Commission Reference Laboratories on Cereals
& Feedingstuff and Single Residues Methods

Report on Proficiency Test on incurred and spiked pesticides in wheat

EUPT-C1-SRM2 2007

Final report
December 2007

Technical University of Denmark

EU PROFICIENCY TESTS
C1/SRM2, 2007

Pesticide Residues in Cereals using Multi and Single Residue Methods

Final Report

Mette Erecius Poulsen
Michelangelo Anastassiades
Drazen Kostelac
Hanne Bjerre Christensen
Susan Herrmann

December 2007

Organisers:

Mette Erecius Poulsen
Head of CRL Cereals and Feedingstuff
National Food Institute
Department of Food Chemistry
Danish Technical University
Moerkhoej Bygade 19
DK-2860 Soeborg
Phone: +45 7234 7463
Fax: +45 7234 7448
E-Mail: mep@food.dtu.dk

Dr. Michelangelo Anastassiades
Head of CRL Single Residue Methods
CVUA Stuttgart
Schaflandstrasse 3/2
D-70736 Fellbach
Phone: 49-711-3426-1124
Fax: 49-711-588176
E-Mail: Michelangelo.Anastassiades@cvuas.bwl.de

http://www.crl-pesticides.eu

Organising team in collaboration with the Organisers:

Dr. Hanne Bjerre Christensen, Chemist	CRL for Cereals and Feedingstuff
Ms. Susan Herrmann, Chemist	CRL for Cereals and Feedingstuff
Ms. Merete B. Ludwigsen, Chemical Technician	CRL for Cereals and Feedingstuff
Inge Schröder, Chemical Technician	CRL for Cereals and Feedingstuff
Lisbet Pilhkjær, Chemical Technician	CRL for Cereals and Feedingstuff
Marianne Graf, Secretary	CRL for Cereals and Feedingstuff
Arne Bent Jensen, System Developer	CRL for Cereals and Feedingstuff
Dr. Drazen Kostelac, Chemist	CRL for Single Residue Methods
Dr. Hubert Zipper, Chemist	CRL for Single Residue Methods
Ms. Dorothea Mack, Chemical Technician	CRL for Single Residue Methods
Mr. Bünyamin Tasdelen, Chemical Technician	CRL for Single Residue Methods
Ms. Marianne Kaebel, Chemical Technician	CRL for Single Residue Methods

Scientific Group:

Dr. Antonio Valverde, Professor	University of Almería, Spain.
Mr. Arne Andersson, Head of Division	National Food Administration, Uppsala, Sweden.
Dr. Amadeo R. Fernández-Alba, Professor	University of Almeria, Spain
Dr. Miguel Gamón, senior Chemist	Pesticide Residue Laboratory of the Generalitat Valenciana, Valencia, Spain.
Dr. André de Kok, senior Chemist	Food and Consumer Product Safety Authority (VWA), Amsterdam, The Netherlands.
Dr. Tuija Pihlström, Chemist	National Food Administration, Uppsala, Sweden.
Mr. Stewart Reynolds, senior Chemist	Central Science Laboratory, York, United Kingdom.
Dr. Ralf Lippold, senior Chemist	Chemisches und Veterinäruntersuchungsamt (CVUA) Freiburg

FOREWORD

The Council Directives 86/362/EEC [1] and 90/642/EEC [2] provide for the organisation and financial support for regular proficiency testing (PT) of those laboratories that perform analyses for their official national monitoring programmes. These proficiency tests are performed in order to ensure the quality, accuracy and comparability of the residue data sent by EU Member States to the European Commission, as well as to the other Member States. With the recent establishment of Community Reference Laboratories (CRLs) for food, feed and animal health, EU proficiency testing has been given a new broader framework. According to Regulation (EC) No 882/2004 [3], which specifies the general responsibilities of the CRLs, the organisation of comparative tests is on of the CRL's main tasks.

The EU PTs organized to date have been focusing on fruit and vegetable commodities and the use of multi residue methods (with a few exceptions). In 2006 the first proficiency test on single or group-specific residue methods (EUPT-SRM-01) was organised. The present proficiency test EUPT- C1/SRM02 was the first test on cereals and required the use both multi and single residue methods. The test was organised between the CRL for pesticides in cereals and feedingstuff (CRL-CF) and the CRL for pesticide residue analysis using Single Residue Methods (CRL-SRM) located in Copenhagen and Stuttgart, respectively. Participation was open to all official analytical laboratories involved in the determination of pesticide residues in cereals or feedingstuff within the EU.

CONTENTS

EUROPEAN COMMISSION CRL - PROFICIENCY TEST ON PESTICIDE RESIDUES USING MULTI AND SINGLE RESIDUE METHODS IN CEREALS, EUPT-C1/SRM2, 2007

INTRODUCTION

On 16 January 2007, 157official laboratories as well as the contact points of the EU Member States were sent an invitation to participate in this 1st European Commission's Proficiency Test on cereals using multi residue and single residue methods. A list of 40 possible pesticides (ANNEX I), which potentially were present in the test material along with their corresponding minimum required performance levels (MPRLs), was also included in the invitation. Following this call, 63 laboratories from 25 countries agreed to participate in this PT.

This proficiency test was performed using wheat flour of Danish origin, which had been partly treated in the field and partly spiked post-harvest with pesticides. The Faculty of Agricultural Sciences, University of Aarhus performed the field spraying, based on application rates and application times decided by the CRL-CF. Participating laboratories were provided with 250 g portions of treated whole wheat flour test material and 200 g of blank whole wheat flour. Twelve laboratories asked for extra test material. The test materials were shipped to participants on 14 May 2007 and the deadline for submission of results to the Organiser was 17 June 2007. The participants were asked to analyse the treated test material as well as the ‘blank‘ material and report the concentrations of any pesticide residues they found which were included in the list (ANNEX I). 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 the results were performed online via a website developed by the CRL-CF.

The median values of the analytical data submitted were used to obtain the assigned (true) values for each of the pesticide residues present. A fit-for-purpose target relative standard deviation (FFP RSD) of 25%, based on the experience of the Advisory Group, was chosen to calculate the target standard deviations (σ) as well as the z-scores of the compounds present. For informative purposes, the concentration dependent Horwitz equation standard deviation as well as the robust (Qn) standard deviation were additionally used to calculate the target standard deviations.

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 sample material:

- For GC amenable MRM pesticides: Ethyl acetate extraction with GPC clean-up and determination on GC/MS, GC/MS/MS, GC-ECD and GC- NPD [4]
- For LC amenable MRM pesticides: Methanol extraction follow by filtration and determination on LC/MS/MS [5]
- For acidic pesticides (as free acids): QuEChERS-method [6, 7], involving extraction with acetonitrile, partitioning after addition of salts, and direct determination by LC-MS/MS.
- For acidic pesticides (including esters and conjugates): QuEChERS-method [NEW REF www.crl-pesticides.eu], involving an initial alkaline hydrolysis and neutralization and followed by the normal procedure (this method was placed online well before the PT exercise).
- For chlormequat: In-house-method based on [8], involving addition of an isotopically labeled internal standard, extraction with methanol, centrifugation, filtration and direct determinative analysis by LC-MS/MS.

1.2 Preparation of the treated test material

Before preparing the test material, the pesticides and suitable residue levels for the study were selected. The application rate and application time for nine pesticides from the possible pesticide list was assessed based on data from supervised residue trials. The Faculty of Agricultural Sciences, University of Aarhus, performed the field spraying. One hundred kilos of wheat were delivered for preparation of the test material. Following a preliminary analysis of the material it was decided to additionally spike it in the laboratory with three pesticides where the incurred residues were too low (carbendazim, deltamethrin, pirimiphos-methyl) as well as with two additional pesticides (mecoprop and propiconazole). One pesticide with low residue level (pirimicarb) was not spike in the lab in order not to raise the number of pesticides present in the material too much (see **Table 1**). Pesticide formulations were used for spiking. One kilo of wheat was spiked with one of the pesticide formulations and a second kilo of wheat was spiked with another pesticide formulation, etc. The resulting 5 kilos were mixed with the remaining 55 kg and homogenised thoroughly. The 60 kg mixed wheat where milled, 4 kilo portions at a time. The portions were stirred thoroughly individually, and additionally two by two to ensure a well-homogenised bulk with respect to both incurred and spiked pesticide residues. Portions of 250 g were weighed out into screw-capped polyethylene plastic bottles, sealed, numbered, and stored in a freezer at about - 20 °C prior to homogeneity test and distribution to the participants.

1.3 Preparation of the 'blank' test material

The wheat 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. Portions of 200 g 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
Azoxystrobin	x	
Carbendazim	x	x
Chlormequat	x	
Deltamethrin	x	x
Diazinon	x	
Endosulfan	x	
MCPA	x	
Mecoprop		x
Pirimicarb	x	
Pirimiphos-methyl	x	x
Propiconazole		x

1.4 Homogeneity test

Ten bottles 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 Protocol published by IUPAC, ISO and AOAC [9]. The individual residue data from the homogeneity tests are given in Appendix 1. The results of the statistical analyses are given in **Table 2** and **Table 3**. The acceptance criteria for the test material to be sufficiently homogenous for the proficiency test was that $S_s/\sigma < 0.3$, with S_s being the between sampling standard deviation and σ = RSD (25%) x the mean concentration of each pesticide.

All the pesticides passed the homogeneity test and the test material was considered to be homogeneous and suitable for use in the PT-C1/SMR2.

Table 2. Statistical evaluation of the MRM homogeneity test data (n = 20 analyses)

	Azoxystrobin	Carbendazim	Deltamethrin	Diazinon	Pirimiphos-methyl	Propiconazole
Mean (mg/kg)	0.036	0.098	0.403	0.071	6.44	0.236
S_s/σ	0.11	0.22	0.29	0.056	0.084	0.23
Pass/Fail	Pass	Pass	Pass	Pass	Pass	Pass

S_s: Between Sampling Standard Deviation

Table 3. Statistical evaluation of the SRM homogeneity test data (n = 20 analyses)

	Chlormequat	MCPA	MCPA (optional)	Mecoprop	Mecoprop (optional)
Mean (mg/kg)	1.232	0.045	0.259	0.410	0.530
S_s/σ	0.03	0.20	0.18	0.12	0.11
Pass/Fail	Pass	Pass	Pass	Pass	Pass

S_s: Between Sampling Standard Deviation

1.5 Stability tests

The analytical methods described briefly above (in section 1.1) were also used for the stability tests.

The tests were performed on two occasions, one before the start of the PT-exercise and one after the completion date. The test material was kept frozen prior to the initial analysis. Three different storage conditions were compared room temperature, 4°C and -18°C. In all cases the analyses were performed on 5 randomly chosen samples employing duplicate measurement.

The two occasions were:

- Day 1: shortly after the first sample shipment,
  on 22 May 2007 for MRM, on 29 May 2007 for SRM.
- Day 2: after a period of approximately one or two months,
  on 27 June 2007 for MRM, on 29 July 2007 for SRM.

The individual results are given in Table 4 and Table 5.

All the MRM pesticides were stable at -18 ºC, which was the recommended temperature for storage of the test material. Also storage at 4 ºC seems adequate for all of the pesticides. However, after 1 month storage at room temperature azoxystrobin, carbendazim, diazinon and some - pirimiphos-methyl showed some decline up to 24%. For azoxystrobin, the stability test were in contrast to the homogeneity test performed by adding water prior to the extraction, which explains the differences between the mean values.

Table 4. Statistical test to demonstrate stability at different storage temperatures for MRM-compounds

Azoxystrobin (mg/kg)	Carbendazim	Deltamethrin	Diazinon	Pirimiphos-	Propiconazole	
Day 1 (mean)	0.253	0.096	0.341	0.077	6.08	0.237
Room temperature						
Day 2 mean)	0.222	0.0856	0.361	0.068	4.60	0.235
%	-13	-10	6	-12	-24	-1
4°C						
Day 2 (mean)	0.257	0.096	0.352	0.08	5.70	0.233
%	1	0	3	3	-6	- 1
-18°C						
Day 2 (mean)	0.265	0.098	0.351	0.078	6.21	0.227
%	5	2	3	2	2	-4

At the -18°C storage temperature the tests did not show any significant decrease in the pesticide levels, which indicated that at these storage conditions the pesticides present in the test material remained stable for the entire duration of the Proficiency Test. At 4°C storage the level of MCPA declined by about 15 % a value slightly greater than one would expect at quintuplicate analysis. Therefore this storage condition is recommended for the test material. Anyhow, the laboratories were asked to store the samples in the freezer until analysis is performed. The stability test at room temperature was not performed due to the growing of fungus in the material.

For Chlormequat, the homogeneity test was performed with a different standard solution than the stability test and this may have introduced the 20% difference in the mean value (1.232 mg/kg vs, 0.910 mg/kg).

Table 5. Statistical test to demonstrate stability at different storage temperatures for SRM-Compounds

	Chlormequat (mg/kg)	**MCPA** (mg/kg)	**MCPA (optional)** (mg/kg)	**Mecoprop** (mg/kg)	**Mecoprop (optional)** (mg/kg)
Day 1 (mean)	0.910	0.045	0.259	0.410	0.530
4°C					
Day 2 (mean)	0.989	0.038	0.251	0.391	0.583
%	8.7	-15.5	-3.1	-4.6	10.0
-18°C					
Day 2 (mean)	0.954	0.042	0.272	0.376	0.520
%	4.8	-6.7	5.0	-8.3	-1.9

1.6 Organizational details

1.6.1 Access of documents and confidentiality

After the participants registered for the PT, they received a laboratory code, a password and the link for the online result submission website. Additionally, the participants received username and password to access all the relevant documents that were placed at the CRL web-page. This ensured that confidentiality was maintained throughout the entire duration of the PT.

1.6.2 Submission of results

An online submission tool was developed that allowed participants to submit their results via the Internet. All participants had access to the webpage, http://thor.dfvf.dk/ptc, from the day of sample shipment until the deadline for the submission of results. Participants were asked to give information, not only of their results, but also regarding accreditation, reporting limits and details about the analytical methods used for analysing the test material.

1.6.3 Distribution of the test material

One bottle of treated test material (250g) and one bottle of 'blank' material (200g) were shipped on the 14 May 2007 to each participant in boxes containing a freezing unit.

2. STATISTICAL METHODS

2.1 False positives and negatives

2.1.1 False positives

In principle, results indicating the presence of pesticides that were included in the pesticide list, and which were (i) not used in the preparation of the test material, (ii) and not detected by the organiser, even following a repeat analysis, were treated as false positives, if they were reported at concentrations at or above the Minimum Required Performance Level (MRPL) stipulated by the Organisers. Results reported that were lower than 0.01mg/kg were ignored by the Organisers and not considered as false positives. No z-score value was calculated for these results.

2.1.2 False negatives

Pesticides that were analyzed but not reported by the laboratories, although they were used by the Organiser to treat the test material and were subsequently detected at, or above, the MRPL by the Organiser (and the majority of participating laboratories) were considered to be false negatives. z-Scores were not only calculated for all pesticides detected at levels exceeding the MRPL but also for the false negatives, in the latter case using the MRPL for calculation (exception MCPA, see 4.1.2).

2.2 Estimation of the assigned values

To establish the assigned values, the median levels of all the reported results, excluding the outliers, were used. For azoxystrobin also results from participants not adding water to the samples prior to the extraction were excluded (see 3.1.3).

2.3 Fixed target standard deviation

Based on previous experience from EU proficiency test on fruit and vegetables a fixed fit-for-purpose relative standard deviation (FFP RSD) of 25 % was used. The target standard deviation (σ) for each individual pesticide was calculated by multiplying this FFP RSD by the assigned value. In addition, the concentration dependent Horwitz standard deviation was also calculated for informative purposes. In addition, the concentration dependent Horwitz standard deviation σ_H as well as the robust Qn standard deviations were also calculated for informative purposes.

2.4 z-Scores

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

$$z = (x-X) / \sigma \qquad \text{Eq.1}$$

or

$$z = (x-X) / \sigma_H \qquad \text{Eq.2}$$

Where:

- x is the result reported by the participant or the specific reporting limit of the lab for those labs not having detected the pesticide present in the test material
- X is the assigned value or true concentration
- σ is the target standard deviation obtained by multiplying the median by the FFP RSD of 25%
- σ_H is the target standard deviation calculated using the Horwitz equation

The z-Score classification was as follows:

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

- Any z-score values of $|z| > 5$ have been reported as '+5', or '-5'.
- In case of false negative pesticide residues, the reporting limit (RL) of the corresponding lab was taken into account. For MRM pesticide residues, z-scores were calculated using the MRPL values as the value for x.
- No calculation of z-score was performed for any false positive result.

2.5 Weighted Sum of z-Scores

In order to evaluate each laboratory's overall performance, and taking into account all the MRM pesticides analysed the Weighted Sum of z-scores (WSZ) has been calculated. This function was only applied to laboratories with sufficient scope, i.e. those labs that have reported 5 or more of the total number of MRM pesticides present in the sample and no false positives. The weighting factor ω is defined as follows:

$$\omega|Z_i| = \begin{cases} 1 & \text{if} \quad |Z| \le 2 \\ 3 & \text{if} \quad 2 < |Z| \le 3 \\ 5 & \text{if} \quad |Z| > 3 \end{cases}$$

Therefore, the 'Weighted Sum of z-Scores' |z| formula is:

$$\text{'Weighted sum of z-scores '} |z| = \frac{\sum_{i=1}^{n} |Z_i| \omega(Z_i)}{n}$$

So for each lab:

- The first term is the sum of absolute values of z-scores between zero and two, multiplied by one.
- The second term is the sum of absolute values of z-scores greater than two, but less than or equal to three, multiplied by three.
- The third factor is the sum of absolute values of z-scores greater than three, multiplied by five.

The sum is then divided by the number of reported results (n) from each lab.

3. RESULTS - MRM PESTICIDES

Sixty-three laboratories representing 25 countries agreed to participate in this proficiency test, and only one laboratory did not submit results. Five Member States did not participate, Bulgaria, Cyprus, Malta, Rumania and Spain. Only 25 of the laboratories submitted results for all seven MRM pesticides in the test material. The participating laboratories are listed in ANNEX II .

All data reported by the participants is shown in the appendices. An overview of the results for MRM pesticides is reported in **Table 6**. All analytical results reported can be seen in **Table 9**; the methods used are shown in **Appendix 4**.

3.1 Results of MRM pesticides

An overview of the results can be seen in **Table 6**. Results for deltamethrin, diazinon, endosulfan and pirimiphos were reported by 94-97% of the participants. Azoxystrobin and propiconazole were reported by 84-87%, while carbendazim were only reported by 63% of the participants. After assessment of the results it transpired that the estimated assigned value for endosulfan was below the MRPL given to the participant prior to the test and no further assessments were performed for endosulfan.

Table 6. Overview of results for MRM pesticides

Pesticides	No. of reported Results	No. of reported NA [1)]	No. of reported ND [2)]	% of labs that reported results [3)]
Azoxystrobin	55	7	2	89
Carbendazim	39	23	3	63
Deltamethrin	58	4	1	94
Diazinon	59	3	1	95
Endosulfan[4)]	59	3	0	95
Pirimiphos-methyl	60	2	1	97
Propiconazole	52	10	0	84

1) NA = Not analysed
2) ND = Not detected, possibly false negatives
3) The % of labs that reported results has been calculated using the number of laboratories that reported results from the total number of laboratories submitting results (n=62).
4) The assigned value for Endosulfan was assessed to be below MRPL and no z-scores were calculated

3.1.1 False positives

One laboratory reported a false positive result for pirimicarb. Although pirimicarb was actually applied to the wheat in the field, the submitted result at 8.1 mg/kg was considered to be a false positive, because no other laboratory reported result for this pesticide.

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 how many laboratories reported false negatives for each pesticide.

3.1.3 Distribution of data

The distributions of the concentration of the MRM pesticide residues reported by the laboratories have been plotted as histograms (see **Appendix 2**). For carbendazim, deltamethrin, diazinon, pirimiphos-methyl and propiconazole the distributions were unimodal. However, the distribution for azoxystrobin was not unimodal, indicating that two sets of data were involved. By analysing the method information given by the participants it was noted that the laboratories adding water to sample prior to extraction were, in general, reporting higher concentrations than the others (see **Figure 1**). After consulting the Advisory Group it was decided to calculate the assigned value for azoxystrobin based only on results from participant who had added water to the sample prior to extraction.

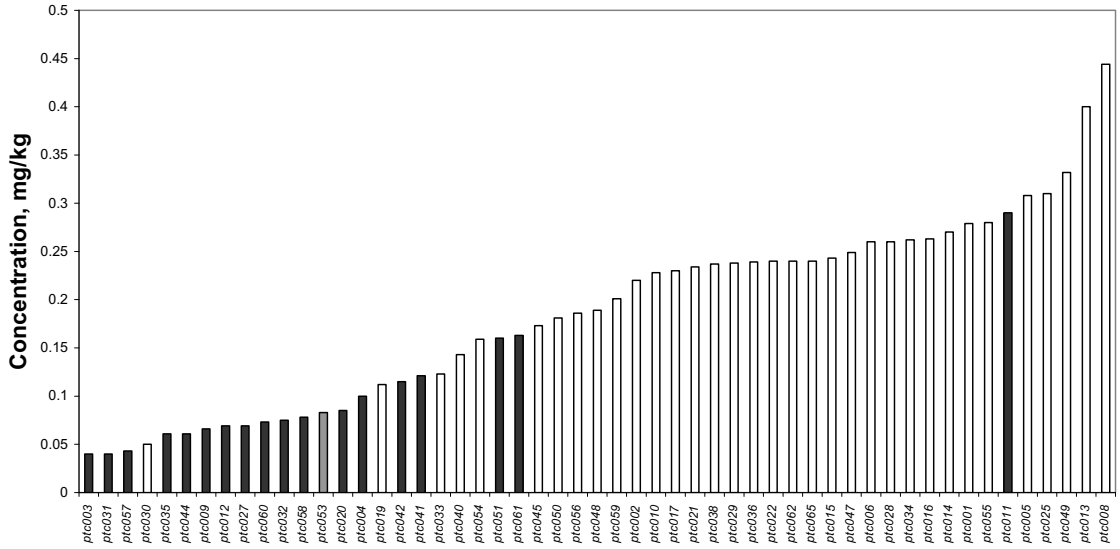


Figure 1. Azoxystrobin results reported by the participants. Bars in white: water addition, bars in black: no water addition and bars in grey: no information given.

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. The following results were regarded as outliers; carbendazim two results were excluded (0.49 mg/kg and 0.664 mg/kg), diazinon one result (0.306 mg/kg) and propiconazole two results (0.807 mg/kg and 1.1 mg/kg). For azoxystrobin also results from participants not adding water to the samples prior to the extraction were excluded (see above). All median 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, a robust standard deviation (Qn) as well as the concentration dependent Horwitz RSDs were also calculated for informative purposes. These RSDs can be seen in **Table 7**.

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

Pesticides	MRPL	Median [1)] (mg/kg)	FFP RSD (%)	Qn RSD (%)	Horwitz RSD (%)
Azoxystrobin	0.05	0.240	25	22	21
Carbendazim	0.1	0.126	25	31	22
Deltamethrin	0.05	0.342	25	24	19
Diazinon	0.02	0.078	25	28	23
Pirimiphos-methyl	0.05	6.33	25	22	12
Propiconazole	0.05	0.353	25	27	19

1) Assigned Value

3.3 Assessment of laboratory performance

3.3.1 z-sores

The z-Scores have been calculated for the quantified pesticides by using the FFP RSD of 25%. **Table 8** shows an overview of the result and **Table 9** shows the individual z-scores together with the median for each laboratory and pesticide. The corresponding graphs are shown in **Appendix 3.**

Table 8. Acceptable, questionable and unacceptable z-scores for MRM pesticides

Pesticides	Acceptable z-scores	Questionable z-scores	Unacceptable z-scores	False negatives
Azoxystrobin	34	14	5	2
Carbendazim	31	1	4	3
Deltamethrin	54	2	1	1
Diazinon	55	1	2	1
Pirimiphos-methyl	56	3	0	1
Propiconazole	50	0	2	0

The assigned value for azoxystrobin was calculated using only the results based on water addition prior to extraction. Only 62 % of the laboratories obtained acceptable z-scores. For deltamethrin, diazinon, propiconazole, pirimiphos-methyl, acceptable results were obtained by 93-96% of the laboratories. However, for carbendazim, acceptable z-scores were obtained by only 79% of the laboratories. The assigned value for carbendazim was close to the MRPL, which could be one of the reasons for the relative high percentage of not acceptable results. However, the MRPL was set at 0.1 mg/kg, which can easily be achieved using available methods. The results show that the extraction efficiency is improved by adding water to the test material and it is therefore recommended to do so when analysing cereal matrices.

Table 9 MRM-Results given by the laboratories (mg/kg) and their calculated *z*-scores using FFP RSD 25%

Lab Code	Azoxystrobin	z-Score (FFP RSD 25%)	Carbendazim	z-Score (FFP RSD 25%)	Deltamethrin	z-Score (FFP RSD 25%)	Diazinon	z-Score (FFP RSD 25%)	Pirimiphos-methyl	z-Score (FFP RSD 25%)	Propiconazole	z-Score (FFP RSD 25%)
MRPL (mg/kg)	0.05		0.1		0.05		0.02		0.05		0.05	
Median (mg/kg)	**0.240**		**0.126**		**0.342**		**0.078**		**6.33**		**0.353**	
ptc001	0.279	0.7	0.119	-0.2	0.266	-0.9	0.066	-0.6	7.47	0.7	0.405	0.6
ptc002	0.220	-0.3	0.130	0.1	NA		0.098	1.0	6.49	0.1	0.300	-0.6
ptc003	0.040	-3.3	0.082	-1.4	0.252	-1.1	0.041	-1.9	4.81	-1.0	0.217	-1.5
ptc004	0.100	-2.3	0.135	0.3	0.335	-0.1	0.050	-1.4	6.33	0.0	0.460	1.2
ptc005	0.308	1.1	0.101	-0.8	0.398	0.7	0.107	1.5	7.39	0.7	0.411	0.6
ptc006	0.260	0.3	0.100	-0.8	0.320	-0.3	0.090	0.6	7.51	0.7	0.460	1.2
ptc008	0.444	3.4	0.180	1.7	0.350	0.1	0.075	-0.2	6.78	0.3	0.462	1.2
ptc009	0.066	-2.9	0.246	3.8	0.275	-0.8	0.070	-0.4	6.40	0.0	0.323	-0.3
ptc010	0.228	-0.2	0.155	0.9	0.363	0.2	0.099	1.1	5.55	-0.5	0.378	0.3
ptc011	0.290	0.8	0.490	11.6	0.490	1.7	0.109	1.6	9.60	2.1	1.100	8.3
ptc012	0.069	-2.9	0.069	-1.8	0.363	0.2	0.104	1.3	5.73	-0.4	0.300	-0.6
ptc013	0.400	2.7	0.130	0.1	0.310	-0.4	0.060	-0.9	5.66	-0.4	0.340	-0.1
ptc014	0.270	0.5	0.175	1.6	0.405	0.7	0.075	-0.2	4.51	-1.2	0.440	1.0
ptc015	0.243	0.1	0.099	-0.8	0.332	-0.1	0.090	0.6	6.96	0.4	0.349	0.0
ptc016	0.263	0.4	0.664	17.2	0.375	0.4	0.128	2.6	9.32	1.9	0.448	1.1
ptc017	0.230	-0.2	NA		0.540	2.3	0.160	4.2	10.20	2.4	0.290	-0.7
ptc018	ND	-3.2	NA		0.362	0.2	0.057	-1.1	5.61	-0.5	0.189	-1.8
ptc019	0.112	-2.1	NA		0.358	0.2	0.078	0.0	5.82	-0.3	0.255	-1.1
ptc020	0.085	-2.6	NA		0.183	-1.9	0.080	0.1	5.25	-0.7	0.480	1.4
ptc021	0.234	-0.1	0.113	-0.4	0.436	1.1	0.087	0.5	7.26	0.6	0.314	-0.4
ptc022	0.240	0.0	0.107	-0.6	0.314	-0.3	0.074	-0.2	5.86	-0.3	0.362	0.1
ptc023	NA		NA		0.231	-1.3	0.069	-0.5	4.56	-1.1	NA	
ptc025	0.310	1.2	NA		0.410	0.8	0.110	1.6	6.80	0.3	NA	
ptc027	0.069	-2.9	NA		0.374	0.4	0.061	-0.9	5.56	-0.5	0.208	-1.6
ptc028	0.260	0.3	0.159	1.1	0.168	-2.1	0.071	-0.4	5.49	-0.5	0.282	-0.8
ptc029	0.238	0.0	NA		0.334	-0.1	0.095	0.9	6.24	-0.1	0.392	0.4
ptc030	0.050	-3.2	NA		0.346	0.0	0.076	-0.1	6.57	0.2	0.201	-1.7
ptc031	0.040	-3.3	NA		0.178	-1.9	0.045	-1.7	3.75	-1.6	0.231	-1.4
ptc032	0.075	-2.8	NA		0.305	-0.4	0.066	-0.6	4.20	-1.3	0.280	-0.8
ptc033	0.123	-2.0	0.191	2.1	0.274	-0.8	0.053	-1.3	4.82	-1.0	0.285	-0.8
ptc034	0.262	0.4	0.113	-0.4	0.318	-0.3	0.073	-0.3	8.29	1.2	0.435	0.9
ptc035	0.061	-3.0	NA		0.301	-0.5	0.059	-1.0	10.30	2.5	NA	
ptc036	0.239	0.0	0.100	-0.8	0.260	-1.0	0.100	1.1	6.80	0.3	0.426	0.8

Lab Code	Azoxystrobin	z-Score (FFP RSD 25%)	Carbendazim	z-Score (FFP RSD 25%)	Deltamethrin	z-Score (FFP RSD 25%)	Diazinon	z-Score (FFP RSD 25%)	Pirimiphos-methyl	z-Score (FFP RSD 25%)	Propiconazole	z-Score (FFP RSD 25%)
MRPL (mg/kg)	0.05		0.1		0.05		0.02		0.05		0.05	
Median (mg/kg)	**0.240**		**0.126**		**0.342**		**0.078**		**6.33**		**0.353**	
ptc037	NA		0.180	1.7	0.210	-1.6	NA		6.90	0.4	NA	
ptc038	0.237	-0.1	0.108	-0.6	0.342	0.0	0.082	0.2	6.48	0.1	0.491	1.5
ptc040	0.143	-1.6	ND	-0.8	0.411	0.8	0.042	-1.8	5.17	-0.7	0.227	-1.4
ptc041	0.121	-2.0	0.074	-1.6	0.346	0.0	0.082	0.2	8.29	1.2	NA	
ptc042	0.115	-2.1	0.149	0.7	0.470	1.5	0.077	-0.1	6.11	-0.1	0.314	-0.4
ptc043	NA		NA		0.230	-1.3	ND	-3.0	4.90	-0.9	NA	
ptc044	0.061	-3.0	NA		0.370	0.3	0.076	-0.1	6.36	0.0	0.221	-1.5
ptc045	0.173	-1.1	0.121	-0.1	0.378	0.4	0.102	1.2	6.22	-0.1	0.398	0.5
ptc047	0.249	0.2	0.100	-0.8	0.324	-0.2	0.057	-1.1	7.00	0.4	0.304	-0.5
ptc048	0.189	-0.9	0.134	0.3	0.357	0.2	0.084	0.3	5.43	-0.6	0.380	0.3
ptc049	0.332	1.5	0.148	0.7	0.415	0.9	0.112	1.7	7.85	1.0	0.518	1.8
ptc050	0.181	-1.0	ND	-0.8	0.384	0.5	0.103	1.3	7.67	0.8	0.397	0.5
ptc051	0.160	-1.3	NA		0.380	0.4	0.092	0.7	ND	-4.0	0.380	0.3
ptc052	NA		0.154	0.9	0.282	-0.7	0.054	-1.2	5.15	-0.7	NA	
ptc053	0.083	-2.6	0.101	-0.8	0.346	0.0	0.088	0.5	6.53	0.1	0.324	-0.3
ptc054	0.159	-1.4	NA		0.190	-1.8	0.080	0.1	5.32	-0.6	0.249	-1.2
ptc055	0.280	0.7	0.220	3.0	0.435	1.1	0.115	1.9	6.35	0.0	0.475	1.4
ptc056	0.186	-0.9	0.144	0.6	0.330	-0.1	0.059	-1.0	5.11	-0.8	0.807	5.1
ptc057	0.043	-3.3	NA		0.383	0.5	0.306	11.7	4.61	-1.1	NA	
ptc058	0.078	-2.7	NA		0.272	-0.8	0.077	-0.1	5.16	-0.7	0.357	0.0
ptc059	0.201	-0.7	NA		0.393	0.6	0.103	1.3	7.91	1.0	0.406	0.6
ptc060	0.073	-2.8	NA		0.220	-1.4	0.098	1.0	7.93	1.0	0.277	-0.8
ptc061	0.163	-1.3	0.120	-0.2	0.465	1.5	0.090	0.6	5.68	-0.4	0.376	0.3
ptc062	0.240	0.0	0.120	-0.2	0.070	-3.2	0.110	1.6	7.25	0.6	0.400	0.5
ptc063	ND	-3.2	NA		0.275	-0.8	0.072	-0.3	3.85	-1.6	0.490	1.5
ptc064	0.240	0.0	ND	-0.8	ND	-3.5	0.078	0.0	6.12	-0.1	0.246	-1.2
ptc065	0.279	0.7	0.135	0.3	0.266	-0.9	0.068	-0.5	6.78	0.3	0.340	-0.1

3.3.2 Weighted Sum of z-sores

The MRM results were additionally evaluated by calculating the Weighted Summed z-Scores (WSZ). The WSZ values were calculated only for the 49 laboratories that reported 5 or more results for MRM pesticides (excluding endosulfan). The z-scores can be seen in **Table 10** and the corresponding graph in **Appendix 3**. In totally, 32 participants obtained WSZ values at or below 2 (65%) and 6 participants obtained WSZ above 2 but below or at 3 (12%). Eleven participants obtained WSZ values above 3 (22%). No differences were seen between National Reference laboratories (NRL) and official laboratories.

Table 10. Weighted Sum of z-scores for laboratories which reported 5 or more results, no. of pesticide analysed, false negatives reported and status as NRL

Lab code	WSZ	No. of pesticide analysed	False negative	NRL	Lab code	WSZ	No. of pesticide analysed	False negative	NRL
ptc065	0.2	5		x	**ptc028**	1.5	6		x
ptc022	0.3	6		x	**ptc019**	1.6	5		
ptc029	0.3	5		x	**ptc053**	1.6	6		
ptc015	0.3	6			**ptc004**	1.7	6		
ptc038	0.4	6			**ptc013**	1.7	6		x
ptc048	0.4	6		x	**ptc058**	2.0	5		
ptc002	0.4	5			**ptc033**	2.0	6		
ptc021	0.5	6		x	**ptc012**	2.2	6		
ptc047	0.5	6			**ptc044**	2.2	5		
ptc010	0.5	6			**ptc032**	2.3	5		
ptc034	0.6	6			**ptc020**	2.4	5		
ptc045	0.6	6			**ptc027**	2.4	5		
ptc001	0.6	6		x	**ptc060**	2.5	5		
ptc006	0.7	6		x	**ptc062**	3.2	6		x
ptc036	0.7	6			**ptc055**	3.3	6		
ptc061	0.7	6		x	**ptc008**	3.4	6		x
ptc050	0.8	6	x	x	**ptc030**	3.6	5		
ptc059	0.8	5			**ptc003**	3.9	6		x
ptc014	0.8	6			**ptc031**	4.7	5		
ptc005	0.9	6			**ptc056**	4.7	6		x
ptc054	1.0	5			**ptc009**	4.9	6		
ptc041	1.0	5			**ptc011**	5.0	6		x
ptc040	1.2	6	x	x	**ptc016**	5.0	6		x
ptc049	1.3	6			**ptc017**	5.0	5		
ptc042	1.5	6							

3.4 Analytical methods used for MRM pesticides

Detailed information regarding the methods used by the participants is presented in **Appendix 4**. An overview of the reference methods used is given in **Table 11**. The table shows the methods used by 3 or more laboratories. All methods used by less than 3 laboratories are categorised in the group 'other'. The QuEChERS method was used by 8-13 participants and was the most used method, although different versions were applied. A few participants used the Dutch method, The CEN method EN 12393, Luke, the German official method and the Italian method. However, the participants used a broad variety of methods and a summery is therefore given below.

Table 11 Overview of the methods used.

Pesticide	QuEChERS 1)	Dutch 2)	EN 12393	Luke	German 3)	Italian 4)	Other 5)	Total
Azoxystrobin	13	2	4	3	4	2	25	**53**
Carbendazim	12	1	2	0	0	0	21	**36**
Deltamethrin	8	3	5	3	6	3	29	**57**
Diazinon	10	3	5	3	5	3	29	**58**
Pirimiphos-methyl	10	3	5	3	4	3	31	**59**
Propiconazole	12	2	3	2	4	3	26	**52**

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) Italian: Rapporto ISTISAN 97/23, 1997. Metodo multiresiduo per l'analisi di residui di antiparassitari in prodotti vegetalil
5) 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 12**. The LC systems used were mainly LC/MS/MS. Only a few results were based on LC detectors like DAD and Fluorescence. However, half of the results obtained with GC-systems were detected with selective detectors like NPD, FPD or ECD. The rest were obtained by MS of these were only a few analysed by GC-MS/MS. Carbendazim was only detected by LC-systems. Deltamethrin, diazinon and pirimiphos-methyl were predominately detected on GC-systems although some participants did use LC. For azoxystrobin and propiconazole 40% and 25% of the participants, respectively, used LC-systems.

Table 12. Overview on the detection systems used by the participants. Number of participant, which used 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	**34**	4		18	9	2	1	**22**	1	20	1	**3**	
Carbendazim								**36**	6	30			
Deltamethrin	**56**	2		32	16	5	1[b]	**3**		2	1	**2**	
Diazinon	**52**	17	5	12	15	3		**8**		7	1	**3**	1
Pirimiphos-methyl	**54**	22	5	6	17	4		**7**		6	1	**3**	1
Propiconazole	**39**	7		18	11	2	1	**14**		14		**2**	1

a) DAD or Fluorescence
b) TOF

Between 54-67% of the participants were accredited for the individual pesticides present in the test material (see **Table 13**). Only 54% of the participants were accredited for propiconazole, a pesticide commonly applied to wheat, while 67% were accredited for diazinon, which is not commonly applied for cereals.

Table 13 Accredited laboratories, LODs and Sub-Sample Weights (SW) listed for MRM pesticides. The numbers in brackets are those laboratories that are not accredited. The LODs are given in mg/kg and SW in g.

Pesticide	ACCREDITED	LOD ≤ 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	29 (24)	18	17	17		19	12	5	5	6	4
Carbendazim	22 (14)	19	11	6	2	19	6	6	2	1	2
Deltamethrin	36 (21)	15	17	24	1	15	16	4	7	6	6
Diazinon	39 (19)	23	31	4		18	13	6	7	7	5
Pirimiphos-methyl	37 (22)	23	13	23		19	12	5	7	7	7
Propiconazole	28 (24)	18	14	20		23	9	4	7	4	3

More than half of the laboratories used 10 g or less of test material for the analysis of MRM pesticides. Probably by mistake, one lab reported to have used a sample weight of 0 g another of 500 g. These data are not included in the **Table 13**. Twelve participants ask for extra test material, although at least ten of them, according to their method details reported, had no justifiable need to request more.

Table 14. Method information about water addition prior to extraction, clean-up and calibration . The numbers in brackets are participants that did not add water to the sample prior to extraction (see ANNEX III – 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	34 (18)	15	9	7	12	2	1	5	33	7	10	2	
Carbendazim	31 (4)	1	7	4	12	1	6	2	23	2	10	1	
Deltamethrin	28 (27)	26	7	8	8	1	2	4	33	7	13	4	
Diazinon	32 (24)	25	8	8	6	2	2	4	31	7	13	5	1
Pirimiphos-methyl	32 (24)	24	8	8	7	2	3	4	32	6	14	5	1
Propiconazole	33 (18)	16	9	8	8	2	2	4	31	6	10	3	1

3.5 Conclusions on MRM pesticides

A homogenous test material of wheat, including both incurred and spiked samples, was successfully prepared. The wheat was sprayed in the field and spiked in the laboratory following harvest with commercially available pesticide formulations. The test material included the following MRM pesticides: azoxystrobin, carbendazim, deltamethrin, endosulfan, diazinon, pirimiphos-methyl and propiconazole. Sixty-three laboratories representing 25 countries agreed to participate in this proficiency test, and only one laboratory did not submit results. Five Member States did not participate.

The z-Scores have been calculated for the quantified pesticides by using the FFP RSD of 25%. The assigned value for endosulfan was below the MRPL at 0.05 and consequently no z-scores were calculated. The distribution for azoxystrobin was not unimodal, indicating that two distinct data sets were involved. By analysing the method information given by the participants it was noted that the laboratories adding water to the sample prior to extraction were reporting higher

residue levels than the laboratories that did not add water and consequently, the assigned value for azoxystrobin was calculated only based on results from participant who had added water to the test material prior to extraction. Therefore only 62 % of the laboratories obtained acceptable z-scores for azoxystrobin. For deltamethrin, diazinon, propiconazole, pirimiphos-methyl, acceptable results were obtained by 93-96% of the laboratories. However, for carbendazim, acceptable z-scores were obtained by only 79% of the laboratories. One false positive and eight false negative results were reported.

The participants used a broad variety of methods. The QuEChERS method was used by 8-13 participants and was the most frequently used method. Carbendazim was analysed by LC-systems by all participants. Azoxystrobin, deltamethrin, diazinon, pirimiphos-methyl and propiconazole were predominately analysed on GC-systems.

3.6 Future proficiency test on cereals

Proficiency tests for pesticide residues in cereals will continue to be performed in the future for various important pesticides and commodities. In future PTs the list of pesticides to be sought will be expanded and will be made known to the laboratories well in advance of the test, so that laboratories have time to include new pesticides in their scope. The list will include compulsory pesticides to be analysed in order to be classified as a Category A laboratory. The pesticide residues will be incurred wherever practical. However, weather conditions may influence the levels of residues, so that additional laboratory spiking may be necessary.

The online web submission will be continued and expanded with an online registration website.

4. RESULTS – SRM PESTICIDES

Sixty-three laboratories agreed to participate in this proficiency test, while one of them did not submit any results. Only 30 of those laboratories representing 18 countries did analyse for SRM pesticides. Appendix 9 shows the different laboratories that analyzed for every specific SRM-pesticide sorted by country and status as NRL or not.
All laboratories that agreed to participate are listed in ANNEX II. All data reported by the participants are shown in the tables below. **Table 15** and **Table 16** give an overview of the results reported for each pesticide. All analytical results reported can be seen in **Table 17** and **Table 18**. The analytical methods used by the laboratories and the recoveries achieved are shown in **Appendix 8**.

4.1 Overview of results for SRM-pesticides

Table 15 gives an overview of the results reported for each pesticide. Clearly fewer laboratories have analyzed for SRM- than for MRM-compounds. Chlormequat, which is part of the monitoring program was analyzed by 40% of the laboratories. The acidic pesticides MCPA and mecoprop were analyzed by roughly 30 % of the labs as free acids with half of them also performing the optional exercise of analyzing those compounds following alkaline hydrolysis to cover covalently bound residues (esters and conjugates).

Table 15. Summary of Results (SRM)

Pesticides	No. of reported Results	No. of reported NA [1)]	No. of reported ND [2)]	% of labs that reported results [3)]
Chlormequat	25	37	1	40
MCPA	19	43	6	31
MCPA (optional)	10	52	0	16
Mecoprop	18	44	0	29
Mecoprop (optional)	10	52	0	16

1) NA = Not analysed
2) ND = Not detected, possibly false negatives
3) The % of labs that reported results has been calculated using the number of laborarories that reported results from the total number of laboratories submitting results (n=62).

4.1.1 False positives

In the present test one laboratory has detected 2,4-D (following alkaline hydrolysis) in the matrix, while 8 other labs, that analysed for this pesticide as well, did not detect this residue. This re-ported result was therefore considered to be a false positive. It should be taken into account, however, that this analysis was optional.

4.1.2 False negatives

One laboratory reported chlormequat as ND while all other labs that analysed for this compound (24) have detected it in the matrix. Since the median of all results (1.01 mg/kg) is well above the reporting limit of that laboratory (0.01 mg/kg) as well as the MRPL (0.05 mg/kg), the result is considered as false negative.

In the case of MCPA six of the 19 labs, that analysed for this compound, reported an ND. Considering the fact that the obtained median of 0.04 mg/kg is below the MRPL of 0.05 mg/kg, these results were not considered as false negatives even in those three cases, where the laboratories indicated a reporting limit below the assigned value. Z-scores were calculatedfor the remaining 13 labs, that have reported results for MCPA. It was shown, however, that even if these 3 laboratories would have been included in the population (by setting their reporting levels as their analytical result), the median concentration and the z-scores would have only shifted marginally, not affecting the classification of all other laboratory results into satisfactory, questionable and unsatisfactory.

4.2 Assigned values and target standard deviations

To establish the assigned values, the medians of all the reported results were used. A statistical programme was used to calculate the medians.

The target standard deviation was obtained using a fixed FFP RSD value of 25%. In parallel, the robust standard deviation (Qn) as well as the concentration dependent Horwitz RSDs were calculated for informative purposes. These RSDs are given in **Table 16**.

In the case of MCPA one value was excluded from the median calculation since it was very distant from the second largest result reported. However, the median would not have been significantly shifted even if this outlier had been included. All median values for the SRM pesticides are given in **Table 16**.

Table 16. Median values and RSDs for all SRM pesticides present in the test material

Pesticide	Median* (mg/kg)	FFP RSD (%)	Horwitz RSD (%)	Qn RSD (%)
Chlormequat	1.01	25	16	32
MCPA	0.04	25	26	56
MCPA (optional)	0.284	25	19	16
Mecoprop	0.312	25	19	24
Mecoprop (optional)	0.454	25	18	28

* Assigned Value

In general the FFP-RSD matches well with the Qn RSD with exception of the MCPA, which was the analyte contained in the sample at the lowest concentration, in fact even lower than the MRPL.

4.3 Assessment of laboratory performance

4.3.1 z-Scores

z-Scores have been calculated for the quantified pesticides in two different ways.

- Using the FFP RSD of 25%; **Table 17** shows the individual z-scores together with the median for each laboratory and pesticide, and **Appendix 7** the corresponding graphs.
- Using Horwitz Equation; **Table 18** shows the individual z-scores together with the median for each laboratory and pesticide and **Appendix 7** the corresponding graphs.

The SRM results are treated individually and no laboratory ranking based on Weighted Summed z-Scores (WSZ) was calculated.

Table 17 SRM-Results given by the laboratories (mg/kg) and their calculated z-scores using FFP RSD 25%

Lab Code	Chlormequat	z-Score (FFP RSD 25%)	MCPA	z-Score (FFP RSD 25%)	MCPA (optional)	z-Score (FFP RSD 25%)	Mecoprop	z-Score (FFP RSD 25%)	Mecoprop (optional)	z-Score (FFP RSD 25%)
MRPL (mg/kg)	**0.05**		**0.05**		**0.05**		**0.05**		**0.05**	
Median (mg/kg)	**1.01**		**0.04**		**0.284**		**0.312**		**0.454**	
001	1.21	0.8	0.039	-0.1	NA		0.332	0.3	NA	
002	NA		0.036	-0.4	0.27	-0.2	0.15	-2.1	0.3	-1.4
003	ND	-3.8	0.014	-2.6	0.338	0.8	0.095	-2.8	0.337	-1.0
004	0.136	-3.5	NA		NA		NA		NA	
006	1.02	0.0	ND	--	0.355	1.0	0.306	-0.1	0.457	0.0
010	0.686	-1.3	NA		NA		NA		NA	
012	0.88	-0.5	0.051	1.1	0.247	-0.5	0.286	-0.3	0.451	0.0
014	1.285	1.1	ND	--	NA		NA		NA	
015	0.91	-0.4	ND	--	NA		0.688	4.8	NA	
016	1.162	0.6	NA		NA		NA		NA	
021	NA		0.515	5.0	NA		0.069	-3.1	NA	
022	0.943	-0.3	NA		NA		NA		NA	
027	1.43	1.7	NA		NA		NA		NA	
028	1.082	0.3	0.026	-1.4	0.2	-1.2	0.3	-0.1	0.657	1.8
034	1.41	1.6	0.04	0.0	0.262	-0.3	0.322	0.1	0.434	-0.2
036	NA		0.059	1.9	0.286	0.0	0.382	0.9	0.4	-0.5
038	1.24	0.9	0.04	0.0	0.292	0.1	0.327	0.2	0.625	1.5
039	1.578	2.2	NA		NA		NA		NA	
040	0.54	-1.9	NA		NA		NA		NA	
042	0.809	-0.8	NA		0.29	0.1	NA		0.668	1.9
045	1.0	-0.1	ND	--	NA		0.26	-0.7	NA	
048	NA		0.05	1.0	NA		0.273	-0.5	NA	
050	0.868	-0.6	0.046	0.6	0.282	0.0	0.317	0.1	0.505	0.4
051	1.1	0.4	NA		NA		NA		NA	
053	0.773	-0.9	ND	--	NA		0.295	-0.2	NA	
055	1.19	0.7	ND	--	NA		0.37	0.8	NA	
056	NA		0.027	-1.3	NA		0.324	0.2	NA	
059	0.811	-0.8	0.181	5.0	NA		0.478	2.1	NA	
061	1.12	0.4	NA		NA		NA		NA	
062	0.72	-1.1	NA		NA		NA		NA	

Note: Only laboratories that have analyzed at least one of the SRM pesticides were included in this list

Table 18 SRM-Results given by the laboratories (mg/kg) and their calculated z-scores using Horwitz Equation

Lab Code	Chlormequat	z-Score (16%)	MCPA	z-Score (26%)	MCPA (optional)	z-Score (19%)	Mecoprop	z-Score (19%)	Mecoprop (optional)	z-Score (18%)
MRPL (mg/kg)	**0.05**		**0.05**		**0.05**		**0.05**		**0.05**	
Median (mg/kg)	**1.01**		**0.04**		**0.284**		**0.312**		**0.454**	
001	1.21	1.2	0.039	-0.1	NA		0.332	0.3	NA	
002	NA		0.036	-0.4	0.27	-0.3	0.15	-2.7	0.3	-1.9
003	ND	-5.0	0.014	-2.5	0.338	1.0	0.095	-3.7	0.337	-1.4
004	0.136	-5.0	NA		NA		NA		NA	
006	1.02	0.1	ND	--	0.355	1.3	0.306	-0.1	0.457	0.0
010	0.686	-2.0	NA		NA		NA		NA	
012	0.88	-0.8	0.051	1.1	0.247	-0.7	0.286	-0.4	0.451	0.0
014	1.285	1.7	ND	--	NA		NA		NA	
015	0.91	-0.6	ND	--	NA		0.688	5.0	NA	
016	1.162	0.9	NA		NA		NA		NA	
021	NA		0.515	5.0	NA		0.069	-4.1	NA	
022	0.943	-0.4	NA		NA		NA		NA	
027	1.43	2.6	NA		NA		NA		NA	
028	1.082	0.4	0.026	-1.3	0.2	-1.6	0.3	-0.2	0.657	2.5
034	1.41	2.5	0.04	0.0	0.262	-0.4	0.322	0.2	0.434	-0.2
036	NA		0.059	1.3	0.286	0.0	0.382	1.2	0.4	-0.7
038	1.24	1.4	0.04	0.0	0.292	0.1	0.327	0.3	0.625	2.1
039	1.578	3.5	NA		NA		NA		NA	
040	0.54	-2.9	NA		NA		NA		NA	
042	0.809	-1.2	NA		0.29	0.1	NA		0.668	2.6
045	1.0	-0.1	ND	--	NA		0.26	-0.9	NA	
048	NA		0.05	1.0	NA		0.273	-0.7	NA	
050	0.868	-0.9	0.046	0.6	0.282	0.0	0.317	0.1	0.505	0.6
051	1.1	0.6	NA		NA		NA		NA	
053	0.773	-1.5	ND	--	NA		0.295	-0.3	NA	
055	1.19	1.1	ND	--	NA		0.37	1.0	NA	
056	NA		0.027	-1.3	NA		0.324	0.2	NA	
059	0.811	-1.2	0.181	5.0	NA		0.478	2.8	NA	
061	1.12	0.7	NA		NA		NA		NA	
062	0.72	-1.8	NA		NA		NA		NA	

Note: Only laboratories that have analyzed at least one of the SRM pesticides were included in this list

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 8**.

In the case of chlormequat 23 out of the 25 laboratories that analyzed this compound used methanol for extraction. 8 laboratories indicated that they have use isotopically labeled chlormequat as internal standard, 9 laboratories that they did not use and 7 did not give any answer. Determinative analysis was performed by LC-MS/MS in all cases. Just 5 of the labs indicated that they were not accredited for this compound.

In the case of MCPA, out of the 13 laboratories that have been considered in the evaluation 8 have employed the QuEChERS-method. Out of the remaining 5 laboratories 3 indicated that they did use other methods (in-house, EPA) and 2 labs did not indicate the method. In the determinative step 11 laboratories employed LC-MS/MS, two laboratories GC-MS and the remaining 5 did not disclose their procedure.

In the case of mecoprop 10 out of the 18 laboratories that have targeted this compound employed the QuEChERS-method. Out of the remaining 8 laboratories, 5 used other methods (in-house, EPA, ChemElut, SLV) and 3 did not indicate their method. In the determinative step 11 laboratories employed LC-MS/MS, two laboratories GC-MS and the remaining five did not disclose their technique.

In the case of MCPA and mecoprop involving alkaline hydrolysis 8 out of the 10 laboratories that participated in this optional exercise indicated that they employed the QuEChERS method. A modified QuEChERS procedure involving alkaline hydrolysis was published in the CRL-website.

The sample amounts employed by the participants for the analysis of MCPA and mecoprop ranged between 2 and 15 g and for chlormequat between 1 and 25 g. It should be noted that the homogeneity test was performed using 5 g sample portions for the acidic pesticides and 10 g portions for chlormequat. Higher sub-sampling variations are to be expected when smaller sample amounts are used.

4.5 Conclusions on SRM pesticides

As this is just the second EU-PT focusing on analytes typically covered by single residue methods and the first one focusing on a cereal matrix and taking into account that many laboratories have only recently introduced these compounds in their target spectrum this exercise is considered as an introductory one primarily aiming at helping the laboratories to assess their performance and to take corrective measures where necessary.

The sample material contained two pesticides sprayed in the field (MCPA and chlormequat) and one pesticide spiked in the laboratory (mecoprop). Out of 63 laboratories representing 25 countries agreed to participate in this proficiency test, only 30 laboratories representing 18 countries did submit results for SRM analytes copared to 25 in the PT-SRM01.

25 labs from 17 countries have analyzed for chlormequat, which is included in the EU-coordinated monitoring program (one false negative, 22 satisfactory, 1 questionalble and 1 un-

acceptable results), 19 labs from 13 countries for MCPA (10 satisfactory, 1 questionable, 2 unacceptable and 6 not detected) and 18 labs from 13 countries for mecoprop (14 satisfactory, 2 questionable and 2 unacceptable results). One false positive result was reported (2,4-D).

The z-Scores have been calculated for the quantified pesticides using the FFP RSD of 25%. The variation of the results was generally higher than in the PT-SRM01, but in general within the limits typically seen in multi residue exercises (Qn(Chlormequat) = 32%, Qn(MCPA) = 56%, Qn(mecoprop) = 24%). It should be noted, that in the case of MCPA (as free acid) the assigned concentration (0.04 mg/kg) was lower than the stated Minimum Required Performance Level (MRPL) of 0.05 mg/kg. Furthermore, some laboratories may have employed methods involving steps releasing esters and/or conjugates (e.g. acid-base cleanup, high extraction pH), which may also have contributed to the increased variation.. It should also be noted that the cereals are considered as more challenging matrix than the apple juice, which was used in PT-SRM01.

In the case of the optional exercise involving alkaline hydrolysis to release bound residues of MCPA and mecoprop the results were more satisfying (Qn(MCPA) = 16%, Qn(mecoprop) = 28%), with all 10 laboratories for MCPA (optional) and mecoprop (optional), being within the satisfactory range (z< +/-2). One of the reasons for the better performance here compared to the free acid analysis is surely the fact that the analysis involving alkaline hydrolysis is more defined. The fact that better performing laboratories have participated in this optional exercise may have also contributed to this.

An interesting aspect noticed is the ca. 7-fold increase of the MCPA residue (median) following alkaline hydrolysis (MCPA = 0.04 mg/kg, MCPA(optional) = 0.284 mg/kg), which indicates that roughly 86% of the field-sprayed residue was bound. In the case of mecoprop, which was added post-harvest, the increase of the median following alkaline hydrolysis was just ca 1.4-fold (mecoprop = 0.312 mg/kg, mecoprop(optional) = 0.454 mg/kg), indicating roughly 30% bound residues.

The relatively low participation rate in this proficiency test indicates that the use of single residue or group specific methods in the EU member states may still be rather limited. This is surely related to the fact that such methods often involve laborious and troublesome sample preparation steps or require special instrumentation, which in many laboratories is either non-existent or is fully occupied with other analyses. If at all, single residue methods are often only performed when they are specifically required (e.g. when these pesticides are included in the monitoring list).

In any case it is obvious, that appropriate measures should be taken to ensure that compounds currently not amenable to multi residue methods are also satisfactorily monitored. Such measures may include the development and validation of simple-to-use, fast and cheap methodologies for such compounds.

4.6 Suggestions for future work for SRM pesticides

Proficiency tests for SRM pesticide residues in cereals should continue to be performed in the future focusing on pesticides of high relevance and particularly on those included in current or upcoming EU-coordinated monitoring programs. In future PTs the list of pesticides to be sought for should be expanded and distributed to the laboratories well in advance of the test, so that laboratories have time to include new pesticides in their scope.

5. ACKNOWLEDGEMENTS

The Organisers wishes to thank the members of the Scientific Committee for their valuable advice. Additionally, the Organiser wishes to give special thanks Amadeo R. Fernández-Alba, Paula Medina and Octavio Malato from CRL of Fruit and Vegetables for all the help in the preparation of the PT and the assesment of the data.

6. REFERENCES

1. Council Directive 86/362/EEC of 24 July 1986 on the fixing of maximum levels for pesticide residues in and on cereals. Published at OJ of the EU 221, 7.8.1986, p. 37. Directive as last amended by Commission Directive 2006/62/EC (OJ L 206, 27.7.2006, p. 27).

2. Council Directive 90/642/EEC of 27 November 1990 on the fixing of maximum levels for pesticide residues in and on certain products of plant origin, including fruit and vegetables. Published at OJ L 350, 14.12.1990, p. 71. Directive as last amended by Commission Directive 2006/62/EC.

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

4. Granby K., Andersen J.H., Christensen H.B.,2004. Analysis of pesticides in fruit, vegetables and cereals using methanolic extraction and detection by liquid chromatography-tandem mass spectrometry. Analytica Chimica Acta, 520, 165-176.

5. Granby K., Andersen J.H., Christensen H.B.,2004. Analysis of pesticides in fruit, vegetables and cereals using methanolic extraction and detection by liquid chromatography-tandem mass spectrometry. Analytica Chimica Acta, 520, 165-176.

6. EN 15662 'Foods of plant origin - Determination of pesticide residues using GC-MS(/MS) and/or LC-MS/MS following acetonitrile extraction/partitioning and clean-up by dispersive SPE - QuEChERS-method, CEN/TC 275/WG 4

7. M. Anastassiades, S. J. Lehotay, D. Stajnbaher, F. J. Schenck: Fast and Easy Multiresidue Method Employing Acetonitile Extraction/Partitioning and “Dispersive Solid-Phase Extraction” for the Determination of Pesticide Residues in Produce, J. AOAC Int., 86 (2003) 412-431

8. Amtliche Sammlung von Untersuchungsverfahren nach § 64 LFGB L 00.00-76, Untersuchung von Le-bensmitteln - Bestimmung von Chlormequat und Mepiquat in Lebensmitteln mittels HPLC-MS/MS

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

7. APPENDICES

MRM APPENDICES

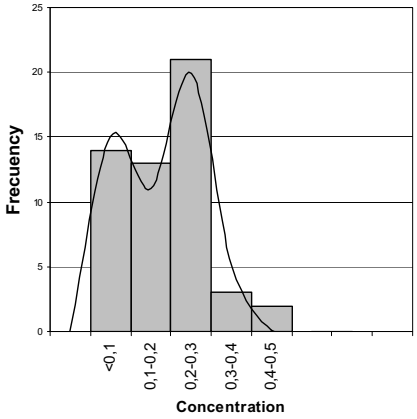
Appendix 1 Homogeneity data

Azoxystrobin (mg/kg)		
Sample	Portion 1	Portion 2
1	0.043	0.034
2	0.038	0.033
3	0.034	0.035
4	0.034	0.036
5	0.033	0.029
6	0.036	0.040
7	0.038	0.038
8	0.045	0.033
9	0.036	0.038
10	0.032	0.035
Carbendazim (mg/kg)		
Sample	Portion 1	Portion 2
1	0.077	0.109
2	0.099	0.083
3	0.078	0.096
4	0.104	0.119
5	0.097	0.099
6	0.101	0.089
7	0.116	0.120
8	0.103	0.074
9	0.098	0.095
10	0.110	0.100
Deltamethrin (mg/kg)		
Sample	Portion 1	Portion 2
1	0.342	0.412
2	0.377	0.410
3	0.436	0.473
4	0.411	0.394
5	0.460	0.345
6	0.405	0.424
7	0.373	0.355
8	0.319	0.483
9	0.361	0.445
10	0.396	0.434

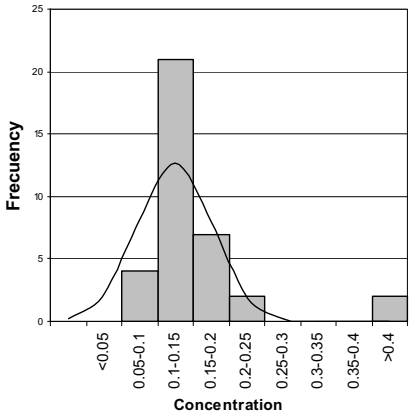
Diazinon (mg/kg)		
Sample	Portion 1	Portion 2
1	0.059	0.064
2	0.074	0.065
3	0.084	0.063
4	0.072	0.067
5	0.068	0.068
6	0.066	0.071
7	0.070	0.069
8	0.068	0.078
9	0.071	0.082
10	0.076	0.076
Pirimiphos-methyl (mg/kg)		
Sample	Portion 1	Portion 2
1	6.163	6.726
2	5.874	6.650
3	5.813	6.467
4	5.509	6.011
5	6.209	5.554
6	8.598	6.574
7	6.772	5.752
8	6.559	6.878
9	6.498	6.924
10	7.502	5.859
Propiconazole (mg/kg)		
Sample	Portion 1	Portion 2
1	0.217	0.202
2	0.280	0.216
3	0.234	0.280
4	0.230	0.220
5	0.244	0.194
6	0.253	0.293
7	0.209	0.202
8	0.211	0.257
9	0.243	0.298
10	0.204	0.237

Appendix 2 Histograms

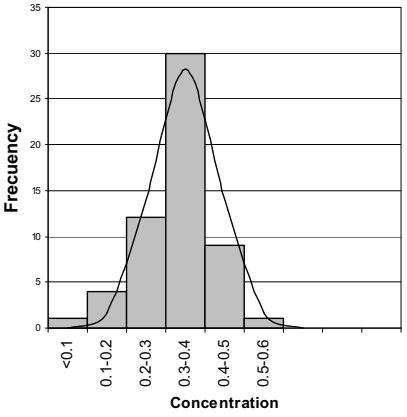
Azozystrobin



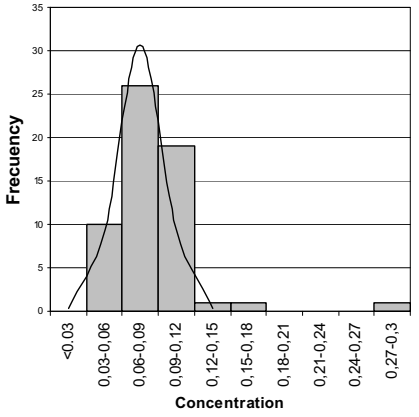
Carbendazim



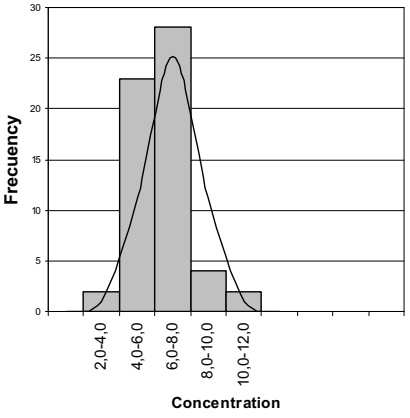
Deltamethrin



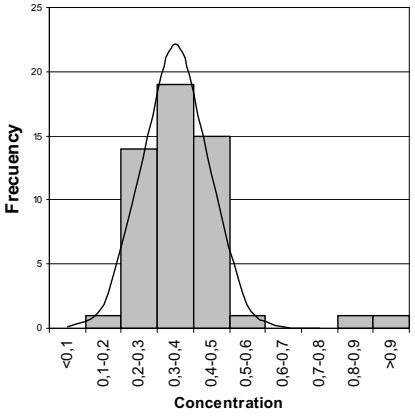
Diazinon



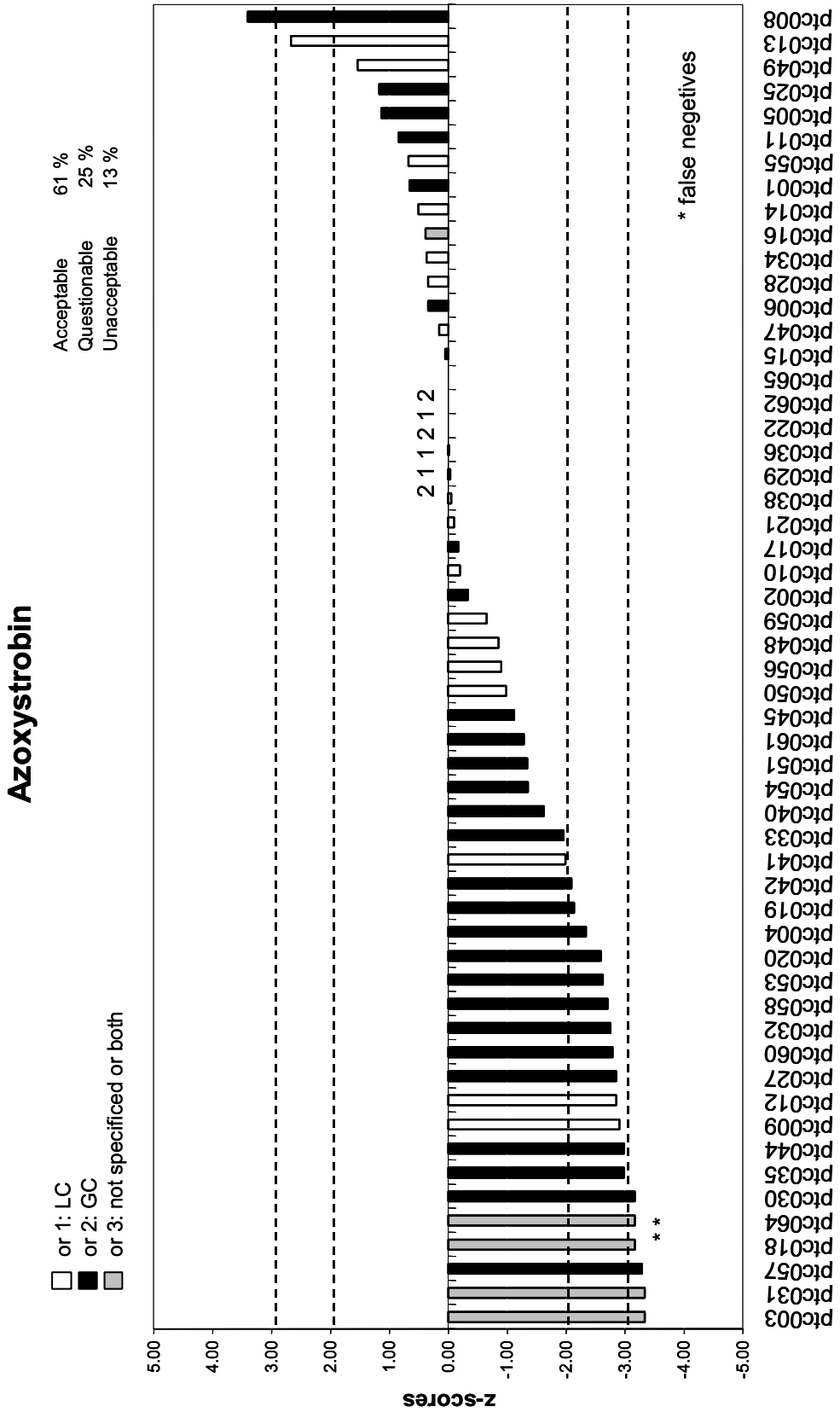
Pirimiphos-methyl

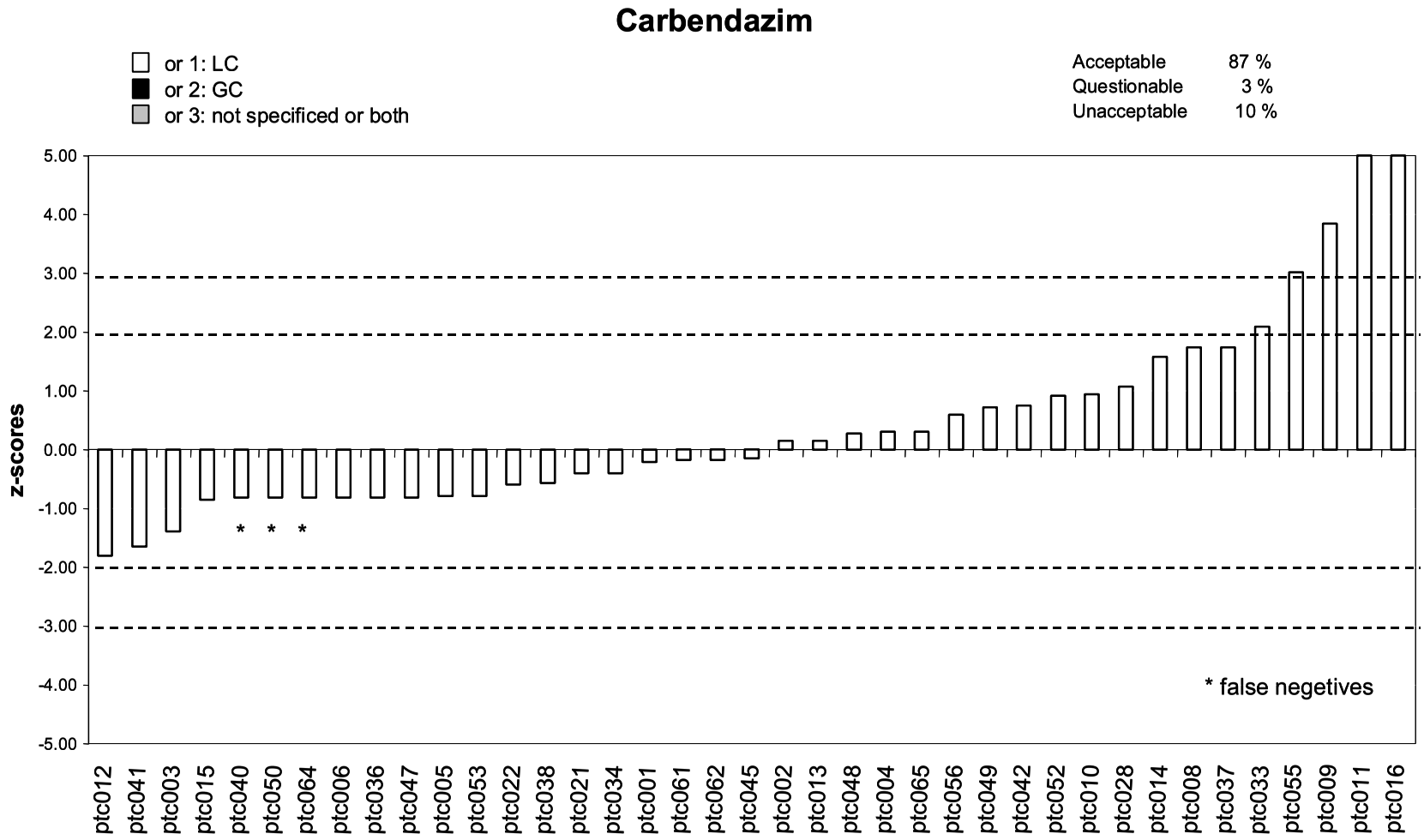


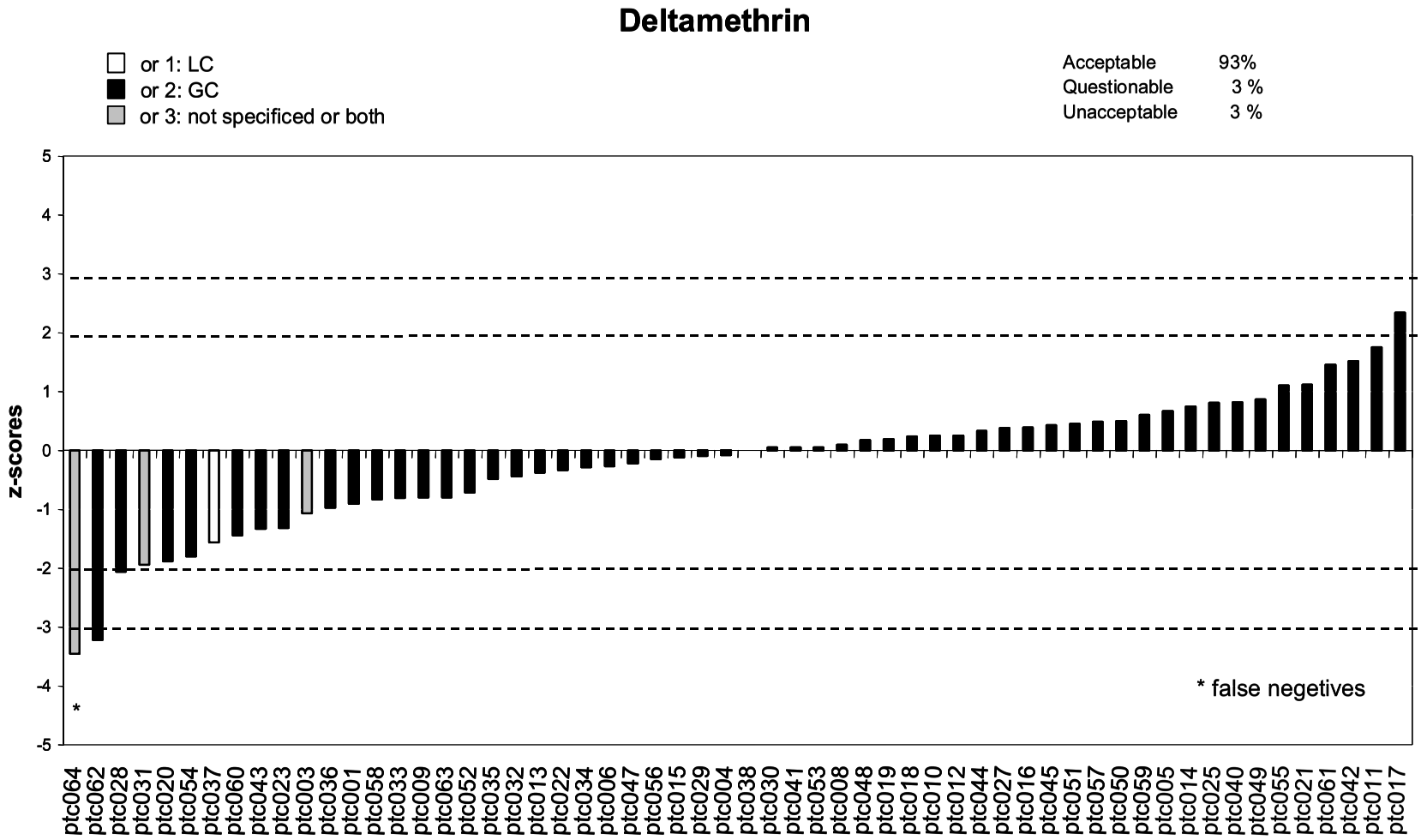
Propizonazol

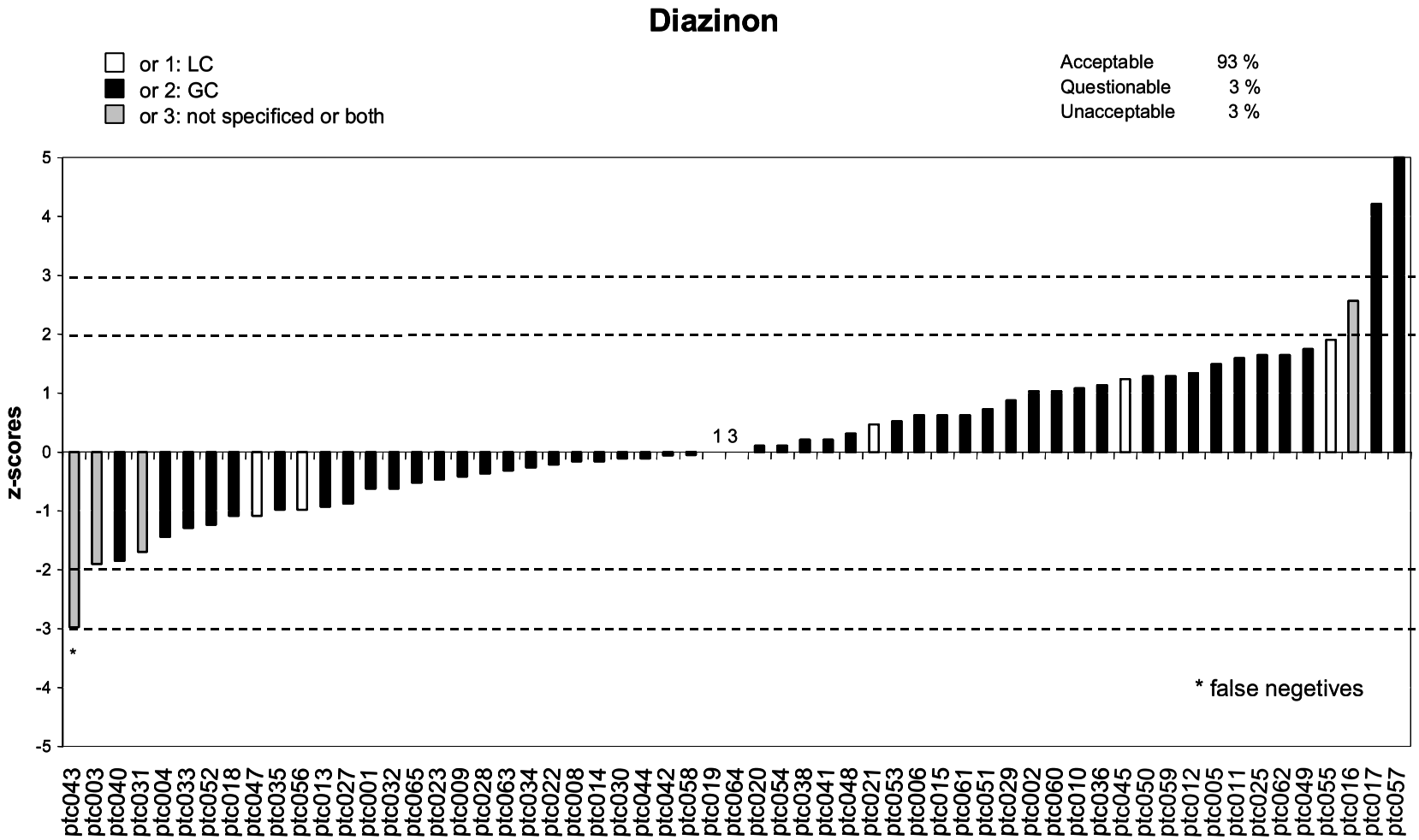


Appendix 3 **Graphical presentation of z-scores for each pesticide**

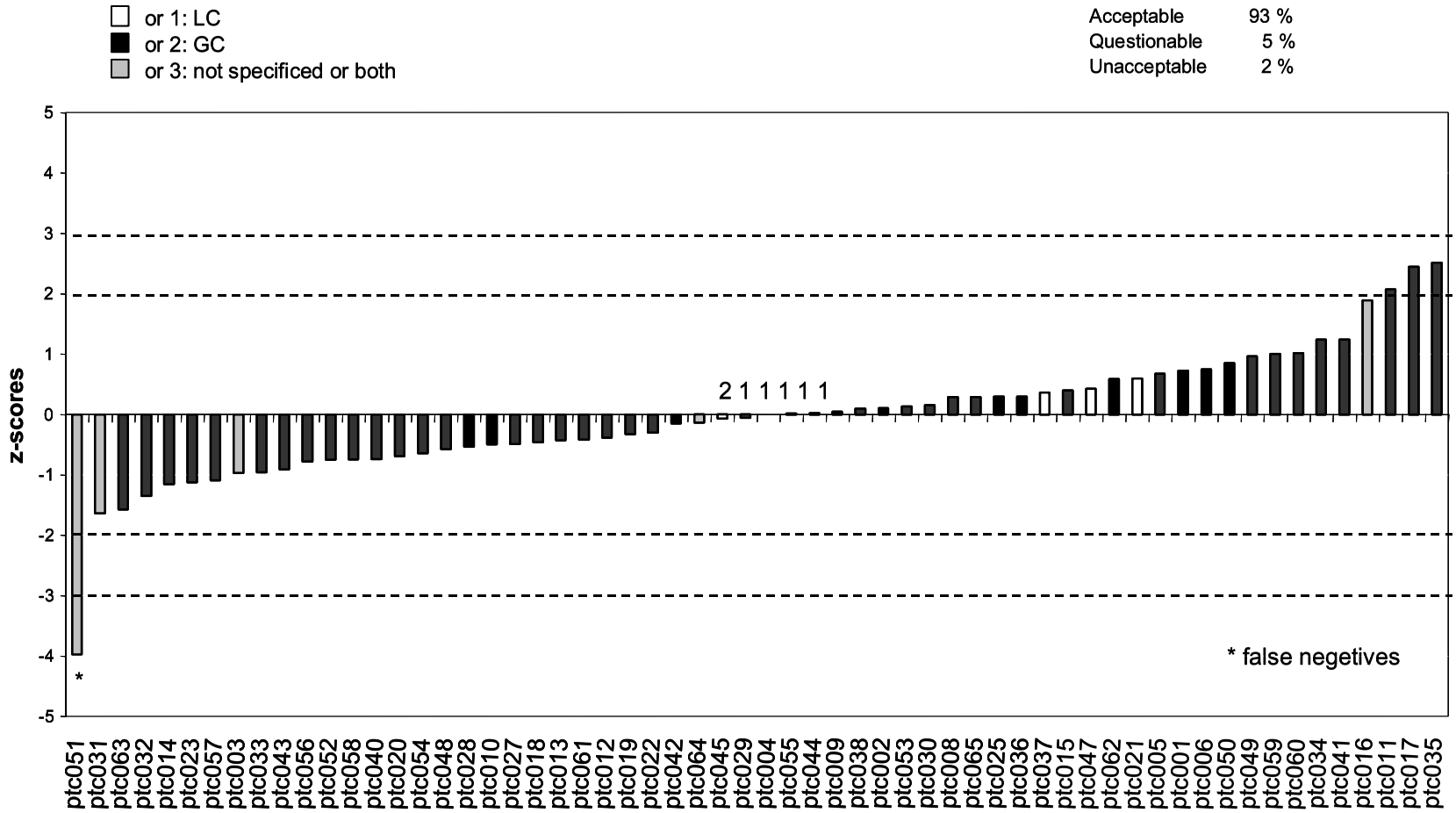


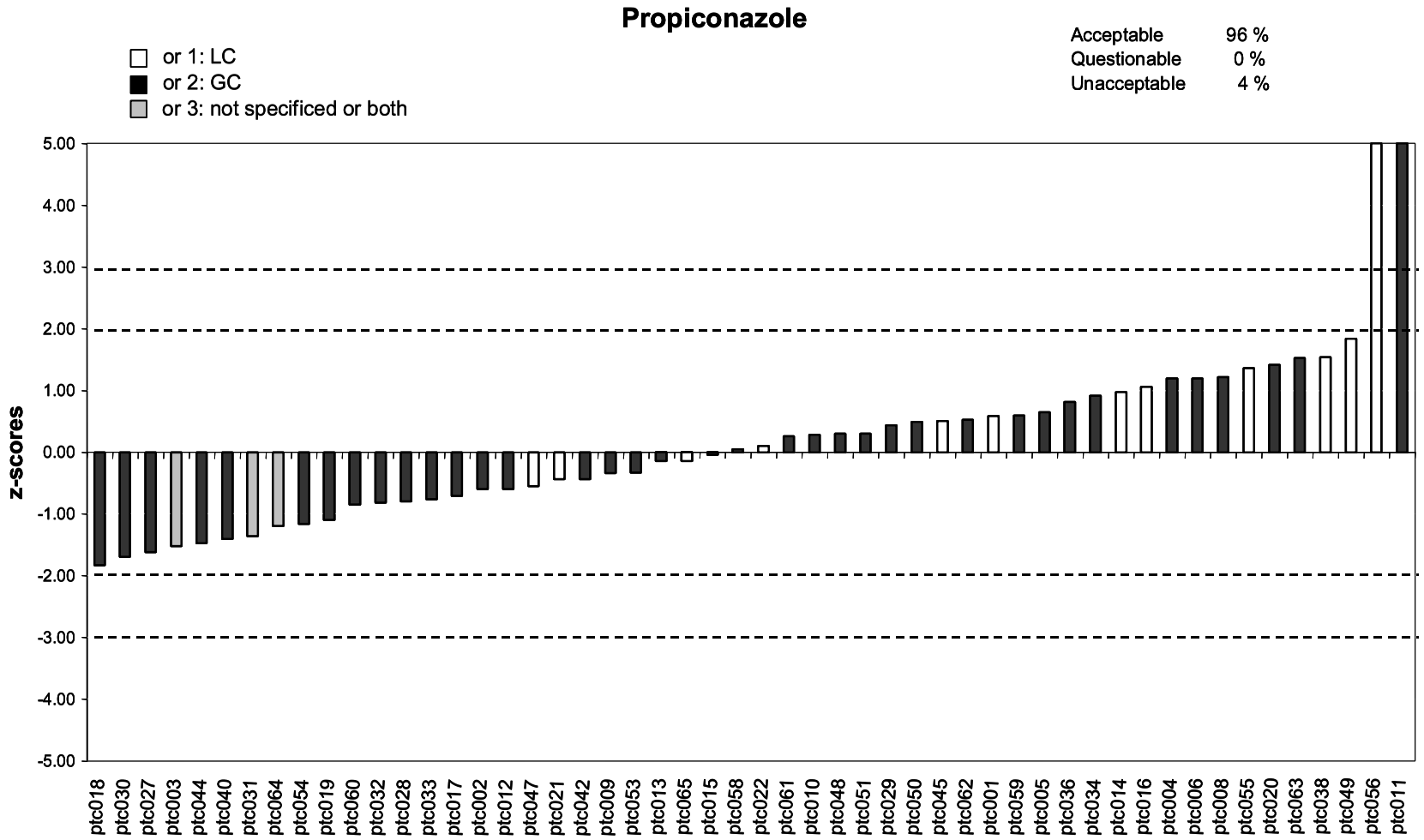

Carbendazim
or 1: LC
or 2: GC
or 3: not specificed or both
Acceptable 87 %
Questionable 3 %
Unacceptable 10 %
z-scores
5.00
4.00
3.00
2.00
1.00
0.00
-1.00
-2.00
-3.00
-4.00
-5.00
* false negetives
ptc012
ptc041
ptc003
ptc015
ptc040
ptc050
ptc064
ptc006
ptc036
ptc047
ptc005
ptc053
ptc022
ptc038
ptc021
ptc034
ptc001
ptc061
ptc062
ptc045
ptc002
ptc013
ptc048
ptc004
ptc065
ptc056
ptc049
ptc042
ptc052
ptc010
ptc028
ptc014
ptc008
ptc037
ptc033
ptc055
ptc009
ptc011
ptc016

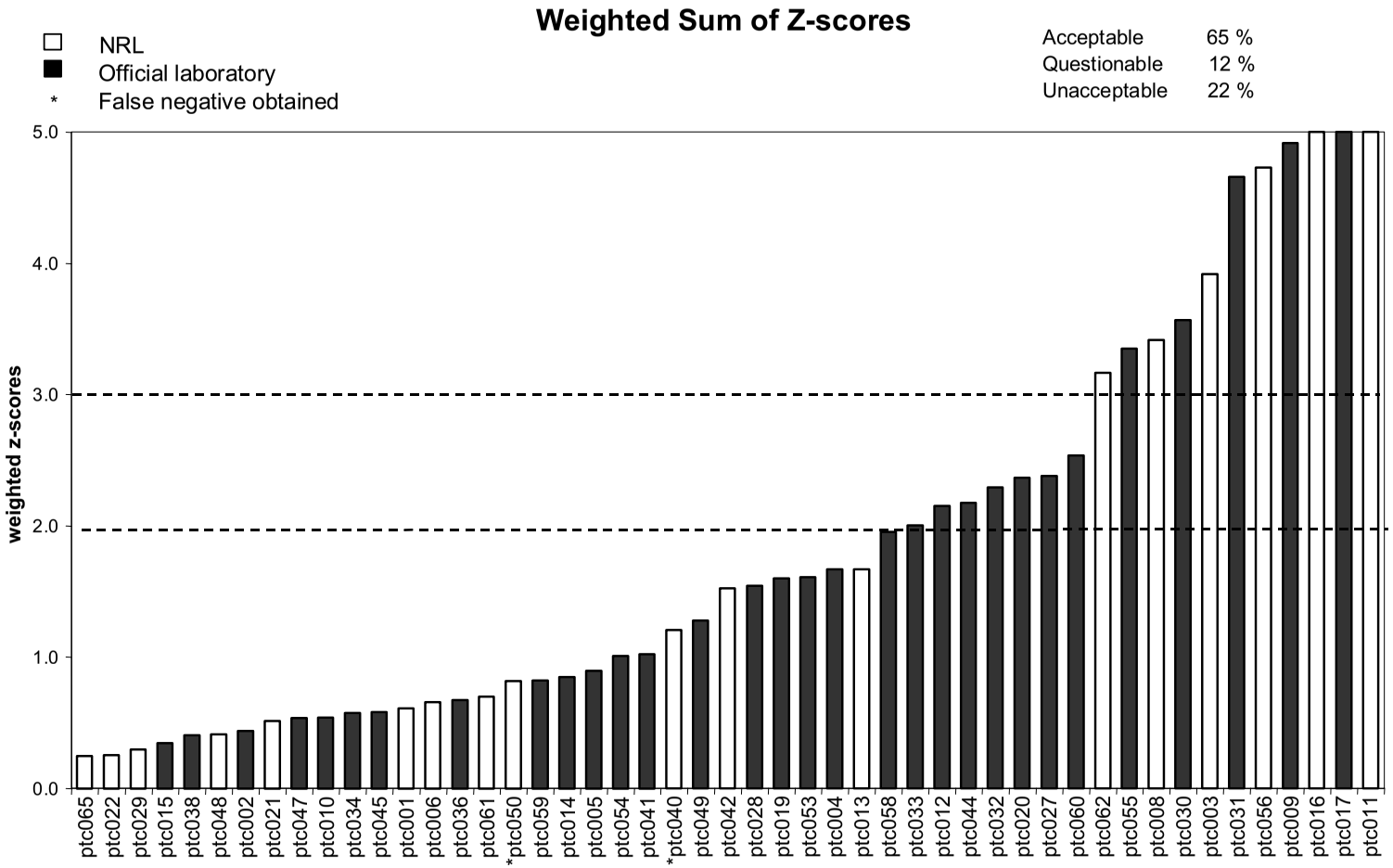

Deltamethrin
or 1: LC
or 2: GC
or 3: not specificed or both
Acceptable 93%
Questionable 3 %
Unacceptable 3 %
z-scores
5
4
3
2
1
0
-1
-2
-3
-4
-5
* false negetives
*
ptc064
ptc062
ptc028
ptc031
ptc020
ptc054
ptc037
ptc060
ptc043
ptc023
ptc003
ptc036
ptc001
ptc058
ptc033
ptc009
ptc063
ptc052
ptc035
ptc032
ptc013
ptc022
ptc034
ptc006
ptc047
ptc056
ptc015
ptc029
ptc004
ptc038
ptc030
ptc041
ptc053
ptc008
ptc048
ptc019
ptc018
ptc010
ptc012
ptc044
ptc027
ptc016
ptc045
ptc051
ptc057
ptc050
ptc059
ptc005
ptc014
ptc025
ptc040
ptc049
ptc055
ptc021
ptc061
ptc042
ptc011
ptc017


Diazinon
or 1: LC
or 2: GC
or 3: not specificed or both
Acceptable 93 %
Questionable 3 %
Unacceptable 3 %
z-scores
5
4
3
2
1
0
-1
-2
-3
-4
-5
1 3
*
* false negetives
ptc043 ptc003 ptc040 ptc031 ptc004 ptc033 ptc052 ptc018 ptc047 ptc035 ptc056 ptc013 ptc027 ptc001 ptc032 ptc065 ptc023 ptc009 ptc028 ptc063 ptc034 ptc022 ptc008 ptc014 ptc030 ptc044 ptc042 ptc058 ptc019 ptc064 ptc020 ptc054 ptc038 ptc041 ptc048 ptc021 ptc053 ptc006 ptc015 ptc061 ptc051 ptc029 ptc002 ptc060 ptc010 ptc036 ptc045 ptc050 ptc059 ptc012 ptc005 ptc011 ptc025 ptc062 ptc049 ptc055 ptc016 ptc017 ptc057

Pirimiphos-methyl




Propiconazole
Acceptable 96 %
Questionable 0 %
Unacceptable 4 %
or 1: LC
or 2: GC
or 3: not specificed or both
z-scores
5.00
4.00
3.00
2.00
1.00
0.00
-1.00
-2.00
-3.00
-4.00
-5.00
ptc018 ptc030 ptc027 ptc003 ptc044 ptc040 ptc031 ptc064 ptc054 ptc019 ptc060 ptc032 ptc028 ptc033 ptc017 ptc002 ptc012 ptc047 ptc021 ptc042 ptc009 ptc053 ptc013 ptc065 ptc015 ptc058 ptc022 ptc061 ptc010 ptc048 ptc051 ptc029 ptc050 ptc045 ptc062 ptc001 ptc059 ptc005 ptc036 ptc034 ptc014 ptc016 ptc004 ptc006 ptc008 ptc055 ptc020 ptc063 ptc038 ptc049 ptc056 ptc011


Weighted Sum of Z-scores
NRL
Official laboratory
* False negative obtained
Acceptable 65 %
Questionable 12 %
Unacceptable 22 %
weighted z-scores
5.0
4.0
3.0
2.0
1.0
0.0
ptc065 ptc022 ptc029 ptc015 ptc038 ptc048 ptc002 ptc021 ptc047 ptc010 ptc034 ptc045 ptc001 ptc006 ptc036 ptc061 *ptc050 ptc059 ptc014 ptc005 ptc054 ptc041 *ptc040 ptc049 ptc042 ptc028 ptc019 ptc053 ptc004 ptc013 ptc058 ptc033 ptc012 ptc044 ptc032 ptc020 ptc027 ptc060 ptc062 ptc055 ptc008 ptc030 ptc003 ptc031 ptc056 ptc009 ptc016 ptc017 ptc011

Appendix 4 MRM-Methods of the participating Laboratories

Participants	Pesticide	Accredited	Reporting level	Reference method	Sample weight	Extraction solvent 1	Extraction solvent 2	Extraction solvent 3	Water addition	Hydrolysis	pH adjusted	Clean up	Calibration	GC detector	HPLC detector	Confirmation
ptc001	Azoxystrobin	No	0.03	Anastassiades JAOAC 86 (2003)	5	Acetonitrile			Yes	No	Yes	DSPE	MM-SL	NPD		GC/MS
	Carbendazim	No	0.01	Anastassiades JAOAC 86 (2003)	5	Acetonitrile			Yes	No	Yes	DSPE	MM-ML		MS/MS	LC/MS/MS
	Deltamethrin	No	0.05	Anastassiades JAOAC 86 (2003)	5	Acetonitrile			Yes	No	Yes	DSPE	MM-SL	ECD		GC/MS
	Diazinon	No	0.02	Anastassiades JAOAC 86 (2003)	5	Acetonitrile			Yes	No	Yes	DSPE	MM-SL	NPD		GC/MS
	Pirimiphos-methyl	No	0.05	Anastassiades JAOAC 86 (2003)	5	Acetonitrile			Yes	No	Yes	DSPE	PS-SL	NPD		GC/MS
	Propiconazole	No	0.01	Anastassiades JAOAC 86 (2003)	5	Acetonitrile			Yes	No	Yes	DSPE	MM-ML		MS/MS	LC/MS/MS
ptc002	Azoxystrobin	Yes	0.02	M. Anastassiades et al. JAOAC 86 (2003)	5	Acetonitrile			Yes	No	pH 5	DSPE	MM-SL	MSD		None
	Carbendazim	Yes	0.01	M. Anastassiades et al. JAOAC 86 (2003)	5	Acetonitrile			Yes	No	pH 5	DSPE	PS-SL		DAD	None
	Diazinon	Yes	0.01	M. Anastassiades et al. JAOAC 86 (2003)	5	Acetonitrile			Yes	No	pH 5	DSPE	MM-SL	MSD		None
	Pirimiphos-methyl	Yes	0.005	M. Anastassiades et al. JAOAC 86 (2003)	5	Acetonitrile			Yes	No	pH 5	DSPE	MM-SL	MSD		None
	Propiconazole	Yes	0.01	M. Anastassiades et al. JAOAC 86 (2003)	5	Acetonitrile			Yes	No	pH 5	DSPE	MM-SL	MSD		None
ptc003	Azoxystrobin	Yes	0.02	EN 12393-1,2,3:1998	25	Ethyl acetate			No	No	No	GPC	MM-ML	NPD	MS/MS	GC/MS
	Carbendazim	No	0.05	EN 12393-1,2,3:1998	10	Ethyl acetate			Yes	No	pH 9	None	PS-ML		MS/MS	LC/MS/MS
	Deltamethrin	Yes	0.02	EN 12393-1,2,3:1998	25	Ethyl acetate			No	No	No	GPC	MM-ML	NPD	MS/MS	GC/MS
	Diazinon	Yes	0.02	EN 12393-1,2,3:1998	25	Ethyl acetate			No	No	No	GPC	MM-ML	NPD	MS/MS	GC/MS
	Pirimiphos-methyl	Yes	0.02	EN 12393-1,2,3:1998	25	Ethyl acetate			No	No	No	GPC	MM-ML	NPD	MS/MS	GC/MS
	Propiconazole	Yes	0.02	EN 12393-1,2,3:1998	25	Ethyl acetate			No	No	No	GPC	MM-ML	NPD	MS/MS	GC/MS

Participants	Pesticide	Accredited	Reporting level	Reference method	Sample weight	Extraction solvent 1	Extraction solvent 2	Extraction solvent 3	Water addition	Hydrolysis	pH adjusted	Clean up	Calibration	GC detector	HPLC detector	Confirmation
ptc004	Azoxystrobin	Yes	0.05		40	Dichloro-methane			No	No	No	GPC	PS-ML	MSD		GC/MS
	Deltamethrin	Yes	0.05		40	Dichloromethane			No	No	No	GPC	PS-ML	MSD		GC/MS
	Diazinon	Yes	0.05		40	Dichloromethane			No	No	No	GPC	PS-ML	MSD		GC/MS
	Pirimiphos-methyl	Yes	0.05		40	Dichloromethane			No	No	No	GPC	PS-ML	MSD		GC/MS
	Propiconazole	Yes	0.05		40	Dichloromethane			No	No	No	GPC	PS-ML	MSD		GC/MS
	Carbendazim	Yes	0.04		20	Ethyl acetate			No	No	Yes	liq./liq part.	PS-ML		DAD	LC/MS
ptc005	Carbendazim	No	0.01	Annastasiades et al.	5	Acetonitrile			Yes	No	No	None	MM-ML		MS/MS	
	Azoxystrobin	No	0.02	In-house method	5	Acetonitrile			Yes	No	No	SPE	MM-ML	MSD		
	Deltamethrin	No	0.02	In-house method	5	Acetonitrile			Yes	No	No	SPE	MM-ML	MSD		
	Diazinon	No	0.02	In-house method	5	Acetonitrile			Yes	No	No	SPE	MM-ML	MSD		
	Pirimiphos-methyl	No	0.02	In-house method	5	Acetonitrile			Yes	No	No	SPE	MM-ML	MSD		
	Propiconazole	No	0.02	In-house method	5	Acetonitrile			Yes	No	No	SPE	MM-ML	MSD		
ptc006	Azoxystrobin	Yes	0.05	AOAC 86 (2003) 412-431	5	Acetonitrile			Yes		Yes	DSPE	MM-SL	ECD		GC/MS
	Carbendazim	Yes	0.05	AOAC 86 (2003) 412-431	5	Acetonitrile			Yes	Yes	Yes	DSPE	MM-SL		MS/MS	LC/MS/MS
	Deltamethrin	Yes	0.05	AOAC 86 (2003) 412-431	5	Acetonitrile			Yes		Yes	DSPE	MM-SL	ECD		GC/MS
	Diazinon	Yes	0.02	AOAC 86 (2003) 412-431	5	Acetonitrile			Yes		Yes	DSPE	MM-SL	ECD		GC/MS
	Pirimiphos-methyl	Yes	0.05	AOAC 86 (2003) 412-431	5	Acetonitrile			Yes		Yes	DSPE	MM-SL	MSD		GC/MS
	Propiconazole	Yes	0.05	AOAC 86 (2003) 412-431	5	Acetonitrile			Yes		Yes	DSPE	MM-SL	ECD		GC/MS
ptc008	Azoxystrobin	No		Dutch method	15	Acetone	Dichloromethane	Other	Yes	No	No	None	MM-ML	NPD		GC/MS
	Carbendazim	No	0.05	Dutch method	10	Acetone	Dichloromethane	Other	Yes	No	No	None	MM-ML		MS/MS	LC/MS/MS
	Deltamethrin	Yes	0.05	Dutch method	15	Acetone	Dichloromethane	Other	Yes	No	No	None	MM-ML	ECD		GC/MS
	Diazinon	Yes	0.02	Dutch method	15	Acetone	Dichloromethane	Other	Yes	No	No	None	MM-ML	NPD		GC/MS

Participants	Pesticide	Accredited	Reporting level	Reference method	Sample weight	Extraction solvent 1	Extraction solvent 2	Extraction solvent 3	Water addition	Hydrolysis	pH adjusted	Clean up	Calibration	GC detector	HPLC detector	Confirmation
ptc008 cont.	Pirimiphos-methyl	No	0.05	Dutch method	15	Acetone	Dichloromethane	Other	Yes	No	No	None	MM-ML	NPD		GC/MS
	Propiconazole	No	0.05	Dutch method	15	Acetone	Dichloromethane	Other	Yes	No	No	None	MM-ML	NPD		GC/MS
ptc009	Carbendazim	Yes	0.02	EN 14333-1	5	Acetone	Dichloromethane		Yes	No	No	SPE	PS-ML		MS/MS	None
	Azoxystrobin	No	0.01	Internal method	4	Other			No	No	No	SPE	PS-ML		MS/MS	None
	Deltamethrin	No	0.01	Internal method	4	Other			No	No	No	SPE	PS-ML	ECD		GC/MS
	Diazinon	No	0.01	Internal method	4	Other			No	No	No	SPE	PS-ML	FPD		GC/MS
	Pirimiphos-methyl	No	0.01	Internal method	4	Other			No	No	No	SPE	PS-ML	FPD		GC/MS
	Propiconazole	No	0.01	Internal method	4	Other			No	No	No	SPE	PS-ML	NPD		GC/MS
ptc010	Deltamethrin	Yes	0.02	§64 L00.00-34	50	Acetone	Ethyl acetate		Yes			GPC	PS-SL	ECD		GC/MS
	Diazinon	Yes	0.02	§64 L00.00-34	50	Acetone	Ethyl acetate		Yes			GPC	PS-SL	ECD		GC/MS
	Pirimiphos-methyl	Yes	0.02	§64 L00.00-34	50	Acetone	Ethyl acetate		Yes			GPC	MM-ML	MSD		
	Propiconazole	Yes	0.02	§64 L00.00-34	50	Acetone	Ethyl acetate		Yes			GPC	MM-ML	MSD		
	Azoxystrobin	Yes	0.02	Housemethod	50	Acetone	Ethyl acetate		Yes			None	MM-ML		MS/MS	
	Carbendazim	Yes	0.02	Housemethod	50	Acetone	Ethyl acetate		Yes			None	MM-ML		MS/MS	
ptc011	Carbendazim	Yes	0.02		10	Acetone	Dichloromethane	Other	No	No	Yes	SPE	PS-ML		Fluor.	
	Azoxystrobin	Yes	0.02		25	Ethyl acetate	Cyclo-hexane	Acetone	No	No	No	None	PS-ML	MS/MS		GC/MS/MS
	Deltamethrin	Yes	0.03		25	Ethyl acetate	Cyclohexane	Acetone	No	No	No	None	PS-ML	MS/MS		GC/MS/MS
	Diazinon	Yes	0.01		25	Ethyl acetate	Cyclohexane	Acetone	No	No	No	None	PS-ML	MS/MS		GC/MS/MS
	Pirimiphos-methyl	Yes	0.01		25	Ethyl acetate	Cyclohexane	Acetone	No	No	No	None	PS-ML	MS/MS		GC/MS/MS
	Propiconazole	Yes	0.02		25	Ethyl acetate	Cyclohexane	Acetone	No	No	No	None	PS-ML	MS/MS		GC/MS/MS
ptc012	Azoxystrobin	Yes	0.01	Internal method	10	Dichloromethane	Other		No	No	No	None	MM-ML		MS/MS	LC/MS/MS
	Carbendazim	Yes	0.01	Internal method	10	Dichloromethane	Other		No	No	No	None	MM-ML		MS/MS	LC/MS/MS
	Deltamethrin	Yes	0.05	Internal method	10	Dichloromethane	Other		No	No	No	GPC	PS-SL	NPD		GC/MS
	Diazinon	Yes	0.02	Internal method	10	Dichloromethane	Other		No	No	No	GPC	PS-SL	NPD		GC/MS

Participants	Pesticide	Accredited	Reporting level	Reference method	Sample weight	Extraction solvent 1	Extraction solvent 2	Extraction solvent 3	Water addition	Hydrolysis	pH adjusted	Clean up	Calibration	GC detector	HPLC detector	Confirmation
ptc012 cont.	Pirimiphos-methyl	Yes	0.02	Internal method	10	Dichloromethane	Other		No	No	No	GPC	PS-SL	NPD		GC/MS
	Propiconazole	Yes	0.05	Internal method	10	Dichloromethane	Other		No	No	No	GPC	PS-ML	NPD		GC/MS
ptc013	Deltamethrin	Yes	0.05		20	Ethyl acetate			Yes	No	No	GPC	MM-ML	MSD		
	Diazinon	Yes	0.02		20	Ethyl acetate			Yes	No	No	GPC	MM-ML	MSD		
	Pirimiphos-methyl	Yes	0.02		20	Ethyl acetate			Yes	No	No	GPC	MM-ML	MSD		
	Propiconazole	Yes	0.02		20	Ethyl acetate			Yes	No	No	GPC	MM-ML	MSD		
	Azoxystrobin	No	0.01	Leothay, S. et al. JAOAC 88 (2005)	15	Acetonitrile			Yes	No	Yes	Other	MM-ML		MS/MS	
	Carbendazim	No	0.01	Leothay, S. et al. JAOAC 88 (2005)	15	Acetonitrile			Yes	No	Yes	Other	MM-ML		MS/MS	
ptc014	Azoxystrobin	Yes	0.01		5	Acetonitrile			Yes	No	No	None	MM-ML		MS/MS	LC/MS/MS
	Carbendazim	Yes	0.01		5	Acetonitrile			Yes	No	No	None	MM-ML		MS/MS	LC/MS/MS
	Propiconazole	Yes	0.01		5	Acetonitrile			Yes	No	No	None	MM-ML		MS/MS	LC/MS/MS
	Deltamethrin	Yes	0.01		12	Ethyl acetate			No	No	No	GPC	MM-ML	MSD		GC/MS
	Diazinon	Yes	0.01		12	Ethyl acetate			No	No	No	GPC	MM-ML	MSD		GC/MS
	Pirimiphos-methyl	Yes	0.01		12	Ethyl acetate			No	No	No	GPC	MM-ML	MSD		GC/MS
ptc015	Carbendazim	Yes	0.04	inhouse validated method	25	Acetone	Ethyl acetate	Dichloromethane	Yes	No	pH 7	liq./liq part.	MM-ML		DAD	None
	Azoxystrobin	Yes	0.01	Modulare Multimethode gem 64 LFGB (S 19)	25	Acetone	Cyclohexane	Ethyl acetate	Yes	No	No	GPC	MM-ML	ECD		GC/MS/MS
	Deltamethrin	Yes	0.005	Modulare Multimethode gemÃ¤ÃŸ Â§ 64 LFGB	25	Acetone	Cyclohexane	Ethyl acetate	Yes	No	No	GPC	MM-ML	ECD		GC/MS/MS
	Diazinon	Yes	0.005	Modulare Multimethode gemÃ¤ÃŸ Â§ 64 LFGB	25	Acetone	Cyclohexane	Ethyl acetate	Yes	No	No	GPC	MM-ML	NPD		GC/MS/MS
	Pirimiphos-methyl	Yes	0.005	Modulare Multimethode gemÃ¤ÃŸ Â§ 64 LFGB	25	Acetone	Cyclohexane	Ethyl acetate	Yes	No	No	GPC	MM-ML	NPD		GC/MS/MS
	Propiconazole	Yes	0.01	Modulare Multimethode gemÃ¤ÃŸ Â§ 64 LFGB	25	Acetone	Cyclohexane	Ethyl acetate	Yes	No	No	GPC	MM-ML	ECD		GC/MS/MS
ptc016	Deltamethrin	Yes	0.05	Inernal method	25	Acetone	Cyclohexane	Ethyl acetate	Yes	No	pH 7	GPC	MM-ML	ECD		GC/MS

Participants	Pesticide	Accredited	Reporting level	Reference method	Sample weight	Extraction solvent 1	Extraction solvent 2	Extraction solvent 3	Water addition	Hydrolysis	pH adjusted	Clean up	Calibration	GC detector	HPLC detector	Confirmation
ptc016 cont.	Carbendazim	Yes	0.01	Inetrnal method	5	Methanol			Yes	No	No	None	MM-ML		MS/MS	LC/MS/MS
	Propiconazole	Yes	0.05	Inetrnal method	5	Methanol			Yes	No	No	None	MM-ML		MS/MS	LC/MS/MS
	Azoxystrobin	Yes	0.05	Internal method	25	Acetone	Cyclohexane	Ethyl acetate	Yes	No	pH 7	GPC	MM-ML	ECD	MS/MS	GC/MS
	Diazinon	Yes	0.02	Internal method	25	Acetone	Cyclohexane	Ethyl acetate	Yes	No	pH 7	GPC	MM-ML	ECD	MS/MS	GC/MS
	Pirimiphos-methyl	Yes	0.05	Internal method	25	Acetone	Cyclohexane	Ethyl acetate	Yes	No	pH 7	GPC	MM-ML	ECD	MS/MS	GC/MS
ptc017	Azoxystrobin	No	0.05	internal method	20	Acetone			Yes	No	No	SPE	MM-ML	MSD		GC/MS
	Deltamethrin	No	0.1	internal method	20	Acetone			Yes	No	No	SPE	MM-ML	MSD		GC/MS
	Diazinon	No	0.05	internal method	20	Acetone			Yes	No	No	SPE	MM-ML	MSD		GC/MS
	Pirimiphos-methyl	No	0.05	internal method	20	Acetone			Yes	No	No	SPE	MM-ML	MSD		GC/MS
	Propiconazole	No	0.05	internal method	20	Acetone			Yes	No	No	SPE	MM-ML	MSD		GC/MS
ptc018	Deltamethrin	No	0.02	internal method	5	Acetone	Dichloromethane		No	No	No	SPE	PS-ML	ECD		None
	Diazinon	No	0.02	internal method	5	Acetone	Dichloromethane		No	No	No	SPE	PS-ML	NPD		Two col.
	Pirimiphos-methyl	No	0.02	internal method	5	Acetone	Dichloromethane		No	No	No	SPE	PS-ML	NPD		Two col.
	Propiconazole	No	0.02	internal method	5	Acetone	Dichloromethane		No	No	No	SPE	PS-ML	NPD		Two col.
ptc019	Azoxystrobin	No	0.05	A	10	Acetone	Other	Other	Yes	No	No	Other	MM-SL	ECD		Two col.
	Deltamethrin	No	0.05	A	10	Acetone	Other	Other	Yes	No	No	Other	MM-SL	ECD		Two col.
	Diazinon	No	0.02	A	10	Acetone	Other	Other	Yes	No	No	Other	MM-SL	NPD		Two col.
	Pirimiphos-methyl	No	0.02	A	10	Acetone	Other	Other	Yes	No	No	Other	MM-SL	NPD		Two col.
	Propiconazole	No	0.05	A	10	Acetone	Other	Other	Yes	No	No	Other	MM-SL	ECD		Two col.
ptc020	Azoxystrobin	No	0.05	internal method	10	Acetonitrile			No	No	No	None	PS-SL	ECD		GC/MS
	Deltamethrin	No	0.05	internal method	10	Acetonitrile			No	No	No	None	PS-SL	ECD		GC/MS
	Diazinon	No	0.02	internal method	10	Acetonitrile			No	No	No	None	PS-SL	NPD		GC/MS
	Pirimiphos-methyl	No	0.05	internal method	10	Acetonitrile			No	No	No	None	PS-SL	NPD		GC/MS

Participants	Pesticide	Accredited	Reporting level	Reference method	Sample weight	Extraction solvent 1	Extraction solvent 2	Extraction solvent 3	Water addition	Hydrolysis	pH adjusted	Clean up	Calibration	GC detector	HPLC detector	Confirmation
ptc020 cont.	Propiconazole	No	0.05	internal method	10	Acetonitrile			No	No	No	None	PS-SL	ECD		GC/MS
ptc021	Azoxystrobin	No	0.01		3	Acetonitrile			Yes	No	No	liq./liq part.	PS-ML		MS/MS	
	Carbendazim	No	0.01		3	Acetonitrile			Yes	No	No	liq./liq part.	PS-ML		MS/MS	
	Diazinon	No	0.01		3	Acetonitrile			Yes	No	No	liq./liq part.	PS-ML		MS/MS	
	Pirimiphos-methyl	No	0.05		3	Acetonitrile			Yes	No	No	liq./liq part.	PS-ML		MS/MS	
	Propiconazole	No	0.02		3	Acetonitrile			Yes	No	No	liq./liq part.	PS-ML		MS/MS	
	Deltamethrin	No	0.05		10	Ethyl acetate			No	No	No	GPC	MM-SL	TOF		
ptc022	Deltamethrin	Yes	0.05	Anal. meth for pesticide Res. in Foodst., Min.of Health (AMPRF) The Haque,1996	2	Acetone	Dichloromethane	Other	Yes	No	No	None	MM-ML	ECD		GC/MS
	Diazinon	Yes	0.02	Anal. meth for pesticide Res. in Foodst., Min.of Health (AMPRF) The Haque,1996	2	Acetone	Dichloromethane	Other	Yes	No	No	None	MM-ML	ITD		GC/MS
	Pirimiphos-methyl	Yes	0.05	Anal. meth for pesticide Res. in Foodst., Min.of Health (AMPRF) The Haque,1996	2	Acetone	Dichloromethane	Other	Yes	No	No	None	MM-ML	ITD		GC/MS
	Azoxystrobin	Yes	0.02	J. Chromatogr. A, 1154(2007)3-25	2	Acetone	Dichloromethane	Other	Yes	No	No	None	MM-ML		MS/MS	LC/MS/MS
	Carbendazim	Yes	0.02	J. Chromatogr. A, 1154(2007)3-25	2	Acetone	Dichloromethane	Other	Yes	No	No	None	MM-ML		MS/MS	LC/MS/MS
	Propiconazole	Yes	0.02	J. Chromatogr. A, 1154(2007)3-25	2	Acetone	Dichloromethane	Other	Yes	No	No	None	MM-ML		MS/MS	LC/MS/MS
ptc023	Deltamethrin	Yes	0.05	internal method	50	Ethyl acetate						GPC	PS-ML	MSD		GC/MS
	Diazinon	Yes	0.02	internal method	50	Ethyl acetate						GPC	PS-ML	MSD		GC/MS
	Pirimiphos-methyl	Yes	0.05	internal method	50	Ethyl acetate						GPC	PS-ML	MSD		GC/MS
ptc025	Azoxystrobin	Yes	0.05	DFG S19	0	Acetone	Cyclohexane	Ethyl acetate	Yes	No	No	GPC	MM-ML	MSD		GC/MS
	Deltamethrin	No	0.05	DFG S19	0	Acetone	Cyclohexane	Ethyl acetate	Yes	No	No	GPC	MM-ML	MSD		GC/MS

Participants	Pesticide	Accredited	Reporting level	Reference method	Sample weight	Extraction solvent 1	Extraction solvent 2	Extraction solvent 3	Water addition	Hydrolysis	pH adjusted	Clean up	Calibration	GC detector	HPLC detector	Confirmation
ptc025 cont.	Diazinon	Yes	0.05	DFG S19	0	Acetone	Cyclohexane	Ethyl acetate	Yes	No	No	GPC	MM-ML	MSD		GC/MS
	Pirimiphos-methyl	Yes	0.05	DFG S19	0	Acetone	Cyclohexane	Ethyl acetate	Yes	No	No	GPC	MM-ML	MSD		GC/MS
ptc027	Azoxystrobin	Yes	0.008	FP004	500	Ethyl acetate			No	No	No	GPC	MM-ML	ECD		Two col.
	Deltamethrin	Yes	0.008	FP004	500	Ethyl acetate			No	No	No	GPC	MM-ML	ECD		Two col.
	Diazinon	Yes	0.008	FP004	500	Ethyl acetate			No	No	No	GPC	MM-ML	ECD		Two col.
	Pirimiphos-methyl	Yes	0.042	FP004	500	Ethyl acetate			No	No	No	GPC	MM-ML	ECD		Two col.
	Propiconazole	Yes	0.008	FP004	500	Ethyl acetate			No	No	No	GPC	MM-ML	ECD		Two col.
ptc028	Deltamethrin	No	0.02		20	Acetone			Yes	No	No	liq./liq part.	MM-ML	ECD		GC/MS
	Diazinon	No	0.02		20	Acetone			Yes	No	No	liq./liq part.	MM-ML	ECD		GC/MS
	Pirimiphos-methyl	No	0.02		20	Acetone			Yes	No	No	liq./liq part.	MM-ML	ECD		GC/MS
	Propiconazole	No	0.02		20	Acetone			Yes	No	No	liq./liq part.	MM-ML	ECD		GC/MS
	Azoxystrobin	No	0.01		5	Methanol			Yes	No	Yes	Freezing out	MM-ML		MS/MS	LC/MS/MS
	Carbendazim	No	0.01		5	Methanol			Yes	No	Yes	Freezing out	MM-ML		MS/MS	LC/MS/MS
ptc029	Azoxystrobin	No	0.05	EN-12393	10	Acetone	Dichloromethane		Yes	No	No	SPE	MM-ML	ECD		GC/MS/MS
	Deltamethrin	No	0.02	EN-12393	10	Acetone	Dichloromethane		Yes	No	No	SPE	MM-ML	ECD		GC/MS/MS
	Diazinon	No	0.01	EN-12393	10	Acetone	Dichloromethane		Yes	No	No	SPE	MM-ML	NPD		GC/MS/MS
	Pirimiphos-methyl	No	0.01	EN-12393	10	Acetone	Dichloromethane		Yes	No	No	SPE	MM-ML	NPD		GC/MS/MS
	Propiconazole	No	0.05	EN-12393	10	Acetone	Dichloromethane		Yes	No	No	SPE	MM-ML	ECD		GC/MS/MS
ptc030	Azoxystrobin	No	0.05	Luke	20	Acetone	Other		Yes	No	No	Other	MM-SL	ECD		None
	Deltamethrin	No	0.05	Luke	20	Acetone	Other		Yes	No	No	Other	MM-SL	ECD		None
	Diazinon	No	0.02	Luke	20	Acetone	Other		Yes	No	No	Other	MM-SL	ECD		Two col.
	Pirimiphos-methyl	No	0.05	Luke	20	Acetone	Other		Yes	No	No	Other	MM-SL	NPD		Two col.
	Propiconazole	No	0.05	Luke	20	Acetone	Other		Yes	No	No	Other	MM-SL	ECD		Two col.

Participants	Pesticide	Accredited	Reporting level	Reference method	Sample weight	Extraction solvent 1	Extraction solvent 2	Extraction solvent 3	Water addition	Hydrolysis	pH adjusted	Clean up	Calibration	GC detector	HPLC detector	Confirmation
ptc031	Azoxystrobin	No	0.02	Internal	10	Ethyl acetate			No	No	No	GPC	MM-ML	Other	MS/MS	LC/MS/MS
	Deltamethrin	No	0.05	Internal	10	Ethyl acetate			No	No	No	GPC	MM-ML	MSD	Other	GC/MS
	Diazinon	No	0.02	Internal	10	Ethyl acetate			No	No	No	GPC	MM-ML	MSD	Other	GC/MS
	Pirimiphos-methyl	No	0.02	Internal	10	Ethyl acetate			No	No	No	GPC	MM-ML	MSD	Other	GC/MS
	Propiconazole	No	0.02	Internal	10	Ethyl acetate			No	No	No	GPC	MM-ML	Other	MS/MS	LC/MS/MS
ptc032	Azoxystrobin	No	0.01	ISTISAN	10	Ethyl acetate			No	No	No	GPC	PS-SL	ECD		GC/MS
	Deltamethrin	No	0.01	ISTISAN	10	Ethyl acetate			No	No	No	GPC	PS-SL	ECD		GC/MS
	Diazinon	No	0.01	ISTISAN	10	Ethyl acetate			No	No	No	GPC	PS-SL	NPD		GC/MS
	Pirimiphos-methyl	No	0.01	ISTISAN	10	Ethyl acetate			No	No	No	GPC	PS-SL	NPD		GC/MS
	Propiconazole	No	0.01	ISTISAN	10	Ethyl acetate			No	No	No	GPC	PS-SL	ECD		GC/MS
ptc033	Carbendazim	No	0.02	EN 12393 P method	50	Ethyl acetate			Yes	No	Yes	GPC	PS-ML		DAD	
	Azoxystrobin	No	0.05	EN 12393 P method	50	Ethyl acetate			Yes	No	No	GPC	MM-ML	ECD		GC/MS
	Deltamethrin	Yes	0.05	EN 12393 P method	50	Ethyl acetate			Yes	No	No	GPC	MM-ML	ECD		GC/MS
	Diazinon	Yes	0.02	EN 12393 P method	50	Ethyl acetate			Yes			GPC	MM-ML	NPD		GC/MS
	Pirimiphos-methyl	Yes	0.05	EN 12393 P method	50	Ethyl acetate			Yes			GPC	MM-ML	NPD		GC/MS
	Propiconazole	No	0.05	EN 12393 P method	50	Ethyl acetate			Yes			GPC	MM-ML	NPD		GC/MS
ptc034	Azoxystrobin	Yes	0.01	Anastassiades (10.05.2007) incl. optional alkaline hydrolysis	5	Acetonitrile			Yes	No	No	DSPE	MM-ML		MS/MS	LC/MS/MS
	Carbendazim	Yes	0.01	Anastassiades (10.05.2007) incl. optional alkaline hydrolysis	5	Acetonitrile			Yes	No	No	DSPE	MM-ML		MS/MS	LC/MS/MS
	Deltamethrin	Yes	0.02	Anastassiades et al. JAOAC 86 (2003) modified	5	Acetonitrile			Yes	No		DSPE	MM-ML	MSD		GC/MS

Participants	Pesticide	Accredited	Reporting level	Reference method	Sample weight	Extraction solvent 1	Extraction solvent 2	Extraction solvent 3	Water addition	Hydrolysis	pH adjusted	Clean up	Calibration	GC detector	HPLC detector	Confirmation
ptc034 cont.	Diazinon	Yes	0.01	Anastassiades et al. JAOAC 86 (2003) modified	5	Acetonitrile			Yes	No		DSPE	MM-ML	MSD		GC/MS
	Pirimiphos-methyl	Yes	0.01	Anastassiades et al. JAOAC 86 (2003) modified	5	Acetonitrile			Yes	No		DSPE	MM-ML	MSD		GC/MS
	Propiconazole	Yes	0.01	Anastassiades et al. JAOAC 86 (2003) modified	5	Acetonitrile			Yes	No		DSPE	MM-ML	MSD		GC/MS
ptc035	Azoxystrobin	No	0.05	Luke	75	Acetone	Dichloromethane	Other	No	No	No	GPC	PS-ML	ECD		GC/MS
	Deltamethrin	No	0.05	Luke	75	Acetone	Dichloromethane	Other	No	No	No	GPC	PS-ML	ECD		GC/MS
	Diazinon	No	0.05	Luke	75	Acetone	Dichloromethane	Other	No	No	No	GPC	PS-ML	ECD		GC/MS
	Pirimiphos-methyl	No	0.05	Luke	75	Acetone	Dichloromethane	Other	No	No	No	GPC	PS-ML	ECD		GC/MS
ptc036	Azoxystrobin	No	0.05	FILLION	25	Acetonitrile			Yes	No	Yes	SPE	PS-ML	NPD		LC/MS/MS
	Deltamethrin	Yes	0.05	FILLION	25	Acetonitrile			Yes	No	Yes	SPE	PS-ML	ECD		LC/MS/MS
	Diazinon	Yes	0.02	FILLION	25	Acetonitrile			Yes	No	Yes	SPE	PS-ML	ECD		LC/MS/MS
	Pirimiphos-methyl	No	0.05	FILLION	25	Acetonitrile			Yes	No	Yes	SPE	PS-ML	ECD		LC/MS/MS
	Propiconazole	No	0.05	FILLION	25	Acetonitrile			Yes	No	Yes	SPE	PS-ML	ECD		LC/MS/MS
	Carbendazim	No	0.1	INTERNO	13	Acetonitrile			Yes	No	Yes	SPE	PS-ML		MS/MS	
ptc037	Carbendazim	No	0.01	J. Klein and L. Alder; J AOAC International 86, 1015-37 (2003)	5	Methanol	Dichloromethane		Yes	No	No	liq./liq part.	MM-ML		MS/MS	LC/MS/MS
ptc037	Deltamethrin	No	0.01	J. Klein and L. Alder; J AOAC International 86, 1015-37 (2003)	5	Methanol	Dichloromethane		Yes	No	No	liq./liq part.	MM-ML		MS/MS	LC/MS/MS
	Pirimiphos-methyl	No	0.01	J. Klein and L. Alder; J AOAC International 86, 1015-37 (2003)	5	Methanol	Dichloromethane		Yes	No	No	liq./liq part.	MM-ML		MS/MS	LC/MS/MS
ptc038	Azoxystrobin	Yes	0.005	QuEChERS	5	Acetonitrile			Yes	No	pH 5	DSPE	MM-ML		MS/MS	GC/MS
	Carbendazim	Yes	0.005	QuEChERS	5	Acetonitrile			Yes	No	pH 5	DSPE	MM-ML		MS/MS	

Participants	Pesticide	Accredited	Reporting level	Reference method	Sample weight	Extraction solvent 1	Extraction solvent 2	Extraction solvent 3	Water addition	Hydrolysis	pH adjusted	Clean up	Calibration	GC detector	HPLC detector	Confirmation
ptc038 cont.	Deltamethrin	Yes	0.03	QuEChERS	5	Acetonitrile			Yes	No	pH 5	DSPE	MM-ML	MSD		GC/MS
	Diazinon	Yes	0.01	QuEChERS	5	Acetonitrile			Yes	No	pH 5	DSPE	MM-ML	FPD		GC/MS/MS
	Pirimiphos-methyl	Yes	0.01	QuEChERS	5	Acetonitrile			Yes	No	pH 5	DSPE	MM-ML	FPD		GC/MS/MS
	Propiconazole	Yes	0.005	QuEChERS	5	Acetonitrile			Yes	No	pH 5	DSPE	MM-ML		MS/MS	GC/MS
ptc040	Azoxystrobin	Yes	0.04	Anastasiades	5	Acetonitrile			Yes		No	DSPE	MM-ML	ECD		GC/MS/MS
	Propiconazole	Yes	0.01	Anastasiades	5	Acetonitrile			Yes		No	DSPE	MM-ML	ECD		GC/MS/MS
	Deltamethrin	Yes	0.02	EN EN-12393	10	Ethyl acetate			No			GPC	MM-ML	ECD		GC/MS/MS
	Diazinon	Yes	0.01	EN EN-12393	10	Ethyl acetate			No			GPC	MM-ML	ECD		GC/MS/MS
	Pirimiphos-methyl	Yes	0.01	EN EN-12393	10	Ethyl acetate			No			GPC	MM-ML	ECD		GC/MS/MS
ptc041	Azoxystrobin	Yes	0.01	Internal method LMS	10	Acetone	Dichloromethane	Other	No	No	No	None	PS-ML		MS/MS	LC/MS/MS
	Carbendazim	Yes	0.01	Internal method LMS	10	Acetone	Dichloromethane	Other	No	No	No	None	PS-ML		MS/MS	LC/MS/MS
	Diazinon	Yes	0.01	Internal method P2	10	Ethyl acetate	Cyclohexane		No	Yes	No	GPC	PS-ML	FPD		
	Pirimiphos-methyl	Yes	0.01	Internal method P2	10	Ethyl acetate	Cyclohexane		No	Yes	No	GPC	PS-ML	FPD		
	Deltamethrin	Yes	0.01	Internal method PYR2	10	Other			No	Yes	No	GPC	PS-ML	ECD		
ptc042	Azoxystrobin	Yes	0.02	Bottomley and Baker, Analyst, vol 109, pp85-90, 1984	10	Acetone	Methanol		No	No	No	GPC	MM-ML	MSD		GC/MS
	Deltamethrin	Yes	0.02	Bottomley and Baker, Analyst, vol 109, pp85-90, 1984	10	Acetone	Methanol		No	No	No	GPC	MM-ML	MSD		GC/MS
	Diazinon	Yes	0.02	Bottomley and Baker, Analyst, vol 109, pp85-90, 1984	10	Acetone	Methanol		No	No	No	GPC	MM-ML	MSD		GC/MS
	Pirimiphos-methyl	Yes	0.02	Bottomley and Baker, Analyst, vol 109, pp85-90, 1984	10	Acetone	Methanol		No	No	No	GPC	MM-ML	MSD		GC/MS
	Propiconazole	Yes	0.02	Bottomley and Baker, Analyst, vol 109, pp85-90, 1984	10	Acetone	Methanol		No	No	No	GPC	MM-ML	MSD		GC/MS

Participants	Pesticide	Accredited	Reporting level	Reference method	Sample weight	Extraction solvent 1	Extraction solvent 2	Extraction solvent 3	Water addition	Hydrolysis	pH adjusted	Clean up	Calibration	GC detector	HPLC detector	Confirmation
ptc042 cont.	Carbendazim	Yes	0.02	quechers	5	Acetonitrile			Yes	No			MM-ML		MS/MS	LC/MS/MS
ptc043	Deltamethrin	Yes	0.01		10	Acetone	Dichloromethane	Ethyl acetate	Yes	No	No	Other	PS-ML	ECD		GC/MS
	Pirimiphos-methyl	Yes	0.01		50	Acetone	Dichloromethane	Ethyl acetate	Yes	No	No	Other	PS-ML	NPD		GC/MS
ptc044	Azoxystrobin	No	0.05	Luke	20	Acetone	Other		No	No	No	Other	MM-SL	ECD		None
	Deltamethrin	No	0.05	Luke	20	Acetone	Other		No	No	No	Other	MM-SL	ECD		None
	Diazinon	No	0.02	Luke	20	Acetone	Other		No	No	No	Other	MM-SL	ECD		None
	Pirimiphos-methyl	No	0.05	Luke	20	Acetone	Other		No	No	No	Other	MM-SL	NPD		None
	Propiconazole	No	0.05	Luke	20	Acetone	Other		No	No	No	Other	MM-SL	ECD		None
ptc045	Deltamethrin	Yes	0.025	DFG (S19)	10	Other			No	No	No	None	MM-ML	MSD		LC/MS/MS
	Azoxystrobin	Yes	0.020	KLEIN, J., ALDER, L. (2003): Applicability of gradient liquid chroma-tography with tandem mass spectr	5	Methanol			Yes	No	No	None	MM-ML	MSD		LC/MS/MS
	Carbendazim	Yes	0.010	KLEIN, J., ALDER, L. (2003): Applicability of gradient liquid chroma-tography with tandem mass spectr	5	Methanol			Yes	No	No	None	PS-ML		MS/MS	LC/MS/MS
	Diazinon	Yes	0.010	Quetchers	5	Acetonitrile			Yes	No	No	Freezing out	Standard addition		MS/MS	GC/MS
	Pirimiphos-methyl	Yes	0.010	Quetchers	5	Acetonitrile			Yes	No	No	Freezing out	Standard addition		MS/MS	GC/MS
	Propiconazole	Yes	0.015	Quetchers	5	Acetonitrile			Yes	No	No	Freezing out	Standard addition		MS/MS	GC/MS
ptc047	Deltamethrin	Yes	0.01	Â§64 LFBG	10	Acetonitrile	Acetone		No	No	No	GPC	MM-ML	MSD		GC/MS
	Azoxystrobin	Yes	0.01	QUECHERS	5	Acetonitrile			Yes	No	No	DSPE	MM-ML		MS/MS	LC/MS/MS
	Carbendazim	Yes	0.01	QUECHERS	5	Acetonitrile			Yes	No	No	DSPE	MM-ML		MS/MS	LC/MS/MS
	Diazinon	Yes	0.01	QUECHERS	5	Acetonitrile			Yes	No	No	DSPE	MM-ML		MS/MS	LC/MS/MS
	Pirimiphos-methyl	Yes	0.01	QUECHERS	5	Acetonitrile			Yes	No	No	DSPE	MM-ML		MS/MS	LC/MS/MS

Participants	Pesticide	Accredited	Reporting level	Reference method	Sample weight	Extraction solvent 1	Extraction solvent 2	Extraction solvent 3	Water addition	Hydrolysis	pH adjusted	Clean up	Calibration	GC detector	HPLC detector	Confirmation
ptc047 cont.	Propiconazole	Yes	0.01	QUECHERS	5	Acetonitrile			Yes	No	No	DSPE	MM-ML		MS/MS	LC/MS/MS
ptc048	Azoxystrobin	No	0.037	QuChERS; TC 275 WI 00275	5	Acetonitrile			Yes	No	Yes	None	MM-ML		MS/MS	
	Carbendazim	No	0.037	QuChERS; TC 275 WI 00275	5	Acetonitrile			Yes	No	Yes	None	MM-ML		MS/MS	
	Deltamethrin	Yes	0.005	QuChERS; TC 275 WI 00275	5	Acetonitrile			Yes	No	Yes	DSPE	MM-ML	MSD		GC/MS/MS
	Diazinon	Yes	0.001	QuChERS; TC 275 WI 00275	5	Acetonitrile			Yes	No	Yes	DSPE	MM-ML	MSD		GC/MS/MS
ptc048	Pirimiphos-methyl	Yes	0.001	QuChERS; TC 275 WI 00275	5	Acetonitrile			Yes	No	Yes	DSPE	MM-ML	MSD		LC/MS/MS
	Propiconazole	No	0.014	QuChERS; TC 275 WI 00275	5	Acetonitrile			Yes	No	Yes	DSPE	MM-ML	MSD		LC/MS/MS
ptc049	Azoxystrobin	No	0.01	Leothay, S. Et al. JAOAC 88 (2005)	10	Acetonitrile			Yes	No	Yes	DSPE	MM-ML		MS/MS	LC/MS/MS
	Carbendazim	No	0.01	Leothay, S. Et al. JAOAC 88 (2005)	10	Acetonitrile			Yes	No	Yes	DSPE	MM-ML		MS/MS	LC/MS/MS
	Deltamethrin	No	0.02	Leothay, S. Et al. JAOAC 88 (2005)	10	Acetonitrile			Yes	No	Yes	DSPE	MM-ML	MS/MS		GC/MS/MS
	Diazinon	No	0.01	Leothay, S. Et al. JAOAC 88 (2005)	10	Acetonitrile			Yes	No	Yes	DSPE	MM-ML	MS/MS		GC/MS/MS
	Pirimiphos-methyl	No	0.01	Leothay, S. Et al. JAOAC 88 (2005)	10	Acetonitrile			Yes	No	Yes	DSPE	MM-ML	MS/MS		GC/MS/MS
	Propiconazole	No	0.02	Leothay, S. Et al. JAOAC 88 (2005)	10	Acetonitrile			Yes	No	Yes	DSPE	MM-ML		MS/MS	LC/MS/MS
ptc050	Diazinon	Yes	0.02	Internal method	5	Dichloromethane	Acetone		Yes	No	No	GPC	PS-ML	NPD		GC/MS
	Pirimiphos-methyl	Yes	0.05	Internal method	5	Dichloromethane	Acetone		Yes	No	No	GPC	PS-ML	NPD		GC/MS
	Propiconazole	Yes	0.05	Internal method	5	Dichloromethane	Acetone		Yes	No	No	GPC	PS-ML	NPD		GC/MS
	Azoxystrobin	Yes	0.05	QuEChERS Anastas-siades	5	Acetonitrile			Yes	No	Yes	DSPE	PS-ML		DAD	Other
	Deltamethrin	Yes	0.05	QuEChERS Anastas-siades	5	Acetonitrile			Yes	No	Yes	DSPE	PS-ML	ECD		Two col.
ptc051	Azoxystrobin	Yes	0.025	Â§64LFGB	8	Acetone	Dichloromethane		No	No	No	GPC	MM-ML	MS/MS		GC/MS/MS

Participants	Pesticide	Accredited	Reporting level	Reference method	Sample weight	Extraction solvent 1	Extraction solvent 2	Extraction solvent 3	Water addition	Hydrolysis	pH adjusted	Clean up	Calibration	GC detector	HPLC detector	Confirmation
ptc051 cont.	Deltamethrin	Yes	0.025	Â§64LFGB	8	Acetone	Dichloromethane		No	No	No	GPC	MM-ML	MS/MS		GC/MS/MS
	Diazinon	Yes	0.025	Â§64LFGB	8	Acetone	Dichloromethane		No	No	No	GPC	MM-ML	MS/MS		GC/MS/MS
	Propiconazole	Yes	0.025	Â§64LFGB	8	Acetone	Dichloromethane		No	No	No	GPC	MM-ML	MS/MS		GC/MS/MS
ptc052	Deltamethrin	Yes	0.05		5	Acetone	Dichloromethane	Dichloromethane	No			GPC	MM-ML	ECD		None
	Diazinon	Yes	0.02		5	Acetone	Dichloromethane	Dichloromethane	No			GPC	MM-ML	ECD		Two col.
	Pirimiphos-methyl	Yes	0.05		5	Acetone	Dichloromethane	Dichloromethane	No			GPC	MM-ML	NPD		Two col.
	Carbendazim	No	0.05		15	Ethyl acetate	Other	Ethyl acetate	Yes	Yes	Yes	liq./liq part.	PS-ML		Fluor.	Other
ptc053	Azoxystrobin	No	0.05	INTERNAL METHOD	15	Dichloromethane							MM-ML	ECD		GC/MS
	Carbendazim	No	0.1	INTERNAL METHOD	15	Dichloromethane							MM-ML		MS/MS	LC/MS/MS
	Deltamethrin	No	0.05	INTERNAL METHOD	15	Dichloromethane							MM-ML	ECD		GC/MS
	Diazinon	No	0.02	INTERNAL METHOD	15	Dichloromethane							MM-ML	NPD		GC/MS
	Pirimiphos-methyl	No	0.05	INTERNAL METHOD	15	Dichloromethane							MM-ML	NPD		GC/MS
ptc053	Propiconazole	No	0.05	INTERNAL METHOD	15	Dichloromethane							MM-ML	ECD		GC/MS
ptc054	Azoxystrobin	Yes	0.01	Â§35 00.00-34	10	Acetone	Ethyl acetate	Cyclohexane	Yes	No	Yes	GPC	MM-ML	ECD		GC/MS
	Deltamethrin	Yes	0.01	Â§35 00.00-34	10	Acetone	Ethyl acetate	Cyclohexane	Yes	No	Yes	GPC	MM-ML	ECD		GC/MS
	Diazinon	Yes	0.01	Â§35 00.00-34	10	Acetone	Ethyl acetate	Cyclohexane	Yes	No	Yes	GPC	MM-ML	NPD		GC/MS
	Pirimiphos-methyl	Yes	0.01	Â§35 00.00-34	10	Acetone	Ethyl acetate	Cyclohexane	Yes	No	Yes	GPC	MM-ML	NPD		GC/MS
	Procymidone	Yes	0.01	Â§35 00.00-34	10	Acetone	Ethyl acetate	Cyclohexane	Yes	No	Yes	GPC	MM-ML	ECD		GC/MS
	Propiconazole	Yes	0.01	Â§35 00.00-34	10	Acetone	Ethyl acetate	Cyclohexane	Yes	No	Yes	GPC	MM-ML	ECD		GC/MS
ptc055	Deltamethrin	Yes	0.01	SLV M914	48	Ethyl acetate			No	No	No	None	MM-SL	MS/MS		GC/MS/MS
	Pirimiphos-methyl	Yes	0.01	SLV M914	48	Ethyl acetate			No	No	No	None	MM-SL	MS/MS		GC/MS/MS
	Azoxystrobin	Yes	0.01	SLV M915	15	Acetonitrile			Yes	No	No	Other	MM-SL		MS/MS	LC/MS/MS
	Carbendazim	Yes	0.01	SLV M915	15	Acetonitrile			Yes	No	No	Other	MM-SL		MS/MS	LC/MS/MS

Participants	Pesticide	Accredited	Reporting level	Reference method	Sample weight	Extraction solvent 1	Extraction solvent 2	Extraction solvent 3	Water addition	Hydrolysis	pH adjusted	Clean up	Calibration	GC detector	HPLC detector	Confirmation
ptc055 cont.	Diazinon	Yes	0.01	SLV M915	15	Acetonitrile			Yes	No	No	Other	MM-SL		MS/MS	LC/MS/MS
	Propiconazole	Yes	0.01	SLV M915	15	Acetonitrile			Yes	No	No	Other	MM-SL		MS/MS	LC/MS/MS
ptc056	Azoxystrobin	Yes	0.01		15	Acetonitrile			Yes		pH 4	None	MM-ML		MS/MS	LC/MS/MS
	Carbendazim	Yes	0.01		15	Acetonitrile			Yes		pH 4	None	MM-ML		MS/MS	LC/MS/MS
	Deltamethrin	Yes	0.01		15	Acetonitrile			Yes		pH 4	None	MM-ML	MS/MS		GC/MS/MS
	Diazinon	Yes	0.01		15	Acetonitrile			Yes		pH 4	None	MM-ML		MS/MS	LC/MS/MS
	Pirimiphos-methyl	Yes	0.01		15	Acetonitrile			Yes		pH 4	None	MM-ML	MS/MS		GC/MS/MS
	Propiconazole	Yes	0.01		15	Acetonitrile			Yes		pH 4	None	MM-ML		MS/MS	LC/MS/MS
ptc057	Azoxystrobin	No	0.01	PN-EN 12393-1,2,3	25	Acetone	Dichloromethane		No	No	No	GPC	MM-ML	ECD		GC/MS
	Deltamethrin	Yes	0.01	PN-EN 12393-1,2,3	25	Acetone	Dichloromethane		No	No	No	GPC	MM-ML	ECD		GC/MS
	Diazinon	Yes	0.01	PN-EN 12393-1,2,3	25	Acetone	Dichloromethane		No	No	No	GPC	MM-ML	ECD		GC/MS
	Pirimiphos-methyl	Yes	0.01	PN-EN 12393-1,2,3	25	Acetone	Dichloromethane		No	No	No	GPC	MM-ML	NPD		GC/MS
ptc058	Azoxystrobin	No	0.05	istisan 97/23	10	Dichloromethane			No	No	No	GPC	PS-ML	ECD		GC/MS
	Deltamethrin	Yes	0.05	istisan 97/23	10	Dichloromethane			No	No	No	GPC	PS-ML	ECD		GC/MS
	Diazinon	Yes	0.02	istisan 97/23	10	Dichloromethane			No	No	No	GPC	PS-SL	NPD		GC/MS
	Pirimiphos-methyl	Yes	0.05	istisan 97/23	10	Dichloromethane			No	No	No	GPC	PS-SL	NPD		GC/MS
	Propiconazole	No	0.05	istisan 97/23	10	Dichloromethane			No	No	No	GPC	PS-SL	ECD		GC/MS
ptc059	Azoxystrobin	Yes	0.05	M. Anastassiades et al JAOAC 86 (2003)	5	Acetonitrile			Yes	No	No	Freezing out	PS-ML		DAD	GC/MS
	Deltamethrin	Yes	0.02	M. Anastassiades et al JAOAC 86 (2003)	5	Acetonitrile			Yes	No	No	Freezing out	PS-ML	ECD		GC/MS
	Diazinon	Yes	0.02	M. Anastassiades et al JAOAC 86 (2003)	5	Acetonitrile			Yes	No	No	Freezing out	PS-ML	NPD		GC/MS
	Pirimiphos-methyl	Yes	0.05	M. Anastassiades et al JAOAC 86 (2003)	5	Acetonitrile			Yes	No	No	Freezing out	PS-ML	NPD		GC/MS
	Propiconazole	Yes	0.05	M. Anastassiades et al JAOAC 86 (2003)	5	Acetonitrile			Yes	No	No	Freezing out	PS-ML	ECD		GC/MS

Participants	Pesticide	Accredited	Reporting level	Reference method	Sample weight	Extraction solvent 1	Extraction solvent 2	Extraction solvent 3	Water addition	Hydrolysis	pH adjusted	Clean up	Calibration	GC detector	HPLC detector	Confirmation
ptc060	Azoxystrobin	No	0.02		5	Acetone	Other		No	No	No	SPE	MM-ML	MSD		GC/MS
	Deltamethrin	No	0.05		5	Acetone	Other		No	No	No	SPE	MM-ML	MSD		GC/MS
	Diazinon	No	0.02		5	Acetone	Other		No	No	No	SPE	MM-ML	MSD		GC/MS
	Pirimiphos-methyl	No	0.05		5	Acetone	Other		No	No	No	SPE	MM-ML	MSD		GC/MS
	Propiconazole	No	0.05		5	Acetone	Other		No	No	No	SPE	MM-ML	MSD		GC/MS
ptc061	Azoxystrobin	Yes	0.02	Anal.Meth.f.Pest.Res.in Foodst.NL 6th Ed. 1996	20	Acetone	Dichloromethane		No	No	No	None	MM-ML	ECD		GC/MS
	Deltamethrin	Yes	0.02	Anal.Meth.f.Pest.Res.in Foodst.NL 6th Ed. 1996	20	Acetone	Dichloromethane		No	No	No	None	MM-ML	ECD		GC/MS
	Diazinon	Yes	0.01	Anal.Meth.f.Pest.Res.in Foodst.NL 6th Ed. 1996	20	Acetone	Dichloromethane		No	No	No	None	MM-ML	FPD		GC/MS
	Pirimiphos-methyl	Yes	0.01	Anal.Meth.f.Pest.Res.in Foodst.NL 6th Ed. 1996	20	Acetone	Dichloromethane		No	No	No	None	MM-ML	FPD		GC/MS
	Propiconazole	Yes	0.01	Anal.Meth.f.Pest.Res.in Foodst.NL 6th Ed. 1996	20	Acetone	Dichloromethane		No	No	No	None	MM-ML	ECD		GC/MS
	Carbendazim	Yes	0.02	Klein,J.,Alder L., JAOAC 86,1015 (2003)	5	Methanol			Yes	No	No	liq./liq part.	MM-ML		MS/MS	
ptc062	Azoxystrobin	Yes	0.02		20	Acetone			Yes	No	No	SPE	MM-ML	MSD		None
	Deltamethrin	No	0.02		20	Acetone			Yes	No	No	SPE	MM-ML	MSD		None
	Diazinon	Yes	0.02		20	Acetone			Yes	No	No	SPE	MM-ML	MSD		None
	Pirimiphos-methyl	Yes	0.02		20	Acetone			Yes	No	No	SPE	MM-ML	MSD		None
	Propiconazole	No	0.02		20	Acetone			Yes	No	No	SPE	MM-ML	MSD		None
	Carbendazim	Yes	0.01		20	Methanol			Yes		pH 7	SPE	MM-ML		MS/MS	None
ptc063	Deltamethrin	Yes	0.01	ISS 97/23	5	Ethyl acetate			No	No	No	GPC	PS-ML	ECD		Two col.
	Diazinon	Yes	0.01	ISS 97/23	5	Ethyl acetate			No	No	No	GPC	PS-ML	NPD		Two col.
	Pirimiphos-methyl	Yes	0.01	ISS 97/23	5	Ethyl acetate			No	No	No	GPC	PS-ML	NPD		Two col.
	Propiconazole	Yes	0.01	ISS 97/23	5	Ethyl acetate			No	No	No	GPC	PS-ML	ECD		Two col.

Participants	Pesticide	Accredited	Reporting level	Reference method	Sample weight	Extraction solvent 1	Extraction solvent 2	Extraction solvent 3	Water addition	Hydrolysis	pH adjusted	Clean up	Calibration	GC detector	HPLC detector	Confirmation
ptc064	Diazinon	No	0.02	SAR-1-04	12	Acetone	Dichloromethane	Other	No	No	No					
	Pirimiphos-methyl	No	0.05	SAR-1-04	12	Acetone	Dichloromethane	Other	No	No	No					
	Propiconazole	No	0.05	SAR-2-04oc	6	Acetone	Dichloromethane	Other	No	No	No					
ptc065	Azoxystrobin	Yes	0.02		5	Acetone	Dichloromethane	Other	Yes				MM-ML		MS/MS	LC/MS/MS
	Carbendazim	Yes	0.02		5	Acetone	Dichloromethane	Other	Yes				MM-ML		MS/MS	LC/MS/MS
	Diazinon	Yes	0.02		25	Acetone	Dichloromethane						PS-ML	FPD		GC/MS
	Pirimiphos-methyl	Yes	0.02		25	Acetone	Dichloromethane						PS-ML	FPD		GC/MS
	Propiconazole	Yes	0.05		5	Acetone	Dichloromethane	Other	Yes				MM-ML		MS/MS	LC/MS/MS

SRM APPENDICES

Appendix 5 Homogeneity

Chlormequat (mg/kg)		
Sample	Portion 1	Portion 2
1	1.252	1.257
2	1.196	1.257
3	1.204	1.251
4	1.242	1.233
5	1.227	1.214
6	1.233	1.237
7	1.233	1.253
8	1.220	1.236
9	1.225	1.258
10	1.193	1.227

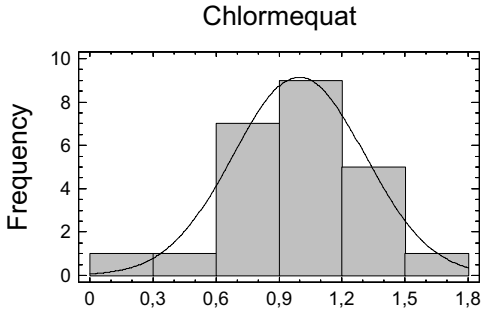
MCPA (mg/kg)		
Sample	Portion 1	Portion 2
1	0.036	0.040
2	0.050	0.048
3	0.046	0.046
4	0.050	0.047
5	0.045	0.050
6	0.042	0.050
7	0.047	0.040
8	0.046	0.045
9	0.045	0.040
10	0.046	0.049

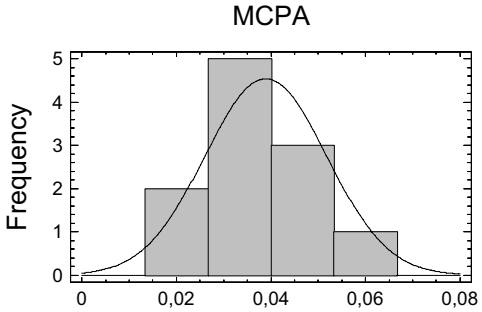
MCPA (optional) (mg/kg)		
Sample	Portion 1	Portion 2
1	0.231	0.277
2	0.299	0.300
3	0.245	0.317
4	0.286	0.283
5	0.232	0.212
6	0.270	0.281
7	0.272	0.228
8	0.218	0.282
9	0.206	0.220
10	0.246	0.275

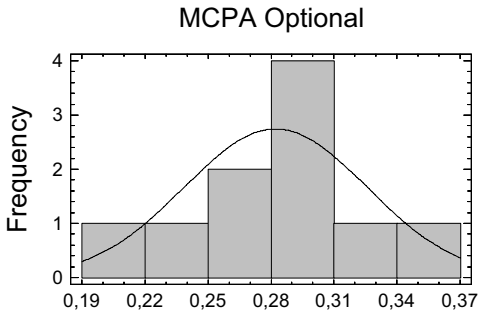
Mecoprop (mg/kg)		
Sample	Portion 1	Portion 2
1	0.394	0.393
2	0.354	0.461
3	0.392	0.385
4	0.404	0.421
5	0.443	0.477
6	0.360	0.378
7	0.378	0.437
8	0.466	0.411
9	0.412	0.408
10	0.409	0.425

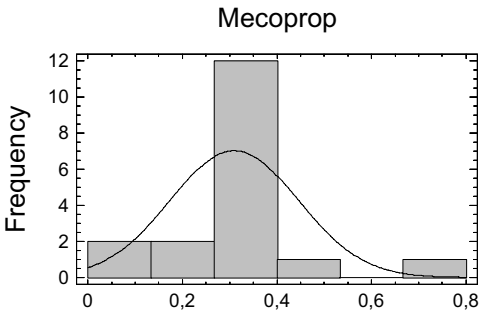
Mecoprop (optional) (mg/kg)		
Sample	Portion 1	Portion 2
1	0.540	0.559
2	0.562	0.524
3	0.517	0.579
4	0.459	0.470
5	0.528	0.585
6	0.495	0.504
7	0.528	0.545
8	0.500	0.549
9	0.552	0.492
10	0.507	0.599

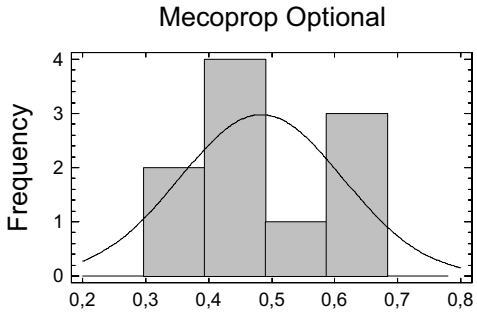
Appendix 6 Histograms







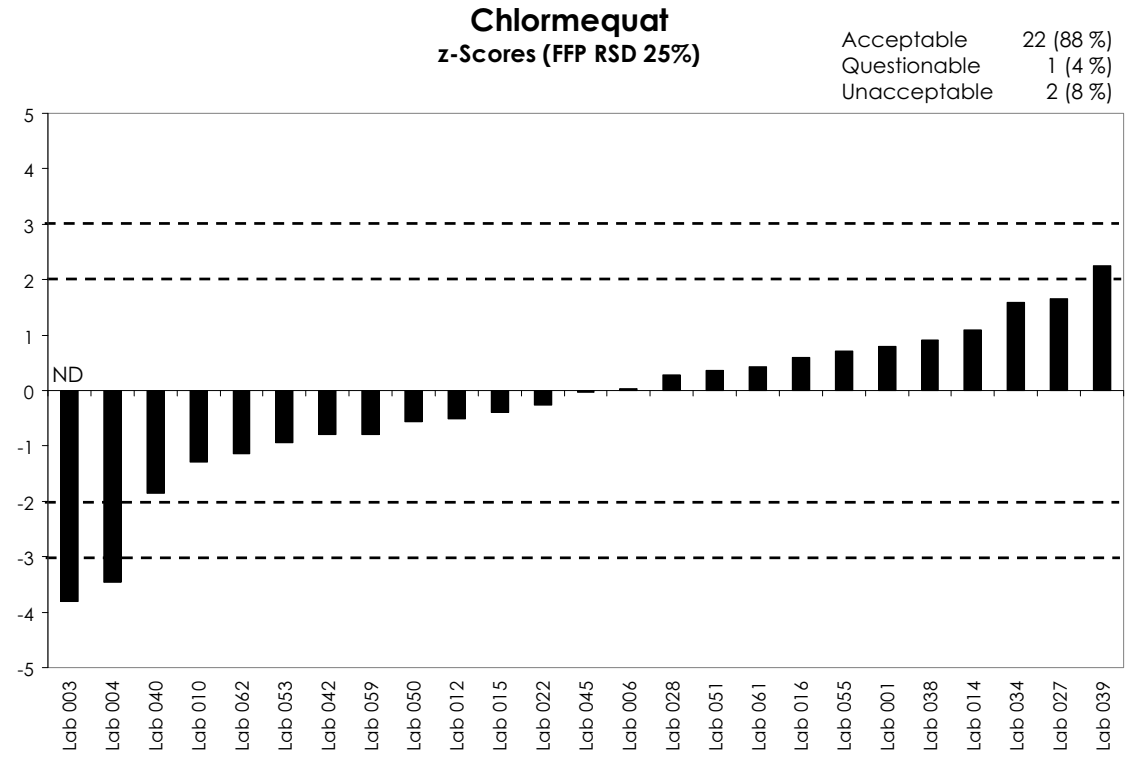




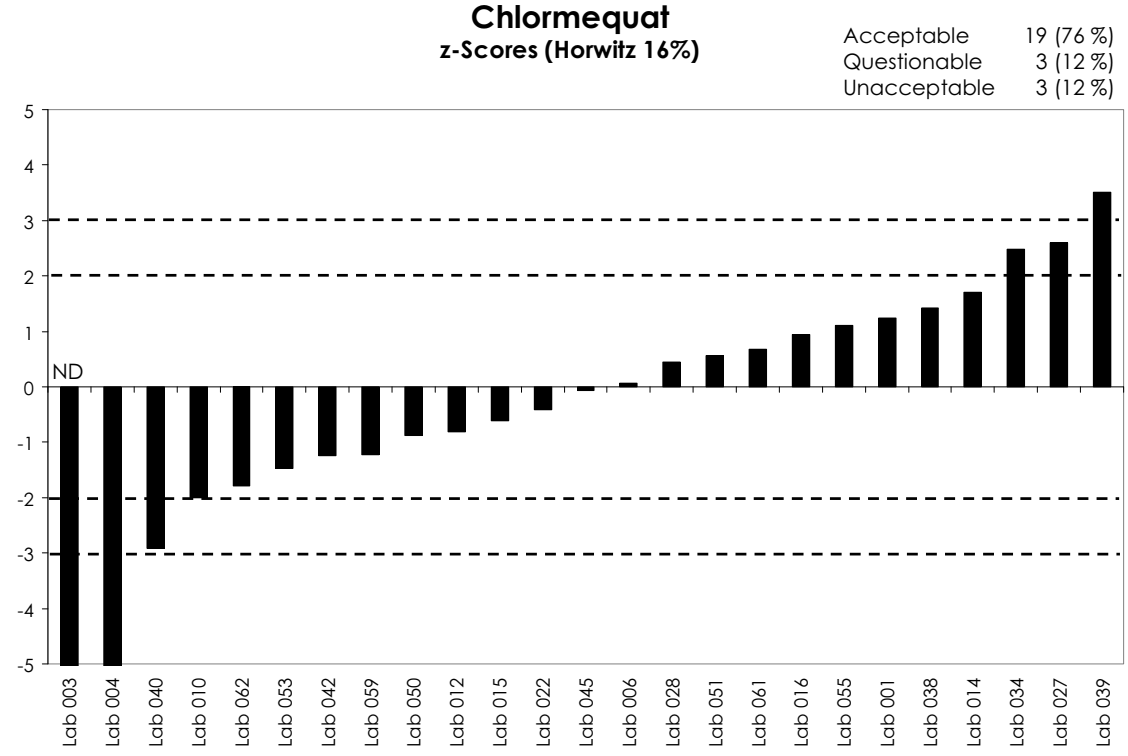
Appendix 7 Graphical presentation of z-scores for each pesticide

z-Scores for Chlormequat:

a) using FFP RSD 25%

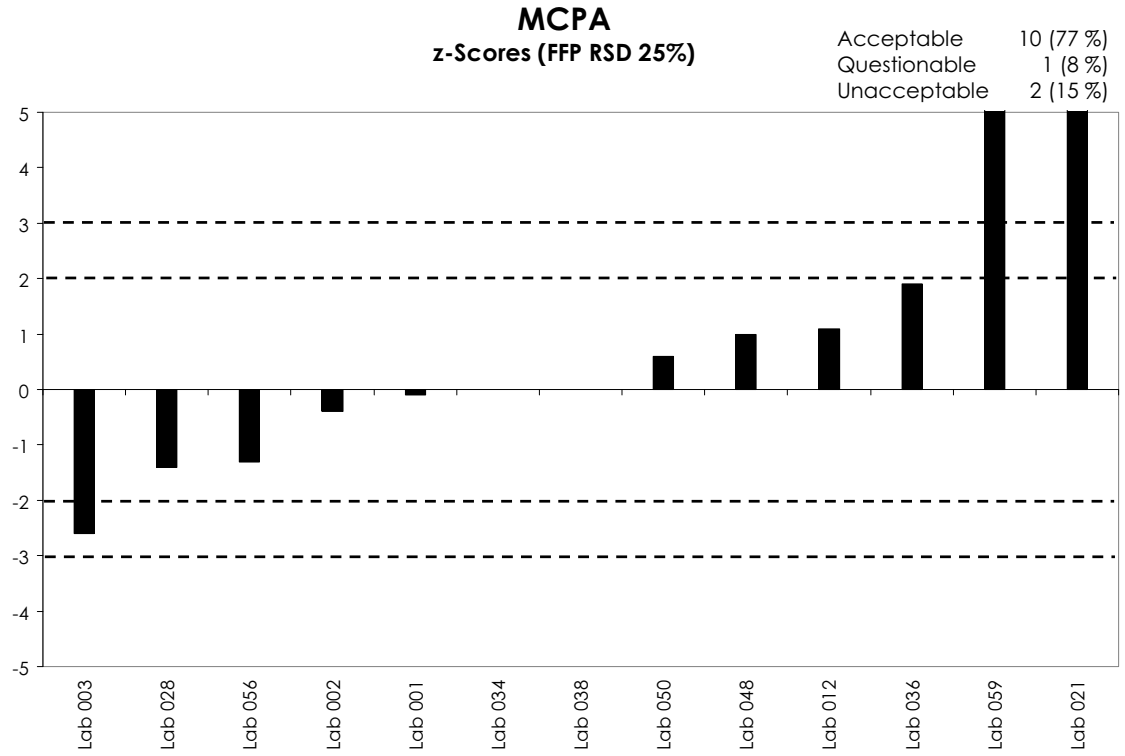


b) using Horwitz-Equation

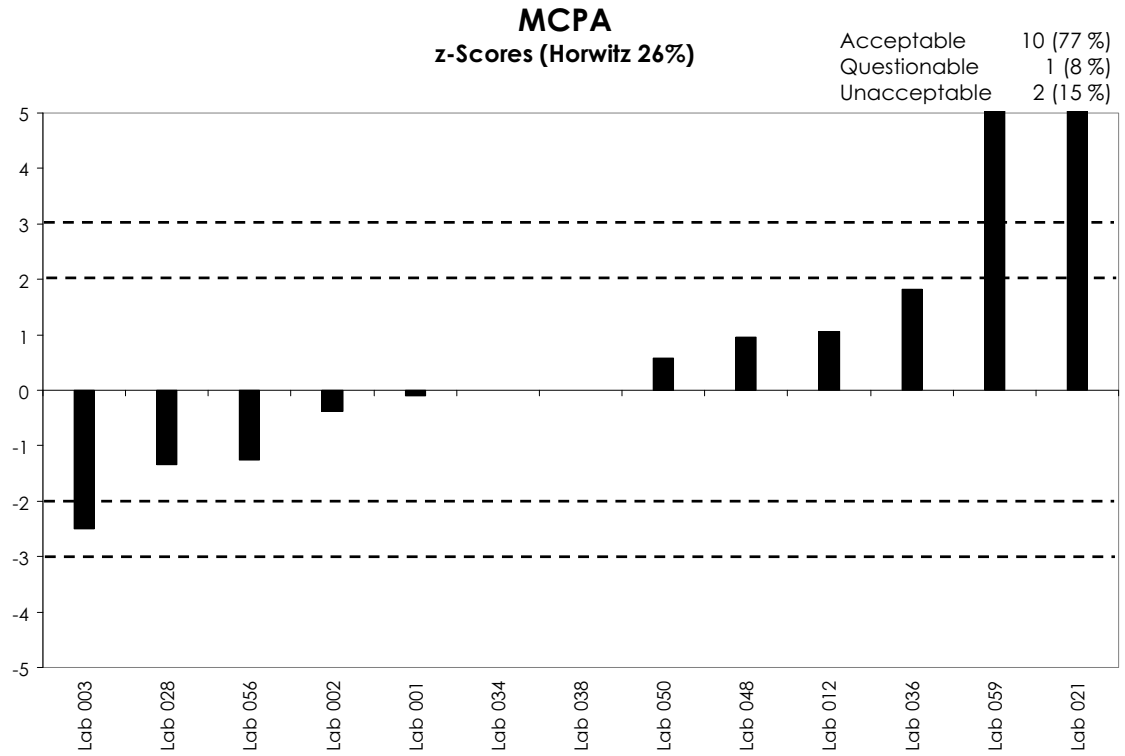


z-Scores for MCPA:

a) using FFP RSD 25%

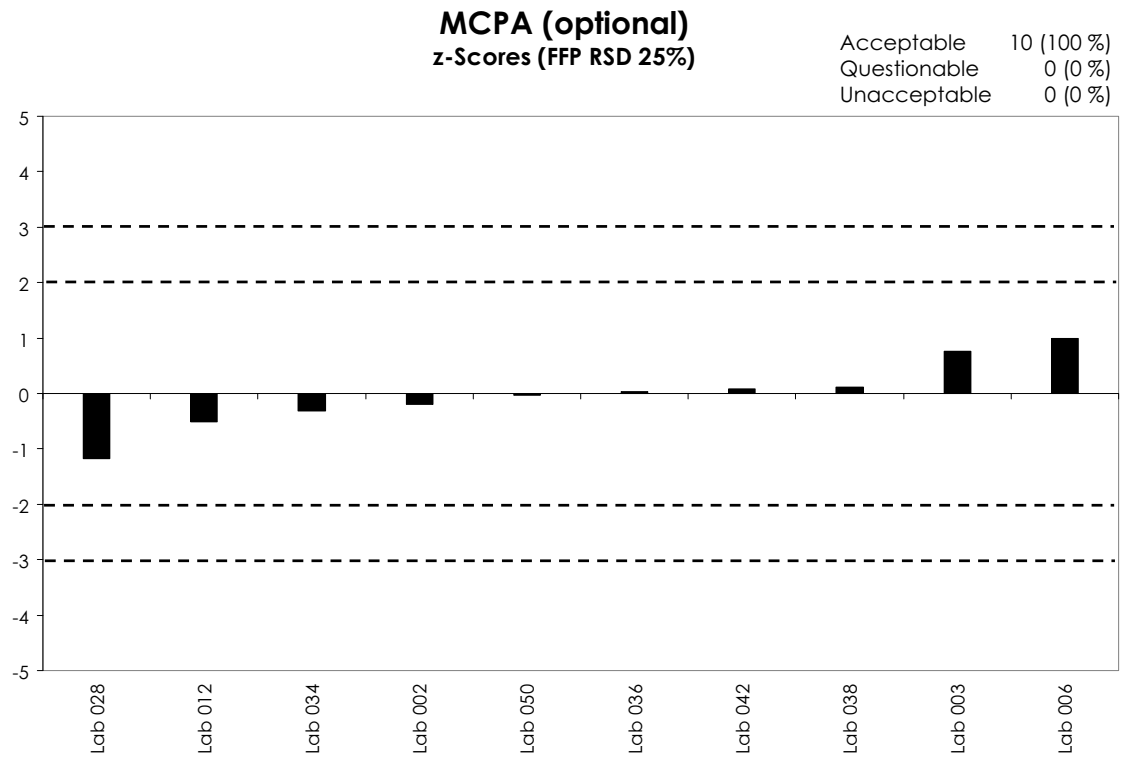


b) using Horwitz-Equation

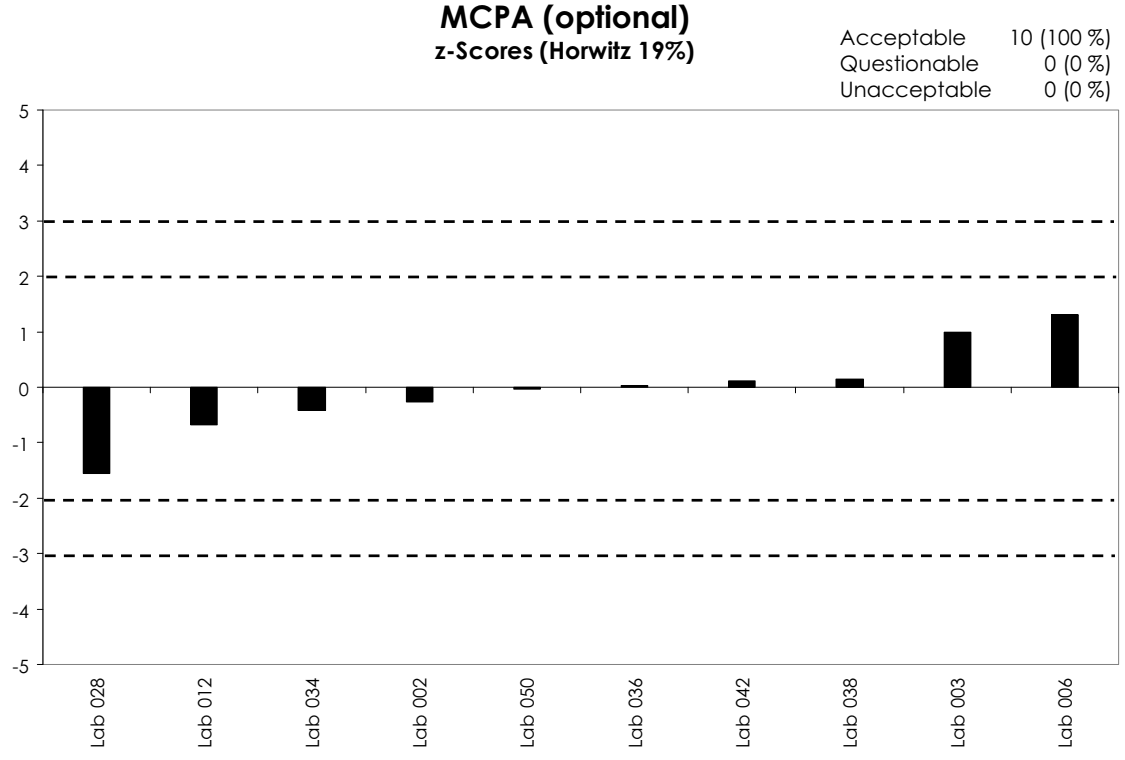


z-Scores for MCPA (optional):

a) using FFP RSD 25%

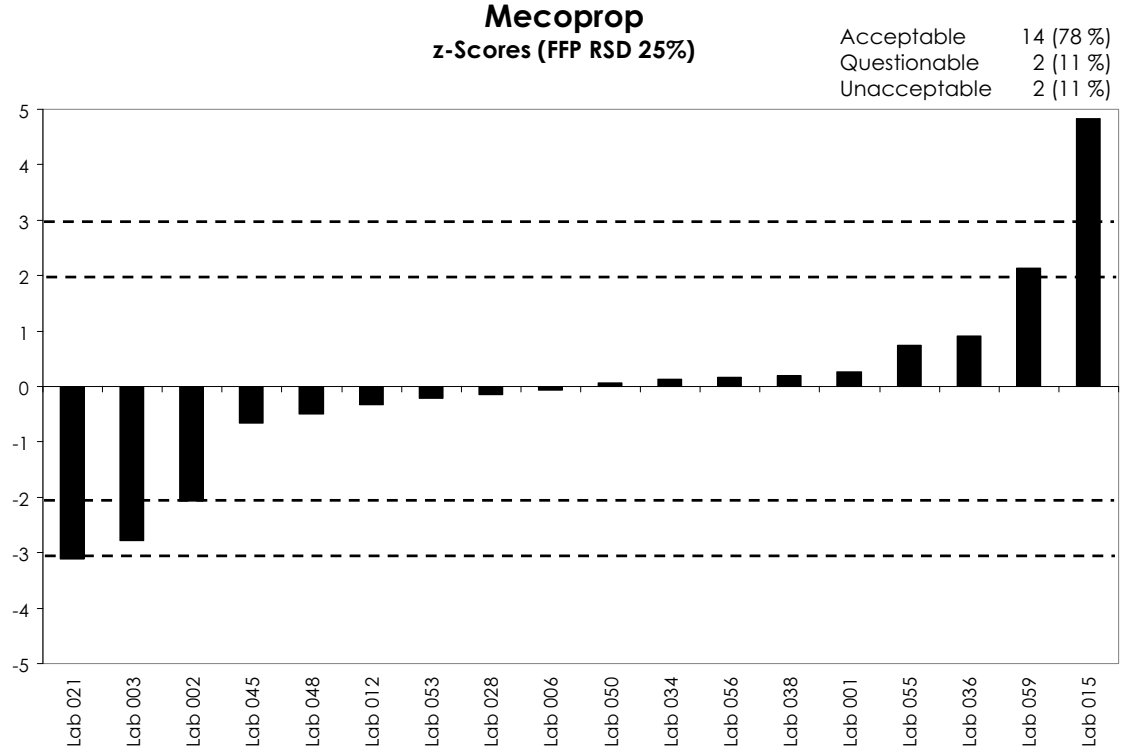


b) using Horwitz-Equation

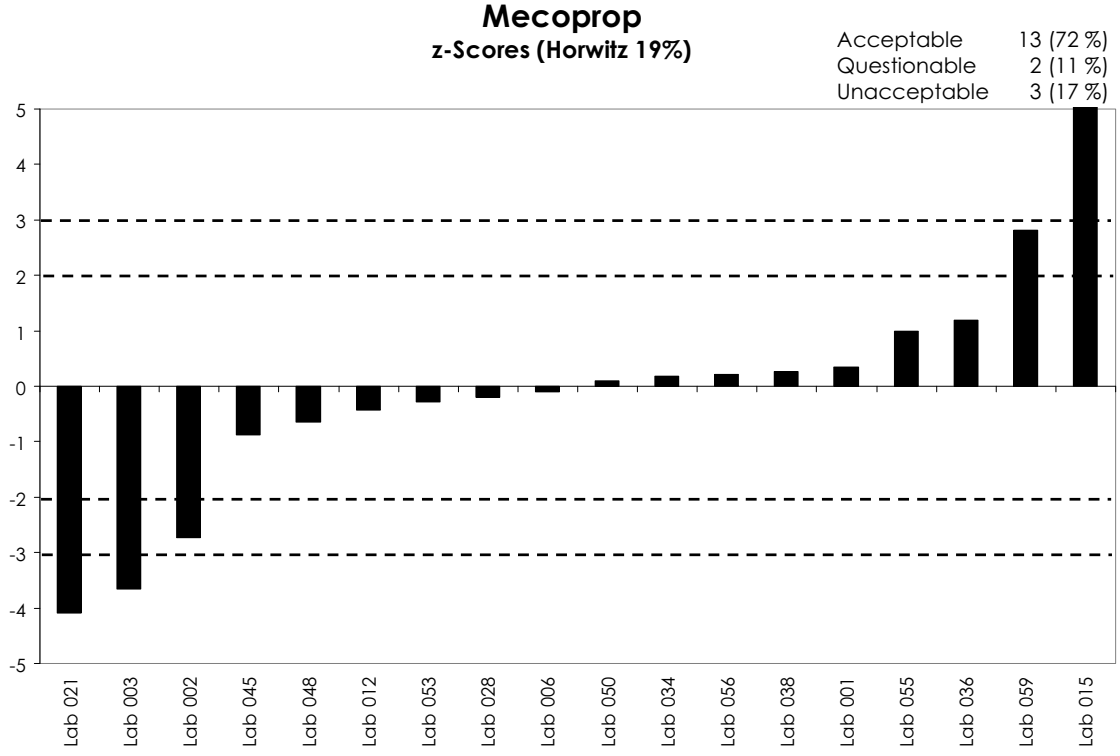


z-Scores for Mecoprop:

a) using FFP RSD 25%

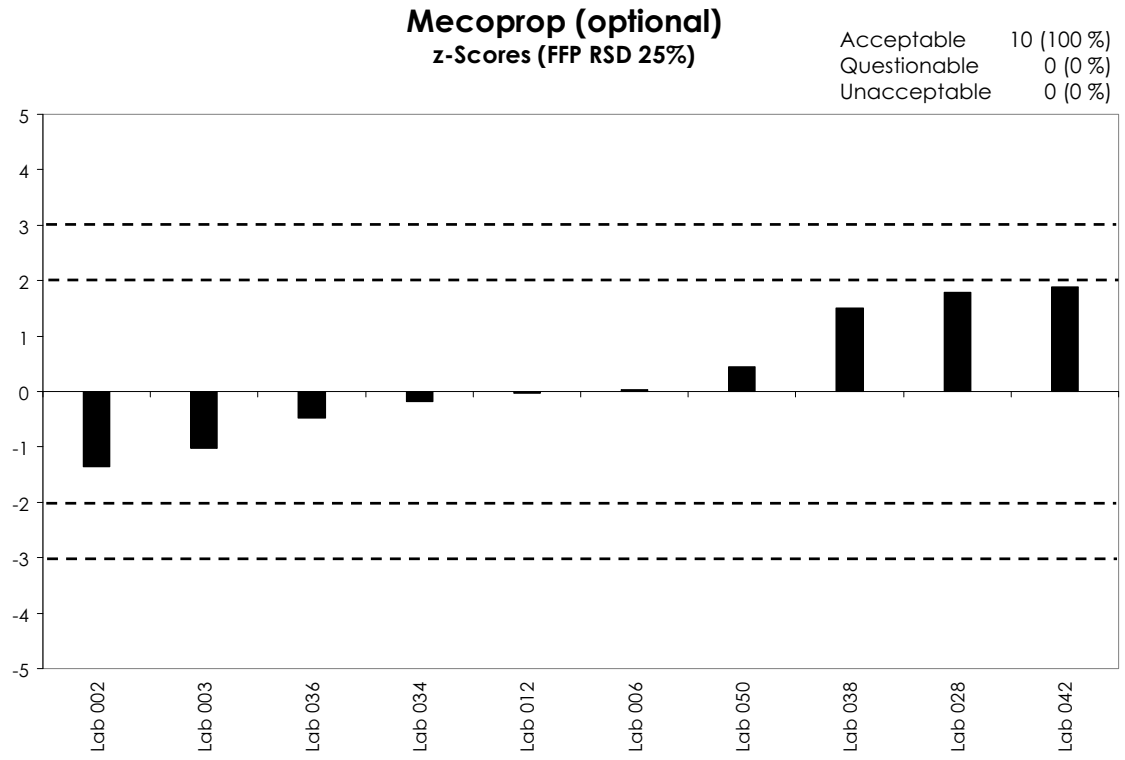


b) using Horwitz-Equation

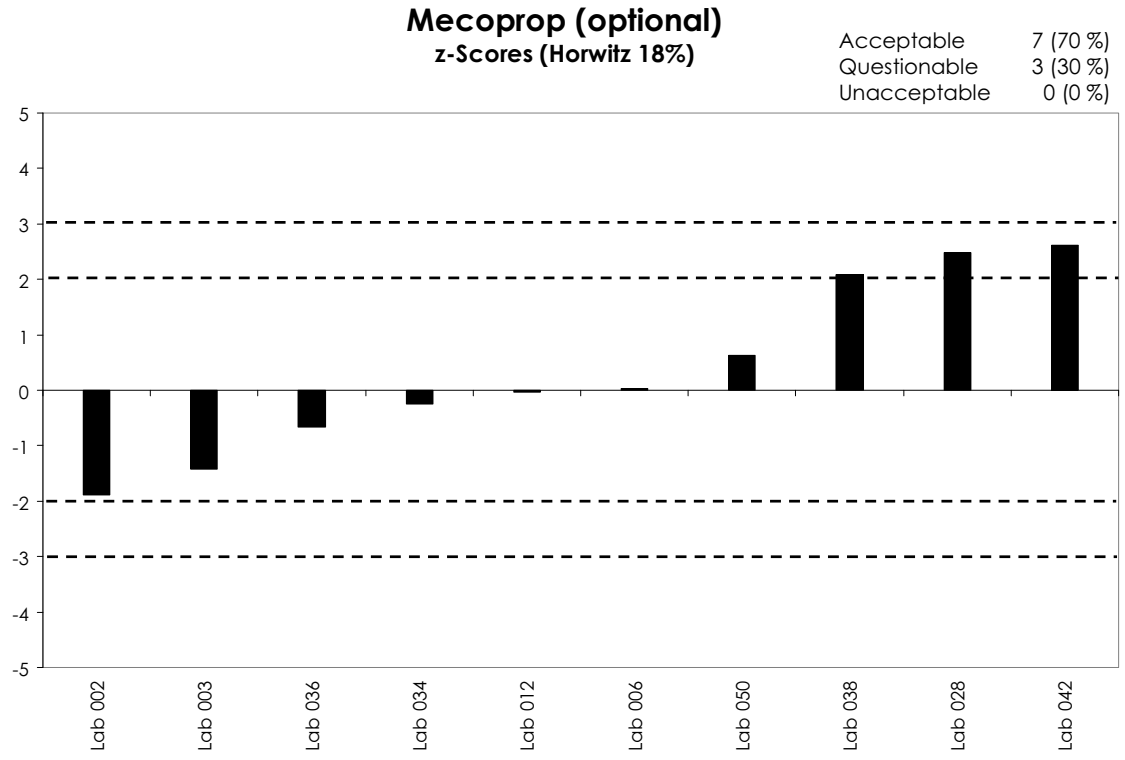


z-Scores for Mecoprop (optional):

a) using FFP RSD 25%



b) using Horwitz-Equation



Appendix 8 SRM-Methods of the participating Laboratories

Participant	Pesticide	Accred.	Reporting level [mg/kg]	Reference method	Sample weight [g]	Extraction solvent 1	Extraction solvent 2	Isotopically labelled Stan-dard?	Water addition	Hydrolysis	pH adjusted	Clean up	Calibration	GC detector	HPLC detector	Confirmation
Lab 001	Chlormequat	Yes	0.01	Startin, Analyst 124 (1999)	25	Methanol			Yes	No	No	None	PS-ML		MS/MS	LC/MS/MS
	MCPA	No	0.01	QuEChERS	5	Acetonitrile			Yes	No	Yes	None	MM-ML		MS/MS	LC/MS/MS
	Mecoprop	No	0.01	QuEChERS	5	Acetonitrile			Yes	No	Yes	None	MM-ML		MS/MS	LC/MS/MS
Lab 002	MCPA	No	0.01	QuEChERS	5	Acetonitrile			Yes	No	pH 5	None	MM-SL		MS/MS	None
	Mecoprop	No	0.01	QuEChERS	5	Acetonitrile			Yes	No	pH 5	None	MM-SL		MS/MS	None
	MCPA (optional)	No	0.01	QuEChERS	5	Acetonitrile			Yes	Yes	pH 5	None	MM-SL		MS/MS	None
	Mecoprop (optional)	No	0.01	QuEChERS	5	Acetonitrile			Yes	Yes	pH 5	None	MM-SL		MS/MS	None
Lab 003	MCPA	No	0.01	EPA Method 3545	5	Acetone	Other		No	No	pH 2	None	PS-ML		MS/MS	LC/MS/MS
	Mecoprop	No	0.02	EPA Method 3545	5	Acetone	Other		No	No	pH 2	None	PS-ML		MS/MS	LC/MS/MS
	MCPA (optional)	No	0.01	QuEChERS	10	Acetonitrile			Yes	Yes	Yes	None	PS-ML		MS/MS	LC/MS/MS
	Mecoprop (optional)	No	0.02	QuEChERS	10	Acetonitrile			Yes	Yes	Yes	None	PS-ML		MS/MS	LC/MS/MS
Lab 004	Chlormequat	No	0.05		10	Methanol		No	No	No	No	None	PS-ML		MS/MS	LC/MS/MS
Lab 006	Chlormequat	No	0.05	NDC-T012-080-2006	5	Methanol		Yes	Yes	No	No	liq./liq part.	MM-SL		MS/MS	LC/MS/MS
	MCPA (optional)	No	0.05	QuEChERS	5	Acetonitrile			Yes	Yes	Yes	DSPE	MM-SL		MS/MS	LC/MS/MS
	Mecoprop	No	0.05	QuEChERS	5	Acetonitrile			Yes	Yes	Yes	DSPE	MM-SL		MS/MS	LC/MS/MS

Participant	Pesticide	Accred.	Reporting level [mg/kg]	Reference method	Sample weight [g]	Extraction solvent 1	Extraction solvent 2	Isotopically labelled Stan-dard?	Water addition	Hydrolysis	pH adjusted	Clean up	Calibration	GC detector	HPLC detector	Confirmation
	Mecoprop (optional)	No	0.05	QuEChERS	5	Acetonitrile			Yes	Yes	Yes	DSPE	MM-SL		MS/MS	LC/MS/MS
Lab 010	Chlormequat	Yes	0.02	§64, LFGB, L00.00 76	10	Methanol		No	Yes			None	MM-ML		MS/MS	
Lab 012	Chlormequat	Yes	0.05	EN 15054	20	Methanol		No	Yes	No	No	None	PS-ML		MS	LC/MS
	MCPA	No	0.01	Internal method	10	Other			No	No	Yes	liq./liq part.	PS-ML	MSD		GC/MS
	MCPA (optional)	No	0.01	Internal method	10	Other			Yes	Yes	Yes	liq./liq part.	PS-ML	MSD		GC/MS
	Mecoprop	No	0.01	Internal method	10	Other			No	No	Yes	liq./liq part.	PS-ML	MSD		GC/MS
	Mecoprop (optional)	No	0.01	Internal method	10	Other			Yes	Yes	Yes	liq./liq part.	PS-ML	MSD		GC/MS
Lab 014	Chlormequat	Yes	0.01		5	Methanol		--	Yes	No	pH 2	None	MM-ML		MS/MS	LC/MS/MS
Lab 015	Chlormequat	Yes	0.02	§ 64 LFGB, L00.00-76	10	Methanol		Yes	Yes	No	No	None	MM-ML		MS/MS	LC/MS/MS
	Mecoprop	Yes	0.005	modified BfR-method (ChemElut, pH4,5)	5	Methanol	Dichloromethane		Yes	No	No	liq./liq part.	MM-ML		MS/MS	LC/MS/MS
Lab 016	Chlormequat	No	0.05	Inetrnal method	5	Methanol		Yes	Yes	No	No	None	MM-ML		MS/MS	LC/MS/MS
Lab 021	MCPA	No	0.05		3	Acetonitrile			Yes	No	No	liq./liq part.	MM-SL		MS/MS	
	Mecoprop	No	0.05		3	Acetonitrile			Yes	No	No	liq./liq part.	MM-SL		MS/MS	
Lab 022	Chlormequat	Yes	0.05	CEN	25	Methanol		Yes	No	No	No	None	MM-SL		MS/MS	LC/MS/MS
Lab 027	Chlormequat	Yes	0.01	FP045	10	Methanol		--	No	No	No	SPE	MM-ML		MS/MS	LC/MS/MS
Lab 028	Chlormequat	No	0.02		0			No								

Participant	Pesticide	Accred.	Reporting level [mg/kg]	Reference method	Sample weight [g]	Extraction solvent 1	Extraction solvent 2	Isotopically labelled Stan-dard?	Water addition	Hydrolysis	pH adjusted	Clean up	Calibration	GC detector	HPLC detector	Confirmation
	MCPA	No	0.02		5	Methanol			Yes	No	Yes	Freezing out	MM-ML		MS/MS	LC/MS/MS
	MCPA (optional)	No	0.02		5	Methanol			Yes	Yes	Yes	Freezing out	MM-ML		MS/MS	LC/MS/MS
	Mecoprop	No	0.02		5	Methanol			Yes	No	Yes	Freezing out	MM-ML		MS/MS	LC/MS/MS
	Mecoprop (optional)	No	0.02		5	Methanol			Yes	Yes	Yes	Freezing out	MM-ML		MS/MS	LC/MS/MS
Lab 034	MCPA	Yes	0.01	QuEChERS	5	Acetonitrile		Yes	Yes	No	No	DSPE	MM-ML		MS/MS	LC/MS/MS
	MCPA (optional)	No	0.01	QuEChERS	5	Acetonitrile			Yes	Yes	Yes	DSPE	MM-ML		MS/MS	LC/MS/MS
	Mecoprop	Yes	0.01	QuEChERS	5	Acetonitrile			Yes	No	No	DSPE	MM-ML		MS/MS	LC/MS/MS
	Mecoprop (optional)	No	0.01	QuEChERS	5	Acetonitrile			Yes	Yes	Yes	DSPE	MM-ML		MS/MS	LC/MS/MS
	Chlormequat	Yes	0.01	internal method	4	Acetone	Other	--				liq./liq part.	PS-ML		MS/MS	LC/MS/MS
Lab 036	MCPA	No	0.05	QuEChERS	2	Acetonitrile			Yes	No	Yes	liq./liq part.	MM-ML		MS/MS	
	MCPA (optional)	No	0.05	QuEChERS	2	Acetonitrile			Yes	No	Yes	liq./liq part.	MM-ML		MS/MS	
	Mecoprop	No	0.05	QuEChERS	2	Acetonitrile			Yes	No	Yes	liq./liq part.	MM-ML		MS/MS	
	Mecoprop (optional)	No	0.05	QuEChERS	2	Acetonitrile			Yes	No	Yes	liq./liq part.	MM-ML		MS/MS	
Lab 038	Chlormequat	Yes	0.01	EN 15055:2006	10	Methanol		Yes	Yes	No	No	None	MM-ML		MS/MS	
	MCPA	Yes	0.005	QuEChERS	5	Acetonitrile			Yes	No	pH 5	Freezing out	MM-ML		MS/MS	
	MCPA (optional)	Yes	0.005	QuEChERS	5	Acetonitrile			Yes	Yes	pH 5	Freezing out	MM-ML		MS/MS	

Participant	Pesticide	Accred.	Reporting level [mg/kg]	Reference method	Sample weight [g]	Extraction solvent 1	Extraction solvent 2	Isotopically labelled Stan-dard?	Water addition	Hydrolysis	pH adjusted	Clean up	Calibration	GC detector	HPLC detector	Confirmation
	Mecoprop	Yes	0.005	QuEChERS	5	Acetonitrile			Yes	No	pH 5	Freezing out	MM-ML		MS/MS	
	Mecoprop (optional)	Yes	0.005	QuEChERS	5	Acetonitrile			Yes	Yes	pH 5	Freezing out	MM-ML		MS/MS	
Lab 039	Chlormequat	Yes	0.005		1	Methanol		--	Yes	No	Yes	None	PS-ML		MS/MS	LC/MS/MS
Lab 040	Chlormequat	No	0.008		5	Methanol	Other	No	Yes				MM-ML			LC/MS/MS
Lab 042	Chlormequat	Yes	0.02	in-house	20	Methanol	Other	No	No	No	No		MM-ML		MS/MS	LC/MS/MS
	MCPA (optional)	No	0.02	QuEChERS	5	Acetonitrile			Yes	Yes			MM-ML		MS/MS	LC/MS/MS
	Mecoprop (optional)	No	0.02	QuEChERS	5	Acetonitrile			Yes	Yes			MM-ML		MS/MS	LC/MS/MS
Lab 045	Chlormequat	Yes	0.020	Lutz Alder	10	Methanol		Yes	Yes	No	No	None	MM-ML		MS/MS	LC/MS/MS
	Mecoprop	Yes	0.020	QuEChERS	5	Acetonitrile			Yes	No	No	Freezing out	Standard addition		MS/MS	LC/MS/MS
Lab 048	MCPA	No	0.048	QuEChERS	5	Acetonitrile			Yes	No	Yes	None	MM-ML		MS/MS	
	Mecoprop	No	0.020	QuEChERS	5	Acetonitrile			Yes	No	Yes	None	MM-ML		MS/MS	
Lab 050	Chlormequat	Yes	0.05	EN 15055:2006	10	Methanol		No	Yes	No	No	None	MM-ML		MS/MS	None
	MCPA	Yes	0.02	QuEChERS	5	Acetonitrile			Yes	No	Yes	Freezing out	MM-ML		MS/MS	None
	MCPA (optional)	Yes	0.02	QuEChERS	5	Acetonitrile			Yes	Yes	Yes	Freezing out	MM-ML		MS/MS	None
	Mecoprop	Yes	0.05	QuEChERS	5	Acetonitrile			Yes	No	Yes	Freezing out	PS-ML		MS/MS	None
	Mecoprop (optional)	Yes	0.05	QuEChERS	5	Acetonitrile			Yes	Yes	Yes	Freezing out	PS-ML		MS/MS	None

Participant	Pesticide	Accred.	Reporting level [mg/kg]	Reference method	Sample weight [g]	Extraction solvent 1	Extraction solvent 2	Isotopically labelled Stan-dard?	Water addition	Hydrolysis	pH adjusted	Clean up	Calibration	GC detector	HPLC detector	Confirmation
Lab 051	Chlormequat	Yes	0.10	no	10	Methanol		--	Yes	No	No	None	MM-ML		MS/MS	LC/MS/MS
Lab 053	Mecoprop	No	0.05	Internal method	10	Acetonitrile				Yes			MM-ML		MS/MS	LC/MS/MS
	Chlormequat	No	0.05	Internal method	10	Methanol		No					MM-ML		MS/MS	LC/MS/MS
Lab 055	Chlormequat	Yes	0.005	SLM M030	10	Methanol		Yes	Yes	No	No	Other	PS-ML		MS/MS	LC/MS/MS
	Mecoprop	Yes	0.01	SLV M915	15	Acetonitrile			Yes	No	No	Other	MM-SL		MS/MS	LC/MS/MS
Lab 056	MCPA	Yes	0.01		15	Acetonitrile			Yes		pH 4	None	MM-ML		MS/MS	LC/MS/MS
	Mecoprop	Yes	0.01		15	Acetonitrile			Yes		pH 4	None	MM-ML		MS/MS	LC/MS/MS
Lab 059	Chlormequat	Yes	0.1	EN 15055:2006	10	Methanol		No	Yes	No	No	Freezing out	MM-SL		MS/MS	
	MCPA	Yes	0.05	QuEChERS	5	Acetonitrile			Yes	No	pH 2	Freezing out	MM-SL	ECD		GC/MS
	Mecoprop	Yes	0.05	QuEChERS	5	Acetonitrile			Yes	No	pH 2	Freezing out	MM-SL	ECD		GC/MS
Lab 061	Chlormequat	Yes	0.01	CEN/TC 275 EN 15054 (2005)	20	Methanol		Yes	Yes	No	No	None	MM-ML		MS/MS	
Lab 062	Chlormequat	Yes	0.01		20	Methanol		--	Yes		pH 7		MM-ML		MS/MS	None

Appendix 9 SRM - Countries, Laboratories and NRLs

	Country	No. of participating Labs	No. of NRLs
		CHLORMEQUAT	
	Austria	2	2
	Belgium	2	1
	Czech Republic	1	0
	Denmark	1	0
	Estonia	1	1
	Finland	1	1
	Germany	5	0
	Hungary	2	2
	Italy	2	0
	Latvia	1	1
	Lithuania	1	1
	Norway	1	1
	Slovakia	1	1
	Slovenia	1	1
	Sweden	1	0
	The Netherlands	1	1
	UK	1	1
SUM	**17**	**25**	**14**
		MCPA	
	Austria	1	1
	Belgium	1	1
	Czech Republic	2	1
	Estonia	1	1
	Finland	1	1
	Germany	3	0
	Hungary	2	2
	Italy	2	0
	Latvia	1	1
	Norway	1	1
	Poland	1	0
	Sweden	2	1
	The Netherlands	1	1
SUM	**13**	**19**	**11**
		MCPA (optional)	
	Belgium	1	1
	Estonia	1	1
	Germany	2	0
	Hungary	1	1
	Italy	1	0
	Latvia	1	1
	Norway	1	1
	Poland	1	0
	UK	1	1
SUM	**9**	**10**	**6**
		MECOPROP	
	Austria	1	1
	Belgium	1	1
	Czech Republic	1	1
	Estonia	1	1
	Finland	1	1
	Germany	3	0
	Hungary	2	2
	Italy	2	0
	Latvia	1	1
	Norway	1	1
	Poland	1	0
	Sweden	2	1
	The Netherlands	1	1
SUM	**13**	**18**	**11**

	Country	No. of participating Labs	No. of NRLs
MECOPROP (optional)			
	Belgium	1	1
	Estonia	1	1
	Germany	2	0
	Hungary	1	1
	Italy	1	0
	Latvia	1	1
	Norway	1	1
	Poland	1	0
	UK	1	1
SUM	**9**	**10**	**6**

ANNEX I Pesticide list

Pesticides	MRPL (mg/kg)
2,4-D (free acid)	0.1
2,4-D (free acid plus covalently bound) (optional)	0.1
Azoxystrobin	0.05
Bifenthrin	0.05
Carbaryl	0.05
Carbendazim (Benomyl + Carbendazim, expressed as Carbendazim)	0.1
Chlormequat (expressed as cation)	0.05
Chlorpyrifos	0.05
Chlorpyrifos-methyl	0.05
Cypermethrin	0.05
Deltamethrin	0.05
Diazinon	0.02
Endosulfan (α + ß + Sulphate expressed as Endosulfan)	0.05
Fenhexamid	0.05
Fenpropimorph	0.05
Imazalil	0.02
Iprodione	0.05
Kresoxim-methyl	0.05
Lambda-cyhalothrin	0.02
Lindane	0.01
Malathion (Malathion + Malaoxon,expressed as Malathion)	0.05
MCPA (free acid)	0.05
MCPA (free acid plus covalently bound) (optional)	0.05
Mecoprop (Mecoprop-P + Mecoprop, expressed as Mecoprop) (free acids)	0.05
Mecoprop (Mecoprop-P + Mecoprop, expressed as Mecoprop) (free acids plus covalently	0.05
Mepiquat (expressed as cation)	0.05
Methacrifos	0.05
Methomyl (Methomyl + Thiodicarb,expressed as Methomyl)	0.05
Parathion	0.05
Penconazole	0.05
Pirimicarb	0.05
Pirimiphos-methyl	0.05

Pesticides	MRPL (mg/kg)
Prochloraz	0.05
Procymidone	0.02
Propiconazole	0.05
Spiroxamine	0.05
Thiabendazole	0.05
Thiodicarb see Methomyl	0.05
Thiophanate-methyl	0.1
Triadimefon (Triadimefon + Triadimenol expressed as Triadimefon)	0.02
Triadimenol see Triadimefon	0.05
Triazophos	0.1
Vinclozolin (only parent compound)	0.1

ANNEX II: List of laboratories registered to participate in the PTs

COUNTRY	LABORATORY NAME	REPORTED RESULTS
Austria	AGES Competence Center Pesticide Residues	Yes
Austria	AGES Competence Centre Residue Analysis Vienna	Yes
Belgium	Scientific Institute of Public Health	Yes
Belgium	ERC NV	Yes
Belgium	Fytolab	Yes
Belgium	CER Groupe - Laboratoire d'Hormonologie	Yes
Czech Republic	Institute of Chemical Technology, Department of Food Chemistry and Analysis	Yes
Czech Republic	CISTA/NRL Brno	Yes
Denmark	Danish Vet. And Food Adm., Region East	Yes
Estonia	Agricultural Research Centre Lab for Res.	Yes
Finland	Finnish Customs Laboratory	Yes
France	Laboratoire de SCL de Montpellier	Yes
France	SCL Laboratoire d'Ile de France	Yes
Germany	Chemisches Landes- und Staatliches Veterinäruntersuchungsamt	Yes
Germany	Landesamt für Verbraucherschutz Sachsen-Anhalt	Yes
Germany	LAVES Lebensmittelinstitut Oldenburg	Yes
Germany	BVL	Yes
Germany	Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit	Yes
Germany	Sächsische Landesanstalt für Landwirtschaft Fachbereich 8	Yes
Germany	Thür. Landesanstalt für Landwirtschaft	Yes
Germany	Landesanstalt für Landwirtschaft, Forsten und Gartenbau des Landes Sachsen-Anhalt; PB Rückstandsanalytik	Yes
Greece	Benaki Phytopathological Institute Pesticide Residues Laboratory	Yes
Greece	General Chemical State Lab	Yes

COUNTRY	LABORATORY NAME	REPORTED RESULTS
Hungary	Agricultural Office of County Fejér, Plant Protection and Soil Conservation Directorate, Pesticide Residue Analytical Laboratory	Yes
Hungary	Agricultural Office of Somogy County Plant Protection and Soil Conservation Directorate	Yes
Iceland	Matis ohf	No
Ireland	Pesticide Control Service	Yes
Italy	Arpa Piemonte	Yes
Italy	Arpa FVG- Dipartimento di Pordenone	Yes
Italy	Appa Trento Settore Laboratorio e Controlli	Yes
Italy	Arpa Ferrara	Yes
Italy	Arpa Puglia - Dipartimento di Bari	Yes
Italy	Arpav Dipartimento di Vicenza	Yes
Italy	CReAA	Yes
Italy	AUSL N.7 Ragusa Arpa Sicilia Dap Ragusa	Yes
Italy	Arpa Marche - Dip. Macerata	Yes
Italy	Arpa Sardegna Dip. Cagliari	Yes
Italy	Arpat Dipartimento prov.le di Livorno	Yes
Latvia	National Diagnostic Centre	Yes
Lithuania	National Veterinary Laboratory	Yes
Luxemburg	Contrôle des Denrées alimentaires, LNS	Yes
Norway	Norwegian Institute for Agricultural and Environmental Research, Bioforsk Laboratory	Yes
Poland	Research Institute of Pomology and Floriculture, Food Safety Laboratory	Yes
Poland	Plant Protection Institute	Yes
Poland	Instytut Ochrony Roslin, Laboratorium Badania Pozostalosci Srodkow Ochrony Roslin	Yes
Poland	Department of Pesticide Residue Research The Institute of Plant Protection	Yes
Poland	Institute of Plant Protection, Experimental Station	Yes
Poland	Wojewódzka Stacja Sanitarno-Epidemiologiczna w Rzeszowie	Yes

COUNTRY	LABORATORY NAME	REPORTED RESULTS
Poland	Instytut Ochrony Roślin	Yes
Poland	Wojewódzka Stacja Sanitarno- Epidemiologiczna	Yes
Poland	Wojewódzka Stacja Sanitarno-Epidemiologiczna Laboratorium Badania Żywności i Przedmiotów Użytku	Yes
Portugal	Pesticide Residues Laboratory/Direcção-Geral de Protecção das Culturas	Yes
Portugal	Laboratorio Qualidade Agricola	Yes
Slovakia	State Veterinary and Food Institute Bratislava	Yes
Slovenia	Institute of Public Health Maribor	Yes
Slovenia	Institute for public health	Yes
Spain	Laboratori Agroalimetari Dar (Generalitat de Catalunya)	Yes
Spain	Lab. Agrario Fraisoro	Yes
Sweden	Lantmännen Analycen AB	Yes
Sweden	National Food Administration Chemistry Division 1	Yes
The Netherlands	Rikilt Institute of Food Safety	Yes
The Netherlands	VWA - Food and Consumer Product Safety Authority	Yes
UK	Central Science Laboratory	Yes

ANNEX III List of abbreviations

The following abreviations has been used in the report and in the appendices.

Abreviation	Describsion
DAD	Diode Array Detector
DSPE	Dispersive Solid Phase Extraction
ECD	Electron Capture Detector
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
liq./liq part.	Liquid / Liquid Partitioning
MM-ML	Matrix Matched Multi Level Calibration
MM-SL	Matrix Matched Single Level Calibration
MRPL	Minimum Required Performance 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
SPE	Solid Phase Extraction
LOD	Limit of determination
SW	Sample weight

Commission Reference Laboratories on Cereals & Feedingstuff and Single Residues Methods

Final report
December 2007

Technical University of Denmark