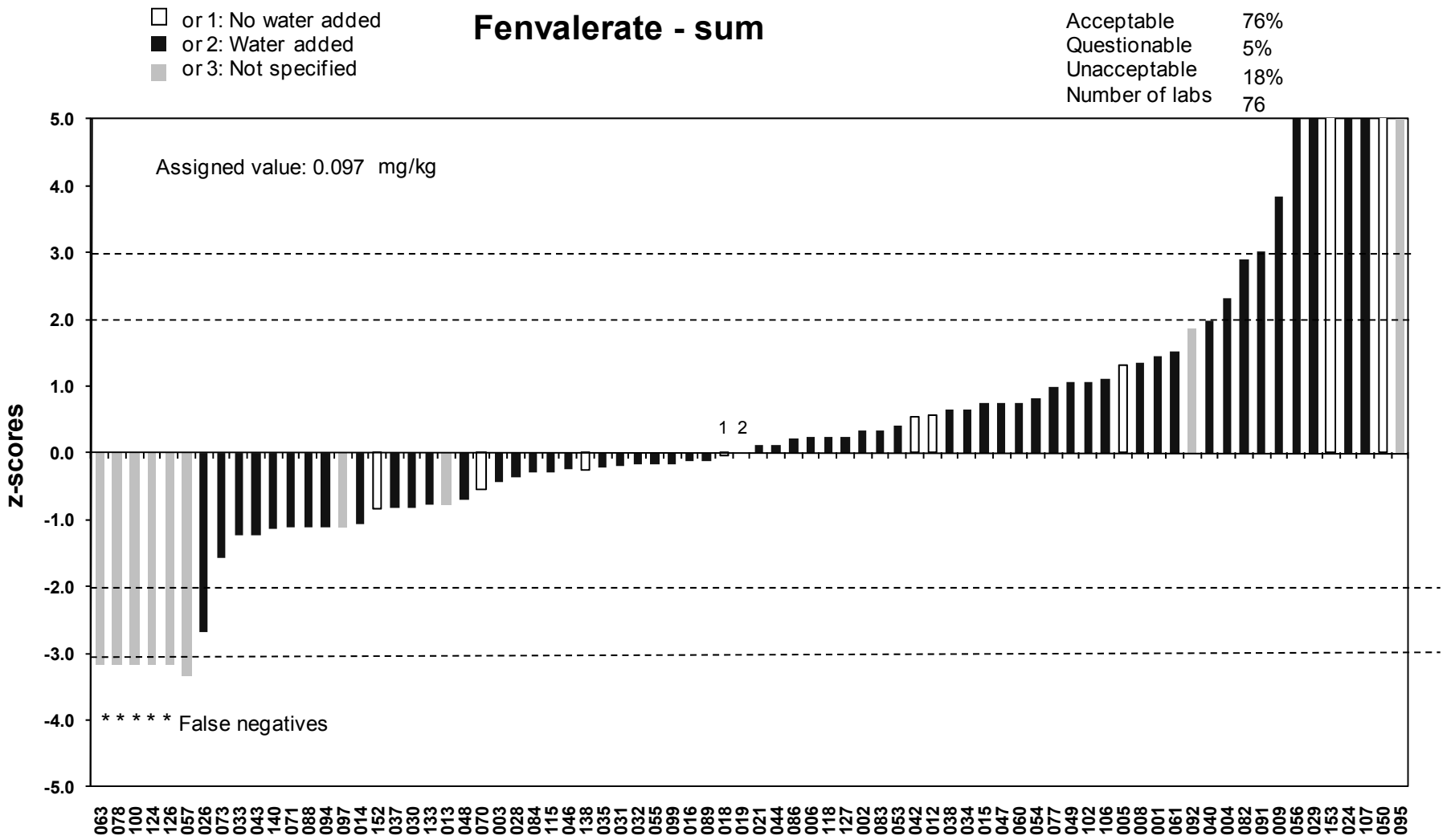

or 1: No water added
or 2: Water added
or 3: Not specified
Cyprodinil
Acceptable 88%
Questionable 11%
Unacceptable 1%
Number of labs 72
Assigned value: 0.076 mg/kg
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
2 2
* * False negatives
097 078 101 118 152 033 133 089 094 037 107 030 026 088 070 044 106 121 048 015 031 005 028 130 014 006 099 003 140 024 046 061 056 102 058 093 032 082 001 084 019 043 049 091 036 095 108 115 127 004 040 018 038 034 029 124 050 047 055 060 063 053 022 042 086 073 002 054 008 100 103 009

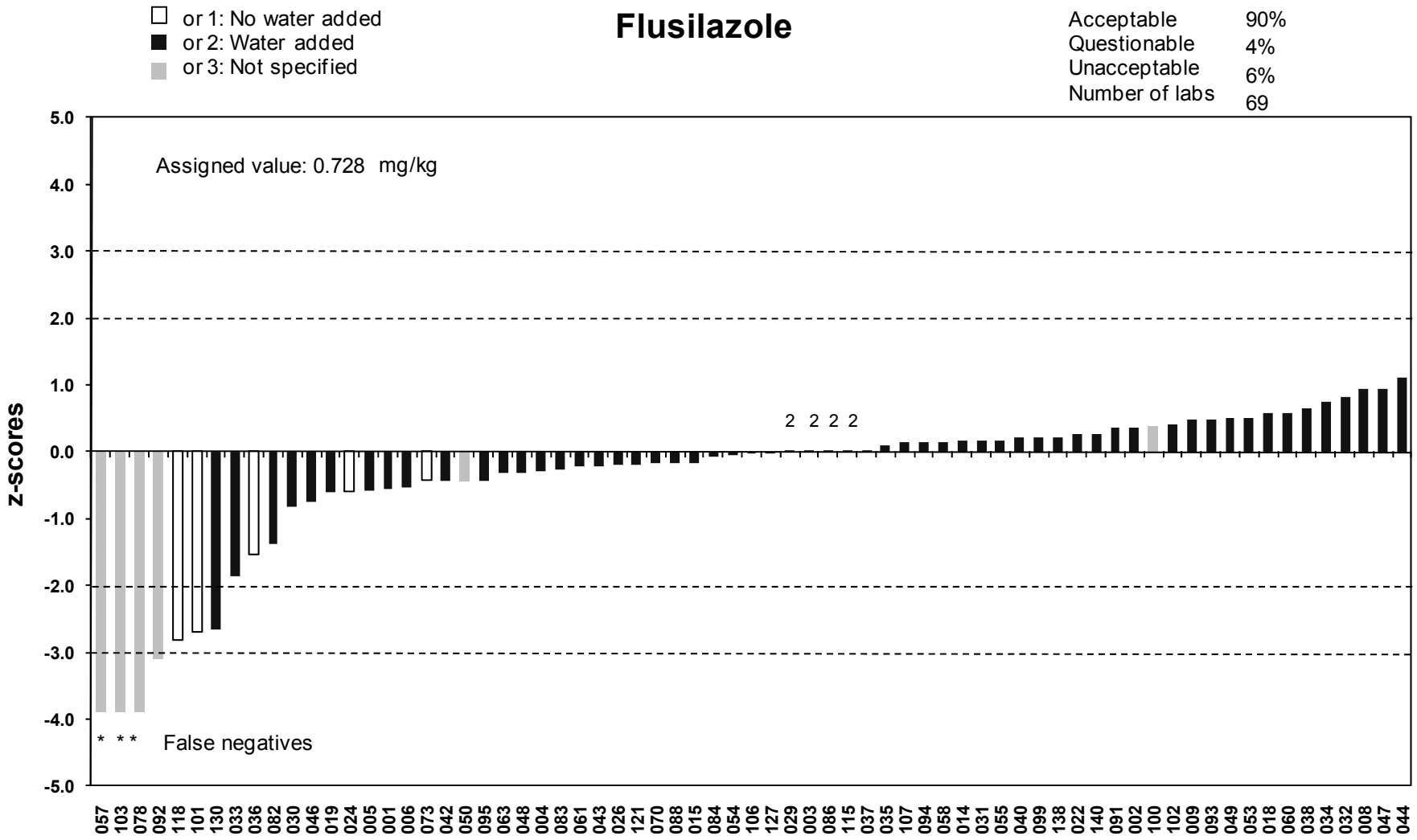

or 1: No water added
or 2: Water added
or 3: Not specified
Fenbuconazole
Acceptable 89%
Questionable 9%
Unacceptable 2%
Number of labs 56
Assigned value: 0.508 mg/kg
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
2 2
* False negative
078 043 012 041 033 101 005 099 130 030 006 070 024 095 019 061 088 103 073 031 050 127 082 040 091 003 048 107 029 046 004 060 106 037 001 138 102 049 053 032 002 014 022 018 034 084 038 140 047 044 026 086 008 009 054 055

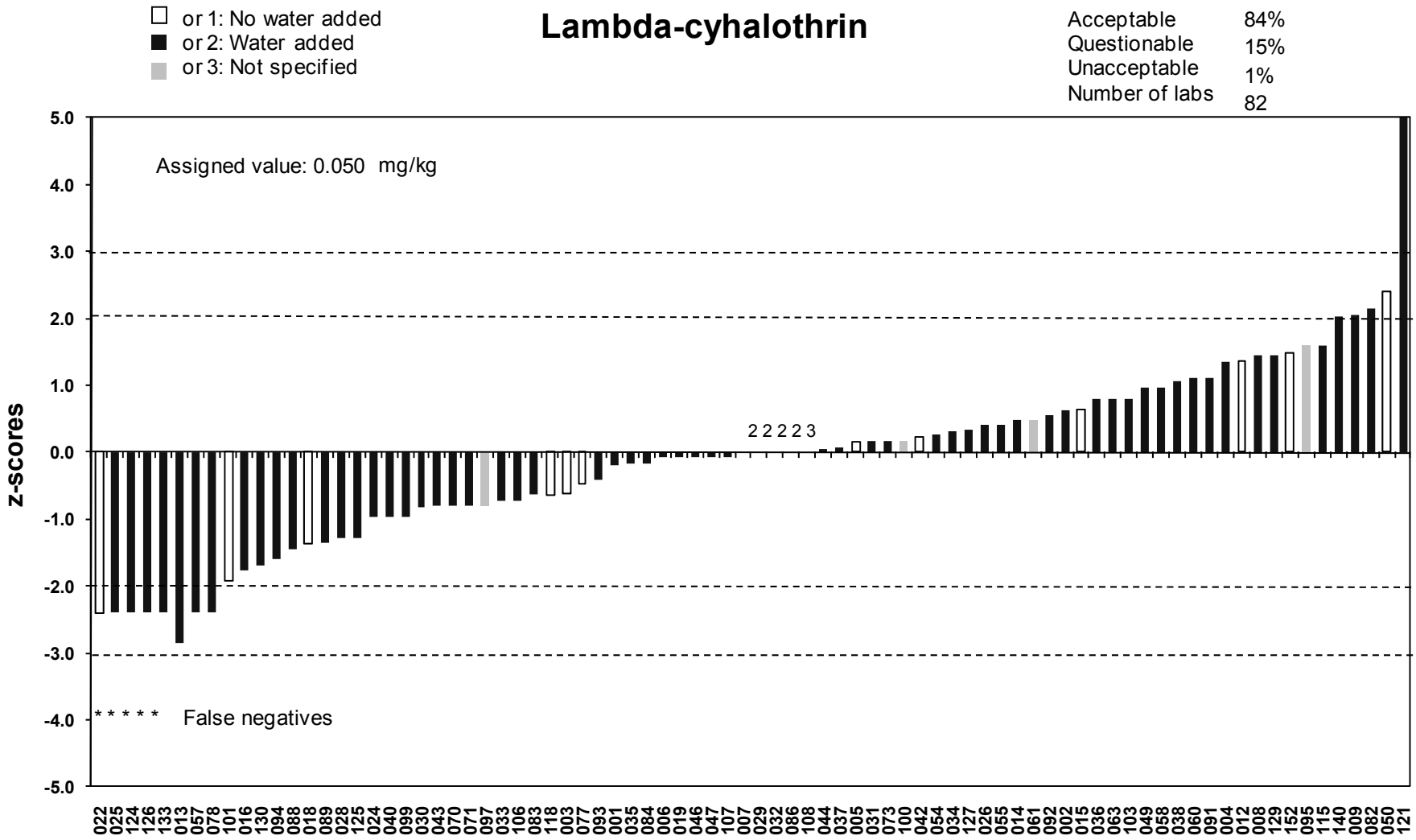

or 1: No water added
or 2: Water added
or 3: Not specified
Fenpropimorph
Acceptable 90%
Questionable 5%
Unacceptable 5%
Number of labs 58
Assigned value: 0.121 mg/kg
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
2 2 2 2
* * False negatives
061 078 118 031 130 003 041 101 058 019 099 107 095 140 127 073 030 070 006 040 048 004 106 001 084 043 060 007 008 022 032 014 088 038 044 018 024 049 091 002 034 082 033 037 050 093 029 063 100 005 086 046 047 054 026 055 009 053

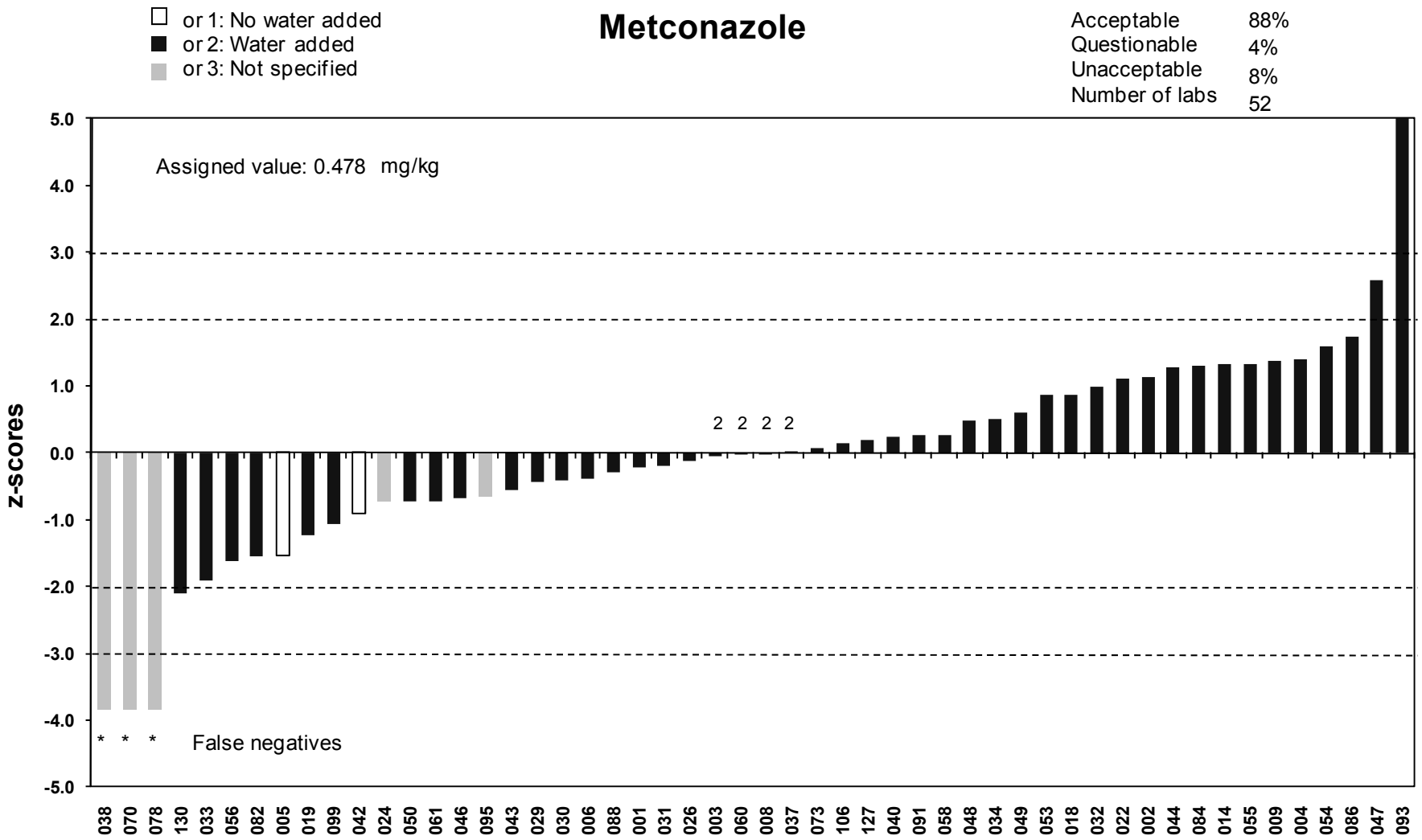
Fludioxonil

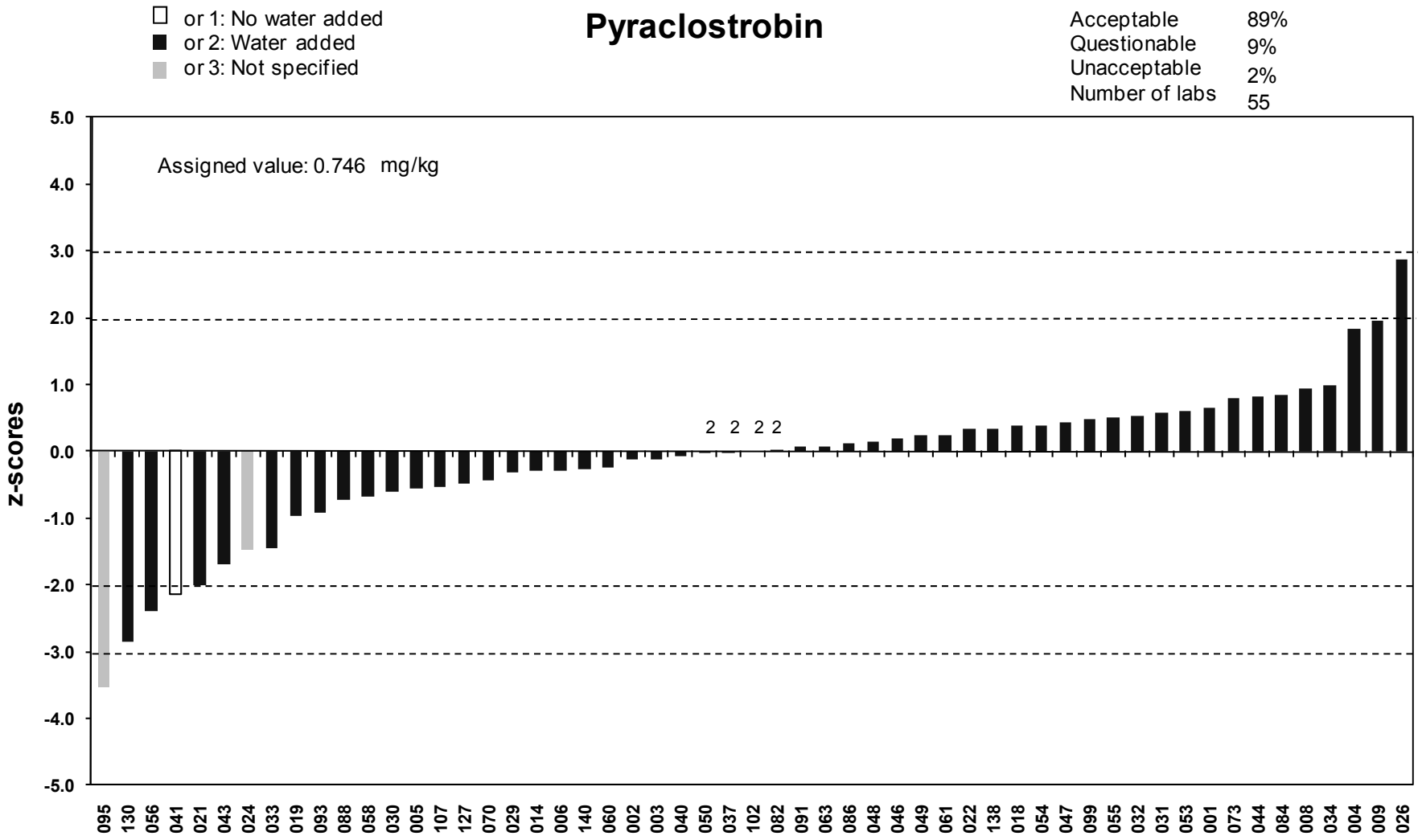



or 1: No water added
or 2: Water added
or 3: Not specified
Fenvalerate - sum
Acceptable 76%
Questionable 5%
Unacceptable 18%
Number of labs 76
Assigned value: 0.097 mg/kg
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
1 2
* * * * * False negatives
063 078 100 124 126 057 026 073 033 043 140 071 088 094 097 014 152 037 030 133 013 048 070 003 028 084 115 046 138 035 031 032 055 099 016 089 018 019 021 044 086 006 118 127 002 083 053 042 012 038 034 015 047 060 054 077 049 102 106 005 008 001 061 092 040 004 082 091 009 056 029 153 024 107 050 095

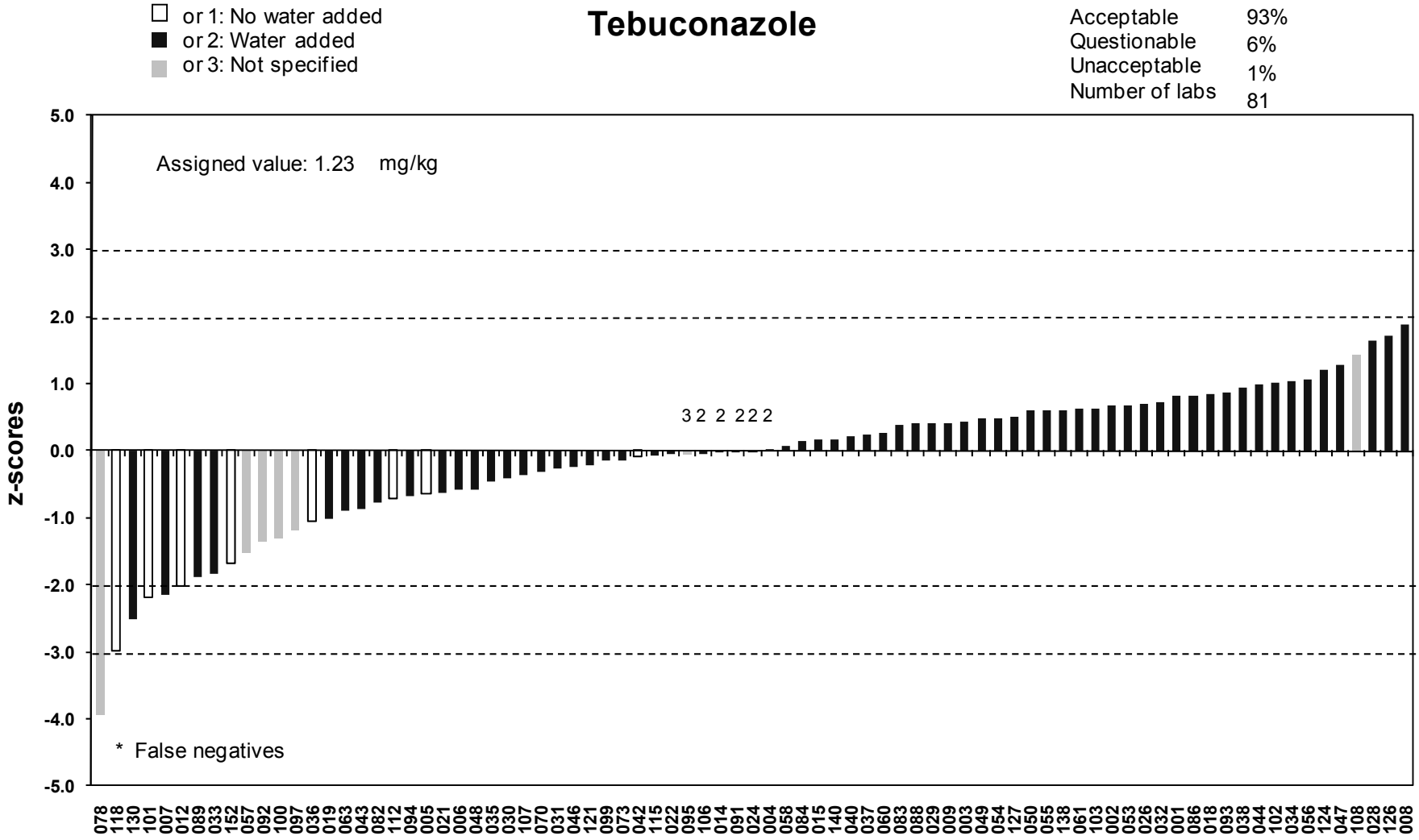

or 1: No water added
or 2: Water added
or 3: Not specified
Flusilazole
Acceptable 90%
Questionable 4%
Unacceptable 6%
Number of labs 69
Assigned value: 0.728 mg/kg
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
2 2 2 2
* * * False negatives
057 103 078 092 118 101 130 033 036 082 030 046 019 024 005 001 006 073 042 050 095 063 048 004 083 061 043 026 121 070 088 015 084 054 106 127 029 003 086 115 037 035 107 094 058 014 031 055 040 099 138 022 140 091 002 100 102 009 093 049 053 018 060 038 034 032 008 047 044


or 1: No water added
or 2: Water added
or 3: Not specified
Lambda-cyhalothrin
Acceptable 84%
Questionable 15%
Unacceptable 1%
Number of labs 82
Assigned value: 0.050 mg/kg
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
2 2 2 2 3
* * * * * False negatives
022 025 124 126 133 013 057 078 101 016 130 094 088 018 089 028 125 024 040 099 030 043 070 071 097 033 106 083 118 003 077 093 001 035 084 006 019 046 047 107 007 029 032 086 108 044 037 005 031 073 100 042 054 034 127 026 055 014 061 092 002 015 036 063 103 049 058 038 060 091 004 012 008 129 152 095 115 140 009 082 050 121


or 1: No water added
or 2: Water added
or 3: Not specified
Metconazole
Acceptable 88%
Questionable 4%
Unacceptable 8%
Number of labs 52
Assigned value: 0.478 mg/kg
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
2 2 2 2
* * * False negatives
038 070 078 130 033 056 082 005 019 099 042 024 050 061 046 095 043 029 030 006 088 001 031 026 003 060 008 037 073 106 127 040 091 058 048 034 049 053 018 032 022 002 044 084 014 055 009 004 054 086 047 093


or 1: No water added
or 2: Water added
or 3: Not specified
Pyraclostrobin
Acceptable 89%
Questionable 9%
Unacceptable 2%
Number of labs 55
Assigned value: 0.746 mg/kg
z-scores
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
2 2 2 2
095 130 056 041 021 043 024 033 019 093 088 058 030 005 107 127 070 029 014 006 140 060 002 003 040 050 037 102 082 091 063 086 048 046 049 061 022 138 018 054 047 099 055 032 031 053 001 073 044 084 008 034 004 009 026

Tebuconazole



Appendix 7 MRM-Methods used by the participating Laboratories

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1)]	Extraction solvent II [1)]	Extraction solvent III [1)]	Water addition	Hydrolysis	pH adjused	Clean up [2)]	Calibration [3)]	GC detector	HPLC detector	Confirmation	Reference method
eupt 001	Azoxystrobin	No	0.188	5	ACN			Yes	No	No	SPE	MM-ML	MSD		GC/MS	
	Carbendazim and benomyl	No	0.41	0	ACN			Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	QuEChERS
	Chlorpyrifos	No	0.924	5	ACN			Yes	No	No	SPE	MM-ML	MSD		GC/MS	
	Cyproconazole	No	0.453	5	ACN			Yes	No	No	SPE	MM-ML	MSD		GC/MS	
	Cyprodinil	No	0.077	5	ACN			Yes	No	No	SPE	MM-ML	MSD		GC/MS	
	Fenvalerate and Esfenvalerate	No	0.132	5	ACN			Yes	No	No	SPE	MM-ML	MSD		GC/MS	
	Fenbuconazole	No	0.534	5	ACN			Yes	No	No	SPE	MM-ML	MSD		GC/MS	
	Fenpropimorph	No	0.116	5	ACN			Yes	No	No	SPE	MM-ML	MSD		GC/MS	
	Fludioxonil	No	0.07	5	ACN			Yes	No	No	SPE	MM-ML	MSD		GC/MS	
	Flusilazole	No	0.626	0	ACN			Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	QuEChERS
	Lambda-cyhalothrin	No	0.0475	5	ACN			Yes	No	No	SPE	MM-ML	MSD		GC/MS	
	Metconazole	No	0.453	5	ACN			Yes	No	No	SPE	MM-ML	MSD		GC/MS	
	Pyraclostrobin	No	0.866	5	ACN			Yes	No	No	SPE	MM-ML	MSD		GC/MS	
	Tebuconazole	No	1,478	5	ACN			Yes	No	No	SPE	MM-ML	MSD		GC/MS	
eupt 002	Azoxystrobin	No	0.216	3	ACN	Other		Yes	No	pH 4	None	MM-SL		MS/MS		
	Carbendazim and benomyl	No	0.63	3	ACN	Other		Yes	No	pH 4	None	MM-SL		MS/MS		
	Chlorpyrifos	No	1.33	3	ACN	Other		Yes	No	pH 4	None	MM-SL		MS/MS		
	Cyproconazole	No	0.6	3	ACN	Other		Yes	No	pH 4	None	MM-SL		MS/MS		
	Cyprodinil	No	0.099	3	ACN	Other		Yes	No	pH 4	None	MM-SL		MS/MS		
	Fenvalerate and Esfenvalerate	No	0.105	3	ACN			Yes	No	pH 4	DSPE	MM-SL	MS/MS			
	Fenbuconazole	No	0.564	3	ACN	Other		Yes	No	pH 4	None	MM-SL		MS/MS		

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1)]	Extraction solvent II [1)]	Extraction solvent III [1)]	Water addition	Hydrolysis	pH adjused	Clean up [2)]	Calibration [3)]	GC detector	HPLC detector	Confirmation	Reference method
	Fenpropimorph	No	0.134	3	ACN	Other		Yes	No	pH 4	None	MM-SL		MS/MS		
	Fludioxonil	No	0.096	3	ACN	Other		Yes	No	pH 4	None	MM-SL		MS/MS		
	Flusilazole	No	0.794	3	ACN	Other		Yes	No	pH 4	None	MM-SL		MS/MS		
	Lambda-cyhalothrin	No	0.058	3	ACN			Yes	No	pH 4	DSPE	MM-SL	MS/MS			
	Metconazole	No	0.613	3	ACN	Other		Yes	No	pH 4	None	MM-SL		MS/MS		
	Pyraclostrobin	No	0.722	3	ACN	Other		Yes	No	pH 4	None	MM-SL		MS/MS		
	Tebuconazole	No	1.43	3	ACN	Other		Yes	No	pH 4	None	MM-SL		MS/MS		
eupt 003	Azoxystrobin	Yes	0.163	5	ACN			Yes	No	Yes	None	MM-ML		MS/MS	LC-MS/MS	Anastassiades et al. JAOAC 86 (2003)
	Carbendazim and benomyl	Yes	0.521	5	ACN			Yes	No	Yes	None	MM-ML		MS/MS	LC-MS/MS	Anastassiades et al. JAOAC 86 (2003)
	Chlorpyrifos	Yes	0.802	25	Acetone	MeOH		No	No	No	GPC	MM-ML	MSD		GC/MS	Bottomley. P. and Bake.r P. (1984).
	Cyproconazole	Yes	0.413	5	ACN			Yes	No	Yes	None	MM-ML		MS/MS	LC-MS/MS	Anastassiades et al. JAOAC 86 (2003)
	Cyprodinil	Yes	0.0705	5	ACN			Yes	No	Yes	None	MM-ML		MS/MS	LC-MS/MS	Anastassiades et al. JAOAC 86 (2003)
	Fenvalerate and Esfenvalerate	Yes	0.0865	25	Acetone	MeOH		No	No	No	GPC	MM-ML	MSD		GC/MS	Bottomley. P. and Bake.r P. (1984).
	Fenbuconazole	Yes	0.494	5	ACN			Yes	No	Yes	None	MM-ML		MS/MS	LC-MS/MS	Anastassiades et al. JAOAC 86 (2003)
	Fenpropimorph	Yes	0.0646	5	ACN			Yes	No	Yes	None	MM-ML		MS/MS	LC-MS/MS	Anastassiades et al. JAOAC 86 (2003)
	Fludioxonil	Yes	0.077	5	ACN			Yes	No	Yes	None	MM-ML		MS/MS	LC-MS/MS	Anastassiades et al. JAOAC 86 (2003)
	Flusilazole	Yes	0.73	5	ACN			Yes	No	Yes	None	MM-ML		MS/MS	LC-MS/MS	Anastassiades et al. JAOAC 86 (2003)
	Lambda-cyhalothrin	Yes	0.0424	25	Acetone	MeOH		No	No	No	GPC	MM-ML	MSD		GC/MS	Bottomley. P. and Bake.r P. (1984). Analyst. 109. 85-106
	Metconazole	Yes	0.473	5	ACN			Yes	No	Yes	None	MM-ML		MS/MS	LC-MS/MS	Anastassiades et al. JAOAC 86 (2003)

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Pyraclostrobin	Yes	0.724	5	ACN			Yes	No	Yes	None	MM-ML		MS/MS	LC-MS/MS	Anastassiades et al. JAOAC 86 (2003)
	Tebuconazole	Yes	1.36	5	ACN			Yes	No	Yes	None	MM-ML		MS/MS	LC-MS/MS	Anastassiades et al. JAOAC 86 (2003)
eupt 004	Azoxystrobin	Yes	0.193	20	Acetone	CH_2Cl_2		Yes	No	No	GPC	MM-ML	MSD	MS/MS	LC-MS/MS	DFG-S19. graphite aminopropyl SPE
	Carbendazim and benomyl	Yes	0.395	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	LC-MS/MS	Quechers
	Chlorpyrifos	Yes	1	20	Acetone	CH_2Cl_2		Yes	No	No	GPC	MM-ML	MSD	MS/MS	LC-MS/MS	DFG-S19. graphite aminopropyl SPE
	Cyproconazole	Yes	0.465	20	Acetone	CH_2Cl_2		Yes	No	No	GPC	MM-ML	MSD	MS/MS	LC-MS/MS	DFG-S19. graphite aminopropyl SPE
	Cyprodinil	Yes	0.084	20	Acetone	CH_2Cl_2		Yes	No	No	GPC	MM-ML	MSD	MS/MS	LC-MS/MS	DFG-S19. graphite aminopropyl SPE
	Fenvalerate and Esfenvalerate	Yes	0.153	20	Acetone	CH_2Cl_2		Yes	No	No	GPC	MM-ML	MSD	MS/MS	LC-MS/MS	DFG-S19. graphite aminopropyl SPE
	Fenbuconazole	Yes	0.52	20	Acetone	CH_2Cl_2		Yes	No	No	GPC	MM-ML	MSD	MS/MS	LC-MS/MS	DFG-S19. graphite aminopropyl SPE
	Fenpropimorph	Yes	0.109	5	ACN	CH_2Cl_2		Yes	No	Yes	DSPE	MM-ML		MS/MS	LC-MS/MS	Quechers
	Fludioxonil	Yes	0.088	20	Acetone	CH_2Cl_2		Yes	No	No	GPC	MM-ML	MSD	MS/MS	LC-MS/MS	DFG-S19. graphite aminopropyl SPE
	Flusilazole	Yes	0.674	20	Acetone	CH_2Cl_2		Yes	No	No	GPC	MM-ML	MSD	MS/MS	LC-MS/MS	DFG-S19. graphite aminopropyl SPE
	Lambda-cyhalothrin	Yes	0.067	20	Acetone	CH_2Cl_2		Yes	No	No	GPC	MM-ML	MSD	MS/MS	LC-MS/MS	DFG-S19. graphite aminopropyl SPE
	Metconazole	Yes	0.644	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	LC-MS/MS	QuEChERS
	Pyraclostrobin	Yes	1.09	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	LC-MS/MS	QuEChERS
	Tebuconazole	Yes	1.23	20	Acetone	CH_2Cl_2		Yes	No	No	GPC	MM-ML	MSD	MS/MS	LC-MS/MS	DFG-S19. graphite aminopropyl SPE
eupt 005	Azoxystrobin	Yes	0.144	20	Acetone	CH_2Cl_2		No	No	No	None	MM-ML	ECD		GC/MS	Anal. Meth. f. Pest. Res. in Foodst. NL 6th Ed. 1996

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Carbendazim and benomyl	Yes	0.458	5	MeOH			Yes	No	No	SPE	MM-ML		MS/MS	None	Klein. J.; Alder. L.; JAOAC; 86.1015 (2003)
	Chlorpyrifos	Yes	1.19	20	Acetone	CH_2Cl_2		No	No	No	None	MM-ML	FPD		GC/MS	Anal. Meth. f. Pest. Res. in Foodst. NL 6th Ed. 1996
	Cyproconazole	Yes	0.404	20	Acetone	CH_2Cl_2		No	No	No	None	MM-ML	NPD		GC/MS	Anal. Meth. f. Pest. Res. in Foodst. NL 6th Ed. 1996
	Cyprodinil	Yes	0.0678	20	Acetone	CH_2Cl_2		No	No	No	None	MM-ML	NPD		GC/MS	Anal. Meth. f. Pest. Res. in Foodst. NL 6th Ed. 1996
	Fenvalerate and Esfenvalerate	Yes	0.129	20	Acetone	CH_2Cl_2		No	No	No	None	MM-ML	ECD		GC/MS	Anal. Meth. f. Pest. Res. in Foodst. NL 6th Ed. 1996
	Fenbuconazole	No	0.339	20	Acetone	CH_2Cl_2		No	No	No	None	MM-ML	NPD		GC/MS	Anal. Meth. f. Pest. Res. in Foodst. NL 6th Ed. 1996
	Fenpropimorph	No	0.158	20	Acetone	CH_2Cl_2		No	No	No	None	MM-ML	NPD		GC/MS	Anal. Meth. f. Pest. Res. in Foodst. NL 6th Ed. 1996
	Fludioxonil	Yes	0.102	20	Acetone	CH_2Cl_2		No	No	No	None	MM-ML	ECD		GC/MS	Anal. Meth. f. Pest. Res. in Foodst. NL 6th Ed. 1996
	Flusilazole	Yes	0.621	20	Acetone	CH_2Cl_2		No	No	No	None	MM-ML	NPD		GC/MS	Anal. Meth. f. Pest. Res. in Foodst. NL 6th Ed. 1996
	Lambda-cyhalothrin	Yes	0.052	20	Acetone	CH_2Cl_2		No	No	No	None	MM-ML	ECD		GC/MS	Anal. Meth. f. Pest. Res. in Foodst. NL 6th Ed. 1996
	Metconazole	No	0.296	20	Acetone	CH_2Cl_2		No	No	No	None	MM-ML	NPD		GC/MS	Anal. Meth. f. Pest. Res. in Foodst. NL 6th Ed. 1996
	Pyraclostrobin	Yes	0.644	5	MeOH			Yes	No	No	SPE	MM-ML		MS/MS	None	Klein. J.; Alder. L.; JAOAC; 86.1015 (2003)

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Tebuconazole	Yes	1.03	20	Acetone	CH_2Cl_2		No	No	No	None	MM-ML	NPD		GC/MS	Anal. Meth. f. Pest. Res. in Foodst. NL 6th Ed. 1996
eupt 006	Carbendazim and benomyl	Yes	0.414	15	ACN			Yes	No	pH 4	None	MM-ML		MS/MS	LC-MS/MS	NFA
	Chlorpyrifos	No	0.947	15	ACN			Yes	No	pH 4	None	MM-ML	MS/MS		GC-MS/MS	
	Cyproconazole	No	0.291	15	ACN			Yes	No	pH 4	None	MM-ML		MS/MS	LC-MS/MS	NFA
	Cyprodinil	No	0.0692	15	ACN			Yes	No	pH 4	None	MM-ML	MS/MS		GC-MS/MS	
	Fenvalerate and Esfenvalerate	No	0.103	15	ACN			Yes	No	pH 4	None	MM-ML	MS/MS		GC-MS/MS	
	Fenbuconazole	Yes	0.369	15	ACN			Yes	No	pH 4	None	MM-ML		MS/MS	LC-MS/MS	NFA
	Fenpropimorph	No	0.102	15	ACN			Yes	No	pH 4	None	MM-ML		MS/MS	LC-MS/MS	NFA
	Fludioxonil	No	0.0806	15	ACN			Yes	No	pH 4	None	MM-ML		MS/MS	LC-MS/MS	NFA
	Flusilazole	Yes	0.631	15	ACN			Yes	No	pH 4	None	MM-ML		MS/MS	LC-MS/MS	NFA
	Lambda-cyhalothrin	No	0.049	15	ACN			Yes	No	pH 4	None	MM-ML	MS/MS		GC-MS/MS	
	Metconazole	No	0.432	15	ACN			Yes	No	pH 4	None	MM-ML		MS/MS	LC-MS/MS	NFA
	Pyraclostrobin	Yes	0.691	15	ACN			Yes	No	pH 4	None	MM-ML		MS/MS	LC-MS/MS	NFA
	Tebuconazole	Yes	1.05	15	ACN			Yes	No	pH 4	None	MM-ML		MS/MS	LC-MS/MS	NFA
eupt 007	Azoxystrobin	Yes	0.14	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		GC/MS	In house
	Carbendazim and benomyl	Yes	0.44	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	LC-MS/MS	In house
	Chlorpyrifos	Yes	1.44	5	ACN			Yes	No	No	DSPE	MM-ML	ECD			In house
	Cyproconazole	Yes	0.42	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		GC/MS	In house
	Fenpropimorph	Yes	0.12	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		GC/MS	In house
	Lambda-cyhalothrin	Yes	0.05	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		GC/MS	In house
	Malathion	Yes	<0.02	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	LC-MS/MS	In house
	Permethrin	Yes	0.04	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		GC/MS	In house
	Prochloraz. only parent	Yes	0.04	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		GC/MS	In house
	Procymidone	Yes	0.04	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		GC/MS	In house
	Tebuconazole	Yes	0.57	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		GC/MS	In house

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1)]	Extraction solvent II [1)]	Extraction solvent III [1)]	Water addition	Hydrolysis	pH adjused	Clean up [2)]	Calibration [3)]	GC detector	HPLC detector	Confirmation	Reference method
eupt 008	Azoxystrobin	Yes	0.22	10	Acetone			Yes	No	No	SPE	MM-ML	MSD			
	Carbendazim and benomyl	Yes	0.426	10	MeOH			Yes	No	Yes	SPE	MM-ML		MS/MS		
	Chlorpyrifos	Yes	1.3	10	Acetone			Yes	No	No	SPE	MM-ML	MSD			
	Cyprodinil	Yes	0.11	10	Acetone			Yes	No	No	SPE	MM-ML	MSD			
	Fenvalerate and Esfenvalerate	No	0.13	10	Acetone			Yes	No	No	SPE	MM-ML	MSD			
	Fenbuconazole	No	0.69	10	Acetone			Yes	No	No	SPE	MM-ML	MSD			
	Fenpropimorph	No	0.12	10	Acetone			Yes	No	No	SPE	MM-ML	MSD			
	Fludioxonil	Yes	0.11	10	Acetone			Yes	No	No	SPE	MM-ML	MSD			
	Flusilazole	No	0.9	10	MeOH			Yes	No	Yes	SPE	MM-ML		MS/MS		
	Lambda-cyhalothrin	Yes	0.068	10	Acetone			Yes	No	No	SPE	MM-ML	MSD			
	Malathion	No	0.02	10	Acetone			Yes	No	No	SPE	MM-ML	MSD			
	Metconazole	No	0.476	10	MeOH			Yes	No	Yes	SPE	MM-ML		MS/MS		
	Pyraclostrobin	No	0.92	10	Acetone			Yes	No	No	SPE	MM-ML	MSD			
	Tebuconazole	No	1.8	10	Acetone			Yes	No	No	SPE	MM-ML	MSD			
eupt 009	Azoxystrobin	No	0.275	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML	ECD		2 colns	
	Carbendazim and benomyl	No	0.997	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	
	Chlorpyrifos	Yes	1.52	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML	ECD		2 colns	
	Cyproconazole	No	0.548	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML	NPD		LC-MS/MS	
	Cyprodinil	No	0.166	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML	NPD		LC-MS/MS	
	Fenvalerate and Esfenvalerate	No	0.19	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML	ECD		2 colns	
	Fenbuconazole	No	0.712	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML	ECD		2 colns	
	Fenpropimorph	No	0.21	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML	NPD		LC-MS/MS	
	Fludioxonil	No	0.12	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML	NPD		LC-MS/MS	
	Flusilazole	No	0.815	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML	NPD		LC-MS/MS	
	Lambda-cyhalothrin	Yes	0.0757	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML	ECD		2 colns	

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1)]	Extraction solvent II [1)]	Extraction solvent III [1)]	Water addition	Hydrolysis	pH adjused	Clean up [2)]	Calibration [3)]	GC detector	HPLC detector	Confirmation	Reference method
	Metconazole	No	0.642	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML	ECD		2 colns	
	Pyraclostrobin	No	1.11	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML	ECD		2 colns	
	Tebuconazole	No	1.35	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML	NPD		LC-MS/MS	
eupt 012	Chlorpyrifos	No	0.68	0	EtOAc			No	No	No	DSPE	MM-ML	MS/MS		GC-MS/MS	Laboratory internal method:and Quechers
	Fenvalerate and Esfenvalerate	No	0.111	0	EtOAc			No	No	No	DSPE	MM-ML	MS/MS		GC-MS/MS	Laboratory internal method:and Quechers
	Fenbuconazole	No	0.204	0	EtOAc			No	No	No	DSPE	MM-ML	MS/MS		GC-MS/MS	Laboratory internal method:and Quechers
	Fludioxonil	No	0.032	0	EtOAc			No	No	No	DSPE	MM-ML	MS/MS		GC-MS/MS	Laboratory internal method:and Quechers
	Lambda-cyhalothrin	No	0.067	0	EtOAc			No	No	No	DSPE	MM-ML	MS/MS		GC-MS/MS	Laboratory internal method:and Quechers
	Tebuconazole	No	0.605	0	EtOAc			No	No	No	DSPE	MM-ML	MS/MS		GC-MS/MS	Laboratory internal method:and Quechers
eupt 013	Azoxystrobin	No	0.0577	15	EtOAc						None	PS-ML	ECD		2 colns	ACETATO DE ETILO
	Chlorpyrifos	No	2,117	15	EtOAc						None	PS-ML	ECD		2 colns	ACETATO DE ETILO
	Fenvalerate and Esfenvalerate	No	0.0783	15	EtOAc						None	PS-ML	ECD		2 colns	ACETATO DE ETILO
	Lambda-cyhalothrin	No	0.0145	15	EtOAc						None	PS-ML	ECD		2 colns	ACETATO DE ETILO
eupt 014	Azoxystrobin	No	0.188	5	ACN			Yes	No	No	Freez.	PS-ML	ECD		GC/MS	QuEChERS
	Carbendazim and benomyl	No	0.55	5	ACN			Yes	No	No	Freez.	PS-ML		MS/MS	LC-MS/MS	QuEChERS
	Chlorpyrifos	No	0.846	5	ACN			Yes	No	No	Freez.	PS-ML	FPD		GC/MS	QuEChERS
	Cyproconazole	No	0.553	5	ACN			Yes	No	No	Freez.	PS-ML	NPD		GC/MS	QuEChERS
	Cyprodinil	No	0.069	5	ACN			Yes	No	No	Freez.	PS-ML	NPD		GC/MS	QuEChERS
	Fenvalerate and Esfenvalerate	No	0.071	5	ACN			Yes	No	No	Freez.	PS-ML	ECD		GC/MS	QuEChERS
	Fenbuconazole	No	0.564	5	ACN			Yes	No	No	Freez.	PS-ML	NPD		GC/MS	QuEChERS
	Fenpropimorph	No	0.123	5	ACN			Yes	No	No	Freez.	PS-ML	MSD			QuEChERS
	Fludioxonil	No	0.076	5	ACN			Yes	No	No	Freez.	PS-ML	NPD		GC/MS	QuEChERS

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Flusilazole	No	0.758	5	ACN			Yes	No	No	Freez.	PS-ML	NPD		GC/MS	QuEChERS
	Lambda-cyhalothrin	No	0.056	5	ACN			Yes	No	No	Freez.	PS-ML	ECD		GC/MS	QuEChERS
	Metconazole	No	0.636	5	ACN			Yes	No	No	Freez.	PS-ML	NPD		GC/MS	QuEChERS
	Pyraclostrobin	No	0.69	5	ACN			Yes	No	No	Freez.	PS-ML		MS/MS	LC-MS/MS	QuEChERS
	Tebuconazole	No	1,214	5	ACN			Yes	No	No	Freez.	PS-ML	NPD		GC/MS	QuEChERS
eupt 015	Azoxystrobin	No	0.192	50	Acetone	CH_2Cl_2		Yes	No	No	Other	MM-SL	ECD		None	Luke
	Chlorpyrifos	No	0.912	50	Acetone	CH_2Cl_2		Yes	No	No	Other	MM-SL	NPD		2 colns	Luke
	Cyproconazole	No	0.443	50	Acetone	CH_2Cl_2		Yes	No	No	Other	MM-SL	NPD		2 colns	Luke
	Cyprodinil	No	0.066	50	Acetone	CH_2Cl_2		Yes	No	No	Other	MM-SL	NPD		2 colns	Luke
	Fenvalerate and Esfenvalerate	No	0.115	50	Acetone	CH_2Cl_2		Yes	No	No	Other	MM-SL	ECD		None	Luke
	Fludioxonil	No	0.066	50	Acetone	CH_2Cl_2		Yes	No	No	Other	MM-SL	NPD		2 colns	Luke
	Flusilazole	No	0.697	50	Acetone	CH_2Cl_2		Yes	No	No	Other	MM-SL	NPD		2 colns	Luke
	Lambda-cyhalothrin	No	0.058	50	Acetone	CH_2Cl_2		Yes	No	No	Other	MM-SL	ECD		None	Luke
	Tebuconazole	No	1,274	50	Acetone	CH_2Cl_2		Yes	No	No	Other	MM-SL	NPD		2 colns	Luke
eupt 016	Azoxystrobin	Yes	0.134	10	Acetone	CH_2Cl_2		Yes	No	No	GPC	MM-ML	ECD		GC/MS	§ 64 LFGB BVL L 00.00-34
	Chlorpyrifos	Yes	0.83	10	Acetone	CH_2Cl_2		Yes	No	No	GPC	MM-ML	ITD		GC/MS	§ 64 LFGB BVL L 00.00-34
	Fenvalerate and Esfenvalerate	Yes	0.094	10	Acetone	CH_2Cl_2		Yes	No	No	GPC	MM-ML	ECD		2 colns	§ 64 LFGB BVL L 00.00-34
	Lambda-cyhalothrin	Yes	0.028	10	Acetone	CH_2Cl_2		Yes	No	No	GPC	MM-ML	ITD		GC/MS	§ 64 LFGB BVL L 00.00-34
eupt 018	Azoxystrobin	Yes	0.225	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	GC/MS	QuEChERS
	Carbendazim and benomyl	Yes	0.532	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	GC/MS	QuEChERS
	Chlorpyrifos	Yes	1.08	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	GC/MS	QuEChERS
	Cyproconazole	Yes	0.527	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	GC/MS	QuEChERS
	Cyprodinil	Yes	0.085	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	GC/MS	QuEChERS

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Fenvalerate and Esfenvalerate	Yes	0.096	10	ACN	Acetone		No	No		GPC	MM-ML	MSD		Other	
	Fenbuconazole	Yes	0.578	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	GC/MS	QuEChERS
	Fenpropimorph	Yes	0.126	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	GC/MS	QuEChERS
	Fludioxonil	Yes	0.087	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	GC/MS	QuEChERS
	Flusilazole	Yes	0.831	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	GC/MS	QuEChERS
	Lambda-cyhalothrin	Yes	0.033	10	ACN	Acetone		No	No		GPC	MM-ML	MSD		Other	
	Metconazole	Yes	0.582	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	GC/MS	QuEChERS
	Pyraclostrobin	Yes	0.817	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	GC/MS	QuEChERS
	Tebuconazole	Yes	1,484	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	GC/MS	QuEChERS
eupt 019	Azoxystrobin	Yes	0.16	250	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML	ECD		GC/MS	§ 64 LFGB; L00.00-34
	Carbendazim and benomyl	Yes	0.633	50	MeOH			Yes	No	No	liq./liq	MM-ML		MS/MS	LC-MS/MS	§ 64 LFGB; L00.00-34
	Chlorpyrifos	Yes	0.887	250	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML	NPD		GC/MS	§ 64 LFGB; L00.00-34
	Cyproconazole	Yes	0.361	250	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML	NPD		GC/MS	§ 64 LFGB; L00.00-34
	Cyprodinil	Yes	0.079	250	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML	NPD		GC/MS	§ 64 LFGB; L00.00-34
	Fenvalerate and Esfenvalerate	Yes	0.097	250	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML	ECD		GC/MS	§ 64 LFGB; L00.00-34
	Fenbuconazole	Yes	0.425	250	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML	ECD		GC/MS	§ 64 LFGB; L00.00-34
	Fenpropimorph	Yes	0.083	250	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML	NPD		GC/MS	§ 64 LFGB; L00.00-34
	Fludioxonil	Yes	0.063	250	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML	NPD		GC/MS	§ 64 LFGB; L00.00-34
	Flusilazole	Yes	0.617	250	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML	NPD		GC/MS	§ 64 LFGB; L00.00-34
	Lambda-cyhalothrin	Yes	0.049	250	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML	ECD		GC/MS	§ 64 LFGB; L00.00-34
	Metconazole	Yes	0.33	250	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML	NPD		GC/MS	§ 64 LFGB; L00.00-34
	Pyraclostrobin	Yes	0.567	50	MeOH			Yes	No	No	liq./liq Part	MM-ML		MS/MS	LC-MS/MS	§ 64 LFGB; L00.00-34
	Tebuconazole	Yes	0.912	250	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML	NPD		GC/MS	§ 64 LFGB; L00.00-34
eupt 021	Azoxystrobin	Yes	0.17	25	Acetone	EtOAc	C6H12	Yes	No	No	GPC	MM-ML				FP004
	Carbendazim and benomyl	Yes	0.37	3	MeOH			Yes	No	No	Other	MM-ML		MS/MS	LC-MS/MS	FP086

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1)]	Extraction solvent II [1)]	Extraction solvent III [1)]	Water addition	Hydrolysis	pH adjused	Clean up [2)]	Calibration [3)]	GC detector	HPLC detector	Confirmation	Reference method
	Chlorpyrifos	Yes	0.77	25	Acetone	EtOAc	C_6H_{12}	Yes	No	No	GPC	MM-ML				FP004
	Fenvalerate and Esfenvalerate	Yes	0.1	25	Acetone	EtOAc	C_6H_{12}	Yes	No	No	GPC	MM-ML				FP004
	Pyraclostrobin	Yes	0.37	3	MeOH			Yes	No	No	Other	MM-ML		MS/MS	LC-MS/MS	FP086
	Tebuconazole	Yes	1.03	25	Acetone	EtOAc	C_6H_{12}	Yes	No	No	GPC	MM-ML				FP004
eupt 022	Carbendazim and benomyl	No	0.683	5	ACN			Yes		Yes		MM-ML		MS/MS	LC-MS/MS	QuEChERS
	Chlorpyrifos	No	0.931	5	ACN			Yes		Yes		MM-ML		MS/MS	LC-MS/MS	QuEChERS
	Cyproconazole	No	0.518	5	ACN			Yes		Yes		MM-ML		MS/MS	LC-MS/MS	QuEChERS
	Cyprodinil	No	0.0926	5	ACN			Yes		Yes		MM-ML		MS/MS	LC-MS/MS	QuEChERS
	Fenbuconazole	No	0.577	5	ACN			Yes		Yes		MM-ML		MS/MS	LC-MS/MS	QuEChERS
	Fenpropimorph	No	0.121	5	ACN			Yes		Yes		MM-ML		MS/MS	LC-MS/MS	QuEChERS
	Fludioxonil	No	0.0987	5	ACN			Yes		Yes		MM-ML		MS/MS	LC-MS/MS	QuEChERS
	Flusilazole	No	0.776	5	ACN			Yes		Yes		MM-ML		MS/MS	LC-MS/MS	QuEChERS
	Metconazole	No	0.611	5	ACN			Yes		Yes		MM-ML		MS/MS	LC-MS/MS	QuEChERS
	Pyraclostrobin	No	0.81	5	ACN			Yes		Yes		MM-ML		MS/MS	LC-MS/MS	QuEChERS
eupt 024	Azoxystrobin	Yes	0.12	20	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	MM-ML	MSD		GC/MS	Mini-Luke
	Carbendazim and benomyl	Yes	0.35	0												
	Chlorpyrifos	Yes	1.05	20	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	MM-ML	MSD		GC/MS	Mini-Luke
	Cyproconazole	Yes	0.36	20	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	MM-ML	MSD		GC/MS	Mini-Luke
	Cyprodinil	Yes	0.072	0												
	Fenvalerate and Esfenvalerate	Yes	0.347	20	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	MM-ML	MSD		GC/MS	Mini-Luke
	Fenbuconazole	Yes	0.4	20	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	MM-ML	MSD		GC/MS	Mini-Luke
	Fenpropimorph	Yes	0.13	0												
	Fludioxonil	Yes	0.07	0												
	Flusilazole	Yes	0.62	20	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	MM-ML	MSD		GC/MS	Mini-Luke
	Lambda-cyhalothrin	Yes	0.038	20	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	MM-ML	MSD		GC/MS	Mini-Luke

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Metconazole	Yes	0.39	0												
	Pyraclostrobin	Yes	0.47	0												
	Tebuconazole	Yes	1.22	20	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	MM-ML	MSD		GC/MS	Mini-Luke
eupt 025	Carbendazim and benomyl	No	0.35	75	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	PS-ML		Diode Array Det.	None	LUKE
	Chlorpyrifos	No	0.499	75	Acetone	CH_2Cl_2	Other	No	No	No	None	MM-ML	MS/MS		GC-MS/MS	LUKE
eupt 026	Azoxystrobin	Yes	0.185	25	ACN			Yes	No	No	SPE	PS-ML	MS/MS		GC-MS/MS	NF 12393
	Carbendazim and benomyl	Yes	0.136	0												
	Chlorpyrifos	Yes	0.735	25	ACN			Yes	No	No	SPE	PS-ML	MS/MS		GC-MS/MS	NF 12393
	Cyproconazole	No	0.441	25	ACN			Yes	No	No	SPE	PS-ML	MS/MS		GC-MS/MS	NF 12393
	Cyprodinil	Yes	0.058	25	ACN			Yes	No	No	SPE	PS-ML	MS/MS		GC-MS/MS	NF 12393
	Fenvalerate and Esfenvalerate	No	0.032	25	ACN			Yes	No	No	SPE	PS-ML	MS/MS		GC-MS/MS	NF 12393
	Fenbuconazole	No	0.659	25	ACN			Yes	No	No	SPE	PS-ML	MS/MS		GC-MS/MS	NF 12393
	Fenpropimorph	No	0.183	25	ACN			Yes	No	No	SPE	PS-ML	MS/MS		GC-MS/MS	NF 12393
	Fludioxonil	Yes	0.078	25	ACN			Yes	No	No	SPE	PS-ML	MS/MS		GC-MS/MS	NF 12393
	Flusilazole	No	0.692	25	ACN			Yes	No	No	SPE	PS-ML	MS/MS		GC-MS/MS	NF 12393
	Lambda-cyhalothrin	Yes	0.055	25	ACN			Yes	No	No	SPE	PS-ML	MS/MS		GC-MS/MS	NF 12393
	Metconazole	No	0.465	25	ACN			Yes	No	No	SPE	PS-ML	MS/MS		GC-MS/MS	NF 12393
	Pyraclostrobin	No	1.28	25	ACN			Yes	No	No	SPE	PS-ML	MS/MS		GC-MS/MS	NF 12393
	Tebuconazole	No	1.44	25	ACN			Yes	No	No	SPE	PS-ML	MS/MS		GC-MS/MS	NF 12393
eupt 028	Azoxystrobin	No	0.153	5	ACN			Yes		Yes	SPE	MM-ML	ITD		GC-MS/MS	QuEChERS
	Carbendazim and benomyl	No	1.15	5	ACN			Yes		Yes	SPE	MM-ML		MS/MS	LC-MS/MS	QuEChERS
	Chlorpyrifos	No	1.05	5	ACN			Yes		Yes	SPE	MM-ML	ITD		GC-MS/MS	QuEChERS
	Cyprodinil	No	0.068	5	ACN			Yes		Yes	SPE	MM-ML	ITD		GC-MS/MS	QuEChERS
	Fenvalerate and Esfenvalerate	No	0.088	5	ACN			Yes		Yes	SPE	MM-ML	ITD		GC-MS/MS	QuEChERS

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Lambda-cyhalothrin	No	0.034	5	ACN			Yes		Yes	SPE	MM-ML	ITD		GC-MS/MS	QuEChERS
	Tebuconazole	No	1.73	5	ACN			Yes		Yes	SPE	MM-ML	ITD		GC-MS/MS	QuEChERS
eupt 029	Azoxystrobin	Yes	0.195	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	PS-ML		MS/MS	LC-MS/MS	LMS
	Carbendazim and benomyl	Yes	0.632	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	PS-ML		MS/MS	LC-MS/MS	LMS
	Chlorpyrifos	Yes	0.893	5	Acetone	EtOAc	Other	Yes	No	No	None	MM-ML	MS/MS		GC-MS/MS	GMS
	Cyproconazole	Yes	0.553	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	PS-ML		MS/MS	LC-MS/MS	LMS
	Cyprodinil	Yes	0.087	5	Acetone	EtOAc	Other	Yes	No	No	None	MM-ML	MS/MS		GC-MS/MS	GMS
	Fenvalerate and Esfenvalerate	Yes	0.28	5	Acetone	EtOAc	Other	Yes	No	No	None	MM-ML	MS/MS		GC-MS/MS	GMS
	Fenbuconazole	Yes	0.508	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	PS-ML		MS/MS	LC-MS/MS	LMS
	Fenpropimorph	Yes	0.146	5	Acetone	EtOAc	Other	Yes	No	No	None	MM-ML	MS/MS		GC-MS/MS	GMS
	Fludioxonil	Yes	0.079	5	Acetone	EtOAc	Other	Yes	No	No	None	MM-ML	MS/MS		GC-MS/MS	GMS
	Flusilazole	Yes	0.728	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	PS-ML		MS/MS	LC-MS/MS	LMS
	Lambda-cyhalothrin	Yes	0.05	5	Acetone	EtOAc	Other	Yes	No	No	None	MM-ML	MS/MS		GC-MS/MS	GMS
	Malathion	Yes	0.01	5	Acetone	EtOAc	Other	Yes	No	No	None	MM-ML	MS/MS		GC-MS/MS	GMS
	Metconazole	Yes	0.426	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	PS-ML		MS/MS	LC-MS/MS	LMS
	Pyraclostrobin	Yes	0.687	5	Acetone	EtOAc	Other	Yes	No	No	None	MM-ML	MS/MS		GC-MS/MS	GMS
	Tebuconazole	Yes	1,349	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	PS-ML		MS/MS	LC-MS/MS	LMS
eupt 030	Azoxystrobin	No	0.134	10	EtOAc			Yes	No	No	None	MM-ML	ECD	MS/MS	LC-MS/MS	1
	Carbendazim and benomyl	No	0.461	10	EtOAc			Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	2
	Chlorpyrifos	Yes	0.905	10	EtOAc			Yes	No	No	GPC	MM-ML	FPD		GC/MS	3
	Cyproconazole	No	0.377	10	EtOAc			Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	2
	Cyprodinil	Yes	0.0578	10	EtOAc			Yes	No	No	GPC	MM-ML	ITD	MS/MS	LC-MS/MS	4
	Fenvalerate and Esfenvalerate	Yes	0.0772	10	EtOAc			Yes	No	No	None	MM-ML	ECD		2 colns	5
	Fenbuconazole	No	0.368	10	EtOAc			Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	2
	Fenpropimorph	No	0.0986	10	EtOAc			Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	2

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Fludioxonil	No	0.0577	10	EtOAc			Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	2
	Flusilazole	No	0.576	10	EtOAc			Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	2
	Lambda-cyhalothrin	Yes	0.0396	10	EtOAc			Yes	No	No	None	MM-ML	ECD		2 colns	5
	Metconazole	No	0.428	10	EtOAc			Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	2
	Pyraclostrobin	No	0.634	10	EtOAc			Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	2
	Tebuconazole	No	1.1	10	EtOAc			Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	2
eupt 031	Azoxystrobin	Yes	0.175	5	ACN			Yes	No	No	DSPE	MM-ML	NPD		GC/MS	EN 12393-1.2.3:1998
	Carbendazim and benomyl	Yes	0.313	5	ACN			Yes	No	No	Freez.	PS-ML		MS/MS	LC-MS/MS	prEN15662:2007
	Chlorpyrifos	Yes	0.91	5	ACN			Yes	No	No	DSPE	MM-ML	NPD		GC/MS	EN 12393-1.2.3:1998
	Cyproconazole	Yes	0.42	5	ACN			Yes	No	No	DSPE	MM-ML	NPD		GC/MS	EN 12393-1.2.3:1998
	Cyprodinil	Yes	0.067	5	ACN			Yes	No	No	DSPE	MM-ML	NPD		GC/MS	EN 12393-1.2.3:1998
	Fenvalerate and Esfenvalerate	Yes	0.0925	5	ACN			Yes	No	No	DSPE	MM-ML	NPD		GC/MS	EN 12393-1.2.3:1998
	Fenbuconazole	No	0.47	5	ACN			Yes	No	No	DSPE	MM-ML	NPD		GC/MS	EN 12393-1.2.3:1998
	Fenpropimorph	Yes	0.0585	5	ACN			Yes	No	No	DSPE	MM-ML	NPD		GC/MS	EN 12393-1.2.3:1998
	Fludioxonil	Yes	0.0775	5	ACN			Yes	No	No	DSPE	MM-ML	NPD		GC/MS	EN 12393-1.2.3:1998
	Flusilazole	Yes	0.76	5	ACN			Yes	No	No	DSPE	MM-ML	NPD		GC/MS	EN 12393-1.2.3:1998
	Lambda-cyhalothrin	Yes	0.052	5	ACN			Yes	No	No	DSPE	MM-ML	NPD		GC/MS	EN 12393-1.2.3:1998
	Malathion	Yes	0.0102	5	ACN			Yes	No	No	DSPE	MM-ML	NPD		GC/MS	EN 12393-1.2.3:1998
	Metconazole	Yes	0.455	5	ACN			Yes	No	No	DSPE	MM-ML	NPD		GC/MS	EN 12393-1.2.3:1998
	Pyraclostrobin	Yes	0.852	5	ACN			Yes	No	No	DSPE	MM-ML	NPD		GC/MS	EN 12393-1.2.3:1998
	Tebuconazole	Yes	1.14	5	ACN			Yes	No	No	DSPE	MM-ML	NPD		GC/MS	EN 12393-1.2.3:1998
eupt 032	Azoxystrobin	No	0.187	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	LC-MS/MS	
	Carbendazim and benomyl	No	0.563	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	LC-MS/MS	
	Chlorpyrifos	No	1,178	5	ACN			Yes	No	Yes	DSPE	MM-ML	MSD		GC/MS	
	Cyproconazole	No	0.356	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	LC-MS/MS	
	Cyprodinil	No	0.076	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	LC-MS/MS	

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Fenvalerate and Esfenvalerate	No	0.093	5	ACN			Yes	No	Yes	DSPE	MM-ML	MSD		GC/MS	
	Fenbuconazole	No	0.555	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	LC-MS/MS	
	Fenpropimorph	No	0.122	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	LC-MS/MS	
	Fludioxonil	No	0.081	5	ACN			Yes	No	Yes	Freez.	MM-ML		MS/MS	LC-MS/MS	
	Flusilazole	No	0.879	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	LC-MS/MS	
	Lambda-cyhalothrin	No	0.05	5	ACN			Yes	No	Yes	DSPE	MM-ML	MSD		GC/MS	
	Metconazole	No	0.596	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	LC-MS/MS	
	Pyraclostrobin	No	0.843	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	LC-MS/MS	
	Tebuconazole	No	1.45	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	LC-MS/MS	
eupt 033	Azoxystrobin	Yes	0.0493	20	Acetone	EtOAc	C_6H_{12}	Yes	No	No	None	MM-ML		MS	GC/MS	ASU L00.00-34 mod.
	Carbendazim and benomyl	Yes	0.131	20	Acetone	EtOAc	C_6H_{12}	Yes	No	No	None	MM-ML		MS	GC/MS	ASU L00.00-34 mod.
	Chlorpyrifos	Yes	1.27	20	Acetone	EtOAc	C_6H_{12}	Yes	No	No	GPC	MM-ML	MSD		GC/MS	ASU L00.00-34 mod.
	Cyproconazole	Yes	0.221	20	Acetone	EtOAc	C_6H_{12}	Yes	No	No	None	MM-ML		MS	GC/MS	ASU L00.00-34 mod.
	Cyprodinil	Yes	0.0377	20	Acetone	EtOAc	C_6H_{12}	Yes	No	No	None	MM-ML		MS	GC/MS	ASU L00.00-34 mod.
	Fenvalerate and Esfenvalerate	Yes	0.067	20	Acetone	EtOAc	C_6H_{12}	Yes	No	No	GPC	MM-ML	MSD		GC/MS	ASU L00.00-34 mod.
	Fenbuconazole	Yes	0.209	20	Acetone	EtOAc	C_6H_{12}	Yes	No	No	GPC	MM-ML	MSD		GC/MS	ASU L00.00-34 mod.
	Fenpropimorph	Yes	0.137	5	ACN			Yes	No	No	Freez.	MM-ML	MSD			ASU L00.00-115 mod.
	Fludioxonil	Yes	0.0532	20	Acetone	EtOAc	C_6H_{12}	Yes	No	No	None	MM-ML		MS	GC/MS	ASU L00.00-34 mod.
	Flusilazole	Yes	0.388	20	Acetone	EtOAc	C_6H_{12}	Yes	No	No	GPC	MM-ML	MSD		GC/MS	ASU L00.00-34 mod.
	Lambda-cyhalothrin	Yes	0.0408	5	ACN			Yes	No	No	Freez.	MM-ML	MSD			ASU L00.00-115 mod.
	Metconazole	Yes	0.251	20	Acetone	EtOAc	C_6H_{12}	Yes	No	No	None	MM-ML		MS	GC/MS	ASU L00.00-34 mod.
	Pyraclostrobin	Yes	0.477	20	Acetone	EtOAc	C_6H_{12}	Yes	No	No	GPC	MM-ML	MSD		GC/MS	ASU L00.00-34 mod.
	Tebuconazole	Yes	0.661	20	Acetone	EtOAc	C_6H_{12}	Yes	No	No	None	MM-ML		MS	GC/MS	ASU L00.00-34 mod.
eupt 034	Azoxystrobin	Yes	0.209	5	ACN			Yes	No	No	DSPE	MM-ML	MSD	MS/MS	GC/MS	QueChERS
	Carbendazim and benomyl	Yes	0.582	5	ACN			Yes	No	No	SPE	MM-ML		MS/MS	None	QueChERS

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1)]	Extraction solvent II [1)]	Extraction solvent III [1)]	Water addition	Hydrolysis	pH adjused	Clean up [2)]	Calibration [3)]	GC detector	HPLC detector	Confirmation	Reference method
	Chlorpyrifos	Yes	1.19	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		GC/MS	QueChERS
	Cyproconazole	Yes	0.499	5	ACN			Yes	No	No	DSPE	MM-ML	MSD	MS/MS	GC/MS	QueChERS
	Cyprodinil	Yes	0.086	5	ACN			Yes	No	No	DSPE	MM-ML	MSD	MS/MS	GC/MS	QueChERS
	Fenvalerate and Esfenvalerate	Yes	0.113	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		GC/MS	QueChERS
	Fenbuconazole	Yes	0.59	5	ACN			Yes	No	No	DSPE	MM-ML	MSD	MS/MS	GC/MS	QueChERS
	Fenpropimorph	Yes	0.134	5	ACN			Yes	No	No	DSPE	MM-ML	MSD	MS/MS	GC/MS	QueChERS
	Fludioxonil	Yes	0.089	5	ACN			Yes	No	No	None	MM-ML	MSD	MS/MS	GC/MS	without DSPE QueChERS
	Flusilazole	Yes	0.863	5	ACN			Yes	No	No	DSPE	MM-ML	MSD	MS/MS	GC/MS	QueChERS
	Lambda-cyhalothrin	Yes	0.054	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		GC/MS	QueChERS
	Metconazole	Yes	0.538	5	ACN			Yes	No	No	DSPE	MM-ML	MSD	MS/MS	GC/MS	QueChERS
	Pyraclostrobin	Yes	0.932	5	ACN			Yes	No	No	DSPE	MM-ML	MSD	MS/MS	GC/MS	QueChERS
	Tebuconazole	Yes	1.54	5	ACN			Yes	No	No	DSPE	MM-ML	MSD	MS/MS	GC/MS	QueChERS
eupt 035	Azoxystrobin	Yes	0.167	10	Acetone	CH_2Cl_2	EtOAc	Yes	No	No	GPC	MM-ML	ECD			
	Carbendazim and benomyl	Yes	0.255	15	EtOAc	EtOAc	Methanol	Yes	No	pH 13	None	PS-ML		FLUOR	Other	
	Chlorpyrifos	Yes	0.851	10	Acetone	CH_2Cl_2	EtOAc	Yes	No	No	GPC	MM-ML	ECD			
	Fenvalerate and Esfenvalerate	No	0.092	10	Acetone	CH_2Cl_2	EtOAc	Yes	No	No	GPC	MM-ML	ECD			
	Flusilazole	Yes	0.747	10	Acetone	CH_2Cl_2	EtOAc	Yes	No	No	GPC	MM-ML	NPD			
	Lambda-cyhalothrin	Yes	0.048	10	Acetone	CH_2Cl_2	EtOAc	Yes	No	No	GPC	MM-ML	ECD			
	Tebuconazole	Yes	1,087	10	Acetone	CH_2Cl_2	EtOAc	Yes	No	No	GPC	MM-ML	NPD			
eupt 036	Azoxystrobin	No	0.16	20	Acetone	Other		No	No	No	SPE	MM-SL	ECD			Luke
	Chlorpyrifos	No	1.1	20	Acetone	Other		No	No	No	SPE	MM-SL	ECD			Luke
	Cyproconazole	No	0.35	20	Acetone	Other		No	No	No	SPE	MM-SL	NPD			Luke
	Cyprodinil	No	0.08	20	Acetone	Other		No	No	No	SPE	MM-SL	NPD			Luke
	Flusilazole	No	0.45	20	Acetone	Other		No	No	No	SPE	MM-SL	NPD			Luke
	Lambda-cyhalothrin	No	0.06	20	Acetone	Other		No	No	No	SPE	MM-SL	ECD			Luke

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Tebuconazole	No	0.9	20	Acetone	Other		No	No	No	SPE	MM-SL	NPD			Luke
eupt 037	Azoxystrobin	Yes	0.159	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	eupt037	QuEChERS
	Carbendazim and benomyl	Yes	0.44	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	eupt037	QuEChERS
	Chlorpyrifos	Yes	1.13	5	ACN			Yes	No	No	DSPE	MM-ML	TOF		eupt037	QuEChERS
	Cyproconazole	Yes	0.527	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	eupt037	QuEChERS
	Cyprodinil	Yes	0.055	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	eupt037	QuEChERS
	Fenvalerate and Esfenvalerate	Yes	0.077	5	ACN			Yes	No	No	DSPE	MM-ML	TOF		eupt037	QuEChERS
	Fenbuconazole	Yes	0.527	5	ACN			Yes	No	No	DSPE	MM-ML	TOF		eupt037	QuEChERS
	Fenpropimorph	Yes	0.144	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	eupt037	QuEChERS
	Fludioxonil	Yes	0.068	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	eupt037	QuEChERS
	Flusilazole	Yes	0.732	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	eupt037	QuEChERS
	Lambda-cyhalothrin	Yes	0.051	5	ACN			Yes	No	No	DSPE	MM-ML	TOF		eupt037	QuEChERS
	Metconazole	Yes	0.48	5	ACN			Yes	No	No	DSPE	MM-ML	TOF		eupt037	QuEChERS
	Pyraclostrobin	Yes	0.745	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	eupt037	QuEChERS
	Tebuconazole	Yes	1.3	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	eupt037	QuEChERS
eupt 038	Azoxystrobin	Yes	0.187	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	eupt038	QuEChERS
	Carbendazim and benomyl	Yes	0.723	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	eupt038	QuEChERS
	Chlorpyrifos	Yes	1.17	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	eupt038	QuEChERS
	Cyproconazole	Yes	0.574	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	eupt038	QuEChERS
	Cyprodinil	Yes	0.0859	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	eupt038	QuEChERS
	Fenvalerate and Esfenvalerate	Yes	0.1126	50	MeOH	CH_2Cl_2		Yes	No	No	GPC	MM-ML	ECD		eupt038	DFG S 19
	Fenbuconazole	Yes	0.626	50	MeOH	CH_2Cl_2		Yes	No	No	GPC	MM-ML	MSD		eupt038	DFG S 19
	Fenpropimorph	Yes	0.124	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	eupt038	QuEChERS
	Fludioxonil	Yes	0.0952	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	eupt038	QuEChERS
	Flusilazole	Yes	0.848	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	eupt038	QuEChERS

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Lambda-cyhalothrin	Yes	0.0633	50	MeOH	CH_2Cl_2		Yes	No	No	GPC	MM-ML	ECD		eupt038	DFG S 19
	Tebuconazole	Yes	1.51	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	eupt038	QuEChERS
eupt 040	Azoxystrobin	Yes	0.219	5	ACN			Yes	No	pH 5	DSPE	PS-ML	MSD		eupt040	QuEChERS
	Carbendazim and benomyl	Yes	0.491	5	ACN			Yes	No	pH 5	DSPE	PS-ML		MS/MS	eupt040	QuEChERS
	Chlorpyrifos	Yes	0.887	5	ACN			Yes	No	pH 5	DSPE	PS-ML	MSD		eupt040	QuEChERS
	Cyproconazole	Yes	0.462	5	ACN			Yes	No	pH 5	DSPE	PS-ML		MS/MS	eupt040	QuEChERS
	Cyprodinil	Yes	0.084	5	ACN			Yes	No	pH 5	DSPE	PS-ML	MSD		eupt040	QuEChERS
	Fenvalerate and Esfenvalerate	No	0.145	5	ACN			Yes	No	pH 5	DSPE	PS-SL	MSD		eupt040	QuEChERS
	Fenbuconazole	Yes	0.485	5	ACN			Yes	No	pH 5	DSPE	PS-ML		MS/MS	eupt040	QuEChERS
	Fenpropimorph	Yes	0.103	5	ACN			Yes	No	pH 5	DSPE	PS-ML		MS/MS	eupt040	QuEChERS
	Fludioxonil	No	0.078	5	ACN			Yes	No	pH 5	DSPE	PS-ML		MS/MS	eupt040	QuEChERS
	Flusilazole	Yes	0.766	5	ACN			Yes	No	pH 5	DSPE	PS-ML		MS/MS	eupt040	QuEChERS
	Lambda-cyhalothrin	Yes	0.038	5	ACN			Yes	No	pH 5	DSPE	PS-ML	MSD		eupt040	QuEChERS
	Metconazole	Yes	0.506	5	ACN			Yes	No	pH 5	DSPE	PS-ML		MS/MS	eupt040	QuEChERS
	Pyraclostrobin	Yes	0.733	5	ACN			Yes	No	pH 5	DSPE	PS-ML		MS/MS	eupt040	QuEChERS
	Tebuconazole	No	1.29	5	ACN			Yes	No	pH 5	DSPE	PS-ML		MS/MS	eupt040	QuEChERS
eupt 041	Carbendazim and benomyl	No	0.443	8	Acetone	CH_2Cl_2	Other	No	No	No	SPE	PS-SL		MS/MS	Other	SIST EN 14333-1:2005 modified
	Cyproconazole	No	0.217	20	Acetone	CH_2Cl_2	Other	No	No	No	GPC	MM-ML		MS/MS	LC-MS/MS	
	Fenbuconazole	No	0.205	20	Acetone	CH_2Cl_2	Other	No	No	No	GPC	MM-ML		MS/MS	LC-MS/MS	
	Fenpropimorph	No	0.066	20	Acetone	CH_2Cl_2	Other	No	No	No	GPC	MM-ML		MS/MS	LC-MS/MS	
	Pyraclostrobin	No	0.349	20	Acetone	CH_2Cl_2	Other	No	No	No	GPC	MM-ML		MS/MS	LC-MS/MS	
eupt 042	Carbendazim and benomyl	Yes	0.021	5	MeOH	Other		No	No	No	liq./liq	MM-ML	MS/MS	MS/MS	LC-MS/MS	
	Chlorpyrifos	Yes	1.3	8	Acetone	CH_2Cl_2		No	No	No	SPE	MM-ML	MS/MS		GC-MS/MS	L.00.0034
	Cyprodinil	Yes	0.094	8	Acetone	CH_2Cl_2		No	No	No	SPE	MM-ML	MS/MS		GC-MS/MS	L.00.0034
	Fenvalerate and Esfenvalerate	Yes	0.11	8	Acetone	CH_2Cl_2		No	No	No	SPE	MM-ML	MS/MS		GC-MS/MS	L.00.0034

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Fludioxonil	Yes	0.08	8	Acetone	CH_2Cl_2		No	No	No	SPE	MM-ML	MS/MS		GC-MS/MS	L.00.0034
	Flusilazole	Yes	0.65	8	Acetone	CH_2Cl_2		No	No	No	SPE	MM-ML	MS/MS		GC-MS/MS	L.00.0034
	Lambda-cyhalothrin	Yes	0.053	8	Acetone	CH_2Cl_2		No	No	No	SPE	MM-ML	MS/MS		GC-MS/MS	L.00.0034
	Metconazole	Yes	0.37	8	Acetone	CH_2Cl_2		No	No	No	SPE	MM-ML	MS/MS		GC-MS/MS	L.00.0034
	Tebuconazole	Yes	1.2	8	Acetone	CH_2Cl_2		No	No	No	SPE	MM-ML	MS/MS		GC-MS/MS	L.00.0034
eupt 043	Carbendazim and benomyl	Yes	0.51	10	EtOAc			Yes		pH 10	GPC	Std-Add.		DAD	Other	EN 12393
	Chlorpyrifos	Yes	0.762	5	ACN			Yes		No	DSPE	Std-Add.	MS/MS		GC-MS/MS	QuEChERS
	Cyproconazole	Yes	0.393	5	ACN			Yes		No	DSPE	Std-Add.	MS/MS		GC-MS/MS	QuEChERS
	Cyprodinil	Yes	0.079	5	ACN			Yes		No	DSPE	Std-Add.	MS/MS		GC-MS/MS	QuEChERS
	Fenvalerate and Esfenvalerate	Yes	0.067	5	ACN			Yes		No	DSPE	Std-Add.	ECD		GC/MS	QuEChERS
	Fenbuconazole	No	0.163	5	ACN			Yes		No	Freez.	Std-Add.		MS/MS	LC-MS/MS	QuEChERS
	Fenpropimorph	Yes	0.118	5	ACN			Yes		No	DSPE	Std-Add.	MS/MS		GC-MS/MS	QuEChERS
	Fludioxonil	Yes	0.064	5	ACN			Yes		No	DSPE	Std-Add.	MS/MS		GC-MS/MS	QuEChERS
	Flusilazole	Yes	0.688	5	ACN			Yes		No	DSPE	Std-Add.	MS/MS		GC-MS/MS	QuEChERS
	Lambda-cyhalothrin	Yes	0.04	5	ACN			Yes		No	DSPE	Std-Add.	ECD		GC/MS	QuEChERS
	Metconazole	Yes	0.412	5	ACN			Yes		No	DSPE	Std-Add.	MS/MS		GC-MS/MS	QuEChERS
	Pyraclostrobin	No	0.43	5	ACN			Yes		No	Freez.	MM-ML		MS/MS	LC-MS/MS	QuEChERS
	Tebuconazole	Yes	0.962	5	ACN			Yes		No	DSPE	Std-Add.	MS/MS		GC-MS/MS	QuEChERS
eupt 044	Azoxystrobin	Yes	0.213	15	ACN			Yes	No	Yes	Other	MM-SL		MS/MS	LC-MS/MS	SLV M915
	Carbendazim and benomyl	Yes	0.589	15	ACN			Yes	No	Yes	Other	MM-SL		MS/MS	LC-MS/MS	SLV M915
	Chlorpyrifos	Yes	0.924	48	EtOAc			Yes	No	No	Other	MM-SL	MS/MS		GC-MS/MS	SLV M916
	Cyproconazole	No	0.576	15	ACN			Yes	No	Yes	Other	MM-SL		MS/MS	LC-MS/MS	SLV M915
	Cyprodinil	No	0.0626	48	EtOAc			Yes	No	No	Other	MM-SL	MS/MS		GC-MS/MS	SLV M916
	Fenvalerate and Esfenvalerate	Yes	0.1	48	EtOAc			Yes	No	No	Other	MM-SL	MS/MS		GC-MS/MS	SLV M916
	Fenbuconazole	Yes	0.648	15	ACN			Yes	No	Yes	Other	MM-SL		MS/MS	LC-MS/MS	SLV M915

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Fenpropimorph	Yes	0.125	15	ACN			Yes	No	Yes	Other	MM-SL		MS/MS	LC-MS/MS	SLV M915
	Fludioxonil	No	0.0641	48	EtOAc			Yes	No	No	Other	MM-SL	MS/MS		GC-MS/MS	SLV M916
	Flusilazole	Yes	0.928	15	ACN			Yes	No	Yes	Other	MM-SL		MS/MS	LC-MS/MS	SLV M915
	Lambda-cyhalothrin	Yes	0.0506	48	EtOAc			Yes	No	No	Other	MM-SL	MS/MS		GC-MS/MS	SLV M916
	Malathion	Yes	0.0077	48	EtOAc			Yes	No	No	Other	MM-SL	MS/MS		GC-MS/MS	SLV M916
	Metconazole	No	0.631	15	ACN			Yes	No	Yes	Other	MM-SL		MS/MS	LC-MS/MS	SLV M915
	Pyraclostrobin	Yes	0.897	48	EtOAc			Yes	No	No	Other	MM-SL	MS/MS		GC-MS/MS	SLV M916
	Tebuconazole	Yes	1.53	15	ACN			Yes	No	Yes	Other	MM-SL		MS/MS	LC-MS/MS	SLV M915
eupt 046	Azoxystrobin	Yes	0.172	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		LC-MS/MS	QuEChERS
	Carbendazim and benomyl	Yes	0.404	5	ACN			Yes	No	No	Freez.	MM-ML		MS/MS	None	QuEChERS
	Chlorpyrifos	Yes	0.929	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		LC-MS/MS	QuEChERS
	Cyproconazole	Yes	0.408	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		None	QuEChERS
	Cyprodinil	Yes	0.072	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		None	QuEChERS
	Fenvalerate and Esfenvalerate	Yes	0.091	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		None	QuEChERS
	Fenbuconazole	Yes	0.512	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		LC-MS/MS	QuEChERS
	Fenpropimorph	Yes	0.162	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		LC-MS/MS	QuEChERS
	Fludioxonil	Yes	0.078	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		LC-MS/MS	QuEChERS
	Flusilazole	Yes	0.593	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		LC-MS/MS	QuEChERS
	Lambda-cyhalothrin	Yes	0.049	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		None	QuEChERS
	Metconazole	Yes	0.398	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		None	QuEChERS
	Pyraclostrobin	Yes	0.784	2	ACN			Yes	No	No	Freez.	Std-Add.		MS/MS	None	QuEChERS
	Tebuconazole	Yes	1,152	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		LC-MS/MS	QuEChERS
eupt 047	Azoxystrobin	Yes	0.224	2	ACN			Yes	No	No	Freez.	MM-SL	ECD		LC-MS/MS	ASU § 64 LFGB L00.00-115
	Carbendazim and benomyl	Yes	0.578	2	ACN			Yes	No	No	Freez.	MM-SL		MS/MS	LC-MS/MS	ASU § 64 LFGB L00.00-115
	Chlorpyrifos	Yes	1,126	2	ACN			Yes	No	No	Freez.	MM-SL	ECD		GC/MS	ASU § 64 LFGB L00.00-115

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1)]	Extraction solvent II [1)]	Extraction solvent III [1)]	Water addition	Hydrolysis	pH adjused	Clean up [2)]	Calibration [3)]	GC detector	HPLC detector	Confirmation	Reference method
	Cyproconazole	Yes	0.494	2	ACN			Yes	No	No	Freez.	MM-SL	MSD		LC-MS/MS	ASU § 64 LFGB L00.00-115
	Cyprodinil	Yes	0.089	2	ACN			Yes	No	No	Freez.	MM-SL	MSD		LC-MS/MS	ASU § 64 LFGB L00.00-115
	Fenvalerate and Esfenvalerate	Yes	0.115	2	ACN			Yes	No	No	Freez.	MM-SL	ECD		GC/MS	ASU § 64 LFGB L00.00-115
	Fenbuconazole	Yes	0.64	2	ACN			Yes	No	No	Freez.	MM-SL		MS/MS	LC-MS/MS	ASU § 64 LFGB L00.00-115
	Fenpropimorph	Yes	0.169	2	ACN			Yes	No	No	Freez.	MM-SL	MSD		LC-MS/MS	ASU § 64 LFGB L00.00-115
	Fludioxonil	Yes	0.112	2	ACN			Yes	No	No	Freez.	MM-SL		MS/MS	LC-MS/MS	ASU § 64 LFGB L00.00-115
	Flusilazole	Yes	0.9	2	ACN			Yes	No	No	Freez.	MM-SL		MS/MS	LC-MS/MS	ASU § 64 LFGB L00.00-115
	Lambda-cyhalothrin	Yes	0.049	2	ACN			Yes	No	No	Freez.	MM-SL	ECD		GC/MS	ASU § 64 LFGB L00.00-115
	Metconazole	Yes	0.788	2	ACN			Yes	No	No	Freez.	MM-SL		MS/MS	LC-MS/MS	ASU § 64 LFGB L00.00-115
	Pyraclostrobin	Yes	0.827	2	ACN			Yes	No	No	Freez.	MM-SL		MS/MS	LC-MS/MS	ASU § 64 LFGB L00.00-115
	Tebuconazole	Yes	1.62	2	ACN			Yes	No	No	Freez.	MM-SL		MS/MS	LC-MS/MS	ASU § 64 LFGB L00.00-115
eupt 048	Carbendazim and benomyl	No	0.852	5	MeOH			Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	
	Chlorpyrifos	No	1.06	20	Acetone			Yes	No	No	liq./liq	MM-ML	MS/MS		GC-MS/MS	
	Cyproconazole	No	0.447	5	MeOH			Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	
	Cyprodinil	No	0.064	5	MeOH			Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	
	Fenvalerate and Esfenvalerate	No	0.08	20	Acetone			Yes	No	No	liq./liq	MM-ML	MS/MS		GC-MS/MS	
	Fenbuconazole	No	0.498	5	MeOH			Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	
	Fenpropimorph	No	0.104	5	MeOH			Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	
	Fludioxonil	No	0.059	5	MeOH			Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Flusilazole	No	0.672	20	Acetone			Yes	No	No	liq./liq	MM-ML	MS/MS		GC-MS/MS	
	Metconazole	No	0.537	5	MeOH			Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	
	Pyraclostrobin	No	0.772	5	MeOH			Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	
	Tebuconazole	No	1.05	5	MeOH			Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	
eupt 049	Azoxystrobin	Yes	0.202	5	ACN			Yes	No	pH 5	DSPE	MM-ML		MS/MS	GC/MS	QuEChERS
	Carbendazim and benomyl	Yes	0.569	5	ACN			Yes	No	pH 5	DSPE	MM-ML		MS/MS		QuEChERS
	Chlorpyrifos	Yes	1,055	5	ACN			Yes	No	pH 5	DSPE	MM-ML	FPD		GC-MS/MS	QuEChERS
	Cyproconazole	Yes	0.488	5	ACN			Yes	No	pH 5	DSPE	MM-ML		MS/MS	GC/MS	QuEChERS
	Cyprodinil	Yes	0.079	5	ACN			Yes	No	pH 5	DSPE	MM-ML		MS/MS	GC/MS	QuEChERS
	Fenvalerate and Esfenvalerate	Yes	0.123	5	ACN			Yes	No	pH 5	DSPE	MM-ML	MSD		GC/MS	QuEChERS
	Fenbuconazole	Yes	0.542	5	ACN			Yes	No	pH 5	DSPE	MM-ML		MS/MS	GC/MS	QuEChERS
	Fenpropimorph	Yes	0.131	5	ACN			Yes	No	pH 5	DSPE	MM-ML		MS/MS	GC/MS	QuEChERS
	Fludioxonil	Yes	0.094	5	ACN			Yes	No	pH 5	DSPE	MM-ML	MSD		LC-MS/MS	QuEChERS
	Flusilazole	Yes	0.819	5	ACN			Yes	No	pH 5	DSPE	MM-ML		MS/MS	GC/MS	QuEChERS
	Lambda-cyhalothrin	Yes	0.062	5	ACN			Yes	No	pH 5	DSPE	MM-ML	MSD		GC/MS	QuEChERS
	Malathion	Yes	0.01	0												calculated from 36
	Metconazole	Yes	0.549	5	ACN			Yes	No	pH 5	DSPE	MM-ML		MS/MS	GC/MS	QuEChERS
	Pyraclostrobin	Yes	0.793	5	ACN			Yes	No	pH 5	DSPE	MM-ML		MS/MS		QuEChERS
	Tebuconazole	Yes	1.37	5	ACN			Yes	No	pH 5	DSPE	MM-ML		MS/MS	GC/MS	QuEChERS
eupt 050	Azoxystrobin	Yes	0.185	2	MeOH			Yes	No	No	Other	Std-Add.	TOF	MS/MS		
	Carbendazim and benomyl	Yes	0.51	2	MeOH			Yes	No	No	Other	Std-Add.		MS/MS	LC-MS/MS	
	Chlorpyrifos	Yes	1.5	2	MeOH			Yes	No	No	Other	Std-Add.	TOF	MS/MS		
	Cyproconazole	Yes	0.51	2	MeOH			Yes	No	No	Other	Std-Add.	TOF	MS/MS		
	Cyprodinil	Yes	0.088	2	MeOH			Yes	No	No	Other	Std-Add.	TOF	MS/MS		
	Fenvalerate and Esfenvalerate	Yes	0.439	4	EtOAc	C_6H_{12}		No	No	No	GPC	Std-Add.	TOF		GC/MS	

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1)]	Extraction solvent II [1)]	Extraction solvent III [1)]	Water addition	Hydrolysis	pH adjused	Clean up [2)]	Calibration [3)]	GC detector	HPLC detector	Confirmation	Reference method
	Fenbuconazole	Yes	0.47	2	MeOH			Yes	No	No	Other	Std-Add.	TOF	MS/MS		
	Fenpropimorph	Yes	0.145	2	MeOH			Yes	No	No	Other	Std-Add.	TOF	MS/MS		
	Fludioxonil	Yes	0.097	2	MeOH			Yes	No	No	Other	Std-Add.	TOF	MS/MS		
	Flusilazole	Yes	0.65	2	MeOH			Yes	No	No	Other	Std-Add.	TOF	MS/MS		
	Lambda-cyhalothrin	Yes	0.08	4	EtOAc	C_6H_{12}		No	No	No	GPC	Std-Add.	TOF		GC/MS	
	Metconazole	Yes	0.39	2	MeOH			Yes	No	No	Other	Std-Add.	TOF	MS/MS		
	Pyraclostrobin	Yes	0.74	2	MeOH			Yes	No	No	Other	Std-Add.	TOF	MS/MS		
	Tebuconazole	Yes	1.41	2	MeOH			Yes	No	No	Other	Std-Add.	TOF	MS/MS		
eupt 053	Azoxystrobin	Yes	0.188	3	EtOAc	C_6H_{12}		Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	ASE extraction. LC
	Carbendazim and benomyl	Yes	0.625	3	EtOAc	C_6H_{12}		Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	ASE extraction. LC
	Chlorpyrifos	Yes	1,135	3	EtOAc	C_6H_{12}		Yes	No	No	GPC	MM-ML	MS/MS		GC-MS/MS	ASE extraction. GPC. GC
	Cyproconazole	Yes	0.503	3	EtOAc	C_6H_{12}		Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	ASE extraction. LC
	Cyprodinil	Yes	0.092	3	EtOAc	C_6H_{12}		Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	ASE extraction. LC
	Fenvalerate and Esfenvalerate	Yes	0.107	3	EtOAc	C_6H_{12}		Yes	No	No	GPC	MM-ML	MS/MS		GC-MS/MS	ASE extraction. GPC. GC
	Fenbuconazole	Yes	0.543	3	EtOAc	C_6H_{12}		Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	ASE extraction. LC
	Fenpropimorph	Yes	0.335	3	EtOAc	C_6H_{12}		Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	ASE extraction. LC
	Fludioxonil	Yes	0.067	3	EtOAc	C_6H_{12}		Yes	No	No	GPC	MM-ML		MS/MS	GC-MS/MS	ASE extraction. GPC. LC
	Flusilazole	Yes	0.82	3	EtOAc	C_6H_{12}		Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	ASE extraction. LC
	Malathion	Yes	<0.02	3	EtOAc	C_6H_{12}		Yes	No	No	GPC	MM-ML	MS/MS	MS/MS		ASE extraction. (GPC). GC+LC
	Metconazole	Yes	0.581	3	EtOAc	C_6H_{12}		Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	ASE extraction. LC
	Pyraclostrobin	Yes	0.859	3	EtOAc	C_6H_{12}		Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	ASE extraction. LC
	Tebuconazole	Yes	1,435	3	EtOAc	C_6H_{12}		Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	ASE extraction. LC
eupt 054	Azoxystrobin	Yes	0.219	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	Other	QuEChERS
	Carbendazim and benomyl	Yes	0.708	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	Other	QuEChERS
	Chlorpyrifos	Yes	1.21	5	ACN			Yes	No	Yes	DSPE	PS-ML	ECD		Other	QuEChERS

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1)]	Extraction solvent II [1)]	Extraction solvent III [1)]	Water addition	Hydrolysis	pH adjused	Clean up [2)]	Calibration [3)]	GC detector	HPLC detector	Confirmation	Reference method
	Cyproconazole	Yes	0.527	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	Other	QuEChERS
	Cyprodinil	Yes	0.1	5	ACN			Yes	No	Yes	DSPE	PS-ML		DAD.	LC-MS/MS	QuEChERS
	Fenvalerate and Esfenvalerate	Yes	0.117	5	ACN			Yes	No	Yes	DSPE	PS-ML	ECD		Other	QuEChERS
	Fenbuconazole	Yes	0.743	5	ACN			Yes	No	Yes	DSPE	PS-ML	ECD		Other	QuEChERS
	Fenpropimorph	Yes	0.17	5	ACN			Yes	No	Yes	DSPE	PS-ML	MS/MS		Other	QuEChERS
	Fludioxonil	Yes	0.092	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	Other	QuEChERS
	Flusilazole	Yes	0.72	5	ACN			Yes	No	Yes	DSPE	PS-ML	NPD		GC/MS	QuEChERS
	Lambda-cyhalothrin	Yes	0.0534	5	ACN			Yes	No	Yes	DSPE	PS-ML	ECD		Other	QuEChERS
	Metconazole	Yes	0.669	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	Other	QuEChERS
	Pyraclostrobin	Yes	0.82	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	Other	QuEChERS
	Tebuconazole	Yes	1.37	5	ACN			Yes	No	Yes	DSPE	PS-ML	NPD		LC-MS/MS	QuEChERS
eupt 055	Azoxystrobin	Yes	0.274	5	ACN			Yes	No	No	Other	MM-ML	NPD		GC/MS	QuEChERS
	Carbendazim and benomyl	Yes	0.457	5	ACN			Yes	No	No	Other	PS-ML		UV		QuEChERS
	Chlorpyrifos	Yes	1.26	5	ACN			Yes	No	No	Other	MM-ML	NPD		GC/MS	QuEChERS
	Cyproconazole	Yes	0.562	5	ACN			Yes	No	No	Other	MM-ML	NPD		GC/MS	QuEChERS
	Cyprodinil	Yes	0.089	5	ACN			Yes	No	No	Other	MM-ML	NPD		GC/MS	QuEChERS
	Fenvalerate and Esfenvalerate	Yes	0.093	5	ACN			Yes	No	No	Other	MM-ML	ECD		GC/MS	QuEChERS
	Fenbuconazole	Yes	0.784	5	ACN			Yes	No	No	Other	MM-ML	NPD		GC/MS	QuEChERS
	Fenpropimorph	Yes	0.183	5	ACN			Yes	No	No	Other	MM-ML	ITD		GC/MS	QuEChERS
	Fludioxonil	Yes	0.094	5	ACN			Yes	No	No	Other	MM-ML	NPD		GC/MS	QuEChERS
	Flusilazole	Yes	0.76	5	ACN			Yes	No	No	Other	MM-ML	NPD		GC/MS	QuEChERS
	Lambda-cyhalothrin	Yes	0.055	5	ACN			Yes	No	No	Other	MM-ML	ECD		GC/MS	QuEChERS
	Metconazole	Yes	0.638	5	ACN			Yes	No	No	Other	MM-ML	NPD		GC/MS	QuEChERS
	Pyraclostrobin	Yes	0.842	5	ACN			Yes	No	No	Other	MM-ML	NPD		GC/MS	QuEChERS
	Tebuconazole	Yes	1.41	5	ACN			Yes	No	No	Other	MM-ML	NPD		GC/MS	QuEChERS
eupt	Azoxystrobin	No	0.042	5	ACN			Yes	No	No	None	MM-ML		MS/MS	None	QuEChers

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
056	Carbendazim and benomyl	No	0.169	5	ACN			Yes	No	No	None	MM-ML		MS/MS	None	QuEChers
	Chlorpyrifos	Yes	1.05	5	ACN			Yes	No	No	None	MM-ML	Other		None	QuEChERS
	Cyprodinil	No	0.073	5	ACN			Yes	No	No	None	MM-ML	MS/MS		None	QuEChERS
	Fenvalerate and Esfenvalerate	No	0.23	5	ACN			Yes	No	No	None	MM-ML	MS/MS		None	QuEChERS
	Fludioxonil	No	0.43	5	ACN			Yes	No	No	None	MM-ML	MS/MS		None	QuEChERS
	Metconazole	No	0.286	5	ACN			Yes	No	No	None	MM-ML		MS/MS	None	QuEChERS
	Pyraclostrobin	No	0.3	5	ACN			Yes	No	No	None	MM-ML		MS/MS	None	QuEChERS
	Tebuconazole	No	1.55	5	ACN			Yes	No	No	None	MM-ML	MS/MS		None	QuEChERS
eupt 057	Fenvalerate and Esfenvalerate	No	0.016	20	EtOAc						GPC	MM-ML	MSD		GC/MS	
	Fludioxonil	No	0.05	20	EtOAc						GPC	MM-ML		MS/MS	LC-MS/MS	
	Lambda-cyhalothrin	No	0.02	20	EtOAc						GPC	MM-ML	MSD		GC/MS	
	Tebuconazole	No	0.758	20	EtOAc						GPC	MM-ML		MS/MS	LC-MS/MS	
eupt 058	Azoxystrobin	Yes	0.203	5	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	
	Carbendazim and benomyl	Yes	0.59	5	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	
	Chlorpyrifos	Yes	0.998	5	Acetone	CH_2Cl_2	Other	Yes	No	No	None	PS-ML	FPD		GC-MS/MS	
	Cyproconazole	Yes	0.453	5	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	
	Cyprodinil	Yes	0.074	5	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	
	Fenpropimorph	Yes	0.074	5	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	
	Fludioxonil	Yes	0.092	5	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	
	Flusilazole	Yes	0.756	5	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	
	Lambda-cyhalothrin	No	0.062	5	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML	ECD		GC-MS/MS	
	Metconazole	Yes	0.51	5	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	
	Pyraclostrobin	No	0.62	5	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	
	Tebuconazole	No	1.25	5	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	
eupt	Azoxystrobin	Yes	0.178	5	ACN			Yes	No	No	None	MM-ML		MS/MS	GC/MS	

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1)]	Extraction solvent II [1)]	Extraction solvent III [1)]	Water addition	Hydrolysis	pH adjused	Clean up [2)]	Calibration [3)]	GC detector	HPLC detector	Confirmation	Reference method
060	Carbendazim and benomyl	Yes	0.589	5	ACN			Yes	No	No	None	MM-ML		MS/MS	LC-MS/MS	
	Chlorpyrifos	Yes	1.16	5	ACN			Yes	No	No	None	MM-ML	ITD	MS/MS	GC-MS/MS	
	Cyproconazole	Yes	0.503	5	ACN			Yes	No	No	None	MM-ML		MS/MS	GC/MS	
	Cyprodinil	Yes	0.09	5	ACN			Yes	No	No	None	MM-ML		MS/MS	GC/MS	
	Fenvalerate and Esfenvalerate	Yes	0.115	5	ACN			Yes	No	No	None	MM-ML	ITD	Other	GC-MS/MS	
	Fenbuconazole	Yes	0.523	5	ACN			Yes	No	No	None	MM-ML		MS/MS	GC/MS	
	Fenpropimorph	Yes	0.119	5	ACN			Yes	No	No	None	MM-ML		MS/MS	GC/MS	
	Fludioxonil	Yes	0.099	5	ACN			Yes	No	No	None	MM-ML		MS/MS	GC/MS	
	Flusilazole	Yes	0.835	5	ACN			Yes	No	No	None	MM-ML		MS/MS	GC/MS	
	Lambda-cyhalothrin	Yes	0.064	5	ACN			Yes	No	No	None	MM-ML	ITD	Other	GC-MS/MS	
	Malathion	Yes	0.011	5	ACN			Yes	No	No	None	MM-ML		MS/MS	GC/MS	
	Metconazole	Yes	0.475	5	ACN			Yes	No	No	None	MM-ML		MS/MS	GC/MS	
	Pyraclostrobin	Yes	0.699	5	ACN			Yes	No	No	None	MM-ML		MS/MS	GC/MS	
	Tebuconazole	Yes	1.31	5	ACN			Yes	No	No	None	MM-ML		MS/MS	GC/MS	
eupt 061	Azoxystrobin	Yes	0.19	5	ACN			Yes	No	No	DSPE	PS-ML		MS/MS	LC-MS/MS	QuEChERS
	Carbendazim and benomyl	Yes	0.433	5	ACN			Yes	No	No	DSPE	PS-ML		MS/MS	LC-MS/MS	QuEChERS
	Chlorpyrifos	Yes	0.722	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		GC/MS	QuEChERS
	Cyproconazole	Yes	0.514	5	ACN			Yes	No	No	DSPE	PS-ML		MS/MS	LC-MS/MS	QuEChERS
	Cyprodinil	Yes	0.072	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		GC/MS	QuEChERS
	Fenvalerate and Esfenvalerate	Yes	0.134	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		GC/MS	QuEChERS
	Fenbuconazole	No	0.425	5	ACN			Yes	No	No	DSPE	PS-ML		MS/MS	LC-MS/MS	QuEChERS
	Fludioxonil	Yes	0.078	5	ACN			Yes	No	No	DSPE	PS-ML		MS/MS	LC-MS/MS	QuEChERS
	Flusilazole	Yes	0.687	5	ACN			Yes	No	No	DSPE	PS-ML		MS/MS	LC-MS/MS	QuEChERS
	Lambda-cyhalothrin	Yes	0.056	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		GC/MS	QuEChERS
	Metconazole	Yes	0.392	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		GC/MS	QuEChERS

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Pyraclostrobin	Yes	0.793	5	ACN			Yes	No	No	DSPE	PS-ML		MS/MS	LC-MS/MS	QuEChERS
	Tebuconazole	Yes	1,416	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		GC/MS	QuEChERS
eupt 062	Chlorpyrifos	Yes	1.47	1	Acetone	CH_2Cl_2		Yes	No	No	SPE	MM-ML	FPD		GC/MS	
eupt 063	Azoxystrobin	Yes	0.177	5	ACN			Yes	No	Yes	Freez.	MM-ML		MS/MS	LC-MS/MS	
	Carbendazim and benomyl	Yes	0.482	5	ACN			Yes	No	Yes	Freez.	MM-ML		MS/MS	LC-MS/MS	
	Chlorpyrifos	Yes	1.08	5	ACN			Yes	No	Yes	Freez.	MM-ML		MS/MS	LC-MS/MS	
	Cyproconazole	Yes	0.434	5	ACN			Yes	No	Yes	Freez.	MM-ML		MS/MS	LC-MS/MS	
	Cyprodinil	Yes	0.09	5	ACN			Yes	No	Yes	Freez.	MM-ML		MS/MS	LC-MS/MS	
	Fenpropimorph	Yes	0.148	5	ACN			Yes	No	Yes	Freez.	MM-ML		MS/MS	LC-MS/MS	
	Fludioxonil	Yes	0.098	5	ACN			Yes	No	Yes	Freez.	MM-ML		MS/MS	LC-MS/MS	
	Flusilazole	Yes	0.67	5	ACN			Yes	No	Yes	Freez.	MM-ML		MS/MS	LC-MS/MS	
	Lambda-cyhalothrin	Yes	0.06	5	ACN			Yes	No	Yes	Freez.	MM-ML	TOF		GC/MS	
	Malathion	Yes	0.02	5	ACN			Yes	No	Yes	Freez.	MM-ML		MS/MS	LC-MS/MS	
	Pyraclostrobin	Yes	0.761	5	ACN			Yes	No	Yes	Freez.	MM-ML		MS/MS	LC-MS/MS	
	Tebuconazole	Yes	0.951	5	ACN			Yes	No	Yes	Freez.	MM-ML		MS/MS	LC-MS/MS	
eupt 064	Chlorpyrifos	Yes	1.45	5	Acetone	CH_2Cl_2		Yes	No	No	SPE	PS-ML	Other		2 colns	
eupt 068	Chlorpyrifos	Yes	1.4	10	Acetone	CH_2Cl_2		Yes	No	No	SPE	PS-ML	NPD		2 colns	
eupt 069	Chlorpyrifos	No	1.35	5	Acetone	CH_2Cl_2		Yes	No	No	SPE	PS-ML	NPD		2 colns	
eupt 070	Azoxystrobin	Yes	0.147	5	ACN			Yes	No	Yes	SPE	MM-SL	MSD			
	Carbendazim and benomyl	Yes	0.432	5	ACN			Yes	No	Yes	SPE	PS-SL		DAD		
	Chlorpyrifos	Yes	0.81	5	ACN			Yes	No	Yes	SPE	MM-SL	MSD			
	Cyprodinil	Yes	0.062	5	ACN			Yes	No	Yes	SPE	MM-SL	MSD			
	Fenvalerate and Esfenvalerate	Yes	0.084	5	ACN			Yes	No	Yes	SPE	MM-SL	MSD			

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Fenbuconazole	No	0.377	5	ACN			Yes	No	Yes	SPE	MM-SL	MSD			
	Fenpropimorph	No	0.099	5	ACN			Yes	No	Yes	SPE	MM-SL	MSD			
	Fludioxonil	Yes	0.053	5	ACN			Yes	No	Yes	SPE	MM-SL	MSD			
	Flusilazole	Yes	0.695	5	ACN			Yes	No	Yes	SPE	MM-SL	MSD			
	Lambda-cyhalothrin	Yes	0.04	5	ACN			Yes	No	Yes	SPE	MM-SL	MSD			
	Pyraclostrobin	No	0.665	5	ACN			Yes	No	Yes	SPE	MM-SL	MSD			
	Tebuconazole	Yes	1.13	5	ACN			Yes	No	Yes	SPE	MM-SL	MSD			
eupt 071	Carbendazim and benomyl	Yes	0.192	25	Acetone	EtOAc				pH 7		PS-ML		DAD.	None	
	Chlorpyrifos	Yes	0.684	50	Acetone	CH_2Cl_2		Yes			SPE	PS-ML	ECD		None	
	Fenvalerate and Esfenvalerate	Yes	0.07	50	Acetone	CH_2Cl_2		Yes			SPE	PS-ML	ECD		None	
	Lambda-cyhalothrin	Yes	0.04	50	Acetone	CH_2Cl_2		Yes			SPE	PS-ML	ECD		None	
eupt 073	Azoxystrobin	Yes	0.142	10	Acetone			Yes	No		SPE	MM-ML	ECD		GC-MS/MS	EN 12393
	Carbendazim and benomyl	No	0.592	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	LC-MS/MS	Walorczyk. 2008.
	Chlorpyrifos	Yes	1,119	10	Acetone			Yes	No		SPE	MM-ML	ECD		GC-MS/MS	EN 12393
	Cyproconazole	Yes	0.494	5	ACN			Yes	No	Yes	DSPE	MM-ML	MS/MS		GC-MS/MS	Walorczyk. 2008.
	Cyprodinil	Yes	0.097	10	Acetone			Yes	No		SPE	MM-ML	ECD		GC-MS/MS	EN 12393
	Fenvalerate and Esfenvalerate	Yes	0.059	5	ACN			Yes	No	Yes	DSPE	MM-ML	MS/MS		GC-MS/MS	Walorczyk. 2008.
	Fenbuconazole	No	0.459	10	Acetone			Yes	No		SPE	MM-ML	ECD		GC-MS/MS	EN 12393
	Fenpropimorph	Yes	0.098	5	ACN			Yes	No	Yes	DSPE	MM-ML	MS/MS		GC-MS/MS	Walorczyk. 2008.
	Fludioxonil	Yes	0.079	5	ACN			Yes	No	Yes	DSPE	MM-ML	MS/MS		GC-MS/MS	Walorczyk. 2008.
	Flusilazole	Yes	0.649	10	Acetone			Yes	No		SPE	MM-ML	ECD		GC-MS/MS	EN 12393
	Lambda-cyhalothrin	Yes	0.052	5	ACN			Yes	No	Yes	DSPE	MM-ML	MS/MS		GC-MS/MS	Walorczyk. 2008.
	Metconazole	Yes	0.486	5	ACN			Yes	No	Yes	DSPE	MM-ML	MS/MS		GC-MS/MS	Walorczyk. 2008.
	Pyraclostrobin	No	0.893	5	ACN			Yes	No	Yes	DSPE	MM-ML		MS/MS	LC-MS/MS	Walorczyk. 2008.
	Tebuconazole	Yes	1,182	10	Acetone			Yes	No		SPE	MM-ML	ECD		GC-MS/MS	EN 12393

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
eupt 074	Chlorpyrifos	No	1.34	10	Acetone	CH_2Cl_2		Yes	No	No	SPE	PS-ML	FPD		2 colns	Yes
eupt 076	Chlorpyrifos	No	1.47	10	Acetone	CH_2Cl_2		Yes	No	No	SPE	PS-ML	NPD		GC/MS	
eupt 077	Azoxystrobin	No	0.5	0												
	Carbendazim and benomyl	Yes	0.3	50	MeOH	CH_2Cl_2		Yes	No	pH 7	liq./liq	PS-ML		UV	Other	
	Chlorpyrifos	Yes	1.17	75	EtOAc			No	No	No	GPC	PS-ML	ECD		2 colns	EN 12393
	Fenvalerate and Esfenvalerate	Yes	0.121	75	EtOAc			No	No	No	GPC	PS-ML	ECD		2 colns	EN 12393
	Lambda-cyhalothrin	Yes	0.044	75	EtOAc			No	No	No	GPC	PS-ML	ECD		2 colns	EN 12393
	Methomyl and Thiodicarb	No		20	CH2Cl2			No	No	No	SPE	PS-ML		FLUOR	None	EN 14185-2
eupt 078	Chlorpyrifos	No	0.208	15	Other						GPC	Std-Add.	NPD		GC-MS/MS	
	Cyproconazole	No	yes	15	ACN						GPC		MSD			
	Cyprodinil	No	yes	15	ACN						GPC		MSD			
	Fenbuconazole	No	yes	15	ACN						GPC		MSD			
	Fenpropimorph	No	yes	15	ACN						GPC		MSD			
	Flusilazole	No	yes	15	ACN						GPC		MSD			
	Lambda-cyhalothrin	No	0.02	0												
	Metconazole	No	yes	15	ACN						GPC		MSD			
	Tebuconazole	No	yes	15	ACN						GPC		MSD			
eupt 079	Chlorpyrifos	No	1.48	10	Acetone	CH_2Cl_2		Yes	No	No	SPE	PS-ML	NPD		None	
eupt 082	Azoxystrobin	Yes	0.303	5	ACN			Yes	No	No	SPE	MM-ML	MSD		None	
	Carbendazim and benomyl	No	0.254	25	EtOAc				Yes	pH 13	liq./liq	MM-ML		FLUOR	None	
	Chlorpyrifos	Yes	0.745	5	ACN			Yes	No	No	SPE	MM-ML	MSD		None	
	Cyproconazole	No	0.417	5	ACN			Yes	No	No	SPE	MM-ML	MSD		None	
	Cyprodinil	Yes	0.076	5	ACN			Yes	No	No	SPE	MM-ML	MSD		None	

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1)]	Extraction solvent II [1)]	Extraction solvent III [1)]	Water addition	Hydrolysis	pH adjused	Clean up [2)]	Calibration [3)]	GC detector	HPLC detector	Confirmation	Reference method
	Fenvalerate and Esfenvalerate	Yes	0.167	5	ACN			Yes	No	No	SPE	MM-ML	MSD		None	
	Fenbuconazole	No	0.478	5	ACN			Yes	No	No	SPE	MM-ML	MSD		None	
	Fenpropimorph	No	0.136	5	ACN			Yes	No	No	SPE	MM-ML	MSD		None	
	Fludioxonil	Yes	0.068	5	ACN			Yes	No	No	SPE	MM-ML	MSD		None	
	Flusilazole	No	0.475	5	ACN			Yes	No	No	SPE	MM-ML	MSD		None	
	Lambda-cyhalothrin	Yes	0.077	5	ACN			Yes	No	No	SPE	MM-ML	MSD		None	
	Malathion	Yes	0.02	5	ACN			Yes	No	No	SPE	MM-ML	MSD		None	
	Metconazole	No	0.293	5	ACN			Yes	No	No	SPE	MM-ML	MSD		None	
	Pyraclostrobin	No	0.75	5	ACN			Yes	No	No	SPE	MM-ML	MSD		None	
	Tebuconazole	No	0.99	5	ACN			Yes	No	No	SPE	MM-ML	MSD		None	
eupt 083	Azoxystrobin	Yes	0.111	20	Acetone	C_6H_{12}	EtOAc	Yes			GPC	PS-ML	ECD		2 colns	
	Carbendazim and benomyl	Yes	0.118	25	Acetone	EtOAc		No		pH 7	GPC	PS-ML		UV	None	
	Chlorpyrifos	Yes	1,103	25	Acetone	CH_2Cl_2		Yes			GPC	PS-ML	NPD		2 colns	
	Fenvalerate and Esfenvalerate	Yes	0.105	25	Acetone	CH_2Cl_2		Yes			GPC	PS-ML	ECD		2 colns	
	Flusilazole	No	0.68	25	Acetone	CH_2Cl_2		Yes			GPC	PS-ML	NPD		2 colns	
	Lambda-cyhalothrin	Yes	0.042	25	Acetone	CH_2Cl_2		Yes			GPC	PS-ML	ECD		2 colns	
	Tebuconazole	Yes	1.34	25	Acetone	CH_2Cl_2		Yes			GPC	PS-ML	NPD		2 colns	
eupt 084	Azoxystrobin	Yes	0.193	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML	MSD	MS/MS	LC-MS/MS	SOP
	Carbendazim and benomyl	Yes	0.626	5	MeOH			Yes	No	No	SPE	MM-ML		MS/MS	LC-MS/MS	SOP
	Chlorpyrifos	Yes	1.08	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML	MSD	MS/MS	LC-MS/MS	SOP
	Cyproconazole	Yes	0.454	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML	MSD	MS/MS	LC-MS/MS	SOP
	Cyprodinil	Yes	0.078	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML	MSD	MS/MS	LC-MS/MS	SOP
	Fenvalerate and Esfenvalerate	Yes	0.09	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML	MSD	MS/MS	LC-MS/MS	SOP
	Fenbuconazole	No	0.59	5	MeOH			Yes	No	No	SPE	MM-ML		MS/MS	LC-MS/MS	SOP
	Fenpropimorph	Yes	0.116	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML	MSD	MS/MS	LC-MS/MS	SOP

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Fludioxonil	Yes	0.08	5	MeOH			Yes	No	No	SPE	MM-ML		MS/MS	LC-MS/MS	SOP
	Flusilazole	Yes	0.714	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML	MSD	MS/MS	LC-MS/MS	SOP
	Lambda-cyhalothrin	Yes	0.048	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML	MSD	MS/MS	LC-MS/MS	SOP
	Metconazole	Yes	0.634	5	MeOH			Yes	No	No	SPE	MM-ML		MS/MS	LC-MS/MS	SOP
	Pyraclostrobin	No	0.903	5	MeOH			Yes	No	No	SPE	MM-ML		MS/MS	LC-MS/MS	SOP
	Tebuconazole	Yes	1,269	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML	MSD	MS/MS	LC-MS/MS	SOP
eupt 086	Azoxystrobin	Yes	0.184	5	ACN			Yes			DSPE	MM-ML		MS/MS	None	
	Carbendazim and benomyl	Yes	0.732	5	ACN			Yes			DSPE	MM-ML		MS/MS	None	
	Chlorpyrifos	Yes	1.14	5	ACN			Yes			DSPE	MM-ML	MS/MS		Other	
	Cyproconazole	Yes	0.435	5	ACN			Yes			DSPE	MM-ML		MS/MS	None	
	Cyprodinil	Yes	0.095	5	ACN			Yes			DSPE	MM-ML		MS/MS	None	
	Fenvalerate and Esfenvalerate	Yes	0.102	5	ACN			Yes			DSPE	MM-ML	MS/MS		Other	
	Fenbuconazole	Yes	0.667	5	ACN			Yes			DSPE	MM-ML		MS/MS	None	
	Fenpropimorph	Yes	0.159	5	ACN			Yes			DSPE	MM-ML	MSD		GC-MS/MS	
	Fludioxonil	Yes	0.094	5	ACN			Yes			DSPE	MM-ML		MS/MS	None	
	Flusilazole	Yes	0.73	5	ACN			Yes			DSPE	MM-ML	MSD		GC-MS/MS	
	Lambda-cyhalothrin	Yes	0.05	5	ACN			Yes			DSPE	MM-ML	ECD		GC-MS/MS	
	Metconazole	Yes	0.686	5	ACN			Yes			DSPE	MM-ML		MS/MS	None	
	Pyraclostrobin	Yes	0.77	5	ACN			Yes			DSPE	MM-ML		MS/MS	None	
	Tebuconazole	Yes	1.48	5	ACN			Yes			DSPE	MM-ML		MS/MS	GC-MS/MS	
eupt 087	Chlorpyrifos	No	0.935	50	EtOAc			No	No	No	GPC	PS-ML	NPD		2 colns	EN12393 Metodo P
eupt 088	Azoxystrobin	Yes	0.193	5	ACN			Yes		Yes	None	MM-SL		MS/MS	GC-MS/MS	QuEChERS
	Carbendazim and benomyl	Yes	0.396	5	ACN			Yes		Yes	None	MM-SL		MS/MS		QuEChERS
	Chlorpyrifos	Yes	1,012	5	ACN			Yes		Yes	DSPE	MM-SL	MS/MS		LC-MS/MS	QuEChERS
	Cyproconazole	Yes	0.461	5	ACN			Yes		Yes	DSPE	MM-SL	MS/MS			QuEChERS

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1)]	Extraction solvent II [1)]	Extraction solvent III [1)]	Water addition	Hydrolysis	pH adjused	Clean up [2)]	Calibration [3)]	GC detector	HPLC detector	Confirmation	Reference method
	Cyprodinil	No	0.059	5	ACN			Yes		Yes	DSPE	MM-SL	MS/MS			QuEChERS
	Fenvalerate and Esfenvalerate	Yes	0.07	5	ACN			Yes		Yes	DSPE	MM-SL	MS/MS			QuEChERS
	Fenbuconazole	No	0.437	5	ACN			Yes		Yes	DSPE	MM-SL	MS/MS			QuEChERS
	Fenpropimorph	Yes	0.123	5	ACN			Yes		Yes	DSPE	MM-SL	MS/MS		LC-MS/MS	QuEChERS
	Fludioxonil	No	0.069	5	ACN			Yes		Yes	DSPE	MM-SL	MS/MS			QuEChERS
	Flusilazole	No	0.696	5	ACN			Yes		Yes	DSPE	MM-SL	MS/MS			QuEChERS
	Lambda-cyhalothrin	Yes	0.032	5	ACN			Yes		Yes	DSPE	MM-SL	MS/MS			QuEChERS
	Malathion	Yes	0.009	5	ACN			Yes		Yes	DSPE	MM-SL	MS/MS		LC-MS/MS	QuEChERS
	Metconazole	Yes	0.442	5	ACN			Yes		Yes	DSPE	MM-SL	MS/MS			QuEChERS
	Pyraclostrobin	No	0.612	5	ACN			Yes		Yes	None	MM-SL		MS/MS		QuEChERS
	Tebuconazole	Yes	1,348	5	ACN			Yes		Yes	DSPE	MM-SL	MS/MS		LC-MS/MS	QuEChERS
eupt 089	Azoxystrobin	Yes	0.092	25	EtOAc			Yes	No	No	GPC	MM-ML	TOF		Other	EN 12393-2 Method P
	Carbendazim and benomyl	Yes	0.586	25	EtOAc			Yes	No	Yes	GPC	PS-ML		DAD	Other	EN 12393-2 Method P
	Chlorpyrifos	Yes	0.703	25	EtOAc			Yes	No	No	GPC	MM-ML	TOF		Other	EN 12393-2 Method P
	Cyproconazole	No	0.287	25	EtOAc			Yes	No	No	GPC	MM-ML	NPD		2 colns	EN 12393-2 Method P
	Cyprodinil	Yes	0.046	25	EtOAc			Yes	No	No	GPC	MM-ML	TOF		Other	EN 12393-2 Method P
	Fenvalerate and Esfenvalerate	Yes	0.094	25	EtOAc			Yes	No	No	GPC	MM-ML	TOF		Other	EN 12393-2 Method P
	Fludioxonil	Yes	0.055	25	EtOAc			Yes	No	Yes	GPC	PS-ML		DAD	Other	EN 12393-2 Method P
	Lambda-cyhalothrin	Yes	0.033	25	EtOAc			Yes	No	No	GPC	MM-ML	TOF		Other	EN 12393-2 Method P
	Tebuconazole	Yes	0.647	25	EtOAc			Yes	No	No	GPC	MM-ML	TOF		Other	EN 12393-2 Method P
eupt 091	Azoxystrobin	Yes	0.183	3	ACN			Yes	No	Yes	None	MM-ML		MS/MS	LC-MS/MS	
	Carbendazim and benomyl	Yes	0.576	3	ACN			Yes	No	Yes	None	MM-ML		MS/MS	LC-MS/MS	
	Chlorpyrifos	Yes	1,123	6	EtOAc			Yes	No	Yes	GPC	MM-ML	MSD			
	Cyproconazole	Yes	0.48	3	ACN			Yes	No	Yes	None	MM-ML		MS/MS	LC-MS/MS	
	Cyprodinil	Yes	0.079	6	EtOAc			Yes	No	Yes	GPC	MM-ML	MSD			

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Fenvalerate and Esfenvalerate	Yes	0.17	6	EtOAc			Yes	No	Yes	GPC	MM-ML	MSD			
	Fenbuconazole	Yes	0.49	3	ACN			Yes	No	Yes	None	MM-ML		MS/MS	LC-MS/MS	
	Fenpropimorph	Yes	0.133	3	ACN			Yes	No	Yes	None	MM-ML		MS/MS	LC-MS/MS	
	Fludioxonil	Yes	0.078	3	ACN			Yes	No	Yes	None	MM-ML		MS/MS	LC-MS/MS	
	Flusilazole	Yes	0.793	3	ACN			Yes	No	Yes	None	MM-ML		MS/MS	LC-MS/MS	
	Lambda-cyhalothrin	Yes	0.064	6	EtOAc			Yes	No	Yes	GPC	MM-ML	MSD			
	Metconazole	Yes	0.509	3	ACN			Yes	No	Yes	None	MM-ML		MS/MS	LC-MS/MS	
	Pyraclostrobin	Yes	0.758	3	ACN			Yes	No	Yes	None	MM-ML		MS/MS	LC-MS/MS	
	Tebuconazole	Yes	1,216	3	ACN			Yes	No	Yes	None	MM-ML		MS/MS	LC-MS/MS	
eupt 092	Azoxystrobin	No	0.128	5	Acetone	CH_2Cl_2					SPE	PS-SL	ECD		GC/MS	
	Chlorpyrifos	No	0.907	5	Acetone	CH_2Cl_2					SPE	PS-SL	ECD		GC/MS	
	Cyproconazole	No	0.122	5	Acetone	CH_2Cl_2					SPE	PS-SL	ECD		GC/MS	
	Fenvalerate and Esfenvalerate	No	0.142	5	Acetone	CH_2Cl_2					SPE	PS-SL	ECD		GC/MS	
	Flusilazole	No	0.163	5	Acetone	CH_2Cl_2					SPE	PS-SL	NPD		GC/MS	
	Lambda-cyhalothrin	No	0.057	5	Acetone	CH_2Cl_2					SPE	PS-SL	ECD		GC/MS	
	Tebuconazole	No	0.809	5	Acetone	CH_2Cl_2					SPE	PS-SL	NPD		GC/MS	
eupt 093	Azoxystrobin	No	0.116	15	Acetone	CH_2Cl_2	Other	Yes				MM-ML		MS/MS		
	Carbendazim and benomyl	No	2,956	15	Acetone	CH_2Cl_2	Other	Yes				MM-ML		MS/MS		
	Chlorpyrifos	Yes	0.88	15	Acetone	CH_2Cl_2	Other	Yes				MM-ML	ITD		GC/MS	
	Cyproconazole	No	1.22	15	Acetone	CH_2Cl_2	Other	Yes				MM-ML		MS/MS		
	Cyprodinil	Yes	0.075	15	Acetone	CH_2Cl_2	Other	Yes				MM-ML	ITD		GC/MS	
	Fenpropimorph	No	0.145	15	Acetone	CH_2Cl_2	Other	Yes				MM-ML	ITD		GC/MS	
	Flusilazole	No	0.816	15	Acetone	CH_2Cl_2	Other	Yes				MM-ML	ITD		GC/MS	
	Lambda-cyhalothrin	Yes	0.045	15	Acetone	CH_2Cl_2	Other	Yes				MM-ML	ITD		GC/MS	
	Metconazole	No	1,152	15	Acetone	CH_2Cl_2	Other	Yes				MM-ML		MS/MS		
	Pyraclostrobin	No	0.575	15	Acetone	CH_2Cl_2	Other	Yes				MM-ML	ITD		GC/MS	

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Tebuconazole	Yes	1,494	15	Acetone	CH_2Cl_2	Other	Yes				MM-ML	ITD		GC/MS	
eupt 094	Azoxystrobin	No	0.125	5	Acetone	CH_2Cl_2	Other	Yes	No	No	SPE	MM-ML	NPD		GC/MS	
	Chlorpyrifos	No	0.761	5	Acetone	CH_2Cl_2	Other	Yes	No	No	SPE	MM-ML	NPD		GC/MS	
	Cyprodinil	No	0.049	5	Acetone	CH_2Cl_2	Other	Yes	No	No	SPE	MM-ML	NPD		GC/MS	
	Fenvalerate and Esfenvalerate	No	0.07	5	Acetone	CH_2Cl_2	Other	Yes	No	No	SPE	MM-ML	NPD		GC/MS	
	Fludioxonil	No	0.047	5	Acetone	CH_2Cl_2	Other	Yes	No	No	SPE	MM-ML	NPD		GC/MS	
	Flusilazole	No	0.755	5	Acetone	CH_2Cl_2	Other	Yes	No	No	SPE	MM-ML	NPD		GC/MS	
	Lambda-cyhalothrin	No	0.03	5	Acetone	CH_2Cl_2	Other	Yes	No	No	SPE	MM-ML	NPD		GC/MS	
	Tebuconazole	No	1.02	5	Acetone	CH_2Cl_2	Other	Yes	No	No	SPE	MM-ML	NPD		GC/MS	
eupt 095	Azoxystrobin	No	0.14	10	EtOAc						GPC	PS-ML	ECD		GC/MS	
	Carbendazim and benomyl	No	0.22	0												
	Chlorpyrifos	No	1.07	10	EtOAc						GPC	PS-ML	ECD		GC/MS	
	Cyproconazole	No	0.42	10	EtOAc						GPC	PS-ML	NPD		GC/MS	
	Cyprodinil	No	0.08	10	EtOAc						GPC	PS-ML	NPD		GC/MS	
	Fenvalerate and Esfenvalerate	No	0.55	10	EtOAc						GPC	PS-ML	ECD		GC/MS	
	Fenbuconazole	No	0.4	10	EtOAc						GPC	PS-ML	NPD		GC/MS	
	Fenpropimorph	No	0.09	0												
	Fludioxonil	No	0.07	10	EtOAc						GPC	PS-ML	NPD		GC/MS	
	Flusilazole	No	0.65	10	EtOAc						GPC	PS-ML	NPD		GC/MS	
	Lambda-cyhalothrin	No	0.07	10	EtOAc						GPC	PS-ML	ECD		GC/MS	
	Metconazole	No	0.4	10	EtOAc						GPC	PS-ML	NPD		GC/MS	
	Pyraclostrobin	No	0.09	10	EtOAc						GPC	PS-ML	ECD		GC/MS	
	Tebuconazole	No	1.21	10	EtOAc						GPC	PS-ML	NPD		GC/MS	
eupt 097	Chlorpyrifos	No	0.76	10	EtOAc						None	MM-ML	MS/MS		GC-MS/MS	
	Fenvalerate and Esfenvalerate	No	0.07	10	EtOAc						None	MM-ML	MS/MS		GC-MS/MS	

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Lambda-cyhalothrin	No	0.04	10	EtOAc						None	MM-ML	MS/MS		GC-MS/MS	
	Tebuconazole	No	0.86	10	EtOAc						None	MM-ML	MS/MS		GC-MS/MS	
eupt 099.	Azoxystrobin	Yes	0.152	15	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	MM-ML	MSD		LC-MS/MS	Luke
	Carbendazim and benomyl	No	0.64	25	EtOAc			Yes	Yes	Yes	liq./liq	MM-ML		MS/MS		
	Chlorpyrifos	Yes	1.22	15	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	MM-ML	FPD		GC/MS	Luke
	Cyproconazole	No	0.326	15	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	MM-ML	MSD		LC-MS/MS	Luke
	Cyprodinil	Yes	0.07	15	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	MM-ML	MSD		LC-MS/MS	Luke
	Fenvalerate and Esfenvalerate	Yes	0.093	15	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	MM-ML	ECD		GC/MS	Luke
	Fenbuconazole	No	0.341	15	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	MM-ML	MSD		LC-MS/MS	Luke
	Fenpropimorph	No	0.083	15	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	MM-ML	MSD		LC-MS/MS	Luke
	Fludioxonil	Yes	0.077	15	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	MM-ML	MSD		LC-MS/MS	Luke
	Flusilazole	Yes	0.766	15	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	MM-ML	MSD		LC-MS/MS	Luke
	Lambda-cyhalothrin	Yes	0.038	15	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	MM-ML	ECD		GC/MS	Luke
	Metconazole	No	0.35	15	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	MM-ML	MSD		LC-MS/MS	Luke
	Pyraclostrobin	No	0.834	15	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	MM-ML	MSD		LC-MS/MS	Luke
	Tebuconazole	Yes	1.18	15	Acetone	CH_2Cl_2	Other	Yes	No	No	GPC	MM-ML	MSD		LC-MS/MS	Luke
eupt 100	Azoxystrobin	No	0.112	10	Acetone	CH_2Cl_2					liq./liq	PS-ML		FLUOR	None	
	Carbendazim and benomyl	No	2,892	10	Acetone	CH_2Cl_2					liq./liq	PS-ML		UV	None	
	Chlorpyrifos	Yes	0.817	10	Acetone	CH_2Cl_2					liq./liq	MM-ML	NPD		2 colns	
	Cyproconazole	No	0.165	10	Acetone	CH_2Cl_2					liq./liq	MM-ML	NPD		None	
	Cyprodinil	No	0.114	10	Acetone	CH_2Cl_2					liq./liq	MM-ML	NPD		None	
	Fenpropimorph	No	0.156	10	Acetone	CH_2Cl_2					liq./liq	MM-ML	NPD		None	
	Flusilazole	No	0.798	10	Acetone	CH_2Cl_2					liq./liq	PS-ML		FLUOR		
	Lambda-cyhalothrin	No	0.052	10	Acetone	CH_2Cl_2					liq./liq	MM-ML	ECD			
	Tebuconazole	No	0.828	10	Acetone	CH_2Cl_2					liq./liq	MM-ML	NPD			
eupt	Azoxystrobin	No	0.069	10	ACN			No	No	No	None	PS-SL	ECD		GC/MS	1

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1)]	Extraction solvent II [1)]	Extraction solvent III [1)]	Water addition	Hydrolysis	pH adjused	Clean up [2)]	Calibration [3)]	GC detector	HPLC detector	Confirmation	Reference method
101	Chlorpyrifos	No	0.648	10	ACN			No	No	No	None	PS-SL	NPD		GC/MS	2
	Cyproconazole	No	0.117	10	ACN			No	No	No	None	PS-SL	NPD		GC/MS	2
	Cyprodinil	No	0.035	10	ACN			No	No	No	None	PS-SL	NPD		GC/MS	2
	Fenbuconazole	No	0.268	10	ACN			No	No	No	None	PS-SL	NPD		GC/MS	2
	Fenpropimorph	No	0.068	10	ACN			No	No	No	None	PS-SL	NPD		GC/MS	2
	Fludioxonil	No	0.045	10	ACN			No	No	No	None	PS-SL	NPD		GC/MS	2
	Flusilazole	No	0.238	10	ACN			No	No	No	None	PS-SL	NPD		GC/MS	2
	Lambda-cyhalothrin	No	0.026	10	ACN			No	No	No	None	PS-SL	ECD		GC/MS	1
	Tebuconazole	No	0.558	10	ACN			No	No	No	None	PS-SL	NPD		GC/MS	2
eupt 102	Azoxystrobin	No	0.145	10	CH_2Cl_2			Yes			GPC	MM-ML	MSD		GC/MS	
	Chlorpyrifos	Yes	0.92	10	CH_2Cl_2			Yes			GPC	MM-ML	NPD		GC/MS	
	Cyproconazole	No	0.475	10	CH_2Cl_2			Yes			GPC	MM-ML	MSD		GC/MS	
	Cyprodinil	No	0.073	10	CH_2Cl_2			Yes			GPC	MM-ML	MSD		GC/MS	
	Fenvalerate and Esfenvalerate	No	0.123	10	CH_2Cl_2			Yes			GPC	MM-ML	MSD		GC/MS	
	Fenbuconazole	No	0.535	10	CH_2Cl_2			Yes			GPC	MM-ML	MSD		GC/MS	
	Fludioxonil	No	0.84	10	CH_2Cl_2			Yes			GPC	MM-ML	MSD		GC/MS	
	Flusilazole	No	0.8	10	CH_2Cl_2			Yes			GPC	MM-ML	MSD		GC/MS	
	Pyraclostrobin	No	0.746	10	CH_2Cl_2			Yes			GPC	MM-ML	MSD		GC/MS	
	Tebuconazole	No	1,535	10	CH_2Cl_2			Yes			GPC	MM-ML	MSD		GC/MS	
eupt 103	Azoxystrobin	No	0.2	5	ACN			Yes	No	Yes	DSPE	PS-ML	MSD		GC/MS	CEN/TC 275 WI 00275154
	Chlorpyrifos	Yes	1.02	5	ACN			Yes	No	Yes	DSPE	PS-ML	MSD		GC/MS	CEN/TC 275 WI 00275154
	Cyprodinil	No	0.12	5	ACN			Yes	No	Yes	DSPE	PS-ML	MSD		GC/MS	CEN/TC 275 WI 00275154
	Fenbuconazole	No	0.448	5	ACN			Yes	No	Yes	DSPE	PS-ML	MSD		GC/MS	CEN/TC 275 WI 00275154
	Lambda-cyhalothrin	Yes	0.06	5	ACN			Yes	No	Yes	DSPE	PS-ML	MSD		GC/MS	CEN/TC 275 WI 00275154

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Tebuconazole	No	1.42	5	ACN			Yes	No	Yes	DSPE	PS-ML	MSD		GC/MS	CEN/TC 275 WI 00275154
eupt 106	Azoxystrobin	No	0.17	5	ACN			Yes	No	No	DSPE	MM-SL	MSD		GC/MS	QuEChERS
	Chlorpyrifos	No	0.85	5	ACN			Yes	No	No	DSPE	MM-SL	FPD		GC/MS	QuEChERS
	Cyproconazole	No	0.495	5	ACN			Yes	No	No	DSPE	MM-SL	MSD		GC/MS	QuEChERS
	Cyprodinil	No	0.063	5	ACN			Yes	No	No	DSPE	MM-SL	MSD		GC/MS	QuEChERS
	Fenvalerate and Esfenvalerate	No	0.124	5	ACN			Yes	No	No	DSPE	MM-SL	MSD		GC/MS	QuEChERS
	Fenbuconazole	No	0.526	5	ACN			Yes	No	No	DSPE	MM-SL	MSD		GC/MS	QuEChERS
	Fenpropimorph	No	0.112	5	ACN			Yes	No	No	DSPE	MM-SL	MSD		GC/MS	QuEChERS
	Fludioxonil	No	0.13	5	ACN			Yes	No	No	DSPE	MM-SL	MSD		GC/MS	QuEChERS
	Flusilazole	No	0.724	5	ACN			Yes	No	No	DSPE	MM-SL	MSD		GC/MS	QuEChERS
	Lambda-cyhalothrin	No	0.041	5	ACN			Yes	No	No	DSPE	MM-SL	MSD		GC/MS	QuEChERS
	Metconazole	No	0.495	5	ACN			Yes	No	No	DSPE	MM-SL	MSD		GC/MS	QuEChERS
	Tebuconazole	No	1.21	5	ACN			Yes	No	No	DSPE	MM-SL	MSD		GC/MS	QuEChERS
eupt 107	Azoxystrobin	No	0.166	10	CH_2Cl_2			Yes	No	No	SPE	PS-SL	NPD		GC/MS	
	Carbendazim and benomyl	No	0.5	10	EtOAc			Yes	No	No	liq./liq	PS-ML		DAD	None	
	Chlorpyrifos	No	0.748	10	CH_2Cl_2			Yes	No	No	SPE	PS-SL	NPD		GC/MS	
	Cyproconazole	No	0.358	10	CH_2Cl_2			Yes	No	No	SPE	PS-SL	NPD		GC/MS	
	Cyprodinil	No	0.055	10	CH_2Cl_2			Yes	No	No	SPE	PS-SL	NPD		GC/MS	
	Fenvalerate and Esfenvalerate	No	0.361	10	CH_2Cl_2			Yes	Yes	No	SPE	PS-SL	ECD		GC/MS	
	Fenbuconazole	No	0.498	10	CH_2Cl_2			Yes	No	No	SPE	PS-SL	NPD		GC/MS	
	Fenpropimorph	No	0.086	10	CH_2Cl_2			Yes	No	No	SPE	PS-SL	NPD		GC/MS	
	Fludioxonil	No	0.021	10	CH_2Cl_2			Yes	No	No	SPE	PS-SL	NPD		GC/MS	
	Flusilazole	No	0.754	10	CH_2Cl_2			Yes	No	No	SPE	PS-SL	NPD		GC/MS	
	Lambda-cyhalothrin	No	0.049	10	CH_2Cl_2			Yes	No	No	SPE	PS-SL	ECD		GC/MS	
	Pyraclostrobin	No	0.648	10	CH_2Cl_2			Yes	No	No	SPE	PS-SL	NPD		2 colns	

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Tebuconazole	No	1,115	10	CH_2Cl_2			Yes	No	No	SPE	PS-SL	NPD		GC/MS	
eupt 108	Azoxystrobin	No	0.25	0												
	Chlorpyrifos	No	0.86	0												
	Cyprodinil	No	0.08	0												
	Fludioxonil	No	0.11	0												
	Lambda-cyhalothrin	No	0.05	0												
eupt 112	Chlorpyrifos	No	0.815	10	ACN			No	No	No	None	PS-ML	MSD		None	
	Fenvalerate and Esfenvalerate. RR. SS	No	0.31	10	ACN			No	No	No	None	PS-ML	MSD		None	
	Tebuconazole	No	1.01	10	ACN			No	No	No	None	PS-ML	MSD		None	
eupt 115	Azoxystrobin	No	0.14	10	Acetone	Other	Other	Yes	No	No	Other	MM-SL	ECD		2 colns	internal method
	Chlorpyrifos	No	1.1	10	Acetone	Other	Other	Yes	No	No	Other	MM-SL	ECD		2 colns	internal method
	Cyproconazole	No	0.33	10	Acetone	Other	Other	Yes	No	No	Other	MM-SL	NPD		2 colns	internal method
	Cyprodinil	No	0.08	10	Acetone	Other	Other	Yes	No	No	Other	MM-SL	NPD		2 colns	internal method
	Fenvalerate and Esfenvalerate	No	0.09	10	Acetone	Other	Other	Yes	No	No	Other	MM-SL	ECD		2 colns	internal method
	Fludioxonil	No	0.08	10	Acetone	Other	Other	Yes	No	No	Other	MM-SL	NPD		2 colns	internal method
	Flusilazole	No	0.73	10	Acetone	Other	Other	Yes	No	No	Other	MM-SL	NPD		2 colns	internal method
	Lambda-cyhalothrin	No	0.07	10	Acetone	Other	Other	Yes	No	No	Other	MM-SL	ECD		2 colns	internal method
	Tebuconazole	No	1.2	10	Acetone	Other	Other	Yes	No	No	Other	MM-SL	NPD		2 colns	internal method
eupt 118	Azoxystrobin	Yes	0.076	15	EtOAc			No	No	No	GPC	PS-ML	MSD		None	Swedish
	Carbendazim and benomyl	Yes	0.042	15	EtOAc			No	Yes	pH 10	GPC	PS-ML		MS/MS	None	Swedish
	Chlorpyrifos	Yes	0.327	15	EtOAc			No	No	No	GPC	PS-ML	MSD		None	Swedish
	Cyprodinil	Yes	0.035	15	EtOAc			No	No	No	GPC	PS-ML	MSD		None	Swedish
	Fenvalerate and Esfenvalerate	Yes	0.103	15	EtOAc			No	No	No	GPC	PS-ML	MSD		None	Swedish
	Fenpropimorph	Yes	0.052	15	EtOAc			No	No	No	GPC	PS-ML	MSD		None	Swedish
	Fludioxonil	Yes	0.046	15	EtOAc			No	No	No	GPC	PS-ML	MSD		None	Swedish

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1]	Extraction solvent II [1]	Extraction solvent III [1]	Water addition	Hydrolysis	pH adjused	Clean up [2]	Calibration [3]	GC detector	HPLC detector	Confirmation	Reference method
	Flusilazole	Yes	0.214	15	EtOAc			No	No	No	GPC	PS-ML	MSD		None	Swedish
	Lambda-cyhalothrin	Yes	0.042	15	EtOAc			No	No	No	GPC	PS-ML	MSD		None	Swedish
	Tebuconazole	Yes	0.308	15	EtOAc			No	No	No	GPC	PS-ML	MSD		None	Swedish
eupt 121	Azoxystrobin	No	0.163	5	ACN			Yes	No	No	DSPE	MM-ML	MSD			QuEChERS
	Chlorpyrifos	No	1.16	5	ACN			Yes	No	No	DSPE	MM-ML	MSD			QuEChERS
	Cyprodinil	No	0.063	5	ACN			Yes	No	No	DSPE	MM-ML	MSD			QuEChERS
	Fludioxonil	No	0.077	5	ACN			Yes	No	No	DSPE	MM-ML	MSD			QuEChERS
	Flusilazole	No	0.692	5	ACN			Yes	No	No	DSPE	MM-ML	MSD			QuEChERS
	Lambda-cyhalothrin	No	0.163	5	ACN			Yes	No	No	DSPE	MM-ML	MSD			QuEChERS
	Tebuconazole	No	1.16	5	ACN			Yes	No	No	DSPE	MM-ML	MSD			QuEChERS
eupt 124	Azoxystrobin	No	0.089	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML	MS/MS		GC-MS/MS	
	Chlorpyrifos	No	1.21	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML	MS/MS		GC-MS/MS	
	Cyprodinil	No	0.087	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML	MS/MS		GC-MS/MS	
	Tebuconazole	No	1,593	10	Acetone	CH_2Cl_2	Other	Yes	No	No	None	MM-ML	MS/MS		GC-MS/MS	
eupt 125	Azoxystrobin	No	0.237	10	ACN			Yes	No	No	SPE	MM-SL	MSD		GC/MS	QuEChERS
	Chlorpyrifos	No	1,033	10	ACN			Yes	No	No	SPE	MM-SL	MSD		GC/MS	QuEChERS
	Lambda-cyhalothrin	No	0.034	10	ACN			Yes	No	No	SPE	MM-SL	MSD		GC/MS	QuEChERS
	Malathion	No	0.021	10	ACN			Yes	No	No	SPE	MM-SL	MSD		GC/MS	QuEChERS
Eupt 126	Chlorpyrifos	No	1.21	5	ACN			Yes			DSPE	MM-ML	NPD		GC/MS	quechers
	Tebuconazole	No	1.75	5	ACN			Yes			DSPE	MM-ML	MSD			quechers
eupt 127	Azoxystrobin	Yes	0.162	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Meth. 3.3.7.1. 2008
	Carbendazim and benomyl	Yes	1.98	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Meth. 3.3.7.1. 2008
	Chlorpyrifos	Yes	1.08	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Meth. 3.3.7.1. 2008
	Cyproconazole	Yes	0.509	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Meth. 3.3.7.1. 2008

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1)]	Extraction solvent II [1)]	Extraction solvent III [1)]	Water addition	Hydrolysis	pH adjused	Clean up [2)]	Calibration [3)]	GC detector	HPLC detector	Confirmation	Reference method
	Cyprodinil	Yes	0.0806	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Meth. 3.3.7.1. 2008
	Fenvalerate and Esfenvalerate	Yes	0.103	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	PS-ML	ECD		GC/MS	VDLUFA-Meth. 3.3.7.1. 2008
	Fenbuconazole	Yes	0.474	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Meth. 3.3.7.1. 2008
	Fenpropimorph	Yes	0.0957	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Meth. 3.3.7.1. 2008
	Fludioxonil	Yes	0.118	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Meth. 3.3.7.1. 2008
	Flusilazole	Yes	0.727	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Meth. 3.3.7.1. 2008
	Lambda-cyhalothrin	Yes	0.0542	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Meth. 3.3.7.1. 2008
	Metconazole	Yes	0.502	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Meth. 3.3.7.1. 2008
	Pyraclostrobin	Yes	0.657	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Meth. 3.3.7.1. 2008
	Tebuconazole	Yes	1.38	25	Acetone	C_6H_{12}	EtOAc	Yes	No	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Meth. 3.3.7.1. 2008
eupt 129	Azoxystrobin	No	0.18	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		None	QuEChERS
	Chlorpyrifos	No	1,038	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		None	QuEChERS
	Lambda-cyhalothrin	Yes	0.068	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		None	QuEChERS
	Malathion	Yes	0.018	5	ACN			Yes	No	No	DSPE	MM-ML	MSD		None	QuEChERS
eupt 130	Azoxystrobin	Yes	0.22	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	None	QuEChERS _LCMS
	Carbendazim and benomyl	Yes	0.27	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	None	QuEChERS _LCMS
	Chlorpyrifos	Yes	1.44	5	ACN			Yes	No	No	DSPE	MM-ML	MS/MS		LC-MS/MS	QuEChERS _GCMS
	Cyproconazole	Yes	0.317	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	None	QuEChERS _LCMS
	Cyprodinil	Yes	0.068	5	ACN			Yes	No	No	DSPE	MM-ML	MS/MS		LC-MS/MS	QuEChERS _GCMS
	Fenbuconazole	Yes	0.356	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	None	QuEChERS _LCMS
	Fenpropimorph	Yes	0.064	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	None	QuEChERS _LCMS

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1)]	Extraction solvent II [1)]	Extraction solvent III [1)]	Water addition	Hydrolysis	pH adjused	Clean up [2)]	Calibration [3)]	GC detector	HPLC detector	Confirmation	Reference method
	Fludioxonil	Yes	0.089	5	ACN			Yes	No	No	DSPE	MM-ML	MS/MS		LC-MS/MS	QuEChERS _GCMS
	Flusilazole	Yes	0.245	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	None	QuEChERS _LCMS
	Lambda-cyhalothrin	Yes	0.029	5	ACN			Yes	No	No	DSPE	MM-ML	MS/MS		LC-MS/MS	QuEChERS _GCMS
	Metconazole	Yes	0.227	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	None	QuEChERS _LCMS
	Pyraclostrobin	Yes	0.214	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	None	QuEChERS _LCMS
	Tebuconazole	Yes	0.453	5	ACN			Yes	No	No	DSPE	MM-ML		MS/MS	None	QuEChERS _LCMS
eupt 133	Chlorpyrifos	Yes	0.598	5	ACN			Yes	No	pH 5	DSPE	MM-ML	MSD		GC/MS	PR EN 15662
	Cyproconazole	Yes	0.359	5	ACN			Yes	No	pH 5	DSPE	MM-ML	MSD		GC/MS	PR EN 15662
	Cyprodinil	Yes	0.044	5	ACN			Yes	No	pH 5	DSPE	MM-ML	MSD		GC/MS	PR EN 15662
	Fenvalerate and Esfenvalerate	Yes	0.078	5	ACN			Yes	No	pH 5	DSPE	MM-ML	MSD		GC/MS	PR EN 15662
eupt 138	Azoxystrobin	No	0.209	5	ACN			Yes			Freez.	MM-ML		MS/MS	LC-MS/MS	QuEChERS
	Chlorpyrifos	No	0.931	20	EtOAc			No			GPC	MM-ML	MSD		GC/MS	Internal Method
	Cyproconazole	No	0.476	5	ACN			Yes			Freez.	MM-ML		MS/MS	LC-MS/MS	QuEChERS
	Fenvalerate and Esfenvalerate	No	0.091	20	EtOAc			No			GPC	MM-ML	MSD		GC/MS	Internal Method
	Fenbuconazole	No	0.534	5	ACN			Yes			Freez.	MM-ML		MS/MS	LC-MS/MS	QuEChERS
	Flusilazole	No	0.769	5	ACN			Yes			Freez.	MM-ML		MS/MS	LC-MS/MS	QuEChERS
	Lambda-cyhalothrin	No	0.048	0												
	Pyraclostrobin	No	0.81	5	ACN			Yes			Freez.	MM-ML		MS/MS	LC-MS/MS	QuEChERS
	Tebuconazole	No	1.41	5	ACN			Yes			Freez.	MM-ML		MS/MS	LC-MS/MS	QuEChERS
eupt 140	Azoxystrobin	No	0.178	5	ACN			Yes	No	No	DSPE	PS-ML		MS/MS	None	QuEChERS
	Carbendazim and benomyl	No	0.36	5	ACN			Yes	No	No	DSPE	PS-ML		MS/MS	None	QuEChERS
	Chlorpyrifos	No	1.02	5	ACN			Yes	No	No	DSPE	PS-ML		MS/MS	None	QuEChERS
	Cyproconazole	No	0.461	5	ACN			Yes	No	No	DSPE	PS-ML		MS/MS	None	QuEChERS
	Cyprodinil	No	0.0716	5	ACN			Yes	No	No	DSPE	PS-ML		MS/MS	None	QuEChERS
	Fenvalerate and Esfenvalerate	No	0.0697	5	ACN			Yes	No	No	DSPE	PS-ML	MSD		None	QuEChERS

Lab-Code	Pesticide	Accredited	Reporting result [mg/kg]	Sample weight [g]	Extraction solvent I [1)]	Extraction solvent II [1)]	Extraction solvent III [1)]	Water addition	Hydrolysis	pH adjused	Clean up [2)]	Calibration [3)]	GC detector	HPLC detector	Confirmation	Reference method
	Fenbuconazole	No	0.636	5	ACN			Yes	No	No	DSPE	PS-ML		MS/MS	None	QuEChERS
	Fenpropimorph	No	0.0953	5	ACN			Yes	No	No	DSPE	PS-ML		MS/MS	None	QuEChERS
	Fludioxonil	No	0.0931	5	ACN			Yes	No	No	DSPE	PS-ML		MS/MS	None	QuEChERS
	Flusilazole	No	0.776	5	ACN			Yes	No	No	DSPE	PS-ML		MS/MS	None	QuEChERS
	Lambda-cyhalothrin	No	0.0754	5	ACN			Yes	No	No	DSPE	PS-ML	MSD		None	QuEChERS
	Malathion	No	0.01	5	ACN			Yes	No	No	DSPE	PS-ML		MS/MS	None	QuEChERS
	- Malathion	No	0.01	5	ACN			Yes	No	No	DSPE	PS-ML		MS/MS	None	QuEChERS
	Pyraclostrobin	No	0.698	5	ACN			Yes	No	No	DSPE	PS-ML		MS/MS	None	QuEChERS
	Tebuconazole	No	1.28	5	ACN			Yes	No	No	DSPE	PS-ML		MS/MS	None	QuEChERS
eupt 141	Chlorpyrifos	No	1.42	5	Acetone	CH_2Cl_2		Yes			SPE		NPD		None	
eupt 152	Azoxystrobin	No	0.127	25	Acetone	CH_2Cl_2	Other	No	No	No	None	PS-ML	ECD		2 colns	SAR-2-04p
	Carbendazim and benomyl	No	0.59	50	EtOAc			No	No	No	liq./liq	PS-ML		UV	2 colns	SAR-1-02
	Chlorpyrifos	No	0.734	25	Acetone	CH_2Cl_2	Other	No	No	No	None	MM-ML	NPD		2 colns	SAR-1-04
	Cyprodinil	No	0.0352	12	EtOAc	CH_2Cl_2		No	No	No	None	MM-ML	NPD		GC/MS	SAR-3-06
	Fenvalerate and Esfenvalerate	No	0.0768	25	Acetone	CH_2Cl_2	Other	No	No	No	None	PS-ML	ECD		2 colns	SAR-2-04p
	Fludioxonil	No	0.0421	12	EtOAc	CH_2Cl_2		No	No	No	None	MM-ML	NPD		GC/MS	SAR-3-06
	Lambda-cyhalothrin	No	0.0687	25	Acetone	CH_2Cl_2	Other	No	No	No	None	PS-ML	ECD		2 colns	SAR-2-04p
	Tebuconazole	No	0.712	25	Acetone	CH_2Cl_2	Other	No	No	No	None	MM-ML	NPD		2 colns	SAR-1-04
eupt 153	Chlorpyrifos	No	1.68	15	ACN			No	No	No	liq./liq	MM-ML	NPD		GC-MS/MS	SR EN 12393
	Deltamethrin. cis-deltamethrin	Yes	0.053	15	ACN			No	No	No	liq./liq	MM-ML	ECD		GC-MS/MS	SR EN 12393
	Fenvalerate and Esfenvalerate	No	0.343	15	ACN			No	No	No	liq./liq	MM-ML	ECD		GC-MS/MS	SR EN 12393
	Malathion	Yes	0.015	15	ACN			No	No	No	liq./liq	MM-ML	NPD		GC-MS/MS	SR EN 12393

1). ACN: Acetonitile; MeOH: Methanol; EtOAc: Ethyl Acetate; C2H6: Cyclohexane; CH2Cl2: Dichlormethane

2). SPE: Solid Phase Ext. col.; DSPE: Disp. Solid Phase Ext.: liq. / liq..: liquid/liquid partitioning; GPC: Gel Perm. Chromatogr.; Freez.: Freezing out

3). MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD-Add.: Standard addition

SRM APPENDICES

Appendix 8 Target Pesticide List of SRM Pesticides

SRM-Parameter	Residue definition within this PT	Optional [2)]	MRRL [1)] [mg/kg]
* 2,4-D	following alkaline hydrolysis		0.02
2,4-D	free acid	Optional	0.02
Bentazone	only parent		0.02
Dicamba	free acid		0.02
Dicamba	following alkaline hydrolysis	Optional	0.02
Dichlorprop incl. Dichlorprop-P	free acids		0.02
Dichlorprop incl. Dichlorprop-P	following alkaline hydrolysis	Optional	0.02
Fluazifop incl. Fluazifop-P	following alkaline hydrolysis		0.02
Fluazifop incl. Fluazifop-P	free acids	Optional	0.02
Fluroxypyr	following alkaline hydrolysis		0.02
Fluroxypyr	free acid	Optional	0.02
Haloxyfop incl. Haloxyfop-R	following alkaline hydrolysis		0.02
Haloxyfop incl. Haloxyfop-R	free acids	Optional	0.02
* MCPA	following alkaline hydrolysis		0.02
MCPA	free acid	Optional	0.02
Mecoprop incl. Mecoprop-P	free acids		0.02
Mecoprop incl. Mecoprop-P	following alkaline hydrolysis	Optional	0.02
Ethephon			0.02
Glyphosate			0.02
* Chlormequat	free cation		0.02
* Mepiquat	free cation		0.02

[1)] MRRL: Minimum Required Reporting Level

[2)] Optional means that the results will be out of competition.

Explanatory note concerning the PT-specific residue definitions for acidic pesticides given above: Where the legal residue definition includes only the free acid the result following alkaline hydrolysis is OPTIONAL. And vice versa, where the legal residue definition includes the esters and/or conjugates the result for the free acid is OPTIONAL. OPTIONAL means that the results will be out competition and mainly used for scientific evaluation for example to support the ongoing discussions concerning the legal residue definitions of acids.

Appendix 9 Homogeneity data of SRM-pesticides

2,4-D (foll. akl. hydr.) [mg/kg]		
Sample	Portion 1	Portion 2
1	0.493	0.502
2	0.476	0.474
3	0.485	0.477
4	0.504	0.490
5	0.498	0.470
6	0.538	0.519
7	0.465	0.471
8	0.496	0.516
9	0.460	0.477
10	0.514	0.490

Dicamba (foll. akl. hydr.) [mg/kg]		
Sample	Portion 1	Portion 2
1	0.276	0.254
2	0.284	0.285
3	0.268	0.287
4	0.262	0.267
5	0.277	0.258
6	0.250	0.242
7	0.291	0.279
8	0.283	0.275
9	0.259	0.274
10	0.292	0.297

Chlormequat [mg/kg]		
Sample	Portion 1	Portion 2
1	0.505	0.496
2	0.531	0.515
3	0.519	0.523
4	0.469	0.480
5	0.503	0.524
6	0.575	0.587
7	0.523	0.532
8	0.509	0.489
9	0.520	0.519
10	0.504	0.507

Ethephone [mg/kg]		
Sample	Portion 1	Portion 2
1	0.532	0.549
2	0.539	0.527
3	0.548	0.549
4	0.501	0.520
5	0.571	0.596
6	0.557	0.556
7	0.564	0.585
8	0.567	0.555
9	0.547	0.540
10	0.545	0.541

Glyphosate [mg/kg]		
Sample	Portion 1	Portion 2
1	1.260	1.143
2	1.152	1.149
3	1.283	1.183
4	1.155	1.167
5	1.297	1.244
6	1.269	1.295
7	1.191	1.195
8	1.252	1.211
9	1.229	1.283
10	1.177	1.223

Appendix 10 Detailed stability data of SRM-pesticides

2,4-D (foll. alk. hydr.) [mg/kg]			
Subsample	10.01.2009	5.02.2009	10.03.2009
1	0.500	0.483	0.428
2	0.479	0.493	0.449
3	0.455	0.490	0.469
4	0.421	0.503	0.432
5	0.483	0.510	0.418
Mean [mg/kg]	0.468	0.496	0.439
Standard Diviation	0.031	0.11	0.02
Difference	**—**	**6 %**	**-6.1 %**

2,4-D (free acid) [mg/kg]			
Subsample	10.01.2009	5.02.2009	10.03.2009
1	0.448	—	0.451
2	0.442	—	0.457
3	0.435	—	0.420
4	0.438	—	0.448
5	0.435	—	0.479
Mean [mg/kg]	0.440	—	0.451
Standard Diviation [mg/kg]	0.005	—	0.022
Difference	**—**	**—**	**2.6 %**

Dicamba (foll. alk. hydr.) [mg/kg]			
Subsample	10.01.2009	5.02.2009	10.03.2009
1	0.291	0.301	0.290
2	0.291	0.305	0.295
3	0.294	0.308	0.290
4	0.270	0.304	0.286
5	0.298	0.306	0.262
Mean [mg/kg]	0.289	0.305	0.285
Standard Diviation [mg/kg]	0.011	0.003	0.013
Difference	**—**	**5.5 %**	**-1.5 %**

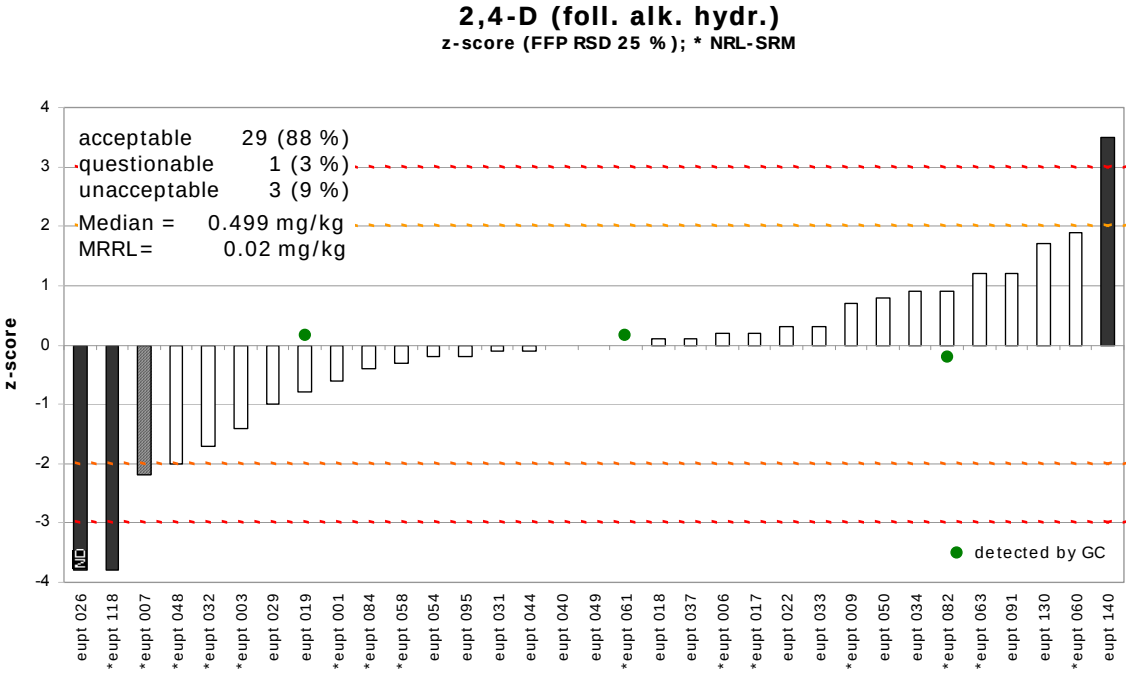
Dicamba (free acid) [mg/kg]			
Subsample	10.01.2009	5.02.2009	10.03.2009
1	0.150	—	0.149
2	0.148	—	0.148
3	0.149	—	0.148
4	0.146	—	0.147
5	0.141	—	0.149
Mean [mg/kg]	0.147	—	0.148
Standard Diviation [mg/kg]	0.004	—	0.001
Difference	—	—	**1.0 %**

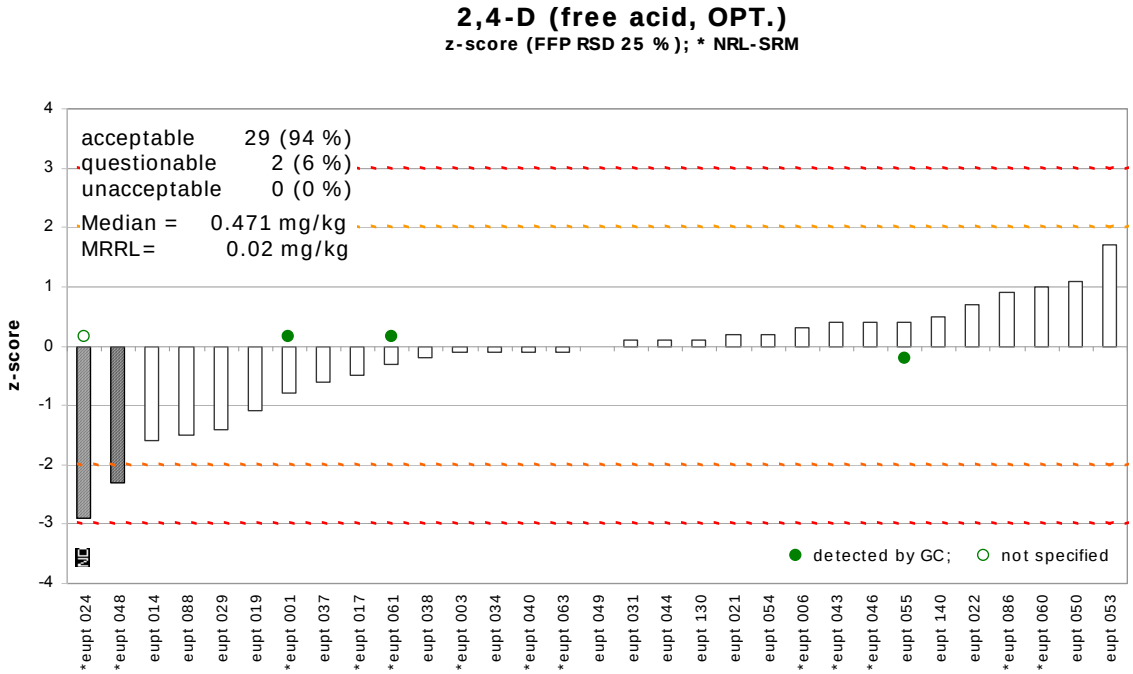
Chlormequat [mg/kg]			
Subsample	10.01.2009	5.02.2009	10.03.2009
1	0.496	0.532	0.480
2	0.492	0.500	0.476
3	0.504	0.512	0.476
4	0.492	0.492	0.480
5	0.532	0.504	0.484
Mean [mg/kg]	0.503	0.508	0.479
Standard Diviation [mg/kg]	0.017	0.015	0.003
Difference	**—**	**1.0 %**	**-4.8 %**

Ethephone [mg/kg]			
Subsample	10.01.2009	5.02.2009	10.03.2009
1	0.580	0.536	0.556
2	0.568	0.528	0.520
3	0.544	0.536	0.544
4	0.548	0.556	0.516
5	0.556	0.556	0.516
Mean [mg/kg]	0.559	0.542	0.530
Standard Diviation [mg/kg]	0.015	0.013	0.018
Difference	**—**	**-3.5 %**	**-5.2 %**

Glyphosate [mg/kg]			
Subsample	10.01.2009	5.02.2009	10.03.2009
1	1.200	1.276	1.348
2	1.200	1.228	1.268
3	1.200	1.284	1.276
4	1.140	1.268	1.236
5	1.208	1.260	1.208
Mean [mg/kg]	1.190	1.263	1.267
Standard Diviation [mg/kg]	0.028	0.022	0.053
Difference	**—**	**6.2 %**	**3.6 %**

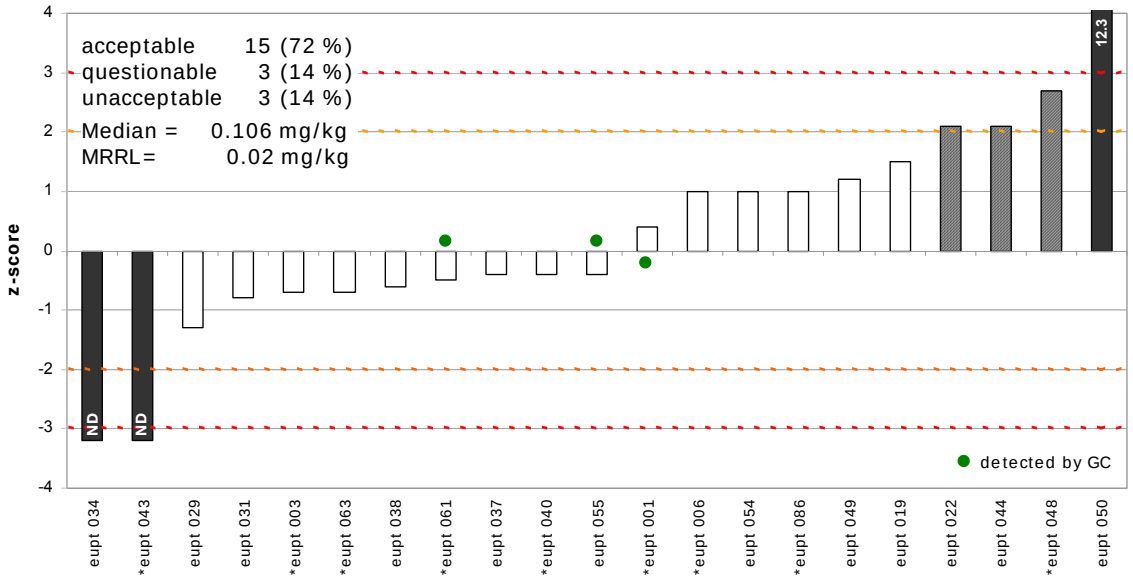
Appendix 11 Graphical presentation of z-scores for each SRM-pesticide





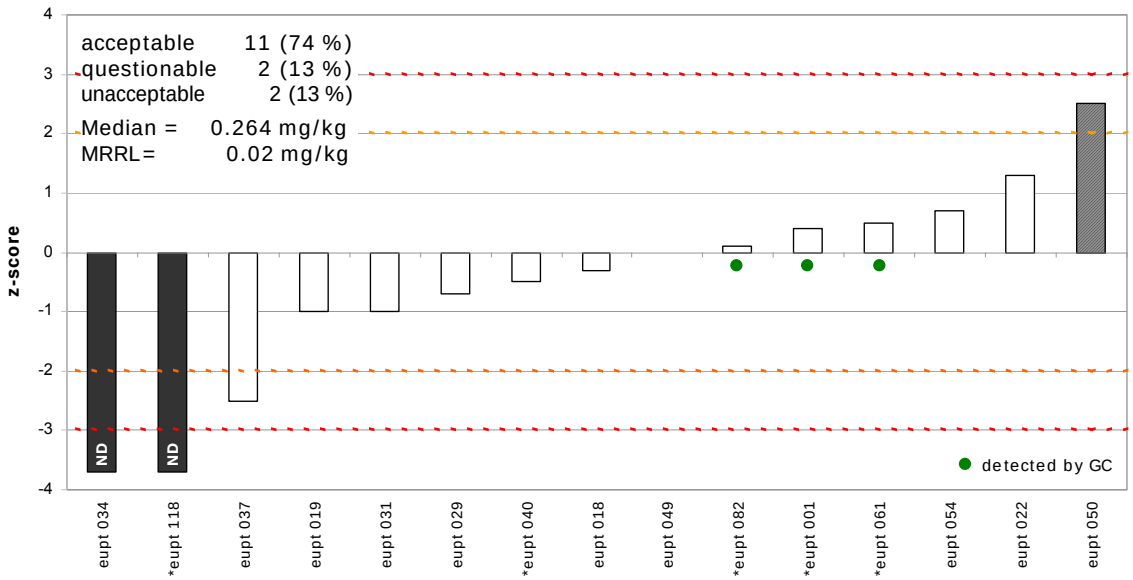
Dicamba (free acid)

z-score (FFP RSD 25 %); * NRL-SRM



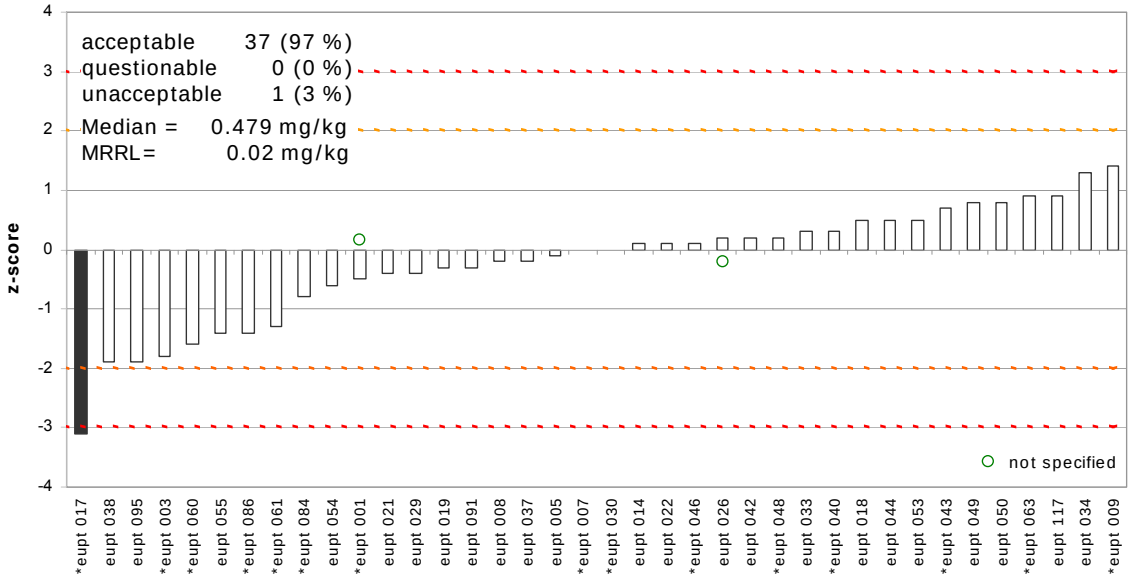
Dicamba (foll. alk. hydr. OPT.)

z-score (FFP RSD 25 %); * NRL-SRM

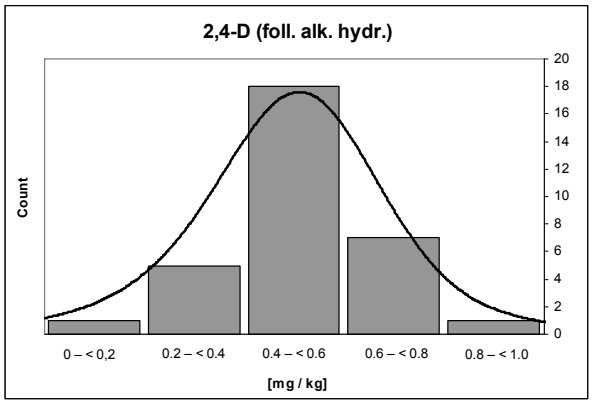


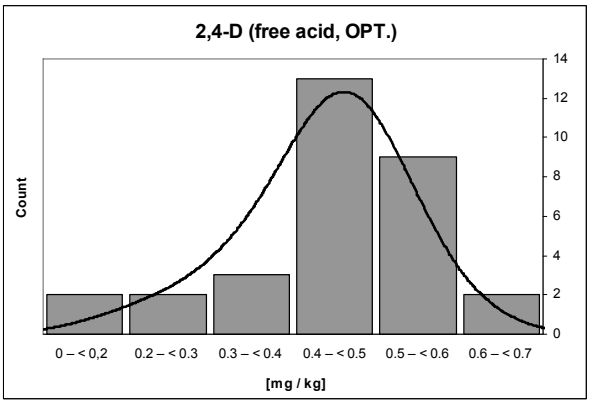
Chlormequat

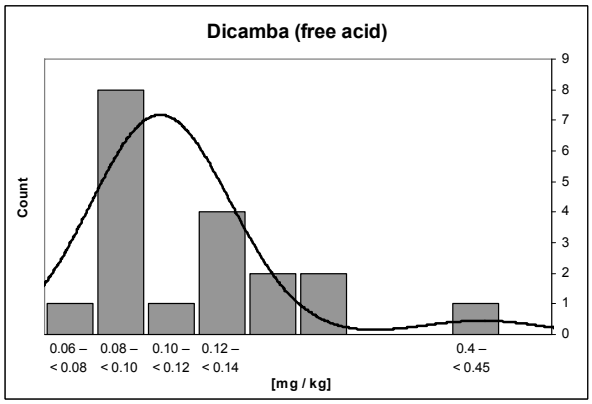
z-score (FFP RSD 25 %); * NRL-SRM

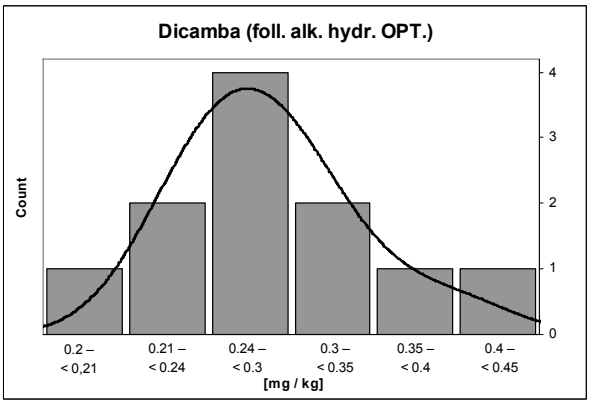


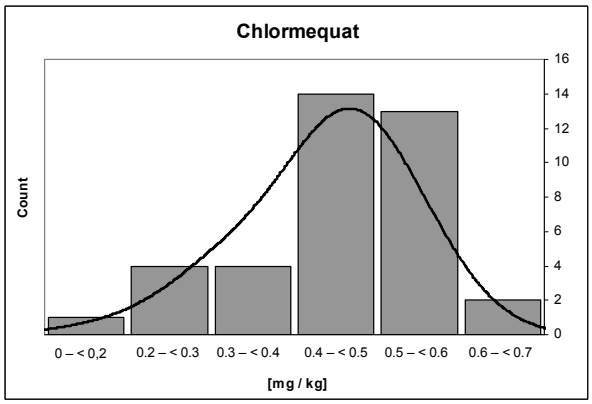
Appendix 12 Histograms of SRM-pesticides











Appendix 13 SRM-Methods used by the participating Laboratories

Lab-Code	Pesticide	Accredited?	Reproted result [mg/kg]	Reporting level [mg/kg]	Sample weight [g]	Water addition	Hydrolysis	Extraction solvent [1]	ISTD used / ISTD details [2]	pH adjusted?	Clean up [3]	Calibration [4]	GC detector	HPLC detector	Confirmation	Recovery approach from	Recovery [%]	Recovery correction	Reference method
eupt 001	2,4-D, foll. alk. hydr.	No	0.427	0.02	5	Yes	Yes	ACN	Yes, isotop. labelled-1	Yes	SPE	MM - ML	MSD		GC/MS	Same batch	79	No	
	2,4-D, free acid, OPT.	No	0.373	0.02	5	Yes	No	ACN	Yes, isotop. labelled-1	Yes	SPE	MM - ML	MSD		GC/MS	Same batch	79	No	
	Chlormequat (free cation)	No	0.115	0.02	0												114	Yes autom.	
	Dicamba, foll. alk. hydr., OPT.	No	0.29	0.02	5	Yes	Yes	ACN	Yes, isotop. labelled-1	Yes	SPE	MM - ML	MSD		GC/MS	Same batch	79	No	
	Dicamba, free acid	No	0.416	0.02	5	Yes	No	ACN	Yes, isotop. labelled-1	Yes	SPE	MM - ML	MSD		GC/MS	Same batch	79	No	
eupt 003	2,4-D, foll. alk. hydr.	No	0.321	0.02	5	Yes	Yes	ACN	No		None	MM - ML		MS/MS	LC-MS/MS	Same batch	69	No	Anastassiades, M. & Lehotay, S.J.(2003), J. AOAC Int. 86, 412-431
	2,4-D, free acid, OPT.	No	0.458	0.02	5	Yes	No	ACN	No	Yes	None	MM - ML		MS/MS	LC-MS/MS	Same batch	89	No	Anastassiades, M. & Lehotay, S.J.(2003), J. AOAC Int. 86, 412-431
	Chlormequat (free cation)	Yes	0.26	0.02	20	No		Other; MeOH				MM - ML		MS/MS	LC-MS/MS	Same batch	65	No	
	Dicamba, free acid	No	0.0876	0.02	5	Yes	No	ACN	No	Yes	None	MM - ML		MS/MS	LC-MS/MS	Same batch	82	No	Anastassiades, M. & Lehotay, S.J.(2003), J. AOAC Int. 86, 412-431
	Glyphosate	Yes	1.1	0.05	25	No	No	Other; MeOH	Yes, Glyphosate labelled with C13 and N15	Yes	DSPE	MM - ML	MSD		LC-MS/MS	Same batch	66	No	
eupt 005	Chlormequat (free cation)	Yes	0.47	0.01	20	Yes	No	MeOH	Yes, isotop. labelled-1	No	None	PS - ML		MS/MS	None	Isotope labelled std.	103	Yes autom.	CEN/TC 275 EN 15054 (2005)
eupt 006	2,4-D, foll. alk. hydr.	No	0.526	0.01	5	Yes	Yes	ACN	No	pH 12	None	MM - SL		MS/MS	LC-MS/MS	Same batch	94	No	
	2,4-D, free acid, OPT.	Yes	0.512	0.01	5	Yes	No	ACN	No	pH 1	None	MM - SL		MS/MS	LC-MS/MS	Same batch	92	No	
	Dicamba, free acid	No	0.131	0.01	5	Yes	No	ACN	No	pH 1	None	MM - SL		MS/MS	LC-MS/MS	Same batch	97	No	

Lab-Code	Pesticide	Accredited?	Reproted result [mg/kg]	Reporting level [mg/kg]	Sample weight [g]	Water addition	Hydrolysis	Extraction solvent [1]	ISTD used / ISTD details [2]	pH adjusted?	Clean up [3]	Calibration [4]	GC detector	HPLC detector	Confirmation	Recovery approach from	Recovery [%]	Recovery correction	Reference method
eupt 007	2,4-D, foll. alk. hydr.	Yes	0.22	0.02	5	Yes	Yes	ACN	No	pH 12	Freez.	MM - ML		MS/MS	LC-MS/MS			No	In house
	Chlormequat (free cation)	Yes	0.48	0.02	5	Yes	No	MeOH	Yes, isotop. labelled-1	No	None	MM - ML		MS/MS	LC-MS/MS			No	In house
eupt 008	Chlormequat (free cation)	Yes	0.455	0.01	10	Yes		MeOH	Yes, isotop. labelled-1	Yes	None	MM - ML		MS/MS		Same batch	85	No	
eupt 009	2,4-D, foll. alk. hydr.	No	0.58	0.02	5	Yes	Yes	ACN	No	No	None	MM - ML		MS/MS	LC-MS/MS	Same batch	120	No	
	Chlormequat (free cation)	No	0.645	0.02	5	Yes	Yes	ACN	No	No	None	MM - ML		MS/MS	LC-MS/MS	Same batch	37	Yes	
eupt 014	2,4-D, free acid, OPT.	No	0.288	0.02	5	Yes	No	ACN	No	No	Freez.	PS - ML		MS/MS	LC-MS/MS	Same batch	69	No	QuEChERS
	Chlormequat (free cation)	No	0.493	0.02	5			MeOH		No	None	PS - ML		MS		Same batch	77	No	ASE
eupt 017	2,4-D, foll. alk. hydr.	No	0.52	0.0003	5	Yes	Yes	ACN	Yes, other (Nicarbazin)	Yes	Freez.	MM - SL		MS/MS	None	Same batch	112.2	No	QuEChERS
	2,4-D, free acid, OPT.	No	0.407	0.0002	5	Yes	No	ACN	Yes, other (Nicarbazin)	No	Freez.	MM - SL		MS/MS	None	Same batch	90.5	No	QuEChERS
	Chlormequat (free cation)	No	0.108	0.0003	5	Yes	No	MeOH	No	No	None	MM - SL		MS/MS	None	Same batch	67	No	Quick method
eupt 018	2,4-D, foll. alk. hydr.	No	0.516	0.02	5	Yes	Yes	ACN	Yes, isotop. labelled-2	Yes	DSPE	MM - ML		MS/MS		Same batch	90	No	
	Chlormequat (free cation)	No	0.538	0.02	10	Yes	No	MeOH	No	No	None	MM - ML		MS/MS		Same batch	105	No	
	Dicamba, foll. alk. hydr., OPT.	No	0.245	0.1	5	Yes	Yes	ACN	Yes, isotop. labelled-2	Yes	DSPE	MM - ML		MS/MS		Same batch	75	No	
eupt 019	2,4-D, foll. alk. hydr.	No	0.4	0.02	50	Yes	Yes	MeOH	No	pH 5	liq. / liq.	MM - ML		MS/MS	LC-MS/MS	Same batch	98.3	No	Modifizierte BfR-Methode (ChemElut, pH4,5) gemäß §64 LFBG-Methodenentwurf
	2,4-D, free acid, OPT.	Yes	0.346	0.02	50	Yes	No	MeOH	No	No	liq. / liq.	MM - ML		MS/MS	LC-MS/MS	Same batch	83.0	No	Modifizierte BfR-Methode (ChemElut, pH4,5) gemäß §64 LFBG-Methodenentwurf
	Chlormequat (free cation)	Yes	0.437	0.02	50	Yes	No	MeOH	Yes, isotop. labelled-1	No	None	MM - ML		MS/MS	LC-MS/MS	Same batch	97.8	No	LC-MS/MS gemäß § 64 LFGB, L00.00-76
	Dicamba, foll. alk. hydr., OPT.	No	0.201	0.04	50	Yes	Yes	MeOH	No	pH 5	liq. / liq.	MM - ML		MS/MS	LC-MS/MS	Same batch	63.0	No	Modifizierte BfR-Methode (ChemElut, pH4,5) gemäß §64 LFBG-Methodenentwurf

Lab-Code	Pesticide	Accredited?	Reproted re-sult [mg/kg]	Reporting level [mg/kg]	Sample weight [g]	Water addition	Hydrolysis	Extraction solvent [1]	ISTD used / ISTD details [2]	pH adjusted?	Clean up [3]	Calibration [4]	GC detector	HPLC detector	Confirmation	Recovery approach from	Recovery [%]	Recovery correction	Reference me-thod
	Dicamba, free acid	Yes	0.146	0.04	50	Yes	No	MeOH	No	No	liq. / liq.	MM - ML		MS/MS	LC-MS/MS	Same batch	68.0	No	Modifizierte BfR-Methode (ChemElut, pH4,5) gemäß §64 LFBG-Methodenentwurf
eupt 021	2,4-D, free acid, OPT.	Yes	0.5	0.15	3	Yes	No	MeOH	Yes, isotop. labelled C13-Carbaryl	No	Other	MM - ML		MS/MS	LC-MS/MS	Same batch	45	No	FP086
	Chlormequat (free cation)	Yes	0.43	0.01	10	No	No	MeOH	Yes, isotop. labelled D4-chlormequat	No	SPE	MM - ML		MS/MS	LC-MS/MS	Same batch	90	No	FP045
eupt 022	2,4-D, foll. alk. hydr.	No	0.54	0.02	5	Yes	Yes	ACN		Yes		MM - ML		MS/MS	LC-MS/MS	Same batch	90	Yes	QuEChERS
	2,4-D, free acid, OPT.	No	0.555	0.02	5	Yes		ACN		Yes		MM - ML		MS/MS	LC-MS/MS	Same batch	90	Yes	QuEChERS
	Chlormequat (free cation)	Yes	0.495	0.01	5	Yes		MeOH	Yes, isotop. labelled-1			PS - ML		MS/MS	LC-MS/MS	Same batch	100	Yes	EN15054
	Dicamba, foll. alk. hydr., OPT.	No	0.35	0.02	5	Yes	Yes	ACN		Yes		MM - ML		MS/MS	LC-MS/MS	Same batch	85	Yes	QuEChERS
	Dicamba, free acid	No	0.16	0.02	5	Yes		ACN		Yes		MM - ML		MS/MS	LC-MS/MS	Same batch	85	Yes	QuEChERS
	Glyphosate	Yes	1.43	0.02	5	Yes		Other	Yes, isotop. labelled-1			PS - ML		MS/MS	LC-MS/MS	Same batch	100	Yes	In house
eupt 024	2,4-D, free acid, OPT.	No	0.13	0.02	0												26.8	No	
eupt 026	2,4-D, foll. alk. hydr.		ND																
	Chlormequat (free cation)	No	0.5	0.02	0												83	No	
eupt 029	2,4-D, foll. alk. hydr.	No	0.372	0.01	10	Yes	Yes	MeOH	No	No	None	PS - ML		MS/MS	LC-MS/MS	Same batch	86.5	No	LMS2_A
	2,4-D, free acid, OPT.	Yes	0.309	0.01	10	No	No	MeOH	No	No	None	PS - ML		MS/MS	LC-MS/MS	Same batch	52.8	No	LMS2
	Chlormequat (free cation)	No	0.43	0.05	5	No	No	MeOH	Yes, isotop. labelled-1	No	None	PS - ML		MS/MS	LC-MS/MS	Same batch	95	Yes autom.	CCC
	Dicamba, foll. alk. hydr., OPT.	No	0.219	0.01	10	Yes	Yes	MeOH	No	No	None	PS - ML		MS/MS	LC-MS/MS	Same batch	90.8	No	LMS2_A
	Dicamba, free acid	Yes	0.071	0.01	10	No	No	MeOH	No	No	None	PS - ML		MS/MS	LC-MS/MS	Same batch	77	No	LMS2
	Ethephon	No	0.79	0.05	5	Yes	No	EtOAc	No	No	None	MM - ML	FPD		None	Same batch	35	Yes	ETHEFON
eupt 030	Chlormequat (free cation)	No	0.477	0.02	10	Yes	No	MeOH	Yes, isotop. labelled D4-Chlormequat Chloride	No	None	PS - ML		MS/MS	LC-MS/MS	Same batch	110	No	As published by the CRL-SRM in the CRL-Website

Lab-Code	Pesticide	Accredited?	Reproted re-sult [mg/kg]	Reporting level [mg/kg]	Sample weight [g]	Water addition	Hydrolysis	Extraction solvent [1]	ISTD used / ISTD details [2]	pH adjusted?	Clean up [3]	Calibration [4]	GC detector	HPLC detector	Confirmation	Recovery approach from	Recovery [%]	Recovery correction	Reference me-thod
eupt 031	2,4-D, foll. alk. hydr.	No	0.491	0.02	5	Yes	Yes	ACN	No	No	Freez.	PS - ML		MS/MS	LC-MS/MS	Same batch	70	No	prEN15662:2007
	2,4-D, free acid, OPT.	Yes	0.477	0.02	5	Yes	No	ACN	No	No	Freez.	PS - ML		MS/MS	LC-MS/MS	Same batch	97	No	prEN15662:2007
	Dicamba, foll. alk. hydr., OPT.	No	0.197	0.02	5	Yes	Yes	ACN	No	No	Freez.	PS - ML		MS/MS	LC-MS/MS	Same batch	85	No	prEN15662:2007
	Dicamba, free acid	Yes	0.085	0.02	5	Yes	No	ACN	No	No	Freez.	PS - ML		MS/MS	LC-MS/MS	Same batch	99	No	prEN15662:2007
	Glyphosate	No	1.24	0.02	3	Yes	No		No	No		PS - ML		MS	LC/MS	Same batch	92	No	Food Additives and Contaminants, Vol.20,No.8, 2003, pp.692-698
eupt 032	2,4-D, foll. alk. hydr.	No	0.29	0.02	5	Yes	Yes	ACN	Yes, other (Nicarbazin)	Yes	Freez.	MM - ML		MS/MS	LC-MS/MS	Same batch	59	No	
eupt 033	2,4-D, foll. alk. hydr.	No	0.533	0.01	5	Yes	Yes	ACN	No	No	None	MM - ML		MS/MS	Other	Same batch	36	Yes	ASU L00.00-115 mod.
	Chlormequat (free cation)	Yes	0.51	0.01	2	Yes	No	MeOH	No	No	None	MM - ML		MS/MS	None	Same batch	10	Yes	ASU L00.00-76 mod.
eupt 034	2,4-D, foll. alk. hydr.	Yes	0.606	0.15	5	Yes	Yes	ACN	Yes, other (Nicarbazin)	Yes	None	MM - ML		MS/MS	None		65	No	alkalic saponify before QuEChERS
	2,4-D, free acid, OPT.	Yes	0.458	0.15	5	Yes	No	ACN	Yes, other (Nicarbazin)	No	None	MM - ML		MS/MS	GC/MS		65	No	without DSPE QuEChERS
	Chlormequat (free cation)	Yes	0.634	0.02	10	Yes		MeOH	Yes, isotop. labelled D4-Chlormequat		None	PS - ML		MS/MS	None		115	No	gemäß §64 Method L00.00-76
	Dicamba, foll. alk. hydr., OPT.	Yes	ND	0,1	5	Yes	Yes	ACN	Yes, other (Nicarbazin)	Yes	None	MM - ML		MS/MS	None		0	No	alkalic saponify before QuEChERS
	Dicamba, free acid	Yes	ND	0,1	5	Yes	No	ACN	Yes, other (Nicarbazin)	No	None	MM - ML		MS/MS	None		0	No	without DSPE QuEChERS
eupt 037	2,4-D, foll. alk. hydr.	Yes	0.516	0.01	5	Yes	Yes	ACN	Yes, other (Nicarbazin)	No	DSPE	MM - ML		MS/MS	LC-MS/MS	Same batch	126	No	QuEChERS
	2,4-D, free acid, OPT.	Yes	0.406	0.01	5	Yes	No	ACN	Yes, isotop. labelled (other substance)	No	DSPE	MM - ML		MS/MS	LC-MS/MS	Same batch	126	No	QuEChERS
	Chlormequat (free cation)	Yes	0.499	0.01	10	Yes	No	MeOH	Yes, isotop. labelled-1	No	None	PS - ML		MS	LC-MS/MS	Same batch	97	No	gemäß §64 LFGB L00.00-76
	Dicamba, foll. alk. hydr., OPT.	Yes	0.098	0.01	5	Yes	Yes	ACN	Yes, other (Nicarbazin	No	DSPE	MM - ML		MS/MS	LC-MS/MS	Same batch		No	QuEChERS

Lab-Code	Pesticide	Accredited?	Reproted result [mg/kg]	Reporting level [mg/kg]	Sample weight [g]	Water addition	Hydrolysis	Extraction solvent [1]	ISTD used / ISTD details [2]	pH adjusted?	Clean up [3]	Calibration [4]	GC detector	HPLC detector	Confirmation	Recovery approach from	Recovery [%]	Recovery correction	Reference method
	Dicamba, free acid	Yes	0.096	0.01	5	Yes	No	ACN	Yes, isotop. labelled-2	No	DSPE	MM - ML		MS/MS	LC-MS/MS	Same batch		No	QuEChERS
	Ethephon	No	0.396	0.05	5	Yes	No	MeOH	Yes, isotop. labelled-1	No	None	MM - ML		MS/MS	LC-MS/MS	Same batch	99	No	High Polar Pesticides CRL Single residue Methods
	Glyphosate	No	1.05	0.05	5	Yes	No	MeOH	Yes, isotop. labelled-1	No	None	MM - ML		MS/MS	LC-MS/MS	Same batch	78	No	High Polar Pesticides CRL Single residue Methods
eupt038	2,4-D, free acid, OPT.	Yes	0.449	0.01	5	Yes	No	ACN	No	No	DSPE	MM - ML		MS/MS	LC-MS/MS	Same batch	61	No	QuEChERS
	Chlormequat (free cation)	Yes	0.25	0.025	5	Yes	No	ACN	No	No	DSPE	MM - ML		MS/MS	LC-MS/MS	Same batch	51	No	QuEChERS
	Dicamba, free acid	Yes	0.0892	0.05	5	Yes	No	ACN	No	No	DSPE	MM - ML		MS/MS	LC-MS/MS	Same batch	63	No	QuEChERS
eupt040	2,4-D, foll. alk. hydr.	No	0.502	0.02	5	Yes	Yes	ACN	No	pH 12	Freez.	MM - ML		MS/MS	LC-MS/MS	Same batch	92	No	QuEChERS
	2,4-D, free acid, OPT.	No	0.461	0.02	5	Yes	No	ACN	No	pH 5	Freez.	MM - ML		MS/MS	LC-MS/MS	Same batch	87	No	QuEChERS
	Chlormequat (free cation)	Yes	0.511	0.02	25	Yes		MeOH	Yes, isotop. labelled-1		None	PS - ML		MS/MS	LC-MS/MS	Same batch	79	No	CEN 15055
	Dicamba, foll. alk. hydr., OPT.	No	0.23	0.02	5	Yes	Yes	ACN	No	pH 12	Freez.	MM - ML		MS/MS	LC-MS/MS	Same batch	91	No	QuEChERS
	Dicamba, free acid	No	0.094	0.02	5	Yes	No	ACN	No	pH 5	Freez.	MM - ML		MS/MS	LC-MS/MS	Same batch	70	No	QuEChERS
	Glyphosate	No	1.51	0.02	3			Other	Yes, isotop. labelled-1		liq. / liq..	PS - ML		MS/MS	LC-MS/MS	Isotope labelled std.	100	Yes autom.	In house
eupt042	Chlormequat (free cation)	Yes	0.5	0.10	10	Yes	No	MeOH	No	No		MM - ML		MS/MS	LC-MS/MS	Same batch	85	No	
eupt043	2,4-D, free acid, OPT.	No	0.522	0.01	5	Yes		ACN			DSPE	STD - Add		MS/MS	LC-MS/MS	Same batch	88	No	QuEChERS
	Chlormequat (free cation)	Yes	0.565	0.01	5			MeOH	Yes, isotop. labelled-1			STD - Add		MS/MS	LC-MS/MS	Same batch	99	No	
	Dicamba, free acid	No	ND	0.1	5	Yes		ACN	No		DSPE			MS					QuEChERS
eupt044	2,4-D, foll. alk. hydr.	No	0.487	0.01	5	Yes	Yes	ACN	No	Yes	Other	MM - SL		MS/MS	LC-MS/MS	Same batch	80	No	
	2,4-D, free acid, OPT.	Yes	0.486	0.01	15	Yes	No	ACN	No	Yes	Other	MM - SL		MS/MS	LC-MS/MS	Same batch	84	No	SLV M915
	Chlormequat (free cation)	Yes	0.54	0.005	10	Yes	No	MeOH	Yes, isotop. labelled-1	No	Other	MM - SL		MS/MS	LC-MS/MS	Same batch	101	No	SLV M030
	Dicamba, free acid	No	0.162	0.01	15	Yes	No	ACN	No	Yes	Other	MM - SL		MS/MS	LC-MS/MS	Same batch	96	No	SLV M915
eupt	2,4-D, free acid, OPT.	Yes	0.519	0.02	2	Yes	No	ACN	No	No	Freez.	STD - Add		MS/MS	None		100	No	Quechers

Lab-Code	Pesticide	Accredited?	Reproted re-sult [mg/kg]	Reporting level [mg/kg]	Sample weight [g]	Water addition	Hydrolysis	Extraction solvent [1]	ISTD used / ISTD details [2]	pH adjusted?	Clean up [3]	Calibration [4]	GC detector	HPLC detector	Confirmation	Recovery approach from	Recovery [%]	Recovery correction	Reference me-thod
046	Chlormequat (free cation)	Yes	0.485	0.01	5	Yes	No	MeOH	Yes, isotop. labelled D4-Chlormequat	No	None	PS - ML		MS/MS	LC-MS/MS	Same batch	102	No	OE-Norm EN15055:2006
eupt 048	2,4-D, foll. alk. hydr.	No	0.244	0.02	5	Yes	Yes	MeOH	No	No	None	MM - ML		MS/MS	LC-MS/MS	Same batch	89	No	
	2,4-D, free acid, OPT.	No	0.195	0.02	5	Yes	No	MeOH	No	No	None	MM - ML		MS/MS	LC-MS/MS	Same batch	96	No	
	Chlormequat (free cation)	No	0.505	0.02	5	Yes	No	MeOH	No	No	None	STD - Add		MS/MS	LC-MS/MS	Same batch	77	Yes autom.	
	Dicamba, free acid	No	0.176	0.08	5	Yes	No	MeOH	No	No	None	MM - ML		MS/MS	LC-MS/MS	Same batch	88	No	
eupt 049	2,4-D, foll. alk. hydr.	Yes	0.495	0.01	5	Yes	Yes	ACN	Yes, isotop. labelled D3-MCPA	pH 5	None	MM - ML		MS/MS				No	QuEChERS
	2,4-D, free acid, OPT.	Yes	0.471	0.01	5	Yes	No	ACN	Yes, isotop. labelled D3-MCPA	pH 5	None	MM - ML		MS/MS				No	QuEChERS
	Chlormequat (free cation)	Yes	0.576	0.01	10	Yes	No	MeOH	Yes, isotop. labelled D4-Chlormequat Chloride	No	None	PS - ML		MS/MS				No	EN 15055:2006
	Dicamba, foll. alk. hydr., OPT.	Yes	0.264	0.01	5	Yes	Yes	ACN	Yes, isotop. labelled D3-MCPA	pH 5	None	MM - ML		MS/MS				No	QuEChERS
	Dicamba, free acid	Yes	0.136	0.01	5	Yes	No	ACN	Yes, isotop. labelled D3-MCPA	pH 5	None	MM - ML		MS/MS				No	QuEChERS
eupt 050	2,4-D, foll. alk. hydr.	Yes	0.6	0.02	2	Yes	Yes	MeOH	No	pH 7	Other	STD - Add		MS/MS	LC-MS/MS	Validation data	81	Yes autom.	
	2,4-D, free acid, OPT.	Yes	0.6	0.02	2	Yes	No	MeOH	No	No	Other	STD - Add		MS/MS	LC-MS/MS	Validation data	81	Yes autom.	
	Chlormequat (free cation)	Yes	0.58	0.02	2	Yes	No	MeOH	Yes, isotop. labelled-1	No	None	STD - Add		MS/MS	LC-MS/MS	Validation data	95	Yes autom.	
	Dicamba, foll. alk. hydr., OPT.	Yes	0.431	0.02	2	Yes	Yes	MeOH	No	pH 7	Other	STD - Add		MS/MS	LC-MS/MS	Validation data	70	Yes autom.	
	Dicamba, free acid	Yes	0.431	0.02	2	Yes	No	MeOH	No	No	Other	STD - Add		MS/MS	LC-MS/MS	Validation data	70	Yes autom.	
	Ethephon	Yes	0.55	0.02	1	No	No	Other	No	pH 3	None	STD - Add		MS/MS	LC-MS/MS	Validation data	90	Yes autom.	

Lab-Code	Pesticide	Accredited?	Reproted result [mg/kg]	Reporting level [mg/kg]	Sample weight [g]	Water addition	Hydrolysis	Extraction solvent [1]	ISTD used / ISTD details [2]	pH adjusted?	Clean up [3]	Calibration [4]	GC detector	HPLC detector	Confirmation	Recovery approach from	Recovery [%]	Recovery correction	Reference method
eupt 053	2,4-D, free acid, OPT.	Yes	0.673	0.02	3	Yes	No	EtOAc; CyHe	No	No	None	MM - ML		MS/MS	LC-MS/MS	Same batch	38	Yes autom.	ASE extraction, LC
	Chlormequat (free cation)	Yes	0.536	0.01	5	No	No	Other	No	No	None	MM - ML		MS/MS	LC-MS/MS	Same batch	111	Yes autom.	Water extraction. LC
eupt 054	2,4-D, foll. alk. hydr.	Yes	0.471	0.02	5	Yes	Yes	ACN	No	Yes	DSPE	MM - ML		MS/MS	None	Same batch	95.0	No	QuEChERS Anastassiades
	2,4-D, free acid, OPT.	Yes	0.496	0.02	5	Yes	No	ACN	No	Yes	DSPE	MM - ML		MS/MS	None	Same batch	91.9	No	QuEChERS Anastassiades
	Chlormequat (free cation)	Yes	0.402	0.02	10	Yes	No	MeOH	No	No	None	MM - ML		MS/MS	None	Same batch	98.1	No	EN 15055:2006
	Dicamba, foll. alk. hydr., OPT.	Yes	0.313	0.02	5	Yes	Yes	ACN	No	Yes	DSPE	MM - ML		MS/MS	None	Same batch	84.5	No	QuEChERS Anastassiades
	Dicamba, free acid	Yes	0.133	0.02	5	Yes	No	ACN	No	Yes	DSPE	MM - ML		MS/MS	None	Same batch	81.5	No	QuEChERS Anastassiades
	Ethephon	No	ND																
	Glyphosate	Yes	ND																
eupt 055	2,4-D, free acid, OPT.	Yes	0.513	0.02	5	Yes	No	ACN	No	pH 2	Other	MM - ML	ITD		GC/MS	Same batch	101	No	QuEChERS and PFBBr derivative
	Chlormequat (free cation)	Yes	0.314	0.05	20	Yes	No	MeOH	No	No	Other	MM - SL		MS		Same batch	93	No	EN15055
	Dicamba, free acid	Yes	0.096	0.02	5	Yes	No	ACN	No	pH 2	Other	MM - ML	ITD		GC/MS	Same batch	88	No	QuEChERS and PFBBr derivative
	Ethephon	Yes	ND																
	Glyphosate	Yes	ND																
eupt 058	2,4-D, foll. alk. hydr.	no	0.462	0.02	5	Yes	Yes	Aceton; CH_2Cl_2; Other	No		None	MM - ML		MS/MS	LC-MS/MS	Same batch	60	No	
eupt 060	2,4-D, foll. alk. hydr.	no	0.74	0.02	5	Yes	Yes	ACN	No	Yes	None	MM - ML		MS/MS	LC-MS/MS	Same batch	26	Yes	
	2,4-D, free acid, OPT.	no	0.583	0.02	5	Yes	Yes	ACN	No	Yes	None	MM - ML		MS/MS	LC-MS/MS	Same batch	33	Yes	
	Chlormequat (free cation)	Yes	0.283	0.02	25	No	No	MeOH	Yes, isotop. labelled-1	No	None	MM - ML	Other	MS	LC/MS	Same batch	90	No	
eupt 061	2,4-D, foll. alk. hydr.	No	0.494	0.01	10	No	Yes	CH_2Cl_2	Yes, other (Fenoprop)	Yes	liq. / liq.	PS - ML	MSD		GC/MS	Same batch	71	No	Internal method
	2,4-D, free acid, OPT.	No	0.436	0.01	10	No	No	CH_2Cl_2	Yes, other (Fenoprop)	Yes	liq. / liq.	PS - ML	MSD		GC/MS	Same batch	63	No	Internal method
	Chlormequat (free cation)	Yes	0.324	0.01	20	Yes	No	MeOH	No	No	None	PS - ML		MS	LC/MS	Same batch	79	No	EN 15054

Lab-Code	Pesticide	Accredited?	Reproted re-sult [mg/kg]	Reporting level [mg/kg]	Sample weight [g]	Water addition	Hydrolysis	Extraction solvent [1]	ISTD used / ISTD details [2]	pH adjusted?	Clean up [3]	Calibration [4]	GC detector	HPLC detector	Confirmation	Recovery approach from	Recovery [%]	Recovery correction	Reference me-thod
	Dicamba, foll. alk. hydr., OPT.	No	0.3	0.01	10	No	Yes	CH_2Cl_2	Yes, other (Fenoprop)	Yes	liq. / liq.	PS - ML	MSD		GC/MS	Same batch	96	No	Internal method
	Dicamba, free acid	No	0.091	0.01	10	No	No	CH_2Cl_2	Yes, other (Fenoprop)	Yes	liq. / liq.	PS - ML	MSD		GC/MS	Same batch	84	No	Internal method
eupt 063	2,4-D, foll. alk. hydr.	No	0.644	0.008	5	Yes	Yes	ACN	Yes, other (Nicarbazin)	Yes	Freez.	MM - ML		MS/MS	LC-MS/MS	Same batch	83	No	
	2,4-D, free acid, OPT.	Yes	0.458	0.008	5	Yes	No	ACN	Yes, other (Nicarbazin)	Yes	Freez.	MM - ML		MS/MS	LC-MS/MS	Same batch	83	No	
	Chlormequat (free cation)	Yes	0.592	0.02	5	Yes	No	MeOH	Yes, isotop. labelled D4-Chlormequat	Yes	Freez.	MM - ML		MS/MS	LC-MS/MS	Same batch	75	No	
	Dicamba, free acid	Yes	0.086	0.04	5	Yes	No	ACN	Yes, other (Nicarbazin)	Yes	Freez.	MM - ML		MS/MS	LC-MS/MS	Same batch	80	No	
	Ethephon	No	0.23	0.1	5	Yes	No	MeOH	No	Yes	Freez.	MM - ML		MS/MS	LC-MS/MS	Same batch	95	No	
	Glyphosate	No	0.575	0.1	5	Yes	No	MeOH	Yes, isotop. labelled 13C215N	Yes	Freez.	MM - ML		MS/MS	LC-MS/MS	Same batch	77	No	
eupt 082	2,4-D, foll. alk. hydr.	Yes	0.612	0.03	5	Yes	Yes	ACN	No	pH 5	Freez.	MM - ML	MSD		GC/ MS/MS	Same batch	94	No	
	Dicamba, foll. alk. hydr., OPT.	Yes	0.27	0.02	5	Yes	Yes	ACN	No	pH 5	Freez.	MM - ML	MSD		GC/ MS/MS	Same batch	78	No	
eupt 084	2,4-D, foll. alk. hydr.	No	0.447	0.02	5	Yes	Yes	ACN		No	Freez.	MM - ML		MS/MS	LC-MS/MS	Same batch	94	No	QuEChERS
	Chlormequat (free cation)	No	0.377	0.02	10	Yes	No	MeOH	Yes, isotop. labelled D4-Chlormequat	No	None	MM - ML		MS/MS	LC-MS/MS	Same batch	102	No	EN 15054
eupt 086	2,4-D, free acid, OPT.	Yes	0.575	0.02	5	Yes		ACN	No			MM - ML		MS/MS	None	Same batch	109.6	No	
	Chlormequat (free cation)	Yes	0.315	0.02	5	Yes		MeOH	No	Yes		MM - ML		MS/MS	None	Same batch	72.4	No	
	Dicamba, free acid	Yes	0.131	0.02	5	Yes		ACN	No			MM - ML		MS/MS	None	Same batch	79.8	No	
	Ethephon	Yes	ND																
	Glyphosate	Yes	ND	0.01	20			Water	No	No	Ion Change		MSD						
eupt 088	2,4-D, free acid, OPT.	Yes	0.292	0.080	5	Yes		ACN	Yes, other (TCP)	Yes	None	MM - SL		MS/MS		Same batch	96	No	QuEChERS
eupt	2,4-D, foll. alk. hydr.	Yes	0.648	0.020	3	Yes	Yes	ACN	No	Yes	None	MM - ML		MS/MS	LC-MS/MS	Same batch	70	No	

Lab-Code	Pesticide	Accredited?	Reproted re-sult [mg/kg]	Reporting level [mg/kg]	Sample weight [g]	Water addition	Hydrolysis	Extraction solvent [1]	ISTD used / ISTD details [2]	pH adjusted?	Clean up [3]	Calibration [4]	GC detector	HPLC detector	Confirmation	Recovery approach from	Recovery [%]	Recovery correction	Reference me-thod
091	Chlormequat (free cation)	Yes	0.443	0.008	5	Yes	No	MeOH	Yes, isotop. labelled-1	Yes	None	MM - ML		MS/MS	LC-MS/MS	Same batch	105	No	
eupt 095	2,4-D, foll. alk. hydr.	No	0.47	0.02	10		Yes	ACN	Yes, other (Ethion)	Yes	None	MM - ML		MS/MS	LC-MS/MS	Same batch	78	No	
	Chlormequat (free cation)	No	0.25	0.02	10		No	MeOH		Yes	None	MM - ML		MS/MS	LC-MS/MS		70	No	
eupt 117	Chlormequat (free cation)	No	0.59	1	10	Yes	No	MeOH	Yes, isotop. labeled D4-Chlormequat Chloride	No	None	PS - ML		MS/MS	LC-MS/MS	Isotope labelled std.	60	Yes autom.	Analysis of Chlor-mequat and Mepi-quat Residues in foods of plant origin (version 2; janvier 2009)
	Mepiquat (free cation)	No	< 0.005	2	10	Yes	No	MeOH	Yes, isotop. labeled D3-Mepiquat Iodode	No	None	PS - ML		MS/MS	LC-MS/MS	Isotope labelled std.	60	Yes autom.	Analysis of Chlor-mequat and Mepi-quat Residues in foods of plant origin (version 2; janvier 2009)
eupt 118	2,4-D, foll. alk. hydr.	No	0.025	0.02	15	No	Yes	EtOAc	No	pH 10	GPC	PS - ML		MS/MS	None	Same batch	72	No	Swedish
	Dicamba, foll. alk. hydr., OPT.		ND						Yes, other (Nicarbazim)										
eupt 130	2,4-D, foll. alk. hydr.	Yes	0.708	0.02	5	Yes	Yes	ACN	Yes, other (Nicarbazim)	No	DSPE	MM - ML		MS/MS	None			No	QuEChERS _LCMS_neg
	2,4-D, free acid, OPT.	Yes	0.48	0.02	5	Yes	No	ACN	No	No	DSPE	MM - ML		MS/MS	None			No	QuEChERS _LCMS_neg
eupt 140	2,4-D, foll. alk. hydr.	No	0.93	0.02	5	Yes	Yes	ACN	No	Yes	DSPE	PS - ML		MS/MS	None	Same batch	100	No	QuEChERS
	2,4-D, free acid, OPT.	No	0.526	0.02	5	Yes	No	ACN		No	DSPE	PS - ML		MS/MS	None	Same batch	95	No	QuEChERS

1). ACN: Acetonitile; MeOH: Methanol; EtOAc: Ethyl Acetate; Cy-He: Cyclohexane; CH_2Cl_2: Dichlormethane

2). isotop. labelled-1: isotopically labeled target pesticide; isotop. labelled-2: isotopically labeled other substance

3). SPE: Solid Phase Extraction; DSPE: Dispersive Solid Phase Extraction; liq. / liq.: liquid/liquid partitioning; GPC: Gel Permeation Chromatography; Freez.: Freezing out

4). MM – ML: Matrix matched – Multiple level; MM – SL: Matrix matched – Single level; PS – ML: Pure solvent – Multiple level; STD Add.: Standard addition approach

Appendix 14 List of Abbreviations

Abreviation	Describsion
AAZ	Average of Absolute z-Score
DAD	Diode Array Detector
DSPE	Dispersive Solid Phase Extraction
ECD	Electron Capture Detector
FFP RSD	Fit for Purpose Relative Standard Diviation
Fluor.	Flouresence Detector
FPD	Flame Photometric Detector
GC/MS	Gas Chromatograph / Mass spectrometer
GC/MS/MS	Gas Chromatograph / Mass spectrometer / Mass spectrometer
GPC	Gel Permeation Chromatography
ITD	Ion Trap Detector
LC	Liquid Chromatography
LC/MS	Liquid Chromatography / Mass spectrometer
LC/MS/MS	Liquid Chromatography / Mass spectrometer / Mass spectrometer
LOD	Limit of Determination
liq./liq part.	Liquid / Liquid Partitioning
MM-ML	Matrix Matched Multi Level Calibration
MM-SL	Matrix Matched Single Level Calibration
MRRL	Minimum Required Reporting Level
MS/MS	Mass spectrometer / Mass spectrometer
MSD	Mass Selective Detector
NPD	Nitrogen Phosphoros Detector
PS-ML	Pure solvent Multi Level Calibration
PS-SL	Pure Solvent Single Level Calibration
Qn	Robust Standard Diviation
SPE	Solid Phase Extraction
SW	Sample Weight
SWZ	Sum of Weighted Z-score

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, Health & Consumer Protection Directorate-General (DG-SANCO) by the four Community Reference Laboratories (CRLs) for pesticide residues in food and feed. These EUPTs are directed at all National Reference Laboratories (NRLs) and Official Laboratories (OfLs) in the EU Member States. Laboratories outside this CRL/NRL/OfL-Network[1] may be permitted to participate on a case-by-case basis after consultation with DG SANCO.

The following four CRLs for pesticides were appointed by DG-SANCO based on regulation 882/2004/EC[2]:

- CRL for Fruits and Vegetables (CRL-FV),
- CRL for Cereals and Feedingstuff (CRL-CF),
- CRL for Food of Animal Origin and Commodities with high Fat Content (CRL-AO) and
- CRL for Single Residue Methods (CRL-SRM)

NRLs are appointed by the National Food or Feed Authorities based on the provisions of Regulation 882/2004/EC, whereas OfLs are laboratories that are actively involved in providing residue data for the national control programme and/or the co-ordinated multiannual Community control programme.

According to Regulation 396/2005/EC[3] all laboratories analysing samples for the official controls on pesticide residues shall participate in the Community proficiency test(s)

[1] For more information about the CRL/NRL/OfL-Network please refer to the CRL-Web-portal under: http://www.crl-pesticides.eu

[2] 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

organised by the Commission. 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 Commission within the framework of the national control programmes and the co-ordinated multiannual community control programme. 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-organisation

EUPTs are organised by individual CRLs or by more than one CRL in cooperation with one another.

For each EUPT an Organising Team is appointed by the CRL(s) that is responsible for the EUPT. This team is then responsible for all administrative and technical matters concerning the organisation of the PT, e.g. PT-announcement, production of the test material, undertaking the homogeneity and stability tests, packing and shipment of test material, and the handling and first assessment of participant's results.

A common Scientific Committee entailing the following two subgroups:

a) An Advisory Group (AG) and

b) An independent Quality Control Group (QCG)

consisting of expert scientists with long experience in pesticide residue analysis that have been appointed by the CRLs and approved by the DG-SANCO.

The role of the AG is to help the organisers in making decisions concerning the design of the EUPT: 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 anf final report. The QCG has the additional function of supervising the quality of the EUPT and to assisting the CRLs with confidential aspects such as the choice of the pesticides, and levels to be present in the test material.

The EUPT-Organising Team, AG and QCG together form the **EUPT-Panel**.

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[3] 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.

Confidentiality:

In each EUPT the laboratories are given a unique code only known to themselves, the Organisers, and DG-SANCO. 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 the Commission may present the results to the Standing Committee on the Food Chain and Animal Health on a country-to-country basis. It is therefore possible that a link between codes and National Reference Laboratories could be made, especially for those Member States where only one laboratory has participated. The owner of all EUPT data is DG SANCO.

Communication

The official language used in all EUPTs is English.

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

Announcement

The announcement of the individual EUPT will be issued at least 3 months before the test material is distributed to the laboratories. The announcement will be published on the CRL portal and distributed via mail to the NRL/OfL mailing list available to the CRLs. The announcement will contain an invitation letter, details on how to register and where to locate additional related documents, and 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.

Specific Protocol

For each PT a Specific Protocol will be published at least 2 weeks before the test material is distributed to the laboratories. This protocol will contain all information the included in the invitation in its final version, information on payment for delivery service and/or participation. Furthermore, it will also include instructions on how to handle the test material upon receipt, on how to submit results, and other relevant information.

General procedures for reporting results

Laboratories are responsible for reporting their results to the Organiser within the stipulated deadlines. Each laboratory must only report one result for each of the pesticides present in the test material, using the analytical procedure(s) that they would routinely use for each compound for monitoring purposes. More than one method may be used to cover all the compounds to be sought. The results (residue levels of the pesticides detected) must be, expressed in mg/kg.

Correction of results for recovery

According to the Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed, (Document SANCO in force each year) residues data should not normally be adjusted for recovery, when the mean recovery is within the range of 70-120%. If residues data are adjusted for recovery, then this must be clearly stated. Therefore laboratories are required to report whether their results were adjusted for recovery and if this was the case, the recovery factor used. No recovery factors are required where recovery adjustments resulted from using the 'standard addition(s)' approach, or from the use of isotopically labelled internal standards (with spiking of the test material at the beginning of the extraction procedures). In this case, the laboratories should report the technique used for calculation of the results instead of the recovery factor.

Evaluation of the Results

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

- ***False Positives***

These are the results that show the apparent presence of pesticides that were listed in the Target Pesticide List, but which were (i) not used in the sample treatment, (ii) and not detected by the organiser, even after a repeat analysis. However, if a number of participants do detect the same additional pesticide, or if the concentration is above the MRRL, then a decision as to whether, or not, this should be considered to be a false

positive result will be made on a case-by-case basis. 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 that no numerical values were given, although they were used by the Organiser to treat the test material and were detected by the majority of participants at or above the MRRL.

- ***Estimation of the true concentration (μ)***

The "true" concentration will be typically estimated using the median of all the results. Therefore a **median value** for every compound present will be calculated and used as the assigned value. In special justifiable cases, the EUPT Panel may decide to use only part of the population of results to establish the median (e.g. using only results with z-scores ≤ 5.0).

- ***Establishing the standard deviation of the assigned value (target standard deviation)***

The target standard deviation (δ) of the median will be calculated using a Fit-For-Purpose Relative Standard Deviation (FFP-RSD) approach, as follows:

$$\delta = b_i * \mu_i \qquad \text{with } b_i \text{ i = FFP-RSD (= 0.25)}$$

The percentage FFP-RSD is typically 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.

- ***z-scores***

This parameter is calculated using the following formula:

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

Where x_i is the value reported by the laboratory, μ_i the assigned value, and δ_i the standard deviation at that level for each pesticide (i).

Any z-scores of > 5 will be reported as "+5" particularly where summed z-scores of many pesticides are calculated (see SWZ below).

z-scores will be interpreted in the following way:

$|z| \leq 2$ Acceptable

$2 < |z| \leq 3$ Questionable

$|z| > 3$ Unacceptable

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

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

However, a z-score will not be calculated for any false positive result.

- ***Category A and B classification***

The EUPT-Panel will decide whether to classify the laboratories in two groups, A and B. Laboratories that detected a sufficiently high percentage of the pesticides present in the test material (e.g. at least 90%), reported no false positives, and sought all the pesticides on the Target Pesticide List marked with an asterisk that were present in the test material, will have demonstrated 'sufficient scope' and will therefore be classified in Category A.

- ***Combined z-scores***

For evaluation of the overall performance of the laboratories within Category A, a ranking according to the sum of weighted z-scores (SWZ) will be calculated.

The sum of weighted z-scores formula uses the z-scores with a fixed maximum value of 5 for individual z-scores, using the following formula:

$$\text{'Sum of weighted z-scores'}\ (Z) = \frac{\sum_{i=0}^{i\leq 2}|z|\cdot 1 + \sum_{i>2}^{i\leq 3}|z|\cdot 3 + \sum_{i>3}^{\infty}|z|\cdot 5}{n}$$

n = number of reported results

So for each laboratory:

- The first summation is the sum of all their /z-scores/ between zero to two, multiplied by 1.
- The second summation is the sum of all their /z-scores/ greater than two but less than or equal to, three, multiplied by 3.
- The third summation is the sum of all their z-scores greater than three, multiplied by 5.

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

$Z \leq 2$ Good

$2 < Z \leq 3$ Satisfactory

$Z > 3$ Unsatisfactory

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

Publication of results

The preliminary results from the EUPTs will be published within 2 months from the deadline for result submission.

The final report will be published shortly after the organiser and the EUPT-Panel have discussed the results. Taking into account that the EUPT-Panel normally only meets once a year, the final report may be published up to 8 months after the deadline for results submission.

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.

and Feeding Stuff & Single Residue Met

Specific protocol
for EU Proficiency Test for Pesticide Residues in Cereals, EUPT-C3/SRM4 (2008)

Introduction

This protocol is complementary to the General protocol for EU proficiency test for pesticide residues in food and feed. The proficiency test covers both pesticides measured by Multi Residue Methods and Single Residue Methods. The Proficiency Test is organised by the CRL for Pesticide Residues in Cereals and Feedingstuff in collaboration with the CRL for Single Residue Methods.

Test material

This proficiency test involves pesticide residues analysis in oats.

The oats were grown in Denmark in 2008 and pesticides were applied in the field. Afterwards, the oats were also spiked with some additional pesticides

Analytical parameters

The test material contains several pesticides from the Target Pesticide List in Annex1. Laboratories should carefully read the Target Pesticide List, where important information about the reporting of the results, as well as the Minimum Required Reporting Levels (MRRLs) is given. Where the residue definition includes more than one component, the results for the individual components, as well as the respective sum of components, calculated as stated in the residue definition, are to be reported.

Community Reference Laboratory for Cereals and Feedingstuff

Technical University of Denmark, National Food Institute, Mørkhøj Bygade 19, Building C, DK-2860 Søborg

Community Reference Laboratory for Single Residue Methods

CVUA Stuttgart, Schaflandstr. 3/2, DE-70736 Fellbach

For each pesticide and the relevant compounds included in the residue definitions, MRRL values have been set. The MRRL values will be used help to identify false negative results and for the calculation of z-scores for false negatives.

Amount of Sample

Participants will receive:

- approximately 150 g of oat test material with incurred and spiked pesticides and
- approximately 150 g of blank oat test material.

All samples will be frozen and packed in thermo boxes together with a freezer block.

Shipment of Samples

The shipment of the test materials will take place on 2 February 2009. The organisers will try to ensure that all packages arrive at the same time. An information message will be sent out by e-mail before shipment.

Instructions on Test Material Handling

Once received, the test material should be stored deep frozen (-18°C or less) before analysis to avoid any deterioration/spoilage of the test material. The test material should be mixed thoroughly (after defrosting), before taking the analytical portion(s).

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

Sample Receipt and Acceptance

Once the laboratory has received the test materials it must report to the organiser via the result submission website (subpage 0), by filling in the date of receipt, the condition of the test material, and its acceptance. The deadline for acceptance, or not, is the 6 February 2009. If the laboratory does not respond before this deadline the organiser will assume that it has received and accepted the test material. If any participants have not received the test material by 6 February 2009, they should inform the organiser immediately by e-mail (crlcereal@food.dtu.dk).

Reporting Results

To report their results, laboratories must access the **Result Submission Website** (sub-pages 1 and 2) a link to which you will find in the CIRCA platform (visit http://www.crl-pesticides.eu/docs/public/tmplt_article.asp?CntID=657&LabID=400&Lang=EN for the CIRCA link). Here you will also find the **Result Submission Guide** with specific instructions on how to enter the data.

Before entering the results please read carefully the Target Pesticide List in Annex 1, since the residue definitions are not given on the Result Submission Website. For pesticides where the residue definition is a sum of a parent pesticide and other components, results for both the sum and the individual components must be reported.

It should not be assumed that only pesticides registered for use on oats are present.

All results must be reported on the online result submission website by 2 March 2009, at the latest. The website will not be accessible after this date, and any results reported after the deadline will not be included in the statistical treatment, or in the final report.

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

Residue levels <0.010 mg/kg;

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

Residue levels ≥ 0.010 mg/kg;

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

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.

Reporting Information on Analytical Methodology

All laboratories are requested to provide information on the analytical method(s) they have used via the Result Submission Website (subpage 3). If no information is given, the organiser has the right not to accept the analytical results reported.

Time Table

Activity	*Who?*	*Dates*
Test Material treatment, milling, homogenisation, storage, homogeneity tests.	Organizers	September-December 2008 January 2008
Stability tests	Organizers	January - March 2009
Invitation to laboratories to participate and instructions on how to register. Opening of the EUPT-C4/SRM3 website with further information including links to “Calendar”, “Target Pesticide List”	Organizers	31 October 2008
Registration of laboratory representatives at CIRCA-platform	Labs	October-December 2008
Provide access to the “Registration Website”	Organizers	November 2008
Deadline for registration	Labs	**14 January 2009**
Provide access to the “PT-Protocols”	Organizers	15 January 2009
Test material distribution and Information to the laboratories about upcoming shipment	Organizers	2 February 2009
Deadline for receipt of results on the “Result Submission Website”	Labs	**2 March 2009**
Preliminary Report	Organizers	April 2009
Final Report	Organizers	November 2009

Participation fee

NRL for 1) Cereals and Feedingstuff and 2) Single Residue Method: Free of charge

Official laboratories: 100 €

Cost for shipment of test material

All laboratories: 110 €

For further information visit the website www.crl-pesticides.eu

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Quality Assurance Group

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Annex 1

Target Pesticide List for the EUPT-C3/SRM4 - 2008

Note: pesticides marked with an asterisk "*" are mandatory (see Category A and B classification rules on page 6 in the General Protocol (https://fis-vl.bund.de/Members/irc/fis-vl/crl-pesticides/library?l=/crl-pesticides-general&vm=detailed&sb=Title).

Pesticides	MRRL
MRM-compounds	
*Azoxystrobin	0.02
*Bifenthrin	0.02
Carbaryl	0.02
*Carbendazim and benomyl (carbendazim + benomyl, expressed as carbendazim)	0.01
Chlorothanlonil	0.01
*Chlorpyrifos	0.02
*Chlorpyrifos-methyl	0.02
*Cypermethrin (sum of isomers)	0.02
-Alpha-cypermethrin	0.02
*Cyproconazole	0.02
Cyprodinil	0.02
*Deltamethrin (cis-deltamethrin)	0.02
Diazinon	0.02
*Difenoconazole	0.02
*Endosulfan (sum of alpha- and beta-isomers and endosulfan-sulphate expressed as endosulfan)	0.02
-Endosulfan α	0.02
-Endosulfan β	0.02
-Endosulfan sulfate	0.02
*Epoxiconazole	0.02
Fenvalerate and Esfenvalerate (Sum of RR/SS and RS/SR isomers)	0.02

Pesticides	MRRL
-Fenvalerate and Esfenvalerate (Sum of RR & SS isomers)	0.02
-Fenvalerate and Esfenvalerate (Sum of RS & SR isomers)	0.02
Fenbuconazole	0.02
Fenhexamid	0.02
Fenitrothion	0.02
*Fenpropimorph	0.02
*Fludioxonil	0.02
Fluquinconazole	0.02
Flusilazole	0.02
*Imazalil	0.02
*Iprodione	0.02
*Kresoxim-methyl	0.02
*Lambda-cyhalothrin	0.02
*Lindane (gamma- isomer of hexachlorocyclohexane (HCH))	0.01
*Malathion (Malathion + Malaoxon, expressed as Malathion)	0.02
-Malathion	0.02
-Malaoxon	0.02
Metconazole	0.02
*Methacrifos	0.02
*Methomyl and Thiodicarb (Methomyl + Thiodicarb, expressed as Methomyl)	0.02
-Methomyl	0.02
-Thiodicarb	0.02
Metribuzin	0.02
*Parathion	0.02
*Penconazole	0.02
Pendimethalin	0.02
*Permethrin	0.02
*Pirimicarb (sum of pirimicarb and desmethyl pirimicarb, expressed as pirimicarb)	0.02

Pesticides	MRRL
-Pirimicarb	0.01
-Desmethyl pirimicarb	0.01
*Pirimiphos-methyl	0.02
*Prochloraz (parent compound only)	0.02
*Procymidone	0.02
*Propiconazole	0.02
Pyraclostrobin	0.02
Spiroxamine	0.02
*Tebuconazole	0.02
*Thiabendazole	0.02
*Thiophanate-methyl	0.01
Triadimefon and triadimenol (sum of triadimefon and triadimenol)	0.02
-Triadimefon	0.02
-Triadimenol	0.02
Triazophos	0.02
*Trifloxystrobin	0.02
Vinclozolin (only parent compound)	0.02
SRM-compounds	
* 2.4-D (following alkaline hydrolysis)	0.02
2.4-D (free acid), OPTIONAL	*0.02*
Bentazone (only parent)	0.02
Dicamba (free acid)	0.02
Dicamba (following alkaline hydrolysis) OPTIONAL	*0.02*
Dichlorprop (2,4-DP) including Dichlorprop-P (free acid)	0.02
Dichlorprop (2,4-DP) including Dichlorprop-P (following alkaline hydrolysis) OPTIONAL	*0.02*
Fluazifop including Fluazifop-P (following alkaline hydrolysis)	0.02
Fluazifop including Fluazifop-P (free acid) OPTIONAL	*0.02*

Pesticides	MRRL
Fluroxypyr (following alkaline hydrolysis)	0.02
Fluroxypyr (free acid) OPTIONAL	*0.02*
Haloxyfop including Haloxyfop-R (following alkaline hydrolysis)	0.02
Haloxyfop including Haloxyfop-R (free acid) OPTIONAL	*0.02*
* MCPA (following alkaline hydrolysis)	0.02
MCPA (free acid) OPTIONAL	*0.02*
Mecoprop (MCPP) including Mecoprop-P (free acid)	0.02
Mecoprop (MCPP) including Mecoprop-P (following alkaline hydrolysis) OPTIONAL	*0.02*
Ethephon	0.02
Glyphosate	0.02
*Chlormequat (free cation)	0.02
*Mepiquat (free cation)	0.02

Explanatory note concerning the PT-specific residue definitions for acidic pesticides given above: Where the legal residue definition includes only the free acid the result following alkaline hydrolysis is OPTIONAL. And vice versa, where the legal residue definition includes the esters and/or conjugates the result for the free acid is OPTIONAL. OPTIONAL means that the results will be out competition and mainly used for scientific evaluation for example to support the ongoing discussions concerning the legal residue definitions of acids.

Commission Reference Laboratory on Cereals & Feedingstuff and Single Residue Methods

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