

**EU Reference Laboratory
on Cereals & Feedingstuff**

**Report on
Proficiency Test on
incurred and spiked
pesticides in rye**

**EUPT-C4
2010**



**Final report
December 2010**

**DTU National Food Institute
Technical University of Denmark**

EU PROFICIENCY TESTS

EUPT-C4, 2010

Pesticide Residues in Cereals

Final Report

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PREFACE

Regulation 882/2004/EC [1], lays down the general tasks and duties of the European Union Reference Laboratories (EURLs) for Food, Feed and Animal including the organisation of comparative tests. These proficiency tests are carried out on an annual basis, and aim to improve the quality, accuracy and comparability of the analytical results generated by EU Member States within the frame of the EU co-ordinated control and national monitoring programmes. At the same time laboratories are provided with an assessment, both of their analytical performance and the reliability of their data - by comparison with the other participating laboratories. This will hopefully result in improvements in the analytical quality of the residue data and increase the scope of the methods used by laboratories.

According to Article 28 of Regulation 396/2005/EC on maximum residue levels of pesticides in, or on, food and feed of plant and animal origin [2], all laboratories analysing samples for the official controls of pesticide residues shall participate in the European Union Proficiency Tests (EUPTs) for pesticide residues facilitated by the Commission as long as the analytical scope of the PT and the laboratory overlap.

The present EUPT was the fourth organized within the frame of the EURL activities with cereal matrices as test materials. The first (EUPT-C1/SRM2) was conducted in 2007 using wheat flour containing both agriculturally incurred and laboratory-spiked pesticides. This test was, co-organised by the EURL for Cereals and Feedingstuff (EURL-CF) and the EURL for Single Residue Methods (EuRL-SRM). The second PT (EUPT-C2) was performed in 2008 by the EURL-CF using a wheat flour test material containing mostly MRM-pesticides as well as two SRM pesticides (chlormequat and glyphosate). The third PT (EUPT-C3/SRM4) was jointly organised by the EURL-CF and EURL-SRM using oat flour focusing on both MRM and SRM pesticides. The present EUPT-C4 used rye flour previously treated with 19 compounds partly in the field and partly post-harvest in the laboratory.

Participation in EUPT-4 was open to all National Reference Laboratories (NRLs) and Official Laboratories (OfLs) within the EU involved in the determination of pesticide residues in cereals using multi- or single residue methods for their national or EU co-ordinated control programmes. Additionally, laboratories from the EFTA countries (Iceland, Norway and Switzerland) were invited to take part in the PT as they also contribute data to the EU-coordinated community control programme. NRLs and OfLs that did not take part in this test were asked to explain the reasons for their non-participation.

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



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1. INTRODUCTION

On 13 November 2009, all relevant National Reference Laboratories (NRLs) of the 27 EU-Member States (MS) as well as all relevant EU-Official Laboratories (OfLs), as far as their contact data were communicated by the NRLs, were sent an invitation to participate in this 4th European Commission's Proficiency Test on Cereals (EUPT-C4). To make sure that all relevant official laboratories became aware of the PT, the NRLs were asked to additionally forward the invitation to the relevant laboratories within their countries. Also included in the invitation was a Specific Protocol, a Calendar, as well as a Target Pesticides List including all compounds that could potentially be present in the test material. The Target Pesticides List included 60 compounds (pesticides, metabolites etc.) requiring the use of multiresidue methods (MRMs) and an additional 13 compounds requiring single residue methods (SRMs), along with a minimum required reporting level (MRRRL) stipulated for each compound. For 8 acidic pesticides included in the SRM list the laboratories had the opportunity to report two results: one derived following alkaline hydrolysis to release bound residues, and a second without the inclusion of this step. A "General Protocol" (see **Annex 1**) containing information that is common to all EUPTs- was also distributed to the laboratories. The laboratories were able to register on-line by 14 January. In total 118 laboratories from 28 countries agreed to participate (see **Appendix 1**).

The present proficiency test was performed using rye flour of Danish origin, which had been partly treated in the field, and partly spiked post-harvest at the facilities of the EURL-CF. The test material contained 20 compounds in total. The Faculty of Agricultural Sciences, University of Aarhus performed the field treatments. The pesticides employed for field treatment were selected by the EURL-CF and the EURL-SRM with the application rates and harvest intervals chosen based on previous experience and data from supervised residue trials. The harvested grain was treated with two pesticides post-harvest, and then checked for homogeneity before shipping to participants. Furthermore, the stabilities of the pesticides in the test material were checked several times during the period of time allowed for laboratories to undertake the PT exercise.

The participating laboratories were provided with 150 g portions of treated whole rye flour test material and 150 g of blank whole rye flour. The test materials were shipped to the participants on 15 February 2010 and the deadline for submission of results to the Organiser was the 15 March 2010. The participants were asked to analyse the treated test material as well as the 'blank' material and report the concentrations of any pesticide residues found which were included in the Target Pesticide List (see **Appendix 2**). Additionally, the 'blank' material could be used for recovery experiments for the pesticides found in the test material, and if necessary, for the preparation of matrix-matched calibration standards. Submission of results was performed online via a website. Eight laboratories did not submit any results.

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

2. TEST MATERIALS

2.1 Analytical methods

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

- For **MRM pesticides**: QuEChERS method and determination using GC-MS/MS and LC-MS/MS. For more details see www.quechers.com
- For **SRM pesticides**:
 - Mepiquat: Methanol–water–acetic acid extraction involving addition of an isotopically labeled internal standard, followed by centrifugation, filtration and determined by LC–MS/MS. [3]
 - Glyphosate: Extraction with water by ultrasonication, with clean up and separation by LC–MS/MS using a polystyrene-based reverse-phase column (clean-up) in series with an ion chromatography column (separation) using NaHCO₃ as eluent. A micro-membrane suppressor was inserted after the separator column to remove the Na⁺ ions before detection. [4]
 - Acidic pesticides (2,4-D): QuEChERS-method involving water addition and extraction with acetonitrile followed by phase partitioning induced by addition of salts, and a direct determination by LC-MS/MS in the ESI-neg. mode. Where required alkaline hydrolysis was performed directly after the water addition step. For this sodium hydroxide solution was added and the mixture was allowed to react for 30 minutes at room temperature before neutralization with sulfuric acid. The procedure was then continued as described for the QuEChERS method mentioned above. For more details see www.crl-pesticides.eu

2.2 Selection of Pesticides for the Target Pesticide List

The pesticides to be included in the target pesticides list were selected by the Organiser and the Scientific Committee taking into account the present and upcoming scope of the EU-coordinated control programme, a pesticide priority list ranking the pesticides according to their relevance and risk-potential, as well as a list of pesticides relevant to the specific commodity (rye). The overall capacity and capability of the laboratories within the EU, as assessed from previous PTs and surveys, was also taken into account. In some cases the residue definitions valid for the test were slightly different from those in the legislation to overcome analytical difficulties (e.g. in the case of prochloraz). The proficiency test covered both MRM and SRM pesticides. However, only analysis of the MRM pesticides was mandatory. The minimum required reporting levels (MRRLs) were set at 0.01 mg/kg for the MRM-compounds and at 0.02 mg/kg for the SRM-compounds.

2.3 Preparation of the treated test material

Before preparing the test material, the pesticides and suitable target residue levels for the study were selected. The application rates and harvest intervals for the 16 pesticides used for treatment in the field were chosen based on data from supervised residue trials. The field spraying was performed by the Faculty of Agricultural Sciences, University of Aarhus. One hundred kilograms of rye grain was delivered for preparation of the test material. Following a preliminary analysis of the material it was decided to addition-

ally spike in the laboratory with 2,4-D, which could not be detected in the harvested material in a preliminary test, as well as carbaryl, fenitrothion and isoproturon which, were not included in the field treatments (see **Table 1**). Spiking in the laboratory was performed using pesticide formulations. One kilogram of oats was spiked with one of the pesticide formulations, and a second kilogram was spiked with the other pesticide formulation. The resulting 2 kg were mixed with 58 kg and homogenised thoroughly. The 60 kg of mixed rye grain were milled as four kilograms portions. The portions were stirred thoroughly individually and additionally following two by two additions to ensure that a well-homogenised bulk with respect to both incurred and spiked pesticide residues was obtained. 150g portions were weighed out into screw-capped polyethylene plastic bottles, sealed, numbered, and stored in a freezer at about -20 °C prior to homogeneity testing and distribution to participants.

2.4 Preparation of the ‘blank’ test material

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

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

Pesticide	Application in the field	Spiked in laboratory	Formulation	Company
MRM-Compounds				
Azoxystrobin	x		Amistar	Syngenta
Carbaryl		x	Sevin WP 85	Bayer
Carbendazim	x		Bavistin FL	BASF
Chlorpyrifos-methyl	x		Reldan 22	DOW
Deltametrin	x		Decis EW 50	Bayer
Fenitrothion		x		Sumitomo
Fenpropimorph	x		Corbel	BASF
Fluquinconazole	x		Diablo	BASF
Flutriafol	x		Flutriafol 125 EC	Cheminova
Isoproturon		x	Arelon Top	Bayer
Kresoxim-methyl	x		Candid	BASF
Lambda-cyhalothrin	x		Karate 2.5 WG	Syngenta
Malathion	x		Fyfanon	Cheminova
Pirimiphos-methyl	x		Actellic 50 SC	Syngenta
Spiroxamine	x		Spiroxamin EC 500	Bayer
Triadimenol	x		Bayfidan	Bayer
SRM-Compounds				
2,4-D	x	x	2,4-D(AH Marks)	Klarsøe
Mepiquat	x		Terpal	BASF
Glyphosate	x		Glyphomax	Dow AgroSciences

2.5 Homogeneity test

Eleven bottles of treated test material were randomly chosen and analyses were performed on duplicate portions taken from each bottle. The sequence of analyses and injection sequence were both randomly chosen. The quantification was performed using a 5-point calibration curve constructed from matrix-matched standards.

The statistical evaluation was performed according to the International Harmonized Protocols published by IUPAC, ISO and AOAC [5]. An overview of the statistical analyses of the homogeneity test is shown in

Table 2. The individual residue data from the homogeneity tests, as well as the results of the statistical analyses, are given in **Appendix 3**.

The acceptance criteria for the test material to be sufficiently homogenous for the proficiency test were that: $S_s^2 < c$ where S_s is the between-bottle sampling standard deviation and $c = F_1\sigma_{all} + F_2s_{2an}$: F_1 and F_2 being constants with values of 1.83 and 0.93, respectively, from the 11 samples taken, and $\sigma_{all} = 0.3 \times \text{FFP RSD}(25\%) \times \text{the analytical sampling mean for all pesticides}$.

As all pesticides passed the homogeneity test, the test material was considered to be sufficiently homogeneous and suitable for the PT-C4.

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

	Azoxystrobin	Carbaryl	Carbendazim	Chlorpyrifos-methyl	Deltamethrin	Fenitrothion	Fenpropimorph	Fluquinazole	Flutriafol	Isoproturon
Mean, mg/kg	0.341	0.163	1.03	0.127	0.069	0.153	2.40	0.77	2.03	0.157
S_s^2	0.00069	0.00000	0.00000	0.00000	0.00005	0.00000	0.001	0.003	0.015	0.00001
c	0.0012	0.0008	0.016	0.0002	0.00009	0.0003	0.071	0.006	0.047	0.0005
Pass/Fail	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass

	Kresoxim-methyl	Lambda-cyhalothrin	Malathion	Pirimiphos-methyl	Spiroxamin	Triadimenol	2,4 D (free acid)	2,4 D (alk.hydro)	Glyphosate	Mepiquat
Mean, mg/kg	0.412	0.052	0.095	0.083	0.959	1.24	0.306	0.299	3.28	0.09
S_s^2	0.00032	0.00001	0.00001	0.00000	0.00000	0.0022	0.00010	0.00000	0.00000	0.00001
c	0.0020	0.0002	0.0003	0.0001	0.015	0.022	0.0041	0.0032	0.14	0.0001
Pass/Fail	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass

S_s : Between Sampling Standard Deviation

2.6 Stability tests

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

Two different storage temperatures were used; room temperature and -18 °C. In all cases the analyses were performed on 5 randomly chosen samples.

The dates of testing were as follows:

For MRM-compounds:

- Day 1: 16 February 2010 (shortly after the first shipment)
- Day 2: 23 February 2010
- Day 3: 2 March 2010
- Day 4: 9 March 2010
- Day 5: 16 March 2010

The average results from each stability test for the MRM pesticides are given in **Table 3**. A graphic presentation of the stability test results, are shown in **Appendix 4**. The tests did not show any significant decrease in the pesticide levels at -18 °C indicating that at these storage conditions the pesticides present in the test material remained stable for the entire duration of the Proficiency Test.

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

	Azoxystrobin	Carbaryl	Carbendazim	Chlorpyriphos-methyl	Deltamethrin	Fenitrothion	Fenpropimorph	Fluquinazole	Flutriafol	Isoproturon
Day 1	0.323	0.180	1.10	0.129	0.064	0.164	2.44	0.853	2.37	0.164
Storage at -18 °C (mean values in mg/kg)										
Day 8	0.333	0.190	1.37	0.134	0.054	0.178	2.38	0.741	2.27	0.206
Day 15	0.299	0.161	1.10	0.124	0.068	0.177	2.33	0.791	2.39	0.192
Day 21	0.332	0.183	1.01	0.143	0.058	0.196	2.68	0.836	2.45	0.173
Day 28	0.349	0.159	1.03	0.139	0.061	0.185	2.13	0.832	2.05	0.166
	pass	pass	pass	pass	pass	pass	pass	pass	pass	Pass

Storage at Room temperature (mean values in mg/kg) – informative purpose only										
Day 8	0.346	0.160	1.14	0.133	0.054	0.164	2.44	0.840	2.31	0.179
Day 28	0.393	0.144	1.05	0.125	0.075	0.166	2.28	0.781	2.44	0.161

	Kresoxim-methyl	Lambda-cyhalothrin	Malathion	Pirimiphos-methyl	Spiroxamin	Triadimenol-sum	2,4- D free acid	2,4-D alk hydro	Mepiquat	Glyphosate
Day 1	0.420	0.084	0.088	0.081	1.02	1.10	0.325	0.394	0.084	3.42
Storage at -18 °C (mean values in mg/kg)										
Day 8	0.449	0.076	0.107	0.091	1.26	1.35	0.457	0.322	0.083	4.36
Day 15	0.439	0.084	0.102	0.077	1.07	1.21	0.365	0.403	0.093	4.74
Day 21	0.428	0.067	0.116	0.090	0.940	1.03	0.371	0.384	0.090	3.43
Day 28	0.378	0.082	0.111	0.087	0.953	1.05	0.349	0.379	0.099	4.24
	pass	pass	pass	pass	Pass	pass	pass	pass	pass	Pass
Storage at Room temperature (mean values in mg/kg) – informative purpose only										
Day 8	0.441	0.078	0.109	0.092	1.13	1.28	0.402	0.364	0.081	4.54
Day 28	0.334	0.096	0.113	0.090	0.994	1.02	0.299	0.396	0.095	4.45

2.7 Organisational details

2.7.1 Access to documents, registration and confidentiality

In the invitation letter on 31 October 2009 all NRLs and OfLs were prompted to register at the CIRCA platform of the pesticide EURLs where the online registration link and all documents related to this EUPT (Calendar, Target Pesticides List, Specific Protocol, General Protocol) were uploaded. All relevant laboratories, independent of whether or not they were intending to participate at this exercise had to register on the webpage by the 30 January 2010. Laboratories that were intending not to participate were given the opportunity to explain the reasons for their non-participation. After registration, the participants were provided with a username, password, laboratory code and the link for the online result submission website. This ensured confidentiality throughout the entire duration of the PT.

2.7.2 Distribution of the test material

One bottle of treated test material (150 g) and one bottle of 'blank' material (150 g) were shipped on 15 February 2010 to each participant in boxes containing a freezer pack. The laboratories were asked to check the state of the sample on receipt and to enter the website (see above) and communicate whether they accept/not accept the test material.

2.7.3 Submission of results

An online submission tool was developed, allowing participants to submit their results via the internet. All participants had access to the result-submission website (<http://thor.dfvf.dk/ptc>) from the day of the sample shipment until the result-submission deadline (15 March 2010). Participants were asked not only to report their analytical results, but also to give information regarding accreditation, reporting limits and details about the methods they used to analyse the test material.

3. EVALUATION OF THE RESULTS

3.1 False positives and negatives

3.1.1 False positives

These are results that show the apparent presence of pesticides that were listed in the Target Pesticide List, but which were (i) not used in the sample treatment, (ii) and not detected by the organiser, even after a repeat analysis. However, if a number of participants did detect the same additional pesticide, or if the concentration was above the MRRL, then a decision as to whether, or not, this should be considered to be a false positive result was made on a case-by-case basis. Any results reported that were lower than the MRRL were not considered as false positives, even though these results should not have been reported.

3.1.2 False negatives

These are results for pesticides reported by the laboratories as “analysed” but that no numerical values were given, although they were used by the Organiser to treat the test material and were detected by the majority of participants at or above the MRRL.

3.2 Estimation of the true concentration (μ)

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

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

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

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

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

3.4 z-Scores

This parameter was calculated using the following formula:

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

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

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

The z-scores were interpreted in the following way:

$|z| \leq 2$ Acceptable

$2 < |z| \leq 3$ Questionable

$|z| > 3$ Unacceptable

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

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

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

3.5 Category A and B classification and combined -Scores (SWZ)

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

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

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

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

n = number of reported results

So for each laboratory:

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

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

- $Z \leq 2$ Good
- $2 < Z \leq 3$ Satisfactory
- $Z > 3$ Unsatisfactory

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

- SWZ ≤ 2 : good;
- $2 < \text{SWZ} \leq 3$: satisfactory;
- SWZ > 3 : unsatisfactory

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

4. RESULTS

In total, 118 laboratories representing 29 countries agreed to participate in this proficiency test, and 115 of the laboratories submitted results for MRM pesticides before 3 March. The participating laboratories are listed in **Appendix 1**. All analytical results reported can be seen in **Table 10**, **Table 11**, **Table 12**, **Table 13**, **Table 14** and **Table 15**; the methods used are shown in **Appendix 7**. The laboratories were asked to give two results for each detected pesticide, a result with and a result without correction for recovery. This following assessment is based on the uncorrected results, unless the participant reported that they routinely corrects for recoveries. **In these cases the corrected results have been assessed.**

4.1 Results

An overview of the results can be seen in **Table 7**. Chlorpyrifos-methyl, malathion, kresoxim-methyl, pirimiphos-methyl, fenitrothion, deltametrin, lambda-cyhalothrin and carbaryl were the most frequently analysed compounds and more than 80 % of the labs submitted results for these compounds. All other pesticides were targeted by less than 80 % of the laboratories.

4.1.1 False positives

Eleven laboratories reported seventeen results above MRRL for additional pesticides that had not been used to treat the test material (see **Table 4**). In all cases the compounds were not detected either by the organizer or by the other participating laboratories. The reported results are therefore considered to be false positives.

Five laboratories report nine results below the MRRL for additional pesticides (see **Table 5**). These results are not considered to be false positives. However, the laboratories should have been more careful in reporting the results as the results were not only lower than the MRRL but most of them also lower than their own reporting limits.

4.1.2 False negatives

Pesticides actually present in the test material but reported as not detected (ND), were considered to be false negatives. **Table 6** summarizes the number of reported false negatives for each pesticide. For the MRM pesticides, 61 false negative results were reported, which represents 4 % of the total number of MRM pesticide results. More than 30% of the participants (40 laboratories) had false negative results. Two laboratories submitted results as <0.01 (triadimenol) or 0.02 (deltamethrin). This value is not in accordance with the protocol and therefore both results were considered to be false negatives. For deltamethrin, 22 participants had false negatives. No method information was collected from these participants. Assuming that they used the same method as for lambda-cyhalothrin, it seems unlikely that extraction and detection method had a significant influence. However, there was a slight predominance of false negatives amongst participants that used acetonitrile as extraction solvent (alone or in combination). Likewise there was a slight predominance of false negatives amongst participant using GC-MS and ITD, while no false negatives were seen amongst laboratories using GC/MS/MS and GC/TOF.

Table 4 False positive results at or at above 0.01 mg/kg, the concentration level detected in mg/kg, determination technique, reporting level and MRRL in mg/kg.

Laboratory code	Pesticide	Concentration, mg/kg	Determination Technique	RL, mg/kg	MRRL, mg/kg
7	Epoxiconazole	0.016	LC-MS/MS	0.01	0.01
16	Metribuzin	0.129	GC-ECD	0.01	0.01
16	Trifluralin	0.0286	GC-MS/MS	0.01	0.01
20	Vinclozolin	0.205	GC-MS/MS	0.01	0.01
35	Diazinon	0.01	GC/MS	0.01	0.01
39	Dichlorvos	0.057	GC-MS/MS	0.01	0.01
39	Methacrifos	0.018	GC-MS/MS	0.01	0.01
39	Trifluralin	0.014	GC-MS/MS	0.01	0.01
40	-Triadimefon	1.75	LC-MS/MS	0.01	0.01
57	Metconazole	0.095	LC-MS/MS	0.01	0.01
58	Procymidone	0.018	GC-ECD	0.01	0.01
101	Cyproconazole	1.506	GC-NPD	0.1	0.01
106	Metribuzin	0.081	GC/MS	0.01	0.01
114	Cypermethrin (sum)	0.01	GC-MS/MS	0.01	0.01
114	Metconazole	0.05	LC-MS/MS	0.01	0.01
114	Permethrin	0.012	GC-MS/MS	0.01	0.01
114	Procymidone	0.01	GC-MS/MS	0.01	0.01

Table 5 False positive results below 0.01 mg/kg, the concentration level detected in mg/kg, determination technique, reporting level and MMRL in mg/kg.

Laboratory code	Pesticide	Concentration (mg/kg)	Determination Technique	RL, mg/kg	MRRL, mg/kg
16	Methomyl	0.0084	LC-MS/MS	0.01	0.01
26	Malaoxon	0.005	GC-MS	0.01	0.01
35	Endosulfan sulfate	0.009	GC-MS	0.01	0.01
48	Malaoxon	0.0081	LC-MS/MS	0.001	0.01
48	Hexaconazole	0.0038	LC-MS/MS	0.01	0.01
114	Bifenthrin	0.009	GC-MS/MS	0.01	0.01
114	Chlorpyrifos	0.008	GC-MS/MS	0.01	0.01
114	Endosulfan sulfate	0.007	GC-MS/MS	0.01	0.01
114	Vinclozolin	0.007	GC-MS/MS	0.01	0.01

Table 6 False negative results.

Lab code	Azoxystrobin	Carbaryl	Chlorpyrifos-methyl	Deltamethrin	Fenpropathrin	Fenpropimorph	Fluquinconazole	Isoproturon	Lambda-cyhalothrin	Malathion (sum)	Malathion	Triadimefon and triadimenol	Triadimenol	2,4-D (foli. alk hydrolysis)	Mepiquat	Glyphosate
001				ND		ND				ND		ND				
003														ND	ND	
005	ND															
007				ND												
010		ND					ND									
011					ND											
018					ND											
022					ND											
023					ND											
024					ND											
025					ND											
027			ND													
035				ND												
040												ND				
046										ND						
056				ND												
057														ND		
061				ND												
066		ND										ND				
070				ND				ND								
076													ND			
077														ND		
082												ND	ND			
083							ND					ND				
089				ND												
090	ND															
093				ND												
095				ND												
096									ND	ND						
098												ND	ND			
099										ND						
100				ND												
103				ND												
106				ND		ND										
108				ND												
110												ND				
114												ND	ND			
116				ND												
117			ND	ND	ND			ND	ND	ND	ND	ND				
119				ND								ND				

Table 7 Overview of number of results, number of not analysed (NA), number of not detected (ND=false negatives) and the percentage of laboratories that reported results for the pesticides in the test material.

Pesticides	No. of results	No. of NA ¹⁾	No. of ND ²⁾	% results ³⁾
Azoxystrobin	96	21	1	83
Carbaryl	94	24	1	81
Carbendazim	78	40		67
Chlorpyrifos-methyl	110	8	4	95
Deltametrin	104	14	22	90
Fenitrothion	106	12	1	91
Fenpropimorph	76	42	1	66
Fluquinconazole	75	43	2	65
Flutriafol	71	47		61
Isoproturon	65	53	2	56
Kresoxim-methyl	95	10		82
Lambda-cyhalothrin	100	18	3	86
Malathion SUM	84	34	5	72
- Malathion	109	9	1	94
Pirimiphos-methyl	108	10		93
Spiroxamine	76	42		66
Triadimenol and triadimefon	87	31	9	75
- Triadimenol	90	28	5	78
2,4-D, free acid	34	84		29
2,4-D, foll. alk. hydr.	21	97	1	18
Mepiquat	37	81	2	32
Glyphosate	15	103	1	13

1) NA = not analysed

2) ND = not detected (false negatives)

3) '% results' has been calculated using the number of laboratories that reported results for that particular compound and the total number of laboratories submitting results (n = 115). False negatives are included in reported results.

4.2 Assigned values and target standard deviations

To establish the assigned values, the median levels of all the reported results, excluding the outliers, were used. However, for those participants that reported that they routinely corrected for recovery, the corrected values were used for the evaluation. Fifteen results were regarded as outliers; azoxystrobin (9.92 mg/kg), carbaryl (0.353, 0.411 and 1.6 mg/kg), carbendazim (3.66, 4.46 and 5.50 mg/kg), deltamethrin (0.175, 0.165 and 0.905 mg/kg), fluquinconazole (1.94 mg/kg), lambda-cyhalothrin (0.285 and 0.35 mg/kg) and spiroxamine (3.07 and 3.13 mg/kg).

All assigned values for the pesticides can be seen in **Table 8**. The target standard deviation was obtained using a fixed FFP RSD value of 25%. In parallel, the robust standard deviation (Qn RSD) was calculated for informative purposes only. The range of Qn-RSD values was from 11 to 39 % but on average the Qn-RSD was 24%, and thus very close to the 25% FFP RSD used for the calculations.

Table 8 Assigned values in mg/kg and Fit For Purpose Relative Standard Deviation (FFP RSD) and Robust Relative Standard Deviation (Qn RSD) for the pesticides present in the test material.

Pesticides	MRRL (mg/kg)	Assigned value (mg/kg)	FFP RSD (%)	Qn RSD all (%)
Azoxystrobin	0.01	0.316	25	28
Carbaryl	0.01	0.160	25	23
Carbendazim	0.01	1.28	25	29
Chlorpyrifos-methyl	0.01	0.125	25	23
Deltametrin	0.01	0.061	25	29
Fenitrothion	0.01	0.188	25	20
Fenpropimorph	0.01	2.10	25	26
Fluquinconazole	0.01	0.74	25	27
Flutriafol	0.01	2.18	25	24
Isoproturon	0.01	0.164	25	19
Kresoxim-methyl	0.01	0.396	25	23
Lambda-cyhalothrin	0.01	0.065	25	29
Malathion SUM	0.01	0.108	25	22
- Malathion	0.01	0.109	25	24
Pirimiphos-methyl	0.01	0.078	25	25
Spiroxamine	0.01	1.10	25	35
Triadimenol and triadimefon	0.01	1.63	25	24
- Triadimenol	0.01	1.62	25	26
2,4-D, free acid	0.02	0.355	25	13
2,4-D, foll. alk. hydr.	0.02	0.367	25	11
Mepiquat	0.02	3.87	25	34
Glyphosate	0.02	0.085	25	23

4.3 Assessment of laboratory performance

4.3.1 z-Scores

The z-scores have been calculated for the quantified pesticides using the FFP RSD of 25 %. **Table 9** shows an overview of the results and **Table 10**, **Table 11**, **Table 12**, **Table 13**, **Table 14** and **Table 15** shows the individual results and z-scores for each laboratory and pesticide together with the assigned value. A graphical representation of the z-scores can be seen in **Appendix 6** and the histograms showing the distribution of the results in **Appendix 5**.

Table 9 Number of acceptable, questionable, unacceptable z-scores and false negatives. The unacceptable z-scores includes the false negatives.

Pesticides	Acceptable z-scores	Questionable z-scores	Unacceptable z-scores	False negatives
Azoxystrobin	84	7	6	1
Carbaryl	85	2	7	1
Carbendazim	68	7	3	
Chlorpyrifos-methyl	99	5	6	4
Deltametrin	69	8	27	22
Fenitrothion	98	8		1
Fenpropimorph	67	6	3	1
Fluquinconazole	65	5	5	2
Flutriafol	64	4	5	
Isoproturon	60	1	4	2
Kresoxim-methyl	100	6	2	
Lambda-cyhalothrin	83	6	11	3
Malathion SUM	74	4	6	5
- Malathion	97	8	4	1
Pirimiphos-methyl	100	6	2	
Spiroxamine	64	4	8	
Triadimenol and triadimefon	70	7	10	9
- Triadimenol	76	8	6	5
2,4-D, free acid	33	1		
2,4-D, foll. alk. hydr.	19		2	1
Mepiquat	34	2	1	2
Glyphosate	13		2	1

For carbaryl, chlorpyrifos-methyl, fenitrothion, flutriafol, isoproturon, kresoxim-methyl and pirimiphos-methyl acceptable results were obtained by 90-94 % of the laboratories. For azoxystrobin, carbendazim, fenpropimorph, fluquinconazole, lambda-cyhalothrin, malathion, spiroxamine and triadimenol acceptable results were obtained by 82-89 % of the laboratories. However, for deltamethrin acceptable z-scores were obtained by only 66 % of the laboratories. For the optional SRM pesticides acceptable z-scores were obtained by 87-97 % of the participants.

Table 10 Results of azoxystrobin, carbaryl, carbendazim and chlorpyrifos-methyl in mg/kg and their calculated z-scores using FFP RSD 25 %.

Laboratory code	Azoxystrobin	Z-scores (FFP RSD (25%))		Z-scores (FFP RSD (25%))		Z-scores (FFP RSD (25%))		Chlorpyrifos-methyl	Z-scores (FFP RSD (25%))		
		Carbaryl		Carbendazim		Chlorpyrifos-methyl					
		MRRL	0.01	0.01	0.01	0.01	0.01				
Assigned value	0.316										
001	0.37	0.7	0.16	0.0	4.46	9.9	0.16	1.1			
002	0.337	0.3	0.165	0.1	1.42	0.4	0.13	0.2			
003	0.31	-0.1	0.225	1.6	1.57	0.9	0.169	1.4			
004	0.403	1.1	0.151	-0.2	2.06	2.4	0.133	0.3			
005	ND	-3.9	0.112	-1.2	0.571	-2.2	0.111	-0.4			
006	0.362	0.6	0.152	-0.2	1.68	1.3	0.108	-0.5			
007	0.393	1.0	0.185	0.6	0.535	-2.3	0.146	0.7			
008	0.363	0.6	0.19	0.8	1.01	-0.8	0.127	0.1			
009											
010	0.382	0.8	0.169	0.2	1.29	0.0	ND	-3.7			
011	0.29	-0.3	0.19	0.8	0.526	-2.4	0.09	-1.1			
012	0.27	-0.6	0.174	0.4	1.35	0.2	0.123	-0.1			
013			0.191	0.8			0.114	-0.4			
014	0.35	0.4	0.11	-1.3	0.864	-1.3	0.125	0.0			
015	0.309	-0.1	0.151	-0.2	1.33	0.2	0.152	0.9			
016	0.467	1.9	0.092	-1.7	0.993	-0.9	0.17	1.4			
017	0.272	-0.6	0.165	0.1	1.267	0.0	0.13	0.2			
018	0.277	-0.5	0.167	0.2	1.246	-0.1	0.115	-0.3			
019	0.354	0.5	0.193	0.8	1.1	-0.6	0.167	1.3			
020	0.36	0.6	0.3	3.5	0.856	-1.3	0.141	0.5			
021	0.0847	-2.9					0.0798	-1.4			
022	0.327	0.1	0.159	0.0	1.02	-0.8	0.131	0.2			
023	0.316	0.0	0.174	0.4	1.88	1.9	0.125	0.0			
024	0.3	-0.2	0.236	1.9	1.3	0.1	0.108	-0.5			
025	0.325	0.1	0.168	0.2	1.376	0.3	0.118	-0.2			
026	0.316	0.0	0.11	-1.3	1.33	0.2	0.136	0.4			
027	0.31	-0.1	0.18	0.5	0.94	-1.1	ND	-3.7			
028	0.33	0.2	0.178	0.5	1.5	0.7	0.124	0.0			
029	0.191	-1.6	0.189	0.7	1.41	0.4	0.0674	-1.8			
030	0.38	0.8	0.173	0.3	1.51	0.7	0.205	2.6			
031											

Laboratory code	Azoxystrobin	Z-scores (FFP RSD (25%))	Carbaryl	Z-scores (FFP RSD (25%))	Carbendazim	Z-scores (FFP RSD (25%))	Chlorpyrifos-methyl	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01	
Assigned value	0.316		0.160		1.28		0.125	
032	0.374	0.7	0.18	0.5	1.32	0.1	0.144	0.6
033	0.448	1.7	0.407	6.2	1.28	0.0	0.17	1.4
034	0.213	-1.3	0.172	0.3	1.43	0.5	0.108	-0.5
035							0.1	-0.8
036	0.37	0.7	0.189	0.7	1.53	0.8	0.126	0.0
037	0.265	-0.6	0.142	-0.5	1.47	0.6	0.107	-0.6
038	0.18	-1.7	0.14	-0.5	1.59	1.0	0.09	-1.1
039	0.214	-1.3	0.11	-1.3			0.082	-1.4
040	0.344	0.4	0.164	0.1	1.41	0.4	0.142	0.5
041	0.158	-2.0	0.222	1.6	1.34	0.2	0.134	0.3
042	0.387	0.9	0.217	1.4	1.39	0.3	0.123	-0.1
044	0.402	1.1	0.183	0.6			0.128	0.1
045							0.158	1.1
046	0.185	-1.7	0.124	-0.9			0.061	-2.0
047	0.307	-0.1	0.123	-0.9			0.142	0.5
048	0.33	0.2	0.191	0.8	0.86	-1.3	0.172	1.5
049								
050	0.305	-0.1	0.155	-0.1	1.44	0.5	0.142	0.5
051	0.419	1.3	0.156	-0.1	0.994	-0.9	0.117	-0.3
052	0.135	-2.3					0.096	-0.9
053	0.203	-1.4					0.107	-0.6
054			0.0672	-2.3			0.117	-0.3
055	0.33	0.2	0.15	-0.3	1.85	1.8	0.12	-0.2
056	0.352	0.5	0.151	-0.2	1.14	-0.4	0.133	0.3
057	0.324	0.1	0.161	0.0	1.36	0.3	0.108	-0.5
058	9.92	>100	0.118	-1.1			0.106	-0.6
059							0.12	-0.2
060	0.217	-1.3	0.197	0.9	1.33	0.2	0.125	0.0
061	0.47	1.9	0.142	-0.5	1.46	0.6	0.146	0.7
062	0.397	1.0	0.176	0.4	1.32	0.1	0.162	1.2
063	0.245	-0.9	0.155	-0.1	1.48	0.6	0.131	0.2
064	0.356	0.5	0.142	-0.5	1.42	0.4	0.133	0.3
065	0.367	0.6	0.16	0.0	1.31	0.1	0.178	1.7

Laboratory code	Azoxystrobin	Z-scores (FFP RSD (25%))	Carbaryl	Z-scores (FFP RSD (25%))	Carbendazim	Z-scores (FFP RSD (25%))	Chlorpyrifos-methyl	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01	
Assigned value	0.316		0.160		1.28		0.125	
066	0.64	4.1	1.74	39.5			ND	-3.7
067	0.39	0.9	0.166	0.2			0.13	0.2
068	0.251	-0.8	0.101	-1.5			0.107	-0.6
069	0.383	0.8	0.144	-0.4	1.09	-0.6	0.134	0.3
070	0.076	-3.0	0.1	-1.5			0.129	0.1
071	0.179	-1.7	0.107	-1.3	0.998	-0.9	0.0882	-1.2
072								
073	0.322	0.1	0.147	-0.3	1.19	-0.3	0.137	0.4
074	0.0505	-3.4	0.0821	-1.9			0.1	-0.8
075	0.231	-1.1	0.168	0.2	0.975	-1.0	0.122	-0.1
076	0.315	0.0	0.095	-1.6			0.115	-0.3
077	0.321	0.1	0.187	0.7	1.618	1.1	0.135	0.3
078								
079	0.235	-1.0	0.15	-0.3	1.7	1.3	0.122	-0.1
080	0.27	-0.6	0.13	-0.8			0.12	-0.2
081							0.145	0.6
082	0.286	-0.4			0.995	-0.9	0.107	-0.6
083	0.3	-0.2	0.15	-0.3	0.58	-2.2	0.07	-1.8
084	0.33	0.2	0.157	-0.1	1.34	0.2	0.0901	-1.1
085	0.402	1.1	0.169	0.2	1.23	-0.2	0.127	0.1
086							0.168	1.4
087	0.34	0.3	0.19	0.8	1.29	0.0	0.16	1.1
088	0.417	1.3			1.178	-0.3	0.142	0.5
089	0.2	-1.5	0.16	0.0	0.95	-1.0	0.1	-0.8
090	0.306	-0.1	ND	-3.7	1.63	1.1	0.07	-1.8
091	0.311	-0.1	0.155	-0.1	0.879	-1.3	0.138	0.4
092	0.503	2.4	0.171	0.3	0.647	-2.0	0.228	3.3
093	0.27	-0.6	0.158	-0.1	1.45	0.5	0.112	-0.4
094								
095	0.349	0.4	0.165	0.1	1.04	-0.8	0.133	0.3
096							0.193	2.2
097	0.351	0.4			0.984	-0.9	0.1	-0.8
098	0.195	-1.5	0.1	-1.5	0.869	-1.3	0.107	-0.6

Laboratory code	Azoxystrobin	Z-scores (FFP RSD (25%))	Carbaryl	Z-scores (FFP RSD (25%))	Carbendazim	Z-scores (FFP RSD (25%))	Chlorpyrifos-methyl	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01	
Assigned value	0.316		0.160		1.28		0.125	
099					1.06	-0.7	0.14	0.5
100	0.379	0.8	0.237	1.9	3.66	7.4	0.069	-1.8
101	0.0979	-2.8	0.158	-0.1			0.284	5.1
102	0.38	0.8	0.31	3.8			0.09	-1.1
103	0.253	-0.8	0.411	6.3			0.128	0.1
105							0.11	-0.5
106	0.344	0.4	0.186	0.7	0.721	-1.7	0.085	-1.3
107								
108	0.189	-1.6	0.141	-0.5			0.0755	-1.6
109			0.222	1.6			0.126	0.0
110			0.0936	-1.7			0.114	-0.4
111							0.11	-0.5
112	0.35	0.4	0.28	3.0	0.65	-2.0		
113	0.313	0.0	0.214	1.4	1.09	-0.6	0.112	-0.4
114	0.084	-2.9	0.026	-3.4	0.38	-2.8	0.041	-2.7
115	0.675	4.5	0.139	-0.5	0.407	-2.7	0.171	1.5
116	0.27	-0.6	0.141	-0.5	0.975	-1.0	0.06	-2.1
117	0.086	-2.9	0.126	-0.9	0.942	-1.1	ND	-3.7
119	0.303	-0.2	0.145	-0.4	5.5	13.2	0.146	0.7
121	0.201	-1.5					0.12	-0.2
122							0.098	-0.9

Table 11 Results for deltamethrin, fenitrothion, fenpropimorph and fluquinconazole in mg/kg and their calculated z-scores using FFP RSD 25 %

Laboratory code	Deltamethrin (cis)	Z-scores (FFP RSD (25%))	Fenitrothion	Z-scores (FFP RSD (25%))	Fenpropimorph	Z-scores (FFP RSD (25%))	Fluquinconazole	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01	
Assigned value	0.061		0.188		2.10		0.740	
001	ND	-3.3	0.23	0.9	ND	-4.0	1.27	2.9
002	0.0787	1.2	0.195	0.1	2.44	0.6	0.996	1.4
003	0.905	55.8	0.19	0.0	2.02	-0.2	0.93	1.0
004	0.062	0.1	0.192	0.1	1.38	-1.4	0.805	0.4
005			0.181	-0.1	1.766	-0.6	0.365	-2.0
006	0.06	0.0	0.178	-0.2	1.75	-0.7	0.658	-0.4
007	ND	-3.3	0.063	-2.7	1.68	-0.8	1.66	5.0
008	0.08	1.3	0.16	-0.6	2.17	0.1	0.825	0.5
009								
010	0.098	2.5	0.233	1.0	1.94	-0.3	ND	-3.9
011	ND	-3.3	0.165	-0.5	2.38	0.5	0.407	-1.8
012	0.0699	0.6	0.194	0.1	2.78	1.3	0.72	-0.1
013	0.027	-2.2	0.223	0.7				
014	0.065	0.3	0.198	0.2	2.57	0.9	0.814	0.4
015	0.0752	1.0	0.15	-0.8	1.88	-0.4	0.885	0.8
016	0.175	7.6	0.287	2.1	1.89	-0.4	0.823	0.4
017	0.062	0.1			2.69	1.1		
018	ND	-3.3	0.19	0.0	1.787	-0.6	0.63	-0.6
019	0.061	0.0	0.261	1.6	2.48	0.7	0.968	1.2
020	0.1	2.6			2.21	0.2	0.985	1.3
021	0.0614	0.1	0.104	-1.8				
022	ND	-3.3	0.204	0.3	2.42	0.6	0.752	0.1
023	ND	-3.3	0.184	-0.1	2.37	0.5	0.739	0.0
024	ND	-3.3	0.184	-0.1	2.1	0.0	0.563	-1.0
025	ND	-3.3	0.114	-1.6	2.947	1.6	0.745	0.0
026	0.063	0.2	0.213	0.5	2.88	1.5	0.811	0.4
027	0.069	0.6	0.19	0.0	2.1	0.0	0.7	-0.2
028	0.059	-0.1	0.17	-0.4	2.26	0.3	0.835	0.5
029	0.0577	-0.2	0.131	-1.2	2.08	0.0	0.554	-1.0
030	0.0605	0.0	0.22	0.7	2.05	-0.1	1.05	1.7
031			0.218	0.6				

Laboratory code	Deltamethrin (cis)	Z-scores (FFP RSD (25%))	Fenitrothion	Z-scores (FFP RSD (25%))	Fenpropimorph	Z-scores (FFP RSD (25%))	Fluquinconazole	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01	
Assigned value	0.061		0.188		2.10		0.740	
032	0.058	-0.2	0.21	0.5	2.76	1.3	1.08	1.8
033	0.062	0.1	0.27	1.7	2.55	0.9	0.904	0.9
034	0.045	-1.0	0.146	-0.9	2.06	-0.1	0.556	-1.0
035	ND	-3.3						
036	0.05	-0.7	0.161	-0.6	2.28	0.3	0.919	1.0
037	0.054	-0.4	0.172	-0.3	1.987	-0.2	0.684	-0.3
038	0.05	-0.7	0.15	-0.8	3.61	2.9	1.79	5.7
039	0.04	-1.4	0.194	0.1	0.983	-2.1	0.601	-0.8
040	0.039	-1.4	0.22	0.7	2.15	0.1	0.855	0.6
041	0.0471	-0.9	0.244	1.2			0.469	-1.5
042	0.058	-0.2	0.204	0.3	2.5	0.8	0.713	-0.1
044	0.0821	1.4	0.201	0.3				
045			0.168	-0.4				
046	0.034	-1.8	0.135	-1.1				
047	0.0495	-0.7	0.189	0.0	2.42	0.6	0.789	0.3
048	0.084	1.6	0.307	2.5	1.051	-2.0	0.778	0.2
049			0.208	0.4				
050	0.0849	1.6	0.149	-0.8	2.41	0.6	0.707	-0.2
051	0.073	0.8	0.209	0.4	2.03	-0.1	0.736	0.0
052	0.055	-0.4	0.183	-0.1	2.36	0.5	0.488	-1.4
053	0.054	-0.4	0.168	-0.4	2.383	0.5	0.693	-0.3
054								
055	0.054	-0.4	0.19	0.0	1.95	-0.3	0.73	-0.1
056	ND	-3.3	0.207	0.4	1.96	-0.3	0.775	0.2
057	0.061	0.0	0.169	-0.4	1.96	-0.3	0.93	1.0
058	0.058	-0.2						
059			0.18	-0.2	2	-0.2	0.7	-0.2
060	0.057	-0.2	0.189	0.0				
061	ND	-3.3	0.186	0.0	2.72	1.2	0.457	-1.5
062	0.083	1.5	0.273	1.8	2.46	0.7	0.853	0.6
063	0.07	0.6	0.179	-0.2				
064	0.091	2.0	0.189	0.0	2.35	0.5	0.741	0.0
065	0.072	0.8	0.278	1.9	2.07	-0.1	0.803	0.3

Laboratory code	Deltamethrin (cis)	Z-scores (FFP RSD (25%))	Fenitrothion	Z-scores (FFP RSD (25%))	Fenpropimorph	Z-scores (FFP RSD (25%))	Fluquinconazole	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01	
Assigned value	0.061		0.188		2.10		0.740	
066	0.07	0.6	0.18	-0.2				
067	0.095	2.3	0.208	0.4				
068	0.05	-0.7	0.169	-0.4				
069	0.073	0.8	0.215	0.6	2.33	0.4	0.753	0.1
070	ND	-3.3	0.219	0.7	1.7	-0.8	0.522	-1.2
071	0.047	-0.9	0.133	-1.2	0.93	-2.2	0.494	-1.3
072			0.195	0.1				
073	0.0677	0.5	0.179	-0.2	1.79	-0.6	0.719	-0.1
074	0.0502	-0.7	0.141	-1.0	1.65	-0.9	0.26	-2.6
075	0.092	2.1	0.149	-0.8			0.612	-0.7
076	0.061	0.0	0.251	1.3	1.81	-0.6	0.612	-0.7
077	0.062	0.1	0.231	0.9	2.787	1.3	0.777	0.2
078								
079	0.05	-0.7	0.215	0.6	1.44	-1.3	0.616	-0.7
080	0.08	1.3	0.2	0.3				
081			0.196	0.2				
082	0.0504	-0.7	0.164	-0.5				
083	0.025	-2.3	0.13	-1.2	0.4	-3.2	0.25	-2.6
084	0.0391	-1.4	0.182	-0.1	1.93	-0.3	0.747	0.0
085	0.069	0.6	0.204	0.3	2.65	1.0	0.911	0.9
086			0.215	0.6				
087	0.061	0.0	0.21	0.5	2.2	0.2	0.84	0.5
088	0.052	-0.6	0.206	0.4				
089	ND	-3.3	0.14	-1.0	1	-2.1	0.52	-1.2
090	0.053	-0.5	0.1	-1.9				
091	0.0937	2.2	0.174	-0.3	1.99	-0.2	0.67	-0.4
092	0.116	3.7			3.45	2.6		
093	ND	-3.3	0.159	-0.6				
094			0.195	0.1				
095	ND	-3.3	0.203	0.3	2.44	0.6	0.733	0.0
096	0.054	-0.4	0.309	2.6				
097	0.047	-0.9	0.157	-0.7				
098	0.062	0.1	0.183	-0.1				

Laboratory code	Deltamethrin (cis)	Z-scores (FFP RSD (25%))	Fenitrothion	Z-scores (FFP RSD (25%))	Fenpropimorph	Z-scores (FFP RSD (25%))	Fluquinconazole	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01	
Assigned value	0.061		0.188		2.10		0.740	
099	0.059	-0.1	0.18	-0.2				
100	ND	-3.3	0.085	-2.2	2.11	0.0		
101	0.0423	-1.2	0.292	2.2				
102	0.11	3.3	0.16	-0.6				
103	ND	-3.3	0.219	0.7	2.76	1.3	1.94	6.5
105	0.07	0.6	0.19	0.0	1.3	-1.5	0.64	-0.5
106	ND	-3.3	0.135	-1.1	1.207	-1.7	ND	-3.9
107								
108	ND	-3.3	0.215	0.6				
109	0.042	-1.2						
110	0.036	-1.6	0.177	-0.2				
111	0.07	0.6						
112	0.3	15.8	0.17	-0.4	2.45	0.7	0.82	0.4
113	0.05	-0.7	0.176	-0.3	2.05	-0.1	0.902	0.9
114	0.033	-1.8	0.077	-2.4	0.36	-3.3	0.45	-1.6
115	0.076	1.0	0.168	-0.4	3.22	2.1	0.852	0.6
116	ND	-3.3	0.116	-1.5				
117	ND	-3.3	ND	-3.8	1.28	-1.6	0.364	-2.0
119	ND	-3.3	0.144	-0.9	2.403	0.6	0.684	-0.3
121	0.052	-0.6	0.175	-0.3				
122			0.138	-1.1				

Table 12 Results of flutriafol, isoproturon, kresoxim-methyl and lambda-cyhalothrin in mg/kg and their calculated z-scores using FFP RSD 25%.

Laboratory code	Flutriafol	Z-scores (FFP RSD (25%))	Isoproturon	Z-scores (FFP RSD (25%))	Kresoxim-methyl	Z-scores (FFP RSD (25%))	Lambda-cyhalothrin	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01	
Assigned value	2.18		0.164		0.396		0.065	
001	3.45	2.3	0.11	-1.3	0.5	1.1	0.09	1.5
002	2.39	0.4	0.17	0.1	0.404	0.1	0.0739	0.5
003	2.78	1.1	0.18	0.4	0.411	0.2	0.102	2.3
004	2.13	-0.1	0.217	1.3	0.423	0.3	0.065	0.0
005	0.354	-3.4	0.101	-1.5	0.253	-1.4	0.084	1.2
006	2.26	0.1	0.206	1.0	0.342	-0.5	0.095	1.8
007	2.99	1.5	0.161	-0.1	0.86	4.7	0.087	1.4
008	2.34	0.3	0.176	0.3	0.46	0.6	0.094	1.8
009								
010	2.03	-0.3	0.171	0.2	0.443	0.5	0.081	1.0
011	1.45	-1.3	0.14	-0.6	0.216	-1.8	0.047	-1.1
012	2.2	0.0	0.142	-0.5	0.344	-0.5	0.0783	0.8
013								
014	2.07	-0.2	0.11	-1.3	0.414	0.2	0.08	0.9
015	2.25	0.1	0.127	-0.9	0.371	-0.3	0.117	3.2
016	2.43	0.5	0.159	-0.1	0.486	0.9	0.115	3.1
017			0.176	0.3	0.434	0.4	0.063	-0.1
018	2.745	1.0	0.166	0.0	0.383	-0.1	0.08	0.9
019	1.18	-1.8	0.172	0.2	0.488	0.9	0.096	1.9
020	2.41	0.4	0.19	0.6	0.453	0.6	0.1	2.2
021								
022	1.96	-0.4	0.174	0.2	0.469	0.7	0.061	-0.2
023	2.17	0.0	0.175	0.3	0.379	-0.2		
024	1.76	-0.8	0.16	-0.1	0.365	-0.3	0.056	-0.6
025	2.375	0.4	0.175	0.3	0.411	0.2	0.063	-0.1
026	1.92	-0.5	0.147	-0.4	0.412	0.2	0.061	-0.2
027	1.8	-0.7	0.19	0.6	0.41	0.1	0.078	0.8
028	2.26	0.1	0.201	0.9	0.382	-0.1	0.059	-0.4
029	1.89	-0.5	0.165	0.0	0.293	-1.0	0.0533	-0.7
030	2.35	0.3	0.165	0.0	0.482	0.9	0.0855	1.3
031								

Laboratory code	Flutriafol	Z-scores (FFP RSD (25%))	Isoproturon	Z-scores (FFP RSD (25%))	Kresoxim-methyl	Z-scores (FFP RSD (25%))	Lambda-cyhalothrin	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01	
Assigned value	2.18		0.164		0.396		0.065	
032	2.19	0.0	0.176	0.3	0.447	0.5	0.09	1.5
033	2.48	0.6			0.45	0.5	0.055	-0.6
034	1.72	-0.8	0.156	-0.2	0.224	-1.7	0.054	-0.7
035								
036	2.42	0.4	0.205	1.0	0.411	0.2	0.065	0.0
037	2.43	0.5	0.168	0.1	0.374	-0.2	0.062	-0.2
038	6.19	7.4	0.18	0.4	0.26	-1.4	0.06	-0.3
039	1.12	-1.9	0.088	-1.9			0.041	-1.5
040	2.31	0.2	0.182	0.4	0.457	0.6	0.06	-0.3
041	1.14	-1.9	0.158	-0.1	0.301	-1.0	0.0731	0.5
042	2.84	1.2	0.191	0.7	0.689	3.0	0.082	1.0
044					0.471	0.8	0.0622	-0.2
045								
046			0.027	-3.3	0.211	-1.9	0.045	-1.2
047	2.19	0.0			0.455	0.6	0.0721	0.4
048	2.771	1.1	0.184	0.5	0.412	0.2	0.073	0.5
049								
050	2.23	0.1	0.162	0.0	0.378	-0.2	0.126	3.8
051	1.58	-1.1	0.184	0.5	0.479	0.8	0.083	1.1
052					0.32	-0.8	0.086	1.3
053					0.393	0.0	0.096	1.9
054								
055	2.06	-0.2	0.14	-0.6	0.34	-0.6	0.085	1.2
056	2.08	-0.2	0.144	-0.5	0.476	0.8	0.059	-0.4
057	1.69	-0.9	0.156	-0.2	0.386	-0.1	0.105	2.5
058							0.041	-1.5
059					0.32	-0.8	0.09	1.5
060					0.405	0.1	0.072	0.4
061	2.6	0.8	0.188	0.6	0.448	0.5	0.062	-0.2
062	2.45	0.5	0.11	-1.3	0.493	1.0	0.114	3.0
063					0.387	-0.1	0.076	0.7
064	3.26	2.0	0.146	-0.4	0.432	0.4	0.053	-0.7
065	2.3	0.2	0.203	1.0	0.456	0.6	0.0528	-0.8

Laboratory code	Flutriafol	Z-scores (FFP RSD (25%))	Isoproturon	Z-scores (FFP RSD (25%))	Kresoxim-methyl	Z-scores (FFP RSD (25%))	Lambda-cyhalothrin	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01	
Assigned value	2.18		0.164		0.396		0.065	
066							0.05	-0.9
067					0.454	0.6	0.061	-0.2
068					0.352	-0.4	0.059	-0.4
069	2.05	-0.2	0.14	-0.6	0.373	-0.2	0.102	2.3
070					0.389	-0.1	ND	-3.4
071	1.38	-1.5	0.102	-1.5	0.276	-1.2	0.0518	-0.8
072								
073	2.12	-0.1	0.154	-0.2	0.41	0.1	0.0793	0.9
074					0.15	-2.5	0.0571	-0.5
075	1.17	-1.9			0.386	-0.1	0.145	4.9
076					0.395	0.0	0.062	-0.2
077	2.007	-0.3	0.215	1.2	0.436	0.4	0.073	0.5
078								
079	1.22	-1.8	0.289	3.0	0.372	-0.2	0.045	-1.2
080					0.34	-0.6	0.06	-0.3
081								
082					0.336	-0.6	0.0642	0.0
083	0.18	-3.7	ND	-3.8	0.57	1.8	0.06	-0.3
084	2.22	0.1	0.156	-0.2	0.353	-0.4	0.0696	0.3
085	2.57	0.7	0.162	0.0	0.511	1.2	0.094	1.8
086								
087	2	-0.3	0.13	-0.8	0.47	0.7	0.082	1.0
088					0.395	0.0	0.063	-0.1
089	2.31	0.2	0.12	-1.1	0.22	-1.8	0.05	-0.9
090			0.132	-0.8	0.305	-0.9		
091	1.89	-0.5	0.145	-0.5	0.404	0.1	0.107	2.6
092	2.51	0.6			0.598	2.0	0.114	3.0
093					0.39	-0.1	0.066	0.1
094								
095	2.09	-0.2	0.142	-0.5	0.436	0.4	0.062	-0.2
096							ND	-3.4
097					0.376	-0.2	0.061	-0.2
098					0.24	-1.6	0.046	-1.2

Laboratory code	Flutriafol	Z-scores (FFP RSD (25%))		Isoproturon		Z-scores (FFP RSD (25%))		Kresoxim-methyl		Z-scores (FFP RSD (25%))		Lambda-cyhalothrin		Z-scores (FFP RSD (25%))	
MRRL	0.01			0.01				0.01				0.01			
Assigned value	2.18			0.164				0.396				0.065			
099													0.072	0.4	
100	2.71	1.0						0.571	1.8	0.032	-2.0				
101								0.185	-2.1	0.0503	-0.9				
102								0.47	0.7	0.05	-0.9				
103	2.33	0.3						0.309	-0.9	0.0337	-1.9				
105								0.29	-1.1	0.09	1.5				
106	2.07	-0.2	0.12	-1.1	0.274			-1.2	0.078	0.078	0.8				
107															
108						0.322		-0.7	0.047	0.047	-1.1				
109										0.08	0.9				
110										0.0636	-0.1				
111										0.07	0.3				
112	1.45	-1.3	0.28	2.8	0.45			0.5	0.35	0.35	17.5				
113	2.14	-0.1	0.164	0.0	0.463			0.7	0.054	0.054	-0.7				
114	0.35	-3.4			0.18			-2.2							
115	2.8	1.1			0.483			0.9	0.06	0.06	-0.3				
116	1.87	-0.6			0.396			0.0	0.285	0.285	13.5				
117	0.295	-3.5	ND	-3.8	0.198			-2.0	ND	ND	-3.4				
119	2.718	1.0	0.21	1.1	0.465			0.7	0.059	0.059	-0.4				
121					0.327			-0.7	0.053	0.053	-0.7				
122															

Table 13 Results of malathion-SUM, -malathion, -malaoxon and pirimiphos-methyl in mg/kg and their calculated z-scores using FFP RSD 25%.

Laboratory code	Malathion-SUM	Z-scores (FFP RSD (25%))	-malathion	Z-scores (FFP RSD (25%))	-malaoxon	Z-scores (FFP RSD (25%))	Pirimiphos-methyl	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01	
Assigned value	0.108		0.109				0.078	
001	ND	-3.6	0.14	1.2			0.1	1.1
002	0.111	0.1	0.111	0.1			0.0802	0.1
003	0.119	0.4	0.119	0.4			0.121	2.2
004	0.108	0.0	0.108	0.0			0.097	1.0
005							0.081	0.2
006	0.092	-0.6	0.092	-0.6			0.062	-0.8
007	0.074	-1.3	0.074	-1.3			0.118	2.1
008	0.127	0.7	0.127	0.7			0.103	1.3
009								
010	0.127	0.7	0.127	0.7			0.078	0.0
011	0.08	-1.0	0.08	-1.1			0.07	-0.4
012	0.0895	-0.7	0.0895	-0.7			0.075	-0.2
013			0.041	-2.5			0.086	0.4
014	0.11	0.1	0.11	0.1			0.071	-0.4
015	0.104	-0.1	0.104	-0.2			0.0829	0.3
016	0.162	2.0	0.162	2.0			0.114	1.8
017	0.104	-0.1	0.104	-0.2			0.083	0.3
018	0.09	-0.7	0.09	-0.7			0.074	-0.2
019	0.122	0.5	0.122	0.5			0.099	1.1
020	0.115	0.3	0.1115	0.1			0.084	0.3
021			0.0359	-2.7			0.0344	-2.2
022	0.102	-0.2	0.102	-0.2			0.092	0.7
023			0.109	0.0			0.077	-0.1
024	0.075	-1.2	0.075	-1.2			0.07	-0.4
025	0.065	-1.6	0.065	-1.6			0.066	-0.6
026	0.122	0.5	0.117	0.3	0.005		0.083	0.3
027	0.1	-0.3	0.1	-0.3			0.084	0.3
028	0.114	0.2	0.114	0.2			0.076	-0.1
029	0.047	-2.3	0.047	-2.3			0.0443	-1.7
030	0.12	0.4	0.12	0.4			0.099	1.1
031			0.128	0.7				

Laboratory code	Malathion-SUM	Z-scores (FFP RSD (25%))	-malathion	Z-scores (FFP RSD (25%))	-malaoxon	Z-scores (FFP RSD (25%))	Pirimiphos-methyl	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01	
Assigned value	0.108		0.109				0.078	
032	0.134	1.0	0.134	0.9			0.084	0.3
033	0.147	1.4	0.147	1.4			0.08	0.1
034	0.075	-1.2	0.075	-1.2			0.068	-0.5
035								
036	0.115	0.3	0.115	0.2			0.081	0.2
037	0.082	-1.0	0.082	-1.0			0.069	-0.5
038	0.05	-2.1	0.05	-2.2			0.06	-0.9
039	0.121	0.5	0.085	-0.9	0.034		0.048	-1.5
040	0.096	-0.4	0.096	-0.5			0.078	0.0
041	0.0733	-1.3	0.0733	-1.3			0.093	0.8
042	0.114	0.2	0.114	0.2			0.087	0.5
044			0.119	0.4			0.0791	0.1
045			0.114	0.2			0.095	0.9
046	ND	-3.6	0.02	-3.3			0.043	-1.8
047	0.118	0.4	0.118	0.4			0.0837	0.3
048	0.139	1.1	0.13	0.8	0.0081		0.089	0.6
049			0.115	0.2			0.091	0.7
050	0.102	-0.2	0.102	-0.2			0.0785	0.0
051	0.114	0.2	0.114	0.2			0.101	1.2
052			0.078	-1.1			0.072	-0.3
053			0.088	-0.8			0.065	-0.7
054								
055	0.11	0.1	0.11	0.1			0.082	0.2
056	0.111	0.1	0.111	0.1			0.0986	1.1
057	0.105	-0.1	0.105	-0.1			0.083	0.3
058	0.15	1.6	0.15	1.5			0.048	-1.5
059			0.08	-1.1			0.06	-0.9
060			0.13	0.8			0.075	-0.2
061	0.111	0.1	0.111	0.1			0.107	1.5
062	0.14	1.2	0.14	1.2			0.128	2.6
063	0.081	-1.0	0.081	-1.0			0.077	-0.1
064	0.112	0.1	0.112	0.1			0.075	-0.2
065	0.131	0.9	0.131	0.8			0.102	1.2

Laboratory code	Malathion-SUM	Z-scores (FFP RSD (25%))	-malathion	Z-scores (FFP RSD (25%))	-malaoxon	Z-scores (FFP RSD (25%))	Pirimiphos-methyl	Z-scores (FFP RSD (25%))
MRRL	0.01		0.01		0.01		0.01	
Assigned value	0.108		0.109				0.078	
066			0.11	0.1			0.06	-0.9
067	0.104	-0.1	0.104	-0.2			0.078	0.0
068			0.105	-0.1			0.074	-0.2
069	0.114	0.2	0.114	0.2			0.077	-0.1
070	0.097	-0.4	0.097	-0.4			0.1	1.1
071	0.066	-1.6	0.066	-1.6			0.0508	-1.4
072			0.112	0.1				
073	0.102	-0.2	0.102	-0.2			0.0757	-0.1
074	0.0511	-2.1	0.0511	-2.1			0.0521	-1.3
075	0.092	-0.6	0.092	-0.6			0.061	-0.9
076	0.121	0.5	0.121	0.5			0.151	3.7
077	0.117	0.3	0.117	0.3			0.084	0.3
078								
079	0.106	-0.1	0.106	-0.1			0.087	0.5
080			0.12	0.4			0.16	4.2
081			0.118	0.4			0.105	1.4
082	0.0778	-1.1	0.0778	-1.1			0.0583	-1.0
083	0.03	-2.9	0.03	-2.9			0.055	-1.2
084	0.0762	-1.2	0.0762	-1.2			0.0652	-0.7
085	0.129	0.8	0.129	0.8			0.094	0.8
086			0.165	2.1			0.1	1.1
087	0.11	0.1	0.11	0.1			0.09	0.6
088	0.098	-0.4	0.098	-0.4			0.062	-0.8
089	0.1	-0.3	0.1	-0.3			0.06	-0.9
090	0.08	-1.0	0.08	-1.1			0.052	-1.3
091	0.088	-0.7	0.088	-0.8			0.0707	-0.4
092	0.129	0.8	0.129	0.8			0.0941	0.8
093	0.117	0.3	0.117	0.3			0.069	-0.5
094			0.115	0.2				
095	0.108	0.0	0.108	0.0			0.075	-0.2
096	ND	-3.6	0.155	1.7				
097			0.11	0.1			0.057	-1.1
098			0.044	-2.4			0.077	-0.1

Laboratory code	Malathion-SUM	Z-scores (FFP RSD (25%))	-malathion		Z-scores (FFP RSD (25%))	-malaoxon		Z-scores (FFP RSD (25%))	Pirimiphos-methyl	Z-scores (FFP RSD (25%))
			MRRL	0.01		Assigned value	0.108	0.01	0.109	
099	ND	-3.6	0.102	-0.2						0.082
100	0.147	1.4	0.147	1.4						0.0954
101			0.152	1.6						0.126
102			0.1	-0.3						0.05
103	0.101	-0.3	0.101	-0.3						0.094
105	0.09	-0.7								0.06
106	0.096	-0.4	0.076	-1.2	0.02					0.053
107										
108	0.09	-0.7	0.09	-0.7						0.0535
109			0.093	-0.6						0.062
110	0.0946	-0.5	0.0946	-0.5						0.0788
111										0.08
112										
113	0.111	0.1	0.111	0.1						0.075
114			0.027	-3.0						0.04
115	0.193	3.1	0.193	3.1						0.0812
116			0.051	-2.1						0.053
117	ND	-3.6	ND	-3.6						0.056
119	0.119	0.4	0.119	0.4						0.078
121	0.141	1.2	0.141	1.2						0.073
122			0.087	-0.8						0.125
										2.4

Table 14 Results of spiroxamin, 'triadimenol and triadimefon', '-triadimenol' and '-triadimefon' in mg/kg and their calculated z-scores using FFP RSD 25%.

Laboratory code	Spiroxamine	Z-scores (FFP RSD (25%))	Triadimenol and triadimefon		Z-scores (FFP RSD (25%))	-triadimenol	Z-scores (FFP RSD (25%))	-triadimefon	Z-scores (FFP RSD (25%))
			MRRL	0.01					
			Assigned value	1.10					
001	3.07	7.2	ND	-4.0	2.48	2.1			
002	1.1	0.0	1.71	0.2	1.71	0.2			
003	1.11	0.1	2.47	2.1	2.47	2.1			
004	1.87	2.8	1.53	-0.2	1.53	-0.2			
005	0.564	-1.9	0.602	-2.5	0.602	-2.5			
006	1.007	-0.3	1.51	-0.3	1.51	-0.3			
007	0.812	-1.0	2.08	1.1	2.05	1.1	0.031		
008	0.881	-0.8	1.83	0.5	1.83	0.5			
009									
010	1.06	-0.1	1.61	0.0	1.61	0.0			
011	0.935	-0.6	0.835	-1.9	0.835	-1.9			
012	1.04	-0.2	1.45	-0.4	1.45	-0.4			
013									
014	0.928	-0.6	1.65	0.1	1.65	0.1			
015	0.968	-0.5	1.79	0.4	1.78	0.4	0.0112		
016	3.13	7.4	1.86	0.6	1.84	0.5	0.0166		
017	1.186	0.3	1.762	0.3	1.752	0.3	0.01		
018	1.255	0.6	1.458	-0.4	1.458	-0.4			
019	1.28	0.7	2.39	1.9	2.38	1.9	0.011		
020	1.21	0.4	1.95	0.8	1.95	0.8			
021									
022	1.5	1.5	2.13	1.2	2.13	1.3			
023	1.12	0.1	1.63	0.0	1.63	0.0			
024	0.883	-0.8	1.45	-0.4	1.45	-0.4			
025	1.525	1.6	1.802	0.4	1.802	0.4			
026	1.18	0.3	1.8	0.4	1.79	0.4	0.011		
027	1.5	1.5	1.5	-0.3	1.5	-0.3			
028	1.11	0.1	1.54	-0.2	1.54	-0.2			
029	0.867	-0.8	1.2	-1.0	1.2	-1.0			
030	1.03	-0.2	1.97	0.8	1.97	0.9			
031									

Laboratory code	Spiroxamine	Z-scores (FFP RSD (25%))	Triademefon and triademetron		Z-scores (FFP RSD (25%))	-triademefon	Z-scores (FFP RSD (25%))	-triademetron	Z-scores (FFP RSD (25%))
			0.01	1.63					
MRRL	0.01								
Assigned value	1.10								
032	1.27	0.6	1.78	0.4	1.78	0.4			
033	1.364	1.0	1.378	-0.6	1.378	-0.6			
034	0.824	-1.0	1.45	-0.4	1.45	-0.4			
035									
036	1.14	0.2	2.54	2.3	2.54	2.3			
037	0.956	-0.5	1.144	-1.2	1.144	-1.2			
038	0.98	-0.4	0.81	-2.0	0.81	-2.0			
039	0.882	-0.8							
040	0.456	-2.3	1.75	0.3	0		1.75		
041	0.92	-0.6	1.62	0.0	1.62	0.0			
042	1.05	-0.2	2.15	1.3	2.15	1.3			
044			1.3	-0.8	1.3	-0.8			
045									
046									
047	1.35	0.9	1.79	0.4	1.79	0.4			
048	1.555	1.7	1.73	0.3	1.721	0.2	0.0092		
049									
050	1.24	0.5	1.56	-0.2	1.56	-0.1			
051	1.29	0.7	1.92	0.7	1.91	0.7	0.010		
052									
053			1.124	-1.2	1.124	-1.2			
054									
055	0.87	-0.8	1.52	-0.3	1.52	-0.2			
056	1.45	1.3	1.84	0.5	1.84	0.5			
057	1.13	0.1	1.49	-0.3	1.49	-0.3			
058			0.79	-2.1	0.79	-2.0			
059									
060			1.61	0.0	1.61	0.0			
061	1.64	2.0	1.79	0.4	1.79	0.4			
062	0.52	-2.1	1.71	0.2	1.69	0.2	0.020		
063			1.61	0.0	1.6	0.0	0.014		
064	1.37	1.0	2.87	3.1	2.86	3.1	0.010		
065	1.56	1.7	1.69	0.2	1.68	0.1	0.0100		

Laboratory code	Spiroxamine	Z-scores (FFP RSD (25%))	Triademefon and triadimenol		Z-scores (FFP RSD (25%))	-triademenol	Z-scores (FFP RSD (25%))	-triademefon	Z-scores (FFP RSD (25%))
			MRRL	0.01					
			Assigned value	1.10					
066			ND	-4.0	1.6	0.0			
067			1.29	-0.8	1.29	-0.8			
068	0.224	-3.2							
069	1.12	0.1	1.7	0.2	1.69	0.2	0.011		
070			1.004	-1.5	1.004	-1.5			
071			1.01	-1.5	1	-1.5	0.0053		
072									
073	0.878	-0.8	1.54	-0.2	1.54	-0.2			
074			1.52	-0.3	1.52	-0.2			
075	0.66	-1.6	1.01	-1.5	1.01	-1.5			
076	1.01	-0.3	1.25	-0.9	ND	-4.0			
077	1.467	1.4	1.825	0.5	1.825	0.5			
078									
079	0.635	-1.7	1.28	-0.8	1.28	-0.8			
080	1.59	1.8			1.73	0.3			
081									
082	0.76	-1.2	ND	-4.0	ND	-4.0			
083	0.9	-0.7	ND	-4.0	0.7	-2.3			
084	1.34	0.9	1.66	0.1	1.66	0.1			
085	1.26	0.6	2.08	1.1	2.08	1.1			
086									
087	1.2	0.4	1.86	0.6	1.85	0.6	0.01		
088			1.33	-0.7	1.33	-0.7			
089	0.88	-0.8	2.3	1.7	2.3	1.7			
090	1.44	1.3							
091	1.05	-0.2	1.63	0.0	1.62	0.0	0.0087		
092					1.67	0.1			
093	0.848	-0.9	1.43	-0.5	1.43	-0.5			
094									
095	1.32	0.8	1.86	0.6	1.86	0.6			
096									
097	0.467	-2.3			1.26	-0.9			
098			ND	-4.0	ND	-4.0			

Laboratory code	Spiroxamine	Z-scores (FFP RSD (25%))		Triademefon and triademetron		Z-scores (FFP RSD (25%))		-triademefon		Z-scores (FFP RSD (25%))	
MRRL	0.01		0.01		0.01		0.01		0.01		
Assigned value	1.10		1.63		1.62		-0.9		-0.9		
099											
100			1.93	0.8	1.93	0.8					
101					1.271	-0.9					
102											
103	1.86	2.8	1.01	-1.5	1.01	-1.5					
105											
106	0.251	-3.1	2.639	2.5	2.628	2.5	0.011				
107											
108			1.34	-0.7	1.34	-0.7					
109											
110			ND	-4.0							
111											
112	1.21	0.4	0.6	-2.5	0.99	-1.6					
113	1.09	0.0	1.73	0.3	1.73	0.3	0.010				
114	0.19	-3.3	ND	-4.0	ND	-4.0					
115	2.11	3.7	1.64	0.0	1.64	0.0					
116	1.07	-0.1	1.05	-1.4	1.05	-1.4					
117	0.579	-1.9	ND	-4.0	0.467	-2.8					
119	1.205	0.4	ND	-4.0	2.088	1.2					
121			1.13	-1.2	1.13	-1.2					
122											

Table 15 Results of 2,4-D (free acid), 2,4-D (followed by alk. hydrolysis), glyphosate and mepiquat in mg/kg and their calculated z-scores using FFP RSD 25%.

Laboratory code	2,4- D (free acid)	Z-scores (FFP RSD (25%))		2,4- D (f oll. alk. hydr.)	Z-scores (FFP RSD (25%))		Glyphosate	Z-scores (FFP RSD (25%))		Mepiquat (free cation)	Z-scores (FFP RSD (25%))
MRRL	0.02			0.02			0.02			0.02	
Assigned value	0.355			0.367			3.87			0.089	
001	0.4	0.5								0.11	0.9
002	0.345	-0.1	0.35	-0.2						0.0991	0.5
003			ND	-3.8						ND	-3.1
004					4.25	0.4	0.123	1.5			
005											
006									0.054	-1.6	
007			0.478	1.2	3.98	0.1	0.079	-0.4			
008											
009	0.356	0.0	0.329	-0.4							
010	0.332	-0.3							0.11	0.9	
011											
012	0.336	-0.2	0.343	-0.3					0.0938	0.2	
013											
014	0.358	0.0	0.356	-0.1	2.933	-1.0	0.073	-0.7			
015	0.373	0.2									
016											
017											
018											
019	0.478	1.4	0.462	1.0					0.065	-1.1	
020	0.431	0.9	0.451	0.9	8.07	4.4	0.098	0.4			
021											
022											
023											
024	0.328	-0.3							0.055	-1.5	
025	0.449	1.1							0.085	-0.2	
026									0.085	-0.2	
027											
028									0.059	-1.3	
029	0.446	1.0	0.345	-0.2							
030	0.511	1.8	0.675	3.4	3.33	-0.6	0.144	2.5			
031											

Laboratory code	2,4- D (free acid)	Z-scores (FFP RSD (25%))		Z-scores (FFP RSD (25%))		Glyphosate	Z-scores (FFP RSD (25%))		Z-scores (FFP RSD (25%))
MRRL	0.02		0.02		0.02		0.02		
Assigned value	0.355		0.367		3.87		0.089		
032									
033									
034	0.331	-0.3	0.353	-0.2			0.118	1.3	
035									
036	0.35	-0.1	0.365	0.0	3.2	-0.7	0.114	1.1	
037	0.213	-1.6	0.443	0.8			0.063	-1.2	
038	0.17	-2.1			1.98	-2.0	0.04	-2.2	
039	0.374	0.2							
040	0.32	-0.4			4.3	0.5	0.084	-0.2	
041							0.0605	-1.3	
042	0.13	-2.5							
044									
045									
046									
047									
048									
049									
050					3.58	-0.3	0.062	-1.2	
051	0.351	0.0	0.343	-0.3	4.63	0.8	0.108	0.9	
052									
053									
054									
055									
056	0.334	-0.2	0.346	-0.2			0.126	1.7	
057			0.4	0.4	ND	-4.0	0.088	0.0	
058									
059									
060									
061	0.349	-0.1	0.358	-0.1			0.109	0.9	
062									
063									
064	0.35	-0.1					0.094	0.2	
065	0.353	0.0	0.369	0.0	3.75	-0.1	0.097	0.4	

Laboratory code	2.4- D (free acid)	Z-scores (FFP RSD (25%))			Glyphosate	Z-scores (FFP RSD (25%))		Z-scores (FFP RSD (25%))
MRRL	0.02		0.02		0.02		0.02	0.02
Assigned value	0.355		0.367		3.87		0.089	0.089
066								
067								
068								
069	0.327	-0.3						
070								
071								
072								
073								
074								
075							0.089	0.0
076								
077	0.367	0.1	0.45	0.9			ND	-3.1
078								
079								
080								
081								
082								
083								
084	0.407	0.6	0.398	0.3			0.081	-0.4
085	0.371	0.2	0.4	0.4	3.2	-0.7	0.091	0.1
086								
087	0.48	1.4					0.07	-0.9
088								
089								
090	0.324	-0.3			4.1	0.2	0.07	-0.9
091	0.365	0.1					0.109	0.9
092								
093								
094								
095								
096								
097								
098								

Laboratory code	Z-scores (FFP RSD (25%))		Z-scores (FFP RSD (25%))		Z-scores (FFP RSD (25%))	
MRRL	2.4- D (free acid)	0.02	2.4- D (f oll. alk. hydr.)	0.367	0.02	Glyphosate
Assigned value	0.355				3.87	
099						
100						
101						
102						
103						
105						
106						
107						
108						
109						
110						
111						0.1 0.5
112	0.43	0.9		4.2	0.3	
113	0.423	0.8				
114						
115						
116						
117						
119						
121						
122						

4.3.2 Sum of Weighted z-Scores (SWZ) – Category A

The MRM results were additionally evaluated by calculating the sum of weighted z-scores (SWZ). The SWZ values were calculated for the 53 laboratories that:

- 1) reported 90 % of the pesticide present in the test material (14-16 results including false negatives),
- 2) analysed for all the * mark pesticide present in the test material and
- 3) did not report any false positives for MRM pesticides.

For malathion and triadimenol, only the 'sum' results were included in the calculation (malathion-sum, triadimenol and tridimefon). In total, 41 participants (77 %) obtained SWZ scores at or below 2 (good) and 4 participants (8 %) obtained SWZ scores above 2 but below or at 3 (satisfactory) and 8 participants (15 %) obtained SWZ scores above 3 (unsatisfactory). The SWZ scores achieved by the labs can be seen in

Table 16.

Table 17 shows the laboratories in Category B, the number of results reported, and the number of acceptable z-scores. The table also include information on false negative and false positive results. **For malathion and triadimenol, only the 'sum' results were included in the calculation (malathion-sum, triadimenol and tridimefon).**

Table 16 Sum of Weighted z-Scores (SWZ) for laboratories in Category A, number of pesticide analysed by the laboratory, false negatives reported and Classification as good, satisfactory and unsatisfactory.

Lab code	No. of pesticides analysed	SWZ	False negative	Classification
028	16	0.3		Good
073	16	0.3		Good
002	16	0.4		Good
113	16	0.4		Good
012	16	0.4		Good
026	16	0.4		Good
055	16	0.4		Good
084	16	0.5		Good
037	16	0.5		Good
087	16	0.5		Good
014	16	0.5		Good
006	16	0.6		Good
077	16	0.6		Good
051	16	0.6		Good
032	16	0.7		Good
008	16	0.7		Good
085	16	0.7		Good
065	16	0.7		Good
034	16	0.8		Good
069	16	0.8		Good
036	16	0.9		Good
019	16	1.0		Good
030	16	1.1		Good

Lab code	No. of pesticides analysed	SWZ	False negative	Classification
042	16	1.1		Good
091	16	1.2		Good
029	16	1.2		Good
048	16	1.3		Good
095	16	1.4	Yes	Good
004	16	1.4		Good
015	16	1.4		Good
018	16	1.4	Yes	Good
056	16	1.5	Yes	Good
022	16	1.5	Yes	Good
064	16	1.5		Good
050	16	1.5		Good
024	16	1.6	Yes	Good
027	16	1.6	Yes	Good
025	16	1.6	Yes	Good
079	16	1.7		Good
061	16	1.8	Yes	Good
089	16	2.3	Yes	Satisfactory
011	16	2.4	Yes	Satisfactory
062	16	2.5		Satisfactory
010	16	3.2	Yes	Unsatisfactory
003	16	3.3		Unsatisfactory
119	16	4.3	Yes	Unsatisfactory
038	16	5.0		Unsatisfactory
083	16	>5.0	Yes	Unsatisfactory
001	16	>5.0	Yes	Unsatisfactory
071	15	1.6		Good
033	15	2.4		Satisfactory
115	15	>5.0		Unsatisfactory

Table 17 Number of pesticides analysed, number of acceptable z-scores, false negative and positive for the laboratories in Category B.

Lab code	No. of pesticides analysed	No. of acceptable z-scores	False negative	False positive
005	14	9	Yes	
007	16	10	Yes	Yes
009	0	0		
013	5	4		
016	16	12		Yes
017	13	13		
020	15	12		Yes
021	5	3		
023	14	13	Yes	
031	1	1		
035	2	1	Yes	Yes
039	13	12		Yes
040	16	16	Yes	Yes
041	15	14		
044	9	9		
045	3	3		
046	10	7	Yes	
047	14	14		
049	2	2		
052	9	8		
053	10	10		
054	2	1		
057	16	15	Yes	Yes
058	8	6		Yes
059	7	7		
060	10	10		
063	11	11		
066	8	4	Yes	
067	10	9		
068	9	8		
070	12	9	Yes	
072	1	1		
074	12	8		
075	14	12		
076	13	12	Yes	
078	0	0		
080	9	8		
081	3	2		
082	11	10	Yes	
086	3	3		
088	10	10		
090	11	10	Yes	

Lab code	No. of pesticides analysed	No. of acceptable z-scores	False negative	False positive
092	11	5		
093	12	11	Yes	
094	1	1		
096	5	1	Yes	
097	9	8		
098	10	9	Yes	
099	7	6	Yes	
100	13	9	Yes	
101	8	3		Yes
102	8	6		
103	14	10	Yes	
105	9	9		
106	16	12	Yes	Yes
107	0	0		
108	10	9	Yes	
109	5	5		
110	8	7	Yes	
111	4	4		
112	13	8		
114	13	3	Yes	Yes
116	12	9	Yes	
117	16	6	Yes	
121	9	9		
122	3	2		

4.4 Trends in number of participating laboratories and their performance

The number of laboratories participating in the EUPTs with cereals test material increased from 62 in EUPT-C1, 72 in EUPT-C2, 102 in EUPT-C3 and 115 in EUPT-C4 (see **Table 18**) The target pesticide list has increased from 34 MRM pesticides to 65 MRM pesticides and the number of pesticides, spiked or incurred in the test material has increased from 7 to 16.

The scope is still low for many laboratories, where 25% of the laboratories submitted results for less than 10 pesticides. No significant improvement in performance has been seen when looking at the percentage of acceptable, questionable, unacceptable z-scores and false negative results. The percentage of Category A laboratories has fallen from 60 to 46 % from EUPT-C2 to EUPT-C3 and in EUPT-C4 it is still 46%. Nevertheless, it is difficult to assess any improvement/deterioration in laboratory performance between the different proficiency tests, because the pesticides in the test materials and the laboratories participating in the PTs have changed. However, azoxystrobin and carbendazim have been included in all three test materials, and can therefore be used as indicators of accuracy of performance.

The percentage of acceptable z-scores for azoxystrobin has increased from 62 % to 88 % from EUPT-C1 to EUPT-C4. This is probably due to the recommendation to add water to the sample before extraction. In fact, the percentage of laboratories adding water has also increased from 65 % to 86 %. For carbendazim, the percentage of acceptable z-scores has not increased, but decreased slightly from 79 % to 74 %. However, many of the participating laboratories in EUPT-C4 have not participated before, and it is not fully correct to include their results in a trend analysis. Focussing on the results only from laboratories that have participated in all the PTs, the laboratory performance has improved for both compounds. For azoxystrobin the percentage of acceptable results has increased from 62 % to 95 % and for carbendazim from 79 % to 88 %. It is therefore reasonable to conclude that the PTs have been valuable for improving analytical accuracy.

4.5 Summary, conclusions and prospect for the EUPTs on pesticide residues in cereals

EUP4 was the fourth PT focussing on cereal-based test materials. A homogenous test material of rye flour, including both incurred and spiked pesticides, was successfully prepared. The rye was sprayed in the field and spiked in the laboratory following harvest with commercially available pesticide formulations. The test material included the following pesticides: azoxystrobin, carbaryl, carbendazim, chlorpyrifos-methyl, deltametrin, fenitrothion, fenpropimorph, fluquinconazole, flutriafol, isoproturon, kresoxim-methyl, lambda-cyhalothrin, malathion, pirimiphos-methyl, spiroxamine, triadimenol, 2,4-D, mepiquat and glyphosate. One hundred and eighteen laboratories, representing 29 countries, agreed to participate in the proficiency test, but two laboratories failed to submit results. All Member States and NRLs participated, however Malta had delegated their obligations to a laboratory in the UK. Out of the 103 laboratories, 102 submitted at least one result involving MRM-compounds. One laboratory submitted only results for SRM-pesticides. The target pesticide list distributed to the laboratories prior to the test contained 64 MRM pesticides and 11 SRM pesticides, excluding isomers and degradation products.

Table 18 Number of participants, pesticides, reported results, z-scores, number of Category A laboratories and SWZs for the 4 Cereal PTs .

	EUPT-C1	EUPT-C2	EUPT-C3	EUPT-C4
Type of test material	Wheat flour	Wheat flour	Oat flour	Rye flour
Participants submitting results	63	72	102	115
MRM pesticides in the Target Pesticide List ¹⁾	34	43	51	64
MRM pesticides in the test material	7	13	14	16
No. of results for MRM pesticides	323	830	981	1624
Range of 'reported results', % ²⁾	63 – 95	60 - 96	48 - 95	55 - 95
Acceptable z-scores, %	87	85	87	87
Questionable z-scores, %	7	12	8	6
Unacceptable z-scores, %	6	3	5	7
False negatives, %	2	3	3	4
Number of false positives	1	2	3	17
Category A, % of participating laboratories		60	46	46
Good SWZ, %		70	72	77
Satisfactory SWZ, %		9	15	8
Unsatisfactory SWZ, %		21	13	15

- 1) Number of pesticides, excluding isomers and degradation products
- 2) The range of reported results have been calculated using the number of laboratories that reported results from the total number of laboratories submitting results (see **Table 7**)

Both the number of false positives and false negatives has increased compared to the previous EUPTs. The 17 false positives were cypermethrin, ciproconazole, diazinon, dichlorvos, epoxiconazole, metconazole (2), methacrifos, metribuzin (2), permethrin, procymidone, procymidone (2), triadimefon, trifluralin (2) and vinclozolin. The 61 false negatives were azoxystrobin (1), carbaryl (1), chlorpyrifos-methyl (4), delta-methrin (22), fenitrothion (1), fenpropimorph (1), fluquinconazole (2), isoproturon (2), lambda-cyhalothrin (3), malathion (sum) (5), malathion (1), triadimefon and triadimenol (9), triadimenol (5), 2,4-D (foll. alk hydrolysis) (1), mepiquat and glyphosate (1). The average Qn-RSD (robust RSD) was 24 % close to the FFP-RSD of 25 % and on average ranging from 11 to 39 %.

For each laboratory/pesticide combination, z-scores based on the FFP RSD of 25 % were calculated and classified as either 'acceptable', 'questionable' or 'unacceptable'. For carbaryl, chlorpyrifos-methyl, fenitrothion, flutriafol, isoproturon, kresoxim-methyl and pirimiphos-methyl acceptable results were obtained by 90-94 % of the laboratories. For azoxystrobin, carbendazim, fenpropimorph, fluquinconazole, lambda-cyhalothrin, malathion, spiroxamine and triadimenol, acceptable results were obtained by 82-89 % of the laboratories. However, for deltamethrin acceptable z-scores were obtained by only 66 % of the laboratories. For the optional SRM pesticides acceptable z-scores were obtained by 87-97 % of participants.

Overall, the performance of the laboratories appears not to have significantly improved compared to the previous PTs on cereals. The percentage of acceptable, z-scores has not increased and the percentage of Category A laboratories is still 46 %. However, when focusing on azoxystrobin and carbendazim that has

been included in all PTs, and only looking at results from laboratories participating in all the PTs then a significant improvement in their performance can be seen.

In future PTs, the selection of pesticides will continue to focus on pesticides included in the scope of the EU coordinated control programme as well as additional pesticides of relevance for cereal production in Europe and in other parts of the world from where significant quantities of cereals are imported. The pesticide residues will be incurred wherever practical and the commodities will be alternated between the most important commodities. In 2011 the test material will be rice. The target pesticide list for 2011 will be increased by the addition of 22 pesticides: acephate, azinphos-methyl, captan, carbofuran (sum of carbofuran and 3-Hydroxycarbofuran expressed as carbofuran), chlorgenvinphos, chlorpropham (only parent compound), cyfluthrin (cyfluthrin incl. other mixtures of constituent isomers (sum of isomers)), DDT (sum of p,p'-DDT, o,p'-DDT, p,p'-DDE and p,p'-TDE (DDD) expressed as DDT), diflubenzuron, dimethoate (sum of dimethoate and omethoate expressed as dimethoate), ethion, fenthion (fenthion and its oxygen analogue, their sulfoxides and sulfone expressed as fenthion), fipronil (parent compound), hexachlorocyclohexane (HCH) (sum of isomers, except the gamma isomer), imidacloprid, isoporthiolane, linuron, oxydemeton-methyl (sum of oxydemeton-methyl and demeton-S-methylsulfone expressed as oxydemeton-methyl), quinoxifen, thiacloprid, thiamethoxam (sum of thiamethoxam and clothianidin expressed as thiamethoxam) and tricyclazole .

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

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

5. ACKNOWLEDGEMENTS

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

6. REFERENCES

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APPENDICES

Appendix 1 List of Laboratories registered to participate in the EUPT-C4

* Represented by NRL-SRM of UK

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

Appendix 2 Target- Pesticide List of MRM-pesticides

Appendix 3 Homogeneity data

Appendix 4 Stability figures

Appendix 5 Histograms of residue data

Appendix 6 Graphical presentation of z-scores

Appendix 7 Method used by the participants

Annex 1 General protocol

Annex 2 Specific protocol

APPENDICES

Appendix 1 List of Laboratories registered to participate in the EUPT-C4

Country	Institution	City	NRL-CF
Austria	Austrian Agency for Health and Food Safety, Competence Centre for Residue Analysis	Vienna	X
Austria	AGES CC PSMR Innsbruck	Innsbruck	
Belgium	Fytolab	Zwijnaarde	
Belgium	Scientific Institute of Public Health, FOOD	Bruxelles	X
Bulgaria	Plant Protection Institute, Toxicology	Kostinbrod	X
Bulgaria	National Center of Public Health Protection, Food and Nutrition, Department of Chemical Contaminants and Food Additives	Sofia	X
Cyprus	State General Laboratory	Nicosia	X
Czech Republic	Czech Agriculture and Food Inspection Authority, Laboratory Department	Prague	X
Czech Republic	Central Institute for Supervising and Testing in Agriculture, UKZUZ-NRL	Brno	X
Czech Republic	Institute of Chemical Technology Prague, Department of Food Chemistry and Analysis	Prague	
Denmark	Danish Veterinary and Food Administration, Region East	Ringsted	
Denmark	The Danish Plant Directorate	Lyngby	X
Estonia	AGRICULTURAL RESEARCH CENTRE,	Saku	X
Estonia	Health Protection Inspectorate	Tartu	
Finland	Finnish Customs Laboratory	Espoo	X
Finland	MetropoliLab	Helsinki	
France	Laboratoire du SCL de Montpellier	Montpellier	
France	SCL laboratoire d'Ile de France Massy, Residus de Pesticides	Massy Cedex	X
France	Laboratoire SCL, Pesticides	Rennes	
France	LDA 56, Chimie	Saint-Ave	
France	LABORATOIRE Departemental de la SARTHE, CHIMIE,	Le Mans	
France	LABORATOIRE de DEVELOPPEMENT et D' ANALYSE des COTES D' ARMOR (L.D.A 22), AAE, CHROMATOGRAPHIE	Ploufragan	
Germany	LUFA Speyer, Rueckstandsanalytik	Speyer	
Germany	LLFG, Agricultural Analysis	Halle	
Germany	Betriebsgesellschaft für Umwelt und Landwirtschaft, GB 6 - Labore Landwirtschaft	Leipzig	
Germany	Landesamt für Verbraucherschutz, Sachsen-Anhalt	Halle/Saale	
Germany	Federal Office of Consumer Protection and Food Safety (BVL), Unit 501	Berlin	X
Germany	Chemisches- und Veterinäruntersuchungsamt Münsterland-Emscher-Lippe (CVUA-MEL), Fachbereich 6-3	Münster	
Germany	Niedersaechsisches Landesamt fuer Verbraucherschutz und Lebensmittelsicherheit	Oldenburg	

Country	Institution	City	NRL-CF
Germany	Thüringer Landesanstalt für Landwirtschaft (TLL),	Jena	
Germany	Amt fuer Umwelt, Verbraucherschutz und Lokale Agenda der Stadt Bonn, Amtliche Lebensmittel-untersuchung	Bonn	
Germany	Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit	Erlangen	
Germany	Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit, Dienststelle Oberschleissheim	Oberschleissheim	
Germany	Landeslabor Berlin-Brandenburg, FB II-4	Potsdam	
Germany	Institut fuer Hygiene und Umwelt, Residues and Contaminants	Hamburg	
Germany	Landwirtschaftliches Technologiezentrum Augustenberg , Abteilung 2: Chemische Analysen, Saatgut- und Futtermitteluntersuchungen	Augustenberg	
Germany	LUFA-ITL GmbH	Kiel	
Germany	CVUA Stuttgart	Stuttgart	
Germany	Landesuntersuchungsanstalt für das Gesundheits- und Veterinaerwesen Sachsen, , FG 6.5 Pestizidlabor	Dresden	
Germany	CVUA-OWL, Dez. 6.2	Bielefeld	
Germany	Landesamt für Landwirtschaft, Lebensmittelsicherheit und Fischerei Mecklenburg-Vorpommern, , Schadstoff- und Rückstandsanalytik	Rostock	
Germany	Landesbetrieb Hessisches Landeslabor, Fachgebiet 41,	Kassel	
Germany	Landesuntersuchungsamt für Chemie, Hygiene und Veterinärmedizin, Referat 40, Zentrale Analytik	Bremen	
Germany	Landesuntersuchungsamt, Institut für Lebensmittelchemie	Speyer	
Greece	Benaki Phytopathological Institute,	Athens	X
Greece	Regional Centre for Crop Protection and Quality Control of Ioannina, Laboratory of Pesticide analysis	Ioannina	
Greece	General Chemical State Laboratory, D Division	Athens	X
Hungary	Agricultural Office of County Fejer, PPSCD	Velence	X
Hungary	Agricultural Office of County Vas Plant Protection and Soil Conservation Directorate	Tanakajd	
Hungary	Agricultural Office of Somogy County, Plant Protection and Soil Conservation Directorate	Kaposvár	
Hungary	Plant Protection and Soil Conservation Directorate of Jasz-Nagykun-Szolnok County, Pesticide Residue Analytical	Szolnok	
Ireland	Department of Agriculture, Food and Fisheries	Kildare	X
Italy	National Institute of Health, Environmental and Primary Prevention, Pesticide Unit	Rome	
Italy	Agenzia Regionale per la Protezione Ambientale della Toscana (ARPAT), Dipartimento di Arezzo,	Arezzo	
Italy	APPA Bolzano, , Laboratorio Analisi Aria e Rumore	Bolzano	
Italy	Istituto Zooprofilattico Sperimentale	Genova	X
Italy	ARPA-VENETO, DIP. REG. LABORATORI, S.L. VERONA	Verona	
Italy	ARPA-FVG, Dipartimento di Pordenone, Tematico analitico - Alimenti	Pordenone	
Italy	ARPA Puglia, Dipartimento di Bari,	Bari	
Italy	ASL PROVINCIA DI BERGAMO, DIPARTIMENTO MEDICO, LABORATORIO DI SANITA' PUBBLICA	Bergamo	
Italy	Istituto Zooprofilattico Sperimentale dell' Abruzzo e del Molise "G. CAPORALE", Food and Feed Hygiene, Chemistry	Teramo	

Country	Institution	City	NRL-CF
Italy	ARPA MARCHE, DIP. MACERATA, ALIMENTI	Villapotenza – Macerata	
Italy	IZS Sardegna, Igiene Allevamenti, Chimica Ambientale	Sassari	
Italy	Istituto Zooprofilattico Sperimentale Umbria Marche, Food Safety, Environmental Contaminants	Perugia	
Italy	Istituto Zooprofilattico Sperimentali Lazio e Toscana, Direzione Operativa Chimica , Laboratorio Alimenti per Animali	Rome	
Italy	Istituto Zooprofilattico Sperimentale delle Venezie, Struttura Complessa 2 - Chimica, Laboratorio Contaminanti	Legnaro	
Italy	ARPAT, Dipartimento Provinciale di Livorno, Microinquinanti Organici	Livorno	
Italy	Istituto Zooprofilattico Sperimentale Lombardia Emilia Romagna , Reparto Chimica degli alimenti di Origine Animale, Laboratorio Contaminanti Ambientali	Brescia	
Italy	Arpa Regione Emilia-Romagna, RAR - Riferimento Analitico Regionale	Ferrara	
Italy	CE.FI.T. SRL, SETTORE FITOFARMACI, CENTRO FITOFARMACI E TECNOLOGIE AGROALIMENTARI	Avola	
Italy	APPA TRENTO, Settore Laboratorio e Controlli,	Trento	
Latvia	Institute of Food Safety, Animal Health and Environment (BIOR)	Riga	X
Lithuania	National food and veterinary risk assessment institute, Laboratory department	Vilnius	X
Norway	Bioforsk, Plant Health and Plant Protection	Aas	X
Poland	Instytut Ochrony Roslin - PIB, Terenowa Stacja Doswiadczeniowa,	Trzebnica	
Poland	Wojewodzki Inspektorat Weterynarii, , Zaklad Higieny Weterynaryjnej	Poznan	
Poland	Wojewódzka Stacja Sanitarno-Epidemiologiczna, ,	Gdansk	
Poland	Wojewodzka Stacja Sanitarno-Epidemiologiczna w Rzeszowie, ,	Wierzbowa	
Poland	Institute of Plant Protection, Department of Pesticide Residue Research,	Poznan	X
Poland	Wojewodzki Inspektorat Weterynarii, ZHW Wrocław, Chemia Srodowisk Spozywczych i Pasz	Wroclaw	
Poland	Wojewodzki Inspektorat Weterynarii, Opole, Zaklad Higieny Weterynaryjnej	Opole	
Poland	Wojewodzka Stacja Sanitarno-Epidemiologiczna, Dzial Laboratoryjny, Oddzial Laboratoryjny Analiz Instrumentalnych	Krakow	
Poland	INSTYTUT OCHRONY ROSLIN PANSTWOWY INSTYTUT BADAWCZY, ,	Bialystok	
Poland	Voivodship Sanitary-Epidemiological Station in Warsaw, , Pesticide Residues Laboratory	Warsaw	
Poland	Wojewodzki Inspektorat Weterynarii, Zaklad Higieny Weterynaryjnej, Pracownia Analityki Chemicznej	Szczecin	
Poland	Wojewódzki Inspektorat Weterynarii, Zaklad Higieny Weterynaryjnej, Pracownia Badan Chemicznych	Katowice	
Poland	Institute of Plant Protection, Experimental Station in Rzeszow,	Rzeszow	
Poland	Voivodship Veterinary Inspection, Department of Pesticide Residues, Veterinary Hygiene Laboratory	Warszawa	
Poland	Wojewodzka Stacja Sanitarno-Epidemiologiczna w Opolu , ,	Opole	
Poland	Institute of Plant Protection - National Research Institute Sosnowcowice	Sosnowcowice	X

Country	Institution	City	NRL-CF
	Branch, Zaklad Analizy Srodowisk Ochrony Roslin, Laboratorium Badania Pozostalosci Pestycydow		
Poland	Voivodship Veterinary Inspektion, Department of Pesticide Residues, Veterinary Higiene Laboratory	Bialystok	
Poland	Voivodship Veterinary Inspection, Department of Pesticide Residues, Veterinary Hygiene Laboratory	Gdansk	
Portugal	Direccao Regional Agricultura Pescas Norte, Div.Exp.Qualificao e Apoio Laboratorial	Senhora da Hora	
Portugal	INRB-L-INIA, Plant Protection Research Unit	Oeiras	X
Portugal	Direccao de Servicos de Laboratorios Agro-Alimentares, Divisao de Analises de Residuos, Laboratorio Regional de Veterinaria e Segurança Alimentar	Funchal	
Romania	Central Laboratory for Pesticides Residues Control	Bucharest	X
Romania	Sanitary Veterinary and Food Safety Laboratory Bucharest	Bucharest	
Romania	Hygiene and Veterinary Public Health Institute, Residues Department, Pesticides Laboratory	Bucharest	
Romania	Sanitary Veterinary and Food Safety Directorate Cluj, Sanitary Veterinary and Food Safety Laboratory Cluj, Gas-Chromatography Laboratory	Cluj	
Slovakia	State Veterinary and Food Institute, chromatography	Bratislava	X
Slovakia	Public health authority of the Slovak republic, Department for Materialization of Living Condition	Bratislava	
Slovenia	Institute of Public Health Maribor, Environmental Protection Institute	Maribor	
Slovenia	Institute of public health	Ljubljana	X
Slovenia	Agricultural Institute of Slovenia, Central Laboratories	Ljubljana	X
Spain	Instituto Tecnologico de Canarias, Analisis Ambiental	Las Palmas	
Spain	Junta de Castilla y León, Dirección General de Producción Agropecuaria	Burgos	
Spain	GOBIERNO DE ARAGÓN, AGRICULTURA Y ALIMENTACIÓN	Zaragoza	
Spain	LABORATORI AGROALIMENTARI - DAR, Generalitat de Catalunya	Cabrilis (Barcelona)	
Spain	MINISTERIO DE MEDIO AMBIENTE MEDIO RURAL Y MARINO, LABORATORIO ARBITRAL AGROALIMENTARIO, Departamento de Residuos de Pesticidas	Madrid	X
Spain	A.E.S.A.N. (C.N.A.)	Majadahonda	X
Sweden	National Food Administration, Research and Development	Uppsala	X
Sweden	Eurofins Food/Agro Sweden AB **	Lidköping	
Switzerland	Kantonales Labor Zürich, Abteilung Pestizide	Zurich	
Switzerland	Service de la consommation et des affaires vétérinaires (SCAV), Recherche de substances étrangères,	Geneve	
The Netherlands	RIKILT Institute of Food Safety	Wageningen	X
The Netherlands	VWA - Food and Consumer Product Safety Authority, NRL for Pesticides in Food	Amsterdam	X
The Netherlands	Dr A Verwey / Silliker NL, Chromatography, Dr A Verwey	Rotterdam	
United Kingdom	Central Science Laboratory, Food Science Group	York	X

Appendix 2 Target Pesticide List

MRM-Parameter	MRRL mg/kg
* Azoxystrobin	0.01
* Bifenthrin	0.01
* Carbaryl	0.01
* Carbendazim and benomyl (carbendazim + benomyl, expressed as carbendazim)	0.01
* Chlorothanlonil	0.01
* Chloryrifos	0.01
* Chloryrifos-methyl	0.01
* Cypermethrin (sum of isomers)	0.01
* -Alpha-cypermethrin	0.01
* Cyproconazole	0.01
* Cyprodinil	0.01
* Deltamethrin (cis-deltamethrin)	0.01
Diazinon	0.01
* Dichlorvos	0.01
* Difenoconazole	0.01
* Endosulfan (sum of alpha- and beta-isomers and endosulfan-sulphate expressed as endosulfan)	0.01
-Endosulfan α	0.01
-Endosulfan β	0.01
-Endosulfan sulfate	0.01
* Epoxiconazole	0.01
Fenvalerate and Esfenvalerate (Sum of RR/SS and RS/SR isomers)	0.01
Fenbuconazole	0.01
Fenhexamid	0.01
Fenitrothion	0.01
* Fenpropimorph	0.01
* Fludioxonil	0.01
Fluquinconazole	0.01
Flusilazole	0.01
* Flutriafol	0.01
Hexaconazole	0.01
* Imazalil	0.01
* Iprodione	0.01
Isoproturon	0.01
* Kresoxim-methyl	0.01
* Lambda-cyhalothrin	0.01
* Lindane (gamma- isomer of hexachlorocyclohexane (HCH))	0.01
* Malathion (Malathion + Malaoxon, expressed as Malathion)	0.01
-Malathion	0.01
-Malaoxon	0.01
Metconazole	0.01
* Methacrifos	0.01
* Methomyl and Thiodicarb (Methomyl + Thiodicarb, expressed as Methomyl)	0.01
-Methomyl	0.01
-Thiodicarb	0.01

Metribuzin	0.01
* Parathion	0.01
* Penconazole	0.01
Pendimethalin	0.01
* Permethrin	0.01
* Pirimicarb (sum of pirimicarb and desmethyl pirimicarb, expressed as pirimicarb)	0.01
-Pirimicarb	0.01
-Desmethyl pirimicarb	0.01
* Pirimiphos-methyl	0.01
* Prochloraz (parent compound only)	0.01
* Procymidone	0.01
* Propiconazole	0.01
Pyraclostrobin	0.01
Pyrimethanil	0.01
Spiroxamine	0.01
* Tebuconazole	0.01
Tebufenozide	0.01
* Thiabendazole	0.01
* Thiophanate-methyl	0.01
Triadimefon and triadimenol (sum of triadimefon and triadimenol)	0.01
-Triadimefon	0.01
-Triadimenol	0.01
Triazophos	0.01
* Trifloxystrobin	0.01
Trifluralin	0.01
* Triticonazole	0.01
Vinclozolin (only parent compound)	0.01

SRM-Parameter	
2,4-D (free acid)	0.02
2,4-D (following alkaline hydrolysis)	0.02
Dicamba (free acid)	0.02
Dicamba (following alkaline hydrolysis)	0.02
Dichlorprop (2,4-DP) including Dichlorprop-P (free acid)	0.02
Dichlorprop (2,4-DP) including Dichlorprop-P (following alkaline hydrolysis)	0.02
Fluazifop including Fluazifop-P (free acid)	0.02
Fluazifop including Fluazifop-P (following alkaline hydrolysis)	0.02
Fluroxypyr (free acid)	0.02
Fluroxypyr (following alkaline hydrolysis)	0.02
Haloxifop including Haloxifop-R (free acid)	0.02
Haloxifop including Haloxifop-R (following alkaline hydrolysis)	0.02
MCPA (free acid)	0.02
MCPA (following alkaline hydrolysis)	0.02
Mecoprop (MCPP) including Mecoprop-P (free acid)	0.02
Mecoprop (MCPP) including Mecoprop-P (following alkaline hydrolysis)	0.02
Glyphosate	0.02
Chlormequat (free cation)	0.02

Appendix 3 Homogeneity data of MRM-pesticides

Azoxystrobin mg/kg		
Sample no.	Portion 1	Portion 2
19	0.342	0.330
35	0.337	0.339
38	0.432	0.412
49	0.331	0.335
69	0.322	0.333
77	0.318	0.335
105	0.343	0.326
127	0.333	0.329
134	0.337	0.322
179	0.325	0.345
184	0.345	0.338

Carbaryl mg/kg		
Sample no.	Portion 1	Portion 2
19	0.143	0.179
35	0.150	0.186
38	0.174	0.150
49	0.154	0.148
69	0.143	0.199
77	0.156	0.200
105	0.165	0.149
127	0.172	0.177
134	0.184	0.140
179	0.139	0.139
184	0.187	0.151

Carbendazim mg/kg		
Sample no.	Portion 1	Portion 2
19	1.014	1.128
35	1.017	1.101
38	1.007	1.038
49	1.048	1.024
69	0.918	1.105
77	0.995	1.169
105	0.914	1.065
127	1.040	1.005
134	1.011	0.952
179	1.041	1.031
184	1.038	1.059

Chlorpyrifos-methyl mg/kg		
Sample no.	Portion 1	Portion 2
19	0.128	0.118
35	0.126	0.132
38	0.133	0.130
49	0.131	0.133
69	0.118	0.128
77	0.127	0.122
105	0.122	0.132
127	0.121	0.132
134	0.122	0.124
179	0.132	0.117
184	0.137	0.126

Deltamethrin mg/kg		
Sample no.	Portion 1	Portion 2
19	0.076	0.072
35	0.061	0.060
38	0.091	0.091
49	0.062	0.071
69	0.066	0.073
77	0.058	0.067
105	0.073	0.061
127	0.072	0.070
134	0.075	0.054
179	0.058	0.061
184	0.062	0.075

Fenitrothion mg/kg		
Sample no.	Portion 1	Portion 2
19	0.154	0.143
35	0.159	0.164
38	0.171	0.149
49	0.158	0.155
69	0.132	0.161
77	0.156	0.151
105	0.141	0.159
127	0.149	0.154
134	0.143	0.144
179	0.159	0.141
184	0.164	0.154

Fenpropimorph mg/kg		
Sample no.	Portion 1	Portion 2
19	2.544	2.436
35	2.487	2.424
38	2.560	2.434
49	2.507	2.490
69	2.335	2.535
77	2.287	2.362
105	2.447	2.238
127	2.348	2.503
134	2.309	2.384
179	2.312	2.212
184	2.498	2.170

Fluquinazole mg/kg		
Sample no.	Portion 1	Portion 2
19	0.766	0.752
35	0.753	0.736
38	0.942	0.916
49	0.746	0.762
69	0.747	0.755
77	0.745	0.728
105	0.759	0.765
127	0.749	0.739
134	0.786	0.776
179	0.742	0.764
184	0.785	0.766

Flutriafol mg/kg		
Sample no.	Portion 1	Portion 2
19	2.072	1.964
35	2.077	1.947
38	2.443	2.355
49	1.968	1.920
69	1.977	2.022
77	1.840	1.964
105	2.049	2.029
127	1.974	1.960
134	1.995	2.002
179	1.905	2.080
184	2.142	2.012

Isoproturon mg/kg		
Sample no.	Portion 1	Portion 2
19	0.131	0.150
35	0.150	0.185
38	0.153	0.175
49	0.157	0.146
69	0.147	0.144
77	0.172	0.168
105	0.131	0.175
127	0.182	0.158
134	0.137	0.147
179	0.167	0.146
184	0.167	0.168

Kresoxim-methyl mg/kg		
Sample no.	Portion 1	Portion 2
19	0.417	0.410
35	0.428	0.396
38	0.471	0.450
49	0.412	0.405
69	0.406	0.408
77	0.372	0.378
105	0.428	0.404
127	0.419	0.424
134	0.409	0.419
179	0.387	0.399
184	0.438	0.390

Lambda-cyhalothrin mg/kg		
Sample no.	Portion 1	Portion 2
19	0.050	0.058
35	0.050	0.035
38	0.056	0.057
49	0.038	0.037
69	0.070	0.064
77	0.034	0.073
105	0.052	0.046
127	0.035	0.044
134	0.062	0.065
179	0.044	0.075
184	0.050	0.058

Malathion mg/kg		
Sample no.	Portion 1	Portion 2
19	0.101	0.093
35	0.099	0.102
38	0.102	0.109
49	0.100	0.100
69	0.093	0.050
77	0.100	0.093
105	0.097	0.105
127	0.100	0.105
134	0.094	0.101
179	0.099	0.092
184	0.053	0.100

Pirimiphos-methyl mg/kg		
Sample no.	Portion 1	Portion 2
19	0.085	0.075
35	0.082	0.082
38	0.081	0.087
49	0.094	0.085
69	0.081	0.082
77	0.081	0.076
105	0.094	0.079
127	0.083	0.093
134	0.081	0.090
179	0.083	0.078
184	0.090	0.077

Spiroxamin mg/kg		
Sample no.	Portion 1	Portion 2
19	0.989	1.001
35	0.988	1.089
38	0.915	1.031
49	1.073	0.958
69	0.888	1.044
77	0.902	1.117
105	0.836	0.990
127	0.956	0.911
134	0.916	0.897
179	0.931	0.867
184	0.876	0.927

Triadimenol mg/kg		
Sample no.	Portion 1	Portion 2
19	1.241	1.218
35	1.267	1.136
38	1.429	1.450
49	1.189	1.160
69	1.185	1.254
77	1.065	1.263
105	1.317	1.161
127	1.248	1.212
134	1.231	1.307
179	1.135	1.289
184	1.296	1.141

2,4-D (free acid) mg/kg		
Sample no.	Portion 1	Portion 2
19	0.318	0.357
35	0.284	0.417
38	0.277	0.346
49	0.243	0.217
69	0.276	0.293
77	0.233	0.299
105	0.257	0.401
127	0.270	0.329
134	0.249	0.265
179	0.259	0.404
184	0.355	0.378

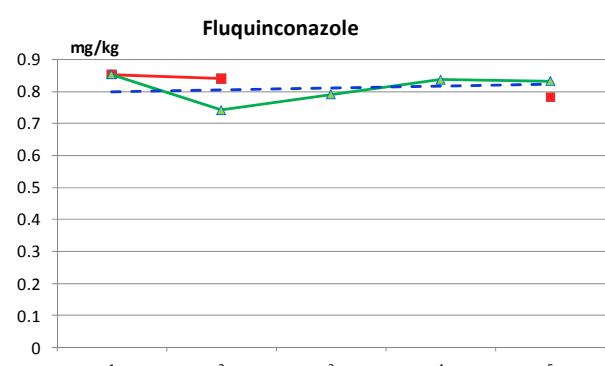
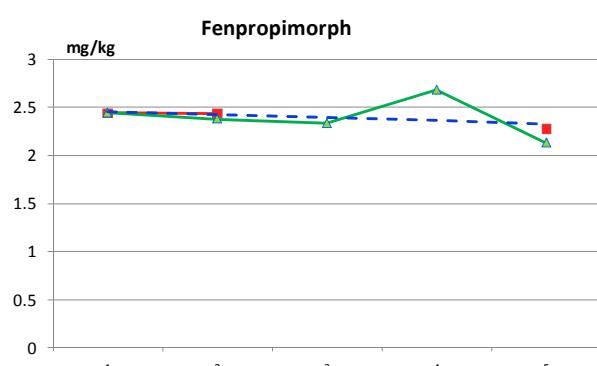
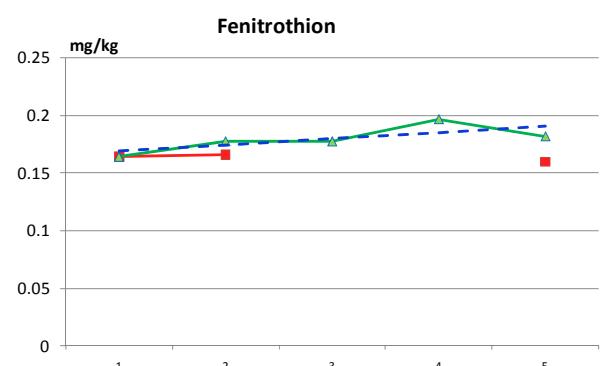
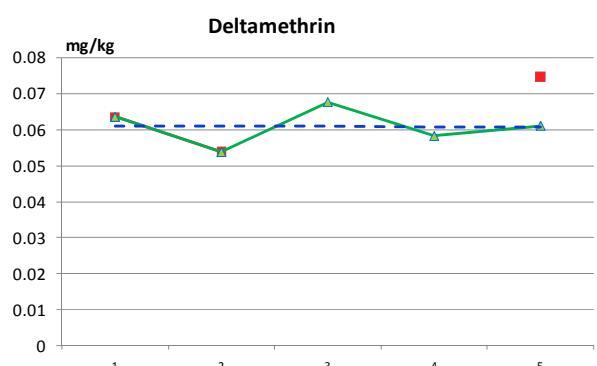
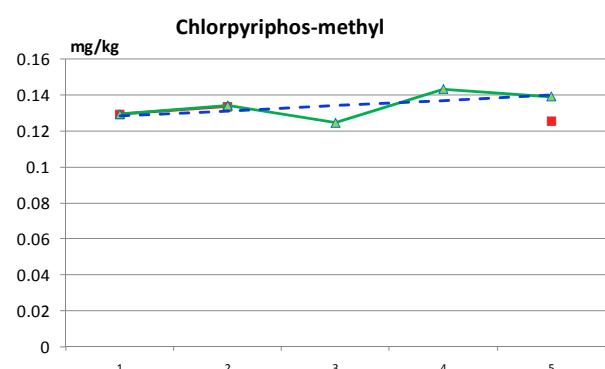
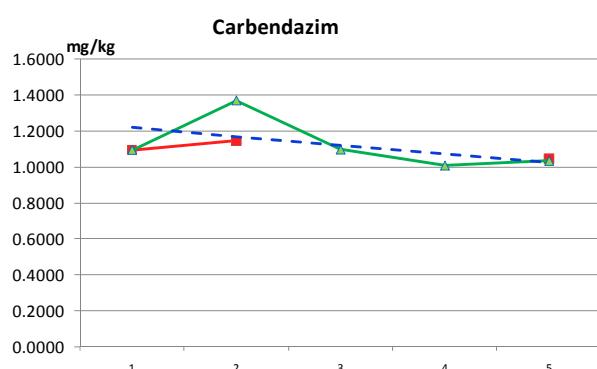
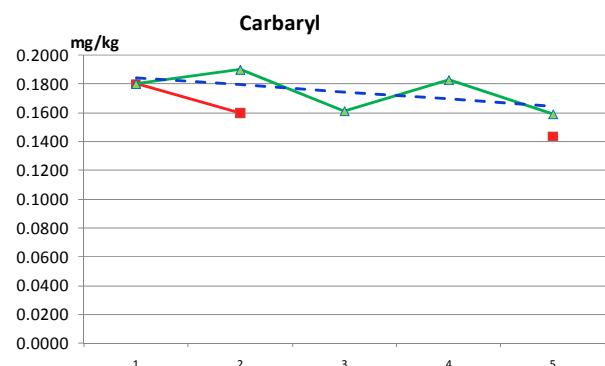
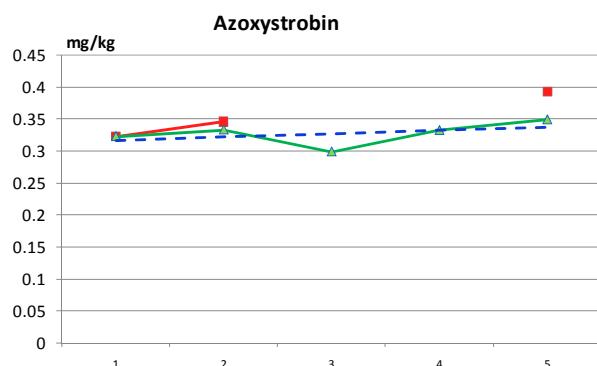
2,4-D (alk. hydro) mg/kg		
Sample no.	Portion 1	Portion 2
19	0.310	0.249
35	0.274	0.339
38	0.341	0.353
49	0.282	0.242
69	0.264	0.305
77	0.388	0.245
105	0.334	0.347
127	0.268	0.359
134	0.238	0.333
179	0.258	0.314
184	0.275	0.250

Glyphosate mg/kg		
Sample no.	Portion 1	Portion 2
19	3.249	3.160
35	3.299	3.560
38	3.071	3.315
49	3.335	3.380
69	3.196	3.301
77	3.170	3.588
105	3.427	3.035
127	3.225	3.097
134	3.282	3.111
179	3.212	3.495
184	3.216	3.444

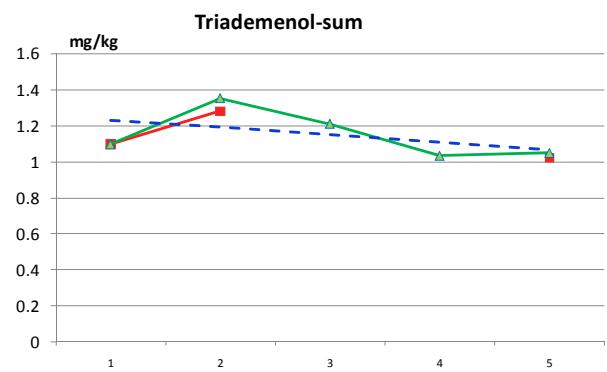
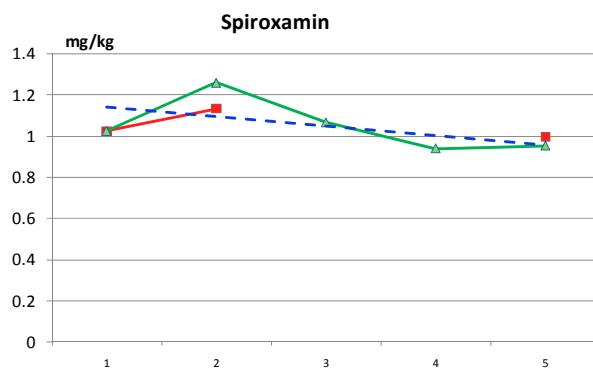
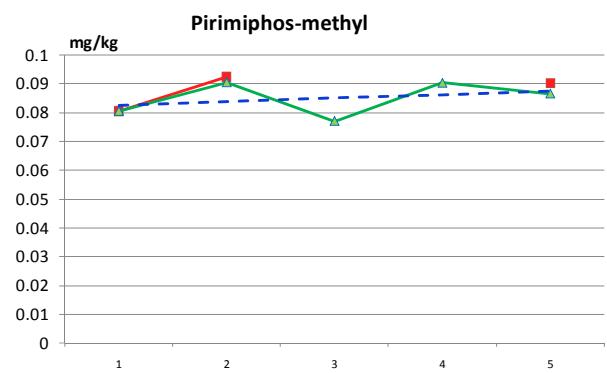
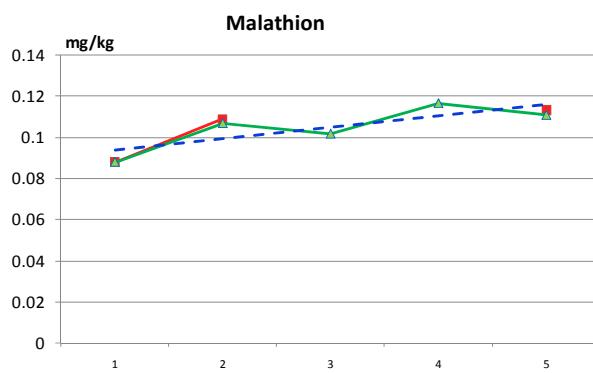
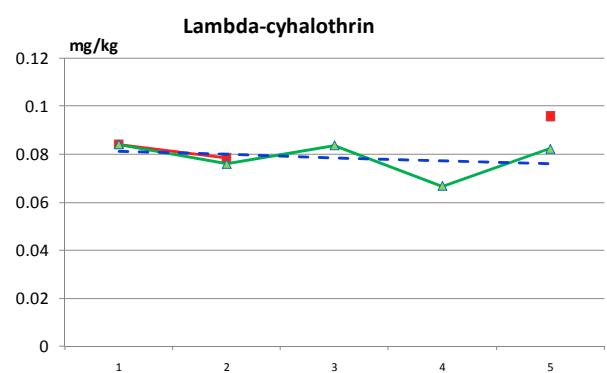
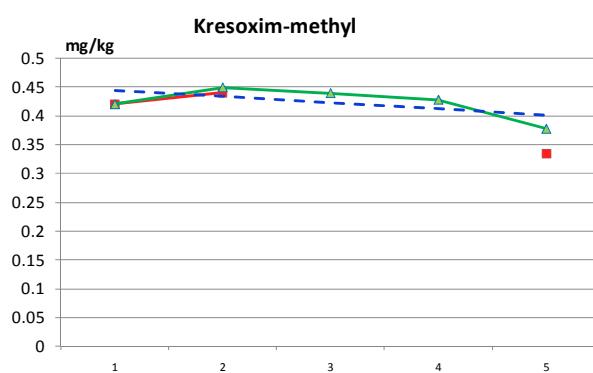
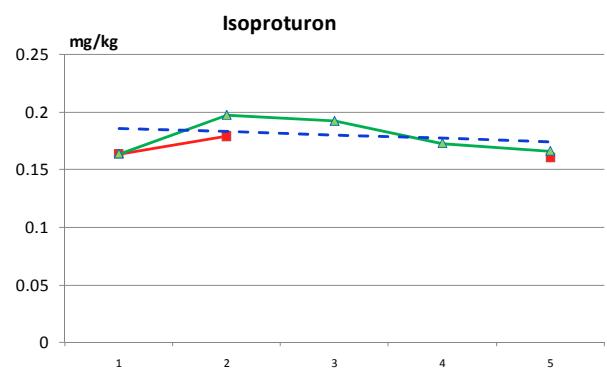
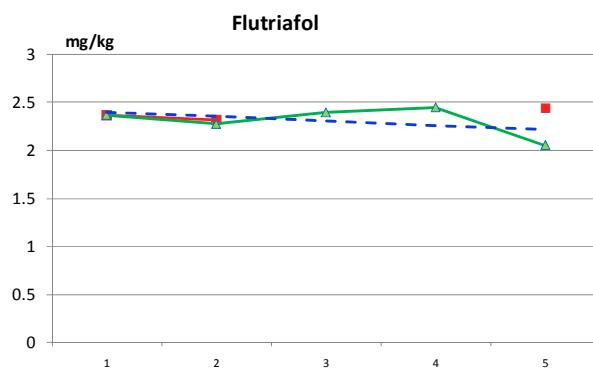
Mepiquat mg/kg		
Sample no.	Portion 1	Portion 2
19	0.094	0.096
35	0.096	0.092
38	0.084	0.092
49	0.092	0.100
69	0.092	0.074
77	0.096	0.100
105	0.102	0.096
127	0.086	0.098
134	0.098	0.094
179	0.088	0.098
184	0.100	0.100

Appendix 4 Stability figures

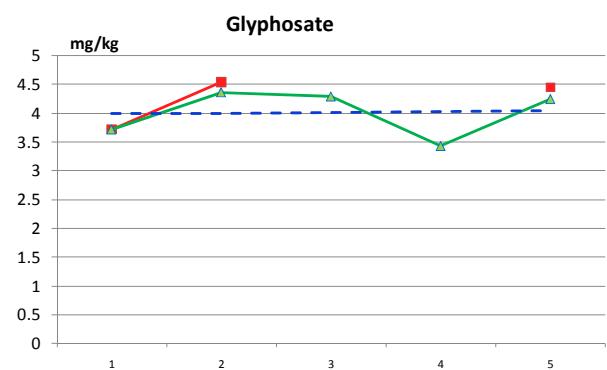
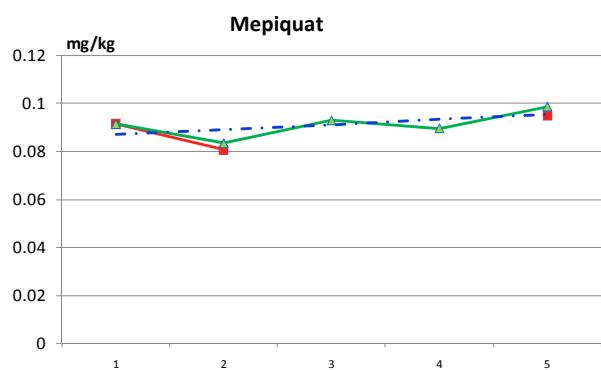
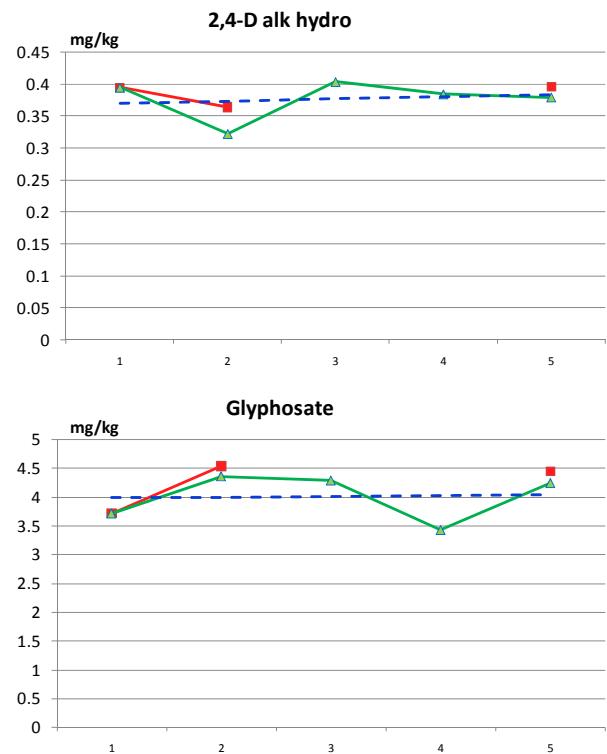
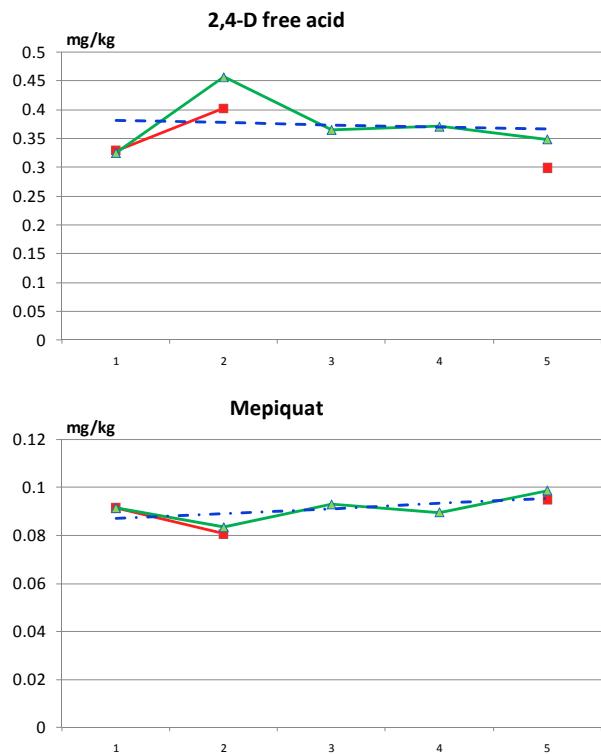
room temperature freezer regression freezer



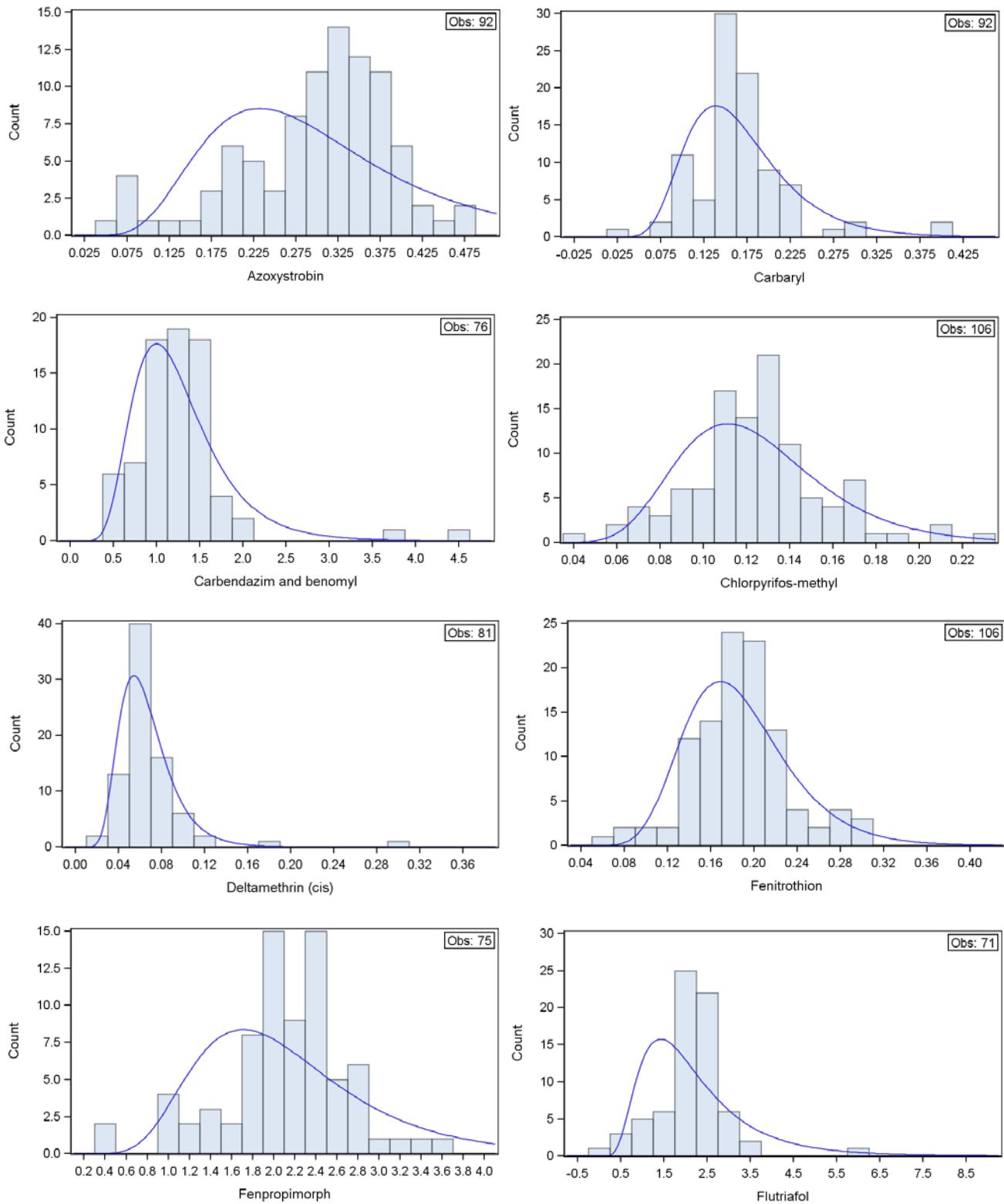
room temperature freezer regression freezer

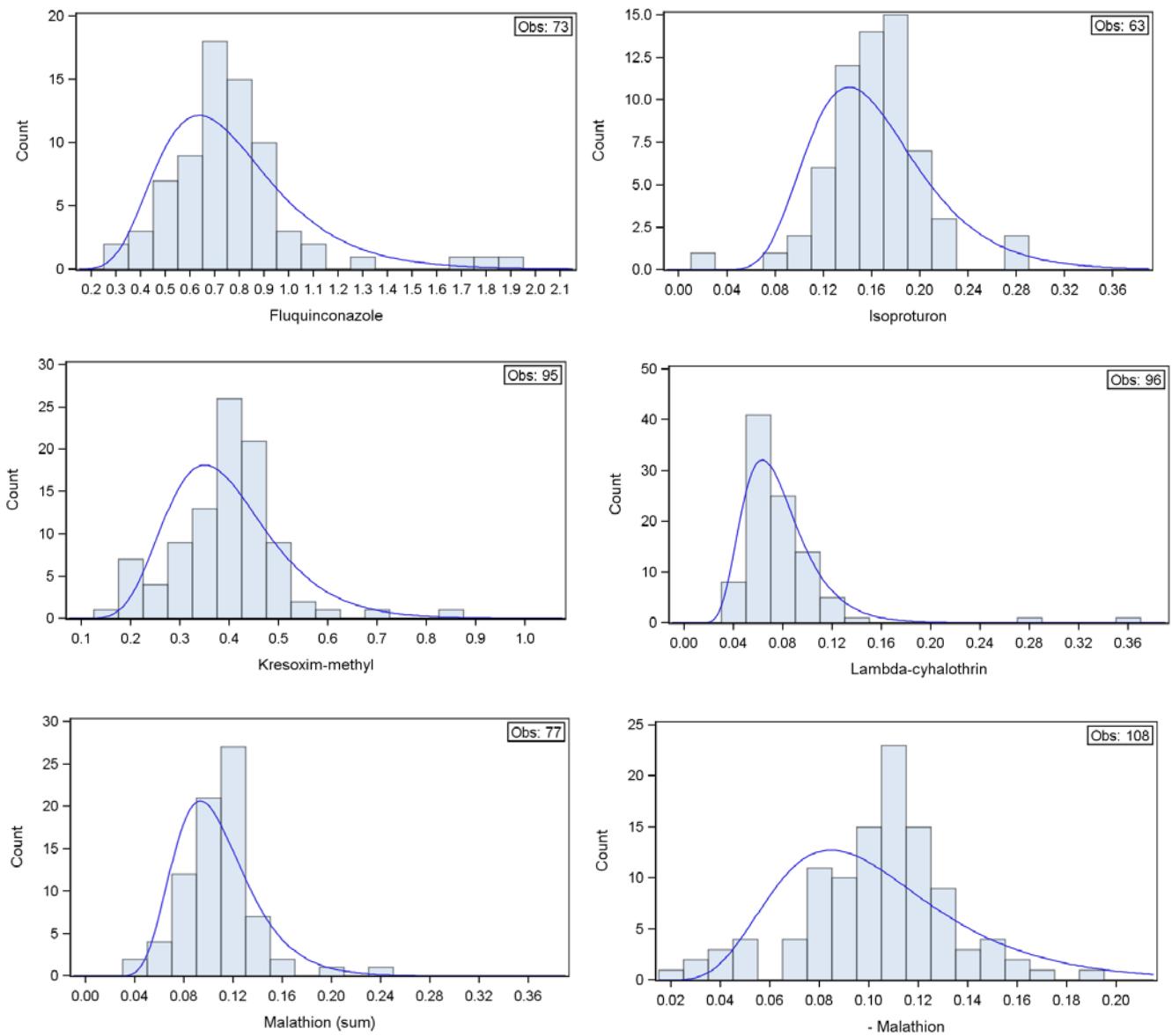


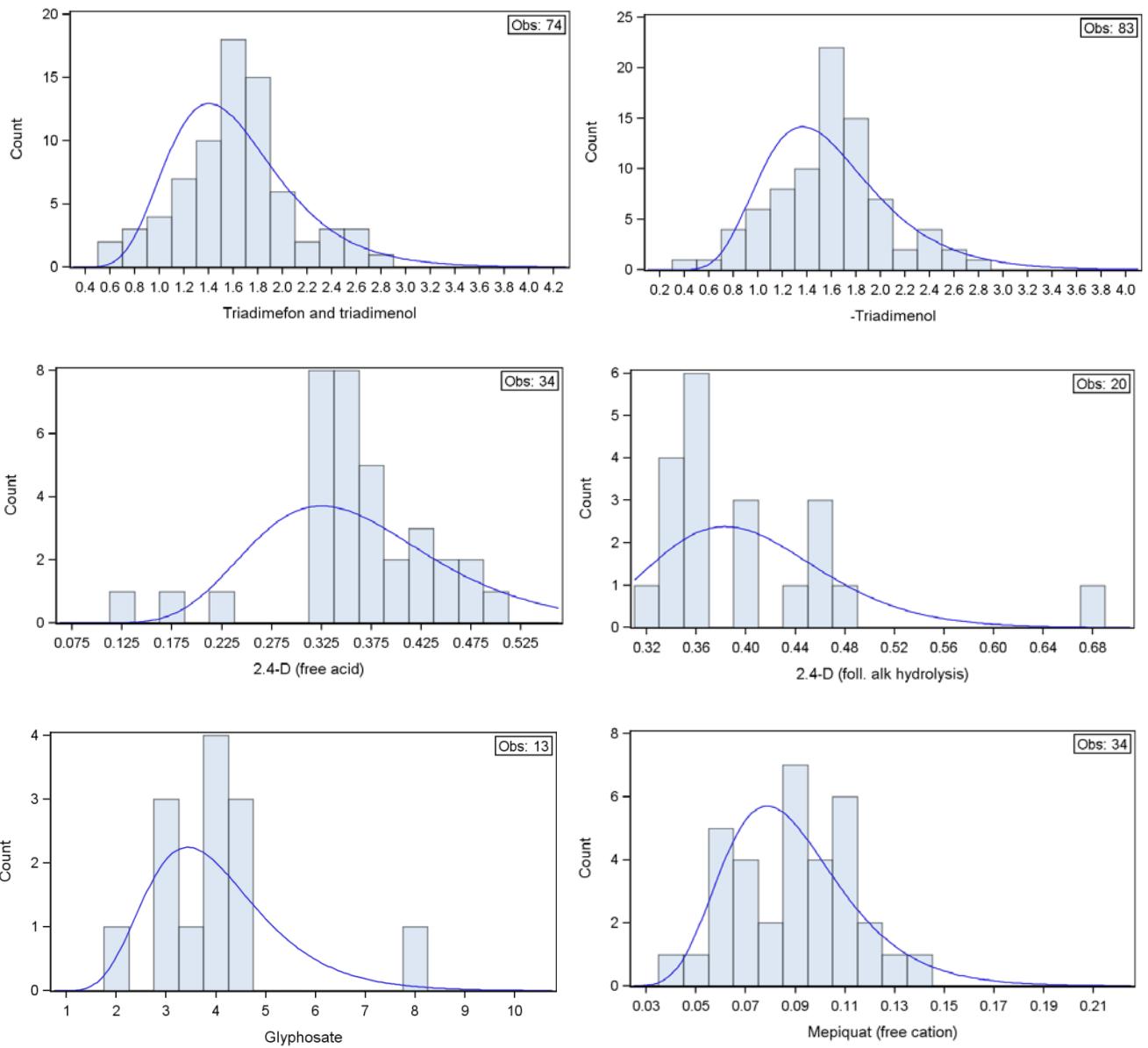
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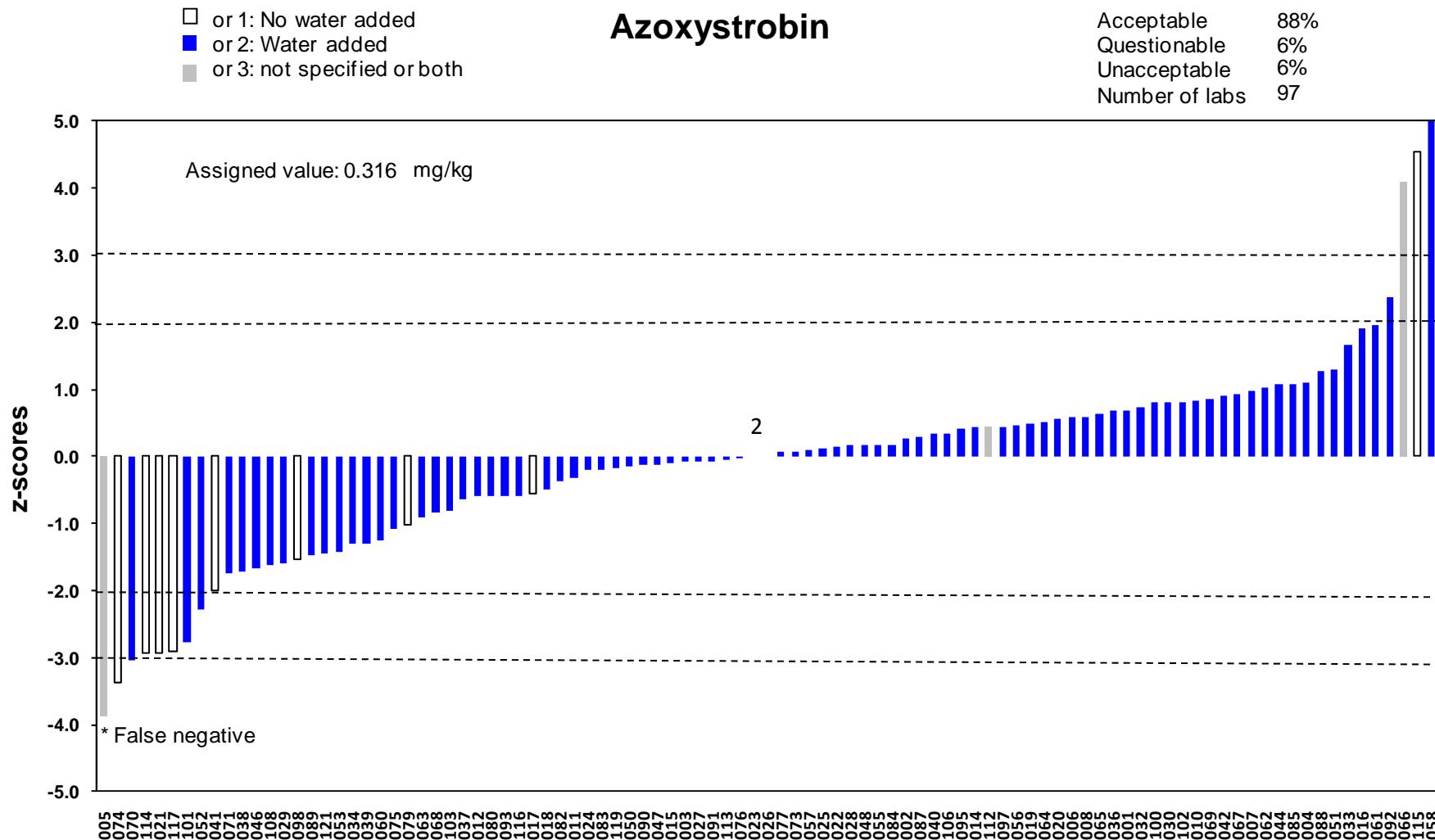
Appendix 5 Histograms of residue data







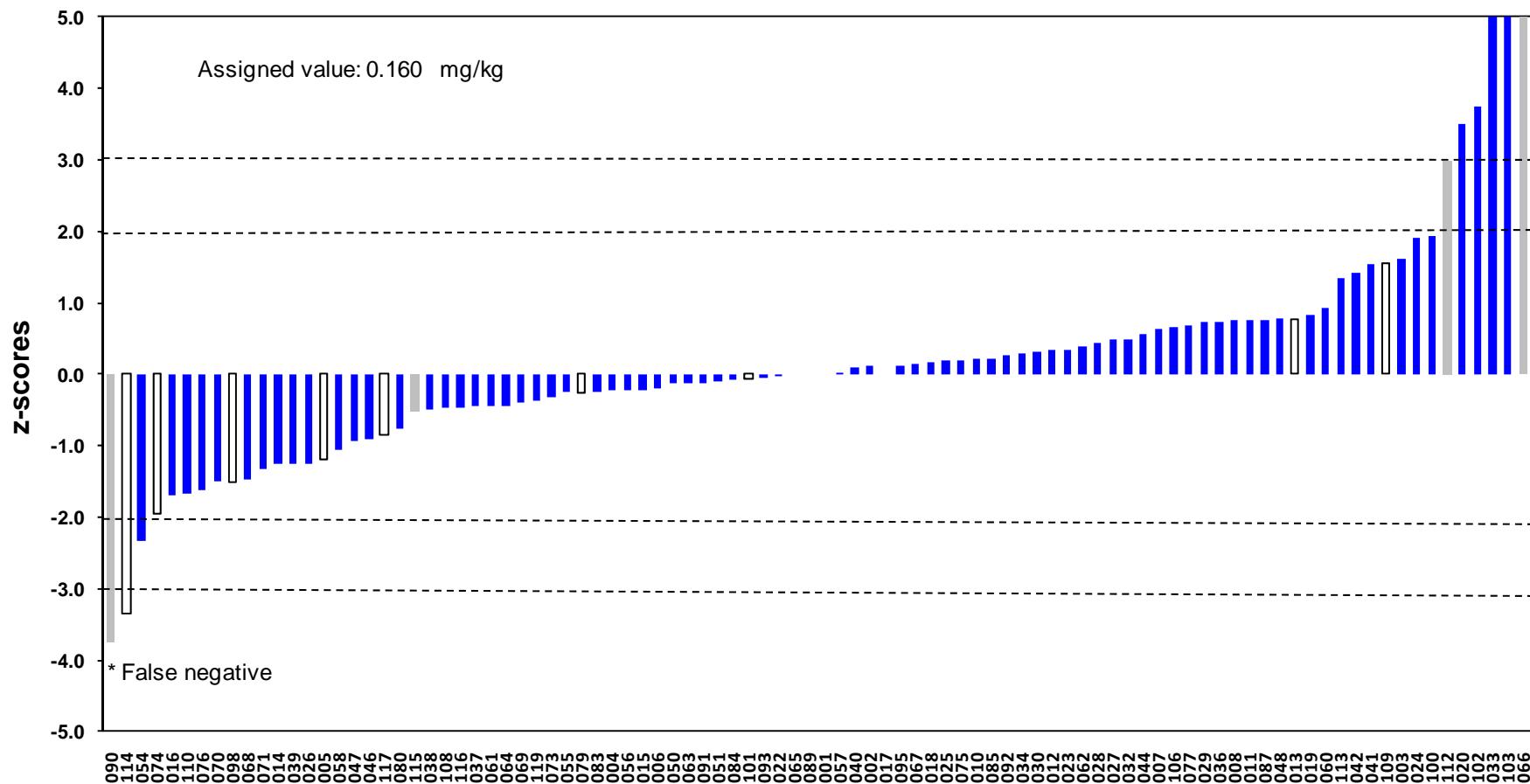
Appendix 6 Graphical presentation of z-scores

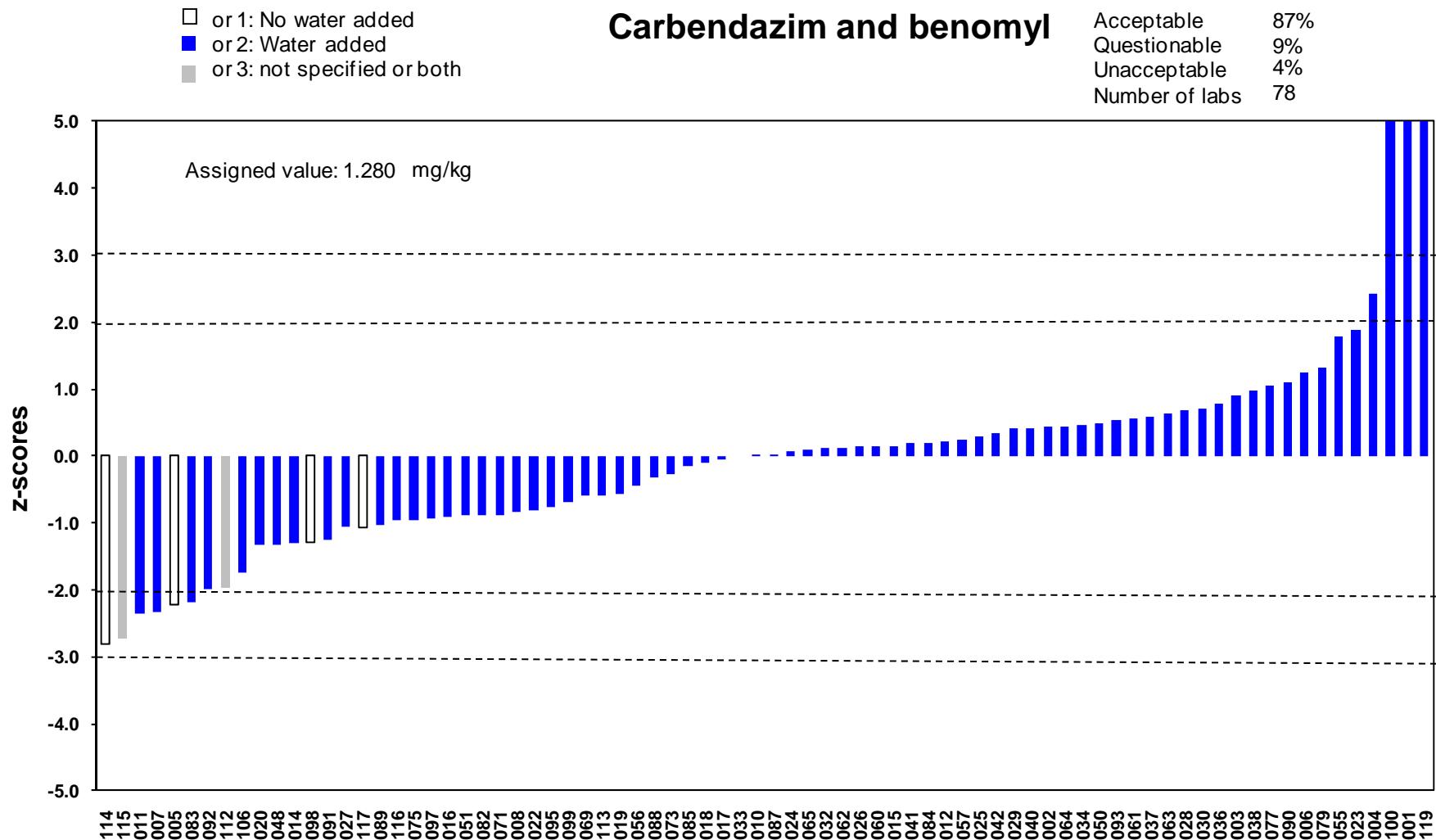


- or 1: No water added
- or 2: Water added
- or 3: not specified or both

Carbaryl

Acceptable	90%
Questionable	2%
Unacceptable	7%
Number of labs	94

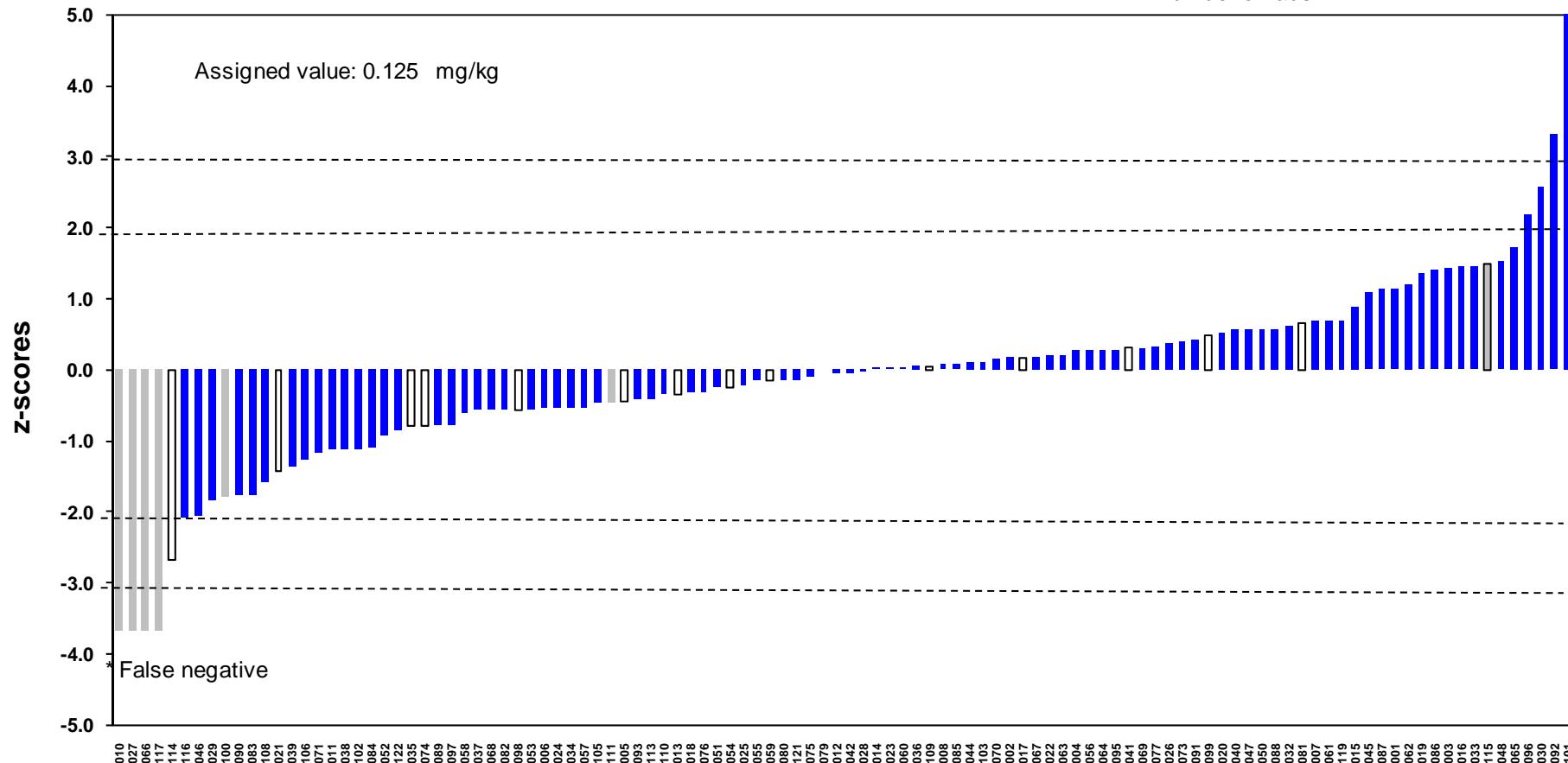


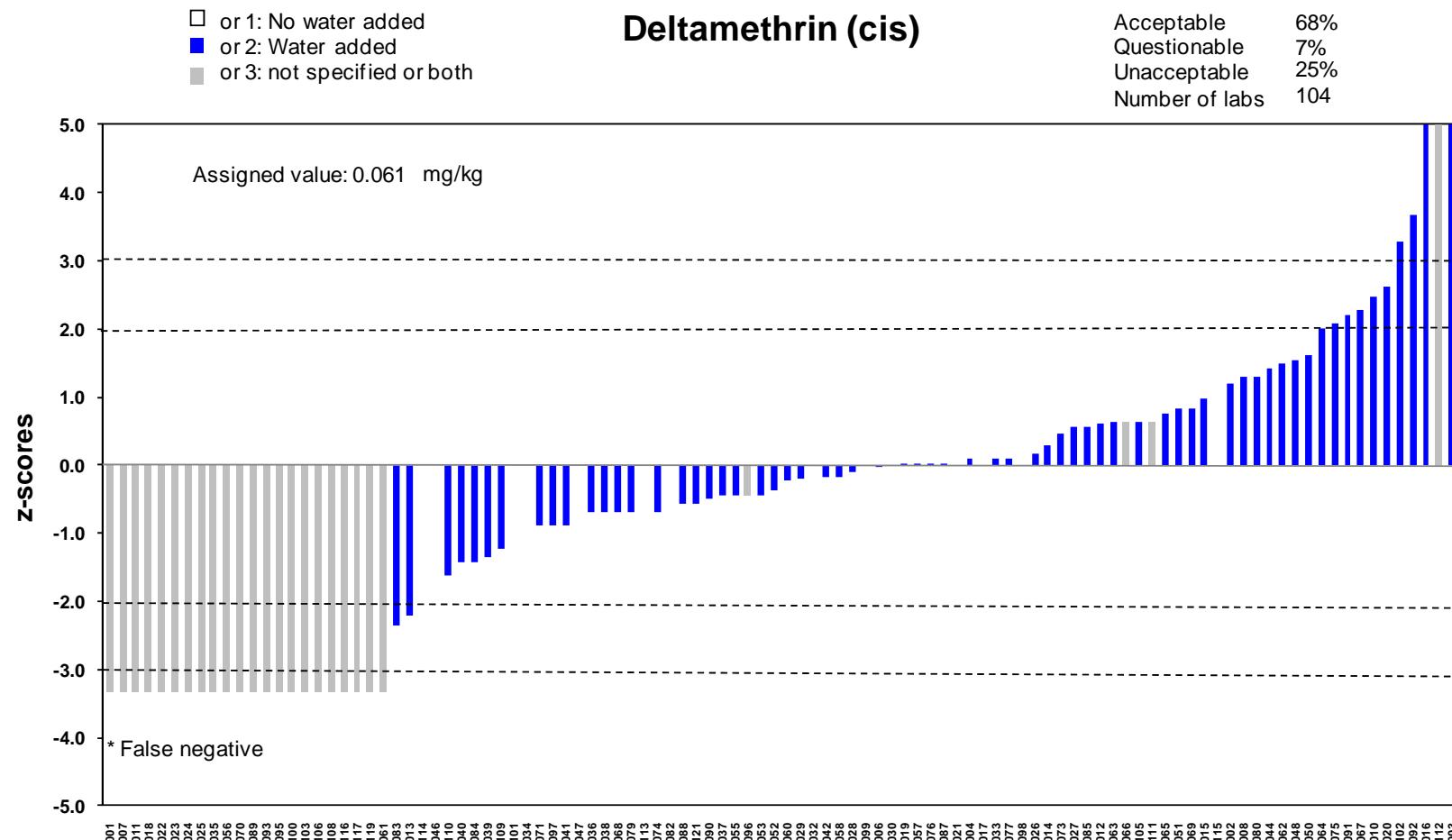


or 1: No water added
 or 2: Water added
 or 3: not specified or both

Chlorpyrifos-methyl

Acceptable	90%
Questionable	5%
Unacceptable	5%
Number of labs	
110	

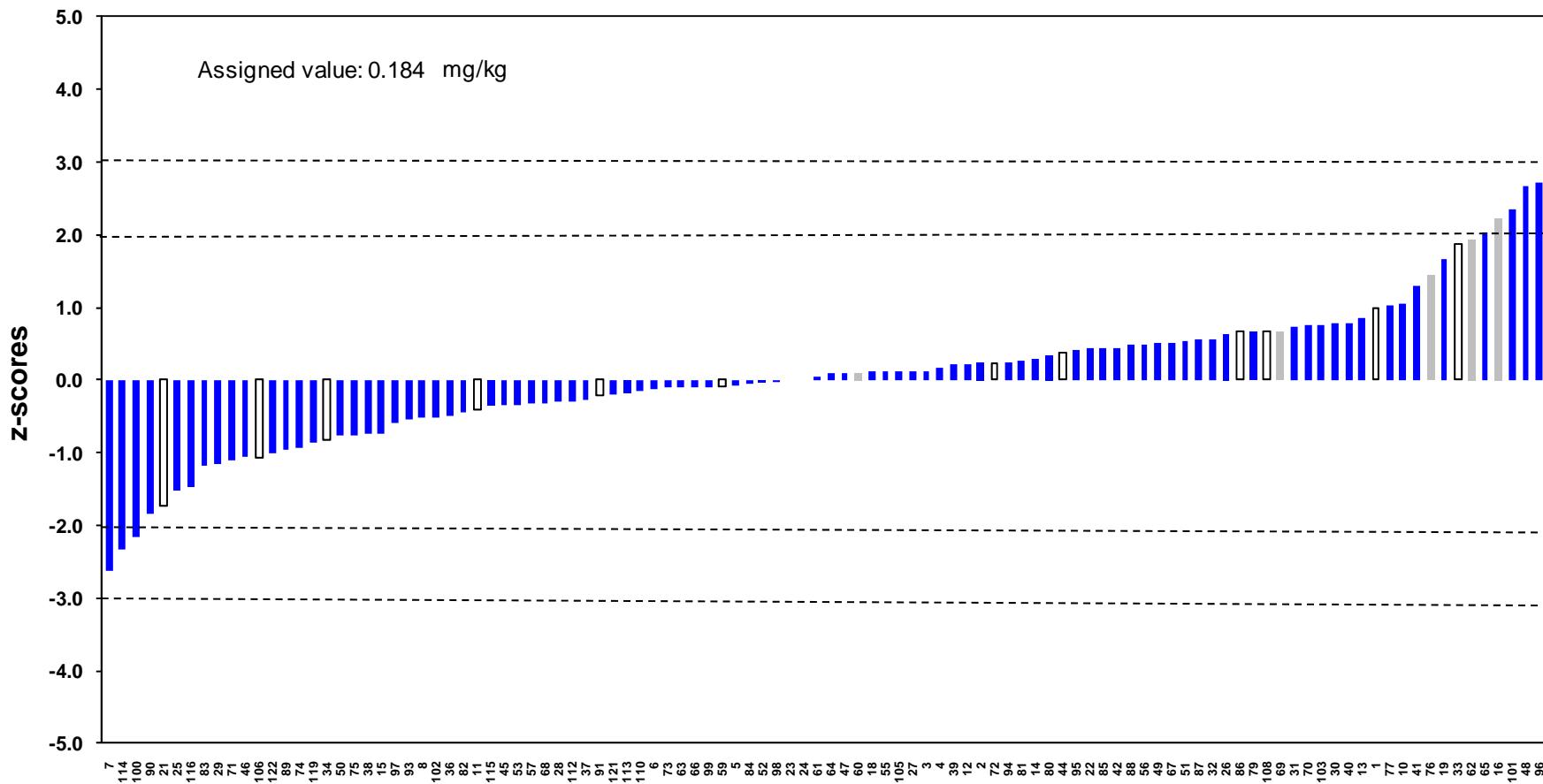


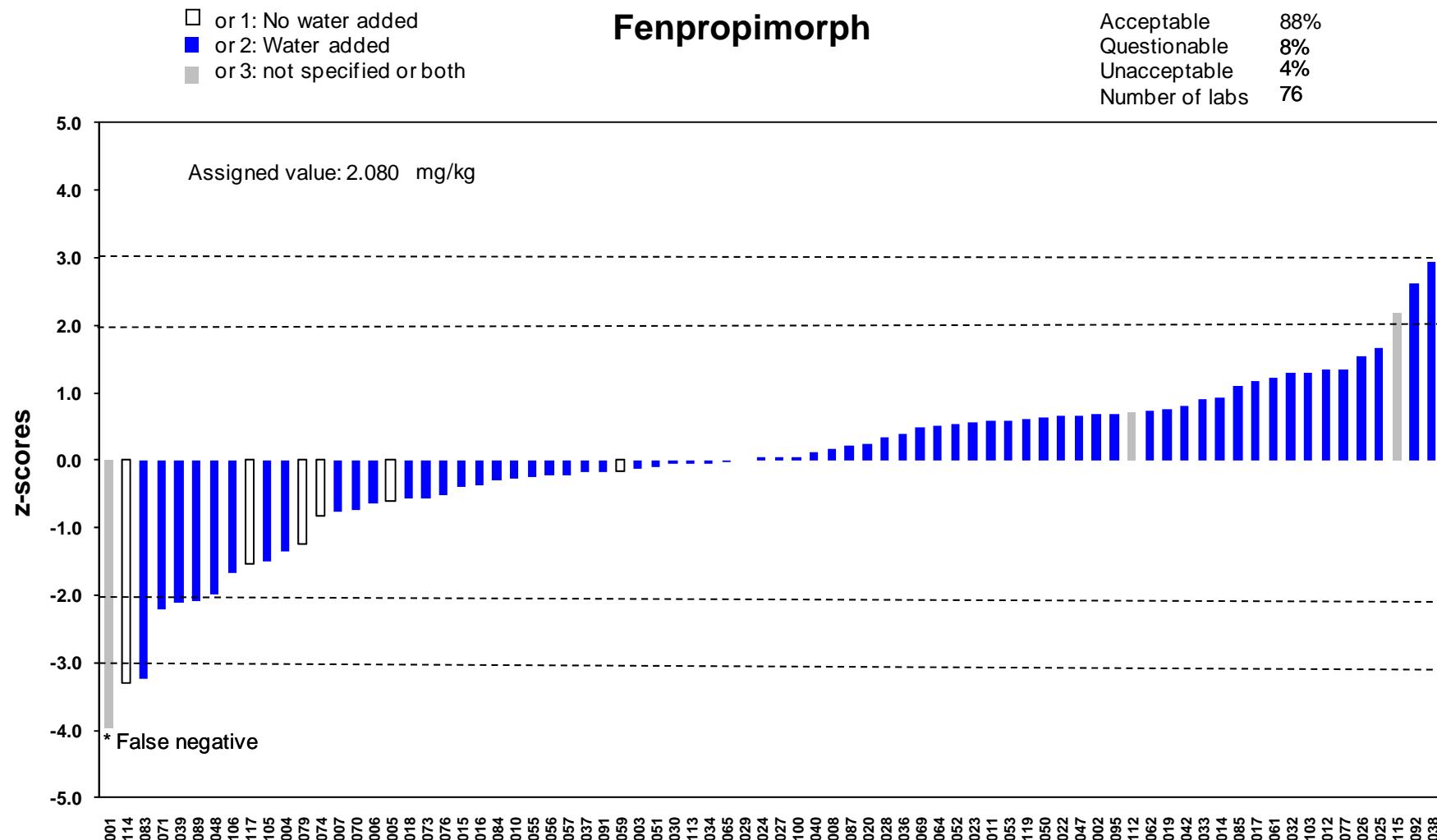


□ or 1: No water added
■ or 2: Water added
■ or 3: not specified or both

Fenitrothion

Acceptable 92%
Questionable 8%
Unacceptable 0%
Number of labs 106

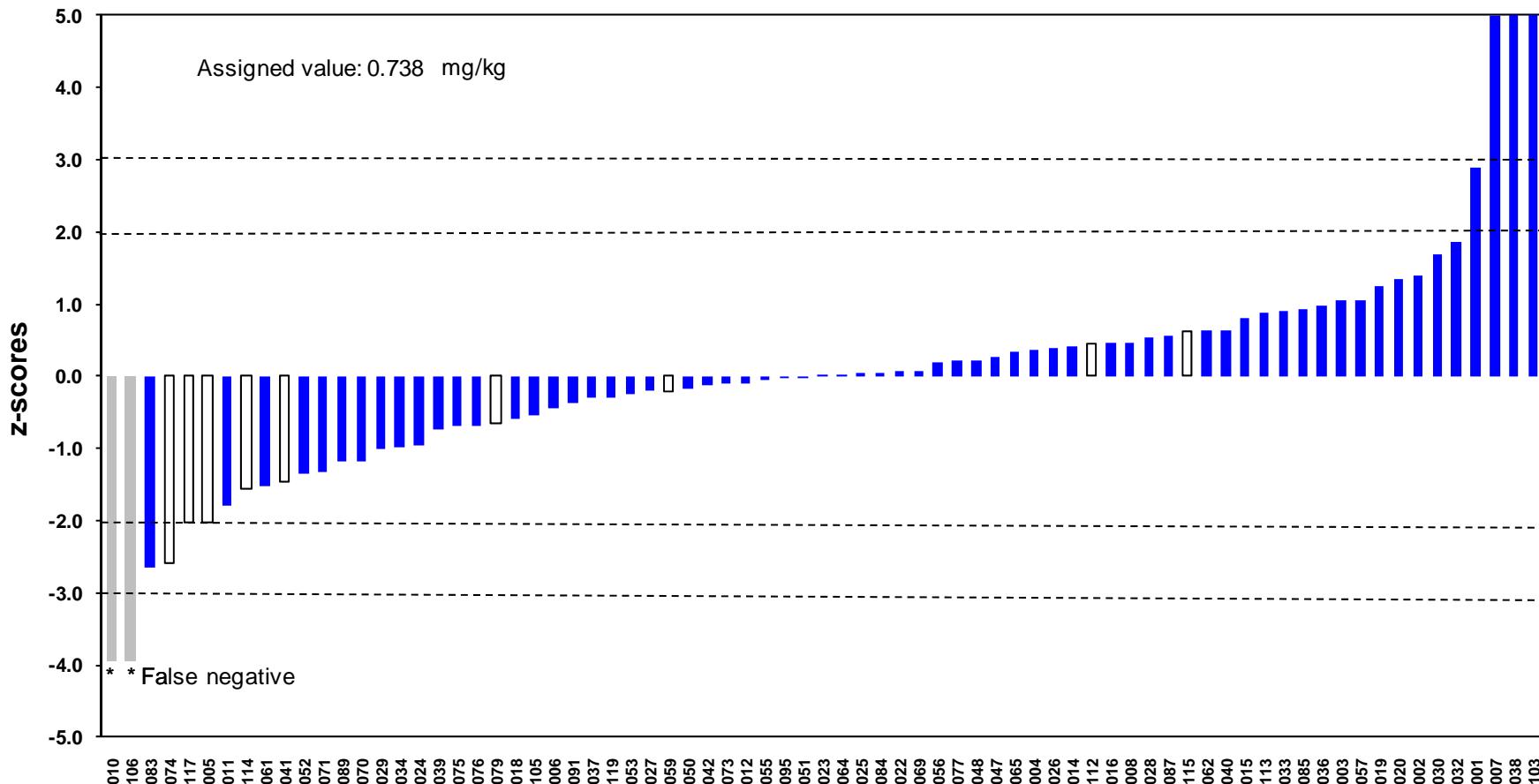


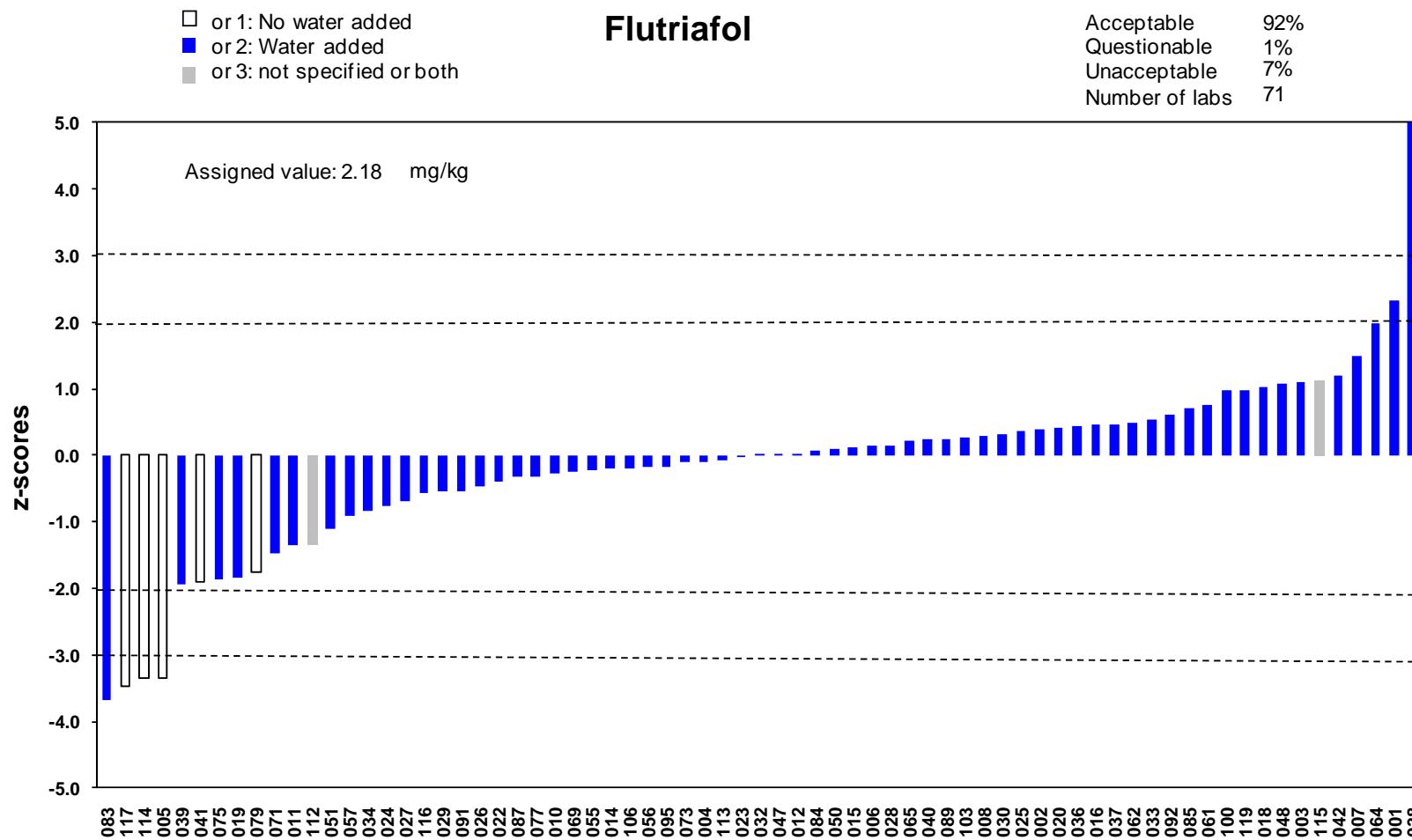


- or 1: No water added
- or 2: Water added
- or 3: not specified or both

Fluquinconazole

Acceptable	87%
Questionable	7%
Unacceptable	7%
Number of labs	75

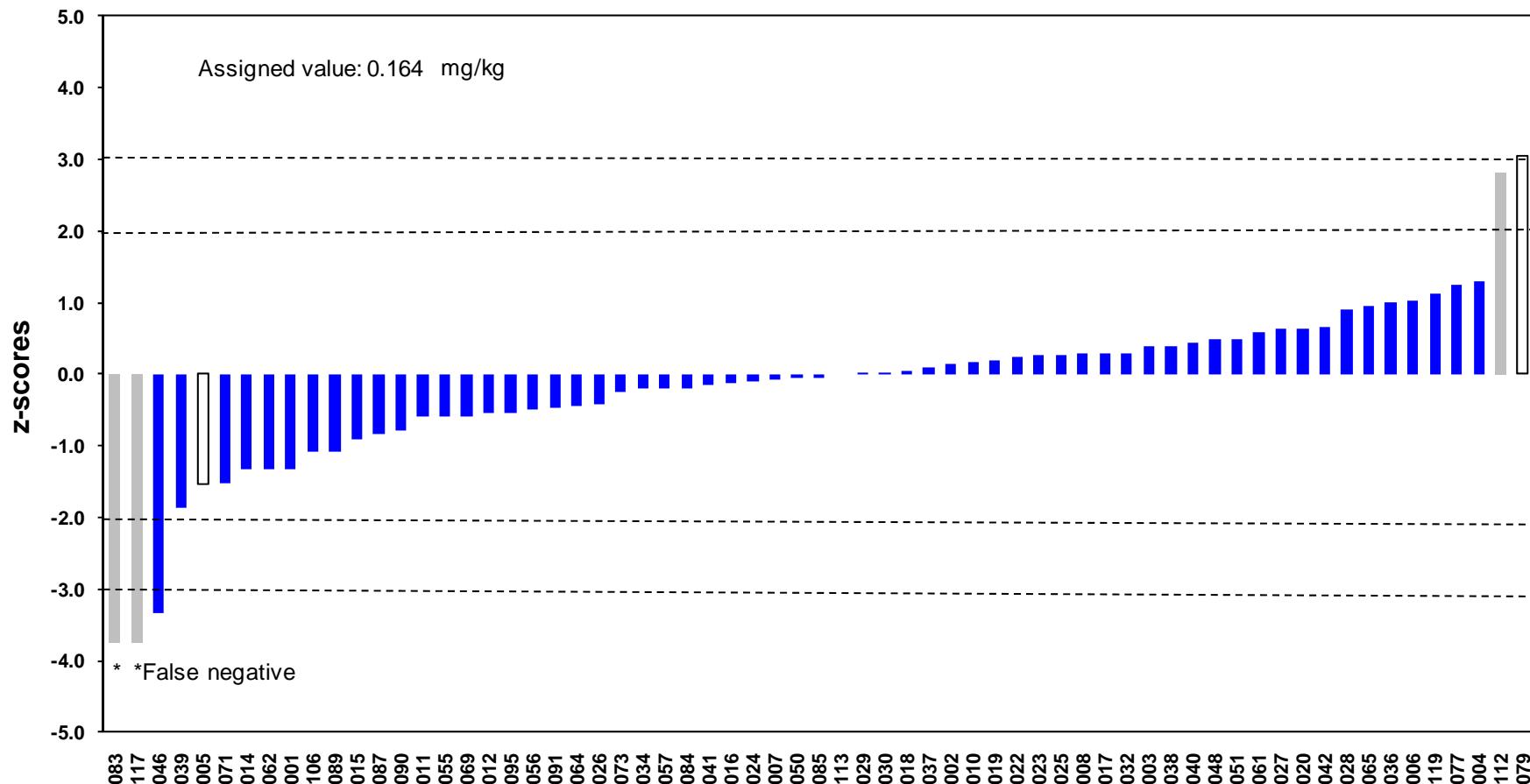


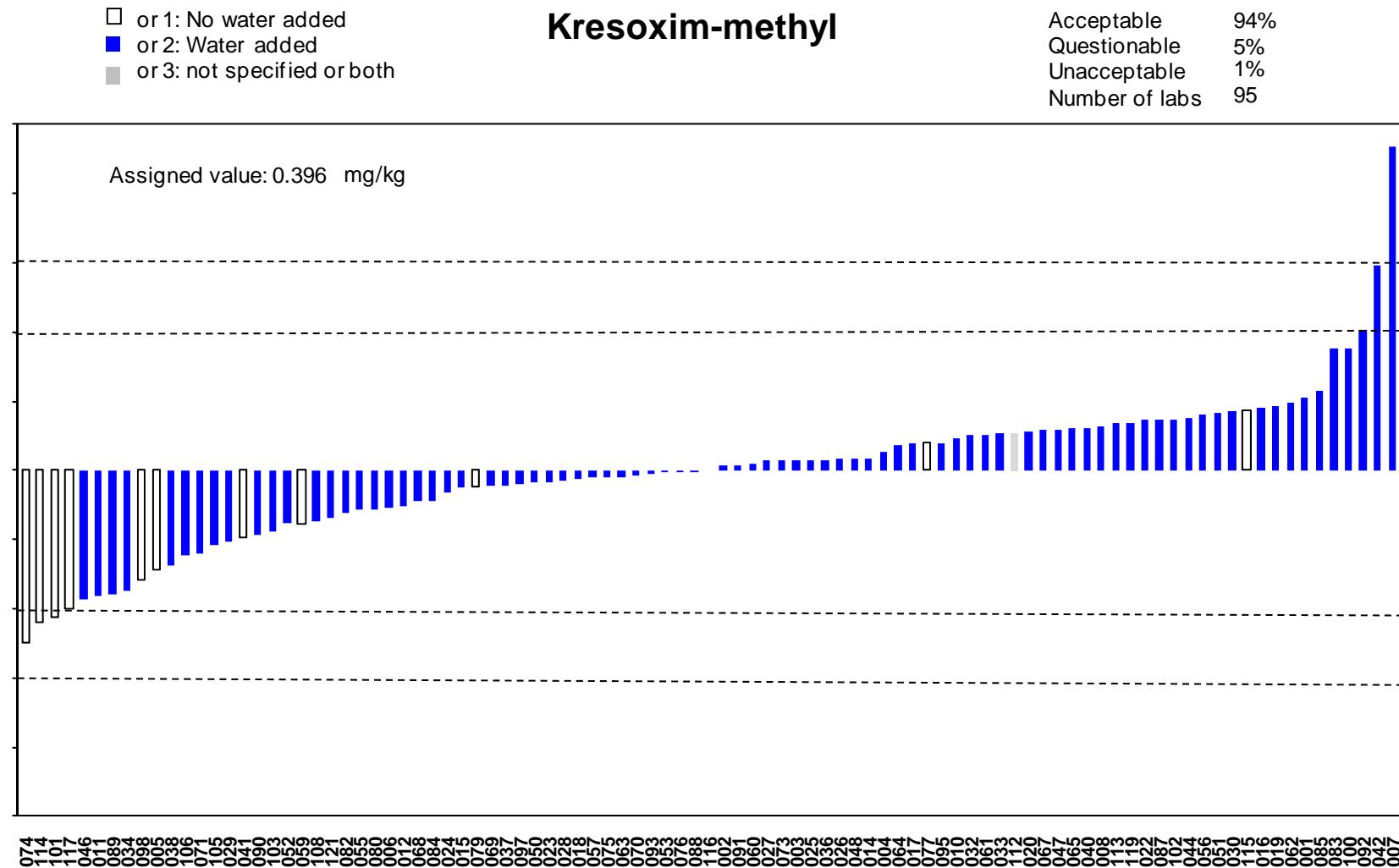


or 1: No water added
 or 2: Water added
 or 3: not specified or both

Isoproturon

Acceptable 92%
 Questionable 2%
 Unacceptable 6%
 Number of labs 65

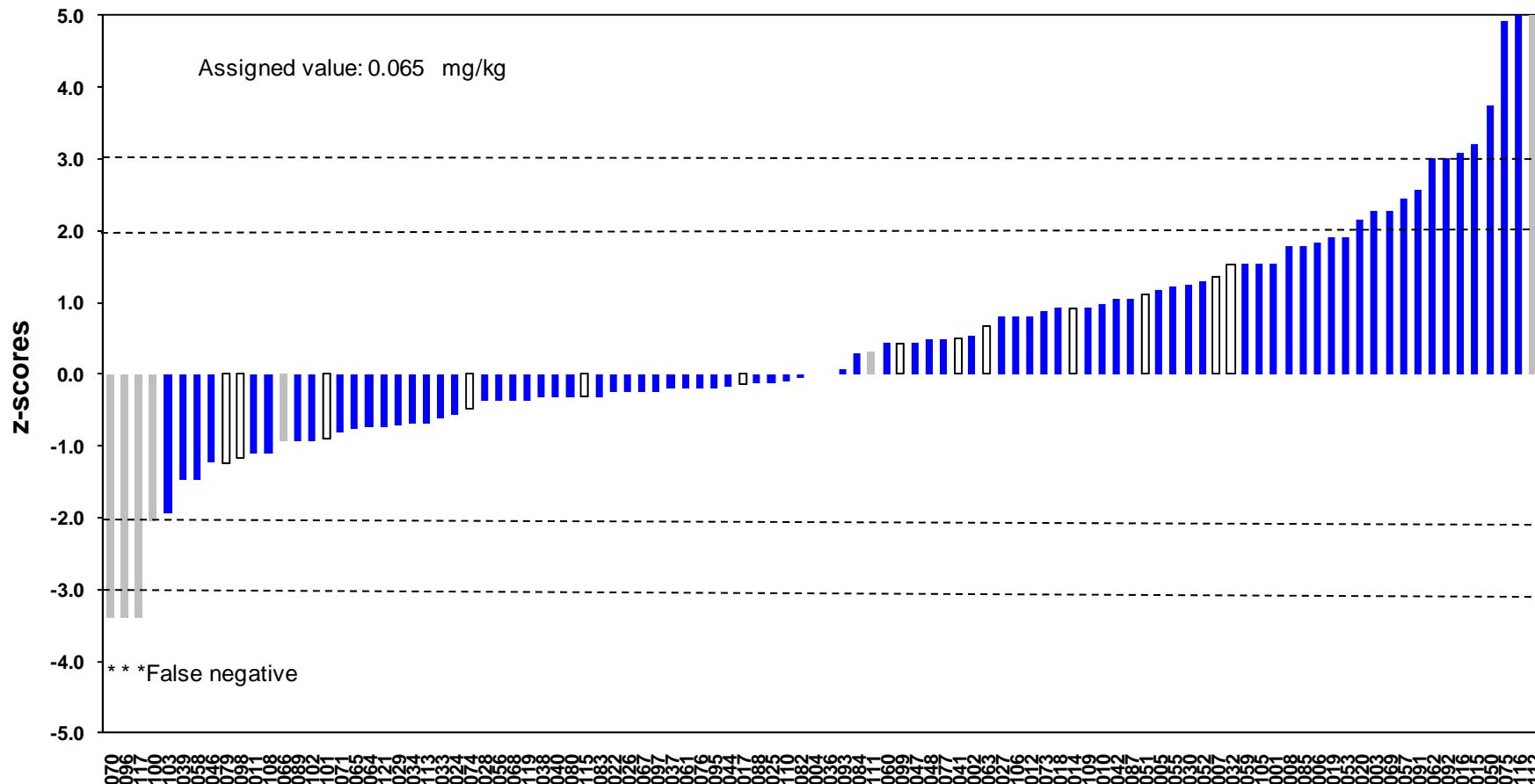


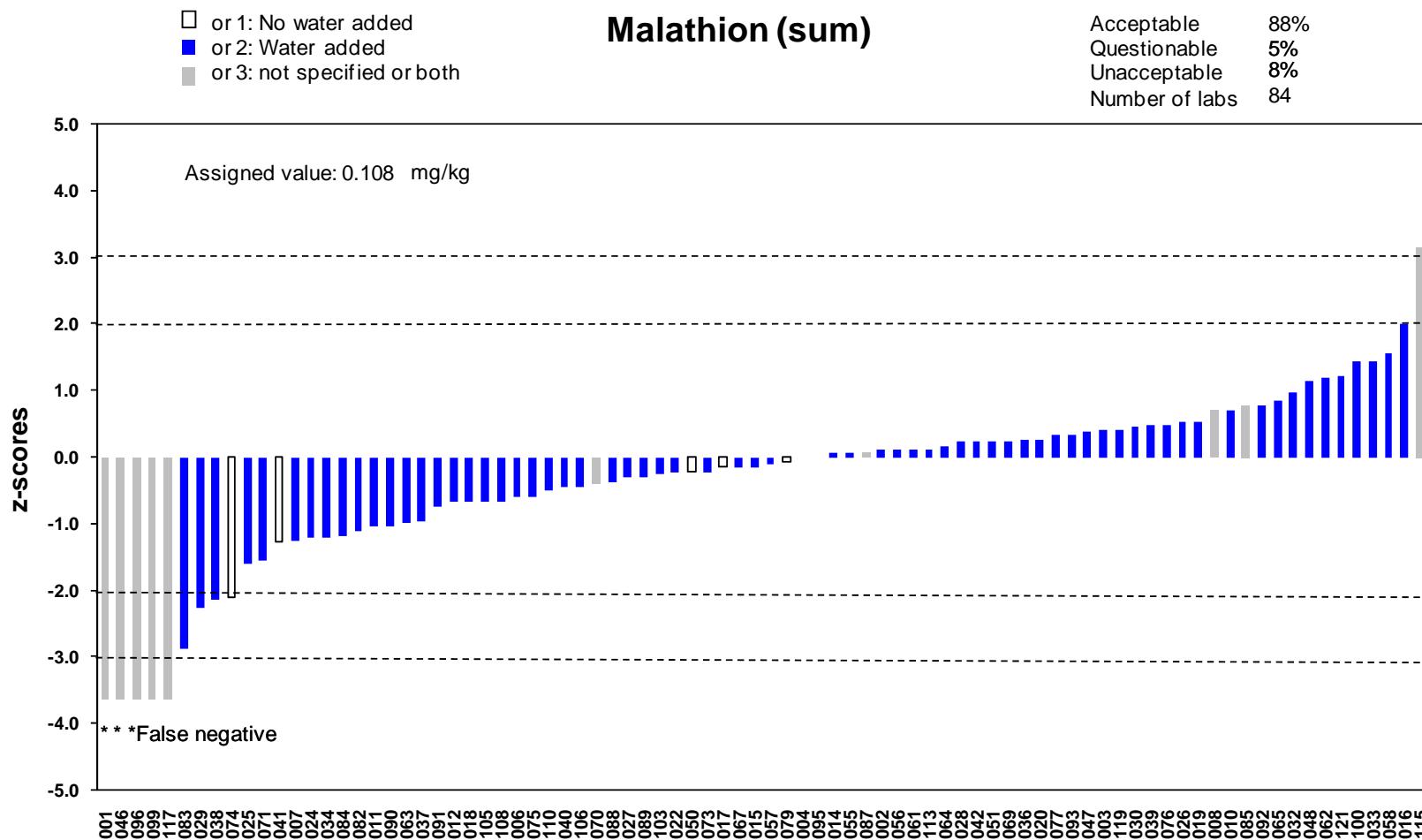


- or 1: No water added
- or 2: Water added
- or 3: not specified or both

Lambda-cyhalothrin

Acceptable	83%
Questionable	6%
Unacceptable	11%
Number of labs	100

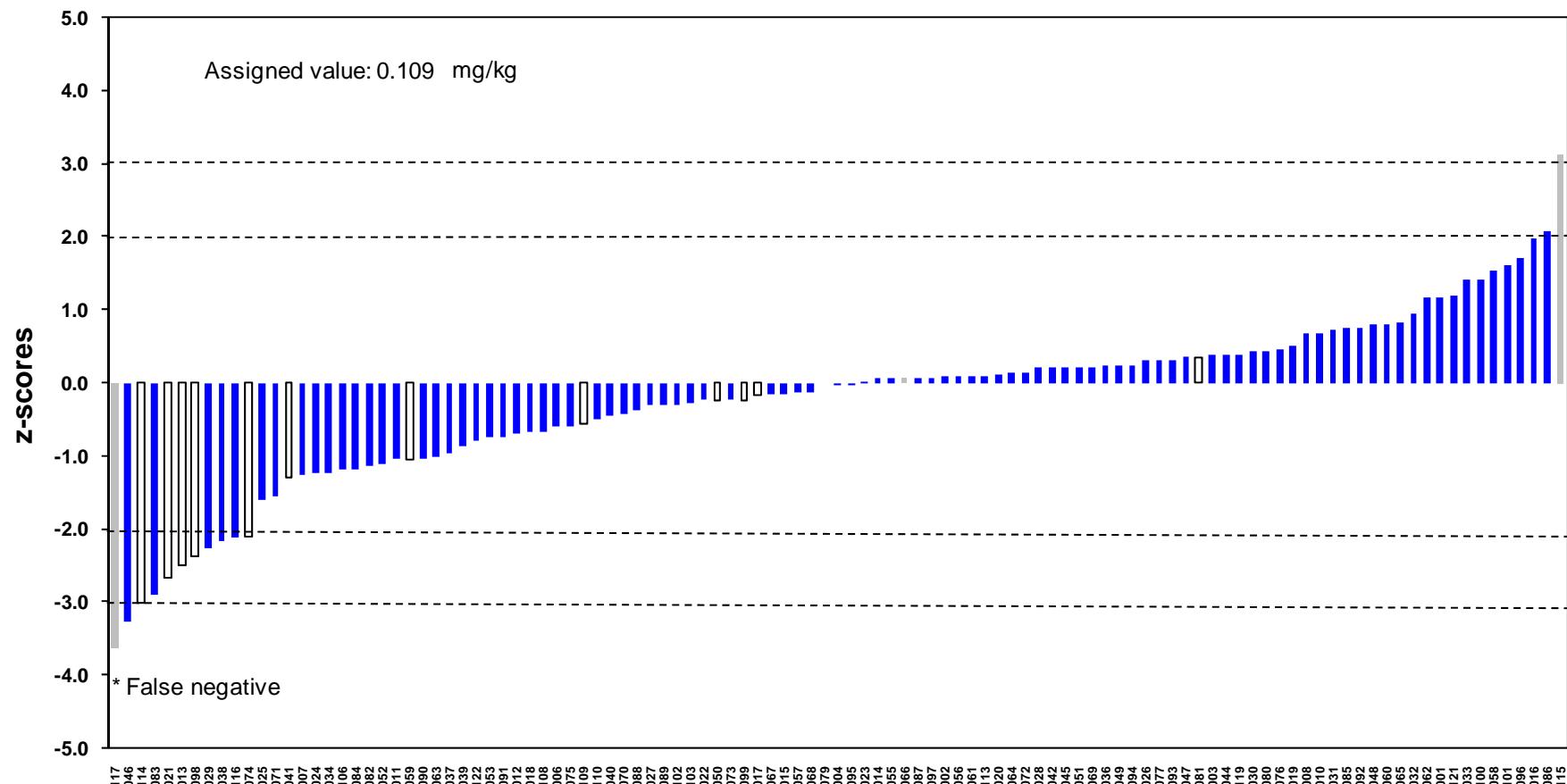




or 1: No water added
 or 2: Water added
 or 3: not specified or both

- Malathion

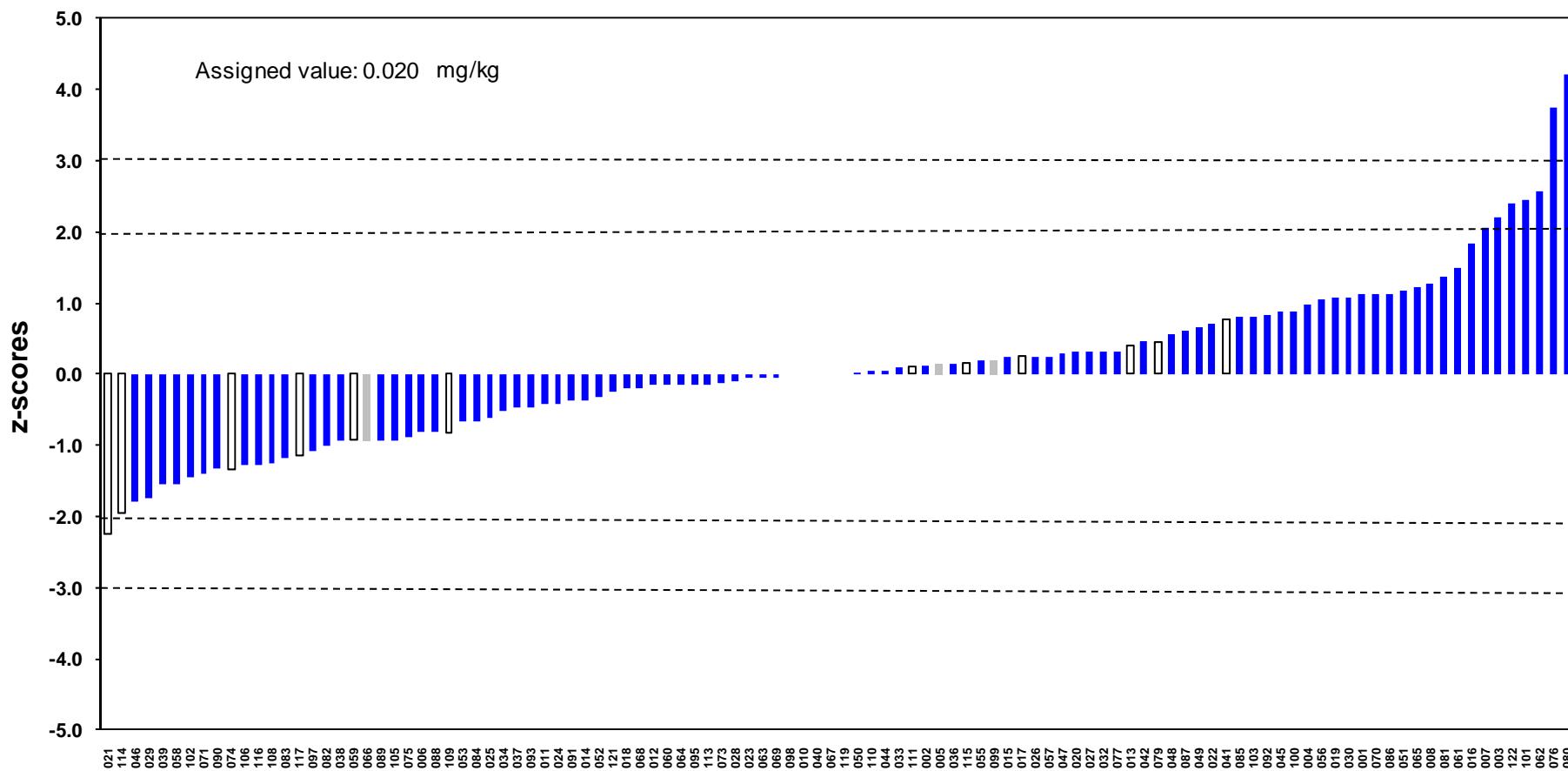
Acceptable	88%
Questionable	8%
Unacceptable	4%
Number of labs	



- or 1: No water added
- or 2: Water added
- or 3: not specified or both

Pirimiphos-methyl

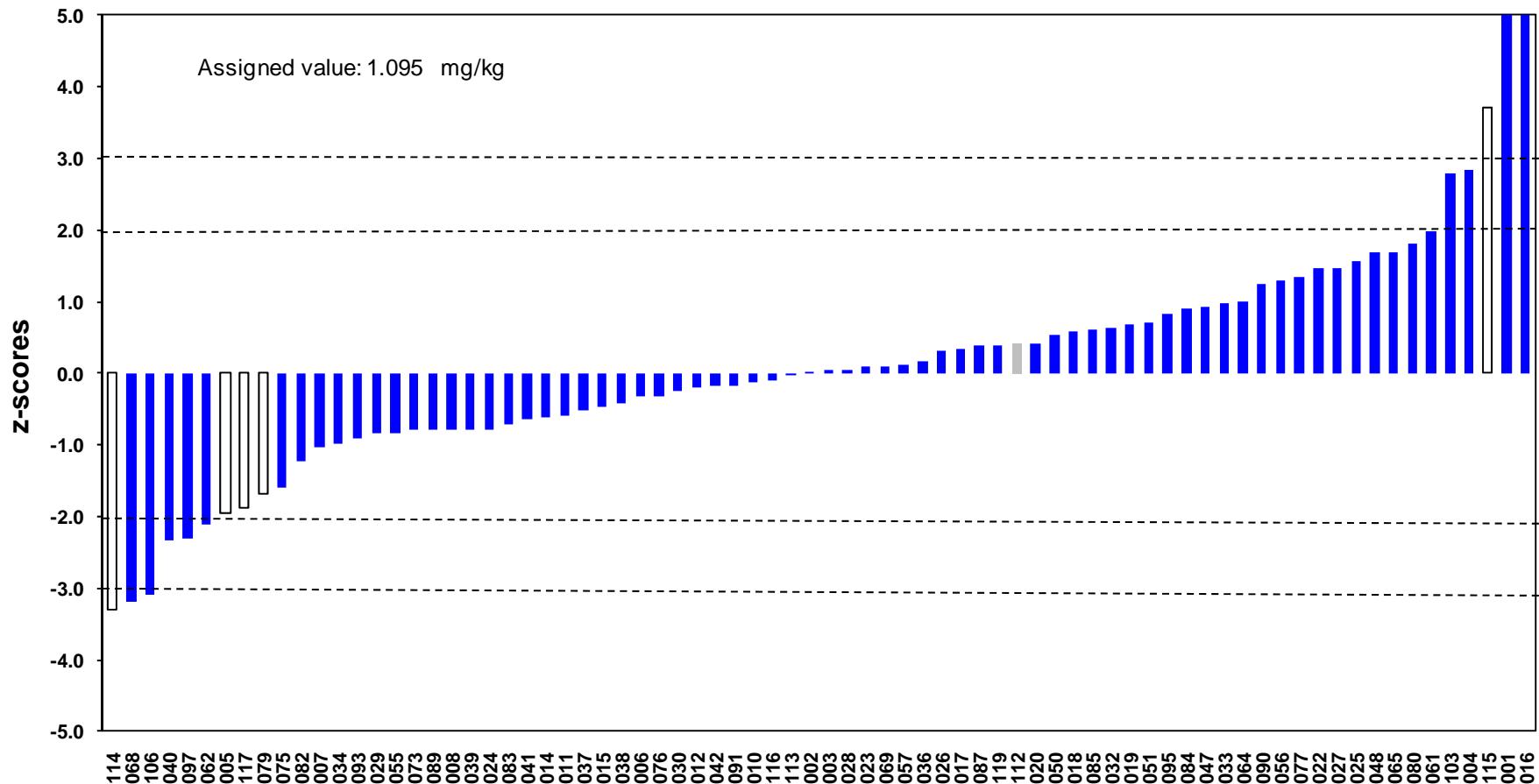
Acceptable	93%
Questionable	6%
Unacceptable	2%
Number of labs	108

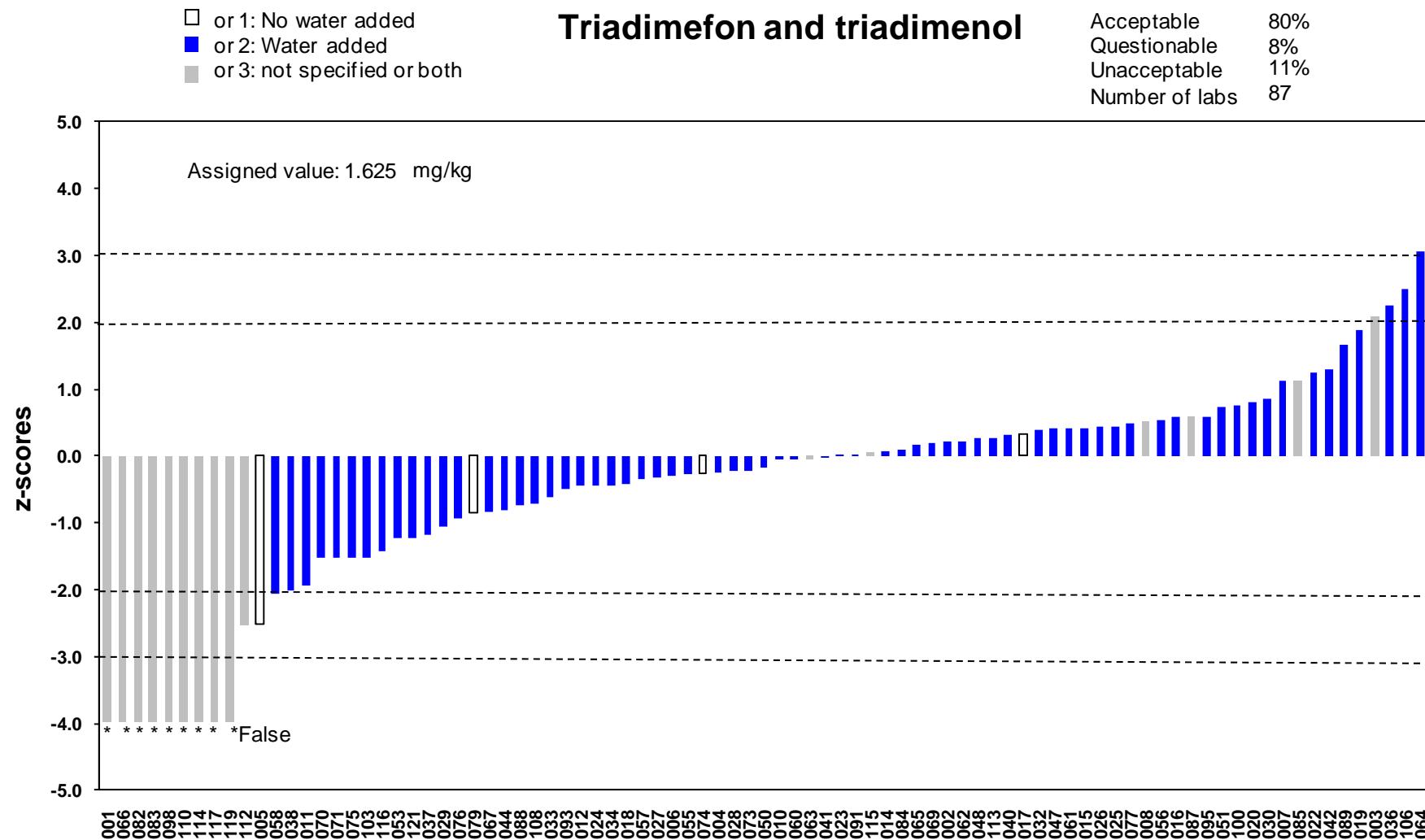


- or 1: No water added
- or 2: Water added
- or 3: not specified or both

Spiroxamine

Acceptable	86%
Questionable	7%
Unacceptable	8%
Number of labs	76

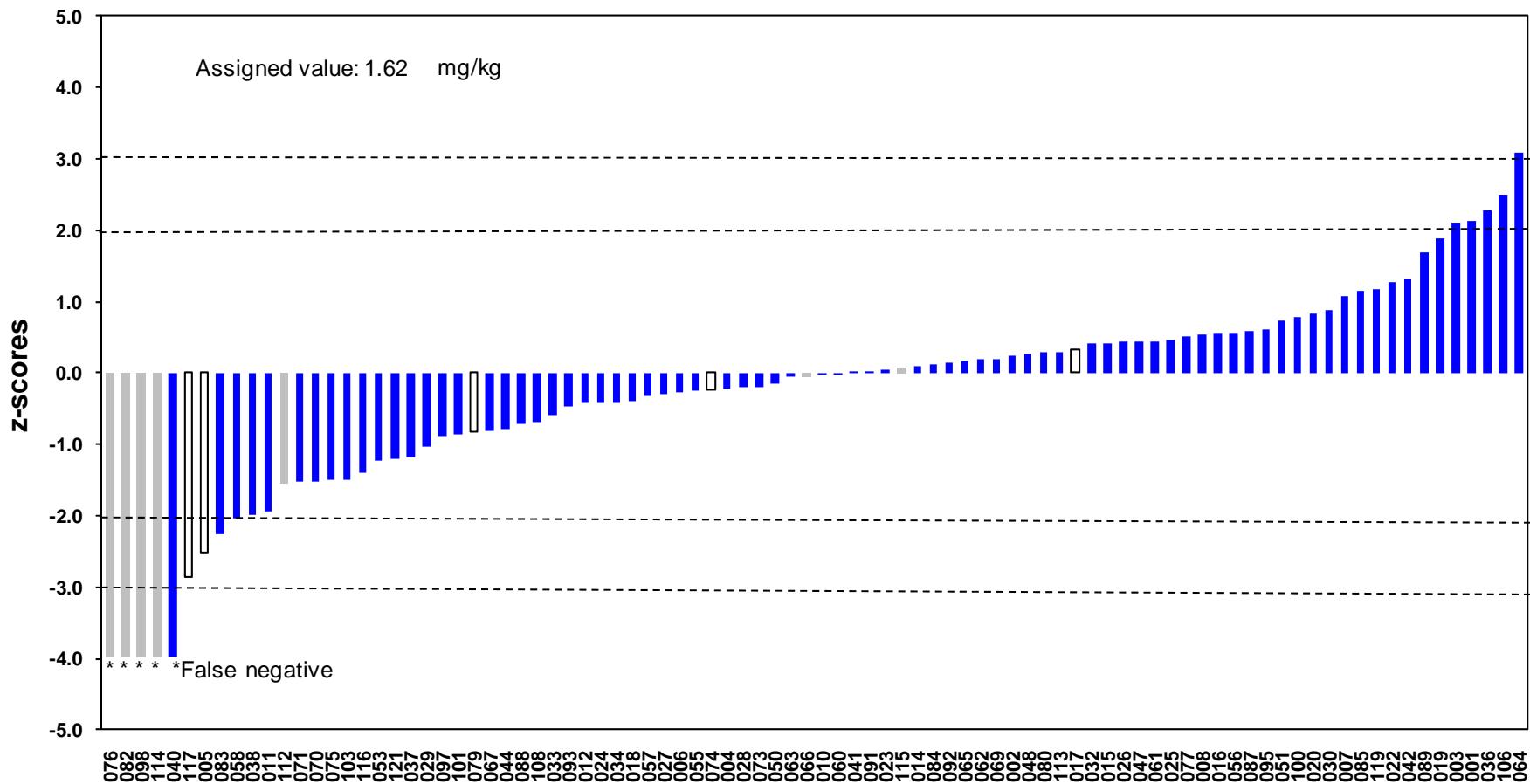




- or 1: No water added
- or 2: Water added
- or 3: not specified or both

-Triadimenol

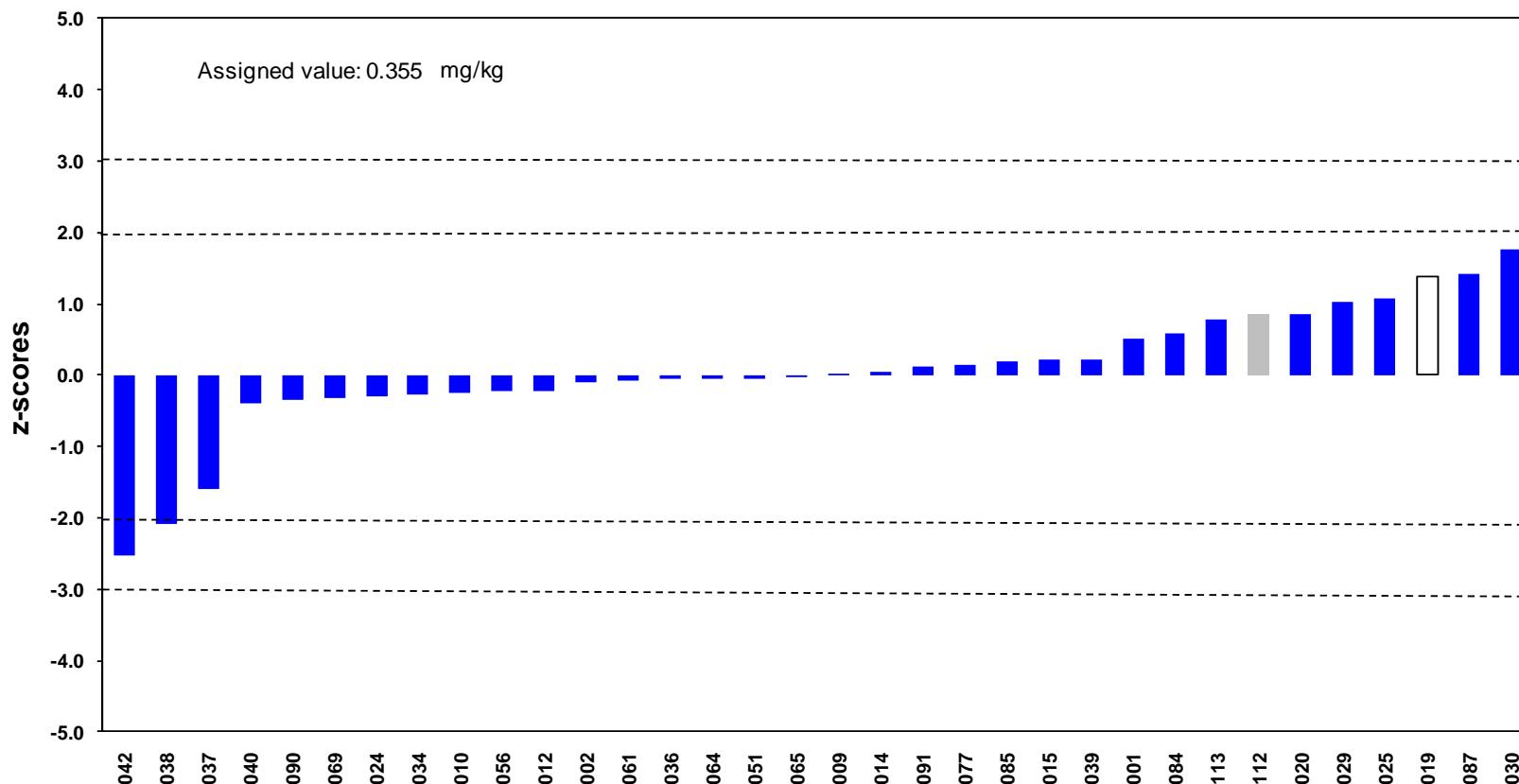
Acceptable	84%
Questionable	9%
Unacceptable	7%
Number of labs	90



□ or 1: No water added
■ or 2: Water added
■ or 3: not specified or both

2,4-D (free acid)

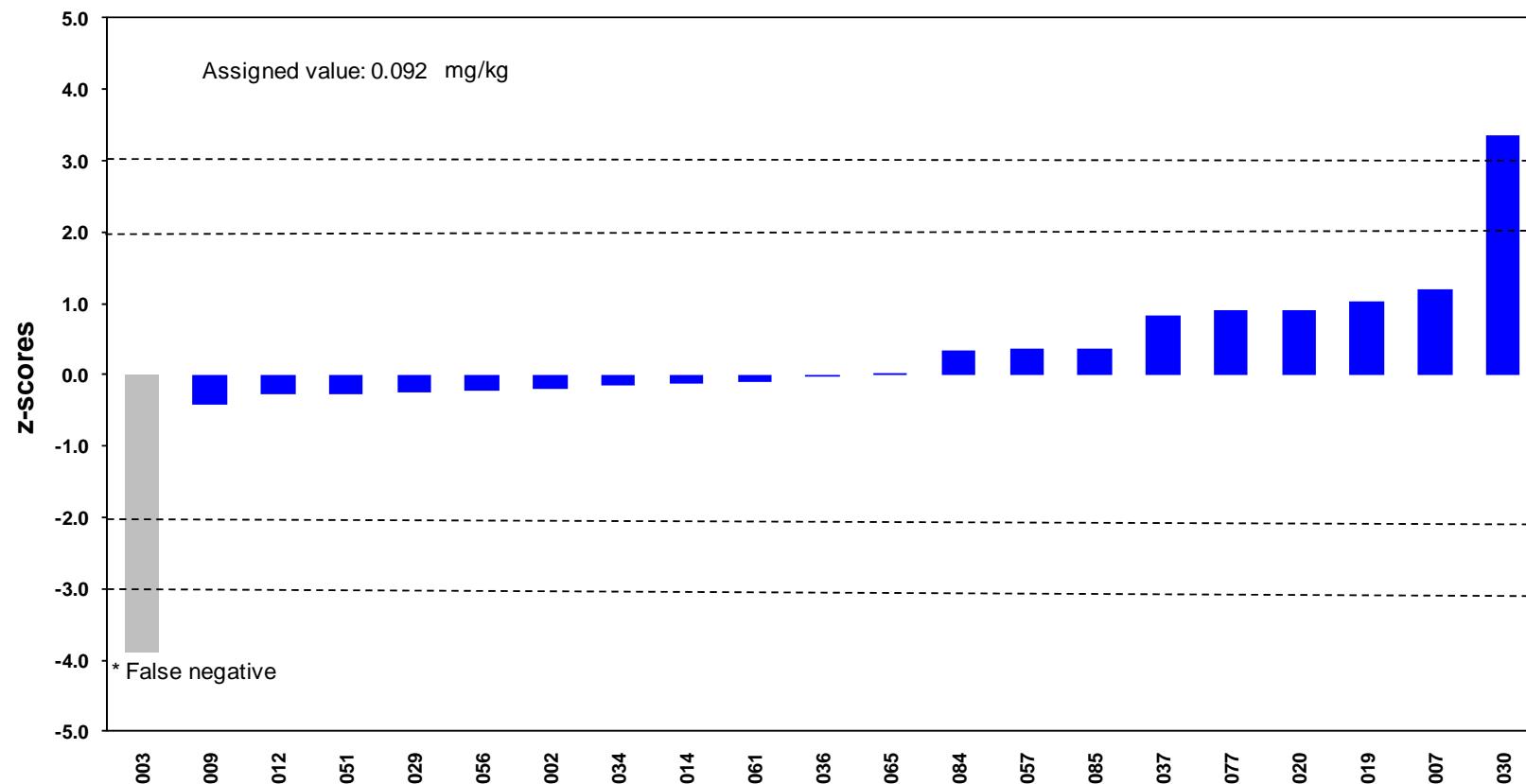
Acceptable 94%
Questionable 6%
Unacceptable 0%
Number of labs 34

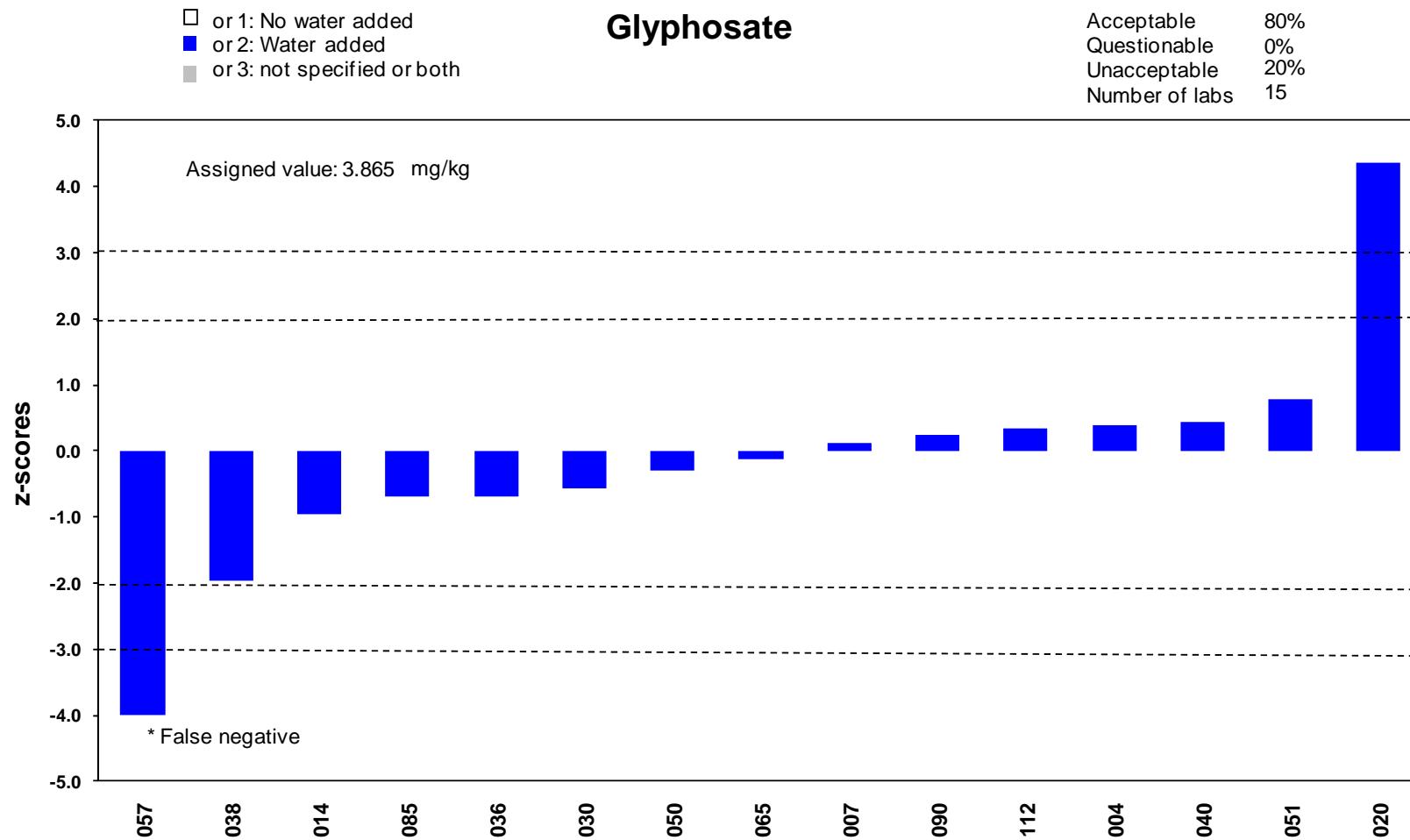


□ or 1: No water added
■ or 2: Water added
■ or 3: not specified or both

2,4-D (foll. alk hydrolysis)

Acceptable 90%
Questionable 0%
Unacceptable 10%
Number of labs 21

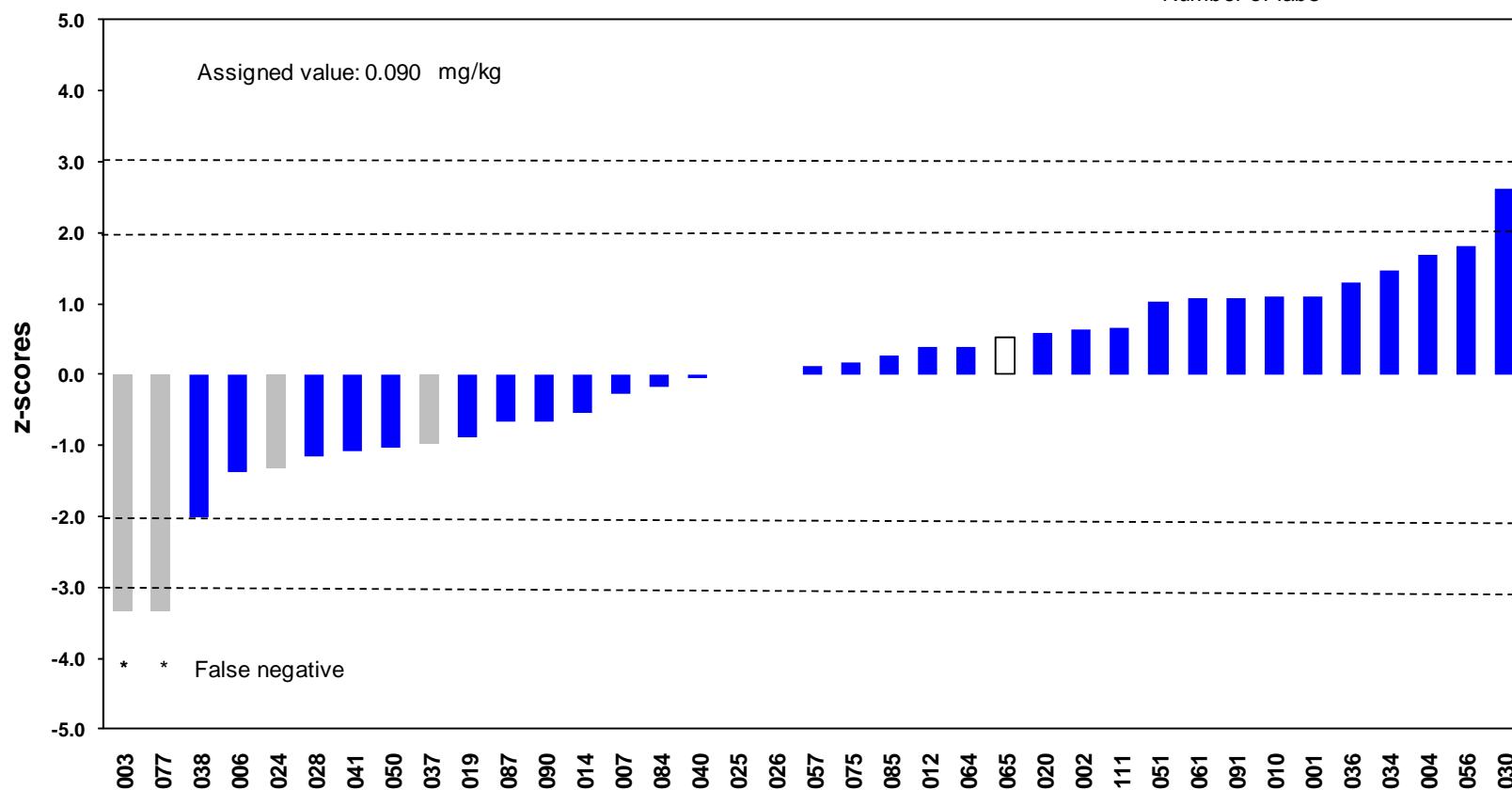




□ or 1: No water added
■ or 2: Water added
■ or 3: not specified or both

Mepiquat (free cation)

Acceptable 92%
Questionable 3%
Unacceptable 5%
Number of labs 37



Appendix 7 Methods used by the participating Laboratories

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery, %	Recovery approach	Sample weight g	Extraction solvent 1) ACN	Extraction solvent 2) ACN	Extraction solvent 3) ACN	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
1	Azoxystrobin	0.37	Yes	94	Same batch	5					Yes	No	DSPE	MM-ML	No	MSD	MS/MS		
1	Carbaryl	0.16	Yes	95	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MSD	MS/MS		
1	Carbendazim and benomyl	4.46	Yes	90	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No		MS/MS		
1	Chlorpyrifos-methyl	0.16	Yes	97	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MSD			
1	Fenitrothion	0.23	Yes	87	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MSD			
1	Fluquinconazole	1.27	Yes	91	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MSD	MS/MS		
1	Flutriafol	3.45	Yes	84	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MSD	MS/MS		
1	Isoproturon	0.11	Yes	101	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MSD			
1	Kresoxim-methyl	0.50	Yes	93	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MSD	MS/MS		
1	Lambda-cyhalothrin	0.09	Yes	88	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MSD			
1	Malathion	0.14	Yes	95	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MSD	MS/MS		
1	Pirimiphos-methyl	0.10	Yes	98	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MSD	MS/MS		
1	Spiroxamine	3.07	Yes	79	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MSD	MS/MS		
1	Triadimenol	2.48	Yes	86	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MSD	MS/MS		
1	2,4-D (free acid)	0.40	Yes	82	Same batch	5	ACN				Yes	No	Freez.	MM-ML	No		MS/MS		
1	Mepiquat (free cation)	0.11	Yes	102	Same batch	5	MeOH				Yes	No	None	MM-ML	Isotop. lab.1		MS/MS		
2	Azoxystrobin	0.337	No	93	Same batch	3	ACN				Yes	No	None	MM-ML	TPP		MS/MS	GC-MS	QuEChERS
2	Carbaryl	0.165	No	93	Same batch	3	ACN				Yes	No	None	MM-ML	TPP		MS/MS	GC-MS	QuEChERS
2	Carbendazim and benomyl	1.42	No	88	Same batch	3	ACN				Yes	No	None	MM-ML	TPP		MS/MS	None	QuEChERS
2	Chlorpyrifos-methyl	0.130	No	94	Same batch	3	ACN				Yes	No	None	MM-ML	TPP		MS/MS	GC-MS	QuEChERS
2	Deltamethrin (cis)	0.0787	No	107	Same batch	3	ACN				Yes	No	None	MM-ML	TPP		MS/MS	GC-MS	QuEChERS
2	Fenitrothion	0.195	No	81	Same batch	6	EtOAc				Yes	No	GPC	MM-ML	TPP	MSD		None	

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
2	Fenpropimorph	2.44	No	102	Same batch	3	ACN				Yes	No	None	MM-ML	TPP		MS/MS	None	QuEChERS
2	Fluquinconazole	0.996	No	86	Same batch	3	ACN				Yes	No	None	MM-ML	TPP		MS/MS	None	QuEChERS
2	Flutriafol	2.39	No	89	Same batch	3	ACN				Yes	No	None	MM-ML	TPP		MS/MS	GC-MS	QuEChERS
2	Isoproturon	0.170	No	95	Same batch	3	ACN				Yes	No	None	MM-ML	TPP		MS/MS	None	QuEChERS
2	Kresoxim-methyl	0.404	No	96	Same batch	3	ACN				Yes	No	None	MM-ML	TPP		MS/MS	GC-MS	QuEChERS
2	Lambda-cyhalothrin	0.0739	No	87	Same batch	6	EtOAc				Yes	No	GPC	MM-ML	TPP	MSD	LC-MS/MS		
2	Malathion	0.111	No	95	Same batch	3	ACN				Yes	No	None	MM-ML	TPP		MS/MS	GC-MS	QuEChERS
2	Pirimiphos-methyl	0.0802	No	91	Same batch	3	ACN				Yes	No	None	MM-ML	TPP		MS/MS	GC-MS	QuEChERS
2	Spiroxamine	1.10	No	96	Same batch	3	ACN				Yes	No	None	MM-ML	TPP		MS/MS	None	QuEChERS
2	Triadimenol	1.71	No	100	Same batch	3	ACN				Yes	No	None	MM-ML	TPP		MS/MS	GC-MS	QuEChERS
2	2,4-D (free acid)	0.345	No	95	Same batch	3	ACN				Yes	No	None	MM-ML	TPP		MS/MS	None	QuEChERS
2	2,4-D (foll. alk hydrolysis)	0.350	No	96	Same batch	3	ACN				Yes	Yes	None	MM-ML	TPP		MS/MS	None	QuEChERS
2	Mepiquat (free cation)	0.0991	No	119	Same batch	5	MeOH				Yes	No		MM-ML	Isotop. lab.1		MS/MS	None	
3	Azoxystrobin	0.310	No	84	Same batch	10	AC	CH ₂ Cl	PE		Yes	No	None	MM-ML		ECD	LC-MS/MS		
3	Carbaryl	0.225	No	103	Same batch	10	AC	CH ₂ Cl	PE		Yes	No	None	MM-ML		NPD	LC-MS/MS		
3	Carbendazim and benomyl	1.57	No	99	Same batch	10	AC	CH ₂ Cl	PE		Yes	No	None	MM-ML		MS/MS	LC-MS/MS		
3	Chlorpyrifos-methyl	0.169	No	93	Same batch	10	AC	CH ₂ Cl	PE		Yes	No	None	MM-ML		ECD	LC-MS/MS		
3	Deltamethrin (cis)	0.905	No	90	Same batch	10	AC	CH ₂ Cl	PE		Yes	Yes	None	MM-ML		ECD	Two columns		
3	Fenitrothion	0.190	No	108	Same batch	10	AC	CH ₂ Cl	PE		Yes	No	None	MM-ML		MS/MS	LC-MS/MS		
3	Fenpropimorph	2.02	No	99	Same batch	10	AC	CH ₂ Cl	PE		Yes	No	None	MM-ML		NPD	LC-MS/MS		
3	Fluquinconazole	0.93	No	85	Same batch	10	AC	CH ₂ Cl	PE		Yes	No	None	MM-ML		ECD	LC-MS/MS		
3	Flutriafol	2.78	No	114	Same batch	10	AC	CH ₂ Cl	PE		Yes	No	None	MM-ML		MS/MS	LC-MS/MS		
3	Isoproturon	0.18	No	100	Same batch	10	AC	CH ₂ Cl	PE		Yes	No	None	MM-ML		ECD	LC-MS/MS		
3	Kresoxim-methyl	0.411	No	84	Same batch	10	AC	CH ₂ Cl	PE		Yes	No	None	MM-ML		ECD	LC-MS/MS		
3	Lambda-cyhalothrin	0.102	No	93	Same batch	10	AC	CH ₂ Cl	PE		Yes	Yes	None	MM-ML		ECD	Two columns		

LAB CODE	Pesticide	Reported result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) AC	Extraction solvent 2) CH2Cl	Extraction solvent 3) PE	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
3	Malathion	0.119	No	80	Same batch	10	AC	CH2Cl	PE		Yes	No	None	MM-ML		ECD	LC-MS/MS		
3	Pirimiphos-methyl	0.121	No	97	Same batch	10	AC	CH2Cl	PE		Yes	No	None	MM-ML		ECD	LC-MS/MS		
3	Spiroxamine	1.11	No	98	Same batch	10	AC	CH2Cl	PE		Yes	No	None	MM-ML		NPD	LC-MS/MS		
3	Triadimenol	2.47	No	90	Same batch	10	AC	CH2Cl	PE		Yes	No	None	MM-ML		MS/MS	LC-MS/MS		
4	Azoxystrobin	0.403	No	96	Same batch	5	ACN				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD	GC-MS		
4	Carbaryl	0.151	No	98	Same batch	5	ACN				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD	GC-MS		
4	Carbendazim and benomyl	2.06	No	97	Same batch	5	ACN				Yes	No	None	MM-ML	No	MS/MS	LC-MS/MS	Quechers	
4	Chlorpyrifos-methyl	0.133	No	98	Same batch	5	ACN				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD	GC-MS		
4	Deltamethrin (cis)	0.062	No	92	Same batch	5	ACN				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD	GC-MS		
4	Fenitrothion	0.192	No	102	Same batch	5	ACN				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD	GC-MS		
4	Fenpropimorph	1.38	No	68	Same batch	5	ACN				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD	GC-MS		
4	Fluquinconazole	0.805	No	95	Same batch	5	ACN				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD	GC-MS		
4	Flutriafol	2.13	No	93	Same batch	5	ACN				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD	GC-MS		
4	Isoproturon	0.217	No	106	Same batch	5	ACN				Yes	No	None	MM-ML	No	MS/MS	LC-MS/MS	Quechers	
4	Kresoxim-methyl	0.423	No	101	Same batch	5	ACN				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD	GC-MS		
4	Lambda-cyhalothrin	0.065	No	97	Same batch	5	ACN				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD	GC-MS		
4	Malathion	0.108	No	100	Same batch	5	ACN				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD	GC-MS		
4	Pirimiphos-methyl	0.097	No	98	Same batch	5	ACN				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD	GC-MS		
4	Spiroxamine	1.87	No	119	Same batch	5	ACN				Yes	No	None	MM-ML	No	MS/MS	LC-MS/MS	Quechers	
4	Triadimenol	1.53	No	92	Same batch	5	ACN				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD	GC-MS		
4	Glyphosate	3.20	Yes automatic		isotop. lab.	5	MeOH				Yes	No	None	PS-ML	Isotop. lab.1	MS/MS	LC-MS/MS	CRL SRM	
4	Mepiquat (free cation)	0.123	Yes automatic		isotop. lab.	10	MeOH				Yes	No	None	PS-ML	Isotop. lab.1	MS/MS	LC-MS/MS	EN15055	
5	Carbaryl	0.112	No	105.4	Same batch	20	AC	CH2Cl	PE		No	No	GPC	MM-ML	No	MSD	GC-MS	S19	
5	Carbendazim and benomyl	0.571	No	108.3	Same batch	20	AC	CH2Cl	PE		No	No	GPC	MM-ML	No	MS/MS	LC-MS/MS	S19	
5	Chlorpyrifos-methyl	0.111	No	105.3	Same batch	20	AC	CH2Cl	PE		No	No	GPC	MM-ML	No	MSD	GC-MS	S19	

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
5	Fenitrothion	0.181	No	95.1	Same batch	20	AC	CH2Cl	PE		No	No	GPC	MM-ML	No	MSD		GC-MS	S19
5	Fenpropimorph	1.766	No	91.1	Same batch	20	AC	CH2Cl	PE		No	No	GPC	MM-ML	No		MS/MS	LC-MS/MS	S19
5	Fluquinconazole	0.365	No	109.0	Same batch	20	AC	CH2Cl	PE		No	No	GPC	MM-ML	No	MSD		GC-MS	S19
5	Flutriafol	0.354	No	102.2	Same batch	20	AC	CH2Cl	PE		No	No	GPC	MM-ML	No		MS/MS	LC-MS/MS	S19
5	Isoproturon	0.101	No	109.0	Same batch	20	AC	CH2Cl	PE		No	No	GPC	MM-ML	No		MS/MS	LC-MS/MS	S19
5	Kresoxim-methyl	0.253	No	96.4	Same batch	20	AC	CH2Cl	PE		No	No	GPC	MM-ML	No	MSD		GC-MS	S19
5	Lambda-cyhalothrin	0.084	No	95.8	Same batch	20	AC	CH2Cl	PE		No	No	GPC	MM-ML	No	MSD		GC-MS	S19
5	Pirimiphos-methyl	0.081	No	105.8	Same batch	20	AC	CH2Cl	PE		No	No	GPC	MM-ML	No	MSD		GC-MS	S19
5	Spiroxamine	0.564	No	97.3	Same batch	20	AC	CH2Cl	PE		No	No	GPC	MM-ML	No	MSD		GC-MS	S19
5	Triadimenol	0.602	No	104.2	Same batch	20	AC	CH2Cl	PE		No	No	GPC	MM-ML	No	MSD		GC-MS	S19
6	Azoxystrobin	0.362	No	106	Same batch	5	MeOH	Water			Yes	No	None	MM-ML	Oxfendazole		MS/MS	LC-MS/MS	
6	Carbaryl	0.152	No	106	Same batch	5	MeOH	Water			Yes	No	None	MM-ML	Oxfendazole		MS/MS	LC-MS/MS	
6	Carbendazim and benomyl	1.68	No	108	Same batch	5	MeOH	Water			Yes	No	None	MM-ML	Oxfendazole		MS/MS	LC-MS/MS	
6	Chlorpyrifos-methyl	0.108	No	95	Same batch	10	EtOAc				Yes	No	DSPE PSA C18	MM-ML	Diethyl-ethyl	MS/MS		GC-MS/MS	
6	Deltamethrin (cis)	0.060	No	87	Same batch	10	EtOAc				Yes	No	DSPE PSA C18	MM-ML	Diethyl-ethyl	MS/MS		GC-MS/MS	
6	Fenitrothion	0.178	No	93	Same batch	10	EtOAc				Yes	No	DSPE PSA C18	MM-ML	Diethyl-ethyl	MS/MS		GC-MS/MS	
6	Fenpropimorph	1.75	No	128	Same batch	5	MeOH	Water			Yes	No	None	MM-ML	Oxfendazole		MS/MS	LC-MS/MS	
6	Fluquinconazole	0.658	No	89	Same batch	10	EtOAc				Yes	No	DSPE PSA C18	MM-ML	Diethyl-ethyl	MS/MS		GC-MS/MS	
6	Flutriafol	2.26	No	102	Same batch	5	MeOH	Water			Yes	No	None	MM-ML	Oxfendazole		MS/MS	LC-MS/MS	
6	Isoproturon	0.206	No	118	Same batch	5	MeOH	Water			Yes	No	None	MM-ML	Oxfendazole		MS/MS	LC-MS/MS	
6	Kresoxim-methyl	0.342	No	91	Same batch	10	EtOAc				Yes	No	DSPE PSA C18	MM-ML	Diethyl-ethyl	MS/MS		GC-MS/MS	
6	Lambda-cyhalothrin	0.095	No	84	Same batch	10	EtOAc				Yes	No	DSPE PSA C18	MM-ML	Diethyl-ethyl	MS/MS		GC-MS/MS	
6	Malathion	0.092	No	87	Same batch	10	EtOAc				Yes	No	DSPE PSA C18	MM-ML	Diethyl-ethyl	MS/MS		GC-MS/MS	
6	Pirimiphos-methyl	0.062	No	92	Same batch	10	EtOAc				Yes	No	DSPE PSA C18	MM-ML	Diethyl-ethyl	MS/MS		GC-MS/MS	
6	Spiroxamine	1.007	No	113	Same batch	5	MeOH	Water			Yes	No	None	MM-ML	Oxfendazole		MS/MS	LC-MS/MS	

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) EtOAc	Extraction solvent 2) MeOH	Extraction solvent 3) Water	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
6	Triadimenol	1.51	No Yes	91 automatic	Same batch	10					Yes	No	DSPE PSA C18	MM-ML	Diethyl-ethyl	MS/MS		GC-MS/MS	
6	Mepiquat (free cation)	0.054			St. Ad.	5					Yes	No	None	St. Ad.	No		MS/MS	LC-MS/MS	
7	Azoxystrobin	0.393	No	102	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD		LC-MS/MS	SOP
7	Carbaryl	0.185	No	99	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MS/MS	LC-MS/MS	SOP	
7	Carbendazim and benomyl	0.535	No	99	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MS/MS	LC-MS/MS	SOP	
7	Chlorpyrifos-methyl	0.146	No	85	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD		LC-MS/MS	SOP
7	Fenitrothion	0.063	No	102	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD		LC-MS/MS	SOP
7	Fenpropimorph	1.68	No	90	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD		LC-MS/MS	SOP
7	Fluquinconazole	1.66	No	92	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MS/MS	LC-MS/MS	SOP	
7	Flutriafol	2.99	No	109	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD		LC-MS/MS	SOP
7	Isoproturon	0.161	No	98	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MS/MS	LC-MS/MS	SOP	
7	Kresoxim-methyl	0.860	No	70	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MS/MS	LC-MS/MS	SOP	
7	Lambda-cyhalothrin	0.087	No	119	Same batch	25	AC	Cy-He	EtOAc		Yes	No	GPC	MM-ML	TPP, Mirex,	ECD		GC-MS	SOP
7	Malathion	0.074	No	74	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MS/MS	LC-MS/MS	SOP	
7	Pirimiphos-methyl	0.118	No	100	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MS/MS	LC-MS/MS	SOP	
7	Spiroxamine	0.812	No	80	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD		LC-MS/MS	SOP
7	-Triadimefon	0.031	No	114	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD		LC-MS/MS	SOP
7	Triadimenol	2.05	No	118	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD		LC-MS/MS	SOP
7	2,4-D (foll. alk hydrolysis)	0.478	No	107	Same batch	5	ACN				Yes	Yes	Freez.	MM-ML	Nicarbazin	MS/MS	LC-MS/MS	SOP	
7	Glyphosate	4.63	No	94	Same batch	5	MeOH				Yes	No	None	MM-ML	Isotop. lab.1	MS/MS	LC-MS/MS	SOP	
7	Mepiquat (free cation)	0.079	No	121	Same batch	5	MeOH				Yes	No	None	MM-ML	Isotop. lab.1	MS/MS	LC-MS/MS	SOP	
8	Azoxystrobin	0.363	No	108	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No	MS/MS	LC-MS/MS	ASU Â§64 LFGB L00.00-115	
8	Carbaryl	0.190	No	108	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No	MS/MS	LC-MS/MS	ASU Â§64 LFGB L00.00-115	
8	Carbendazim and benomyl	1.01	No	100	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No	MS/MS	LC-MS/MS	ASU Â§64 LFGB L00.00-115	
8	Chlorpyrifos-methyl	0.127	No	105	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No	MS/MS	LC-MS/MS	ASU Â§64 LFGB L00.00-115	

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
8	Deltamethrin (cis)	0.0800	No	88	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No		MS/MS	LC-MS/MS	ASU §64 LFGB L00.00-115
8	Fenitrothion	0.160	No	103	Same batch	25	AC	Cy-He	EtOAc		Yes	No	GPC	PS-ML	No	MSD	ECD	VDLUFA-Meth. 3.3.7.1. 2008	
8	Fenpropimorph	2.17	No	80	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No	MS/MS	LC-MS/MS	ASU §64 LFGB L00.00-115	
8	Fluquinconazole	0.825	No	108	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No	MS/MS	LC-MS/MS	ASU §64 LFGB L00.00-115	
8	Flutriafol	2.34	No	103	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No	MS/MS	LC-MS/MS	ASU §64 LFGB L00.00-115	
8	Isoproturon	0.176	No	99	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No	MS/MS	LC-MS/MS	ASU §64 LFGB L00.00-115	
8	Kresoxim-methyl	0.460	No	111	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No	MS/MS	LC-MS/MS	ASU §64 LFGB L00.00-115	
8	Lambda-cyhalothrin	0.0940	No	99	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No	MS/MS	LC-MS/MS	ASU §64 LFGB L00.00-115	
8	Malathion	0.127	No	111	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No	MS/MS	LC-MS/MS	ASU §64 LFGB L00.00-115	
8	Pirimiphos-methyl	0.103	No	105	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No	MS/MS	LC-MS/MS	ASU §64 LFGB L00.00-115	
8	Spiroxamine	0.881	No	100	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No	MS/MS	LC-MS/MS	ASU §64 LFGB L00.00-115	
8	Triadimenol	1.83	No	100	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No	MS/MS	LC-MS/MS	ASU §64 LFGB L00.00-115	
9	2,4-D (free acid)	0.356	No	101	Validation	5	ACN				Yes	No	Freez.	MM-SL	Nicarbazin	MS/MS	None	QuEChERS	
9	2,4-D (foll. alk hydrolysis)	0.329	No	88	Validation	5	ACN				Yes	Yes	Freez.	MM-SL	Nicarbazin	MS/MS	None	QuEChERS	
10	Azoxystrobin	0.382	No	96.5	Same batch	5	ACN				Yes	No	DSPE	MM-ML		MS/MS	None	ASU § 64 LFGB L 00.00-115	
10	Carbaryl	0.169	No	87.5	Same batch	5	ACN				Yes	No	DSPE	MM-ML		MS/MS	None	ASU § 64 LFGB L 00.00-115	
10	Carbendazim and benomyl	1.29	No	84	Same batch	5	ACN				Yes	No	DSPE	MM-ML		MS/MS	None	ASU § 64 LFGB L 00.00-115 ASU § 64 LFGB L 00.00-34 without silica gel cleanup	
10	Deltamethrin (cis)	0.098	Yes	65	Same batch	25	MeOH	CH ₂ Cl			Yes	No	GPC	MM-ML		ECD	GC-MS		
10	Fenitrothion	0.233	No	85.5	Same batch	5	ACN				Yes	No	DSPE	MM-ML		MS/MS	None	ASU § 64 LFGB L 00.00-115	
10	Fenpropimorph	1.94	No	89	Same batch	5	ACN				Yes	No	DSPE	MM-ML		MS/MS	None	ASU § 64 LFGB L 00.00-115	
10	Flutriafol	2.03	No	88.5	Same batch	5	ACN				Yes	No	DSPE	MM-ML		MS/MS	None	ASU § 64 LFGB L 00.00-115	
10	Isoproturon	0.171	No	88.5	Same batch	5	ACN				Yes	No	DSPE	MM-ML		MS/MS	None	ASU § 64 LFGB L 00.00-115	
10	Kresoxim-methyl	0.443	No	89	Same batch	5	ACN				Yes	No	DSPE	MM-ML		MS/MS	None	ASU § 64 LFGB L 00.00-115 ASU § 64 LFGB L 00.00-34 without silica gel cleanup	
10	Lambda-cyhalothrin	0.081	No	85	Same batch	25	MeOH	CH ₂ Cl			Yes	No	GPC	MM-ML		ECD	GC-MS		
10	Malathion	0.127	No	76	Same batch	5	ACN				Yes	No	DSPE	MM-ML		MS/MS	None	ASU § 64 LFGB L 00.00-115	

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) ACN	Extraction solvent 2) ACN	Extraction solvent 3) ACN	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method	
10	Pirimiphos-methyl	0.078	No	85.5	Same batch	5					Yes	No	DSPE	MM-ML		MS/MS	None	ASU Â§ 64 LFGB L 00.00-115		
10	Spiroxamine	1.06	No	87	Same batch	5	ACN				Yes	No	DSPE	MM-ML		MS/MS	None	ASU Â§ 64 LFGB L 00.00-115		
10	Triadimenol	1.61	No	89.5	Same batch	5	ACN				Yes	No	DSPE	MM-ML		MS/MS	None	ASU Â§ 64 LFGB L 00.00-115		
10	2,4-D (free acid)	0.332	No	77	Same batch	5	ACN				Yes	No	DSPE	MM-ML		MS/MS	None	ASU Â§ 64 LFGB L 00.00-115		
10	Mepiquat (free cation)	0.110	Yes	37	Same batch	5	ACN				Yes	No	DSPE	MM-ML		MS/MS	None	ASU Â§ 64 LFGB L 00.00-115		
11	Azoxystrobin	0.29	No	107	Same batch	25	ACN				Yes	No	None	PS-ML	Bromophos	MSD	GC-MS			
11	Carbaryl	0.19	No	103	Same batch	25	ACN				Yes	No	None	PS-ML	Bromophos	MSD	GC-MS			
11	Carbendazim and benomyl	0.526	No	85	Same batch	10	ACN				Yes	No	PS-ML			MS/MS	LC-MS/MS			
11	Chlorpyrifos-methyl	0.09	No	102	Same batch	25	ACN				Yes	No	None	PS-ML	Bromophos	MSD	GC-MS			
11	Fenitrothion	0.165	No	96	Same batch	25	ACN				Yes	No	None	PS-ML	Bromophos	MSD	GC-MS			
11	Fenpropimorph	2.38	No	94	Same batch	25	ACN				Yes	No	None	PS-ML	Bromophos	MSD	GC-MS			
11	Fluquinconazole	0.407	No	87	Same batch	25	ACN				Yes	No	None	PS-ML	Bromophos	MSD	GC-MS			
11	Flutriafol	1.45	No	99	Same batch	25	ACN				Yes	No	None	PS-ML	Bromophos	MSD	GC-MS			
11	Isoproturon	0.14	No	90	Same batch	10	ACN				Yes	No	PS-ML			MS/MS	LC-MS/MS			
11	Kresoxim-methyl	0.216	No	91	Same batch	25	ACN				Yes	No	None	PS-ML	Bromophos	MSD	GC-MS			
11	Lambda-cyhalothrin	0.047	No	85	Same batch	25	ACN				Yes	No	None	PS-ML	Bromophos	MSD	GC-MS			
11	Malathion	0.08	No	86	Same batch	25	ACN				Yes	No	None	PS-ML	Bromophos	MSD	GC-MS			
11	Pirimiphos-methyl	0.07	No	95	Same batch	25	ACN				Yes	No	None	PS-ML	Bromophos	MSD	GC-MS			
11	Spiroxamine	0.935	No	98	Same batch	10	ACN				Yes	No	PS-ML			MS/MS	LC-MS/MS			
11	Triadimenol	0.835	No	98	Same batch	25	ACN				Yes	No	None	PS-ML	Bromophos	MSD	GC-MS			
12	Azoxystrobin	0.270	No	104	Same batch	5	EtOAc				Yes	No	Other Filter	MM-SL	Isotop. lab.2	MS/MS				
12	Carbaryl	0.174	No	100	Same batch	5	EtOAc				Yes	No	Other Filter	MM-SL	Isotop. lab.2	MS/MS				
12	Carbendazim and benomyl	1.35	No	95	Same batch	5	EtOAc				Yes	No	Other Filter	MM-SL	Isotop. lab.2	MS/MS				
12	Chlorpyrifos-methyl	0.123	No	85	Same batch	5	EtOAc				Yes	No	Other Filter	MM-SL	Isotop. lab.2	MS/MS				
12	Deltamethrin (cis)	0.0699	No	91	Same batch	5	EtOAc				Yes	No	Other Filter	MM-SL	Isotop. lab.2	MS/MS				

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) EtOAc	Extraction solvent 2) EtOAc	Extraction solvent 3) EtOAc	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method	
12	Nenitrothion	0.194	No	79	Same batch	5					Yes	No	Other Filter	MM-SL	Isotop. lab.2	MS/MS				
12	Fenpropimorph	2.78	No	105	Same batch	5	EtOAc				Yes	No	Other Filter	MM-SL	Isotop. lab.2	MS/MS				
12	Fluquinconazole	0.720	No	107	Same batch	5	EtOAc				Yes	No	Other Filter	MM-SL	Isotop. lab.2	MS/MS				
12	Flutriafol	2.20	No	89	Same batch	5	EtOAc				Yes	No	Other Filter	MM-SL	Isotop. lab.2	MS/MS				
12	Isoproturon	0.142	No	102	Same batch	5	EtOAc				Yes	No	Other Filter	MM-SL	Isotop. lab.2	MS/MS				
12	Kresoxim-methyl	0.344	No	99	Same batch	5	EtOAc				Yes	No	Other Filter	MM-SL	Isotop. lab.2	MS/MS				
12	Lambda-cyhalothrin	0.0783	No	85	Same batch	5	EtOAc				Yes	No	Other Filter	MM-SL	Isotop. lab.2	MS/MS				
12	Malathion	0.0895	No	83	Same batch	5	EtOAc				Yes	No	Other Filter	MM-SL	Isotop. lab.2	MS/MS				
12	Pirimiphos-methyl	0.0750	No	87	Same batch	5	EtOAc				Yes	No	Other Filter	MM-SL	Isotop. lab.2	MS/MS				
12	Spiroxamine	1.04	No	88	Same batch	5	EtOAc				Yes	No	Other Filter	MM-SL	Isotop. lab.2	MS/MS				
12	Triadimenol	1.45	No	102	Same batch	5	EtOAc				Yes	No	Other Filter	MM-SL	Isotop. lab.2	MS/MS				
12	2,4-D (free acid)	0.336	No	80	Same batch	5	EtOAc				Yes	No	Other Filter	MM-SL	Isotop. lab.2	MS/MS				
12	2,4-D (foll. alk hydrolysis)	0.343	No	77	Same batch	5	EtOAc				Yes	Yes	Other Filter	MM-SL	No	MS/MS				
12	Mepiquat (free cation)	0.0938	No Yes automatic	84	Same batch	10	MeOH				Yes	No	Other Filter	PS-ML	Isotop. lab.1	MS/MS				
13	Carbaryl	0.191	78 Yes automatic	78	Same batch	20	AC	CH2Cl	HEX		No	No	L/I part.	MM-ML	TPP	NPD	GC-MS/MS			
13	Chlorpyrifos-methyl	0.114	91 Yes automatic	91	Same batch	20	AC	CH2Cl	HEX		No	No	L/I part.	MM-ML	TPP	NPD	GC-MS/MS			
13	Deltamethrin (cis)	0.027	80 Yes automatic	80	Same batch	20	AC	CH2Cl	HEX		No	No	L/I part.	MM-ML	Mirex	ECD	GC-MS/MS			
13	Nenitrothion	0.223	92 Yes automatic	92	Same batch	20	AC	CH2Cl	HEX		No	No	L/I part.	MM-ML	TPP	NPD	GC-MS/MS			
13	Malathion	0.041	94 Yes automatic	94	Same batch	20	AC	CH2Cl	HEX		No	No	L/I part.	MM-ML	TPP	NPD	GC-MS/MS			
13	Pirimiphos-methyl	0.086	89 Yes automatic	89	Same batch	20	AC	CH2Cl	HEX		No	No	L/I part.	MM-ML	TPP	NPD	GC-MS/MS			
14	Azoxystrobin	0.350	No	94.7	Same batch	5	ACN				Yes	No	Freez.	MM-ML	No	ECD	GC-MS	EN15662:2008		
14	Carbaryl	0.110	No	102	Same batch	5	ACN				Yes	No	Freez.	MM-ML	No	ECD	GC-MS	EN15662:2008		
14	Carbendazim and benomyl	0.864	No	90	Same batch	5	ACN				Yes	No	Freez.	MM-ML	No	MS/MS	LC-MS/MS	EN15662:2008		
14	Chlorpyrifos-methyl	0.125	No	88.5	Same batch	5	ACN				Yes	No	Freez.	MM-ML	No	ECD	GC-MS	EN15662:2008		
14	Deltamethrin (cis)	0.065	No	85.8	Same batch	5	ACN				Yes	No	Freez.	MM-ML	No	ECD	GC-MS	EN15662:2008		

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) ACN	Extraction solvent 2) ACN	Extraction solvent 3) ACN	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method	
14	Fenitrothion	0.198	No	92.5	Same batch	5					Yes	No	Freez.	MM-ML	No	ECD	GC-MS	EN15662:2008		
14	Fenpropimorph	2.57	No	89.7	Same batch	5	ACN				Yes	No	Freez.	MM-ML	No	ECD	GC-MS	EN15662:2008		
14	Fluquinconazole	0.814	No	94.3	Same batch	5	ACN				Yes	No	Freez.	MM-ML	No	ECD	GC-MS	EN15662:2008		
14	Flutriafol	2.07	No	92.1	Same batch	5	ACN				Yes	No	Freez.	MM-ML	No	ECD	GC-MS	EN15662:2008		
14	Isoproturon	0.110	No	82	Same batch	5	ACN				Yes	No	Freez.	MM-ML	No		MS/MS	LC-MS/MS	EN15662:2008	
14	Kresoxim-methyl	0.414	No	92.2	Same batch	5	ACN				Yes	No	Freez.	MM-ML	No	ECD	GC-MS	EN15662:2008		
14	Lambda-cyhalothrin	0.080	No	84.8	Same batch	5	ACN				Yes	No	Freez.	MM-ML	No	ECD	GC-MS	EN15662:2008		
14	Malathion	0.110	No	90.9	Same batch	5	ACN				Yes	No	Freez.	MM-ML	No	ECD	GC-MS	EN15662:2008		
14	Pirimiphos-methyl	0.071	No	90.8	Same batch	5	ACN				Yes	No	Freez.	MM-ML	No	ECD	GC-MS	EN15662:2008		
14	Spiroxamine	0.928	No	80	Same batch	5	ACN				Yes	No	Freez.	MM-ML	No		MS/MS	LC-MS/MS	EN15662:2008	
14	Triadimenol	1.65	No	95.4	Same batch	5	ACN				Yes	No	Freez.	MM-ML	No	ECD	GC-MS	EN15662:2008		
14	2,4-D (free acid)	0.358	No	78	Same batch	5	ACN				Yes	No	Freez.	MM-ML	No		MS/MS	LC-MS/MS	EN15662:2008	
14	2,4-D (foll. alk hydrolysis)	0.356	No	78	Same batch	5	ACN				Yes	Yes	Freez.	MM-ML	No		MS/MS	LC-MS/MS	15662.2008	
14	Glyphosate	2.933	No	80.3	Same batch	3	Water				No	No	None	PS-ML	No		MS	LC-MS	JFood Add and Cont.,Vol 20, No.8, 2003 CRL-SRM, Analysis of Chlormequat and mepiquat residues in Food of Plant origin, ver.2,2009	
14	Mepiquat (free cation)	0.073	No	97.2	Same batch	5	MeOH				Yes	No	None	PS-ML	No		MS	LC-MS		
15	Azoxystrobin	0.309	Yes	111	Same batch	2	ACN	Water			Yes	No	Other	PS-SL	No		MS/MS	LC-MS/MS		
15	Carbaryl	0.151	Yes	112	Same batch	2	ACN	Water			Yes	No	None	MM-SL	No		MS/MS	LC-MS/MS		
15	Carbendazim and benomyl	1.33	Yes	97	Same batch	2	ACN	Water			Yes	No	Other	PS-SL	No		MS/MS	LC-MS/MS		
15	Chlorpyrifos-methyl	0.152	Yes	95	Same batch	2	ACN	Water			Yes	No	None	MM-SL	No		MS/MS	LC-MS/MS		
15	Deltamethrin (cis)	0.0752	Yes	100	Same batch	2	ACN	Water			Yes	No	None	MM-SL	No		MS/MS	LC-MS/MS		
15	Fenitrothion	0.150	Yes	103	Same batch	2	ACN	Water			Yes	No	DSPE PSA C18	MM-SL	No		MS/MS	GC-MS/MS		
15	Fenpropimorph	1.88	Yes	109	Same batch	2	ACN	Water			Yes	No	Other	PS-SL	No		MS/MS	LC-MS/MS		
15	Fluquinconazole	0.885	Yes	98	Same batch	2	ACN	Water			Yes	No	Other	PS-SL	No		MS/MS	LC-MS/MS		
15	Flutriafol	2.25	Yes	104	Same batch	2	ACN	Water			Yes	No	Other	PS-SL	No		MS/MS	LC-MS/MS		
15	Isoproturon	0.127	Yes	110	Same batch	2	ACN	Water			Yes	No	None	MM-SL	No		MS/MS	LC-MS/MS		

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
15	Kresoxim-methyl	0.371	Yes	121	Same batch	2	ACN	Water			Yes	No	Other	PS-SL	No		MS/MS	LC-MS/MS	
15	Lambda-cyhalothrin	0.117	Yes	87	Same batch	2	ACN	Water			Yes	No	DSPE PSA C18	MM-SL	No	MS/MS	GC-MS/MS		
15	Malathion	0.104	Yes	112	Same batch	2	ACN	Water			Yes	No	None	MM-SL	No	MS/MS	LC-MS/MS		
15	Pirimiphos-methyl	0.0829	Yes	106	Same batch	2	ACN	Water			Yes	No	None	MM-SL	No	MS/MS	LC-MS/MS		
15	Spiroxamine	0.968	Yes	109	Same batch	2	ACN	Water			Yes	No	Other	PS-SL	No	MS/MS	LC-MS/MS		
15	-Triadimefon	0.0103	Yes	109	Same batch	2	ACN	Water			Yes	No	None	MM-SL	No	MS/MS	LC-MS/MS		
15	Triadimenol	1.78	Yes	105	Same batch	2	ACN	Water			Yes	No	Other	PS-SL	No	MS/MS	LC-MS/MS		
15	2,4-D (free acid)	0.373	Yes	97	Same batch	2	ACN	Water			Yes	No	Other	PS-SL	No	MS/MS	LC-MS/MS		
16	Azoxystrobin	0.467	No	-		5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS	None	QuEChERS	
16	Carbaryl	0.0920	No	-		5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS	None	QuEChERS	
16	Carbendazim and benomyl	0.993	No	-		5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS	None	QuEChERS	
16	Chlorpyrifos-methyl	0.170	No	90	Validation	5	ACN				Yes	No	DSPE	MM-ML		Other		None	QuEChERS
16	Deltamethrin (cis)	0.175	No	76	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	ECD		None	QuEChERS
16	Fenitrothion	0.287	No	90	Validation	5	ACN				Yes	No	DSPE	MM-ML		Other		None	QuEChERS
16	Fenpropimorph	1.89	No	-		5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS	None	QuEChERS	
16	Fluquinconazole	0.823	No	89	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	ECD		None	QuEChERS
16	Flutriafol	2.43	No	101	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS		None	QuEChERS
16	Isoproturon	0.159	No	-		5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS	None	QuEChERS	
16	Kresoxim-methyl	0.486	No	93	Validation	5	ACN				Yes	No	DSPE	MM-ML	No	MSD		None	QuEChERS
16	Lambda-cyhalothrin	0.115	No	72	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	ECD		None	QuEChERS
16	Malathion	0.162	No	92	Validation	5	ACN				Yes	No	DSPE	MM-ML		Other		None	QuEChERS
16	Pirimiphos-methyl	0.114	No	93	Validation	5	ACN				Yes	No	DSPE	MM-ML		Other		None	QuEChERS
16	Spiroxamine	3.13	No	93	Validation	5	ACN				Yes	No	DSPE	MM-ML	No	MSD		None	QuEChERS
16	-Triadimefon	0.0166	No	98	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS		None	QuEChERS
16	Triadimenol	1.84	No	120	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS		None	QuEChERS

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) EtOAc	Extraction solvent 2) EtOAc	Extraction solvent 3) ACN	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
17	Azoxystrobin	0.272	No	86.9	Same batch	10	Cy-He	EtOAc		+	No	No	GPC SPE	MM-ML	No	MSD	GC-MS/MS	VDLUFA MB VII 3.3.7.1	
17	Carbaryl	0.165	No	83.2	Same batch	10	Cy-He	EtOAc		+	No	No	GPC SPE	MM-ML	No	MSD	GC-MS/MS	VDLUFA MB VII 3.3.7.1	
17	Carbendazim and benomyl	1.267	No	86	Same batch	5	ACN				Yes	No	Freez. DSPE	MM-ML	TPP	MS/MS	LC-MS/MS	Quechers	
17	Chlorpyrifos-methyl	0.13	No	85	Same batch	10	Cy-He	EtOAc		+	No	No	GPC SPE	MM-ML	No	MSD	GC-MS/MS	VDLUFA MB VII 3.3.7.1	
17	Deltamethrin (cis)	0.062	No	84.2	Same batch	10	Cy-He	EtOAc		+	No	No	GPC SPE	MM-ML	No	MSD	GC-MS/MS	VDLUFA MB VII 3.3.7.1	
17	Fenpropimorph	2.69	No	98.8	Same batch	5	ACN				Yes	No	Freez. DSPE	MM-ML	TPP	MS/MS	LC-MS/MS	Quechers	
17	Isoproturon	0.176	No	100	Same batch	5	ACN				Yes	No	Freez. DSPE	MM-ML	TPP	MS/MS	LC-MS/MS	Quechers	
17	Kresoxim-methyl	0.434	No	94.2	Same batch	10	Cy-He	EtOAc		+	No	No	GPC SPE	MM-ML	No	MSD	GC-MS/MS	VDLUFA MB VII 3.3.7.1	
17	Lambda-cyhalothrin	0.063	No	84	Same batch	10	Cy-He	EtOAc		+	No	No	GPC SPE	MM-ML	No	MSD	GC-MS/MS	VDLUFA MB VII 3.3.7.1	
17	Malathion	0.104	No	83.4	Same batch	10	Cy-He	EtOAc		+	No	No	GPC SPE	MM-ML	No	MSD	GC-MS/MS	VDLUFA MB VII 3.3.7.1	
17	Pirimiphos-methyl	0.083	No	83	Same batch	10	Cy-He	EtOAc		+	No	No	GPC SPE	MM-ML	No	MSD	GC-MS/MS	VDLUFA MB VII 3.3.7.1	
17	Spiroxamine	1.186	No	98	Same batch	5	ACN				Yes	No	Freez. DSPE	MM-ML	TPP	MS/MS	LC-MS/MS	Quechers	
17	-Triadimefon	0.01	No	96.4	Same batch	10	Cy-He	EtOAc		+	No	No	GPC SPE	MM-ML	No	MSD	GC-MS/MS	VDLUFA MB VII 3.3.7.1	
17	Triadimenol	1.752	No	82.7	Same batch	10	Cy-He	EtOAc		+	No	No	GPC SPE	MM-ML	No	MSD	GC-MS/MS	VDLUFA MB VII 3.3.7.1	
18	Azoxystrobin	0.277	98.2	Same batch	5	ACN					Yes		DSPE	MM-ML	TPP, PCT,	MSD	LC-MS/MS		
18	Carbaryl	0.167	112.7	Same batch	5	ACN					Yes		DSPE	MM-ML	TPP, D5-	MS/MS			
18	Carbendazim and benomyl	1.246	109.3	Same batch	5	ACN					Yes		DSPE	MM-ML	TPP, D5-	MS/MS			
18	Chlorpyrifos-methyl	0.115	94.7	Same batch	5	ACN					Yes		DSPE	MM-ML	TPP, PCT,	MSD			
18	Fenitrothion	0.190	109.2	Same batch	5	ACN					Yes		DSPE	MM-ML	TPP, PCT,	MSD			
18	Fenpropimorph	1.787	94.6	Same batch	5	ACN					Yes		DSPE	MM-ML	TPP, PCT,	MSD	LC-MS/MS		
18	Fluquinconazole	0.63	97.7	Same batch	5	ACN					Yes		DSPE	MM-ML	TPP, PCT,	MSD			
18	Flutriafol	2.745	97.5	Same batch	5	ACN					Yes		DSPE	MM-ML	TPP, D5-	MS/MS			
18	Isoproturon	0.166	111.6	Same batch	5	ACN					Yes		DSPE	MM-ML	TPP, D5-	MS/MS			
18	Kresoxim-methyl	0.383	95.6	Same batch	5	ACN					Yes		DSPE	MM-ML	TPP, D5-	MS/MS			
18	Lambda-cyhalothrin	0.080	103.7	Same batch	5	ACN					Yes		DSPE	MM-ML	TPP, PCT,	MSD			

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
18	Malathion	0.09		95.2	Same batch	5	ACN				Yes		DSPE	MM-ML	TPP, PCT,	MSD		LC-MS/MS	
18	Pirimiphos-methyl	0.074		94.2	Same batch	5	ACN				Yes		DSPE	MM-ML	TPP, PCT,	MSD		LC-MS/MS	
18	Spiroxamine	1.255		106.9	Same batch	5	ACN				Yes		DSPE	MM-ML	TPP, D5-		MS/MS		
18	Triadimenol	1.458		103.5	Same batch	5	ACN				Yes		DSPE	MM-ML	TPP, PCT,	MSD		LC-MS/MS	
19	Azoxystrobin	0.354	No	116.9	Same batch	5	ACN				Yes	No	Freez.	PS-ML	No	MS/MS	LC-MS/MS	Quechers	
19	Carbaryl	0.193	No	112.5	Same batch	5	ACN				Yes	No	Freez.	PS-ML	No	MS/MS	LC-MS/MS	Quechers	
19	Carbendazim and benomyl	1.10	No	91.9	Same batch	5	ACN				Yes	No	Freez.	PS-ML	No	MS/MS	LC-MS/MS	Quechers	
19	Chlorpyrifos-methyl	0.167	No	140.2	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	TPP	MSD		GC-MS	Quechers
19	Deltamethrin (cis)	0.061	No	150.4	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	TPP	MSD		GC-MS	Quechers
19	Fenitrothion	0.261	No	133.2	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	TPP	MSD		GC-MS	Quechers
19	Fenpropimorph	2.48	No	116.0	Same batch	5	ACN				Yes	No	Freez.	PS-ML	No	MS/MS	LC-MS/MS	Quechers	
19	Fluquinconazole	0.968	No	107.6	Same batch	5	ACN				Yes	No	Freez.	PS-ML	No	MS/MS	LC-MS/MS	Quechers	
19	Flutriafol	1.18	No	145.0	Same batch	5	ACN				Yes	No	Freez.	PS-ML	No	MS/MS	LC-MS/MS	Quechers	
19	Isoproturon	0.172	No	107.8	Same batch	5	ACN				Yes	No	Freez.	PS-ML	No	MS/MS	LC-MS/MS	Quechers	
19	Kresoxim-methyl	0.488	No	113.6	Same batch	5	ACN				Yes	No	Freez.	PS-ML	No	MS/MS	LC-MS/MS	Quechers	
19	Lambda-cyhalothrin	0.096	No	138.2	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	TPP	MSD		GC-MS	Quechers
19	Malathion	0.122	No	111.0	Same batch	5	ACN				Yes	No	Freez.	PS-ML	No	MS/MS	LC-MS/MS	Quechers	
19	Pirimiphos-methyl	0.099	No	163.8	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	TPP	MSD		GC-MS	Quechers
19	Spiroxamine	1.28	No	116.8	Same batch	5	ACN				Yes	No	Freez.	PS-ML	No	MS/MS	LC-MS/MS	Quechers	
19	-Triadimefon	0.011	No	107.4	Same batch	5	ACN				Yes	No	Freez.	PS-ML	No	MS/MS	LC-MS/MS	Quechers	
19	Triadimenol	2.38	No	116.2	Same batch	5	ACN				Yes	No	Freez.	PS-ML	No	MS/MS	LC-MS/MS	Quechers	
19	2,4-D (free acid)	0.478	No	104.0	Same batch	10	EOH				No	No	L/I part.	PS-ML	Fenoprop	MSD		GC-MS	Internal method
19	2,4-D (foll. alk hydrolysis)	0.462	No	100.0	Same batch	10	EOH				Yes	Yes	L/I part.	PS-ML	Fenoprop	MSD		GC-MS	Internal method
19	Mepiquat (free cation)	0.065	No	79.0	Same batch	20	MeOH				Yes	No	None	PS-ML	No	MS	LC-MS	EN15054	
20	Azoxystrobin	0.360	Yes automatic	100	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS	LC-MS/MS	QuEChERS	

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) %	Extraction solvent 2) %	Extraction solvent 3) %	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
20	Carbaryl	0.300	Yes automatic	100	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS	LC-MS/MS	QuEChERS	
20	Carbendazim and benomyl	0.856	Yes automatic	90	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS	LC-MS/MS	QuEChERS	
20	Chlorpyrifos-methyl	0.141	Yes automatic	85	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS	LC-MS/MS	QuEChERS	
20	Deltamethrin (cis)	0.100	Yes automatic	90	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS	LC-MS/MS	QuEChERS	
20	Fenpropimorph	2.21	Yes automatic	90	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS	LC-MS/MS	QuEChERS	
20	Fluquinconazole	0.985	Yes automatic	100	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS	LC-MS/MS	QuEChERS	
20	Flutriafol	2.41	Yes automatic	100	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS	LC-MS/MS	QuEChERS	
20	Isoproturon	0.190	Yes automatic	100	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS	LC-MS/MS	QuEChERS	
20	Kresoxim-methyl	0.453	Yes automatic	95	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS	LC-MS/MS	QuEChERS	
20	Lambda-cyhalothrin	0.100	Yes automatic	100	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS	LC-MS/MS	QuEChERS	
20	Malathion	0.1115	Yes automatic	100	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS	LC-MS/MS	QuEChERS	
20	Pirimiphos-methyl	0.084	Yes automatic	95	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS	LC-MS/MS	QuEChERS	
20	Spiroxamine	1.21	Yes automatic	100	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS	LC-MS/MS	QuEChERS	
20	Triadimenol	1.95	Yes automatic	90	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS	LC-MS/MS	QuEChERS	
20	2,4-D (free acid)	0.431	Yes automatic	93	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MS/MS	LC-MS/MS	QuEChERS	
20	2,4-D (foll. alk hydrolysis)	0.451	Yes automatic	93	Same batch	5	ACN				Yes	Yes	DSPE	MM-ML	No	MS/MS	LC-MS/MS	QuEChERS	
20	Glyphosate	4.10	Yes automatic	100	Same batch	5	Water				Yes	No	None	PS-SL	Isotop. lab.1	MS/MS	LC-MS/MS	In House	
20	Mepiquat (free cation)	0.098	Yes automatic	100	Same batch	5	MeOH	Water			Yes	No	None	PS-ML	Isotop. lab.1	MS/MS	LC-MS/MS	EN15054	
21	Azoxystrobin	0.0847	No	93	Same batch	50	EtOAc				No	No	GPC	PS-ML	No	ECD	Two columns EN12393-method P		
21	Chlorpyrifos-methyl	0.0798	No	105	Same batch	50	EtOAc				No	No	GPC	PS-ML	No	NPD	Two columns EN12393-method P		
21	Deltamethrin (cis)	0.0614	No	101	Same batch	50	EtOAc				No	No	GPC	PS-ML	No	ECD	Two columns EN12393-method P		
21	Fenitrothion	0.104	No	106	Same batch	50	EtOAc				No	No	GPC	PS-ML	No	NPD	Two columns EN12393-method P		
21	Malathion	0.0359	No	111	Same batch	50	EtOAc				No	No	GPC	PS-ML	No	NPD	Two columns EN12393-method P		
21	Pirimiphos-methyl	0.0344	No	102	Same batch	50	EtOAc				No	No	GPC	PS-ML	No	NPD	Two columns EN12393-method P		
22	Azoxystrobin	0.327	No	94	Same batch	5	ACN				Yes	No	Freez. DSPE	PS-ML	No	ECD	GC-MS		

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
22	Carbaryl	0.159	No	81	Same batch	5	ACN				Yes	No	Freez. DSPE	PS-ML	No		UV	GC-MS	
22	Carbendazim and benomyl	1.02	No	95	Same batch	5	ACN				Yes	No	Freez. DSPE	PS-ML	No		UV	Diode Array Detector	
22	Chlorpyrifos-methyl	0.131	No	98	Same batch	5	ACN				Yes	No	Freez. DSPE	PS-ML	No	FPD		GC-MS	
22	Fenitrothion	0.204	No	101	Same batch	5	ACN				Yes	No	Freez. DSPE	PS-ML	No	FPD		GC-MS	
22	Fenpropimorph	2.42	No	118	Same batch	5	ACN				Yes	No	Freez. DSPE	PS-ML	No	NPD		GC-MS	
22	Fluquinconazole	0.752	No	78	Same batch	5	ACN				Yes	No	Freez. DSPE	PS-ML	No	NPD		GC-MS	
22	Flutriafol	1.96	No	92	Same batch	5	ACN				Yes	No	Freez. DSPE	PS-ML	No	NPD		GC-MS	
22	Isoproturon	0.174	No	98	Same batch	5	ACN				Yes	No	Freez. DSPE	PS-ML	No	NPD		GC-MS	
22	Kresoxim-methyl	0.469	No	107	Same batch	5	ACN				Yes	No	Freez. DSPE	PS-ML	No	NPD		GC-MS	
22	Lambda-cyhalothrin	0.061	No	102	Same batch	5	ACN				Yes	No	Freez. DSPE	PS-ML	No	ECD		GC-MS	
22	Malathion	0.102	No	94	Same batch	5	ACN				Yes	No	Freez. DSPE	PS-ML	No	FPD		GC-MS	
22	Pirimiphos-methyl	0.092	No	89	Same batch	5	ACN				Yes	No	Freez. DSPE	PS-ML	No	FPD		GC-MS	
22	Spiroxamine	1.50	No	112	Same batch	5	ACN				Yes	No	Freez. DSPE	PS-ML	No	NPD		GC-MS	
22	Triadimenol	2.13	No	112	Same batch	5	ACN				Yes	No	Freez. DSPE	PS-ML	No	NPD		GC-MS	
23	Azoxystrobin	0.316	No	105.3	Same batch	10	AC				Yes		L/I part. SPE	MM-ML	No	ECD		GC-MS/MS EN 12393	
23	Carbaryl	0.174	No	77.1	Same batch	5	ACN				Yes		DSPE	MM-ML	Isotop. lab.2 TPP	MS/MS		GC-MS/MS Walorczyk, 2008	
23	Carbendazim and benomyl	1.88	No	106.4	Same batch	5	ACN				Yes		DSPE	MM-ML	Isotop. lab.2		MS/MS	LC-MS/MS Walorczyk 2008	
23	Chlorpyrifos-methyl	0.125	No	102.1	Same batch	10	AC				Yes		L/I part. SPE	MM-ML	No	ECD		GC-MS/MS EN 12393	
23	Fenitrothion	0.184	No	95.2	Same batch	10	AC				Yes		L/I part. SPE	MM-ML	No	ECD		GC-MS/MS EN 12393	
23	Fenpropimorph	2.37	No	77.9	Same batch	5	ACN				Yes		DSPE	MM-ML	Isotop. lab.2 TPP	MS/MS		GC-MS/MS Walorczyk, 2008	
23	Fluquinconazole	0.739	No	94.1	Same batch	5	ACN				Yes		DSPE	MM-ML	Isotop. lab.2 TPP	MS/MS		GC-MS/MS Walorczyk, 2008	
23	Flutriafol	2.17	No	97.2	Same batch	5	ACN				Yes		DSPE	MM-ML	Isotop. lab.2 TPP	MS/MS		GC-MS/MS Walorczyk, 2008	
23	Isoproturon	0.175	No	106.9	Same batch	5	ACN				Yes		DSPE	MM-ML	Isotop. lab.2		MS/MS	LC-MS/MS Walorczyk 2008	
23	Kresoxim-methyl	0.379	No	94.9	Same batch	10	AC				Yes		L/I part. SPE	MM-ML	No	ECD		GC-MS/MS EN 12393	
23	Malathion	0.109	No	95.0	Same batch	10	AC				Yes		L/I part. SPE	MM-ML	No	ECD		GC-MS/MS EN 12393	

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) ACN	Extraction solvent 2) ACN	Extraction solvent 3) ACN	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
23	Pirimiphos-methyl	0.077	No	82.8	Same batch	5					Yes		DSPE	MM-ML	Isotop. lab.2 TPP	MS/MS	GC-MS/MS	Walorczyk, 2008	
23	Spiroxamine	1.12	No	91.7	Same batch	5	ACN				Yes		DSPE	MM-ML	Isotop. lab.2	MS/MS	LC-MS/MS	Walorczyk 2008	
23	Triadimenol	1.63	No	76.6	Same batch	5	ACN				Yes		DSPE	MM-ML	Isotop. lab.2 TPP	MS/MS	GC-MS/MS	Walorczyk, 2008	
24	Azoxystrobin	0.3	No	92	Same batch	10	ACN				Yes		Freez. DSPE	PS-ML		MS/MS	GC-MS	NF EN 15662	
24	Carbaryl	0.236	No	98	Same batch	10	ACN				Yes		Freez. DSPE	PS-ML		MS/MS	GC-MS	NF EN 15662	
24	Carbendazim and benomyl	1.3	No	88	Same batch	10	ACN				Yes		Freez. DSPE	PS-ML		MS/MS		NF EN 15662	
24	Chlorpyrifos-methyl	0.108	No	88.9	Same batch	10	ACN				Yes		Freez. DSPE	PS-ML	bromophos	FPD	GC-MS	NF EN 15662	
24	Fenitrothion	0.184	No	82	Same batch	10	ACN				Yes		Freez. DSPE	PS-ML	bromophos	FPD	GC-MS	NF EN 15662	
24	Fenpropimorph	2.1	No	97	Same batch	10	ACN				Yes		Freez. DSPE	PS-ML	bromophos	ITD	GC-MS	NF EN 15662	
24	Fluquinconazole	0.563	No	90	Same batch	10	ACN				Yes		Freez. DSPE	PS-ML	bromophos	ITD	GC-MS	NF EN 15662	
24	Flutriafol	1.76	No	95	Same batch	10	ACN				Yes		Freez. DSPE	PS-ML	bromophos	ITD	GC-MS	NF EN 15662	
24	Isoproturon	0.160	No	100	Same batch	10	ACN				Yes		Freez. DSPE	PS-ML		MS/MS		NF EN 15662	
24	Kresoxim-methyl	0.365	No	89	Same batch	10	ACN				Yes		Freez. DSPE	PS-ML	bromophos	ITD	GC-MS	NF EN 15662	
24	Lambda-cyhalothrin	0.056	No	75	Same batch	10	ACN				Yes		Freez. DSPE	PS-ML	bromophos	ITD	GC-MS	NF EN 15662	
24	Malathion	0.075	No	96	Same batch	10	ACN				Yes		Freez. DSPE	PS-ML	bromophos	FPD	GC-MS	NF EN 15662	
24	Pirimiphos-methyl	0.070	No	99	Same batch	10	ACN				Yes		Freez. DSPE	PS-ML	bromophos	FPD	GC-MS	NF EN 15662	
24	Spiroxamine	0.883	No	93	Same batch	10	ACN				Yes		Freez. DSPE	PS-ML		MS/MS	GC-MS	NF EN 15662	
24	Triadimenol	1.45	No	86	Same batch	10	ACN				Yes		Freez. DSPE	PS-ML	bromophos	ITD	GC-MS	NF EN 15662	
24	2,4-D (free acid)	0.328	No	82	Same batch	10	ACN				Yes		Freez. DSPE	PS-ML		MS/MS		NF EN 15662	
24	Mepiquat (free cation)	0.055	No	78		10	MeOH	Water	+					PS-ML		ITQ		NF EN 15054	
25	Azoxystrobin	0.325	No	108	Same batch	10	AC	Cy-He			Yes	No	GPC	MM-SL	No	MSD	GC-MS	Â§ 64 LFGB L 00.00-34	
25	Carbaryl	0.168	No	95.7	Same batch	10	AC	Cy-He			Yes	No	GPC	MM-SL	No	MSD	GC-MS	Â§ 64 LFGB L 00.00-34	
25	Carbendazim and benomyl	1.376	No	81.8	Same batch	10	ACN				Yes	No	SPE	MM-SL	No	MS/MS	LC-MS/MS	Â§ 64 LFGB L 00.00-115	
25	Chlorpyrifos-methyl	0.118	No	112	Same batch	10	AC	Cy-He			Yes	No	GPC	MM-SL	No	MSD	GC-MS	Â§ 64 LFGB L 00.00-34	
25	Fenitrothion	0.114	No	79	Same batch	10	AC	Cy-He			Yes	No	GPC	MM-SL	No	MSD	GC-MS	Â§ 64 LFGB L 00.00-34	

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
25	Fenpropimorph	2.947	No	96	Same batch	10	AC	Cy-He			Yes	No	GPC	MM-SL	No	MSD	GC-MS	Å§ 64 LFGB L 00.00-34	
25	Fluquinconazole	0.745	No	90	Same batch	10	AC	Cy-He			Yes	No	GPC	MM-SL	No	MSD	GC-MS	Å§ 64 LFGB L 00.00-34	
25	Flutriafol	2.375	No	103	Same batch	10	AC	Cy-He			Yes	No	GPC	MM-SL	No	MSD	GC-MS	Å§ 64 LFGB L 00.00-34	
25	Isoproturon	0.175	No	94.9	Same batch	10	ACN				Yes	No	SPE	MM-SL	No	MS/MS	LC-MS/MS	Å§ 64 LFGB L 00.00-115	
25	Kresoxim-methyl	0.411	No	103	Same batch	10	AC	Cy-He			Yes	No	GPC	MM-SL	No	MSD	GC-MS	Å§ 64 LFGB L 00.00-34	
25	Lambda-cyhalothrin	0.041	Yes	64	Same batch	10	AC	Cy-He			Yes	No	GPC	MM-SL	No	MSD	GC-MS	Å§ 64 LFGB L 00.00-34	
25	Malathion	0.065	No	83	Same batch	10	AC	Cy-He			Yes	No	GPC	MM-SL	No	MSD	GC-MS	Å§ 64 LFGB L 00.00-34	
25	Pirimiphos-methyl	0.066	No	97	Same batch	10	AC	Cy-He			Yes	No	GPC	MM-SL	No	MSD	GC-MS	Å§ 64 LFGB L 00.00-34	
25	Spiroxamine	1.525	No	94	Same batch	10	AC	Cy-He			Yes	No	GPC	MM-SL	No	MSD	GC-MS	Å§ 64 LFGB L 00.00-34	
25	Triadimenol	1.802	No	101	Same batch	10	AC	Cy-He			Yes	No	GPC	MM-SL	No	MSD	GC-MS	Å§ 64 LFGB L 00.00-34	
25	2,4-D (free acid)	0.449	No	97.8	Same batch	10	ACN				Yes	No	SPE	MM-SL	No	MS/MS	LC-MS/MS	Å§ 64 LFGB L 00.00-115	
25	Mepiquat (free cation)	0.085	No	91.1		0									Isotop. lab.1			Å§ 64 LFGB L 00.00-76	
26	Azoxystrobin	0.237	Yes	75	Same batch	10	AC				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD		None	
26	Carbaryl	0.078	Yes	70	Same batch	10	AC				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD		None	
26	Carbendazim and benomyl	1.33	No	100	Same batch	10	Water	MeOH			Yes	No	SPE	MM-ML		MS/MS		None	
26	Chlorpyrifos-methyl	0.136	No	97	Same batch	10	AC				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD		None	
26	Deltamethrin (cis)	0.044	Yes	70	Same batch	10	AC				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD		None	
26	Fenitrothion	0.213	No	110	Same batch	10	AC				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD		None	
26	Fenpropimorph	2.88	No	103	Same batch	10	AC				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD		None	
26	Fluquinconazole	0.811	No	98	Same batch	10	AC				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD		None	
26	Flutriafol	1.92	No	84	Same batch	10	Water	MeOH			Yes	No	SPE	MM-ML		MS/MS		None	
26	Isoproturon	0.147	No	89	Same batch	10	Water	MeOH			Yes	No	SPE	MM-ML		MS/MS		None	
26	Kresoxim-methyl	0.412	No	103	Same batch	10	AC				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD		None	
26	Lambda-cyhalothrin	0.061	No	93	Same batch	10	AC				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD		None	
26	Malathion	0.117	No	90	Same batch	10	AC				Yes	No	SPE	MM-ML	Isotop. lab.2	MSD		None	

LAB CODE	Pesticide	Reported result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) ACN	Extraction solvent 2) ACN	Extraction solvent 3) CH2Cl	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
26 - Malaoxon	0.005	Yes	45	Same batch	10	AC					Yes	No	SPE	MM-ML	Isotop. lab.2	MSD		None	
26 Pirimiphos-methyl	0.083	No	104	Same batch	10	AC					Yes	No	SPE	MM-ML	Isotop. lab.2	MSD		None	
26 Spiroxamine	1.18	No	98	Same batch	10	AC					Yes	No	SPE	MM-ML	Isotop. lab.2	MSD		None	
26 -Triadimefon	0.011	No	85	Same batch	10	AC					Yes	No	SPE	MM-ML	Isotop. lab.2	MSD		None	
26 Triadimenol	1.79	No	105	Same batch	10	AC					Yes	No	SPE	MM-ML	Isotop. lab.2	MSD		None	
26 Mepiquat (free cation)	0.085	No	98	Same batch	10	Water	MeOH				Yes	No	PS-ML	Isotop. lab.1		MS/MS		None	
27 Azoxystrobin	0.31	No	113	Same batch	5	ACN					Yes	No	DSPE PSA GCB	MM-ML	No	MS/MS	GC-MS/MS		
27 Carbaryl	0.18	No	92	Same batch	5	ACN					Yes	No	DSPE PSA GCB	MM-ML	No	MS/MS	GC-MS/MS		
27 Carbendazim and benomyl	0.94	No	90	Same batch	5	ACN					Yes	No	DSPE PSA GCB	MM-ML	No	MS/MS	LC-MS/MS		
27 Deltamethrin (cis)	0.069	No	110	Same batch	5	ACN					Yes	No	DSPE PSA GCB	MM-ML	No	MS/MS	GC-MS/MS		
27 Fenitrothion	0.19	No	106	Same batch	5	ACN					Yes	No	DSPE PSA GCB	MM-ML	No	MS/MS	GC-MS/MS		
27 Fenpropimorph	2.1	No	100	Same batch	5	ACN					Yes	No	DSPE PSA GCB	MM-ML	No	MS/MS	GC-MS/MS		
27 Fluquinconazole	0.70	No	111	Same batch	5	ACN					Yes	No	DSPE PSA GCB	MM-ML	No	MS/MS	GC-MS/MS		
27 Flutriafol	1.8	No	90	Same batch	5	ACN					Yes	No	DSPE PSA GCB	MM-ML	No	MS/MS	GC-MS/MS		
27 Isoproturon	0.19	No	90	Same batch	5	ACN					Yes	No	DSPE PSA GCB	MM-ML	No	MS/MS	LC-MS/MS		
27 Kresoxim-methyl	0.41	No	104	Same batch	5	ACN					Yes	No	DSPE PSA GCB	MM-ML	No	MS/MS	GC-MS/MS		
27 Lambda-cyhalothrin	0.078	No	96	Same batch	8	AC	CH2Cl	+		No	No	GPC SPE	MM-ML	No	MS/MS	GC-MS/MS			
27 Malathion	0.10	No	94	Same batch	5	ACN					Yes	No	DSPE PSA GCB	MM-ML	No	MS/MS	GC-MS/MS		
27 Pirimiphos-methyl	0.084	No	100	Same batch	5	ACN					Yes	No	DSPE PSA GCB	MM-ML	No	MS/MS	GC-MS/MS		
27 Spiroxamine	1.5	Yes	50	Same batch	5	ACN					Yes	No	DSPE PSA C18	MM-ML	No	MS/MS	LC-MS/MS		
27 Triadimenol	1.5	No	98	Same batch	5	ACN					Yes	No	DSPE PSA GCB	MM-ML	No	MS/MS	GC-MS/MS		
28 Azoxystrobin	0.330	No	101.9	Validation	5	ACN					Yes	No	DSPE Freez.	MM-ML	No	MS/MS		Â§64 L00.00-115	
28 Carbaryl	0.178	No	103.0	Validation	5	ACN					Yes	No	DSPE Freez.	MM-ML	No	MS/MS		Â§64 L00.00-115	
28 Carbendazim and benomyl	1.50	No	78.8	Validation	5	ACN					Yes	No	DSPE Freez.	MM-ML	No	MS/MS		Â§64 L00.00-115	
28 Chlorpyrifos-methyl	0.124	No	84.0	Same batch	5	ACN					Yes	No	DSPE Freez.	MM-ML	No	MSD		Â§64 L00.00-115	

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
28	Deltamethrin (cis)	0.059	No	44.0	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MSD			Å\$64 L00.00-115
28	Fenitrothion	0.170	No	69.0	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MSD			Å\$64 L00.00-115
28	Fenpropimorph	2.26	No	72.0	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MSD			Å\$64 L00.00-115
28	Fluquinconazole	0.835	No	81.0	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MSD			Å\$64 L00.00-115
28	Flutriafol	2.26	No	78.0	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MSD			Å\$64 L00.00-115
28	Isoproturon	0.201	No	70.0	Validation	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MS/MS			Å\$64 L00.00-115
28	Kresoxim-methyl	0.382	No	84.0	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MSD			Å\$64 L00.00-115
28	Lambda-cyhalothrin	0.059	No	91.0	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MSD			Å\$64 L00.00-115
28	Malathion	0.114	No	82.0	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MSD			Å\$64 L00.00-115
28	Pirimiphos-methyl	0.076	No	82.0	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MSD			Å\$64 L00.00-115
28	Spiroxamine	1.11	No	101.0	Validation	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MS/MS			Å\$64 L00.00-115
28	Triadimenol	1.54	No	96.0	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MSD			Å\$64 L00.00-115
28	Mepiquat (free cation)	0.059	No	68.0	Same batch	10	MeOH				Yes	No		MM-ML	No	MS/MS			Å\$64L00.00-76
29	Azoxystrobin	0.191	No	82		5	EtOAc				Yes	No	None			MS/MS			
29	Carbaryl	0.189	No	84		5	EtOAc				Yes	No	None			MS/MS			
29	Carbendazim and benomyl	1.41	No	84		5	EtOAc				Yes	No	None			MS/MS			
29	Chlorpyrifos-methyl	0.0674	No	74		5	EtOAc				Yes	No	None			MS/MS			
29	Deltamethrin (cis)	0.0577	No	80		5	EtOAc				Yes	No	None			MS/MS			
29	Fenitrothion	0.131	No	78		5	EtOAc				Yes	No	None			MS/MS			
29	Fenpropimorph	2.08	No	80		5	EtOAc				Yes	No	None			MS/MS			
29	Fluquinconazole	0.554	No	101		5	EtOAc				Yes	No	None			MS/MS			
29	Flutriafol	1.89	No	80		5	EtOAc				Yes	No	None			MS/MS			
29	Isoproturon	0.165	No	91		5	EtOAc				Yes	No	None			MS/MS			
29	Kresoxim-methyl	0.293	No	86		5	EtOAc				Yes	No	None			MS/MS			
29	Lambda-cyhalothrin	0.0533	No	75		5	EtOAc				Yes	No	None			MS/MS			

LAB CODE	Pesticide	Reported result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) EtOAc	Extraction solvent 2) EtOAc	Extraction solvent 3) EtOAc	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
29	Malathion	0.0470	No	69		5					Yes	No	None			MS/MS			
29	Pirimiphos-methyl	0.0443	No	75		5	EtOAc				Yes	No	None			MS/MS			
29	Spiroxamine	0.867	No	81		5	EtOAc				Yes	No	None			MS/MS			
29	Triadimenol	1.20	No	122		5	EtOAc				Yes	No	None			MS/MS			
29	2,4-D (free acid)	0.446	No	74		5	EtOAc				Yes	No	None			MS/MS			
29	2,4-D (foli. alk hydrolysis)	0.345	No	70		5	EtOAc				Yes	Yes	None			MS/MS			
30	Azoxystrobin	0.380	No	100	Validation	5	ACN				Yes		DSPE	PS-ML	No	MS/MS	GC-MS	quechers	
30	Carbaryl	0.173	No	99	Validation	5	ACN				Yes		DSPE	PS-ML	No	MS/MS		quechers	
30	Carbendazim and benomyl	1.51	No	89	Validation	5	ACN				Yes		DSPE	PS-ML	No	MS/MS		quechers	
30	Chlorpyrifos-methyl	0.205	No	100	Validation	5	ACN				Yes		DSPE	PS-ML	No	MSD	LC-MS/MS	quechers	
30	Deltamethrin (cis)	0.0605	No	100	Validation	5	ACN				Yes		DSPE	PS-ML	No	MS/MS		quechers	
30	Fenitrothion	0.220	No	100	Validation	5	ACN				Yes		DSPE	PS-ML	No	MSD	LC-MS/MS	quechers	
30	Fenpropimorph	2.05	No	97	Validation	5	ACN				Yes		DSPE	PS-ML	No	MS/MS	GC-MS	quechers	
30	Fluquinconazole	1.05	No	94	Validation	5	ACN				Yes		DSPE	PS-ML	No	MS/MS	GC-MS	quechers	
30	Flutriafol	2.35	No	96	Validation	5	ACN				Yes		DSPE	PS-ML	No	MS/MS	GC-MS	quechers	
30	Isoproturon	0.165	No	95	Validation	5	ACN				Yes		DSPE	PS-ML	No	MS/MS		quechers	
30	Kresoxim-methyl	0.482	No	101	Validation	5	ACN				Yes		DSPE	PS-ML	No	MS/MS	GC-MS	quechers	
30	Lambda-cyhalothrin	0.0855	No	102	Validation	5	ACN				Yes		DSPE	PS-ML	No	MS/MS	GC-MS	quechers	
30	Malathion	0.120	No	100	Validation	5	ACN				Yes		DSPE	PS-ML	No	MS/MS	GC-MS	quechers	
30	Pirimiphos-methyl	0.0990	No	100	Validation	5	ACN				Yes		DSPE	PS-ML	No	MS/MS	GC-MS	quechers	
30	Spiroxamine	1.03	No	96	Validation	5	ACN				Yes		None	PS-ML	No	MS/MS	GC-MS	quechers	
30	Triadimenol	1.97	No	98	Validation	5	ACN				Yes		DSPE	PS-ML	No	MS/MS	GC-MS	quechers	
30	2,4-D (free acid)	0.511	No	95	Validation	5	ACN				Yes		None	PS-ML	No	MS/MS		quechers	
30	2,4-D (foli. alk hydrolysis)	0.675	No	96	Validation	5	ACN				Yes	Yes	Freez.	PS-ML	No	MS/MS		quechers	
30	Glyphosate	3.75	No	110	Validation	3	Water				Yes		MM-ML	No		MS/MS			

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
30	Mepiquat (free cation)	0.144	No	102	Validation	5	MeOH				Yes		None	MM-ML	No		MS/MS		CRL SRM
31	Fenitrothion	0.218	No	104	Same batch	5	AC	CH ₂ Cl			Yes	No	SPE	PS-ML		NPD		Two columns	
31	Malathion	0.128	No	110	Same batch	5	AC	CH ₂ Cl			Yes	No	SPE	PS-ML		NPD		Two columns	
32	Azoxystrobin	0.374	No	98	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2		MS/MS	LC-MS/MS	Quechers
32	Carbaryl	0.180	No	99	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2		MS/MS	LC-MS/MS	Quechers
32	Carbendazim and benomyl	1.32	No	89	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2		MS/MS	LC-MS/MS	Quechers
32	Chlorpyrifos-methyl	0.144	No	90	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2		MS/MS	LC-MS/MS	Quechers
32	Deltamethrin (cis)	0.058	No	110	Same batch	10	ACN	AC			No	No	GPC SPE	St. Ad.	Mirex	MSD		negative CI	VDLUFA 3.3.7.1
32	Fenitrothion	0.210	No	110	Same batch	10	ACN	AC			No	No	GPC SPE	St. Ad.	Mirex	MSD		negative CI	VDLUFA 3.3.7.1
32	Fenpropimorph	2.76	No	87	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2		MS/MS	LC-MS/MS	Quechers
32	Fluquinconazole	1.08	No	112	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2		MS/MS	LC-MS/MS	Quechers
32	Flutriafol	2.19	No	95	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2		MS/MS	LC-MS/MS	Quechers
32	Isoproturon	0.176	No	95	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2		MS/MS	LC-MS/MS	Quechers
32	Kresoxim-methyl	0.447	No	86	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2		MS/MS	LC-MS/MS	Quechers
32	Lambda-cyhalothrin	0.090	No	110	Same batch	10	ACN	AC			No	No	GPC SPE	St. Ad.	Mirex	MSD		negative CI	VDLUFA 3.3.7.1
32	Malathion	0.134	No	99	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2		MS/MS	LC-MS/MS	Quechers
32	Pirimiphos-methyl	0.084	No	82	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2		MS/MS	LC-MS/MS	Quechers
32	Spiroxamine	1.27	No	91	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2		MS/MS	LC-MS/MS	Quechers
32	Triadimenol	1.78	No	98	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2		MS/MS	LC-MS/MS	Quechers
33	Azoxystrobin	0.448	No	107.7	Same batch	5	ACN				Yes	No	DSPE C18	MM-ML	TPP	NPD		GC-MS	quechers
33	Carbaryl	0.407	No	115.3	Same batch	5	ACN				Yes	No	DSPE C18	MM-ML	TPP	NPD		GC-MS	quechers
33	Carbendazim and benomyl	1.28	No	83.5	Same batch	5	ACN				Yes	No	DSPE C18	MM-ML	TPP	NPD		GC-MS	quechers
33	Chlorpyrifos-methyl	0.17	No	106.8	Same batch	5	ACN				Yes	No	DSPE C18	MM-ML	TPP	NPD		GC-MS	quechers
33	Deltamethrin (cis)	0.062	No	90.9	Same batch	5	ACN				Yes	No	DSPE C18	MM-ML	TPP	NPD		GC-MS	quechers
33	Fenitrothion	0.27	No	102.5	Same batch	5	ACN				Yes	No	DSPE C18	MM-ML	TPP	NPD		GC-MS	quechers

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) ACN	Extraction solvent 2) ACN	Extraction solvent 3) ACN	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
33	Fenpropimorph	2.55	No	96	Same batch	5					Yes	No	DSPE C18	MM-ML	TPP	NPD	GC-MS	quechers	
33	Fluquinconazole	0.904	No	99.6	Same batch	5					Yes	No	DSPE C18	MM-ML	TPP	NPD	GC-MS	quechers	
33	Flutriafol	2.48	No	111.5	Same batch	5					Yes	No	DSPE C18	MM-ML	TPP	NPD	GC-MS	quechers	
33	Kresoxim-methyl	0.45	No	106	Same batch	5					Yes	No	DSPE C18	MM-ML	TPP	NPD	GC-MS	quechers	
33	Lambda-cyhalothrin	0.055	No	95	Same batch	5					Yes	No	DSPE C18	MM-ML	TPP	NPD	GC-MS	quechers	
33	Malathion	0.147	No	105.8	Same batch	5					Yes	No	DSPE C18	MM-ML	TPP	NPD	GC-MS	quechers	
33	Pirimiphos-methyl	0.08	No	85	Same batch	5					Yes	No	DSPE C18	MM-ML	TPP	NPD	GC-MS	quechers	
33	Spiroxamine	1.364	No	92.5	Same batch	5					Yes	No	DSPE C18	MM-ML	TPP	NPD	GC-MS	quechers	
33	Triadimenol	1.378	No	101.5	Same batch	5					Yes	No	DSPE C18	MM-ML	TPP	NPD	GC-MS	quechers	
34	Azoxystrobin	0.213	No	80.5	Same batch	25	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML	No	ECD	GC-MS	modulare Multimethode gem ASU §64 LFGB, L 00.00-34	
34	Carbaryl	0.172	No	120.0	Same batch	5	MeOH				Yes	No	L/I part.	MM-ML	No	MS/MS	LC-MS/MS	"BFR"-Methode gem ASU §64 LFGB, L 00.00-113	
34	Carbendazim and benomyl	1.43	No	77.9	Same batch	5	MeOH				Yes	No	L/I part.	MM-ML	No	MS/MS	LC-MS/MS	"BFR"-Methode gem ASU §64 LFGB, L 00.00-113	
34	Chlorpyrifos-methyl	0.108	No	83.3	Same batch	25	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML	No	NPD	GC-MS	modulare Multimethode gem ASU §64 LFGB, L 00.00-34	
34	Deltamethrin (cis)	0.045	No	75.7	Same batch	25	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML	No	ECD	GC-MS	modulare Multimethode gem ASU §64 LFGB, L 00.00-34	
34	Fenitrothion	0.146	No	81.0	Same batch	25	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML	No	NPD	GC-MS	modulare Multimethode gem ASU §64 LFGB, L 00.00-34	
34	Fenpropimorph	2.06	No	90.3	Same batch	25	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML	No	NPD	GC-MS	modulare Multimethode gem ASU §64 LFGB, L 00.00-34	
34	Fluquinconazole	0.556	No	91.8	Same batch	25	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML	No	NPD	GC-MS	modulare Multimethode gem ASU §64 LFGB, L 00.00-34	
34	Flutriafol	1.72	No	93.9	Same batch	25	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML	No	NPD	GC-MS	modulare Multimethode gem ASU §64 LFGB, L 00.00-34	
34	Isoproturon	0.156	No	103.0	Same batch	5	MeOH				Yes	No	L/I part.	MM-ML	No	MS/MS	LC-MS/MS	"BFR"-Methode gem ASU §64 LFGB, L 00.00-113	
34	Kresoxim-methyl	0.224	No	81.9	Same batch	25	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML	No	ECD	GC-MS	modulare Multimethode gem ASU §64 LFGB, L 00.00-34	
34	Lambda-cyhalothrin	0.054	No	75.3	Same batch	25	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML	No	ECD	GC-MS	modulare Multimethode gem ASU §64 LFGB, L 00.00-34	
34	Malathion	0.075	No	90.9	Same batch	25	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML	No	NPD	GC-MS	modulare Multimethode gem ASU §64 LFGB, L 00.00-34	
34	Pirimiphos-methyl	0.068	No	86.8	Same batch	25	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML	No	NPD	GC-MS	modulare Multimethode gem ASU §64 LFGB, L 00.00-34	
34	Spiroxamine	0.824	No	92.1	Same batch	5	MeOH				Yes	No	L/I part.	MM-ML	No	MS/MS	LC-MS/MS	"BFR"-Methode gem ASU §64 LFGB, L 00.00-113	
34	Triadimenol	1.45	No	83.6	Same batch	25	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML	No	ECD	GC-MS	modulare Multimethode gem ASU §64 LFGB, L 00.00-34	

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
34	2,4-D (free acid)	0.331	No	82.8	Same batch	5	MeOH				Yes	No	L/I part.	MM-ML	No		MS/MS	LC-MS/MS	modifizierte "Bfr"-Methode gem ASU §64 LFGB, L 00.00-113
34	2,4-D (foll. alk hydrolysis)	0.353	No	92.1	Same batch	5	MeOH				Yes	Yes	L/I part.	MM-ML	No		MS/MS	LC-MS/MS	modifizierte "Bfr"-Methode gem ASU §64 LFGB, L 00.00-113
34	Mepiquat (free cation)	0.118	No	114.0	Same batch	5	MeOH				Yes	No	None	MM-ML	Isotop. lab.1		MS/MS	LC-MS/MS	Methode gem ASU §64 LFGB, L 00.00-76
35	Chlorpyrifos-methyl	0.10	No	75	Validation	20	AC	HEX	ISO		No	No	SPE	PS-ML	Ethion Isotop. lab.2 Pirimicarb	MSD	GC-MS		
36	Azoxystrobin	0.370	No	124	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2 Pirimicarb	MS/MS	LC-MS/MS	QuEChERS	
36	Carbaryl	0.189	No	114	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2 Pirimicarb	MS/MS	LC-MS/MS	QuEChERS	
36	Carbendazim and benomyl	1.53	No	114	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2 Pirimicarb	MS/MS	LC-MS/MS	QuEChERS	
36	Chlorpyrifos-methyl	0.126	No	106	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2 Pirimicarb , PCB 138	TOF	GC-MS	QuEChERS	
36	Deltamethrin (cis)	0.050	No	112	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2 Pirimicarb , PCB 138	TOF	GC-MS	QuEChERS	
36	Fenitrothion	0.161	No	115	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2 Pirimicarb , PCB 138	TOF	GC-MS	QuEChERS	
36	Fenpropimorph	2.28	No	106	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2 Pirimicarb	MS/MS	LC-MS/MS	QuEChERS	
36	Fluquinconazole	0.919	No	103	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2 Pirimicarb , PCB 138	TOF	GC-MS	QuEChERS	
36	Flutriafol	2.42	No	119	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2 Pirimicarb	MS/MS	LC-MS/MS	QuEChERS	
36	Isoproturon	0.205	No	115	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2 Pirimicarb	MS/MS	LC-MS/MS	QuEChERS	
36	Kresoxim-methyl	0.411	No	110	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2 Pirimicarb , PCB 138	TOF	GC-MS	QuEChERS	
36	Lambda-cyhalothrin	0.065	No	106	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2 Pirimicarb , PCB 138	TOF	GC-MS	QuEChERS	
36	Malathion	0.115	No	111	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2 Pirimicarb	MS/MS	LC-MS/MS	QuEChERS	
36	Pirimiphos-methyl	0.081	No	108	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2 Pirimicarb , PCB 138	TOF	GC-MS	QuEChERS	
36	Spiroxamine	1.14	No	115	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2 Pirimicarb	MS/MS	LC-MS/MS	QuEChERS	
36	Triadimenol	2.54	No	120	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Isotop. lab.2 Pirimicarb , PCB 138	TOF	GC-MS	QuEChERS	
36	2,4-D (free acid)	0.350	No	97	Same batch	5	ACN				Yes	No	None	MM-ML	Nicarbazin	MS/MS	LC-MS/MS	QuEChERS	
36	2,4-D (foll. alk hydrolysis)	0.365	No	106	Same batch	5	ACN				Yes	Yes	None	MM-ML	Nicarbazin	MS/MS	LC-MS/MS	QuEChERS	
36	Glyphosate	3.33	No	95	Same batch	5	MeOH				Yes	No	None	MM-ML	Isotop. lab.1	MS/MS	LC-MS/MS	High Polar Pesticides CRL Single Residue Methods	
36	Mepiquat (free cation)	0.114	No	98	Same batch	5	MeOH				Yes	No	None	MM-ML	Isotop. lab.1	MS/MS	LC-MS/MS	High Polar Pesticides CRL Single Residue Methods	
37	Azoxystrobin	0.265	No	78	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML	No	ECD	GC-MS/MS	quechers	

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) ACN	Extraction solvent 2) ACN	Extraction solvent 3) ACN	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
37	Carbaryl	0.142	No	92	Same batch	5					Yes		DSPE Freez.	MM-ML		ITD	GC-MS/MS	quechers	
37	Carbendazim and benomyl	1.47	No	100	Same batch	5	ACN				Yes		Freez.	MM-ML	No		Diod. Ar.	LC-ITQ	quechers
37	Chlorpyrifos-methyl	0.107	No	100	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML		ITD	GC-MS/MS	quechers	
37	Deltamethrin (cis)	0.054	No	91	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML	No	ECD	GC-MS/MS	quechers	
37	Fenitrothion	0.172	No	95	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML		ITD	GC-MS/MS	quechers	
37	Fenpropimorph	1.987	No	102	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML		ITD	GC-MS/MS	quechers	
37	Fluquinconazole	0.684	No	85	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML		ITD	GC-MS/MS	quechers	
37	Flutriafol	2.43	No	112.5	Same batch	5	ACN				Yes		Freez.	MM-ML	No		LC-MS/MS	quechers	
37	Isoproturon	0.168	No	85	Same batch	5	ACN				Yes		Freez.	MM-ML	No		LC-MS/MS	quechers	
37	Kresoxim-methyl	0.374	No	80	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML	No	ECD	GC-MS/MS	quechers	
37	Lambda-cyhalothrin	0.062	No	80	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML	No	ECD	GC-MS/MS	quechers	
37	Malathion	0.082	No	81	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML	No	FPD	GC-MS	quechers	
37	Pirimiphos-methyl	0.069	No	85	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML		ITD	GC-MS/MS	quechers	
37	Spiroxamine	0.956	No	85	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML		ITD	GC-MS/MS	quechers	
37	Triadimenol	1.144	No	87	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML		ITD	GC-MS/MS	quechers	
37	2,4-D (free acid)	0.213	No	60	Same batch	5	ACN				Yes		Freez.	MM-ML			LC-MS/MS	quechers	
37	2,4-D (foll. alk hydrolysis)	0.443	No	90	Same batch	5	ACN				Yes	Yes	Freez.	MM-ML			LC-MS/MS	quechers	
37	Mepiquat (free cation)	0.063	No	70	Same batch	5	MeOH	Water					Filter	MM-ML			LC-MS/MS		
38	Azoxystrobin	0.18	No	102.8	Same batch	15	AC	CH2Cl	PE		Yes	No	GPC	MM-ML	TPP		MS/MS		Mini Luke
38	Carbaryl	0.14	No	95.8	Same batch	15	AC	CH2Cl	PE		Yes	No	GPC	MM-ML	TPP		MS/MS		Mini Luke
38	Carbendazim and benomyl	1.59	No	94.9	Same batch	15	ACN				Yes	No	DSPE	MM-ML	Ethoprophos		MS/MS		Quechers
38	Chlorpyrifos-methyl	0.09	No	97.1	Same batch	15	AC	CH2Cl	PE		Yes	No	GPC	MM-ML	TPP		MS/MS		Mini Luke
38	Deltamethrin (cis)	0.05	No	99.3	Same batch	15	AC	CH2Cl	PE		Yes	No	GPC	MM-ML	TPP		MS/MS		Mini Luke
38	Fenitrothion	0.15	No	97.5	Same batch	15	AC	CH2Cl	PE		Yes	No	GPC	MM-ML	TPP		MS/MS		Mini Luke
38	Fenpropimorph	3.61	No	108.2	Same batch	15	ACN				Yes	No	DSPE	MM-ML	Ethoprophos		MS/MS		Quechers

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
38	Fluquinconazole	1.79	No	187.3	Same batch	15	ACN				Yes	No	DSPE	MM-ML	Ethoprophos		MS/MS		Quechers
38	Flutriafol	6.19	No	132.3	Same batch	15	ACN				Yes	No	DSPE	MM-ML	Ethoprophos		MS/MS		Quechers
38	Isoproturon	0.18	No	94.2	Same batch	15	ACN				Yes	No	DSPE	MM-ML	Ethoprophos		MS/MS		Quechers
38	Kresoxim-methyl	0.26	No	94.0	Same batch	15	AC	CH2Cl	PE		Yes	No	GPC	MM-ML	TPP		MS/MS		Mini Luke
38	Lambda-cyhalothrin	0.06	No	101.2	Same batch	15	AC	CH2Cl	PE		Yes	No	GPC	MM-ML	TPP		MS/MS		Mini Luke
38	Malathion	0.05	No	95.4	Same batch	15	AC	CH2Cl	PE		Yes	No	GPC	MM-ML	TPP		MS/MS		Mini Luke
38	Pirimiphos-methyl	0.06	No	95.5	Same batch	15	AC	CH2Cl	PE		Yes	No	GPC	MM-ML	TPP		MS/MS		Mini Luke
38	Spiroxamine	0.98	No	93.6	Same batch	15	ACN				Yes	No	DSPE	MM-ML	Ethoprophos		MS/MS		Quechers
38	Triadimenol	0.81	No	101.4	Same batch	15	AC	CH2Cl	PE		Yes	No	GPC	MM-ML	TPP		MS/MS		Mini Luke
38	2,4-D (free acid)	0.02	Yes	14.9	Same batch	15	ACN				Yes	No	DSPE	MM-ML	Ethoprophos		MS/MS		Quechers
38	Glyphosate	1.98	No	98.2	Same batch	10	MeOH				Yes	No	None	MM-ML	Isotop. lab.1		MS/MS		CRL Quat
38	Mepiquat (free cation)	0.04	No	100.2	Same batch	10	MeOH				Yes	No	None	MM-ML	Isotop. lab.1		MS/MS		CRL Quat
39	Azoxystrobin	0.214	No	95	Same batch	5	ACN				Yes	No	Freez.	MM-ML	Isoproturon		MS/MS	LC-MS/MS	QuEChERS
39	Carbaryl	0.110	No	101	Same batch	5	ACN				Yes	No	Freez.	MM-ML	Isotop. lab.2 Carbofuran		MS/MS	LC-MS/MS	QuEChERS
39	Chlorpyrifos-methyl	0.082	No	84	Same batch	5	ACN				Yes	No	SPE	MM-ML	Isotop. lab.1		MS/MS	GC-MS/MS	QuEChERS
39	Deltamethrin (cis)	0.040	No	97	Same batch	5	ACN				Yes	No	SPE	MM-ML	Isotop. lab.2 Cypermethrin		MS/MS	GC-MS/MS	QuEChERS
39	Fenitrothion	0.194	No	99	Same batch	5	ACN				Yes	No	SPE	MM-ML	Chlorpyrifos		MS/MS	GC-MS/MS	QuEChERS
39	Fenpropimorph	0.983	No	89	Same batch	5	ACN				Yes	No	Freez.	MM-ML	Isotop. lab.2 Carbofuran		MS/MS	LC-MS/MS	QuEChERS
39	Fluquinconazole	0.601	No	107	Same batch	5	ACN				Yes	No	Freez.	MM-ML	Isotop. lab.2 Propiconazole		MS/MS	LC-MS/MS	QuEChERS
39	Flutriafol	1.12	No	111	Same batch	5	ACN				Yes	No	Freez.	MM-ML	Isotop. lab.2 Carbofuran		MS/MS	LC-MS/MS	QuEChERS
39	Isoproturon	0.088	No	86	Same batch	5	ACN				Yes	No	Freez.	MM-ML	Isotop. lab.1		MS/MS	LC-MS/MS	QuEChERS
39	Lambda-cyhalothrin	0.041	No	91	Same batch	5	ACN				Yes	No	SPE	MM-ML	Isotop. lab.2 Cypermethrin		MS/MS	GC-MS/MS	QuEChERS
39	Malathion	0.085	No	75	Same batch	5	ACN				Yes	No	SPE	MM-ML	Isotop. lab.2 Chlorpyrifos		MS/MS	GC-MS/MS	QuEChERS
39	- Malaoxon	0.034	No	93	Same batch	5	ACN				Yes	No	SPE	MM-ML	Isotop. lab.2 Chlorpyrifos		MS/MS	GC-MS/MS	QuEChERS
39	Pirimiphos-methyl	0.048	No	78	Same batch	5	ACN				Yes	No	SPE	MM-ML	Isotop. lab.2 Chlorpyrifos		MS/MS	GC-MS/MS	QuEChERS

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) ACN	Extraction solvent 2) ACN	Extraction solvent 3) ACN	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
39	Spiroxamine	0.882	No	81	Same batch	5					Yes	No	SPE	MM-ML	Isotop. lab.2 Chlorpyrifos	MS/MS	GC-MS/MS	QuEChERS	
39	2,4-D (free acid)	0.374	No	84	Same batch	5	ACN				Yes	No	Freez.	MM-ML	Isotop. lab.2 Bendazone	MS/MS	LC-MS/MS	QuEChERS	
40	Azoxystrobin	0.344	No	103	Same batch	5	ACN				Yes	No	DSPE PSA C18	MM-ML	Tris	MS/MS	LC-MS/MS		
40	Carbaryl	0.164	No	91	Same batch	5	ACN				Yes	No	DSPE PSA C18	MM-ML	Tris	MS/MS	LC-MS/MS		
40	Carbendazim and benomyl	1.41	No	96	Same batch	5	ACN				Yes	No	DSPE PSA C18	MM-ML	Tris	MS/MS	LC-MS/MS		
40	Chlorpyrifos-methyl	0.142	No	104	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Tris	MSD	GC-MS		
40	Deltamethrin (cis)	0.039	No	73	Same batch	5	ACN				Yes	No	DSPE	MM-ML	PCB209	MSD	GC-MS		
40	Fenitrothion	0.220	No	113	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Tris	MSD	GC-MS		
40	Fenpropimorph	2.15	No	98	Same batch	5	ACN				Yes	No	DSPE PSA C18	MM-ML	Tris	MS/MS	LC-MS/MS		
40	Fluquinconazole	0.855	No	102	Same batch	5	ACN				Yes	No	DSPE PSA C18	MM-ML	Tris	MS/MS	LC-MS/MS		
40	Flutriafol	2.31	No	116	Same batch	5	ACN				Yes	No	DSPE PSA C18	MM-ML	Tris	MS/MS	LC-MS/MS		
40	Isoproturon	0.182	No	105	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Tris	MSD	GC-MS		
40	Kresoxim-methyl	0.457	No	108	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Tris	MSD	GC-MS		
40	Lambda-cyhalothrin	0.060	No	80	Same batch	5	ACN				Yes	No	DSPE	MM-ML	PCB209	MSD	GC-MS		
40	Malathion	0.096	No	107	Same batch	5	ACN				Yes	No	DSPE PSA C18	MM-ML	Tris	MS/MS	LC-MS/MS		
40	- Malaoxon	<0.01			Same batch	5	ACN				Yes	No	DSPE PSA C18	MM-ML	Tris	MS/MS	LC-MS/MS		
40	Pirimiphos-methyl	0.078	No	94	Same batch	5	ACN				Yes	No	DSPE PSA C18	MM-ML	Tris	MS/MS	LC-MS/MS		
40	Spiroxamine	0.456	No	54	Same batch	5	ACN				Yes	No	DSPE PSA C18	MM-ML	Tris	MS/MS	LC-MS/MS		
40	-Triadimefon	1.75	No	116	Same batch	5	ACN				Yes	No	DSPE PSA C18	MM-ML	Tris	MS/MS	LC-MS/MS		
40	Triadimenol	<0.01			Same batch	5	ACN				Yes	No	DSPE PSA C18	MM-ML	Tris	MS/MS	LC-MS/MS		
40	2,4-D (free acid)	0.320	No	104	Same batch	5	ACN				Yes	No	Freez.	MM-ML	nicarbazin	MS/MS	LC-MS/MS		
40	Glyphosate	3.58	Yes automatic	85	Same batch	5	MeOH				Yes	No	Freez.	MM-ML	Isotop. lab.1	MS/MS	LC-MS/MS		
40	Mepiquat (free cation)	0.084	Yes automatic	101	Same batch	5	MeOH				Yes	No	Freez.	MM-ML	Isotop. lab.1	MS/MS	LC-MS/MS		
41	Azoxystrobin	0.158	No	92	Same batch	20	AC	CH2Cl			No	No	None	MM-ML	PCB97 Isotop. lab.2	ECD	GC-MS	Anal.Meth.f.Pest.Res.in Foodst.NL 6th Ed. 1996	
41	Carbaryl	0.222	No	95	Same batch	5	MeOH				Yes	No	SPE	MM-ML	carbendazim	MS/MS	None	Klein, J.; Alder, L.; JAOAC; 86,1015 (2003)	

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
41	Carbendazim and benomyl	1.34	No	100	Same batch	5	MeOH				Yes	No	SPE	MM-ML	Isotop. lab.1		MS/MS	None	Klein, J.; Alder, L.; JAOAC; 86,1015 (2003)
41	Chlorpyrifos-methyl	0.134	No	99	Same batch	20	AC	CH ₂ Cl			No	No	None	MM-ML		FPD	GC-MS		Anal.Meth.f.Pest.Res.in Foodst.NL 6th Ed. 1996
41	Deltamethrin (cis)	0.0471	No	96	Same batch	20	AC	CH ₂ Cl			No	No	None	MM-ML	PCB97	ECD	GC-MS		Anal.Meth.f.Pest.Res.in Foodst.NL 6th Ed. 1996
41	Fenitrothion	0.244	No	100	Same batch	20	AC	CH ₂ Cl			No	No	None	MM-ML		FPD	GC-MS		Anal.Meth.f.Pest.Res.in Foodst.NL 6th Ed. 1996
41	Fluquinconazole	0.469	No	99	Same batch	20	AC	CH ₂ Cl			No	No	None	MM-ML	PCB97	ECD	GC-MS		Anal.Meth.f.Pest.Res.in Foodst.NL 6th Ed. 1996
41	Flutriafol	1.14	No	121	Same batch	20	AC	CH ₂ Cl			No	No	None	MM-ML	Trifluralin	NPD	GC-MS		Anal.Meth.f.Pest.Res.in Foodst.NL 6th Ed. 1996
41	Isoproturon	0.158	No	96	Same batch	5	MeOH				Yes	No	SPE	MM-ML	Isotop. lab.2		MS/MS	None	Klein, J.; Alder, L.; JAOAC; 86,1015 (2003)
41	Kresoxim-methyl	0.301	No	107	Same batch	20	AC	CH ₂ Cl			No	No	None	MM-ML	PCB97	ECD	GC-MS		Anal.Meth.f.Pest.Res.in Foodst.NL 6th Ed. 1996
41	Lambda-cyhalothrin	0.0731	No	103	Same batch	20	AC	CH ₂ Cl			No	No	None	MM-ML	PCB97	ECD	GC-MS		Anal.Meth.f.Pest.Res.in Foodst.NL 6th Ed. 1996
41	Malathion	0.0733	No	98	Same batch	20	AC	CH ₂ Cl			No	No	None	MM-ML		FPD	GC-MS		Anal.Meth.f.Pest.Res.in Foodst.NL 6th Ed. 1996
41	Pirimiphos-methyl	0.0930	No	103	Same batch	20	AC	CH ₂ Cl			No	No	None	MM-ML		FPD	GC-MS		Anal.Meth.f.Pest.Res.in Foodst.NL 6th Ed. 1996
41	Spiroxamine	0.920	No	86	Same batch	5	MeOH				Yes	No	SPE	MM-ML	Isotop. lab.2 carbendazim		MS/MS	None	Klein, J.; Alder, L.; JAOAC; 86,1015 (2003)
41	Triadimenol	1.62	No	95	Same batch	5	MeOH				Yes	No	SPE	MM-ML	Isotop. lab.2 carbendazim		MS/MS	None	Klein, J.; Alder, L.; JAOAC; 86,1015 (2003)
41	Mepiquat (free cation)	0.0605	Yes automatic	100	isotop. lab.	20	MeOH				Yes	No	None	PS-ML	Isotop. lab.1		MS/MS	None	CEN/TC 275 EN 15054 (2005)
42	Azoxystrobin	0.387	No	114	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Desmetryn		MS/MS	None	
42	Carbaryl	0.217	No	111	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Desmetryn		MS/MS	None	
42	Carbendazim and benomyl	1.39	No	95	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Desmetryn		MS/MS	None	
42	Chlorpyrifos-methyl	0.123	No	90	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Desmetryn		MS/MS	None	
42	Deltamethrin (cis)	0.058	No	61	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Desmetryn		MS/MS	None	
42	Fenitrothion	0.204	No	93	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Desmetryn		MS/MS	None	
42	Fenpropimorph	2.50	No	103	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Desmetryn		MS/MS	None	
42	Fluquinconazole	0.713	No	70	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Desmetryn		MS/MS	None	
42	Flutriafol	2.84	No	108	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Desmetryn		MS/MS	None	
42	Isoproturon	0.191	No	110	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Desmetryn		MS/MS	None	
42	Kresoxim-methyl	0.689	No	92	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Desmetryn		MS/MS	None	

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) ACN	Extraction solvent 2) ACN	Extraction solvent 3) ACN	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
42	Lambda-cyhalothrin	0.082	No	83	Same batch	5					Yes	No	DSPE	MM-ML	Desmetryn	MS/MS		None	
42	Malathion	0.114	No	91	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Desmetryn	MS/MS		None	
42	Pirimiphos-methyl	0.087	No	95	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Desmetryn	MS/MS		None	
42	Spiroxamine	1.05	No	110	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Desmetryn	MS/MS		None	
42	Triadimenol	2.15	No	104	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Desmetryn	MS/MS		None	
42	2,4-D (free acid)	0.130	No	54	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Desmetryn	MS/MS		None	
44	Azoxystrobin	0.402	No	87	Same batch	5	ACN				Yes		Freez.	PS-ML	No		MS/MS	LC-MS/MS	EN15662
44	Carbaryl	0.183	No	99	Same batch	5	ACN				Yes		Freez.	PS-ML	No		MS/MS	LC-MS/MS	EN15662
44	Chlorpyrifos-methyl	0.128	No	94	Same batch	5	ACN				Yes		Freez.	PS-ML	No		MS/MS	LC-MS/MS	EN15662
44	Deltamethrin (cis)	0.0821	No	84	Same batch	5	ACN				Yes		Freez.	PS-ML	No		MS/MS	LC-MS/MS	EN15662
44	Fenitrothion	0.201	No	83	Same batch	5	ACN				Yes		Freez.	PS-ML	No		MS/MS	LC-MS/MS	EN15662
44	Kresoxim-methyl	0.471	No	81	Same batch	5	ACN				Yes		Freez.	PS-ML	No		MS/MS	LC-MS/MS	EN15662
44	Lambda-cyhalothrin	0.0622	No	85	Same batch	5	ACN				Yes		Freez.	MM-ML	No	MSD	GC-MS		EN15662
44	Malathion	0.119	No	93	Same batch	5	ACN				Yes		Freez.	PS-ML	No		MS/MS	LC-MS/MS	EN15662
44	Pirimiphos-methyl	0.0791	No	85	Same batch	5	ACN				Yes		Freez.	MM-ML	No	MSD	GC-MS		EN15662
44	Triadimenol	1.30	No	83	Same batch	5	ACN				Yes		Freez.	PS-ML	No		MS/MS	LC-MS/MS	EN15662
45	Chlorpyrifos-methyl	0.158	No	96	Same batch	5	AC	CH2Cl			Yes	No	SPE	PS-ML		NPD		Two columns	
45	Fenitrothion	0.168	No	92	Same batch	5	AC	CH2Cl			Yes	No	SPE	PS-ML		NPD		Two columns	
45	Malathion	0.114	No	94	Same batch	5	AC	CH2Cl			Yes	No	SPE	PS-ML		NPD		Two columns	
45	Pirimiphos-methyl	0.095	No	91	Same batch	5	AC	CH2Cl			Yes	No	SPE	PS-ML		NPD		Two columns	
46	Azoxystrobin	0.185	No	69.7	Same batch	15	AC	CH2Cl	PE		Yes	No	None	MM-ML	HCB	ITD	GC-TOF		
46	Carbaryl	0.124	No	66.7	Same batch	15	AC	CH2Cl	PE		Yes	No	None	MM-ML	HCB	ITD	GC-TOF		
46	Chlorpyrifos-methyl	0.061	No	70	Same batch	15	AC	CH2Cl	PE		Yes	No	None	MM-ML	HCB	ITD	GC-TOF		
46	Deltamethrin (cis)	0.034	No	63.5	Same batch	15	AC	CH2Cl	PE		Yes	No	None	MM-ML	HCB	ITD	GC-TOF		
46	Fenitrothion	0.135	No	69.4	Same batch	15	AC	CH2Cl	PE		Yes	No	None	MM-ML	HCB	ITD	GC-TOF		

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
46	Isoproturon	0.027	No	65	Same batch	10	ACN				Yes		SPE	MM-ML	HCB	ITD		GC-TOF	
46	Kresoxim-methyl	0.211	No	63.5	Same batch	15	AC	CH ₂ Cl	PE		Yes	No	None	MM-ML	HCB	ITD		GC-TOF	
46	Lambda-cyhalothrin	0.045	No	49.5	Same batch	15	AC	CH ₂ Cl	PE		Yes	No	None	MM-ML	HCB	ITD		GC-TOF	
46	Malathion	0.020	No	40.2	Same batch	15	AC	CH ₂ Cl	PE		Yes	No	None	MM-ML	HCB	ITD		GC-TOF	
46	Pirimiphos-methyl	0.043	No	57	Same batch	15	AC	CH ₂ Cl	PE		Yes	No	None	MM-ML	HCB	ITD		GC-TOF	
47	Azoxystrobin	0.307	No	101	Same batch	5	ACN				Yes		SPE Freez.	MM-ML		MSD	None	EN 15662:2008 (E)	
47	Carbaryl	0.123	No	81	Same batch	5	ACN				Yes		SPE Freez.	MM-ML		MSD	None		
47	Chlorpyrifos-methyl	0.142	No	105	Same batch	5	ACN				Yes		SPE Freez.	MM-ML		MSD	None	EN 15662:2008 (E)	
47	Deltamethrin (cis)	0.0495	No	92	Same batch	5	ACN				Yes		SPE Freez.	MM-ML		MSD	None	EN 15662:2008 (E)	
47	Fenitrothion	0.189	No	103	Same batch	5	ACN				Yes		SPE Freez.	MM-ML		MSD	None	EN 15662:2008 (E)	
47	Fenpropimorph	2.42	No	86	Same batch	5	ACN				Yes		SPE Freez.	MM-ML		MSD	None	EN 15662:2008 (E)	
47	Fluquinconazole	0.789	No	89	Same batch	5	ACN				Yes		SPE Freez.	MM-ML		MSD	None	EN 15662:2008 (E)	
47	Flutriafol	2.19	No	87	Same batch	5	ACN				Yes		SPE Freez.	MM-ML		MSD	None	EN 15662:2008 (E)	
47	Kresoxim-methyl	0.455	No	92	Same batch	5	ACN				Yes		SPE Freez.	MM-ML		MSD	None	EN 15662:2008 (E)	
47	Lambda-cyhalothrin	0.0721	No	103	Same batch	5	ACN				Yes		SPE Freez.	MM-ML		MSD	None	EN 15662:2008 (E)	
47	Malathion	0.118	No	104	Same batch	5	ACN				Yes		SPE Freez.	MM-ML		MSD	None	EN 15662:2008 (E)	
47	Pirimiphos-methyl	0.0837	No	103	Same batch	5	ACN				Yes		SPE Freez.	MM-ML		MSD	None	EN 15662:2008 (E)	
47	Spiroxamine	1.35	No	84	Same batch	5	ACN				Yes		SPE Freez.	MM-ML		MSD	None	EN 15662:2008 (E)	
47	Triadimenol	1.79	No	90	Same batch	5	ACN				Yes		SPE Freez.	MM-ML		MSD	None	EN 15662:2008 (E)	
48	Azoxystrobin	0.330	No	102	Same batch	12	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Methode 3.3.7.1	
48	Carbaryl	0.191	No	111	Same batch	12	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Methode 3.3.7.1	
48	Carbendazim and benomyl	0.860	No	94	Same batch	12	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Methode 3.3.7.1	
48	Chlorpyrifos-methyl	0.172	No	102	Same batch	12	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML		MSD	NCI	VDLUFA-Methode 3.3.7.1	
48	Deltamethrin (cis)	0.084	No	104	Same batch	12	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML		MSD	NCI	VDLUFA-Methode 3.3.7.1	
48	Fenitrothion	0.307	No	101	Same batch	12	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Methode 3.3.7.1	

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) AC	Extraction solvent 2) EtOAc	Extraction solvent 3) Cy-He	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
48	Fenpropimorph	1.051	No	77	Same batch	12	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Methode 3.3.7.1	
48	Fluquinconazole	0.778	No	100	Same batch	12	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Methode 3.3.7.1	
48	Flutriafol	2.771	No	108	Same batch	12	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Methode 3.3.7.1	
48	Isoproturon	0.184	No	111	Same batch	12	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Methode 3.3.7.1	
48	Kresoxim-methyl	0.412	No	104	Same batch	12	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Methode 3.3.7.1	
48	Lambda-cyhalothrin	0.073	No	88	Same batch	12	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML	MSD	NCI	VDLUFA-Methode 3.3.7.1		
48	Malathion	0.130	No	106	Same batch	12	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Methode 3.3.7.1	
48	- Malaoxon	0.0081	No	106	Same batch	12	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Methode 3.3.7.1	
48	Pirimiphos-methyl	0.089	No	109	Same batch	12	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Methode 3.3.7.1	
48	Spiroxamine	0.942	Yes	61	Same batch	12	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Methode 3.3.7.1	
48	-Triadimefon	0.0092	No	108	Same batch	12	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Methode 3.3.7.1	
48	Triadimenol	1.721	No	68	Same batch	12	AC	EtOAc	Cy-He		Yes	No	GPC	MM-ML		MS/MS	LC-MS/MS	VDLUFA-Methode 3.3.7.1	
49	Fenitrothion	0.208	No	103	Same batch	10	AC	CH2Cl			Yes	No	SPE	MM-SL	No	NPD	Two columns		
49	Malathion	0.115	No	109	Same batch	10	AC	CH2Cl			Yes	No	SPE	MM-SL	No	NPD	Two columns		
49	Pirimiphos-methyl	0.091	No	106	Same batch	10	AC	CH2Cl			Yes	No	SPE	MM-SL	No	NPD	Two columns		
50	Azoxystrobin	0.305	No	93	Same batch	5	ACN				Yes	No	None	MM-ML	No	MS/MS	LC-MS/MS	J. AOAC Int. 86, 412-431(2003).	
50	Carbaryl	0.155	No	94	Same batch	5	ACN				Yes	No	None	MM-ML	No	MS/MS	LC-MS/MS	J. AOAC Int. 86, 412-431(2003).	
50	Carbendazim and benomyl	1.44	No	93	Same batch	5	ACN				Yes	No	None	MM-ML	No	MS/MS	LC-MS/MS	J. AOAC Int. 86, 412-431(2003).	
50	Chlorpyrifos-methyl	0.142	No	95	Same batch	5	ACN				Yes	No	None	MM-ML	No	MS/MS	LC-MS/MS	J. AOAC Int. 86, 412-431(2003).	
50	Deltamethrin (cis)	0.0849	No	91	Same batch	5	ACN				Yes	No	None	MM-ML	No	MS/MS	LC-MS/MS	J. AOAC Int. 86, 412-431(2003).	
50	Fenitrothion	0.149	No	90	Same batch	25	AC	MeOH			No	No	GPC	MM-ML	No	MSD	GC-MS	Analyst, 109, 85-106 (1984)	
50	Fenpropimorph	2.41	No	98	Same batch	5	ACN				Yes	No	None	MM-ML	No	MS/MS	LC-MS/MS	J. AOAC Int. 86, 412-431(2003).	
50	Fluquinconazole	0.707	No	91	Same batch	5	ACN				Yes	No	None	MM-ML	No	MS/MS	LC-MS/MS	J. AOAC Int. 86, 412-431(2003).	
50	Flutriafol	2.23	No	99	Same batch	5	ACN				Yes	No	None	MM-ML	No	MS/MS	LC-MS/MS	J. AOAC Int. 86, 412-431(2003).	
50	Isoproturon	0.162	No	97	Same batch	5	ACN				Yes	No	None	MM-ML	No	MS/MS	LC-MS/MS	J. AOAC Int. 86, 412-431(2003).	

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
50	Kresoxim-methyl	0.378	No	94	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	LC-MS/MS	J. AOAC Int. 86, 412-431(2003).
50	Lambda-cyhalothrin	0.126	No	90	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	LC-MS/MS	J. AOAC Int. 86, 412-431(2003).
50	Malathion	0.102	No	90	Same batch	25	AC	MeOH			No	No	GPC	MM-ML	No	MSD	GC-MS	Analyst, 109, 85-106 (1984)	
50	Pirimiphos-methyl	0.0785	No	93	Same batch	5	ACN				Yes	No	None	MM-ML	No	MS/MS	LC-MS/MS	J. AOAC Int. 86, 412-431(2003).	
50	Spiroxamine	1.24	No	98	Same batch	5	ACN				Yes	No	None	MM-ML	No	MS/MS	LC-MS/MS	J. AOAC Int. 86, 412-431(2003).	
50	Triadimenol	1.56	No	100	Same batch	5	ACN				Yes	No	None	MM-ML	No	MS/MS	LC-MS/MS	J. AOAC Int. 86, 412-431(2003).	
50	Glyphosate	3.98	No	98	Same batch	25	MeOH	Water					SPE SAX	MM-ML	Isotop. lab.1	MSD	GC-MS	Agric. Food Chem, 1994, 42, 2751-2759 http://www.crl-pesticides.eu/library/docs/srm/meth_ClormequatMepiquat_CrlSrm.pdf	
50	Mepiquat (free cation)	0.0620	No	74	Same batch	10	MeOH				Yes			MM-ML	Isotop. lab.2		MS/MS	LC-MS/MS	
51	Azoxystrobin	0.419	No	105	Same batch	5	ACN				Yes	No	Freez.	MM-ML	TPP		MS/MS	LC-MS/MS	
51	Carbaryl	0.156	No	110	Same batch	5	ACN				Yes	No	Freez.	MM-ML	TPP		MS/MS	LC-MS/MS	
51	Carbendazim and benomyl	0.994	No	92	Same batch	5	ACN				Yes	No	Freez.	MM-ML	TPP		MS/MS	LC-MS/MS	
51	Chlorpyrifos-methyl	0.117	No	90	Same batch	5	ACN				Yes	No	Freez.	MM-ML	TPP		MS/MS	LC-MS/MS	
51	Deltamethrin (cis)	0.073	No	95	Same batch	5	ACN				Yes	No	Freez.	MM-ML	TPP	TOF	GC-TOF		
51	Fenitrothion	0.209	No	98	Same batch	5	ACN				Yes	No	Freez.	MM-ML	TPP	TOF	GC-TOF		
51	Fenpropimorph	2.03	No	97	Same batch	5	ACN				Yes	No	Freez.	MM-ML	TPP		MS/MS	LC-MS/MS	
51	Fluquinconazole	0.736	No	95	Same batch	5	ACN				Yes	No	Freez.	MM-ML	TPP		MS/MS	LC-MS/MS	
51	Flutriafol	1.58	No	95	Same batch	5	ACN				Yes	No	Freez.	MM-ML	TPP		MS/MS	LC-MS/MS	
51	Isoproturon	0.184	No	101	Same batch	5	ACN				Yes	No	Freez.	MM-ML	TPP		MS/MS	LC-MS/MS	
51	Kresoxim-methyl	0.479	No	93	Same batch	5	ACN				Yes	No	Freez.	MM-ML	TPP		MS/MS	LC-MS/MS	
51	Lambda-cyhalothrin	0.083	No	99	Same batch	5	ACN				Yes	No	Freez.	MM-ML	TPP	TOF	GC-TOF		
51	Malathion	0.114	No	99	Same batch	5	ACN				Yes	No	Freez.	MM-ML	TPP		MS/MS	LC-MS/MS	
51	Pirimiphos-methyl	0.101	No	95	Same batch	5	ACN				Yes	No	Freez.	MM-ML	TPP	TOF	GC-TOF		
51	Spiroxamine	1.29	No	108	Same batch	5	ACN				Yes	No	Freez.	MM-ML	TPP		MS/MS	LC-MS/MS	
51	-Triadimefon	0.010	No	113	Same batch	5	ACN				Yes	No	Freez.	MM-ML	TPP	TOF	GC-TOF		

LAB CODE	Pesticide	Reported result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) ACN	Extraction solvent 2) ACN	Extraction solvent 3) Water	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
51	Triadimenol	1.91	No	100	Same batch	5					Yes	No	Freez.	MM-ML	TPP	TOF		GC-TOF	
51	2,4-D (free acid)	0.351	No	103	Same batch	5	ACN				Yes	No	Freez.	MM-ML	Nicarbazin		MS/MS	LC-MS/MS	
51	2,4-D (foll. alk hydrolysis)	0.343	No	103	Same batch	5	ACN				Yes	Yes	Freez.	MM-ML	Nicarbazin		MS/MS	LC-MS/MS	
51	Glyphosate	4.25	Yes automatic	85	Same batch	5	Water				Yes	No	Freez.	PS-ML	Isotop. lab.1		ITQ	LC-ITQ	
51	Mepiquat (free cation)	0.108	Yes automatic	108	Same batch	5	MeOH				Yes	No	Freez.	PS-ML	Isotop. lab.1		ITQ	LC-ITQ	
52	Azoxystrobin	0.135	No	108	Same batch	50	AC	CH2Cl			Yes	No	SPE Florisil	MM-SL	No	ECD		Two columns Luke	
52	Chlorpyrifos-methyl	0.096	No	92	Same batch	50	AC	CH2Cl			Yes	No	SPE Florisil	MM-SL	No	ECD		Two columns Luke	
52	Deltamethrin (cis)	0.055	No	83	Same batch	50	AC	CH2Cl			Yes	No	SPE Florisil	MM-SL	No	ECD		Two columns Luke	
52	Fenitrothion	0.183	No	92	Same batch	50	AC	CH2Cl			Yes	No	SPE Florisil	MM-SL	No	ECD		Two columns Luke	
52	Fenpropimorph	2.36	No	97	Same batch	50	AC	CH2Cl			Yes	No	SPE Florisil	MM-SL	No	NPD		Two columns Luke	
52	Fluquinconazole	0.488	No	96	Same batch	50	AC	CH2Cl			Yes	No	SPE Florisil	MM-SL	No	ECD		Two columns Luke	
52	Kresoxim-methyl	0.320	No	95	Same batch	50	AC	CH2Cl			Yes	No	SPE Florisil	MM-SL	No	ECD		Two columns Luke	
52	Lambda-cyhalothrin	0.086	No	93	Same batch	50	AC	CH2Cl			Yes	No	SPE Florisil	MM-SL	No	ECD		Two columns Luke	
52	Malathion	0.078	No	70	Same batch	50	AC	CH2Cl			Yes	No	SPE Florisil	MM-SL	No	ECD		Two columns Luke	
52	Pirimiphos-methyl	0.072	No	70	Same batch	50	AC	CH2Cl			Yes	No	SPE Florisil	MM-SL	No	NPD		Two columns Luke	
53	Azoxystrobin	0.203	Yes	73.9	Other	10	AC	HEX			Yes	No	SPE	MM-SL		ECD		Two columns internal	
53	Chlorpyrifos-methyl	0.107	Yes	98.0	Other	10	AC	HEX			Yes	No	SPE	MM-SL		ECD		Two columns internal	
53	Deltamethrin (cis)	0.054	Yes	97.5	Other	10	AC	HEX			Yes	No	SPE	MM-SL		ECD		Two columns internal	
53	Fenitrothion	0.168	Yes	101.6	Other	10	AC	HEX			Yes	No	SPE	MM-SL		ECD		Two columns internal	
53	Fenpropimorph	2.383	Yes	96.7	Other	10	AC	HEX			Yes	No	SPE	MM-SL		NPD		Two columns internal	
53	Fluquinconazole	0.693	Yes	95.2	Other	10	AC	HEX			Yes	No	SPE	MM-SL		ECD		Two columns internal	
53	Kresoxim-methyl	0.393	Yes	100.4	Other	10	AC	HEX			Yes	No	SPE	MM-SL		ECD		Two columns internal	
53	Lambda-cyhalothrin	0.096	Yes	78.8	Other	10	AC	HEX			Yes	No	SPE	MM-SL		ECD		Two columns internal	
53	Malathion	0.088	Yes	100.2	Other	10	AC	HEX			Yes	No	SPE	MM-SL		NPD		Two columns internal	
53	Pirimiphos-methyl	0.065	Yes	92.6	Other	10	AC	HEX			Yes	No	SPE	MM-SL		NPD		Two columns internal	

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
53	Triadimenol	1.124	Yes	90.8	Other	10	AC	CH2Cl	HEX		Yes	No	SPE	MM-ML		NPD		Two columns internal	
54	Carbaryl	0.0672	No	73.20	Same batch	50	ACN	CH2Cl	HEX		Yes	No	SPE	St. Ad.	No		Fluor.	GC-MS	531.1 REV. 3.1.1995 National Exposure Research Laboratory , US EPA , Anal.Meth.f.Pest.Res.in Foodst.NL 6th Ed. 1996
54	Chlorpyrifos-methyl	0.117	No	78.00	Same batch	15	AC	CH2Cl	PE		No	No	None	St. Ad.	No	ITD		GC-MS	
55	Azoxystrobin	0.33	No	98	St. Ad.	15	AC	CH2Cl	HEX		Yes	No	GPC	MM-ML	No	ECD		GC-MS/MS	
55	Carbaryl	0.15	No	93	St. Ad.	5	ACN				Yes	No	SPE	MM-ML	No		MS/MS	GC-MS/MS	Quecher
55	Carbendazim and benomyl	1.85	No	95	St. Ad.	5	ACN				Yes	No	SPE	MM-ML	No		MS/MS		Quecher
55	Chlorpyrifos-methyl	0.12	No	87	St. Ad.	15	AC	CH2Cl	HEX		Yes	No	GPC	MM-ML	No	FPD		GC-MS/MS	
55	Deltamethrin (cis)	0.054	No	98	St. Ad.	15	AC	CH2Cl	HEX		Yes	No	GPC	MM-ML	No	ECD		GC-MS/MS	
55	Fenitrothion	0.19	No	88	St. Ad.	15	AC	CH2Cl	HEX		Yes	No	GPC	MM-ML	No	FPD		GC-MS/MS	
55	Fenpropimorph	1.95	No	94	St. Ad.	5	ACN				Yes	No	SPE	MM-ML	No		MS/MS	GC-MS/MS	Quecher
55	Fluquinconazole	0.73	No	85	St. Ad.	5	ACN				Yes	No	SPE	MM-ML	No		MS/MS	GC-MS/MS	Quecher
55	Flutriafol	2.06	No	90	St. Ad.	5	ACN				Yes	No	SPE	MM-ML	No		MS/MS	GC-MS/MS	Quecher
55	Isoproturon	0.14	No	92	St. Ad.	5	ACN				Yes	No	SPE	MM-ML	No		MS/MS		Quecher
55	Kresoxim-methyl	0.34	No	95	St. Ad.	15	AC	CH2Cl	HEX		Yes	No	GPC	MM-ML	No	ECD		GC-MS/MS	
55	Lambda-cyhalothrin	0.085	No	96	St. Ad.	15	AC	CH2Cl	HEX		Yes	No	GPC	MM-ML	No	ECD		GC-MS/MS	
55	Malathion	0.11	No	89	St. Ad.	15	AC	CH2Cl	HEX		Yes	No	GPC	MM-ML	No	FPD		GC-MS/MS	
55	Pirimiphos-methyl	0.082	No	87	St. Ad.	15	AC	CH2Cl	HEX		Yes	No	GPC	MM-ML	No	FPD		GC-MS/MS	
55	Spiroxamine	0.87	No	93	St. Ad.	15	AC	CH2Cl	HEX		Yes	No	GPC	MM-ML	No	MSD		LC-MS/MS	
55	Triadimenol	1.52	No	92	St. Ad.	15	AC	CH2Cl	HEX		Yes	No	GPC	MM-ML	No	MSD		GC-MS/MS	
56	Azoxystrobin	0.352	No	97.8	Same batch	5	ACN				Yes	No	DSPE	PS-ML	Tris-dichlor-isopropyl-phosph.	ECD	LC-MS/MS	EN15662:2008	
56	Carbaryl	0.151	No	87.0	Same batch	5	ACN				Yes	No	DSPE	PS-ML	No		MS/MS	None	EN15662:2008
56	Carbendazim and benomyl	1.14	No	70.2	Same batch	5	ACN				Yes	No	DSPE	PS-ML	No		MS/MS	None	EN15662:2008
56	Chlorpyrifos-methyl	0.133	No	95.8	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Tris-dichlor-isopropyl-phosph.	FPD		GC-ECD	EN15662:2008
56	Fenitrothion	0.207	No	95.0	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Tris-dichlor-isopropyl-phosph.	FPD		GC-ECD	EN15662:2008
56	Fenpropimorph	1.96	No	82.5	Same batch	5	ACN				Yes	No	DSPE	PS-ML	No		MS/MS	GC-NPD	EN15662:2008

LAB CODE	Pesticide	Reported result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) ACN	Extraction solvent 2) ACN	Extraction solvent 3) ACN	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
56	Fluquinconazole	0.775	No	87.7	Same batch	5	ACN				Yes	No	DSPE	PS-ML	No	MS/MS	NPD ECD	EN15662:2008	
56	Flutriafol	2.08	No	90.3	Same batch	5	ACN				Yes	No	DSPE	PS-ML	No	MS/MS	GC-NPD	EN15662:2008	
56	Isoproturon	0.144	No	85.9	Same batch	5	ACN				Yes	No	DSPE	PS-ML	No	MS/MS	None	EN15662:2008	
56	Kresoxim-methyl	0.476	No	97.4	Same batch	5	ACN				Yes	No	DSPE	PS-ML	Tris-dichlor-isopropyl-phosph.	ECD	None	EN15662:2008	
56	Lambda-cyhalothrin	0.0590	No	81.6	Same batch	5	ACN				Yes	No	DSPE	PS-ML	Tris-dichlor-isopropyl-phosph.	ECD	None	EN15662:2008	
56	Malathion	0.111	No	92.0	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Tris-dichlor-isopropyl-phosph.	FPD	GC-ECD	EN15662:2008	
56	Pirimiphos-methyl	0.0986	No	103.2	Same batch	5	ACN				Yes	No	DSPE	MM-ML	Tris-dichlor-isopropyl-phosph.	FPD	GC-NPD	EN15662:2008	
56	Spiroxamine	1.45	No	103.0	Same batch	5	ACN				Yes	No	DSPE	PS-ML	No	MS/MS	None	EN15662:2008	
56	Triadimenol	1.84	No	86.8	Same batch	5	ACN				Yes	No	DSPE	PS-ML	No	MS/MS	GC-NPD	EN15662:2008	
56	2,4-D (free acid)	0.334	No	81.0	Same batch	5	ACN				Yes	No	DSPE	PS-ML	No	MS/MS	None	EN15662:2008	
56	2,4-D (foll. alk hydrolysis)	0.346	No	79.0	Same batch	5	ACN				Yes	No	DSPE	PS-ML	No	MS/MS	None	EN15662:2008	
56	Mepiquat (free cation)	0.126	No	102.1	Same batch	10	MeOH				Yes	No	None	PS-ML	No	MS/MS	None	EN15055:2006	
57	Azoxystrobin	0.324	No	110	Same batch	5	ACN				Yes	No	DSPE PSA	MM-SL	No	MS/MS	GC-MS/MS	QuEChERS-GC	
57	Carbaryl	0.161	No	107	Same batch	5	ACN				Yes	No	DSPE PSA	MM-SL	No	MS/MS	LC-MS/MS	QuEChERS-LC	
57	Carbendazim and benomyl	1.36	No	84	Same batch	5	ACN				Yes	No	DSPE PSA	MM-SL	No	MS/MS	LC-MS/MS	QuEChERS-LC	
57	Chlorpyrifos-methyl	0.108	No	103	Same batch	5	ACN				Yes	No	DSPE PSA	MM-SL	No	MS/MS	GC-MS/MS	QuEChERS-GC	
57	Deltamethrin (cis)	0.061	No	107	Same batch	5	ACN				Yes	No	DSPE PSA	MM-SL	No	MS/MS	GC-MS/MS	QuEChERS-GC	
57	Fenitrothion	0.169	No	103	Same batch	5	ACN				Yes	No	DSPE PSA	MM-SL	No	MS/MS	GC-MS/MS	QuEChERS-GC	
57	Fenpropimorph	1.96	No	85	Same batch	5	ACN				Yes	No	DSPE PSA	MM-SL	No	MS/MS	LC-MS/MS	QuEChERS-LC	
57	Fluquinconazole	0.930	No	106	Same batch	5	ACN				Yes	No	DSPE PSA	MM-SL	No	MS/MS	GC-MS/MS	QuEChERS-GC	
57	Flutriafol	1.69	No	103	Same batch	5	ACN				Yes	No	DSPE PSA	MM-SL	No	MS/MS	GC-MS/MS	QuEChERS-GC	
57	Isoproturon	0.156	No	96	Same batch	5	ACN				Yes	No	DSPE PSA	MM-SL	No	MS/MS	LC-MS/MS	QuEChERS-LC	
57	Kresoxim-methyl	0.386	No	104	Same batch	5	ACN				Yes	No	DSPE PSA	MM-SL	No	MS/MS	GC-MS/MS	QuEChERS-GC	
57	Lambda-cyhalothrin	0.105	No	103	Same batch	5	ACN				Yes	No	DSPE PSA	MM-SL	No	MS/MS	GC-MS/MS	QuEChERS-GC	
57	Malathion	0.105	No	117	Same batch	5	ACN				Yes	No	DSPE PSA	MM-SL	No	MS/MS	GC-MS/MS	QuEChERS-GC	

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method	
57	Pirimiphos-methyl	0.083	No	105	Same batch	5	ACN				Yes	No	DSPE PSA	MM-SL	No	MS/MS	GC-MS/MS	QuEChERS-GC		
57	Spiroxamine	1.13	No	102	Same batch	5	ACN				Yes	No	DSPE PSA	MM-SL	No	MS/MS	GC-MS/MS	QuEChERS-GC		
57	Triadimenol	1.49	No	97	Same batch	5	ACN				Yes	No	DSPE PSA	MM-SL	No	MS/MS	LC-MS/MS	QuEChERS-LC		
57	2,4-D (foll. alk hydrolysis)	0.400	No	100	Same batch	2	ACN				Yes	Yes	Freez.	MM-SL		MS/MS	LC-MS/MS	QuEChERS-LC, alkalische Hydrolyse		
57	Mepiquat (free cation)	0.088	No	71	Same batch	5	MeOH				Yes	No	None	MM-SL		MS/MS	LC-MS/MS	CRL-SRM-Mimi-Methode		
58	Azoxystrobin	9.92	No			5	ACN				Yes	No	DSPE	PS-SL	TPP		LC-MS/MS	15662		
58	Carbaryl	0.118	No			5	ACN				Yes	No	DSPE	PS-SL	TPP		LC-MS/MS	15662		
58	Chlorpyrifos-methyl	0.106	No			5	ACN				Yes	No	DSPE	PS-SL	No	ECD	None	15662		
58	Deltamethrin (cis)	0.058	No			5	ACN				Yes	No	DSPE	PS-SL	No	ECD	None	15662		
58	Lambda-cyhalothrin	0.041	No			5	ACN				Yes	No	DSPE	PS-SL	No	ECD	None	15662		
58	Malathion	0.15	No			5	ACN				Yes	No	DSPE	PS-SL	TPP		LC-MS/MS	15662		
58	Pirimiphos-methyl	0.048	No			5	ACN				Yes	No	DSPE	PS-SL	No	NPD	None	15662		
58	Triadimenol	0.79	No			5	ACN				Yes	No	DSPE	PS-SL	TPP		LC-MS/MS	15662		
59	Chlorpyrifos-methyl	0.12	No	93		20	AC	Other			No	No	SPE	PS-SL		ECD	Two columns Luke			
59	Fenitrothion	0.18	No	91		20	AC	Other			No	No	SPE	PS-SL		NPD	Two columns Luke			
59	Fenpropimorph	2.0	No	89		20	AC	Other			No	No	SPE	PS-SL		NPD	Two columns Luke			
59	Fluquinconazole	0.7	No	92		20	AC	Other			No	No	SPE	PS-SL		ECD	Two columns Luke			
59	Kresoxim-methyl	0.32	No	95		20	AC	Other			No	No	SPE	PS-SL		ECD	Two columns Luke			
59	Lambda-cyhalothrin	0.09	No	91		20	AC	Other			No	No	SPE	PS-SL		ECD	Two columns Luke			
59	Malathion	0.08	No	80		20	AC	Other			No	No	SPE	PS-SL		ECD	Two columns Luke			
59	Pirimiphos-methyl	0.06	No	94		20	AC	Other			No	No	SPE	PS-SL		NPD	Two columns Luke			
60	Azoxystrobin	0.217	No	90	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP		MS/MS	LC-MS/MS	Quechers	
60	Carbaryl	0.197	No	72	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP		MS/MS	LC-MS/MS	Quechers	
60	Carbendazim and benomyl	1.33	No	64	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP		MS/MS	LC-MS/MS	Quechers	
60	Chlorpyrifos-methyl	0.125	No	82	Same batch	5	AC	CH2Cl	PE		Yes	No	None	MM-ML	TPP	ITD	GC-MS/MS	Internal method		

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) ACN	Extraction solvent 2) CH2Cl	Extraction solvent 3) PE	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method	
60	Deltamethrin (cis)	0.057	No	86	Same batch	5	AC	CH2Cl	PE		Yes	No	None	MM-ML	TPP	ITD	GC-MS/MS	Internal method		
60	Fenitrothion	0.189	No	77	Same batch	5	AC	CH2Cl	PE		Yes	No	None	MM-ML	TPP	ITD	GC-MS/MS	Internal method		
60	Kresoxim-methyl	0.405	No	83	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP		MS/MS	LC-MS/MS	Quechers	
60	Lambda-cyhalothrin	0.072	No	77	Same batch	5	AC	CH2Cl	PE		Yes	No	None	MM-ML	TPP	ITD	GC-MS/MS	Internal method		
60	Malathion	0.130	No	59	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP		MS/MS	LC-MS/MS	Quechers	
60	Pirimiphos-methyl	0.075	No	82	Same batch	5	AC	CH2Cl	PE		Yes	No	None	MM-ML	TPP	ITD	GC-MS/MS	Internal method		
60	Triadimenol	1.61	No	106	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP		MS/MS	LC-MS/MS	Quechers	
61	Azoxystrobin	0.470	No	118	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	MS/MS	GC-MS	Quechers	
61	Carbaryl	0.142	No	104	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	MS/MS	GC-MS	Quechers	
61	Carbendazim and benomyl	1.46	No	89	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP		MS/MS	LC-MS/MS	Quechers	
61	Chlorpyrifos-methyl	0.146	No	106	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	GC-MS	Quechers		
61	Deltamethrin (cis)	<0.020	No	81	Validation	5	ACN				Yes	No	DSPE	MM-ML	TPP	ECD	GC-MS	Quechers		
61	Fenitrothion	0.186	No	83	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	GC-MS	Quechers		
61	Fenpropimorph	2.72	No	121	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	MS/MS	GC-MS	Quechers	
61	Fluquinconazole	0.457	No	78	Validation	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	GC-MS	Quechers		
61	Flutriafol	2.60	No	98	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	MS/MS	GC-MS	Quechers	
61	Isoproturon	0.188	No	111	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP		MS/MS	LC-MS/MS	Quechers	
61	Kresoxim-methyl	0.448	No	92	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	MS/MS	GC-MS	Quechers	
61	Lambda-cyhalothrin	0.062	No	78	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	GC-MS	Quechers		
61	Malathion	0.111	No	99	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	MS/MS	GC-MS	Quechers	
61	Pirimiphos-methyl	0.107	No	113	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	MS/MS	GC-MS	Quechers	
61	Spiroxamine	1.64	No	111	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	MS/MS	GC-MS	Quechers	
61	Triadimenol	1.79	No	107	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	MS/MS	GC-MS	Quechers	
61	2,4-D (free acid)	0.349	No	92	Same batch	5	ACN				Yes	No	None	MM-ML	Nicarbazin		MS/MS	LC-MS/MS	Quechers without DSPE	
61	2,4-D (foll. alk hydrolysis)	0.358	No	89	Same batch	5	ACN				Yes	Yes	None	MM-ML	Nicarbazin		MS/MS	LC-MS/MS	Quechers and alk. hydrolysis before extraction	

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
61	Mepiquat (free cation)	0.109	No	118	Same batch	10	MeOH				Yes	No	None	PS-ML	Isotop. lab.1		MS/MS	LC-MS/MS	Å§ 64 method L00.00-76
62	Azoxystrobin	0.397	No	106	Same batch	20	AC	Cy-He	EtOAc		Yes	No	GPC SPE	MM-ML	No	MSD		GC-MS	
62	Carbaryl	0.176	No	90	Same batch	20	AC	Cy-He	EtOAc		Yes	No	GPC SPE	MM-ML	No	MSD		GC-MS	
62	Carbendazim and benomyl	1.32	No	93	Same batch	20	AC	Cy-He	EtOAc		Yes	No	GPC	MM-ML	No	MS/MS	LC-MS/MS		
62	Chlorpyrifos-methyl	0.162	No	86	Same batch	20	AC	Cy-He	EtOAc		Yes	No	GPC	MM-ML	No	MS/MS	LC-MS/MS		
62	Deltamethrin (cis)	0.083	No	85	Same batch	20	AC	Cy-He	EtOAc		Yes	No	GPC SPE	MM-ML	No	MSD		GC-MS	
62	Fenitrothion	0.273	No	97	Same batch	20	AC	Cy-He	EtOAc		Yes	No	GPC SPE	MM-ML	No	MSD		GC-MS	
62	Fenpropimorph	2.46	No	91	Same batch	20	AC	Cy-He	EtOAc		Yes	No	GPC SPE	MM-ML	No	MSD		GC-MS	
62	Fluquinconazole	0.853	No	108	Same batch	20	AC	Cy-He	EtOAc		Yes	No	GPC SPE	MM-ML	No	MSD		GC-MS	
62	Flutriafol	2.45	No	108	Same batch	20	AC	Cy-He	EtOAc		Yes	No	GPC SPE	MM-ML	No	MSD		GC-MS	
62	Isoproturon	0.110	No	101	Same batch	20	AC	Cy-He	EtOAc		Yes	No	GPC	MM-ML	No	MS/MS	LC-MS/MS		
62	Kresoxim-methyl	0.493	No	99	Same batch	20	AC	Cy-He	EtOAc		Yes	No	GPC SPE	MM-ML	No	MSD		GC-MS	
62	Lambda-cyhalothrin	0.114	No	97	Same batch	20	AC	Cy-He	EtOAc		Yes	No	GPC SPE	MM-ML	No	MSD		GC-MS	
62	Malathion	0.140	No	103	Same batch	20	AC	Cy-He	EtOAc		Yes	No	GPC SPE	MM-ML	No	MSD		GC-MS	
62	Pirimiphos-methyl	0.128	No	104	Same batch	20	AC	Cy-He	EtOAc		Yes	No	GPC SPE	MM-ML	No	MSD		GC-MS	
62	Spiroxamine	0.520	No	40	Same batch	20	AC	Cy-He	EtOAc		Yes	No	GPC SPE	MM-ML	No	MSD		GC-MS	
62	-Triadimefon	0.020	No	101	Same batch	20	AC	Cy-He	EtOAc		Yes	No	GPC SPE	MM-ML	No	MSD		GC-MS	
62	Triadimenol	1.69	No	82	Same batch	20	AC	Cy-He	EtOAc		Yes	No	GPC	MM-ML	No	MS/MS	LC-MS/MS		
63	Azoxystrobin	0.245	No	92		5	ACN				Yes	No	DSPE	MM-ML	No	MSD		GC-MS	
63	Carbaryl	0.155	No	60		5	ACN				Yes	No	DSPE	MM-ML	No	MSD		GC-MS	
63	Carbendazim and benomyl	1.48	No	90		5	ACN				Yes	No	DSPE	PS-ML	No		Diod. Ar.		
63	Chlorpyrifos-methyl	0.131	No	83		5	ACN				Yes	No	DSPE	MM-ML	No	MSD		GC-MS	
63	Deltamethrin (cis)	0.070	No	92		5	ACN				Yes	No	DSPE	MM-ML	No	MSD		GC-MS	
63	Fenitrothion	0.179	No	90		5	ACN				Yes	No	DSPE	MM-ML	No	MSD		GC-MS	
63	Kresoxim-methyl	0.387	No	92		5	ACN				Yes	No	DSPE	MM-ML	No	MSD		GC-MS	

LAB CODE	Pesticide	Reported result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) ACN	Extraction solvent 2) ACN	Extraction solvent 3) ACN	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
63	Lambda-cyhalothrin	0.076	No	92		5					Yes	No	DSPE	MM-ML	No	MSD	GC-MS		
63	Malathion	0.081	No	92		5	ACN				Yes	No	DSPE	MM-ML	No	MSD	GC-MS		
63	Pirimiphos-methyl	0.077	No	92		5	ACN				Yes	No	DSPE	MM-ML	No	MSD	GC-MS		
63	-Triadimefon	0.014	No	92		5	ACN				Yes	No	DSPE	MM-ML	No	MSD	GC-MS		
63	Triadimenol	1.60	No	92		5	ACN				Yes	No	DSPE	MM-ML	No	MSD	GC-MS		
64	Azoxystrobin	0.356	No	88	Same batch	5	ACN				Yes	No	None	MM-ML	quinalphos	MS/MS	LC-MS/MS		
64	Carbaryl	0.142	No	86	Same batch	5	ACN				Yes	No	None	MM-ML	quinalphos	MS/MS	LC-MS/MS		
64	Carbendazim and benomyl	1.42	No	80	Same batch	5	ACN				Yes	No	None	MM-ML	quinalphos	MS/MS	LC-MS/MS		
64	Chlorpyrifos-methyl	0.133	No	108	Same batch	5	ACN				Yes	No	None	MM-ML	ethoprophos	ITD	LC-MS/MS		
64	Deltamethrin (cis)	0.091	No	75	Same batch	5	ACN				Yes	No	None	MM-ML	bromofos-ethyl	MSD	GC-MS		
64	Fenitrothion	0.189	No	99	Same batch	5	ACN				Yes	No	None	MM-ML	ethoprophos	ITD	GC-MS		
64	Fenpropimorph	2.35	No	86	Same batch	5	ACN				Yes	No	None	MM-ML	quinalphos	MS/MS	LC-MS/MS		
64	Fluquinconazole	0.741	No	95	Same batch	5	ACN				Yes	No	None	MM-ML	quinalphos	MS/MS	LC-MS/MS		
64	Flutriafol	3.26	No	88	Same batch	5	ACN				Yes	No	None	MM-ML	quinalphos	MS/MS	LC-MS/MS		
64	Isoproturon	0.146	No	87	Same batch	5	ACN				Yes	No	None	MM-ML	quinalphos	MS/MS	LC-MS/MS		
64	Kresoxim-methyl	0.432	No	88	Same batch	5	ACN				Yes	No	None	MM-ML	quinalphos	MS/MS	LC-MS/MS		
64	Lambda-cyhalothrin	0.053	No	89	Same batch	5	ACN				Yes	No	None	MM-ML	bromofos-ethyl	MSD	GC-MS		
64	Malathion	0.112	No	88	Same batch	5	ACN				Yes	No	None	MM-ML	quinalphos	MS/MS	LC-MS/MS		
64	Pirimiphos-methyl	0.075	No	88	Same batch	5	ACN				Yes	No	None	MM-ML	ethoprophos	ITD	LC-MS/MS		
64	Spiroxamine	1.37	No	86	Same batch	5	ACN				Yes	No	None	MM-ML	quinalphos	MS/MS	LC-MS/MS		
64	-Triadimefon	0.010	No	86	Same batch	5	ACN				Yes	No	None	MM-ML	quinalphos	MS/MS	LC-MS/MS		
64	Triadimenol	2.86	No	93	Same batch	5	ACN				Yes	No	None	MM-ML	quinalphos	MS/MS	LC-MS/MS		
64	2,4-D (free acid)	0.350	No	73	Same batch	5	ACN				Yes	No	None	MM-ML	No	MS/MS	LC-MS/MS		
64	Mepiquat (free cation)	0.094	No	92	Same batch	25	MeOH				No	No	None	MM-ML	Isotop. lab.2	MS/MS	LC-MS/MS		
65	Azoxystrobin	0.367	No	83	Validation	5	ACN				Yes		DSPE PSA	MM-ML	TPP	MS/MS	GC-MS/MS	Quechers	

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method	
65	Carbaryl	0.160	No	92	Same batch	5	ACN				Yes		DSPE PSA	PS-ML	No		MS/MS	LC-MS/MS	Quechers	
65	Carbendazim and benomyl	1.31	No	86	Same batch	5	ACN				Yes		DSPE PSA	PS-ML	No		MS/MS	LC-MS/MS	Quechers	
65	Chlorpyrifos-methyl	0.178	No	94	Validation	5	ACN				Yes		DSPE PSA	MM-ML	TPP	MS/MS	GC-MS/MS	Quechers		
65	Deltamethrin (cis)	0.0720	No	80	Same batch	5	ACN				Yes		DSPE PSA	PS-ML	No		MS/MS	LC-MS/MS	Quechers	
65	Fenitrothion	0.278	No	116	Validation	5	ACN				Yes		DSPE PSA	MM-ML	TPP	MS/MS	GC-MS/MS	Quechers		
65	Fenpropimorph	2.07	No	96	Same batch	5	ACN				Yes		DSPE PSA	PS-ML	No		MS/MS	LC-MS/MS	Quechers	
65	Fluquinconazole	0.803	No	80	Same batch	5	ACN				Yes		DSPE PSA	PS-ML	No		MS/MS	LC-MS/MS	Quechers	
65	Flutriafol	2.30	No	98	Same batch	5	ACN				Yes		DSPE PSA	PS-ML	No		MS/MS	LC-MS/MS	Quechers	
65	Isoproturon	0.203	No	98	Same batch	5	ACN				Yes		DSPE PSA	PS-ML	No		MS/MS	LC-MS/MS	Quechers	
65	Kresoxim-methyl	0.456	No	96	Validation	5	ACN				Yes		DSPE PSA	MM-ML	TPP	MS/MS	GC-MS/MS	Quechers		
65	Lambda-cyhalothrin	0.0528	No	89	Validation	5	ACN				Yes		DSPE PSA	MM-ML	TPP	MS/MS	GC-MS/MS	Quechers		
65	Malathion	0.131	No	94	Same batch	5	ACN				Yes		DSPE PSA	PS-ML	No		MS/MS	LC-MS/MS	Quechers	
65	Pirimiphos-methyl	0.102	No	115	Validation	5	ACN				Yes		DSPE PSA	MM-ML	TPP	MS/MS	GC-MS/MS	Quechers		
65	Spiroxamine	1.56	No	114	Same batch	5	ACN				Yes		DSPE PSA	PS-ML	No		MS/MS	LC-MS/MS	Quechers	
65	-Triadimefon	0.0100	No	94	Same batch	5	ACN				Yes		DSPE PSA	PS-ML	No		MS/MS	LC-MS/MS	Quechers	
65	Triadimenol	1.68	No	100	Same batch	5	ACN				Yes		DSPE PSA	PS-ML	No		MS/MS	LC-MS/MS	Quechers	
65	2,4-D (free acid)	0.353	No	88	Same batch	5	ACN				Yes	No	Freez.	MM-ML	No		MS/MS	LC-MS/MS	Quechers	
65	2,4-D (foll. alk hydrolysis)	0.369	No	88	Same batch	5	ACN				Yes	Yes	Freez.	MM-ML	No		MS/MS	LC-MS/MS	Quechers	
65	Glyphosate	4.20	No	100	Same batch	3	Water				No		L/I part.	PS-ML	Isotop. lab.1		MS/MS	LC-MS/MS	In-house	
65	Mepiquat (free cation)	0.097	No	90	Same batch	25	MeOH				Yes		None	PS-ML	Isotop. lab.1		MS/MS	LC-MS/MS	EN 15055	
66	Azoxystrobin	0.64	No	120		0														
66	Carbaryl	1.74	No	110		0														
66	Deltamethrin (cis)	0.07	No	77		0														
66	Fenitrothion	0.18	No	115		0														
66	Lambda-cyhalothrin	0.05	No	73		0														

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) ACN	Extraction solvent 2) ACN	Extraction solvent 3) ACN	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
66	Malathion	0.11	No	110		0													
66	Pirimiphos-methyl	0.06	No	120		0													
66	Triadimenol	1.60	No	80		0													
67	Azoxystrobin	0.390	No	91	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	GC-MS	UNI EN 15662:2009	
67	Carbaryl	0.166	No	95	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	GC-MS	UNI EN 15662:2009	
67	Chlorpyrifos-methyl	0.130	No	87	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	GC-MS	UNI EN 15662:2009	
67	Deltamethrin (cis)	0.095	No	81	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	GC-MS	UNI EN 15662:2009	
67	Fenitrothion	0.208	No	103	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	GC-MS	UNI EN 15662:2009	
67	Kresoxim-methyl	0.454	No	98	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	GC-MS	UNI EN 15662:2009	
67	Lambda-cyhalothrin	0.061	No	75	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	GC-MS	UNI EN 15662:2009	
67	Malathion	0.104	No	78	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	GC-MS	UNI EN 15662:2009	
67	Pirimiphos-methyl	0.078	No	81	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	GC-MS	UNI EN 15662:2009	
67	Triadimenol	1.29	No	80	Same batch	5	ACN				Yes	No	DSPE	MM-ML	TPP	MSD	GC-MS	UNI EN 15662:2009	
68	Azoxystrobin	0.251	No	83	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No		MS/MS	LC-MS/MS	
68	Carbaryl	0.101	No	93	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No		MS/MS	LC-MS/MS	
68	Chlorpyrifos-methyl	0.107	No	81	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No	MSD	GC-MS		
68	Deltamethrin (cis)	0.050	No	75	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No	MSD	GC-MS		
68	Fenitrothion	0.169	No	114	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No	MSD	GC-MS		
68	Kresoxim-methyl	0.352	No	89	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No	MSD	GC-MS		
68	Lambda-cyhalothrin	0.059	No	105	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No	MSD	GC-MS		
68	Malathion	0.105	No	90	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No	MSD	GC-MS		
68	Pirimiphos-methyl	0.074	No	86	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No	MSD	GC-MS		
68	Spiroxamine	0.224	No	51	Same batch	5	ACN				Yes	No	DSPE PSA	MM-ML	No		MS/MS	LC-MS/MS	
69	Azoxystrobin	0.383	No	93	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	LC-MS/MS	
69	Carbaryl	0.144	No	85	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	LC-MS/MS	

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
69	Carbendazim and benomyl	1.09	No	80	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	LC-MS/MS	
69	Chlorpyrifos-methyl	0.134	No	85	Same batch	5	ACN				Yes	No	None	PS-ML	No	FPD		GC-MS	
69	Deltamethrin (cis)	0.073	No	85	Same batch	5	ACN				Yes	No	None	MM-ML	No	ECD		GC-MS	
69	Fenitrothion	0.215	No	87	Same batch	5	ACN				Yes	No	None	PS-ML	No	FPD		GC-MS	
69	Fenpropimorph	2.33	No	103	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	LC-MS/MS	
69	Fluquinconazole	0.753	No	105	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	LC-MS/MS	
69	Flutriafol	2.05	No	113	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	LC-MS/MS	
69	Isoproturon	0.140	No	101	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	LC-MS/MS	
69	Kresoxim-methyl	0.373	No	83	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	LC-MS/MS	
69	Lambda-cyhalothrin	0.102	No	92	Same batch	5	ACN				Yes	No	None	MM-ML	No	ECD		GC-MS	
69	Malathion	0.114	No	76	Same batch	5	ACN				Yes	No	None	PS-ML	No	FPD		GC-MS	
69	Pirimiphos-methyl	0.077	No	75	Same batch	5	ACN				Yes	No	None	PS-ML	No	FPD		GC-MS	
69	Spiroxamine	1.12	No	104	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	LC-MS/MS	
69	-Triadimefon	0.011	No	105	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	LC-MS/MS	
69	Triadimenol	1.69	No	103	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	LC-MS/MS	
69	2,4-D (free acid)	0.327	No	105	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	LC-MS/MS	
70	Azoxystrobin	0.076	No	91	Other	6	ACN				Yes	No	DSPE PSA	MM-ML	No	MSD	None	Waters application note	
70	Carbaryl	0.100	No	100	Other	6	ACN				Yes	No	DSPE PSA	MM-ML	No	MSD	None	Waters application note	
70	Chlorpyrifos-methyl	0.129	No	109	Other	6	ACN				Yes	No	DSPE PSA	MM-ML	No	MSD	None	Waters application note	
70	Fenitrothion	0.219	No	106	Other	6	ACN				Yes	No	DSPE PSA	MM-ML	No	MSD	None	Waters application note	
70	Fenpropimorph	1.70	No	83	Other	6	ACN				Yes	No	DSPE PSA	MM-ML	No	MSD	None	Waters application note	
70	Fluquinconazole	0.522	No	118	Other	6	ACN				Yes	No	DSPE PSA	MM-ML	No	MSD	None	Waters application note	
70	Kresoxim-methyl	0.389	No	107	Other	6	ACN				Yes	No	DSPE PSA	MM-ML	No	MSD	None	Waters application note	
70	Malathion	0.097	No	95	Other	6	ACN				Yes	No	DSPE PSA	MM-ML	No	MSD	None	Waters application note	
70	Pirimiphos-methyl	0.100	No	91	Other	6	ACN				Yes	No	DSPE PSA	MM-ML	No	MSD	None	Waters application note	

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) EtOAc	Extraction solvent 2) EtOAc	Extraction solvent 3) EtOAc	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
70	Triadimenol	1.004	No	102	Other	6	ACN				Yes	No	DSPE PSA	MM-ML	No	MSD		None	Waters application note
71	Azoxystrobin	0.179	No	74	Same batch	10	EtOAc				Yes	No	GPC	MM-ML	No	ECD	MS/MS	GC-MS	Inhouse Validated
71	Carbaryl	0.107	No	83	Same batch	10	EtOAc				Yes	No	GPC	MM-ML	No	ITD	MS/MS	GC-MS	Inhouse Validated
71	Carbendazim and benomyl	0.998	No	74	Same batch	10	EtOAc				Yes	No	None	MM-ML	No	MS/MS	LC-MS/MS	Inhouse Validated	
71	Chlorpyrifos-methyl	0.0882	No	90	Same batch	10	EtOAc				Yes	No	GPC	MM-ML	No	FPD	MS/MS	GC-MS	Inhouse Validated
71	Deltamethrin (cis)	0.0470	No	88	Same batch	10	EtOAc				Yes	No	None	MM-ML	No	ECD		GC-MS	Inhouse Validated
71	Fenitrothion	0.133	No	86	Same batch	10	EtOAc				Yes	No	GPC	MM-ML	No	FPD		GC-MS	Inhouse Validated
71	Fenpropimorph	0.930	No	60	Same batch	10	EtOAc				Yes	No	None	MM-ML	No	MS/MS	LC-MS/MS	Inhouse Validated	
71	Fluquinconazole	0.494	No	79	Same batch	10	EtOAc				Yes	No	None	MM-ML	No	ECD	MS/MS	LC-MS/MS	Inhouse Validated
71	Flutriafol	1.38	No	85	Same batch	10	EtOAc				Yes	No	None	MM-ML	No	MS/MS	LC-MS/MS	Inhouse Validated	
71	Isoproturon	0.102	No	77	Same batch	10	EtOAc				Yes	No	None	MM-ML	No	MS/MS	LC-MS/MS	Inhouse Validated	
71	Kresoxim-methyl	0.276	No	90	Same batch	10	EtOAc				Yes	No	GPC	MM-ML	No	ITD	MS/MS	LC-MS/MS	Inhouse Validated
71	Lambda-cyhalothrin	0.0518	No	98	Same batch	10	EtOAc				Yes	No	None	MM-ML	No	ECD		GC-MS	Inhouse Validated
71	Malathion	0.0660	No	88	Same batch	10	EtOAc				Yes	No	GPC		No	FPD	MS/MS	GC-MS	Inhouse Validated
71	Pirimiphos-methyl	0.0508	No	86	Same batch	10	EtOAc				Yes	No	GPC		No	FPD	MS/MS	GC-MS	Inhouse Validated
71	-Triadimefon	0.0053	No	77	Same batch	10	EtOAc				Yes	No	GPC		No	ITD	MS/MS	LC-MS/MS	Inhouse Validated
71	Triadimenol	1.00	No	83	Same batch	10	EtOAc				Yes	No	GPC		No	ITD	MS/MS	LC-MS/MS	Inhouse Validated
72	Fenitrothion	0.195	No	90		10	AC	CH2Cl			Yes	No	SPE	PS-ML	No	NPD			
72	Malathion	0.112	No	92		10	AC	CH2Cl			Yes	No	SPE	PS-ML	No	NPD			
73	Azoxystrobin	0.322	No	102.9	Same batch	5	ACN				Yes	No	DSPE C18	MM-ML	No	MS/MS	LC-MS/MS	QuEChERS	
73	Carbaryl	0.147	No	95.2	Same batch	5	ACN				Yes	No	None	MM-ML	No	MS/MS	GC-TOF	QuEChERS	
73	Carbendazim and benomyl	1.19	No	93.8	Same batch	5	ACN				Yes	No	None	MM-ML	No	MS/MS	None	QuEChERS	
73	Chlorpyrifos-methyl	0.137	No	106.3	Same batch	5	ACN				Yes	No	DSPE C18	MM-ML	No	MS/MS	LC-MS/MS	QuEChERS	
73	Deltamethrin (cis)	0.0677	No	92.1	Same batch	5	ACN				Yes	No	DSPE C18	MM-ML	No	MS/MS	LC-MS/MS	QuEChERS	
73	Fenitrothion	0.179	No	101.7	Same batch	5	ACN				Yes	No	DSPE C18	MM-ML	No	MSD	GC-TOF	QuEChERS	

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method		
73	Fenpropimorph	1.79	No	104.3	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	GC-TOF	QuEChERS		
73	Fluquinconazole	0.719	No	97.5	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	GC-TOF	QuEChERS		
73	Flutriafol	2.12	No	108.5	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	GC-TOF	QuEChERS		
73	Isoproturon	0.154	No	96.6	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	None	QuEChERS		
73	Kresoxim-methyl	0.410	No	99.4	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	GC-TOF	QuEChERS		
73	Lambda-cyhalothrin	0.0793	No	98.8	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	GC-MS/MS	QuEChERS		
73	Malathion	0.102	No	96.0	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	GC-MS/MS	QuEChERS		
73	Pirimiphos-methyl	0.0757	No	94.4	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	GC-MS/MS	QuEChERS		
73	Spiroxamine	0.878	No	104.1	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	None	QuEChERS		
73	Triadimenol	1.54	No	95.6	Same batch	5	ACN				Yes	No	None	MM-ML	No		MS/MS	GC-TOF	QuEChERS		
74	Azoxystrobin	0.0505	No	90	Same batch	10	ACN				No	No	None	PS-ML	ensulfan lactone	ECD		GC-MS			
74	Carbaryl	0.0821	No	99	Same batch	10	ACN				No	No	None	PS-ML	ethion	NPD		GC-MS			
74	Chlorpyrifos-methyl	0.100	No	100	Same batch	10	ACN				No	No	None	PS-ML	ethion	NPD		GC-MS			
74	Deltamethrin (cis)	0.0502	No	71	Same batch	10	ACN				No	No	None	PS-ML	ensulfan lactone	ECD		GC-MS			
74	Fenitrothion	0.141	No	96	Same batch	10	ACN				No	No	None	PS-ML	ethion	NPD		GC-MS			
74	Fenpropimorph	1.65	No	93	Same batch	10	ACN				No	No	None	PS-ML	ethion	NPD		GC-MS			
74	Fluquinconazole	0.260	No	81	Same batch	10	ACN				No	No	None	PS-ML	ensulfan lactone	ECD		GC-MS			
74	Kresoxim-methyl	0.150	No	95	Same batch	10	ACN				No	No	None	PS-ML	ensulfan lactone	ECD		GC-MS			
74	Lambda-cyhalothrin	0.0571	No	100	Same batch	10	ACN				No	No	None	PS-ML	ensulfan lactone	ECD		GC-MS			
74	Malathion	0.0511	No	93	Same batch	10	ACN				No	No	None	PS-ML	ethion	NPD		GC-MS			
74	Pirimiphos-methyl	0.0521	No	100	Same batch	10	ACN				No	No	None	PS-ML	ethion	NPD		GC-MS			
74	Triadimenol	1.52	No	99	Same batch	10	ACN				No	No	None	PS-ML	ethion	NPD		GC-MS			
75	Azoxystrobin	0.231	Yes automatic	88	Same batch	10	ACN				Yes	No	DSPE PSA C18	PS-ML	TPP		NPD	MS/MS	LC-MS/MS	EN15662	
75	Carbaryl	0.168	No	103	Same batch	10	ACN				Yes	No	DSPE PSA C18	PS-ML	TPP		NPD	MS/MS	LC-MS/MS	EN15662	
75	Carbendazim and benomyl	0.975	Yes automatic	78	Same batch	10	ACN				Yes	No	DSPE PSA C18	PS-ML	TPP		NPD	MS/MS	LC-MS/MS	EN15662	

LAB CODE	Pesticide	Reported result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) ACN	Extraction solvent 2) ACN	Extraction solvent 3) ACN	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
75	Chlorpyrifos-methyl	0.122	No	105	Same batch	10	ACN				Yes	No	DSPE PSA C18	PS-ML	TPP	NPD	MS/MS	LC-MS/MS	EN15662
75	Deltamethrin (cis)	0.092	No	107	Same batch	10	ACN				Yes	No	DSPE PSA C18	PS-ML	TPP	NPD	MS/MS	LC-MS/MS	EN15662
75	Fenitrothion	0.149	No	97	Same batch	10	ACN				Yes	No	DSPE PSA C18	PS-ML	TPP	NPD	MS/MS	LC-MS/MS	EN15662
75	Fluquinconazole	0.612	No	108	Same batch	10	ACN				Yes	No	DSPE PSA C18	PS-ML	TPP	NPD	MS/MS	LC-MS/MS	EN15662
75	Flutriafol	1.17	No	90	Same batch	10	ACN				Yes	No	DSPE PSA C18	PS-ML	TPP	NPD	MS/MS	LC-MS/MS	EN15662
75	Kresoxim-methyl	0.386	No	107	Same batch	10	ACN				Yes	No	DSPE PSA C18	PS-ML	TPP	NPD	MS/MS	LC-MS/MS	EN15662
75	Lambda-cyhalothrin	0.145	No	105	Same batch	10	ACN				Yes	No	DSPE PSA C18	PS-ML	TPP	NPD	MS/MS	LC-MS/MS	EN15662
75	Malathion	0.092	No	96	Same batch	10	ACN				Yes	No	DSPE PSA C18	PS-ML	TPP	NPD	MS/MS	LC-MS/MS	EN15662
75	Pirimiphos-methyl	0.061	No	101	Same batch	10	ACN				Yes	No	DSPE PSA C18	PS-ML	TPP	NPD	MS/MS	LC-MS/MS	EN15662
75	Spiroxamine	0.660	No	94	Same batch	10	ACN				Yes	No	DSPE PSA C18	PS-ML	TPP	NPD	MS/MS	LC-MS/MS	EN15662
75	Triadimenol	1.01	No	95	Same batch	10	ACN				Yes	No	DSPE PSA C18	PS-ML	TPP	NPD	MS/MS	LC-MS/MS	EN15662
75	Mepiquat (free cation)	0.089	Yes automatic 38	95	isotop. lab.	5	ACN				Yes	No	DSPE PSA C18	PS-ML	Isotop. lab.1	MS/MS	None		EN15662
76	Azoxystrobin	0.315	No	91	St. Ad.	5	ACN				Yes	No	DSPE	MM-SL	Tris-dichlor-isopropyl-phosph.	MS/MS		GC-MS/MS	UNI EN 15662
76	Carbaryl	0.095	No	89	St. Ad.	5	ACN				Yes	No	DSPE	MM-SL	Tris-dichlor-isopropyl-phosph.	MS/MS		GC-MS/MS	UNI EN 15662
76	Chlorpyrifos-methyl	0.115	No	95	St. Ad.	5	ACN				Yes	No	DSPE	MM-SL	Tris-dichlor-isopropyl-phosph.	MS/MS		GC-MS/MS	UNI EN 15662
76	Deltamethrin (cis)	0.061	No	85	St. Ad.	5	ACN				Yes	No	DSPE	MM-SL	Tris-dichlor-isopropyl-phosph.	MS/MS		GC-MS/MS	UNI EN 15662
76	Fenitrothion	0.251	No	90	St. Ad.	5	ACN				Yes	No	DSPE	MM-SL	Tris-dichlor-isopropyl-phosph.	MS/MS		GC-MS/MS	UNI EN 15662
76	Fenpropimorph	1.81	No	104	St. Ad.	5	ACN				Yes	No	DSPE	MM-SL	Tris-dichlor-isopropyl-phosph.	MS/MS		GC-MS/MS	UNI EN 15662
76	Fluquinconazole	0.612	No	92	St. Ad.	5	ACN				Yes	No	DSPE	MM-SL	Tris-dichlor-isopropyl-phosph.	MS/MS		GC-MS/MS	UNI EN 15662
76	Kresoxim-methyl	0.395	No	97	St. Ad.	5	ACN				Yes	No	DSPE	MM-SL	Tris-dichlor-isopropyl-phosph.	MS/MS		GC-MS/MS	UNI EN 15662
76	Lambda-cyhalothrin	0.062	No	85	St. Ad.	5	ACN				Yes	No	DSPE	MM-SL	Tris-dichlor-isopropyl-phosph.	MS/MS		GC-MS/MS	UNI EN 15662
76	Malathion	0.121	No	95	St. Ad.	5	ACN				Yes	No	DSPE	MM-SL	Tris-dichlor-isopropyl-phosph.	MS/MS		GC-MS/MS	UNI EN 15662
76	Pirimiphos-methyl	0.151	No	93	St. Ad.	5	ACN				Yes	No	DSPE	MM-SL	Tris-dichlor-isopropyl-phosph.	MS/MS		GC-MS/MS	UNI EN 15662
76	Spiroxamine	1.01	No	103	St. Ad.	5	ACN				Yes	No	DSPE	MM-SL	Tris-dichlor-isopropyl-phosph.	MS/MS		GC-MS/MS	UNI EN 15662
77	Azoxystrobin	0.321	No	108	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MSD		Quechers	

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
77	Carbaryl	0.187	No	100	St. Ad.	5	ACN				Yes	No	None Freez.	St. Ad.	No		MS/MS		Quechers
77	Carbendazim and benomyl	1.618	No	100	St. Ad.	5	ACN				Yes	No	None Freez.	St. Ad.	No		MS/MS		Quechers
77	Chlorpyrifos-methyl	0.135	No	106	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MSD			Quechers
77	Deltamethrin (cis)	0.062	No	111	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MSD			Quechers
77	Fenitrothion	0.231	No	112	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MSD			Quechers
77	Fenpropimorph	2.787	No	117	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MSD			Quechers
77	Fluquinconazole	0.777	No	101	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MSD			Quechers
77	Flutriafol	2.007	No	100	St. Ad.	5	ACN				Yes	No	None Freez.	St. Ad.	No		MS/MS		Quechers
77	Isoproturon	0.215	No	100	St. Ad.	5	ACN				Yes	No	None Freez.	St. Ad.	No		MS/MS		Quechers
77	Kresoxim-methyl	0.436	No	112	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MSD			Quechers
77	Lambda-cyhalothrin	0.073	No	119	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MSD			Quechers
77	Malathion	0.117	No	109	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MSD			Quechers
77	Pirimiphos-methyl	0.084	No	105	Same batch	5	ACN				Yes	No	DSPE Freez.	MM-ML	No	MSD			Quechers
77	Spiroxamine	1.467	No	100	St. Ad.	5	ACN				Yes	No	None Freez.	St. Ad.	No		MS/MS		Quechers
77	Triadimenol	1.825	No	100	St. Ad.	5	ACN				Yes	No	None Freez.	St. Ad.	No		MS/MS		Quechers
77	2,4-D (free acid)	0.367	No	100	St. Ad.	5	ACN				Yes	No	None Freez.	St. Ad.	No		MS/MS		Quechers
77	2,4-D (foll. alk hydrolysis)	0.450	No	100	St. Ad.	5	ACN				Yes	Yes	None Freez.	St. Ad.	No		MS/MS		Quechers
79	Azoxystrobin	0.235	No	96	Same batch	10	EtOAc	Cy-He		+	No	No	GPC PSA	MM-ML	TPP	MS/MS	MS/MS		
79	Carbaryl	0.150	No	99	Same batch	10	EtOAc	Cy-He		+	No	No	GPC PSA	MM-ML	TPP	MS/MS	MS/MS		
79	Carbendazim and benomyl	0.767	Yes	45	Same batch	10	ACN			+	Yes	No	DSPE PSA	MM-ML	No	MS/MS	MS/MS		
79	Chlorpyrifos-methyl	0.122	No	92	Same batch	10	EtOAc	Cy-He		+	No	No	GPC PSA	MM-ML	TPP	MS/MS	MS/MS		
79	Deltamethrin (cis)	0.050	No	90	Same batch	10	EtOAc	Cy-He		+	No	No	GPC PSA	MM-ML	TPP	MS/MS	MS/MS		
79	Fenitrothion	0.215	No	88	Same batch	10	EtOAc	Cy-He		+	No	No	GPC PSA	MM-ML	TPP	MS/MS	MS/MS		
79	Fenpropimorph	1.44	No	94	Same batch	10	EtOAc	Cy-He		+	No	No	GPC PSA	MM-ML	TPP	MS/MS	MS/MS		
79	Fluquinconazole	0.616	No	82	Same batch	10	EtOAc	Cy-He		+	No	No	GPC PSA	MM-ML	TPP	MS/MS	MS/MS		

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) EtOAc	Extraction solvent 2) Cy-He	Extraction solvent 3) Cy-He	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
79	Flutriafol	1.22	No	90	Same batch	10	EtOAc	Cy-He		+	No	No	GPC PSA	MM-ML	TPP	MS/MS	MS/MS		
79	Isoproturon	0.127	Yes	44	Same batch	10	EtOAc	Cy-He		+	No	No	GPC PSA	MM-ML	TPP	MS/MS	MS/MS		
79	Kresoxim-methyl	0.372	No	92	Same batch	10	EtOAc	Cy-He		+	No	No	GPC PSA	MM-ML	TPP	MS/MS	MS/MS		
79	Lambda-cyhalothrin	0.045	No	90	Same batch	10	EtOAc	Cy-He		+	No	No	GPC PSA	MM-ML	TPP	MS/MS	MS/MS		
79	Malathion	0.106	No	105	Same batch	10	EtOAc	Cy-He		+	No	No	GPC PSA	MM-ML	TPP	MS/MS	MS/MS		
79	Pirimiphos-methyl	0.087	No	93	Same batch	10	EtOAc	Cy-He		+	No	No	GPC PSA	MM-ML	TPP	MS/MS	MS/MS		
79	Spiroxamine	0.635	No	86	Same batch	10	EtOAc	Cy-He		+	No	No	GPC PSA	MM-ML	TPP	MS/MS	MS/MS		
79	Triadimenol	1.28	No	93	Same batch	10	EtOAc	Cy-He		+	No	No	GPC PSA	MM-ML	TPP	MS/MS	MS/MS		
80	Azoxystrobin	0.270	No	110	St. Ad.	5	ACN				Yes	No	DSPE Freez.	St. Ad.	TPP	MSD		None	
80	Carbaryl	0.130	No	70	St. Ad.	5	ACN				Yes	No	DSPE Freez.	St. Ad.	TPP	MSD		None	
80	Chlorpyrifos-methyl	0.120	No	70	St. Ad.	5	ACN				Yes	No	DSPE Freez.	St. Ad.	TPP	MSD		None	
80	Deltamethrin (cis)	0.080	No	93	St. Ad.	5	ACN				Yes	No	DSPE Freez.	St. Ad.	TPP	MSD		None	
80	Fenitrothion	0.200	No	102	St. Ad.	5	ACN				Yes	No	DSPE Freez.	St. Ad.	TPP	MSD		None	
80	Kresoxim-methyl	0.340	No	86	St. Ad.	5	ACN				Yes	No	DSPE Freez.	St. Ad.	TPP	MSD		None	
80	Lambda-cyhalothrin	0.060	No	70	St. Ad.	5	ACN				Yes	No	DSPE Freez.	St. Ad.	TPP	MSD		None	
80	Malathion	0.120	No	86	St. Ad.	5	ACN				Yes	No	DSPE Freez.	St. Ad.	TPP	MSD		None	
80	Pirimiphos-methyl	0.160	No	75	St. Ad.	5	ACN				Yes	No	DSPE Freez.	St. Ad.	TPP	MSD		None	
80	Spiroxamine	1.59	No	83	St. Ad.	5	ACN				Yes	No	DSPE Freez.	St. Ad.	TPP	MSD		None	
80	Triadimenol	1.73	No	120	St. Ad.	5	ACN				Yes	No	DSPE Freez.	St. Ad.	TPP	MSD		None	
81	Chlorpyrifos-methyl	0.145	No	106	Validation	10	AC	CH2Cl			No	No	SPE	PS-ML	No	NPD		None	
81	Fenitrothion	0.196	No	110	Validation	10	AC	CH2Cl			No	No	SPE	PS-ML	No	NPD		None	
81	Malathion	0.118	No	105	Validation	10	AC	CH2Cl			No	No	SPE	PS-ML	No	NPD		None	
81	Pirimiphos-methyl	0.105	No	110	Validation	10	AC	CH2Cl			Yes	Yes	SPE	PS-ML	No	NPD		None	
82	Azoxystrobin	0.286	No	137.4	Same batch	25	EtOAc				Yes	No	GPC	MM-ML	No	ECD	GC-TOF	EN 12393-2	
82	Carbendazim and benomyl	0.995	No	101.6	Same batch	25	EtOAc				Yes	No	GPC	PS-ML	No	Diod. Ar.		EN 12393-2 Method P	

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
82	Chlorpyrifos-methyl	0.107	No	101	Same batch	25	EtOAc				Yes	No	GPC	MM-ML	TPP	TOF		NPD	EN 12393-2
82	Deltamethrin (cis)	0.0504	No	103	Same batch	25	EtOAc				Yes	No	GPC	MM-ML	TPP	TOF		NPD	EN 12393-2
82	Fenitrothion	0.164	No	86.4	Same batch	25	EtOAc				Yes	No	GPC	MM-ML	TPP	FPD	GC-TOF	EN 12393-2	
82	Kresoxim-methyl	0.336	No	100	Same batch	25	EtOAc				Yes	No	GPC	MM-ML	TPP	TOF		NPD	EN 12393-2
82	Lambda-cyhalothrin	0.0642	No	102	Same batch	25	EtOAc				Yes	No	GPC	MM-ML	TPP	TOF		NPD	EN 12393-2
82	Malathion	0.0778	No	91.9	Same batch	25	EtOAc				Yes	No	GPC	MM-ML	TPP	FPD	GC-TOF	EN 12393-2	
82	Pirimiphos-methyl	0.0583	No	85.1	Same batch	25	EtOAc				Yes	No	GPC	MM-ML	TPP	FPD	GC-TOF	EN 12393-2	
82	Spiroxamine	0.760	No	89.4	Same batch	25	EtOAc				Yes	No	GPC	MM-ML	TPP	NPD	GC-TOF	EN 12393-2	
83	Azoxystrobin	0.30	Yes	85	St. Ad.	50	AC	CH2Cl			Yes	No	None	St. Ad.	No	ECD		GC-MS	
83	Carbaryl	0.15	Yes	90	St. Ad.	50	AC	CH2Cl			Yes	No	None	St. Ad.	No	MS/MS	MS/MS	LC-MS/MS	
83	Carbendazim and benomyl	0.58	Yes	90	St. Ad.	50	AC	CH2Cl			Yes	No	None	St. Ad.	No	MS/MS	MS/MS	LC-MS/MS	
83	Chlorpyrifos-methyl	0.07	Yes	100	St. Ad.	50	AC	CH2Cl			Yes	No	None	St. Ad.	No	ECD		GC-MS	
83	Deltamethrin (cis)	0.025	Yes	95	St. Ad.	50	AC	CH2Cl			Yes	No	None	St. Ad.	No	ECD		GC-MS	
83	Fenitrothion	0.13	Yes	90	St. Ad.	50	AC	CH2Cl			Yes	No	None	St. Ad.	No	ECD		GC-MS	
83	Fenpropimorph	0.40	Yes	90	St. Ad.	50	AC	CH2Cl			Yes	No	None	St. Ad.	No	ECD		GC-MS	
83	Fluquinconazole	0.25	Yes	90	St. Ad.	50	AC	CH2Cl			Yes	No	None	St. Ad.	No	ECD		GC-MS	
83	Flutriafol	0.18	Yes	90	St. Ad.	50	AC	CH2Cl			Yes	No	None	St. Ad.	No	ECD		GC-MS	
83	Kresoxim-methyl	0.57	Yes	95	St. Ad.	50	AC	CH2Cl			Yes	No	None	St. Ad.	No	ECD		GC-MS	
83	Lambda-cyhalothrin	0.06	Yes	95	St. Ad.	50	AC	CH2Cl			Yes	No	None	St. Ad.	No	ECD		GC-MS	
83	Malathion	0.03	Yes	90	Same batch	50	AC	CH2Cl			Yes	No	None	St. Ad.	No	ECD		GC-MS	
83	Pirimiphos-methyl	0.055	Yes	95	Same batch	50	AC	CH2Cl			Yes	No	None	St. Ad.	No	ECD		GC-MS	
83	Spiroxamine	0.90	Yes	90	Same batch	50	AC	CH2Cl			Yes	No	None	St. Ad.	No	MS/MS	MS	LC-MS	
83	Triadimenol	0.70	Yes	85	Same batch	50	AC	CH2Cl			Yes	No	None	St. Ad.	No				
84	Azoxystrobin	0.330	No	88	Same batch	10	AC	CH2Cl	PE		Yes		None	PS-ML	TPP		MS/MS	LC-MS/MS	
84	Carbaryl	0.157	No	73	Same batch	10	AC	CH2Cl	PE		Yes		None	PS-ML	TPP		MS/MS	LC-MS/MS	

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) AC	Extraction solvent 2) EtOAc	Extraction solvent 3) CH2Cl	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method	
84	Carbendazim and benomyl	1.34	No	94	Same batch	10	AC	CH2Cl	PE		Yes		None	PS-ML	TPP		MS/MS	LC-MS/MS		
84	Chlorpyrifos-methyl	0.0901	No	66	Same batch	5	AC	EtOAc	HEX		Yes		None	MM-ML	TPP		MS/MS	GC-MS/MS		
84	Deltamethrin (cis)	0.0391	No	92	Same batch	5	AC	EtOAc	HEX		Yes		None	MM-ML	TPP		MS/MS	GC-MS/MS		
84	Fenitrothion	0.182	No	84	Same batch	5	AC	EtOAc	HEX		Yes		None	MM-ML	TPP		MS/MS	GC-MS/MS		
84	Fenpropimorph	1.93	No	86	Same batch	5	AC	EtOAc	HEX		Yes		None	MM-ML	TPP		MS/MS	GC-MS/MS		
84	Fluquinconazole	0.747	No	70	Same batch	10	AC	CH2Cl	PE		Yes		None	PS-ML	TPP		MS/MS	LC-MS/MS		
84	Flutriafol	2.22	No	98	Same batch	10	AC	CH2Cl	PE		Yes		None	PS-ML	TPP		MS/MS	LC-MS/MS		
84	Isoproturon	0.156	No	97	Same batch	10	AC	CH2Cl	PE		Yes		None	PS-ML	TPP		MS/MS	LC-MS/MS		
84	Kresoxim-methyl	0.353	No	108	Same batch	5	AC	EtOAc	HEX		Yes		None	MM-ML	TPP		MS/MS	GC-MS/MS		
84	Lambda-cyhalothrin	0.0696	No	92	Same batch	5	AC	EtOAc	HEX		Yes		None	MM-ML	TPP		MS/MS	GC-MS/MS		
84	Malathion	0.0762	No	61	Same batch	5	AC	EtOAc	HEX		Yes		None	MM-ML	TPP		MS/MS	GC-MS/MS		
84	Pirimiphos-methyl	0.0652	No	80	Same batch	5	AC	EtOAc	HEX		Yes		None	MM-ML	TPP		MS/MS	GC-MS/MS		
84	Spiroxamine	1.34	No	103	Same batch	10	AC	CH2Cl	PE		Yes		None	PS-ML	TPP		MS/MS	LC-MS/MS		
84	Triadimenol	1.66	No	95	Same batch	10	AC	CH2Cl	PE		Yes		None	PS-ML	TPP		MS/MS	LC-MS/MS		
84	2,4-D (free acid)	0.407	No	109	Same batch	10	MeOH				Yes		None	PS-ML	nicarbazin		MS/MS	LC-MS/MS		
84	2,4-D (foll. alk hydrolysis)	0.398	No		Same batch	10	MeOH				Yes	Yes	None	PS-ML	nicarbazin		MS/MS	LC-MS/MS		
84	Mepiquat (free cation)	0.0810	No	95	Same batch	10	MeOH				Yes		PS-ML	Isotop. lab.1			MS/MS	LC-MS/MS		
85	Azoxystrobin	0.402	No	99	Validation	5	ACN				Yes	No	DSPE	MM-ML	Linuron		MS/MS	GC-MS	QuEChERS	
85	Carbaryl	0.169	No	96	Validation	5	ACN				Yes	No	DSPE	MM-ML	Linuron		MS/MS	GC-MS	QuEChERS	
85	Carbendazim and benomyl	1.23	No	87	Validation	5	ACN				Yes	No	DSPE	MM-ML	Linuron Triphenylmethan		MS/MS	None	QuEChERS	
85	Chlorpyrifos-methyl	0.127	No	102	Validation	5	ACN				Yes	No	DSPE	MM-ML	Triphenylmethan	MSD	GC-MS	QuEChERS		
85	Deltamethrin (cis)	0.069	No	99	Validation	5	ACN				Yes	No	DSPE	MM-ML	Triphenylmethan	MSD	GC-MS	QuEChERS		
85	Fenitrothion	0.204	No	101	Validation	5	ACN				Yes	No	DSPE	MM-ML	Tris-dichlor-isopropyl-phosph.	MSD	GC-MS	QuEChERS		
85	Fenpropimorph	2.65	No	100	Validation	5	ACN				Yes	No	DSPE	MM-ML	Linuron		MS/MS	GC-MS	QuEChERS	
85	Fluquinconazole	0.911	No	106	Validation	5	ACN				Yes	No	DSPE	MM-ML	Linuron		MS/MS	GC-MS	QuEChERS	

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method			
85	Flutriafol	2.57	No	102	Validation	5	ACN				Yes	No	DSPE	MM-ML	Linuron		MS/MS	GC-MS	QuEChERS			
85	Isoproturon	0.162	No	97	Validation	5	ACN				Yes	No	DSPE	MM-ML	Linuron		MS/MS	None	QuEChERS			
85	Kresoxim-methyl	0.511	No	95	Validation	5	ACN				Yes	No	DSPE	MM-ML	Linuron		MS/MS	GC-MS	QuEChERS			
85	Lambda-cyhalothrin	0.094	No	104	Validation	5	ACN				Yes	No	DSPE	MM-ML	Triphenylmethan	MSD	GC-MS	QuEChERS				
85	Malathion	0.129	No	91	Validation	5	ACN				Yes	No	DSPE	MM-ML		FPD	GC-MS	QuEChERS				
85	Pirimiphos-methyl	0.094	No	107	Validation	5	ACN				Yes	No	DSPE	MM-ML	Triphenylmethan	MSD	GC-MS	QuEChERS				
85	Spiroxamine	1.26	No	96	Validation	5	ACN				Yes	No	DSPE	MM-ML	Linuron		MS/MS	GC-MS	QuEChERS			
85	Triadimenol	2.08	No	102	Validation	5	ACN				Yes	No	DSPE	MM-ML	Triphenylmethan	MSD	GC-MS	QuEChERS				
85	2,4-D (free acid)	0.371	No	92	Validation	5	ACN				Yes	No	None	MM-ML	MCPA		MS/MS	None	QuEChERS			
85	2,4-D (foll. alk hydrolysis)	0.400	No	92	Validation	5	ACN				Yes	Yes	None	MM-ML	MCPA		MS/MS	None	QuEChERS			
85	Glyphosate	3.20	No	78	Same batch	5	MeOH				Yes	No	None	MM-ML	Isotop. lab.1		MS/MS	None				
85	Mepiquat (free cation)	0.091	No	78	Validation	10	MeOH				Yes	No	None	PS-ML	Isotop. lab.1		MS/MS	None	EN 15055:2006			
86	Chlorpyrifos-methyl	0.168	No	95.0	Validation	10	AC	CH2Cl			Yes	No	SPE SiOH		No	NPD			PB-35/CHZedycja 02 z dnia 25.02.2010 r.			
86	Fenitrothion	0.215	No	95.0	Validation	10	AC	CH2Cl			Yes	No	SPE SiOH		No	NPD			PB-35/CHZedycja 02 z dnia 25.02.2010 r.			
86	Malathion	0.165	No	97.7	Validation	10	AC	CH2Cl			Yes	No	SPE SiOH		No	NPD			PB-35/CHZedycja 02 z dnia 25.02.2010 r.			
86	Pirimiphos-methyl	0.100	No	90.0	Validation	10	AC	CH2Cl			Yes	No	SPE SiOH		No	NPD			PB-35/CHZedycja 02 z dnia 25.02.2010 r.			
87	Azoxystrobin	0.34	No	105	Validation	5	ACN	AC			Yes	No	Freez. GPC	MM-ML	No	TOF	MS/MS	GC-MS/MS				
87	Carbaryl	0.19	No	110	Validation	5	ACN	AC			Yes	No	Freez. GPC	MM-ML	No	TOF	MS/MS	GC-MS/MS				
87	Carbendazim and benomyl	1.29	No	95	Validation	5	ACN				Yes	No	Freez. GPC	MM-ML	No		MS/MS					
87	Chlorpyrifos-methyl	0.16	No	110	Validation	5	ACN	AC			Yes	No	Freez. GPC	MM-ML	No	TOF	MS/MS	GC-MS/MS				
87	Deltamethrin (cis)	0.061	No	110	Validation	5	ACN	AC			Yes	No	Freez. GPC	MM-ML	No	TOF		GC-TOF				
87	Fenitrothion	0.21	No	95	Validation	5	ACN	AC			Yes	No	Freez. GPC	MM-ML	No	TOF	MS/MS	GC-MS/MS				
87	Fenpropimorph	2.2	No	100	Validation	5	ACN	AC			Yes	No	Freez. GPC	MM-ML	No	TOF	MS/MS	GC-MS/MS				
87	Fluquinconazole	0.84	No	90	Validation	5	ACN	AC			Yes	No	Freez. GPC	MM-ML	No	TOF	MS/MS	GC-MS/MS				
87	Flutriafol	2.0	No	105	Validation	5	ACN	AC			Yes	No	Freez. GPC	MM-ML	No	TOF	MS/MS	GC-MS/MS				

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) ACN	Extraction solvent 2) AC	Extraction solvent 3) ACN	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
87	Isoproturon	0.13	No	102	Validation	5					Yes	No	Freez. GPC	MM-ML	No		MS/MS		
87	Kresoxim-methyl	0.47	No	100	Validation	5	ACN	AC			Yes	No	Freez. GPC	MM-ML	No	TOF	MS/MS	GC-MS/MS	
87	Lambda-cyhalothrin	0.082	No	105	Validation	5	ACN	AC			Yes	No	Freez. GPC	MM-ML	No	TOF		GC-TOF	
87	Malathion	0.11	No	90	Validation	5	ACN	AC			Yes	No	Freez. GPC	MM-ML	No	TOF		GC-TOF	
87	Pirimiphos-methyl	0.090	No	103	Validation	5	ACN	AC			Yes	No	Freez. GPC	MM-ML	No	TOF	MS/MS	GC-MS/MS	
87	Spiroxamine	1.2	No	105	Validation	5	ACN	AC			Yes	No	Freez. GPC	MM-ML	No	TOF	MS/MS	GC-MS/MS	
87	-Triadimefon	0.01	No	103	Validation	5	ACN	AC			Yes	No	Freez. GPC	MM-ML	No	TOF	MS/MS	GC-MS/MS	
87	Triadimenol	1.85	No	90	Validation	5	ACN	AC			Yes	No	Freez. GPC	MM-ML	No	TOF	MS/MS	GC-MS/MS	
87	2,4-D (free acid)	0.48	No	120	Validation	5	ACN				Yes	No	Freez.	MM-ML	No		MS/MS		
87	Mepiquat (free cation)	0.070	No	80	Validation	10	MeOH				Yes	No		PS-ML	Isotop. lab.1		MS/MS		
88	Azoxystrobin	0.417	No	106.5	Same batch	10	AC	CH2Cl	EtOAc		Yes	No	GPC	MM-ML	No	ECD			
88	Carbendazim and benomyl	1.178	No	81.7	Same batch	10	EtOAc	EtOAc	MeOH		Yes	No	L/I part.	PS-ML	No		Fluor.	HPLC-DAD	
88	Chlorpyrifos-methyl	0.142	No	84.1	Same batch	10	AC	CH2Cl	EtOAc		Yes	No	GPC	MM-ML	No	ECD		GC-NPD	
88	Deltamethrin (cis)	0.052	No	82.8	Same batch	10	AC	CH2Cl	EtOAc		Yes	No	GPC	MM-ML	No	ECD			
88	Fenitrothion	0.206	No	84.4	Same batch	10	AC	CH2Cl	EtOAc		Yes	No	GPC	MM-ML	No	ECD		GC-NPD	
88	Kresoxim-methyl	0.395	No	87.4	Same batch	10	AC	CH2Cl	EtOAc		Yes	No	GPC	MM-ML	No	ECD		GC-NPD	
88	Lambda-cyhalothrin	0.063	No	99.0	Same batch	10	AC	CH2Cl	EtOAc		Yes	No	GPC	MM-ML	No	ECD			
88	Malathion	0.098	No	84.1	Same batch	10	AC	CH2Cl	EtOAc		Yes	No	GPC	MM-ML	No	ECD		GC-NPD	
88	Pirimiphos-methyl	0.062	No	72.9	Same batch	10	AC	CH2Cl	EtOAc		Yes	No	GPC	MM-ML	No	NPD		GC-ECD	
88	Triadimenol	1.330	No	78.6	Same batch	10	AC	CH2Cl	EtOAc		Yes	No	GPC	MM-ML	No	NPD			
89	Azoxystrobin	0.20	No	98	Same batch	10	ACN				Yes	No	DSPE	PS-ML	No		MS	GC-ECD	QuEChERS(MS EN 15662:2009)
89	Carbaryl	0.16	No	60	Same batch	10	ACN				Yes	No	DSPE	PS-ML	No		MS		QuEChERS(MS EN 15662:2009)
89	Carbendazim and benomyl	0.95	No	60	Same batch	10	ACN				Yes	No	DSPE	PS-ML	No		MS		QuEChERS(MS EN 15662:2009)
89	Chlorpyrifos-methyl	0.10	No	120	Same batch	10	ACN				Yes	No	DSPE	PS-ML	No	FPD			QuEChERS(MS EN 15662:2009)
89	Fenitrothion	0.14	No	120	Same batch	10	ACN				Yes	No	DSPE	PS-ML	No	FPD			QuEChERS(MS EN 15662:2009)

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery, %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
89	Fenpropimorph	1.0	Yes automatic	100	Validation	10	ACN				Yes	No	DSPE	PS-ML	No		MS		QuEChERS(MS EN 15662:2009)
89	Fluquinconazole	0.52	No Yes automatic	112	Same batch	10	ACN				Yes	No	DSPE	PS-ML	No	ECD			QuEChERS(MS EN 15662:2009)
89	Flutriafol	2.31	Yes automatic	100	Validation	10	ACN				Yes	No	DSPE	PS-ML	No	ECD	LC-MS		QuEChERS(MS EN 15662:2009)
89	Isoproturon	0.12	Yes automatic	100	Validation	10	ACN				Yes	No	DSPE	PS-ML	No		MS		QuEChERS(MS EN 15662:2009)
89	Kresoxim-methyl	0.22	Yes automatic	100	Validation	10	ACN				Yes	No	DSPE	PS-ML	No	ECD	LC-MS		QuEChERS(MS EN 15662:2009)
89	Lambda-cyhalothrin	0.05	No	70	Same batch	10	ACN				Yes	No	DSPE	PS-ML	No	ECD			QuEChERS(MS EN 15662:2009)
89	Malathion	0.10	No	135	Same batch	10	ACN				Yes	No	DSPE	PS-ML	No	FPD	GC-TSD		QuEChERS(MS EN 15662:2009)
89	Pirimiphos-methyl	0.06	No	90	Same batch	10	ACN				Yes	No	DSPE	PS-ML	No	FPD	LC-MS		QuEChERS(MS EN 15662:2009)
89	Spiroxamine	0.35	Yes	40	Same batch	10	ACN				Yes	No	DSPE	PS-ML	No		MS		QuEChERS(MS EN 15662:2009)
89	Triadimenol	2.3	No	79	Same batch	10	ACN				Yes	No	DSPE	PS-ML	No		MS		QuEChERS(MS EN 15662:2009)
90	Azoxystrobin	0.306	No	110	Same batch	5	EtOAc				Yes	No	GPC	MM-ML	No	ECD		Two columns FP017	
90	Carbendazim and benomyl	1.63	No	111	Same batch	3	MeOH				Yes	No	Other Filter	MM-ML	Isotop. lab.2		MS/MS	LC-MS/MS	FP086
90	Chlorpyrifos-methyl	0.0700	No	81	Same batch	5	EtOAc				Yes	No	GPC	MM-ML	No	ECD		Two columns FP017	
90	Deltamethrin (cis)	0.0530	No	101	Same batch	5	EtOAc				Yes	No	GPC	MM-ML	No	ECD		Two columns FP017	
90	Fenitrothion	0.100	No	76	Same batch	5	EtOAc				Yes	No	GPC	MM-ML	No	ECD		Two columns FP017	
90	Isoproturon	0.132	No	104	Same batch	3	MeOH				Yes	No	Other Filter	MM-ML	Isotop. lab.2		MS/MS	LC-MS/MS	FP086
90	Kresoxim-methyl	0.305	No	83	Same batch	5	EtOAc				Yes	No	GPC	MM-ML	No	ECD		Two columns FP017	
90	Malathion	0.0800	No	88	Same batch	5	EtOAc				Yes	No	GPC	MM-ML	No	ECD		Two columns FP017	
90	Pirimiphos-methyl	0.0520	No	85	Same batch	5	EtOAc				Yes	No	GPC	MM-ML	No	ECD		Two columns FP017	
90	Spiroxamine	1.44	No	104	Same batch	3	MeOH				Yes	No	Other Filter	MM-ML	Isotop. lab.2		MS/MS	LC-MS/MS	FP086
90	2,4-D (free acid)	0.324	No	120	Same batch	3	MeOH				Yes	No	Other Filter	MM-ML	Isotop. lab.2		MS/MS	LC-MS/MS	FP086
90	Glyphosate	8.07	No	86	Same batch	3	Water				Yes	No	Other filter	MM-ML	Isotop. lab.1		MS/MS	LC-MS/MS	FP054
90	Mepiquat (free cation)	0.0700	No Yes automatic	89	Same batch	10	MeOH	Water			Yes	No	SPE C18	MM-ML	Isotop. lab.2		MS/MS	LC-MS/MS	Fp045
91	Azoxystrobin	0.311	Yes automatic	100	St. Ad.	3	EtOAc	Cy-He			Yes	No	Other	St. Ad.	No		MS/MS	LC-MS/MS	
91	Carbaryl	0.155	Yes automatic	100	St. Ad.	3	EtOAc	Cy-He			Yes	No	Other	St. Ad.	No		MS/MS	LC-MS/MS	

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) EtOAc	Extraction solvent 2) Cy-He	Extraction solvent 3) Water	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method	
91	Carbendazim and benomyl	0.879	Yes automatic	100	St. Ad.	3	EtOAc	Cy-He			Yes	No	Other	St. Ad.	No	MS/MS	LC-MS/MS			
91	Chlorpyrifos-methyl	0.138	Yes automatic	100	St. Ad.	3	EtOAc	Cy-He			Yes	No	GPC	St. Ad.	No	MS/MS	GC-MS/MS			
91	Deltamethrin (cis)	0.0937	Yes automatic	100	St. Ad.	3	EtOAc	Cy-He			Yes	No	GPC	St. Ad.	No	MS/MS	GC-MS/MS			
91	Fenitrothion	0.174	Yes automatic	100	St. Ad.	3	EtOAc	Cy-He			Yes	No	GPC	St. Ad.	No	MS/MS	GC-MS/MS			
91	Fenpropimorph	1.99	Yes automatic	100	St. Ad.	3	EtOAc	Cy-He			Yes	No	Other	St. Ad.	No	MS/MS	LC-MS/MS			
91	Fluquinconazole	0.670	Yes automatic	100	St. Ad.	3	EtOAc	Cy-He			Yes	No	Other	St. Ad.	No	MS/MS	LC-MS/MS			
91	Flutriafol	1.89	Yes automatic	100	St. Ad.	3	EtOAc	Cy-He			Yes	No	Other	St. Ad.	No	MS/MS	LC-MS/MS			
91	Isoproturon	0.145	Yes automatic	100	St. Ad.	3	EtOAc	Cy-He			Yes	No	Other	St. Ad.	No	MS/MS	LC-MS/MS			
91	Kresoxim-methyl	0.404	Yes automatic	100	St. Ad.	3	EtOAc	Cy-He			Yes	No	GPC	St. Ad.	No	MS/MS	GC-MS/MS			
91	Lambda-cyhalothrin	0.107	Yes automatic	100	St. Ad.	3	EtOAc	Cy-He			Yes	No	GPC	St. Ad.	No	MS/MS	GC-MS/MS			
91	Malathion	0.0880	Yes automatic	100	St. Ad.	3	EtOAc	Cy-He			Yes	No	GPC	St. Ad.	No	MS/MS	GC-MS/MS			
91	Pirimiphos-methyl	0.0707	Yes automatic	100	St. Ad.	3	EtOAc	Cy-He			Yes	No	GPC	St. Ad.	No	MS/MS	GC-MS/MS			
91	Spiroxamine	1.05	Yes automatic	100	St. Ad.	3	EtOAc	Cy-He			Yes	No	Other	St. Ad.	No	MS/MS	LC-MS/MS			
91	-Triadimefon	0.0087	Yes automatic	100	St. Ad.	3	EtOAc	Cy-He			Yes	No	Other	St. Ad.	No	MS/MS	LC-MS/MS			
91	Triadimenol	1.62	Yes automatic	100	St. Ad.	3	EtOAc	Cy-He			Yes	No	Other	St. Ad.	No	MS/MS	LC-MS/MS			
91	2,4-D (free acid)	0.365	Yes automatic	100	St. Ad.	3	EtOAc	Cy-He			Yes	No	Other	St. Ad.	No	MS/MS	LC-MS/MS			
91	Mepiquat (free cation)	0.109	Yes automatic	100	St. Ad.	0	Water				Yes	No	Other	St. Ad.	No	MS/MS	LC-MS/MS			
92	Azoxystrobin	0.503	Yes	102.9	Same batch	5	ACN					Yes	No	DSPE PSA C18	MM-ML	No	MS/MS	GC-MS/MS		
92	Carbaryl	0.171	Yes	105.9	Same batch	5	ACN					Yes	No	DSPE PSA C18	MM-ML	No	MS/MS	LC-MS/MS		
92	Carbendazim and benomyl	0.647	Yes	104.5	Same batch	5	ACN					Yes	No	DSPE PSA C18	MM-ML	No	MS/MS	LC-MS/MS		
92	Chlorpyrifos-methyl	0.228	Yes	69	Same batch	5	ACN					Yes	No	DSPE PSA C18	MM-ML	No	MS/MS	GC-MS/MS		
92	Deltamethrin (cis)	0.116	Yes	81.2	Same batch	5	ACN					Yes	No	DSPE PSA C18	MM-ML	No	MS/MS	GC-MS/MS		
92	Fenpropimorph	3.45	Yes	77.9	Same batch	5	ACN					Yes	No	DSPE PSA C18	MM-ML	No	MS/MS	GC-MS/MS		
92	Flutriafol	2.51	Yes	103.7	Same batch	5	ACN					Yes	No	DSPE PSA C18	MM-ML	No	MS/MS	LC-MS/MS		
92	Kresoxim-methyl	0.598	Yes	102.5	Same batch	5	ACN					Yes	No	DSPE PSA C18	MM-ML	No	MS/MS	GC-MS/MS		

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
92	Lambda-cyhalothrin	0.114	Yes	79.8	Same batch	5	ACN				Yes	No	DSPE PSA C18	MM-ML	No	MS/MS		GC-MS/MS	
92	Malathion	0.129	Yes	106.7	Same batch	5	ACN				Yes	No	DSPE PSA C18	MM-ML	No	MS/MS	LC-MS/MS		
92	Pirimiphos-methyl	0.0941	Yes	106.6	Same batch	5	ACN				Yes	No	DSPE PSA C18	MM-ML	No	MS/MS	LC-MS/MS		
92	Triadimenol	1.67	Yes	103.8	Same batch	5	ACN				Yes	No	DSPE PSA C18	MM-ML	No	MS/MS	LC-MS/MS		
93	Azoxystrobin	0.270	No			5	ACN				Yes	No	DSPE PSA C18	PS-ML	No	MS/MS	ITD	NF EN 15662	
93	Carbaryl	0.158	No			5	ACN				Yes	No	DSPE PSA C18	PS-ML	No	MS/MS		NF EN 15662	
93	Carbendazim and benomyl	1.45	No			5	ACN				Yes	No	DSPE PSA C18	PS-ML	No	MS/MS		NF EN 15662	
93	Chlorpyrifos-methyl	0.112	No			5	ACN				Yes	No	DSPE PSA C18	PS-ML	trichloronate	ITD			NF EN 15662
93	Fenitrothion	0.159	No			5	ACN				Yes	No	DSPE PSA C18	PS-ML	trichloronate	ITD			NF EN 15662
93	Kresoxim-methyl	0.390	No			5	ACN				Yes	No	SPE PSA C18	PS-ML	trichloronate	ITD	LC-MS/MS	NF EN 15662	
93	Lambda-cyhalothrin	0.066	No			5	ACN				Yes	No	DSPE PSA C18	PS-ML	trichloronate	ITD			NF EN 15662
93	Malathion	0.117	No			5	ACN				Yes	No	DSPE PSA C18	PS-ML	No	MS/MS	ITD	NF EN 15662	
93	Pirimiphos-methyl	0.069	No			5	ACN				Yes	No	DSPE PSA C18	PS-ML	trichloronate	ITD			NF EN 15662
93	Spiroxamine	0.848	No			5	ACN				Yes	No	DSPE	PS-ML	No	MS/MS	ITD	NF EN 15662	
93	Triadimenol	1.43	No			5	ACN				Yes	No	SPE	PS-ML	trichloronate	ITD	LC-MS/MS	NF EN 15662	
94	Fenitrothion	0.195	No	97.1		5	AC	CH2Cl			Yes		SPE	PS-ML		NPD			
94	Malathion	0.115	No	94.3		5	AC	CH2Cl			Yes		SPE	PS-ML		NPD			
95	Azoxystrobin	0.349	No	95.5	Same batch	5	ACN				Yes		DSPE	PS-ML	No	ECD	Diod. Ar.	Two columns EN 15662:2008	
95	Carbaryl	0.165	No	96.5	Same batch	5	ACN				Yes		DSPE	PS-ML	No		Diod. Ar.	Two columns EN 15662:2008	
95	Carbendazim and benomyl	1.04	No	87.0	Same batch	5	ACN				Yes		DSPE	PS-ML	No		Diod. Ar.	Two columns EN 15662:2008	
95	Chlorpyrifos-methyl	0.133	No	105.0	Same batch	5	ACN				Yes		DSPE	PS-ML	No	NPD		Two columns EN 15662:2008	
95	Fenitrothion	0.203	No	102.5	Same batch	5	ACN				Yes		DSPE	PS-ML	No	NPD		Two columns EN 15662:2008	
95	Fenpropimorph	2.44	No	83.6	Same batch	5	ACN				Yes		DSPE	PS-ML	No	NPD		Two columns EN 15662:2008	
95	Fluquinconazole	0.733	No	96.0	Same batch	5	ACN				Yes		DSPE	PS-ML	No	ECD		Two columns EN 15662:2008	
95	Flutriafol	2.09	No	87.0	Same batch	0	ACN				Yes		DSPE	PS-ML	No	ECD		Two columns EN 15662:2008	

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) ACN	Extraction solvent 2) ACN	Extraction solvent 3) ACN	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
95	Isoproturon	0.142	No	96.0	Same batch	5					Yes		DSPE	PS-ML	No	NPD	Two columns EN 15662:2008		
95	Kresoxim-methyl	0.436	No	103.0	Same batch	5	ACN				Yes		DSPE	MM-ML	No	NPD	Diod. Ar.	Two columns EN 15662:2008	
95	Lambda-cyhalothrin	0.062	No	102.0	Same batch	5	ACN				Yes		DSPE	MM-ML	No	ECD	Two columns EN 15662:2008		
95	Malathion	0.108	No	99.7	Same batch	5	ACN				Yes		DSPE	PS-ML	No	NPD	Two columns EN 15662:2008		
95	Pirimiphos-methyl	0.075	No	93.5	Same batch	5	ACN				Yes		DSPE	PS-ML	No	NPD	Two columns EN 15662:2008		
95	Spiroxamine	1.32	No	95.0	Same batch	5	ACN				Yes		DSPE	PS-ML	No	NPD	Two columns EN 15662:2008		
95	Triadimenol	1.86	No	90.0	Same batch	5	ACN				Yes		DSPE	PS-ML	No	ECD	Two columns EN 15662:2008		
96	Chlorpyrifos-methyl	0.193	No	90	Same batch	5	ACN				Yes		DSPE	PS-ML	PCB100	ECD	GC-MS		
96	Deltamethrin (cis)	0.054	Yes	90	Same batch	1	AC	HEX					SPE	PS-ML	PCB100	ECD	GC-MS		
96	Fenitrothion	0.309	No	90	Same batch	5	ACN				Yes		DSPE	PS-ML	PCB100	ECD	GC-MS		
96	Malathion	0.155	No	90	Same batch	5	ACN				Yes		DSPE	PS-ML	PCB100	ECD	GC-MS		
97	Azoxystrobin	0.351	No	97	Validation	20	AC	CH2Cl			Yes		GPC	PS-ML		ECD	None	EN 12393	
97	Carbendazim and benomyl	0.984	No	98	Validation	25	AC	EtOAc			Yes		GPC	PS-ML		UV	None		
97	Chlorpyrifos-methyl	0.100	No	97	Validation	25	AC	CH2Cl			Yes	No	GPC	PS-ML		NPD	Two columns		
97	Deltamethrin (cis)	0.047	No	95	Validation	25	AC	CH2Cl			Yes	No	GPC	PS-ML		ECD	Two columns		
97	Fenitrothion	0.157	Yes	89	Validation	25	AC	CH2Cl			Yes	No	GPC	PS-ML		NPD	Two columns		
97	Kresoxim-methyl	0.376	Yes	89	Validation	25	AC	CH2Cl			Yes	No	GPC	PS-ML		NPD	Two columns		
97	Lambda-cyhalothrin	0.061	Yes	89	Validation	25	AC	CH2Cl			Yes	No	GPC	PS-ML		ECD	Two columns		
97	Malathion	0.110	No	106	Validation	25	AC	CH2Cl			Yes	No	GPC	PS-ML		NPD	Two columns		
97	Pirimiphos-methyl	0.057	Yes	86	Validation	25	AC	CH2Cl			Yes	No	GPC	PS-ML		NPD	Two columns		
97	Spiroxamine	0.467	Yes	60	Validation	25	AC	CH2Cl			Yes	No	GPC	PS-ML		NPD	Two columns		
97	Triadimenol	1.26	Yes	89	Validation	25	AC	CH2Cl			Yes	No	GPC	PS-ML		NPD	Two columns		
98	Azoxystrobin	0.195	No	65	Validation	50	EtOAc				No	No	GPC	PS-ML	No	ECD	Two columns EN-12393		
98	Carbaryl	0.100	Yes	60	Same batch	25	CH2Cl				No	No	SPE	PS-ML	No	Fluor.	None	EN 14185-2	
98	Carbendazim and benomyl	0.869	No	80	Validation	25	MeOH	CH2Cl	CH2Cl		No	No	L/I part.	PS-ML	No	UV	Fluor.		

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
98	Chlorpyrifos-methyl	0.107	No	105	Validation	50	EtOAc				No	No	GPC	PS-ML	No	ECD	Two columns EN-12393		
98	Deltamethrin (cis)	0.062	No	102	Validation	50	EtOAc				No	No	GPC	PS-ML	No	ECD	Two columns EN-12393		
98	Fenitrothion	0.183	No	106	Validation	50	EtOAc				No	No	GPC	PS-ML	No	ECD	Two columns EN-12393		
98	Kresoxim-methyl	0.240	No	87	Validation	50	EtOAc				No	No	GPC	PS-ML	No	ECD	Two columns EN-12393		
98	Lambda-cyhalothrin	0.046	No	71	Validation	50	EtOAc				No	No	GPC	PS-ML	No	ECD	Two columns EN-12393		
98	Malathion	0.044	No	97	Validation	50	EtOAc				No	No	GPC	PS-ML	No	ECD	Two columns EN-12393		
98	Pirimiphos-methyl	0.077	No	81	Validation	50	EtOAc				No	No	GPC	PS-ML	No	ECD	Two columns EN-12393		
99	Carbendazim and benomyl	1.060	No	70	Validation	30	EtOAc	EtOAc			Yes	No	L/I part.	PS-ML			Diod. Ar.		
99	Chlorpyrifos-methyl	0.140	No	94	Validation	50	AC	CH2Cl			No	No	SPE	PS-ML		ECD			
99	Deltamethrin (cis)	0.059	No	102	Validation	50	AC	CH2Cl			No	No	SPE	PS-ML		ECD			
99	Fenitrothion	0.180	No	94	Validation	50	AC	CH2Cl			No	No	SPE	PS-ML		ECD			
99	Lambda-cyhalothrin	0.072	No	93	Validation	50	AC	CH2Cl			No	No	SPE	PS-ML		ECD			
99	Malathion	0.102	No	82	Validation	50	AC	CH2Cl			No	No	SPE	PS-ML		ECD			
99	Pirimiphos-methyl	0.082	No	76	Validation	50	AC	CH2Cl			No	No	SPE	PS-ML	TPP	NPD			
100	Azoxystrobin	0.379	No	118	Same batch	5	ACN				Yes		DSPE PSA	PS-ML	No		MS/MS	LC-MS/MS	
100	Carbaryl	0.237	No	122	Same batch	5	ACN				Yes		DSPE PSA	PS-ML	No		MS/MS	LC-MS/MS	
100	Carbendazim and benomyl	3.66	No	94	Same batch	5	ACN				Yes		DSPE PSA	PS-ML	No		MS/MS	LC-MS/MS	
100	Chlorpyrifos-methyl	0.0690	No	71	Same batch	20	AC						GPC PSA	MM-SL	No	FPD		ITD	
100	Fenitrothion	0.0850	No	87	Same batch	20	AC						GPC PSA	MM-SL	No	FPD		ITD	
100	Fenpropimorph	2.11	No	107	Same batch	5	ACN				Yes		DSPE PSA	PS-ML	No		MS/MS	LC-MS/MS	
100	Flutriafol	2.71	No	100	Same batch	5	ACN				Yes		DSPE PSA	PS-ML	No		MS/MS	LC-MS/MS	
100	Kresoxim-methyl	0.571	No	110	Same batch	5	ACN				Yes		DSPE PSA	PS-ML	No		MS/MS	LC-MS/MS	
100	Lambda-cyhalothrin	0.0320	No	47	Same batch	20	AC						GPC PSA	PS-ML	No	ECD		ITD	
100	Malathion	0.147	No	59	Same batch	5	ACN				Yes		DSPE PSA	PS-ML	No		MS/MS	LC-MS/MS	
100	Pirimiphos-methyl	0.0954	No	112	Same batch	5	ACN				Yes		DSPE PSA	PS-ML	No		MS/MS	LC-MS/MS	

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) ACN	Extraction solvent 2) CH2Cl	Extraction solvent 3) PE	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
100	Triadimenol	1.93	No	100	Same batch	5	ACN				Yes		DSPE PSA	PS-ML	No	MS/MS	LC-MS/MS		
101	Azoxystrobin	0.0979	No	86.6	Same batch	5	ACN				Yes		None	MM-ML	HCB	ECD		Quechers	
101	Carbaryl	0.158	No	72.5	Same batch	15	AC	CH2Cl	PE		No	L/I part.	MM-ML		NPD				
101	Chlorpyrifos-methyl	0.208	No	73	Same batch	5	ACN				Yes		None	MM-ML		NPD		Quechers	
101	Deltamethrin (cis)	0.0423	No	101.8	Same batch	15	AC	CH2Cl	PE		No		MM-ML	HCB	ECD				
101	Fenitrothion	0.292	No	75	Same batch	5	ACN				Yes		None	MM-ML		NPD		Quechers	
101	Kresoxim-methyl	0.1850	No	66.7	Same batch	15	AC	CH2Cl	PE		No	L/I part.	MM-ML	HCB	ECD				
101	Lambda-cyhalothrin	0.0503	No	91.7	Same batch	15	AC	CH2Cl	PE		No	L/I part.	MM-ML	HCB	ECD				
101	Malathion	0.152	No	85	Same batch	5	ACN				Yes		None	MM-ML		NPD		Quechers	
101	Pirimiphos-methyl	0.126	No	77	Same batch	5	ACN				Yes		None	MM-ML		NPD		Quechers	
101	Triadimenol	1.271	No	117	Same batch	5	ACN				Yes		None	PS-ML		NPD		Quechers	
102	Azoxystrobin	0.38	No	101	Validation	5	ACN				Yes	No	DSPE	PS-ML	TPP	MS/MS	GC-MS/MS EN15662		
102	Carbaryl	0.31	No	131	Validation	5	ACN				Yes	No	DSPE	PS-ML	TPP	MS/MS	GC-MS/MS EN15662		
102	Chlorpyrifos-methyl	0.09	No	61	Validation	5	ACN				Yes	No	DSPE	PS-ML	TPP	MS/MS	GC-MS/MS EN15662		
102	Deltamethrin (cis)	0.11	No	99	Validation	5	ACN				Yes	No	DSPE	PS-ML	TPP	MS/MS	GC-MS/MS EN15662		
102	Fenitrothion	0.16	No	88	Validation	5	ACN				Yes	No	DSPE	PS-ML	TPP	MS/MS	GC-MS/MS EN15662		
102	Kresoxim-methyl	0.47	No	79	Validation	5	ACN				Yes	No	DSPE	PS-ML	TPP	MS/MS	GC-MS/MS EN15662		
102	Lambda-cyhalothrin	0.05	No	90	Validation	5	ACN				Yes	No	DSPE	PS-ML	TPP	MS/MS	GC-MS/MS EN15662		
102	Malathion	0.10	No	63	Validation	5	ACN				Yes	No	DSPE	PS-ML	TPP	MS/MS	GC-MS/MS EN15662		
102	Pirimiphos-methyl	0.05	No	90	Validation	5	ACN				Yes	No	DSPE	PS-ML	TPP	MS/MS	GC-MS/MS EN15662		
103	Azoxystrobin	0.253	Yes	102	St. Ad.	5	ACN				Yes	No	SPE	PS-ML	No	MSD	GC-MS		
103	Carbaryl	0.411	Yes	61.6	St. Ad.	5	ACN				Yes	No	SPE	PS-ML	No	MSD	GC-MS		
103	Chlorpyrifos-methyl	0.128	Yes	99.8	St. Ad.	5	ACN				Yes	No	SPE	PS-ML	No	MSD	GC-MS		
103	Fenitrothion	0.219	Yes	81.9	St. Ad.	5	ACN				Yes	No	SPE	PS-ML	No	MSD	GC-MS		
103	Fenpropimorph	2.76	Yes	96.1	St. Ad.	5	ACN				Yes	No	SPE	PS-ML	No	MSD	GC-MS		

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
103	Fluquinconazole	1.94	Yes	79.3	St. Ad.	5	ACN				Yes	No	SPE	PS-ML	No	MSD		GC-MS	
103	Flutriafol	2.33	Yes	85	St. Ad.	5	ACN				Yes	No	SPE	PS-ML	No	MSD		GC-MS	
103	Kresoxim-methyl	0.309	Yes	73.8	St. Ad.	5	ACN				Yes	No	SPE	PS-ML	No	MSD		GC-MS	
103	Malathion	0.101	Yes	102	St. Ad.	5	ACN				Yes	No	SPE	PS-ML	No	MSD		GC-MS	
103	Pirimiphos-methyl	0.094	Yes	92.7	St. Ad.	5	ACN				Yes	No	SPE	PS-ML	No	MSD		GC-MS	
103	Spiroxamine	1.86	Yes	107	St. Ad.	5	ACN				Yes	No	SPE	PS-ML	No	MSD		GC-MS	
103	Triadimenol	1.01	Yes	105	St. Ad.	5	ACN				Yes	No	SPE	PS-ML	No	MSD		GC-MS	
105	Chlorpyrifos-methyl	0.11	No	98	Same batch	30	AC	CH2Cl			Yes	No	SPE	PS-ML	No	ECD	Two columns internal method		
105	Deltamethrin (cis)	0.07	No	89	Same batch	30	AC	CH2Cl			Yes	No	SPE	PS-ML	No	ECD	Two columns internal method		
105	Fenitrothion	0.19	No	105	Same batch	30	AC	CH2Cl			Yes	No	SPE	PS-ML	No	NPD	Two columns internal method		
105	Fenpropimorph	1.3	No	85	Same batch	30	AC	CH2Cl			Yes	No	SPE	PS-ML	No	NPD	Two columns internal method		
105	Fluquinconazole	0.64	No	92	Same batch	30	AC	CH2Cl			Yes	No	SPE	PS-ML	No	NPD	Two columns internal method		
105	Kresoxim-methyl	0.29	No	90	Same batch	30	AC	CH2Cl			Yes	No	SPE	PS-ML	No	NPD	Two columns internal method		
105	Lambda-cyhalothrin	0.09	No	104	Same batch	30	AC	CH2Cl			Yes	No	SPE	PS-ML	No	ECD	Two columns internal method		
105	Pirimiphos-methyl	0.06	No	106	Same batch	30	AC	CH2Cl			Yes	No	SPE	PS-ML	No	NPD	Two columns internal method		
106	Azoxystrobin	0.344	No	105	Same batch	5	EtOAc				Yes		GPC SX-3	MM-ML		MSD	None		
106	Carbaryl	0.186	No	99	Same batch	5	EtOAc				Yes		GPC SX-3	MM-ML		MSD	None		
106	Carbendazim and benomyl	0.721	No	85	Same batch	5	EtOAc				Yes		GPC SX-3	MM-ML	No	MS/MS	None		
106	Chlorpyrifos-methyl	0.085	No	98	Same batch	5	EtOAc				Yes		GPC SX-3	MM-ML		MSD	None		
106	Fenitrothion	0.135	No	99	Same batch	5	EtOAc				Yes		GPC SX-3	MM-ML		MSD	None		
106	Fenpropimorph	1.207	No	99	Same batch	5	EtOAc				Yes		GPC SX-3	MM-ML		MSD	None		
106	Flutriafol	2.070	No	89	Same batch	5	EtOAc				Yes		GPC SX-3	MM-ML		MSD	None		
106	Isoproturon	0.120	No	84	Same batch	5	EtOAc				Yes		GPC SX-3	MM-ML	No	MS/MS	None		
106	Kresoxim-methyl	0.274	No	101	Same batch	5	EtOAc				Yes		GPC SX-3	MM-ML		MSD	None		
106	Lambda-cyhalothrin	0.078	No	110	Same batch	5	EtOAc				Yes		GPC SX-3	MM-ML		MSD	None		

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) EtOAc	Extraction solvent 2) EtOAc	Extraction solvent 3) EtOAc	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
106	Malathion	0.076	No	96	Same batch	5					Yes		GPC SX-3	MM-ML		MSD		None	
106 - Malaoxon		0.020	No	104	Same batch	5					Yes		GPC SX-3	MM-ML		MSD		None	
106	Pirimiphos-methyl	0.053	No	97	Same batch	5					Yes		GPC SX-3	MM-ML		MSD		None	
106	Spiroxamine	0.251	No	115	Same batch	5					Yes		GPC SX-3	MM-ML		MSD		None	
106 -Triadimefon		0.011	No	98	Same batch	5					Yes		GPC SX-3	MM-ML		MSD		None	
106	Triadimenol	2.628	No	98	Same batch	5					Yes		GPC SX-3	MM-ML		MSD		None	
108	Azoxystrobin	0.189	No	70	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MSD	GC-MS	EN15662	
108	Carbaryl	0.141	No	107	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MSD	GC-MS	EN15662	
108	Chlorpyrifos-methyl	0.0755	No	83	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MSD	GC-MS	EN15662	
108	Fenitrothion	0.215	No	112	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MSD	GC-MS	EN15662	
108	Kresoxim-methyl	0.322	No	109	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MSD	GC-MS	EN15662	
108	Lambda-cyhalothrin	0.0470	No	82	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MSD	GC-MS	EN15662	
108	Malathion	0.0900	No	93	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MSD	GC-MS	EN15662	
108	Pirimiphos-methyl	0.0535	No	88	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MSD	GC-MS	EN15662	
108	Triadimenol	1.34	No	129	Same batch	5	ACN				Yes	No	DSPE	MM-ML	No	MSD	GC-MS	EN15662	
109	Carbaryl	0.222	Yes automatic	88.34	Same batch	8	AC	CH ₂ Cl	PE		No	No	Other	MM-ML	TTP	NPD		None	
109	Chlorpyrifos-methyl	0.126	Yes automatic	94.28	Same batch	8	AC	CH ₂ Cl	PE		No	No	Other	MM-ML	TTP	NPD		None	
109	Deltamethrin (cis)	0.042	Yes automatic	101.53	Same batch	8	AC	CH ₂ Cl	PE		No	No	Other	MM-ML	Mirex	ECD		None	
109	Lambda-cyhalothrin	0.080	Yes automatic	99.61	Same batch	8	AC	CH ₂ Cl	PE		No	No	Other	MM-ML	Mirex	ECD		None	
109	Malathion	0.093	Yes automatic	91.61	Same batch	8	AC	CH ₂ Cl	PE		No	No	Other	MM-ML	TTP	NPD		None	
109	Pirimiphos-methyl	0.062	Yes automatic	93.91	Same batch	8	AC	CH ₂ Cl	PE		No	No	Other	MM-ML	TTP	NPD		None	
110	Carbaryl	0.0936	No	82	Same batch	5	ACN				Yes	No	DSPE	MM-ML	PCB153	MSD	GC-MS	EN 15662:2008	
110	Chlorpyrifos-methyl	0.114	No	104	Same batch	5	ACN				Yes	No	DSPE	MM-ML	PCB153	MSD	GC-MS	EN 15662:2008	
110	Deltamethrin (cis)	0.0360	No	75	Same batch	5	ACN				Yes	No	DSPE	MM-ML	PCB153	MSD	GC-MS	EN 15662:2008	
110	Fenitrothion	0.177	No	137	Same batch	5	ACN				Yes	No	DSPE	MM-ML	PCB153	MSD	GC-MS	EN 15662:2008	

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
110	Lambda-cyhalothrin	0.0636	No	89	Same batch	5	ACN				Yes	No	DSPE	MM-ML	PCB153	MSD		GC-MS	EN 15662:2008
110	Malathion	0.0946	No	115	Same batch	5	ACN				Yes	No	DSPE	MM-ML	PCB153	MSD		GC-MS	EN 15662:2008
110	Pirimiphos-methyl	0.0788	No	116	Same batch	5	ACN				Yes	No	DSPE	MM-ML	PCB153	MSD		GC-MS	EN 15662:2008
111	Chlorpyrifos-methyl	0.11	No	83	Same batch	20	AC	CH2Cl					SPE	PS-ML	Transnonachlore	ECD		Two columns	
111	Deltamethrin (cis)	0.07	No	81	Same batch	20	AC	CH2Cl					SPE	PS-ML	Transnonachlore	ECD		Two columns	
111	Lambda-cyhalothrin	0.07	No	97	Same batch	20	AC	CH2Cl					SPE	PS-ML	Transnonachlore	ECD		Two columns	
111	Pirimiphos-methyl	0.08	No	81	Same batch	20	AC	CH2Cl					SPE	PS-ML	Transnonachlore	ECD		Two columns	
111	Mepiquat (free cation)	0.10	No	94	Same batch	10	MeOH				Yes	No		PS-ML	Isotop. lab.1		MS/MS	LC-MS/MS	Method CRL
112	Azoxystrobin	0.350	Yes automatic	55	St. Ad.	20	ACN	MeOH	Other			No	SPE	PS-ML	Isotop. lab.1		MS/MS	LC-MS/MS	
112	Carbaryl	0.280	Yes automatic	50	St. Ad.	20	ACN	MeOH	Other			No	SPE	PS-ML	Isotop. lab.1		MS/MS	LC-MS/MS	
112	Carbendazim and benomyl	0.650	Yes automatic	55	St. Ad.	20	ACN	MeOH	Other			No	SPE	PS-ML	Isotop. lab.1		MS/MS	LC-MS/MS	
112	Deltamethrin (cis)	0.300	Yes automatic	55	St. Ad.	20	ACN	Water	PE			No	L/I part.	PS-ML	PCB	ECD		Two columns	
112	Fenitrothion	0.170	Yes automatic	90	St. Ad.	20	ACN	MeOH	Other			No	SPE	PS-ML	Isotop. lab.1		MS/MS	LC-MS/MS	
112	Fenpropimorph	2.450	Yes automatic	30	St. Ad.	20	ACN	MeOH	Other			No	SPE	PS-ML	Isotop. lab.1		MS/MS	LC-MS/MS	
112	Fluquinconazole	0.820	Yes automatic	50	St. Ad.	20	ACN	MeOH	Other			No	SPE	PS-ML	Isotop. lab.1		MS/MS	LC-MS/MS	
112	Flutriafol	1.450	Yes automatic	50	St. Ad.	20	ACN	MeOH	Other			No	SPE	PS-ML	Isotop. lab.1		MS/MS	LC-MS/MS	
112	Isoproturon	0.280	Yes automatic	60	St. Ad.	20	ACN	MeOH	Other			No	SPE	PS-ML	Isotop. lab.1		MS/MS	LC-MS/MS	
112	Kresoxim-methyl	0.450	Yes automatic	50	St. Ad.	20	ACN	MeOH	Other			No	SPE	PS-ML	Isotop. lab.1		MS/MS	LC-MS/MS	
112	Lambda-cyhalothrin	0.350	Yes automatic	55	St. Ad.	20	Other	Water	PE			No	L/I part.	PS-ML	PCB	ECD		Two columns	
112	Spiroxamine	1.210	Yes automatic	30	St. Ad.	20	ACN	MeOH	Other			No	SPE	PS-ML	Isotop. lab.1		MS/MS	LC-MS/MS	
112	Triadimenol	0.990	Yes automatic	60	St. Ad.	20	ACN	MeOH	Other			No	SPE	PS-ML	Isotop. lab.1		MS/MS	LC-MS/MS	
112	2,4-D (free acid)	0.430	Yes automatic	95	St. Ad.	20	ACN	MeOH	Other			No	SPE	PS-ML	Isotop. lab.1		MS/MS	LC-MS/MS	
112	Glyphosate	4.3	Yes		isotop. lab.	20	Other	Other	Other		Yes	No		MM-ML	Isotop. lab.1		MS/MS	LC-MS/MS	
113	Azoxystrobin	0.313	No	105	Same batch	5	ACN				Yes		None	MM-ML	Tris-dichlor-		MS/MS	GC-MS	
113	Carbaryl	0.214	No	104	Same batch	5	ACN				Yes		None	MM-ML	Tris-dichlor-		MS/MS	GC-MS	

LAB CODE	Pesticide	Reproto result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) ACN	Extraction solvent 2) ACN	Extraction solvent 3) ACN	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method
113	Carbendazim and benomyl	1.09	No	91	Same batch	5					Yes		None	MM-ML	Tris-dichlor-		MS/MS		
113	Chlorpyrifos-methyl	0.112	No	88	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML	Tris-dichlor-	MSD			
113	Deltamethrin (cis)	0.050	No	93	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML	Tris-dichlor-	ITD	GC-MS		
113	Fenitrothion	0.176	No	96	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML	Tris-dichlor-	MSD			
113	Fenpropimorph	2.05	No	82	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML	Tris-dichlor-	MSD	LC-MS/MS		
113	Fluquinconazole	0.902	No	105	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML	Tris-dichlor-	ITD	GC-MS		
113	Flutriafol	2.14	No	118	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML	Tris-dichlor-	MSD			
113	Isoproturon	0.164	No	106	Same batch	5	ACN				Yes		None Freez.	MM-ML	Tris-dichlor-		MS/MS	GC-MS	
113	Kresoxim-methyl	0.463	No	89	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML	Tris-dichlor-	MSD	LC-MS/MS		
113	Lambda-cyhalothrin	0.054	No	94	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML	Tris-dichlor-	ITD	GC-MS		
113	Malathion	0.111	No	97	Same batch	5	ACN				Yes		None Freez.	MM-ML	Tris-dichlor-	MS/MS	GC-MS		
113	Pirimiphos-methyl	0.075	No	105	Same batch	5	ACN				Yes		None Freez.	MM-ML	Tris-dichlor-	MS/MS	GC-MS		
113	Spiroxamine	1.09	No	86	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML	Tris-dichlor-	MSD	LC-MS/MS		
113	-Triadimefon	0.010	No	96	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML	Tris-dichlor-	MSD	LC-MS/MS		
113	Triadimenol	1.73	No	87	Same batch	5	ACN				Yes		DSPE Freez.	MM-ML	Tris-dichlor-	MSD	LC-MS/MS		
113	2,4-D (free acid)	0.423	No	103	Same batch	5	ACN				Yes		None	MM-ML	Tris-dichlor-		MS/MS		
114	Azoxystrobin	0.084	No	85	St. Ad.	15	AC	CH2Cl	PE		No	No	None	PS-ML	No		MS/MS	LC-MS/MS	miniluke
114	Carbaryl	0.026	No	67	St. Ad.	15	AC	CH2Cl	PE		No	No	None	PS-ML	No		MS/MS	LC-MS/MS	miniluke
114	Carbendazim and benomyl	0.38	No	89	St. Ad.	15	AC	CH2Cl	PE		No	No	None	PS-ML	No		MS/MS	LC-MS/MS	miniluke
114	Chlorpyrifos-methyl	0.041	No	83	St. Ad.	15	AC	CH2Cl	PE		No	No	None	PS-ML	TPP	MS/MS	GC-MS/MS	miniluke	
114	Deltamethrin (cis)	0.033	No	71	St. Ad.	15	AC	CH2Cl	PE		No	No	None	PS-ML	TPP	MS/MS	GC-MS/MS	miniluke	
114	Fenitrothion	0.077	No	93	St. Ad.	15	AC	CH2Cl	PE		No	No	None	PS-ML	TPP	MS/MS	GC-MS/MS	miniluke	
114	Fenpropimorph	0.36	No	63	St. Ad.	15	AC	CH2Cl	PE		No	No	None	PS-ML	No		MS/MS	LC-MS/MS	miniluke
114	Fluquinconazole	0.45	No	75	St. Ad.	15	AC	CH2Cl	PE		No	No	None	PS-ML	No		MS/MS	LC-MS/MS	miniluke
114	Flutriafol	0.35	No	106	St. Ad.	15	AC	CH2Cl	PE		No	No	None	PS-ML	No		MS/MS	LC-MS/MS	miniluke

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
114	Kresoxim-methyl	0.18	No	86	St. Ad.	15	AC	CH2Cl	PE		No	No	None	PS-ML	No		MS/MS	LC-MS/MS	miniluke
114	Malathion	0.027	No	91	St. Ad.	15	AC	CH2Cl	PE		No	No	None	PS-ML	TPP	MS/MS	GC-MS/MS	miniluke	
114	Pirimiphos-methyl	0.040	No	86	St. Ad.	15	AC	CH2Cl	PE		No	No	None	PS-ML	TPP	MS/MS	GC-MS/MS	miniluke	
114	Spiroxamine	0.19	No	71	St. Ad.	15	AC	CH2Cl	PE		No	No	None	PS-ML	No		MS/MS	LC-MS/MS	miniluke
115	Azoxystrobin	0.675	No	98.0	Other	6	AC	CH2Cl	PE		No	No	None	PS-SL	No	ECD		GC-TOF	SAR-2-04p
115	Carbaryl	0.139	No	106.5		0													
115	Carbendazim and benomyl	0.407	No	69.9		0													
115	Chlorpyrifos-methyl	0.171	No	110.5		0													
115	Deltamethrin (cis)	0.0760	No	101.4	Other	6	AC	CH2Cl	PE		No	No	None	PS-SL	No	ECD		GC-TOF	SAR-2-04p
115	Fenitrothion	0.168	No	99.1		0													
115	Fenpropimorph	3.22	No	100.8		0													
115	Fluquinconazole	0.852	No	82.5	Other	6	AC	CH2Cl	PE		No	No	None	PS-SL	No	ECD		GC-TOF	SAR-2-04p
115	Flutriafol	2.80	Yes	47.9		0													
115	Kresoxim-methyl	0.483	No	119.0	Other	6	AC	CH2Cl	PE		No	No	None	PS-SL	No	ECD		GC-TOF	SAR-2-04p
115	Lambda-cyhalothrin	0.0600	No	78.7	Other	6	AC	CH2Cl	PE		No	No	None	PS-SL	No	ECD		GC-TOF	SAR-2-04p
115	Malathion	0.193	No	98.2		0													
115	Pirimiphos-methyl	0.0812	No	84.7		0													
115	Spiroxamine	2.11	No	69.9	Other	6	AC	CH2Cl	PE		No	No	None	PS-SL	No	ECD		GC-TOF	SAR-2-04p
115	Triadimenol	1.64	No	74.9		0													
116	Azoxystrobin	0.270	No	65	Same batch	10	ACN				Yes	No	DSPE PSA C18	MM-ML	No		MS/MS	LC-MS/MS	Quechers
116	Carbaryl	0.141	No	82	Same batch	10	ACN				Yes	No	DSPE PSA C18	MM-ML	No		MS/MS	LC-MS/MS	Quechers
116	Carbendazim and benomyl	0.975	No	60	Same batch	10	ACN				Yes	No	DSPE PSA C18	MM-ML	No		MS/MS	LC-MS/MS	Quechers
116	Chlorpyrifos-methyl	0.060	No	78	Same batch	10	ACN				Yes	No	DSPE PSA C18	MM-ML	No	ITD		GC-MS	Quechers
116	Fenitrothion	0.116	No	83	Same batch	10	ACN				Yes	No	DSPE PSA C18	MM-ML	No	ITD		GC-MS	Quechers
116	Flutriafol	1.87	No	89	Same batch	10	ACN				Yes	No	DSPE PSA C18	MM-ML	No		MS/MS	LC-MS/MS	Quechers

LAB CODE	Pesticide	Reported result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1) ACN	Extraction solvent 2) ACN	Extraction solvent 3) ACN	ASE	Water addition	Hydrolysis	Clean up	Calibration	STD	GC detector	HPLC detector	Confirmation	Reference method	
116	Kresoxim-methyl	0.396	No	90	Same batch	10					Yes	No	DSPE PSA C18	MM-ML	No	ITD	GC-MS	Quechers		
116	Lambda-cyhalothrin	0.285	No	66	Same batch	10	ACN				Yes	No	DSPE PSA C18	MM-ML	No	ITD	GC-MS	Quechers		
116	Malathion	0.051	No	72	Same batch	10	ACN				Yes	No	DSPE PSA C18	MM-ML	No	ITD	GC-MS	Quechers		
116	Pirimiphos-methyl	0.053	No	82	Same batch	10	ACN				Yes	No	DSPE PSA C18	MM-ML	No	ITD	GC-MS	Quechers		
116	Spiroxamine	1.07	No	77	Same batch	10	ACN				Yes	No	DSPE PSA C18	MM-ML	No		MS/MS	LC-MS/MS	Quechers	
116	Triadimenol	1.05	No	76	Same batch	10	ACN				Yes	No	DSPE PSA C18	MM-ML	No	ITD	GC-MS	Quechers		
117	Azoxystrobin	0.086	No	71	Same batch	10	ACN				No	No	None	MM-ML	No		MS/MS	LC-MS/MS	QuEChERS	
117	Carbaryl	0.126	No	74	Same batch	10	ACN				No	No	None	MM-ML	No		MS/MS	LC-MS/MS	QuEChERS	
117	Carbendazim and benomyl	0.942	No	65	Same batch	10	ACN				No	No	None	MM-ML	No		MS/MS	LC-MS/MS	QuEChERS	
117	Fenpropimorph	1.28	No	100	Same batch	10	ACN				No	No	None	MM-ML	No		MS/MS	LC-MS/MS	QuEChERS	
117	Fluquinconazole	0.364	No	85	Same batch	10	ACN				No	No	None	MM-ML	No		MS/MS	LC-MS/MS	QuEChERS	
117	Flutriafol	0.295	No	94.0	Same batch	10	ACN				No	No	None	MM-ML	No		MS/MS	LC-MS/MS	QuEChERS	
117	Kresoxim-methyl	0.198	No	98.5	Same batch	10	ACN				No	No	DSPE PSA	MM-ML	No	ITD	Other	QuEChERS		
117	Pirimiphos-methyl	0.056	No	119.5	Same batch	10	ACN				No	No	DSPE PSA	MM-ML	No	ITD	Other	QuEChERS		
117	Spiroxamine	0.579	No	109	Same batch	10	ACN				No	No	None	MM-ML	No		MS/MS	LC-MS/MS	QuEChERS	
117	Triadimenol	0.467	No	86	Same batch	10	ACN				No	No	None	MM-ML	No		MS/MS	LC-MS/MS	QuEChERS	
119	Azoxystrobin	0.303	No	120	Same batch	5	ACN				Yes	No	None	St. Ad.		MSD	GC-MS	QuEChERS		
119	Carbaryl	0.145	No	123	Same batch	5	ACN				Yes	No	None	St. Ad.		MSD	GC-MS	QuEChERS		
119	Carbendazim and benomyl	5.500	No	105	Same batch	2	ACN				Yes	No	None	St. Ad.			MS/MS	LC-MS/MS	QuEChERS	
119	Chlorpyrifos-methyl	0.146	No	118	Same batch	5	ACN				Yes	No	None	St. Ad.		MSD	GC-MS	QuEChERS		
119	Fenitrothion	0.144	No	92	Same batch	5	ACN				Yes	No	None	St. Ad.		MSD	GC-MS	QuEChERS		
119	Fenpropimorph	2.403	No	112	Same batch	5	ACN				Yes	No	None	St. Ad.		MSD	GC-MS	QuEChERS		
119	Fluquinconazole	0.684	No	100	Same batch	5	ACN				Yes	No	None	St. Ad.		MSD	GC-MS	QuEChERS		
119	Flutriafol	2.718	No	124	Same batch	5	ACN				Yes	No	None	St. Ad.		MSD	GC-MS	QuEChERS		
119	Isoproturon	0.210	No	97	Same batch	2	ACN				Yes	No	None	St. Ad.			MS/MS	LC-MS/MS	QuEChERS	

LAB CODE	Pesticide	Reprotox result, mg/kg	Recovery corr.	Recovery %	Recovery approach	Sample weight g	Extraction solvent 1 ¹⁾	Extraction solvent 2 ¹⁾	Extraction solvent 3 ¹⁾	ASE	Water addition	Hydrolysis	Clean up	Calibration	ISTD	GC detector	HPLC detector	Confirmation	Reference method
119	Kresoxim-methyl	0.465	No	126	Same batch	5	ACN				Yes	No	None	St. Ad.		MSD	GC-MS	QuEChERS	
119	Lambda-cyhalothrin	0.059	No	110	Same batch	5	ACN				Yes	No	None	St. Ad.		MSD	GC-MS	QuEChERS	
119	Malathion	0.119	No	103	Same batch	5	ACN				Yes	No	None	St. Ad.		MSD	GC-MS	QuEChERS	
119	Pirimiphos-methyl	0.078	No	114	Same batch	5	ACN				Yes	No	None	St. Ad.		MSD	GC-MS	QuEChERS	
119	Spiroxamine	1.205	No	107	Same batch	5	ACN				Yes	No	None	St. Ad.		MSD	GC-MS	QuEChERS	
119	Triadimenol	2.088	No	123	Same batch	5	ACN				Yes	No	None	St. Ad.		MSD	GC-MS	QuEChERS	
121	Azoxystrobin	0.201	No	65	Same batch	5	AC	CH ₂ Cl	PE		Yes	No	SPE Florisil	MM-ML		ECD	GC-MS	Luke modified	
121	Chlorpyrifos-methyl	0.120	No	100	Same batch	5	AC	CH ₂ Cl	PE		Yes	No	SPE Florisil	MM-ML		FPD	GC-MS	Luke modified	
121	Deltamethrin (cis)	0.052	No	90	Same batch	5	AC	CH ₂ Cl	PE		Yes	No	SPE Florisil	MM-ML		ECD	GC-MS	Luke modified	
121	Fenitrothion	0.175	No	100	Same batch	5	AC	CH ₂ Cl	PE		Yes	No	SPE Florisil	MM-ML		FPD	GC-MS	Luke modified	
121	Kresoxim-methyl	0.327	No	85	Same batch	5	AC	CH ₂ Cl	PE		Yes	No	SPE Florisil	MM-ML		ECD	GC-MS	Luke modified	
121	Lambda-cyhalothrin	0.053	No	90	Same batch	5	AC	CH ₂ Cl	PE		Yes	No	SPE Florisil	MM-ML		ECD	GC-MS	Luke modified	
121	Malathion	0.141	No	115	Same batch	5	AC	CH ₂ Cl	PE		Yes	No	SPE Florisil	MM-ML		FPD	GC-MS	Luke modified	
121	Pirimiphos-methyl	0.073	No	105	Same batch	5	AC	CH ₂ Cl	PE		Yes	No	SPE Florisil	MM-ML		FPD	GC-MS	Luke modified	
121	Triadimenol	1.13	No	85	Same batch	5	AC	CH ₂ Cl	PE		Yes	No	SPE Florisil	MM-ML		ECD	GC-MS	Luke modified	
122	Chlorpyrifos-methyl	0.098	No	63.7	Same batch	5	AC	CH ₂ Cl			Yes	No	SPE	MM-ML		NPD	GC-MS		
122	Fenitrothion	0.138	No	80.4	Same batch	5	AC	CH ₂ Cl			Yes	No	SPE	MM-ML		NPD	GC-MS		
122	Malathion	0.087	No	82.2	Same batch	5	AC	CH ₂ Cl			Yes	No	SPE	MM-ML		NPD	GC-MS		
122	Pirimiphos-methyl	0.125	No	108.2	Same batch	5	AC	CH ₂ Cl			Yes	No	SPE	MM-ML		NPD	GC-MS		

1) AC: Acetone; ACN: Acetonitrile; Cy-He: Cyclohexane; CH₂Cl₂: Dichlormethane; EOH: Ethanol; EtOAc: Ethyl Acetate; HEX: Hexane; MeOH: Methanol; ISO: Isooctane; PE: Petroleum Ether

2) isotop. Lab1: isotopically labeled target pesticide; isotop. lab2: isotopically labeled other substance

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

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



General protocol for EU proficiency Tests for Pesticide Residues in Food and Feed

Introduction

This protocol contains general procedures valid for all European Union proficiency tests (EUPTs) organised on behalf of the European Commission, Health & Consumer Protection Directorate-General (DG-SANCO) by the four Community Reference Laboratories (CRLs) for pesticide residues in food and feed. These EUPTs are directed at all National Reference Laboratories (NRLs) and Official Laboratories (OfLs) in the EU Member States. Laboratories outside this CRL/NRL/OfL-Network¹ may be permitted to participate on a case-by-case basis after consultation with DG SANCO.

The following four CRLs for pesticides were appointed by DG-SANCO based on regulation 882/2004/EC²:

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

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

According to Regulation 396/2005/EC³ all laboratories analysing samples for the official controls on pesticide residues shall participate in the Community proficiency test(s)

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

² Regulation (EC) No 882/2004 of the European Parliament and of the Council on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules. Published at OJ of the EU L191 of 28.05.2004



organised by the Commission. The aim of these EUPTs is to obtain information regarding the quality, accuracy and comparability of the pesticide residue data in food and feed sent to the European Commission within the framework of the national control programmes and the co-ordinated multiannual community control programme. Participating laboratories will be provided with an assessment of their analytical performance and the reliability of their data - compared to the other participating laboratories.

EUPT-organisation

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

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

A common Scientific Committee entailing the following two subgroups:

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

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

The role of the AG is to help the organisers in making decisions concerning the design of the EUPT: selection of pesticides to be included in the Target Pesticide List (see below), the establishment of the minimum required reporting levels (MRRLs), the evaluation and statistical treatment of the results and the drafting of the protocol and final report. The QCG has the additional function of supervising the quality of the EUPT and to assisting the CRLs with confidential aspects such as the choice of the pesticides, and levels to be present in the test material.

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

³ Regulation (EC) No 396/2005, published at OJ of the EU L70 of 16.03.2005, as last amended by Regulation 839/2008 published at OJ of the EU L234 of 30.08.2008.



Confidentiality:

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

Communication

The official language used in all EUPTs is English.

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

Announcement

The announcement of the individual EUPT will be issued at least 3 months before the test material is distributed to the laboratories. The announcement will be published on the CRL portal and distributed via mail to the NRL/OfL mailing list available to the CRLs. The announcement will contain an invitation letter, details on how to register and where to locate additional related documents, and some preliminary information on the specific protocol such as the tentative calendar, the name of the commodity expected to be used, and the tentative Target Pesticide List.

Specific Protocol

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



General procedures for reporting results

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

Correction of results for recovery

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

Evaluation of the Results

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

– *False Positives*

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



positive result will be made on a case-by-case basis. Any results reported that are lower than the MRRL will not be considered as false positives, even though these results should not have been reported.

- ***False Negatives***

These are results for pesticides reported by the laboratories as “analysed” but that no numerical values were given, although they were used by the Organiser to treat the test material and were detected by the majority of participants at or above the MRRL.

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

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

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

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

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

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

- ***z-scores***

This parameter is calculated using the following formula:



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

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

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

z-scores will be interpreted in the following way:

$|z| \leq 2$ Acceptable

$2 < |z| \leq 3$ Questionable

$|z| > 3$ Unacceptable

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

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

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

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

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

- ***Combined z-scores***



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

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

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

n = number of reported results

So for each laboratory:

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

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

$Z \leq 2$ Good

$2 < Z \leq 3$ Satisfactory

$Z > 3$ Unsatisfactory

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



Publication of results

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

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

Disclaimer

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



Specific protocol for EU Proficiency Test for Pesticide Residues in Cereals, EUPT-C4 (2010)

Introduction

This protocol is complementary to the General protocol for EU proficiency tests for pesticide residues in food and feed. The proficiency test covers both pesticides that are determined using both Multi Residue Methods and Single Residue Methods. However, only those pesticides that are amenable to Multi Residue methods are mandatory.

Test material

This proficiency test involves pesticide residues analysis in rye.

The rye was grown in Denmark in 2009 and pesticides were applied in the field. Following harvest, the rye was also spiked with some additional pesticides

Analytical parameters

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

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

Amount of Test Material

Participants will receive:

- approximately 150 g of rye test material with incurred and spiked pesticides and
- approximately 150 g of blank (untreated) rye test material.

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

Shipment of Test Materials

The shipment of the test materials will take place on 15 February 2010. The organisers will aim to ensure that all packages arrive at the same time. An information message will be sent out by e-mail before shipment.

Instructions on Test Material Handling

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

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

Test Material Receipt and Acceptance

Once the laboratory has received the test materials they must report to the organiser, via the result submission website (subpage 0), by filling in the date of receipt, the condition of the test material, and its acceptance. The deadline for acceptance is the 19 February 2010. If the laboratory does not respond before this deadline the organiser will assume that they have received and accepted the test material. If any participants have not received the test material by 19 February 2010, they must inform the organiser immediately by e-mail (crlcereal@food.dtu.dk).

Reporting Results

To report their results, laboratories must access the **Result Submission Website** (sub-pages 1 and subpage 2). A link to the website is uploaded to the CIRCA platform (<https://fis-vl.bund.de/Public/irc/fis-vl/Home/main?f=login&referer=http%3A%2F%2Ffis-vl.bund.de%2FMembers%2Firc%2Ffis-vl%2FHome%2Fmain>). Here you will also find the **Result Submission Guide** with specific instructions on how to enter the data.

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

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

All results must be reported on the online result submission website by 15 March 2010, at the latest. The website will not be accessible after this date, and any results reported after the deadline will not be included in the statistical treatment, or in the final report.

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

Significant Figures:

Residue levels <0.010 mg/kg;

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

Residue levels ≥ 0.010 mg/kg;

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

Results should not be reported where a pesticide was not detected, or was detected below the RL (Reporting Limit) of the laboratory, or below the MRRL.

Result should be reported both with and without correction for recovery.

Reporting Information on Analytical Methodology

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

Time Table

CRL	Release of "Specific PT-Protocol"	January 2010
Participants	Deadline for registration for the EUPT-C4	31 January 2010
CRL	Deadline for "guide to Result Submission Website"	7 February
CRL	Test material distribution and information to the laboratories regarding upcoming shipment	15 February 2010
Participants	Confirmation of test material receipt	19 February 2010
Participants	Reporting of test results and method information	15 March 2010
CRL	Dispatch of a preliminary report to all participants (only results, no statistical treatment)	May 2010
CRL	Dispatch of the final report as pdf-file	November 2010

Participation fee

There is a fee of EUR 150.00 for shipping and handling to all participants. An invoice will be sent to the 'invoice address' stated in connection with the registration.

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

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Annex 1

Target Pesticide List for the EUPT-C4 2010

(last updated 15.01.2010)

Eight new pesticides have been added to the Target Pesticide List from EUPT-C3 (2009), and are marked in bold.

SRM pesticides in the table on page 4 are optional.

Pesticides	MRRL
MRM-compounds	mg/kg
*Azoxystrobin	0.01
*Bifenthrin	0.01
Carbaryl	0.01
*Carbendazim and benomyl (carbendazim + benomyl, expressed as carbendazim)	0.01
Chlorothanlonil	0.01
*Chlorpyrifos	0.01
*Chlorpyrifos-methyl	0.01
*Cypermethrin (sum of isomers)	0.01
-Alpha-cypermethrin	0.01
*Cyproconazole	0.01
Cyprodinil	0.01
*Deltamethrin (cis-deltamethrin)	0.01
Diazinon	0.01
*Dichlorvos	0.01
*Difenoconazole	0.01
*Endosulfan (sum of alpha- and beta-isomers and endosulfan-sulphate expressed as endosulfan)	0.01
-Endosulfan α	0.01
-Endosulfan β	0.01
-Endosulfan sulfate	0.01

Pesticides	MRRL
*Epoxiconazole	0.01
Fenvalerate and Esfenvalerate (Sum of RR/SS and RS/SR isomers)	0.01
Fenbuconazole	0.01
Fenhexamid	0.01
Fenitrothion	0.01
*Fenpropimorph	0.01
*Fludioxonil	0.01
Fluquinconazole	0.01
Flusilazole	0.01
*Flutriafol	0.01
Hexaconazole	0.01
*Imazalil	0.01
*Iprodione	0.01
Isoproturon	0.01
*Kresoxim-methyl	0.01
*Lambda-cyhalothrin	0.01
*Lindane (gamma- isomer of hexachlorocyclohexane (HCH))	0.01
*Malathion (Malathion + Malaoxon, expressed as Malathion)	0.01
<i>-Malathion</i>	0.01
<i>-Malaoxon</i>	0.01
Metconazole	0.01
*Methacrifos	0.01
*Methomyl and Thiodicarb (Methomyl + Thiodicarb, expressed as Methomyl)	0.01
<i>-Methomyl</i>	0.01
<i>-Thiodicarb</i>	0.01
Metribuzin	0.01
*Parathion	0.01
*Penconazole	0.01
Pendimethalin	0.01
*Permethrin	0.01

Pesticides	MRRL
*Pirimicarb (sum of pirimicarb and desmethyl pirimicarb, expressed as pirimicarb)	0.01
-Pirimicarb	0.01
-Desmethyl pirimicarb	0.01
*Pirimiphos-methyl	0.01
*Prochloraz (parent compound only)	0.01
*Procymidone	0.01
*Propiconazole	0.01
Pyraclostrobin	0.01
Pyrimethanil	0.01
Spiroxamine	0.01
*Tebuconazole	0.01
Tebufenozide	0.01
*Thiabendazole	0.01
*Thiophanate-methyl	0.01
Triadimefon and triadimenol (sum of triadimefon and triadimenol)	0.01
-Triadimefon	0.01
-Triadimenol	0.01
Triazophos	0.01
*Trifloxystrobin	0.01
Trifluralin	0.01
*Triticonazole	0.01
Vinclozolin (only parent compound)	0.01

Pesticides	MRRL
SRM-compounds - optional	mg/kg
2,4-D (free acid)	0.02
2,4-D (following alkaline hydrolysis)	0.02
Dicamba (free acid)	0.02
Dicamba (following alkaline hydrolysis)	0.02
Dichlorprop (2,4-DP) including Dichlorprop-P (free acid)	0.02
Dichlorprop (2,4-DP) including Dichlorprop-P (following alkaline hydrolysis)	0.02
Fluazifop including Fluazifop-P (free acid)	0.02
Fluazifop including Fluazifop-P (following alkaline hydrolysis)	0.02
Fluroxypyr (free acid)	0.02
Fluroxypyr (following alkaline hydrolysis)	0.02
Haloxyfop including Haloxyfop-R (free acid)	0.02
Haloxyfop including Haloxyfop-R (following alkaline hydrolysis)	0.02
MCPA (free acid)	0.02
MCPA (following alkaline hydrolysis)	0.02
Mecoprop (MCPP) including Mecoprop-P (free acid)	0.02
Mecoprop (MCPP) including Mecoprop-P (following alkaline hydrolysis)	0.02
Glyphosate	0.02
Chlormequat (free cation)	0.02
Mepiquat (free cation)	0.02

All the SRM pesticides included in the EUPT-C4 Target Pesticide List are optional

This means that the results will not be used in the classification of laboratories and the results used mainly for scientific evaluation, for example to support the ongoing discussions concerning the legal residue definitions of acid pesticides. However, the main purpose will be to give participants an opportunity to test their analytical performance for these particular pesticides.

**EU Reference Laboratory
on Cereals & Feedingstuff**

**Report on
Proficiency Test on
incurred and spiked
pesticides in oat**



**Final report
December 2010**

**DTU National Food Institute
Technical University of Denmark**