

# **PROFICIENCY TEST 7, 2005**

## **Pesticide Residues in Grape Homogenate**

### **Final Report**

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**EUROPEAN COMMISSION'S PROFICIENCY TEST 7**  
**ON PESTICIDE RESIDUES IN FRUIT AND VEGETABLES**  
**2005**

The 7<sup>th</sup> European Commission's Proficiency Test was performed in 2005 using a grape homogenate. The grapes were grown in the south of Spain, in Almeria, and were treated with a post harvest treatment where commercial formulations were applied using a microspray technique. Sixteen pesticides were used for the treatments. Participating laboratories were provided with a 'blank' grape homogenate as well as the treated test material.

One hundred and twenty eight laboratories agreed to participate in this proficiency test.

The median values of the analytical data submitted were used to obtain the assigned (true) concentrations for each of the sixteen residue pesticides present. A fit-for-purpose (FFP) target standard deviation of 25%, based on the experience of the Advisory Group, was chosen to calculate the target standard deviations ( $\sigma$ ) as well as the z-scores of each of the 16 compounds present.

For the assessment of the overall laboratory performance the criteria Weight Sum of z-Score (WSZ) used in the last proficiency test has been applied. Only laboratories that fulfilled the criteria of detecting at least fourteen of the sixteen pesticides (~90%) with no false positives reported, have been classified as having 'sufficient scope', and have therefore been placed in Category A. Within this category, the laboratories have been classified as 'good', 'satisfactory' or 'unsatisfactory'.

All the other laboratories have been placed in Category B, and classified as having 'insufficient scope'. For laboratories in Category B, individual z-scores were calculated, but their overall performance has not been assessed, although this year they have been ordered by the number of pesticides reported and the number of acceptable z-scores achieved.

Furthermore, classical procedures for summing z-scores (SSZ and RSZ) were employed using the individual z-scores of the participating laboratories.

## **1. INTRODUCTION**

The Council Directives (86/362/EEC and 90/642/EEC) provide for the organization and financial support for regular proficiency testing of the laboratories that perform analyses for their official national monitoring programmes. These proficiency tests are performed in order to assure the quality, accuracy and comparability of the residue data sent by EU Member States to the European Commission and to the other Member States on an annual basis. The EU Commission's Proficiency Tests 1-5 were carried out from 1996-2003, and were all organized by the Swedish National Food Administration. This year, as in 2004, the University of Almeria, Spain, organized the European Proficiency Test. Participation in this 7<sup>th</sup> European Proficiency Test was open to all official national or regional analytical laboratories involved in the determination of pesticide residues in fruit and vegetables in Member States of the EU and, additionally, laboratories from Iceland, Norway and Romania were included by invitation.

A total of one hundred and twenty eight laboratories agreed to participate in the 7<sup>th</sup> Proficiency Test. The test material, 300 g of grape homogenate containing pesticide residues, together with 300 g of blank grape homogenate, were shipped to participants on the 5<sup>th</sup>, 6<sup>th</sup> and 11<sup>th</sup> of April, 2005. The deadline for submission of results to the organiser was the 20<sup>th</sup> of May 2005. The participants were provided with a list of sixty-five pesticides (Annex 1), which might be present in the test material and they were asked to determine the levels of all the pesticide residues they detected. Participants were also asked to analyse the blank test material and report residues of any pesticide they found which was included in the list. This blank material was intended for use in performing recovery experiments with the pesticides found in the test material, and if necessary, for the preparation of matrix-matched calibration standards.

## **2. TEST MATERIALS**

### **2.1 Analytical methods**

Two analytical methods, described briefly below, as well as other procedures, were used by the Organisers for the homogeneity and stability tests performed at the laboratory of Coexphal (Almeria):

- GC method [1]:  
ethyl acetate extraction in the presence of sodium sulfate, filtration, addition of more sodium sulfate, evaporation, re-dissolution in cyclohexane and determination by GC-MS/MS.
- LC method [2]:  
ethyl acetate extraction in the presence of sodium sulfate and addition of sodium hydroxide, filtration, addition of more sodium sulfate, evaporation, re-dissolution in methanol, and determination by LC-MS/MS.

Acetamiprid, carbaryl, imidacloprid, methomyl and thiabendazole were determined using the LC-MS/MS method. All other pesticides, were analysed using the GC method. For confirmation purposes, MS/MS spectra were used.

### **2.2 Preparation of the treated test material**

Before preparing the test material, the pesticides and suitable residue levels for the study were selected following recommendations made by the Advisory Group, which had been specifically appointed for Proficiency Test 7. The grapes were grown in the south of Spain, in Almeria. One hundred and fifty kilograms of grapes were sampled and treated post-harvest with a commercial formulation dissolved in water and applied to the grapes using a microspray. Different formulations were applied for each of the pesticides allowing one hour to elapse between applications. A portion was taken and analysed to check the residue levels present in the material. When the grapes contained residue levels close to those recommended by the Advisory Group they were frozen and chopped using liquid nitrogen and a mincer, then mixed in a constantly spinning container especially designed and built by the organiser, until a homogeneous material was obtained. Portions of 300 g, of the well-mixed homogenate, were weighed out into screw-capped polyethylene plastic bottles, sealed, and stored in a freezer at about - 20 °C prior to distribution to participants.

### **2.3 Preparation of the 'blank' test material**

The grapes to be used for the production of the blank test material were organically grown in the same area as the test material, in Almeria, in the south of Spain. A homogenate was prepared in the same way as the treated test material described above. Some very low level traces of pesticides were found in the blank: carbendazim, carbaryl, dimethoate, chlorpyriphos-methyl, pyridaben and bifenthrin. Bifenthrin was not included in the Pesticide List provided by the Organizer. All were found at concentrations below 0.01 mg/Kg.

## 2.4 Homogeneity test

Ten bottles were randomly chosen from those stored in the freezer and analyses were performed on duplicate portions taken from each bottle. The sequence of analyses was determined using a table of randomly generated numbers. The injection sequence of the 20 extracts analysed by GC and LC was also randomly chosen. The quantification by GC and LC was performed using a 3-point calibration curve constructed from matrix-matched standards. One standard mixture was used, for both GC and LC.

The statistical evaluation was performed according to the International Harmonized Protocol published by IUPAC, ISO and AOAC [3]. The individual residue data from the homogeneity tests are given in the Appendix 1. The results of the statistical analyses are given in Tables 1 A-B. The acceptance criteria for the test material to be sufficiently homogenous for the proficiency test were that  $F_{\text{critical}} > F$  for ( $p = 0.05$ ), and that the between sampling standard deviation  $S_s$  was lower than the analytical  $S_a$ , for all pesticides.

Table 1 A. Statistical evaluation of the homogeneity test data (n=20 analyses)

	Acetamiprid	Carbaryl	Cyprodinil	Diazinon	Dimethoate	Fenhexamid	Fludioxonil	Imidacloprid
Mean (mg/Kg)	0.346	1.65	0.443	0.288	0.164	0.671	0.229	0.562
$S_a$ (mg/Kg)	0.0206	0.0208	0.0208	0.00267	0.0239	0.113	0.0232	0.0284
$F_{\text{critical}}$	3.02	3.02	3.02	3.02	3.02	3.02	3.02	3.02
$F$	2.48	2.00	2.89	2.79	1.45	1.37	2.06	1.87
$S_s$ (mg/Kg)	0.0177	0.0147	0.0202	0.00252	0.0113	0.0481	0.0169	0.0188
Pass/Fail	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass

Ss: Between Sampling Standard Deviation

Sa: Analytical Standard Deviation

Table 1 B. Statistical evaluation of the homogeneity test data (n=20 analyses)

	Iprodine	Kresoxim-methyl	Methomyl	Monocrotophos	Procymidone	Pyrimethanil	Tetraconazole	Thiabendazole
Mean (mg/Kg)	0.522	0.432	0.160	0.658	2.18	0.138	0.059	0.638
Sa. (mg/Kg)	0.0262	0.0803	0.0250	0.0278	0.221	0.0107	0.00698	0.0107
F critical	3.02	3.02	3.02	3.02	3.02	3.02	3.02	3.02
F	1.99	1.08	1.18	1.58	1.97	2.63	1.74	2.63
Ss. (mg/Kg)	0.0185	0.0159	0.00742	0.0150	0.154	0.00966	0.00425	0.00966
Pass/Fail	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass

Ss: Between Sampling Standard Deviation

Sa: Analytical Standard Deviation

## 2.5 Stability tests

The two analytical methods described briefly above (in section 2.1) were also used for the stability tests.

The tests were performed on two occasions. On each occasion, a single bottle stored in the freezer at -20°C was chosen randomly and duplicate analyses were performed.

The two occasions were:

- Day 1: coinciding with the first sample shipment, which took place 5<sup>th</sup>/6<sup>th</sup> of April 2005.
- Day 2: shortly after the deadline for reporting results, 20<sup>th</sup> May 2005.

The individual results are given in Tables 2 A-B. In general, these tests did not show any significant decrease in the levels of the pesticides and demonstrated that the pesticide residues present in the test material remained stable for the duration of the proficiency test.

Table 2 A. Statistical test for analytical precision and to demonstrate stability

	Acetamiprid	Carbaryl	Cyprodinil	Diazinon	Dimethoate	Fenhexamid	Fludioxonil	Imidacloprid
Day 1 (mg/Kg) (1 <sup>st</sup> sample)	0.320	1.63	0.440	0.285	0.137	0.700	0.240	0.550
Day 1 (mg/Kg) (2 <sup>nd</sup> sample)	0.312	1.63	0.435	0.298	0.140	0.710	0.235	0.553
Mean 1 (mg/Kg)	0.316	1.63	0.438	0.292	0.139	0.705	0.238	0.552
Day 2 (mg/Kg) (1 <sup>st</sup> sample)	0.316	1.62	0.434	0.283	0.134	0.699	0.238	0.542
Day 2 (mg/Kg) (2 <sup>nd</sup> sample)	0.310	1.63	0.430	0.295	0.137	0.698	0.232	0.551
Mean 2 (mg/Kg)	0.313	1.63	0.432	0.289	0.136	0.699	0.235	0.547
(M1-M2)/M1	0.009	0.002	0.013	0.009	0.022	0.009	0.011	0.009
%	0.95	0.21	1.26	0.86	2.17	0.92	1.05	0.91

Table 2 B. Statistical test for analytical precision and to demonstrate stability

	Iprodine	Kresoxim-methyl	Methomyl	Monocrotophos	Procymidone	Pyrimethanil	Tetraconazole	Thiabendazole
Day 1 (mg/Kg) (1 <sup>st</sup> sample)	0.528	0.464	0.168	0.647	1.91	0.150	0.063	0.654
Day 1 (mg/Kg) (2 <sup>nd</sup> sample)	0.520	0.450	0.149	0.640	1.90	0.155	0.065	0.658
Mean 1 (mg/Kg)	0.524	0.457	0.159	0.644	1.91	0.153	0.064	0.656
Day 2 (mg/Kg) (1 <sup>st</sup> sample)	0.521	0.46	0.164	0.640	1.91	0.148	0.060	0.653
Day 2 (mg/Kg) (2 <sup>nd</sup> sample)	0.517	0.441	0.142	0.635	1.89	0.150	0.061	0.655
Mean 2 (mg/Kg)	0.519	0.451	0.153	0.638	1.90	0.149	0.061	0.654
(M1-M2)/M1	0.010	0.014	0.035	0.009	0.003	0.023	0.055	0.003
%	0.95	1.42	3.47	0.93	0.29	2.30	5.47	0.30

## **2.6 Distribution of test material and protocol to participants**

One bottle of incurred test sample and one bottle of 'blank' material were shipped to each participant in boxes containing dry ice. The samples were sent on the 5<sup>th</sup>, 6<sup>th</sup> and 11<sup>th</sup> of April 2005.

Before sample shipment, the laboratories received full instructions (Annex 1) for the receipt, storage and analysis of the test materials, although they were encouraged to use their normal sample receipt procedure and methods of analysis. These instructions were uploaded onto the EUPT 7 web page constructed especially for this Proficiency Test. A password was required to enter a restricted zone where the Protocol and the Pesticide List with the Minimum Required Performance Level (MRPL) established by the Organizer could be found. This information was notified by e-mail to all participant laboratories at the same time as they were informed that their application form for participation had been accepted. This ensured that confidentiality was maintained throughout the entire Proficiency Test 7.

### **3. STATISTICAL METHODS**

#### **3.1 Background**

##### **3.1.1 Proficiency Tests 1-5**

For the previous Proficiency Tests 1-5, different methods for the estimation of the assigned values and standard deviations (SDs) of the analytical data were used. The arithmetic mean values, after elimination of outliers, were used for Proficiency Tests 1-2, while the SDs were calculated using the Horwitz equation. In Proficiency Test 3, the median of the results was chosen as the best estimate of the true concentration. Estimates of the standard deviations were compared using four methods. The traditional approach (ISO 5725-Part 2) with outlier elimination robust statistics using both the Qn and Swiss methods and a fixed target value (fit-for-purpose) as stipulated by the Advisory Group. The two robust statistical methods, for the estimation of the SD gave only marginal differences in the results, whilst the other two methods produced results that differed significantly.

In Proficiency Test 4, the same two robust statistical methods were compared and again only marginal differences were found.

z-Scores were used in all the previous proficiency tests for the assessment of the laboratory performances for individual pesticides.

The sums of scores, the RSZ (the re-scaled sum of z-scores), SSZ (the squared sum of z-scores) and RLP (relative laboratory performance) were also calculated as for the last Proficiency Test (PT5).

##### **3.1.2 Proficiency Test 6**

In this proficiency test, the median was used to obtain the assigned concentration values for each pesticide.

This was then used to calculate the z-score. The target standard deviations ( $\sigma$ ) were calculated using the FFP (fit-for-purpose) value, which was assigned as 25%. Furthermore the Qn-method was also used to calculate standard deviations.

Sum of z-Scores: the RSZ and SSZ were also used but an additional new criterion was introduced for PT6 with the agreement of the Advisory Group as well as DG Sanco. This was used as the basis for separating the labs into two categories, A and B. To be classified as Category A, sufficient scope had to have been demonstrated. This scope was that 11 or more results (from the 13 pesticides present in the treated test material) must have been submitted with no false positives. All labs reporting less than 11 results, or having reported a false positive were classified as Category B.

##### **3.1.3 Proficiency Test 7**

In this proficiency test, the median has also been used, together with a FFP SD of 25%.

The Weighted Sum of z-Scores (each z-score was multiplied by a weighted function – see 3.6.3) was used to classify laboratories as Category A or Category B depending on the number of pesticide residues reported.

### **3.2 False positives and negatives**

#### 3.2.1 False positives

In principle, reported results that show the presence of pesticides that were included in the pesticide list, and which were (i) not used in the preparation of the test material, (ii) and not detected by the organizer, even following a repeat analysis, were treated as false positives, if they were reported at concentrations at or above the MRPL stipulated by the Organizer. Results reported that were lower than 0.01 mg/Kg have been ignored by the Organiser and have not therefore been considered as false positives. No z-score value has been calculated for these results.

#### 3.2.2 False negatives

Results for pesticides that were not reported by the laboratories, although they were used by the Organiser to treat the test material and were subsequently detected at, or above, the MRPL by the Organiser (and the majority of participating laboratories) have been considered to be false negatives. z-Scores have been calculated for all pesticides detected at levels exceeding the MRPL.

### **3.3 Estimation of the assigned values**

To establish the assigned values, the median levels of all the reported results, excluding the outliers, were used. Individual results without any absolute values reported, such as detected (D), could not be used.

### **3.4 Estimation of the target standard deviation**

To assign the target standard deviations ( $\sigma$ ) for each individual compound a fixed relative standard deviation (fit-for-purpose, FFP), based on individual experience and as recommended by the Advisory Group, was used. The target standard deviation was calculated by multiplying these FFP SDs by the assigned value.

#### 3.4.1 Fixed target standard deviations (fit-for-purpose)

The target relative standard deviation (RSD) was considered to be 25%, as recommended by the Advisory Group, and also as a conclusion from the discussion session on proficiency testing at EPRW 2004 in Stockholm, Sweden. The same target RSD has thus been applied to all the pesticides.

### **3.5 z-Scores**

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

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

Where:

- $x$  is the participant reported result or the MRPL for those labs not having detected the pesticide present in the sample
- $X$  is the assigned value or true concentration
- $\sigma$  is the target standard deviation (using the median and FFP of 25%)

Any z-score values of  $|z| > 5$  has been reported as '+5', or '-5'.

No calculation of z-score has been performed for any false positive result. For false negatives, the MRPL has been used to calculate the z-score.

### 3.6 Combined z-scores

In order to evaluate each laboratory's overall performance, and taking into account all the pesticides analysed, three methods were used to combine z-scores; the re-scaled sum of z-scores (RSZ), the sum of squared z-scores (SSZ) and the procedure for combining z-scores called the 'Weighted sum of z-scores' (WSZ) that was first used in EUPT 6.

#### 3.6.1 RSZ

The RSZ was calculated for all z-score values for each laboratory according to:

$$RSZ = \sum z / (n)^{1/2}$$

Where n is the number of z-scores

The RSZ gives an averaged score for all pesticides analysed and indicates if a laboratory has a consistent bias in its results.

#### 3.6.2 SSZ

The SSZ is the sum of all squared z-scores. It was calculated for all z-scores for each laboratory according to:

$$SSZ = (z\text{-score}_1)^2 + (z\text{-score}_2)^2 + \dots + (z\text{-score}_n)^2$$

Where n is the number of z-scores.

#### 3.6.3 Weighted sum of z-scores

This function was only applied to labs with sufficient scope (those in Category A), i.e. those labs that have reported 14 or more results with no false positives. The weighted function  $\omega$  is defined as follows:

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

Therefore, the 'Weighted sum of z-scores'  $|z|$  formula is:

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

So for each lab the formula will have three terms:

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

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

The 'Weighted sum of z-scores' has then been used to produce an overall classification of laboratories as 'good', 'satisfactory' or 'unsatisfactory' according to:

$ z  \leq 2$	good
$2 <  z  \leq 3$	satisfactory
$ z  > 3$	unsatisfactory

In this way, a simple, single combined value, very similar to the single z-scores, is produced that should help to encourage laboratories to analyse a greater number of target compounds.

This evaluation has not been applied to those participants with insufficient scope, in Category B, which is for the laboratories reporting less than 14 results, or with any false positives.

## 4. RESULTS

### 4.1 Summary of reported results

One hundred and twenty-eight laboratories agreed to participate in this proficiency test and one hundred and twenty-five submitted results.

The results of the one hundred twenty-five participating laboratories are presented in this report.

A summary of the results reported can be seen below in Table 3.

Table 3. Summary of Reported Results

Pesticides	Nº of Results Reported	Nº of NA Reported	Nº of ND Reported (false neg.)	% of the Total Results *
Acetamiprid	56	67	2	46.4
Carbaryl	101	24	0	80.8
Cyprodinil	99	24	2	80.8
Diazinon	123	2	0	98.4
Dimethoate	119	2	4	98.4
Fenhexamid	89	36	0	71.2
Fludioxonil	85	36	4	71.2
Imidacloprid	64	60	1	52.0
Iprodione	113	8	4	93.6
Kresoxim-methyl	104	20	1	84.0
Methomyl	71	45	9	64.0
Monocrotophos	89	31	5	75.2
Procymidone	121	4	0	96.8
Pyrimethanil	98	25	2	80.0
Tetraconazole	70	49	6	60.8
Thiabendazole	104	18	3	85.6

\* The % of the total results has been calculated using only labs that actually reported results for these compounds.

NA = Not analysed

ND = Not detected

The laboratories that agreed to participate are listed in Annex 2. All data reported by the participants is given: the analytical results in Appendix 3, recoveries and analytical methods used in Appendix 7. For an explanation of the symbols used in these tables, see Annex 1.

#### 4.1.1 False positives

Some laboratories reported additional pesticides to those applied to the test material. These pesticides and their residue levels reported are presented in Table 4 A and 4 B.

When the reported concentration of the erroneously detected pesticide was higher than the assigned MRPL value the result was considered to be a false positive (see table 4 A).

No laboratory was classified as Category B solely because it reported a false positive result.

Table 4 A. Laboratories that reported false positives in the treated test material

Pesticide	Lab Code	Concentration (mg/kg)	RL (mg/Kg)	MRPL (mg/Kg)
Dichlofluanid	EUPT 7- 122	0.103	0.006	0.05
Endosulfan	EUPT 7- 125	0.106	0.0034	0.05
Methidathion	EUPT 7- 126	0.176		0.02
Myclobutanol	EUPT 7- 049	0.26	0.01	0.02

If the concentration was below the MRPLs, or if the pesticides did not appear in the pesticide list included in the EUPT 7 (Annex I), then these results were not considered to be false positives. However, these laboratories should have taken more care in reporting their results as the concentrations reported were not only lower than the MRPL but in most cases even lower to their own reporting limit. These can be seen in Table 4 B:

Table 4 B. Laboratories that should take more care in reporting their results

Pesticide	Lab Code	Concentration (mg/kg)	RL (mg/Kg)	MRPL (mg/Kg)
Chlorpyrifos	EUPT 7- 116	0.0016	0.01	0.05
Dichlofluanid	EUPT 7- 097	0.013	0.05	0.05
Myclobutanol	EUPT 7- 125	0.005	0.0006	0.02
Vinclozolin	EUPT 7- 097	0.022	0.05	0.05

#### 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 3 summarizes how many ND results for each pesticide were reported.

#### 4.1.3 Distribution of data

The distributions of the concentration of the sixteen pesticide residues reported by the laboratories have been plotted as histograms. See Appendix 2. A few results fell outside the concentration scale.

## **4.2 Assigned values and target standard deviations**

To establish the assigned values, the medians of all the reported results were used, excluding those values that were very distant from the median. The median did not change even when these outliers were included.

The target standard deviation was obtained using a FFP value of 25%.

The assigned values are given in Appendix 3.

## **4.3 Assessment of the laboratory performance**

### 4.3.1 z-Scores

z-Scores were calculated using the FFP RSD given. The criteria for defining the acceptability of the z-scores are:

$ z  \leq 2$	acceptable
$2 <  z  \leq 3$	questionable
$ z  > 3$	unacceptable

In Appendix 3, the individual z-scores are presented, together with the median for each laboratory and pesticide.

z-Scores for false negatives results have been calculated using the MRPL value.

In Appendix 4, the graphical representations of the z-scores are presented excluding z-scores from false negatives

### 4.3.2 Combined z-Scores

The classical combined z-scores: RSZ and the SSZ values are listed in Appendix 5 for all laboratories.

Appendix 6 shows a table with the values of individual z-scores for each pesticide and the new combined 'Weighted sum of z-scores' (WSZ) for those labs in Category A. In this category are the laboratories that reported 14 or more results, and also did not report any false positives. A graphical representation of the results for these laboratories can also be found.

Sixty-four from the one hundred and twenty-five laboratories have been put into Category A (51.2%), from which 75% were classified as 'good', 4.7% as 'satisfactory' and 20.3% as 'unsatisfactory' (Table 5).

Sixty-one of the one hundred and twenty-five laboratories reported results for less than fourteen pesticides. Several of these labs could also have been classified as 'good', but they failed to report results for the required 14 compounds. Twenty-two of the sixty-one laboratories detected twelve or thirteen pesticides. For Category B, Table 6 gives the number of pesticides reported by each laboratory and the number of results, for which their z-score was acceptable.

Table 5 Laboratories in Category A:  
Those with sufficient scope, having reported 14 to 16 results, and classified as good, satisfactory or unsatisfactory.

Lab Code	Num of Pesticides	WSZ	Classification
1	16	0.4	Good
2	16	0.8	Good
4	16	0.5	Good
5	16	1.6	Good
9	16	1.7	Good
15	16	1.0	Good
19	16	0.9	Good
22	16	0.8	Good
24	16	0.4	Good
25	16	0.5	Good
29	16	0.9	Good
37	16	0.6	Good
43	16	0.3	Good
44	16	0.7	Good
45	16	1.2	Good
48	16	0.4	Good
50	16	0.5	Good
55	16	0.9	Good
56	16	1.3	Good
58	16	0.9	Good
59	16	1.1	Good
63	16	0.7	Good
68	16	0.4	Good
73	16	0.3	Good
75	16	1.3	Good
79	16	1.2	Good
80	16	0.5	Good
83	16	0.4	Good
89	16	0.6	Good
90	16	0.3	Good
92	16	0.4	Good
93	16	0.7	Good
98	16	0.6	Good
102	16	0.5	Good
104	16	1.4	Good
107	16	2.0	Good
111	16	0.5	Good
113	16	0.3	Good
23	16	2.2	Satisfactory
42	16	2.3	Satisfactory
67	16	2.5	Satisfactory
11	16	3.7	Unsatisfactory
28	16	4.5	Unsatisfactory
36	16	3.2	Unsatisfactory
39	16	5.2	Unsatisfactory

Lab Code	Num of Pesticides	WSZ	Classification
61	16	3.2	Unsatisfactory
91	16	4.6	Unsatisfactory
27	15	0.5	Good
35	15	1.3	Good
106	15	0.7	Good
47	15	7.3	Unsatisfactory
53	15	6.1	Unsatisfactory
74	15	4.3	Unsatisfactory
110	15	5.6	Unsatisfactory
26	14	0.7	Good
52	14	0.6	Good
60	14	0.7	Good
88	14	1.6	Good
95	14	1.6	Good
109	14	0.8	Good
115	14	1.9	Good
69	14	8.0	Unsatisfactory
76	14	6.4	Unsatisfactory
114	14	3.1	Unsatisfactory

Table 6 Number of acceptable z-scores for laboratories in Category B

Lab Code	Num of Pesticides	Num of acceptable z-scores
12	13	13
38	13	13
40	13	13
54	13	13
57	13	13
86	13	13
8	13	12
87	13	12
31	13	11
82	13	11
116	13	10
18	13	9
84	13	9
78	13	6
6	12	12
94	12	12
20	12	11
65	12	11
103	12	11
16	12	10
72	12	10
49	12	4
41	11	11

Lab Code	Num of Pesticides	Num of acceptable z-scores
118	11	11
13	11	10
101	11	8
32	11	5
30	11	3
10	10	10
64	10	10
105	10	9
7	9	9
66	9	9
71	9	9
99	9	8
3	8	8
34	8	7
21	8	1
33	7	7
62	7	6
96	7	5
112	7	5
117	7	5
70	7	1
100	6	6
128	6	6
14	6	5
85	6	5
120	6	5
122	6	5
46	5	5
125	5	5
119	5	4
126	5	4
121	5	2
17	5	1
124	4	4
51	2	2
108	2	2
123	2	2
97	0	0
77		No Results
81		No Results
127		No Results

## **5. CONCLUSIONS**

A large number of the invited laboratories submitted results for the analysis of the pesticides present in the grape homogenate test material (125 of 128). For each laboratory/pesticide combination, z-scores based on FFP were calculated. For the FFP, a graphic representation has been prepared. A classification was made using the simple descriptive terms 'acceptable, questionable and unacceptable'.

'The Weighted Sum of z-Scores' (WSZ), a criterion first introduced in last year's proficiency test report, was used to demonstrate the overall performance of the laboratories. Those laboratories reporting fourteen or more results, and not having submitted any false positive results, were classified as having sufficient scope and were therefore placed in Category A. Those laboratories that reported less than fourteen results were considered to have insufficient scope and were placed in Category B. Laboratories in Category A were also classified as 'good', 'satisfactory' or 'unsatisfactory', depending on the values obtained after combining each reported z-score. For the remainder of the labs (the ones belonging to Category B) no combined weighted sum of z-scores was calculated. However, the number of satisfactory z-scores is shown.

The median value of each pesticide, after elimination of outliers, was used to obtain the assigned or "true" concentration, which was also used to calculate the z-scores..

Applying the factors for recovery correction did not improve the overall precision and accuracy of the results.

The presence of some very low level residues in the blank grape homogenate supplied did not affect the results obtained from the treated test material.

Three of the sixteen pesticides used to treat the test material were not included in the monitoring priority list. Acetamiprid and Tetraconazole were considered to be important because of their wide usage in agriculture and the frequent occurrence of MRL-violations. Monocrotophos, was chosen because of its high toxicity.

The decrease in the numbers of false positive and false negative results seen in PT6 was even more apparent in PT7. However, this continued improvement may have been, at least in part, due to some labs that performed poorly in PT6 did not participate in PT7.

Another reason for the improvement could have been that more participants used mass spectrometry, particularly MS/MS.

Participation in this year 7<sup>th</sup> European Proficiency Test involved 24 EU member states, the EU-candidate country Romania, and also Iceland and Norway that regularly participate in the EU-monitoring programmes. This year some laboratories participated for the first time in this Proficiency Test.

## **6. SUGGESTIONS FOR FUTURE WORK**

The Scientific Committee and Advisory Group of the EU Proficiency Test 7 decided to maintain the use of the 25% FFP standard deviation, so that the results from the PTs are more comparable. After this 7th EU Proficiency Test, we believe that it is time for a recapitulation. Recalculating previous PT results using the 25% FFP standard deviation would help to obtain more comparable data and clear information about the trends in the performances of the laboratories throughout all the PTs. This would also help to present a picture of the overall progress in achieving better analytical quality and harmonization within the EU.

## 7. REFERENCES

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5. Muller C H and Uhlig S, Biometrika, 88 (2001), 353-336, Estimation of Variance components with high breakdown point and high efficiency.
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## **8. ACKNOWLEDGEMENTS**

The Organiser is grateful to the European Commission for funding this 7<sup>th</sup> European Proficiency Test.

The Organiser wishes to thank the members of the Advisory Group and the Scientific Committee for their valuable and knowledgeable advice. Many thanks also to the Statistical Group for their cooperation and statistical calculations and evaluations.

The Organiser wishes to give a special thank-you to the University of Almeria and a special mention to Coexphal laboratory for their invaluable work.



**APPENDIX 1. Homogeneity Data**

Acetamiprid		Carbaryl		Cyprodinil		Diazinon	
Sample 1	Sample 2	Sample 1	Sample 2	Sample 1	Sample 2	Sample 1	Sample 2
0.300	0.358	1.660	1.658	0.460	0.458	0.283	0.285
0.350	0.350	1.650	1.650	0.450	0.450	0.290	0.293
0.381	0.352	1.681	1.652	0.481	0.452	0.289	0.285
0.388	0.390	1.688	1.690	0.388	0.390	0.288	0.280
0.332	0.340	1.632	1.640	0.432	0.440	0.291	0.290
0.372	0.380	1.672	1.680	0.472	0.480	0.295	0.290
0.300	0.362	1.600	1.662	0.400	0.462	0.284	0.286
0.330	0.337	1.630	1.637	0.430	0.437	0.287	0.290
0.320	0.334	1.620	1.634	0.420	0.434	0.287	0.284
0.328	0.324	1.628	1.688	0.428	0.488	0.289	0.290

Dimethoate		Fenhexamid		Fludioxonil		Imidacloprid	
Sample 1	Sample 2	Sample 1	Sample 2	Sample 1	Sample 2	Sample 1	Sample 2
0.137	0.134	0.737	0.734	0.269	0.226	0.608	0.626
0.155	0.154	0.755	0.754	0.240	0.208	0.530	0.548
0.184	0.149	0.684	0.649	0.230	0.253	0.540	0.553
0.184	0.193	0.684	0.693	0.230	0.235	0.530	0.535
0.188	0.122	0.688	0.622	0.285	0.200	0.585	0.599
0.143	0.199	0.693	0.699	0.207	0.197	0.507	0.597
0.170	0.175	0.670	0.175	0.230	0.233	0.530	0.533
0.148	0.198	0.648	0.698	0.273	0.270	0.573	0.570
0.126	0.132	0.726	0.732	0.200	0.199	0.540	0.599
0.188	0.193	0.688	0.693	0.200	0.199	0.540	0.599

Iprodione		Kresoxim-methyl		Methomyl		Iprodione	
Sample 1	Sample 2	Sample 1	Sample 2	Sample 1	Sample 2	Sample 1	Sample 2
0.511	0.521	0.506	0.364	0.166	0.164	0.712	0.678
0.518	0.632	0.487	0.440	0.187	0.140	0.682	0.614
0.513	0.514	0.538	0.461	0.138	0.161	0.706	0.670
0.506	0.507	0.387	0.335	0.187	0.135	0.609	0.672
0.491	0.497	0.348	0.386	0.148	0.186	0.685	0.630
0.500	0.499	0.374	0.425	0.174	0.125	0.637	0.637
0.521	0.544	0.400	0.559	0.200	0.159	0.607	0.620
0.557	0.547	0.447	0.617	0.147	0.117	0.671	0.655
0.529	0.530	0.344	0.433	0.144	0.133	0.663	0.692
0.497	0.496	0.308	0.486	0.208	0.186	0.663	0.650

**APPENDIX 1. Homogeneity Data**

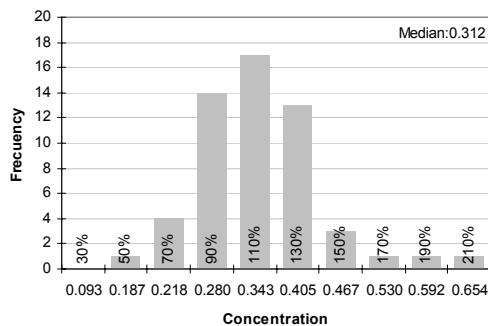
Procymidone		Pyrimethanil		Tetraconazole		Thiabendazole	
Sample 1	Sample 2	Sample 1	Sample 2	Sample 1	Sample 2	Sample 1	Sample 2
2.612	2.178	0.123	0.154	0.063	0.050	0.623	0.654
2.082	2.214	0.128	0.156	0.068	0.056	0.628	0.656
2.606	2.270	0.150	0.129	0.050	0.069	0.650	0.629
2.009	1.872	0.147	0.148	0.067	0.068	0.647	0.648
1.885	2.030	0.130	0.135	0.055	0.065	0.630	0.635
2.037	2.037	0.119	0.119	0.060	0.069	0.619	0.619
2.007	2.620	0.120	0.114	0.060	0.064	0.620	0.614
2.371	2.655	0.159	0.153	0.059	0.050	0.659	0.653
1.863	1.992	0.144	0.147	0.064	0.060	0.644	0.647
1.963	2.350	0.143	0.145	0.043	0.045	0.643	0.645

**APPENDIX 2. Histograms of residue data for each pesticide for all the laboratories.**

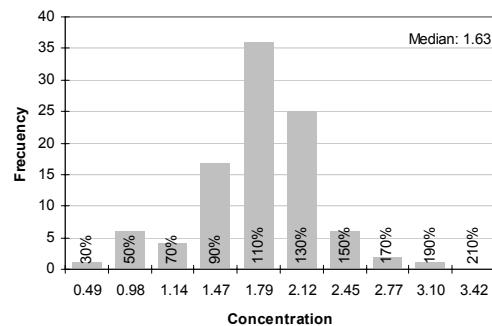
Results presented as histograms (except those results considered to be outliers).

The percentages shown are relative to the median value.

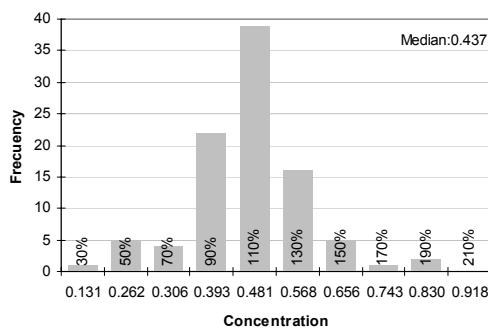
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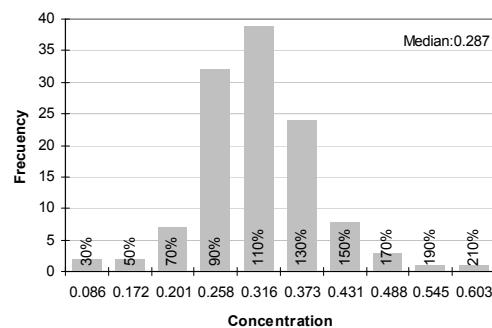
**Carbaryl**



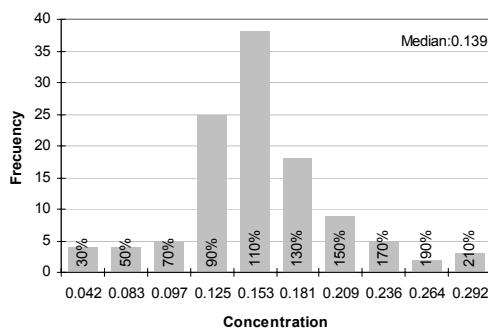
**Cyprodinil**



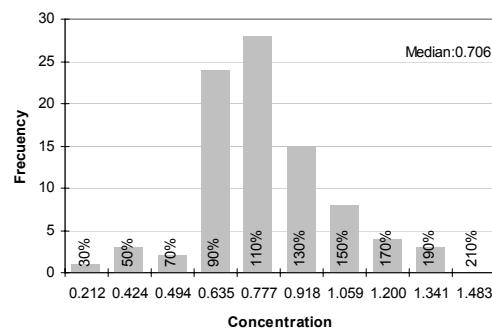
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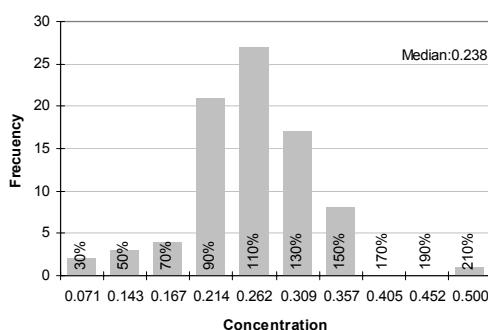
**Dimethoate**



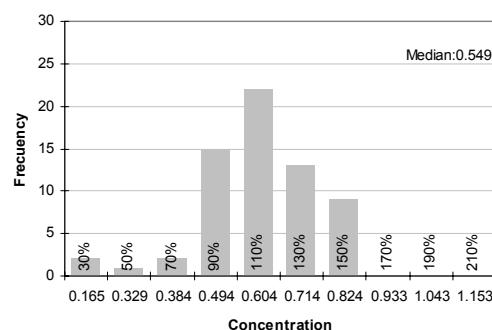
**Fenhexamid**



**Fludioxonil**



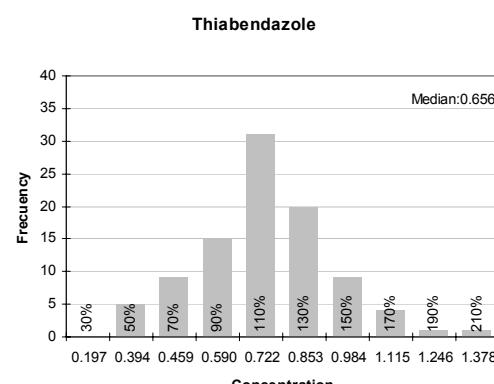
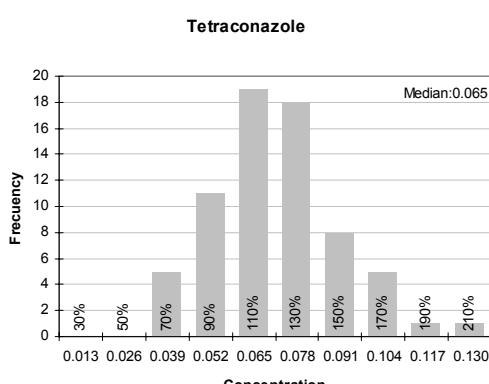
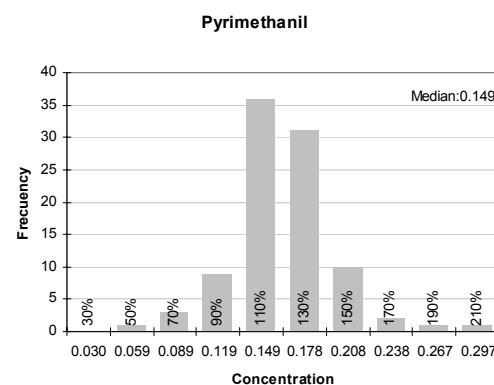
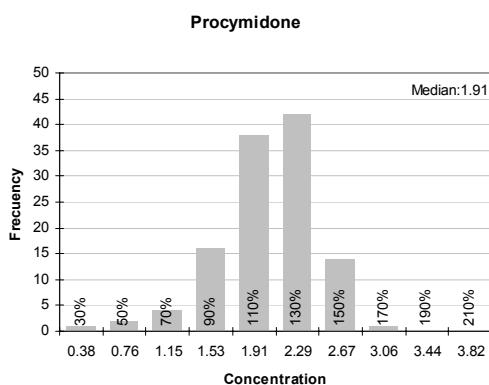
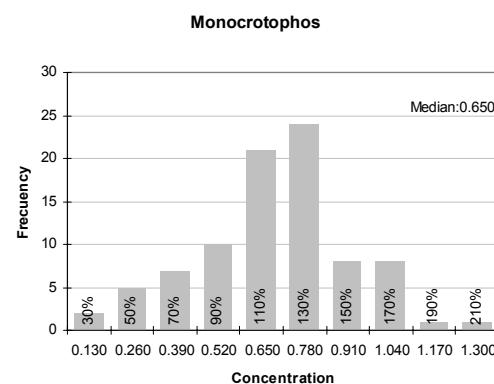
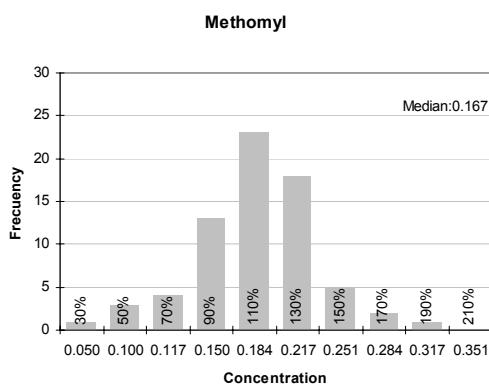
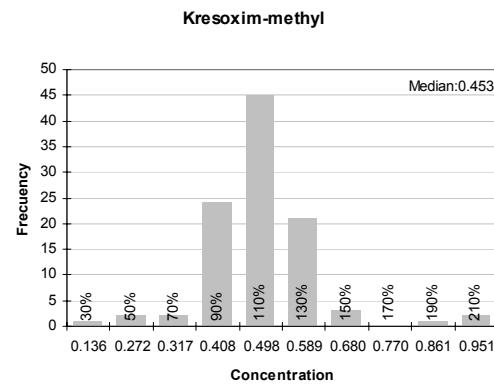
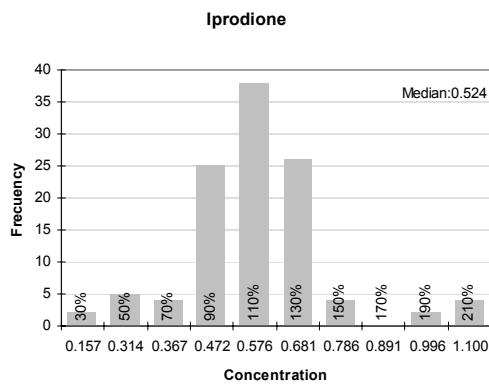
**Imidacloprid**



## APPENDIX 2. Histograms of residue data for each pesticide for all the laboratories.

Results presented as histograms (except those results considered to be outliers).

The percentages shown are relative to the median value.



**APPENDIX 3. Results (mg/kg) and z-scores for FFP**

Lab Code	Acetamiprid	z-Score (FFP 25%)	Carbaryl	z-Score (FFP 25%)	Cyprodinil	z-Score (FFP 25%)	Diazinon	z-Score (FFP 25%)
MRPL	0.05		0.05		0.05		0.02	
Median (mg/kg)	0.312		1.62		0.437		0.287	
1	0.285	-0.3	1.63	0.0	0.385	-0.5	0.287	0.0
2	0.316	0.1	1.56	-0.2	0.519	0.8	0.316	0.4
3	NA		NA		0.330	-1.0	0.267	-0.3
4	0.338	0.3	1.58	-0.1	0.415	-0.2	0.255	-0.4
5	0.312	0.0	1.12	-1.2	0.126	-2.8	0.242	-0.6
6	NA		1.76	0.3	0.470	0.3	0.320	0.5
7	NA		1.50	-0.3	0.566	1.2	0.232	-0.8
8	NA		1.84	0.5	0.620	1.7	0.350	0.9
9	0.344	0.4	1.86	0.6	0.544	1.0	0.339	0.7
10	NA		1.10	-1.3	0.449	0.1	0.232	-0.8
11	0.275	-0.5	1.45	-0.4	0.775	3.1	0.235	-0.7
12	0.172	-1.8	1.49	-0.3	NA		0.246	-0.6
13	0.220	-1.2	NA		NA		0.230	-0.8
14	NA		NA		NA		0.406	1.7
15	0.398	1.1	1.86	0.6	0.537	0.9	0.299	0.2
16	NA		1.60	0.0	0.362	-0.7	0.225	-0.9
17	NA		NA		NA		0.600	4.4
18	NA		0.946	-1.7	0.516	0.7	0.309	0.3
19	0.270	-0.5	1.50	-0.3	0.308	-1.2	0.200	-1.2
20	NA		1.93	0.8	0.437	0.0	0.305	0.3
21	NA		3.98	5.8	1.03	5.4	0.970	9.5
22	0.238	-0.9	1.20	-1.0	0.435	0.0	0.360	1.0
23	0.327	0.2	1.84	0.5	0.457	0.2	0.312	0.3
24	0.293	-0.2	1.56	-0.1	0.473	0.3	0.259	-0.4
25	0.230	-1.0	1.25	-0.9	0.415	-0.2	0.245	-0.6
26	NA		1.79	0.4	0.452	0.1	0.177	-1.5
27	0.300	-0.1	1.50	-0.3	0.420	-0.2	0.250	-0.5
28	0.390	1.0	1.82	0.5	0.471	0.3	0.400	1.6
29	0.391	1.0	0.816	-2.0	0.289	-1.4	0.269	-0.3
30	NA		6.64	12.4	0.573	1.2	0.412	1.7
31	NA		1.96	0.8	0.298	-1.3	0.255	-0.4
32	NA		2.18	1.4	0.460	0.2	0.650	5.1
33	NA		NA		0.525	0.8	0.315	0.4
34	NA		NA		0.398	-0.4	0.354	0.9
35	0.191	-1.5	1.50	-0.3	0.365	-0.7	0.210	-1.1
36	0.343	0.4	1.36	-0.6	0.458	0.2	0.340	0.7
37	0.360	0.6	1.44	-0.4	0.410	-0.2	0.400	1.6
38	NA		1.89	0.7	0.510	0.7	0.348	0.9
39	0.360	0.6	1.86	0.6	ND (0,01)	-3.5	0.190	-1.4
40	NA		1.66	0.1	0.440	0.0	0.223	-0.9
41	0.208	-1.3	1.49	-0.3	0.391	-0.4	0.253	-0.5
42	0.347	0.5	1.81	0.5	0.490	0.5	0.313	0.4
43	0.324	0.2	1.68	0.1	0.472	0.3	0.318	0.4
44	0.376	0.8	2.02	1.0	0.418	-0.2	0.361	1.0
45	0.215	-1.2	1.17	-1.1	0.364	-0.7	0.481	2.7
46	NA		NA		NA		0.379	1.3
47	ND (0,50)	-3.4	1.74	0.3	0.450	0.1	0.290	0.0
48	0.304	-0.1	1.61	0.0	0.476	0.4	0.319	0.4

**APPENDIX 3. Results (mg/Kg) and z-scores for FFP**

Lab Code	Acetamiprid	z-Score (FFP 25%)	Carbaryl	z-Score (FFP 25%)	Cyprodinil	z-Score (FFP 25%)	Diazinon	z-Score (FFP 25%)
49	NA		2.64	2.5	1.03	5.4	0.200	-1.2
50	0.311	0.0	1.72	0.2	0.301	-1.2	0.237	-0.7
51	NA		0.810	-2.0	NA		NA	
52	NA		1.55	-0.2	0.483	0.4	0.400	1.6
53	0.850	6.9	1.93	0.8	0.645	1.9	0.346	0.8
54	NA		2.03	1.0	0.347	-0.8	0.272	-0.2
55	0.365	0.7	1.68	0.1	0.618	1.7	0.390	1.4
56	0.206	-1.4	1.72	0.2	0.690	2.3	0.228	-0.8
57	NA		1.52	-0.2	0.400	-0.3	0.278	-0.1
58	0.429	1.5	2.09	1.2	0.533	0.9	0.242	-0.6
59	0.348	0.5	1.96	0.8	0.510	0.7	0.402	1.6
60	NA		1.30	-0.8	0.360	-0.7	0.360	1.0
61	0.505	2.5	1.60	0.0	0.515	0.7	0.361	1.0
62	NA		NA		0.420	-0.2	0.250	-0.5
63	0.360	0.6	1.94	0.8	0.375	-0.6	0.279	-0.1
64	NA		2.20	1.4	NA		0.302	0.2
65	NA		1.71	0.2	0.420	-0.2	0.331	0.6
66	NA		1.25	-0.9	NA		0.192	-1.3
67	0.258	-0.7	3.03	3.5	0.387	-0.5	0.289	0.0
68	0.239	-0.9	1.27	-0.9	0.467	0.3	0.289	0.0
69	NA		2.71	2.7	0.780	3.1	0.442	2.2
70	NA		0.670	-2.3	NA		0.060	-3.2
71	NA		NA		0.438	0.0	0.346	0.8
72	NA		NA		0.323	-1.0	0.245	-0.6
73	0.271	-0.5	1.42	-0.5	0.430	-0.1	0.293	0.1
74	0.226	-1.1	1.39	-0.6	2.06	14.9	0.241	-0.6
75	0.376	0.8	2.09	1.2	0.392	-0.4	0.337	0.7
76	0.605	3.8	1.86	0.6	0.413	-0.2	0.202	-1.2
77	NO RESULTS							
78	NA		1.85	0.6	ND (0,05)	-3.5	0.770	6.7
79	0.413	1.3	1.44	-0.4	0.473	0.3	0.255	-0.4
80	0.340	0.4	1.38	-0.6	0.260	-1.6	0.290	0.0
81	NO RESULTS							
82	NA		2.02	1.0	0.431	-0.1	0.315	0.4
83	0.269	-0.5	1.50	-0.3	0.395	-0.4	0.260	-0.4
84	NA		0.64	-2.4	0.380	-0.5	0.280	-0.1
85	NA		NA		NA		0.323	0.5
86	NA		1.48	-0.3	0.385	-0.5	0.262	-0.3
87	NA		1.43	-0.5	0.358	-0.7	0.236	-0.7
88	ND (0,01)	-3.4	1.54	-0.2	0.227	-1.9	0.308	0.3
89	0.270	-0.5	1.05	-1.4	0.420	-0.2	0.250	-0.5
90	0.330	0.2	1.74	0.3	0.500	0.6	0.320	0.5
91	0.530	2.8	2.43	2.0	0.252	-1.7	0.357	1.0
92	0.291	-0.3	1.91	0.7	0.518	0.7	0.309	0.3
93	0.266	-0.6	1.20	-1.0	0.334	-0.9	0.268	-0.3
94	NA		1.86	0.6	0.450	0.1	0.300	0.2
95	NA		1.72	0.2	0.383	-0.5	0.261	-0.4
96	NA		NA		NA		0.348	0.9
97	NA		NA		NA		NA	
98	0.235	-1.0	1.61	0.0	0.388	-0.4	0.198	-1.2

**APPENDIX 3. Results (mg/kg) and z-scores for FFP**

Lab Code	Acetamiprid	z-Score (FFP 25%)	Carbaryl	z-Score (FFP 25%)	Cyprodinil	z-Score (FFP 25%)	Diazinon	z-Score (FFP 25%)
99	NA		1.49	-0.3	0.410	-0.2	0.282	-0.1
100	NA		NA		NA		0.230	-0.8
101	NA		1.28	-0.8	0.240	-1.8	0.300	0.2
102	0.266	-0.6	1.60	0.0	0.456	0.2	0.348	0.9
103	NA		2.42	2.0	0.375	-0.6	0.210	-1.1
104	0.294	-0.2	1.55	-0.2	0.471	0.3	0.221	-0.9
105	NA		1.86	0.6	0.362	-0.7	0.316	0.4
106	NA		1.38	-0.6	0.450	0.1	0.310	0.3
107	0.290	-0.3	2.10	1.2	0.490	0.5	0.480	2.7
108	NA		NA		NA		0.240	-0.7
109	NA		1.06	-1.4	0.438	0.0	0.303	0.2
110	0.414	1.3	NA		1.22	7.2	0.502	3.0
111	0.328	0.2	1.84	0.5	0.575	1.3	0.278	-0.1
112	NA		1.63	0.0	NA		0.298	0.2
113	0.310	0.0	1.65	0.1	0.462	0.2	0.265	-0.3
114	NA		0.378	-3.1	0.273	-1.5	0.153	-1.9
115	NA		0.862	-1.9	0.262	-1.6	0.279	-0.1
116	NA		2.30	1.7	0.430	-0.1	0.310	0.3
117	NA		2.22	1.5	0.530	0.9	0.330	0.6
118	NA		NA		0.380	-0.5	0.240	-0.7
119	NA		1.71	0.2	NA		0.262	-0.3
120	NA		NA		NA		0.235	-0.7
121	NA		NA		NA		0.077	-2.9
122	NA		NA		NA		0.185	-1.4
123	NA		NA		NA		0.262	-0.3
124	NA		NA		NA		0.170	-1.6
125	NA		NA		NA		0.255	-0.4
126	NA		NA		NA		0.201	-1.2
127	NO RESULTS							
128	NA		1.20	-1.0	NA		0.275	-0.2

**APPENDIX 3. Results (mg/Kg) and z-scores for FFP**

Lab Code	DIMETHOATE e	z-Score (FFP 25%)	Fenhexamid	z-Score (FFP 25%)	Fludioxonil	z-Score (FFP 25%)	Imidacloprid	z-Score (FFP 25%)
			0.05					
MRPL	0.02							
Median (mg/kg)	0.139		0.706		0.238		0.549	
1	0.142	0.1	0.734	0.2	0.271	0.6	0.413	-1.0
2	0.156	0.5	0.735	0.2	0.273	0.6	0.595	0.3
3	0.109	-0.9	NA		0.216	-0.4	NA	
4	0.156	0.5	0.669	-0.2	0.215	-0.4	0.766	1.6
5	0.122	-0.5	0.506	-1.1	0.139	-1.7	0.508	-0.3
6	0.130	-0.3	0.710	0.0	0.250	0.2	NA	
7	0.139	0.0	0.516	-1.1	NA		NA	
8	0.190	1.5	2.24	8.7	NA		0.768	1.6
9	0.179	1.2	0.857	0.9	0.237	0.0	0.626	0.6
10	0.079	-1.7	NA		0.280	0.7	NA	
11	0.127	-0.3	0.793	0.5	0.483	4.1	0.495	-0.4
12	0.121	-0.5	0.486	-1.2	NA		0.383	-1.2
13	0.100	-1.1	NA		NA		0.290	-1.9
14	0.116	-0.7	NA		NA		NA	
15	0.170	0.9	0.775	0.4	0.243	0.1	0.771	1.6
16	0.111	-0.8	NA		0.183	-0.9	NA	
17	0.280	4.1	NA		NA		NA	
18	ND (0,02)	-3.4	0.423	-1.6	0.137	-1.7	NA	
19	0.113	-0.7	0.573	-0.8	0.155	-1.4	0.480	-0.5
20	0.121	-0.5	0.580	-0.7	NA		NA	
21	0.490	10.1	NA		NA		NA	
22	0.149	0.3	0.441	-1.5	0.153	-1.4	0.490	-0.4
23	0.094	-1.3	0.754	0.3	0.298	1.0	0.653	0.8
24	0.149	0.3	0.614	-0.5	0.229	-0.2	0.503	-0.3
25	0.123	-0.5	0.822	0.7	0.192	-0.8	0.466	-0.6
26	0.096	-1.2	0.645	-0.3	0.338	1.7	0.570	0.2
27	0.140	0.0	0.710	0.0	0.200	-0.6	0.480	-0.5
28	0.278	4.0	1.17	2.6	0.055	-3.1	0.610	0.4
29	0.084	-1.6	0.959	1.4	0.250	0.2	0.743	1.4
30	0.412	7.9	1.08	2.1	ND (0,05)	-3.2	NA	
31	0.140	0.0	0.940	1.3	0.086	-2.6	NA	
32	0.350	6.1	1.25	3.1	0.220	-0.3	NA	
33	0.091	-1.4	NA		0.204	-0.6	NA	
34	0.071	-2.0	NA		0.208	-0.5	NA	
35	0.125	-0.4	0.598	-0.6	0.191	-0.8	NA	
36	0.275	3.9	0.846	0.8	0.244	0.1	0.751	1.5
37	0.140	0.0	0.570	-0.8	0.180	-1.0	0.540	-0.1
38	0.187	1.4	0.610	-0.5	0.298	1.0	NA	
39	0.245	3.1	0.681	-0.1	0.245	0.1	0.674	0.9
40	0.142	0.1	0.715	0.1	0.231	-0.1	NA	
41	0.120	-0.5	0.588	-0.7	NA		NA	
42	0.165	0.7	0.751	0.3	0.294	0.9	0.682	1.0
43	0.144	0.1	0.772	0.4	0.242	0.1	0.598	0.4
44	0.180	1.2	0.797	0.5	0.271	0.6	0.650	0.7
45	0.131	-0.2	0.646	-0.3	0.172	-1.1	0.342	-1.5
46	0.139	0.0	NA		NA		NA	
47	0.400	7.5	0.560	-0.8	0.280	0.7	NA	
48	0.153	0.4	0.801	0.5	0.267	0.5	0.565	0.1

**APPENDIX 3. Results (mg/kg) and z-scores for FFP**

Lab Code	DIMETHOATE e	z-Score (FFP 25%)	Fenhexamid	z-Score (FFP 25%)	Fludioxonil	z-Score (FFP 25%)	Imidacloprid	z-Score (FFP 25%)
MRPL	0.02							
Median (mg/kg)	0.139							
49	0.100	-1.1	NA		NA		0.160	-2.8
50	0.131	-0.2	0.581	-0.7	0.203	-0.6	0.502	-0.3
51	NA		NA		NA		NA	
52	0.126	-0.4	0.574	-0.7	0.201	-0.6	NA	
53	0.257	3.4	1.30	3.4	0.525	4.8	0.640	0.7
54	0.175	1.0	0.757	0.3	0.225	-0.2	0.675	0.9
55	0.133	-0.2	0.893	1.1	0.265	0.5	0.807	1.9
56	0.112	-0.8	0.693	-0.1	0.159	-1.3	0.454	-0.7
57	0.142	0.1	0.733	0.2	NA		0.620	0.5
58	0.150	0.3	1.04	1.9	0.298	1.0	0.781	1.7
59	0.220	2.3	0.860	0.9	0.320	1.4	0.587	0.3
60	0.120	-0.5	1.00	1.7	0.250	0.2	0.430	-0.9
61	0.209	2.0	0.956	1.4	0.194	-0.7	0.753	1.5
62	0.130	-0.3	NA		NA		NA	
63	0.170	0.9	0.662	-0.2	0.169	-1.2	0.612	0.5
64	0.171	0.9	NA		NA		NA	
65	0.150	0.3	0.923	1.2	0.143	-1.6	NA	
66	0.138	0.0	NA		NA		NA	
67	0.129	-0.3	0.547	-0.9	0.340	1.7	0.407	-1.0
68	0.122	-0.5	0.670	-0.2	0.189	-0.8	0.508	-0.3
69	0.179	1.2	1.32	3.5	0.800	9.4	NA	
70	0.030	-3.1	NA		NA		NA	
71	0.156	0.5	0.567	-0.8	NA		NA	
72	0.166	0.8	0.522	-1.0	NA		NA	
73	0.133	-0.2	0.582	-0.7	0.219	-0.3	0.512	-0.3
74	0.105	-1.0	0.706	0.0	0.238	0.0	NA	
75	0.179	1.2	1.09	2.1	0.274	0.6	0.696	1.1
76	0.195	1.6	0.739	0.2	0.318	1.3	NA	
77								
78	0.150	0.3	NA		ND (0,05)	-3.2	ND (0,05)	-3.6
79	0.210	2.0	1.02	1.8	0.256	0.3	0.613	0.5
80	0.150	0.3	0.850	0.8	0.180	-1.0	0.570	0.2
81								
82	0.216	2.2	0.771	0.4	0.313	1.3	NA	
83	0.132	-0.2	0.602	-0.6	0.257	0.3	0.472	-0.6
84	0.040	-2.8	0.530	-1.0	ND (0,05)	-3.2	NA	
85	0.100	-1.1	NA		NA		NA	
86	0.154	0.4	NA		0.324	1.4	0.488	-0.4
87	0.185	1.3	0.814	0.6	0.310	1.2	0.548	0.0
88	0.128	-0.3	0.598	-0.6	0.240	0.0	0.458	-0.7
89	0.110	-0.8	0.640	-0.4	0.220	-0.3	0.540	-0.1
90	0.139	0.0	0.656	-0.3	0.201	-0.6	0.570	0.2
91	0.110	-0.8	0.150	-3.2	ND (0,02)	-3.2	0.150	-2.9
92	0.143	0.1	0.644	-0.4	0.280	0.7	0.521	-0.2
93	0.152	0.4	0.915	1.2	0.182	-0.9	0.415	-1.0
94	0.135	-0.1	0.680	-0.1	NA		NA	
95	0.115	-0.7	0.640	-0.4	0.226	-0.2	NA	
96	0.027	-3.2	0.681	-0.1	0.273	0.6	NA	
97	NA		NA		NA		NA	
98	0.129	-0.3	0.549	-0.9	0.184	-0.9	0.396	-1.1

**APPENDIX 3. Results (mg/Kg) and z-scores for FFP**

Lab Code	DIMETHOATE e		Fenhexamid		Fludioxonil		Imidacloprid	
		<b>z-Score (FFP 25%)</b>		<b>z-Score (FFP 25%)</b>		<b>z-Score (FFP 25%)</b>		<b>z-Score (FFP 25%)</b>
MRPL	0.02		0.05		0.05		0.05	
Median (mg/kg)	0.139	0.706		0.238		0.549		
99	0.127	-0.3	NA		0.193	-0.8	NA	
100	0.160	0.6	NA		NA		NA	
101	0.380	6.9	NA		0.170	-1.1	NA	
102	0.180	1.2	0.631	-0.4	0.245	0.1	0.558	0.1
103	0.130	-0.3	0.805	0.6	0.250	0.2	NA	
104	0.109	-0.9	0.605	-0.6	0.241	0.1	0.576	0.2
105	0.134	-0.1	NA		0.276	0.6	NA	
106	0.110	-0.8	0.525	-1.0	0.225	-0.2	0.490	-0.4
107	0.220	2.3	1.07	2.1	0.270	0.5	0.390	-1.2
108	0.190	1.5	NA		NA		NA	
109	0.204	1.9	0.793	0.5	0.194	-0.7	NA	
110	0.110	-0.8	0.404	-1.7	0.071	-2.8	0.769	1.6
111	0.154	0.4	0.781	0.4	0.248	0.2	0.605	0.4
112	ND (0,01)	-3.4	NA		NA		0.545	0.0
113	0.145	0.2	0.642	-0.4	0.235	-0.1	0.547	0.0
114	0.040	-2.8	0.355	-2.0	0.183	-0.9	0.550	0.0
115	0.131	-0.2	0.823	0.7	0.270	0.5	NA	
116	ND (0,01)	-3.4	0.930	1.3	0.350	1.9	NA	
117	ND (0,03)	-3.4	NA		NA		NA	
118	0.140	0.0	0.550	-0.9	NA		NA	
119	0.197	1.7	NA		NA		NA	
120	0.071	-2.0	NA		NA		NA	
121	0.107	-0.9	NA		NA		NA	
122	0.090	-1.4	NA		NA		NA	
123	0.189	1.4	NA		NA		NA	
124	0.105	-1.0	NA		NA		NA	
125	0.182	1.2	NA		NA		NA	
126	0.075	-1.8	NA		NA		NA	
127								
128	0.130	-0.3	NA		NA		NA	

**APPENDIX 3. Results (mg/kg) and z-scores for FFP**

Lab Code	Iprodione	z-Score (FFP 25%)	Kresoxim-methyl	z-Score (FFP 25%)	Methomyl	z-Score (FFP 25%)	Monocrotophos	z-Score (FFP 25%)
1	0.703	1.4	0.498	0.4	0.203	0.9	0.758	0.7
2	0.632	0.8	0.464	0.1	0.160	-0.2	0.552	-0.6
3	0.533	0.1	0.351	-0.9	NA		0.361	-1.8
4	0.577	0.4	0.440	-0.1	0.169	0.1	0.790	0.9
5	0.442	-0.6	0.454	0.0	0.161	-0.1	0.540	-0.6
6	0.560	0.3	0.450	0.0	NA		0.660	0.1
7	0.302	-1.7	0.341	-1.0	NA		NA	
8	0.655	1.0	0.574	1.1	0.225	1.4	0.720	0.5
9	0.559	0.3	0.554	0.9	0.185	0.4	0.938	1.8
10	0.499	-0.2	0.429	-0.2	NA		NA	
11	0.407	-0.9	0.612	1.4	0.295	3.1	0.963	2.0
12	0.378	-1.1	0.346	-0.9	0.143	-0.6	0.551	-0.6
13	0.390	-1.0	0.390	-0.6	0.110	-1.4	0.250	-2.4
14	0.442	-0.6	NA		NA		0.196	-2.8
15	0.555	0.2	0.414	-0.3	0.257	2.2	0.951	1.9
16	0.582	0.4	0.396	-0.5	NA		0.389	-1.6
17	1.42	6.8	NA		NA		0.710	0.4
18	0.512	-0.1	0.945	4.3	ND (0,05)	-2.8	ND (0,02)	-3.8
19	0.348	-1.3	0.300	-1.4	0.168	0.0	0.449	-1.2
20	0.535	0.1	0.471	0.2	NA		0.214	-2.7
21	1.40	6.7	1.35	7.9	NA		NA	
22	0.660	1.0	0.487	0.3	0.163	-0.1	0.918	1.7
23	0.480	-0.3	0.501	0.4	ND (0,1)	-2.8	ND (0,05)	-3.8
24	0.527	0.0	0.462	0.1	0.186	0.5	0.498	-0.9
25	0.442	-0.6	0.395	-0.5	0.149	-0.4	0.545	-0.6
26	0.478	-0.4	0.489	0.3	0.129	-0.9	0.465	-1.1
27	0.390	-1.0	0.500	0.4	0.190	0.6	NA	
28	0.674	1.1	0.630	1.6	0.200	0.8	1.21	3.5
29	0.643	0.9	0.446	-0.1	0.126	-1.0	0.431	-1.3
30	0.897	2.8	NA		NA		ND (0,05)	-3.8
31	0.587	0.5	0.341	-1.0	NA		0.694	0.3
32	1.01	3.7	0.630	1.6	NA		NA	
33	0.627	0.8	NA		NA		NA	
34	0.170	-2.7	0.366	-0.8	NA		NA	
35	0.350	-1.3	0.335	-1.0	ND (0,05)	-2.8	0.635	0.0
36	0.626	0.8	0.335	-1.0	0.147	-0.5	0.762	0.7
37	0.420	-0.8	0.450	0.0	0.210	1.0	0.850	1.3
38	0.448	-0.6	0.456	0.0	NA		0.610	-0.2
39	0.978	3.5	0.342	-1.0	0.629	11.1	0.700	0.4
40	0.482	-0.3	0.410	-0.4	NA		0.530	-0.7
41	0.472	-0.4	0.414	-0.3	NA		NA	
42	0.634	0.8	0.541	0.8	0.188	0.5	0.935	1.8
43	0.538	0.1	0.502	0.4	0.153	-0.3	0.761	0.7
44	0.699	1.3	0.477	0.2	0.215	1.2	0.666	0.1
45	0.480	-0.3	0.451	0.0	0.112	-1.3	0.588	-0.3
46	0.491	-0.3	NA		NA		NA	
47	1.09	4.3	0.530	0.7	ND (0,05)	-2.8	0.800	1.0

**APPENDIX 3. Results (mg/Kg) and z-scores for FFP**

Lab Code	Iprodione	Kresoxim-methyl	Methomyl	Monocrotophos
		z-Score (FFP 25%)		z-Score (FFP 25%)
MRPL	0.02	0.05	0.05	0.03
Median (mg/kg)	0.524	0.453	0.167	0.643
48	0.600	0.6	0.166	0.620
49	0.110	-3.2	0.040	0.670
50	0.505	-0.1	0.205	0.592
51	NA	NA	0.130	NA
52	0.534	0.1	0.161	0.334
53	0.625	0.8	NA	0.743
54	0.413	-0.8	0.170	NA
55	0.670	1.1	0.193	0.715
56	0.356	-1.3	0.233	0.845
57	0.502	-0.2	0.153	0.600
58	0.610	0.7	0.153	0.607
59	0.510	-0.1	0.210	0.520
60	NA	0.500	0.180	0.610
61	0.559	0.3	0.175	0.992
62	0.560	0.3	NA	0.650
63	0.514	-0.1	0.174	0.375
64	0.454	-0.5	0.190	0.733
65	0.646	0.9	NA	0.490
66	0.536	0.1	0.138	0.569
67	0.580	0.4	0.162	1.00
68	0.567	0.3	0.128	0.576
69	0.673	1.1	ND (0,05)	0.981
70	0.175	-2.7	0.190	NA
71	0.443	-0.6	NA	0.525
72	0.394	-1.0	ND	1.13
73	0.464	-0.5	0.148	0.584
74	0.479	-0.3	ND (0,01)	3.26
75	0.723	1.5	0.214	0.801
76	1.04	3.9	NA	1.78
77				
78	0.550	0.2	ND (0,05)	0.080
79	0.550	0.2	0.222	0.880
80	0.440	-0.6	0.170	0.710
81				
82	0.629	0.8	NA	0.689
83	0.463	-0.5	0.155	0.657
84	0.440	-0.6	NA	ND (0,1)
85	0.354	-1.3	NA	NA
86	0.453	-0.5	0.160	0.750
87	0.586	0.5	0.111	NA
88	0.505	-0.1	NA	0.595
89	0.440	-0.6	0.110	0.510
90	0.524	0.0	0.180	0.750
91	0.665	1.1	0.210	0.297
92	0.507	-0.1	0.204	0.700
93	0.498	-0.2	0.134	0.597
94	0.520	0.0	NA	0.380
95	0.424	-0.8	0.138	0.254

**APPENDIX 3. Results (mg/kg) and z-scores for FFP**

Lab Code	Iprodione	z-Score (FFP 25%)	Kresoxim-methyl	z-Score (FFP 25%)	Methomyl	z-Score (FFP 25%)	Monocrotophos	z-Score (FFP 25%)
MRPL	0.02							
Median (mg/kg)	0.524		0.453		0.167		0.643	
96	0.530	0.0	0.900	3.9	NA		NA	
97	NA		NA		NA		NA	
98	0.438	-0.7	0.353	-0.9	0.163	-0.1	0.670	0.2
99	0.476	-0.4	0.396	-0.5	NA		NA	
100	0.370	-1.2	0.450	0.0	NA		0.760	0.7
101	1.05	4.0	NA		0.070	-2.3	NA	
102	0.586	0.5	0.517	0.6	0.158	-0.2	0.402	-1.5
103	0.120	-3.1	0.335	-1.0	0.150	-0.4	NA	
104	0.557	0.3	0.463	0.1	0.167	0.0	0.737	0.6
105	0.444	-0.6	NA		NA		0.413	-1.4
106	0.475	-0.4	0.320	-1.2	0.090	-1.8	0.330	-1.9
107	0.680	1.2	0.480	0.2	0.230	1.5	0.820	1.1
108	NA		NA		NA		NA	
109	0.432	-0.7	0.484	0.3	0.092	-1.8	0.836	1.2
110	0.575	0.4	0.545	0.8	0.280	2.7	0.105	-3.3
111	0.607	0.6	0.537	0.7	0.185	0.4	0.778	0.8
112	ND (0,05)	-3.8	NA		NA		NA	
113	0.545	0.2	0.570	1.0	0.209	1.0	0.500	-0.9
114	0.281	-1.9	0.200	-2.2	0.160	-0.2	0.770	0.8
115	0.577	0.4	0.409	-0.4	ND (0,01)	-2.8	0.187	-2.8
116	0.600	0.6	0.440	-0.1	0.240	1.8	ND (0,03)	-3.8
117	ND (0,03)	-3.8	NA		NA		NA	
118	0.370	-1.2	0.410	-0.4	NA		0.550	-0.6
119	ND (0,02)	-3.8	NA		NA		D (no standard)	
120	0.759	1.8	NA		ND (0,006)	-2.8	NA	
121	0.165	-2.7	NA		NA		NA	
122	NA		0.400	-0.5	0.147	-0.5	NA	
123	NA		NA		NA		NA	
124	NA		NA		NA		NA	
125	NA		0.314	-1.2	NA		NA	
126	ND (0,010)	-3.8	NA		NA		NA	
127								
128	0.480	-0.3	0.510	0.5	NA		NA	

**APPENDIX 3. Results (mg/Kg) and z-scores for FFP**

Lab Code	Procymidone		Pyrimethamil		Tetraconazole		Thiabendazole	
		<b>z-Score (FFP 25%)</b>		<b>z-Score (FFP 25%)</b>		<b>z-Score (FFP 25%)</b>		<b>z-Score (FFP 25%)</b>
MRPL	0.02		0.05		0.05		0.05	
Median (mg/kg)	1.90		0.149		0.064		0.656	
1	1.90	0.0	0.148	0.0	0.071	0.4	0.679	0.1
2	1.92	0.0	0.172	0.6	0.080	1.0	0.997	2.1
3	1.40	-1.1	NA		NA		NA	
4	1.86	-0.1	0.136	-0.3	0.053	-0.7	0.867	1.3
5	2.12	0.5	0.050	-2.7	0.051	-0.8	0.686	0.2
6	2.14	0.5	0.160	0.3	NA		0.830	1.1
7	1.51	-0.8	NA		NA		0.568	-0.5
8	2.60	1.5	0.183	0.9	NA		0.900	1.5
9	2.07	0.4	0.185	1.0	0.117	3.3	0.862	1.3
10	1.94	0.1	0.139	-0.3	NA		0.502	-0.9
11	2.02	0.3	0.122	-0.7	0.067	0.2	0.645	-0.1
12	1.60	-0.6	0.124	-0.7	Na		0.395	-1.6
13	1.45	-0.9	0.080	-1.8	NA		0.420	-1.4
14	1.32	-1.2	NA		NA		0.630	-0.2
15	2.17	0.6	0.149	0.0	0.064	0.0	0.895	1.5
16	1.66	-0.5	0.245	2.6	0.557	30.8	0.596	-0.4
17	7.73	12.3	NA		NA		NA	
18	2.37	1.0	0.173	0.7	NA		0.596	-0.4
19	1.34	-1.2	0.105	-1.2	0.049	-0.9	0.626	-0.2
20	1.72	-0.4	0.140	-0.2	0.064	0.0	0.331	-2.0
21	5.74	8.1	0.110	-1.0	NA		NA	
22	2.53	1.3	0.146	-0.1	ND (0,05)	-0.9	0.630	-0.2
23	2.22	0.7	0.115	-0.9	0.060	-0.3	0.770	0.7
24	2.11	0.4	0.167	0.5	0.059	-0.3	0.753	0.6
25	1.73	-0.4	0.137	-0.3	0.062	-0.1	0.603	-0.3
26	1.52	-0.8	0.126	-0.6	NA		0.681	0.2
27	2.09	0.4	0.080	-1.8	0.068	0.3	0.650	0.0
28	1.94	0.1	0.213	1.7	0.077	0.8	0.514	-0.9
29	1.45	-0.9	0.143	-0.1	0.048	-1.0	0.732	0.5
30	1.74	-0.3	0.319	4.6	NA		2.03	8.4
31	1.75	-0.3	0.190	1.1	0.104	2.5	0.440	-1.3
32	1.94	0.1	0.430	7.6	NA		ND (0,5)	-3.7
33	2.72	1.7	0.202	1.4	NA		NA	
34	2.05	0.3	0.160	0.3	NA		NA	
35	1.52	-0.8	0.110	-1.0	0.040	-1.5	0.610	-0.3
36	2.38	1.0	0.304	4.2	ND (0,05)	-0.9	0.488	-1.0
37	2.04	0.3	0.180	0.8	0.060	-0.3	0.720	0.4
38	2.44	1.1	0.167	0.5	0.072	0.5	0.762	0.6
39	1.59	-0.7	0.149	0.0	0.087	1.4	0.873	1.3
40	1.70	-0.4	0.125	-0.6	0.076	0.8	0.465	-1.2
41	1.72	-0.4	0.130	-0.5	0.054	-0.6	NA	
42	2.43	1.1	0.165	0.4	0.040	-1.5	1.50	5.1
43	1.98	0.2	0.148	0.0	0.076	0.8	0.745	0.5
44	1.96	0.1	0.175	0.7	0.086	1.4	0.750	0.6
45	1.75	-0.3	0.143	-0.1	0.049	-0.9	0.546	-0.7
46	2.06	0.3	NA		NA		0.629	-0.2

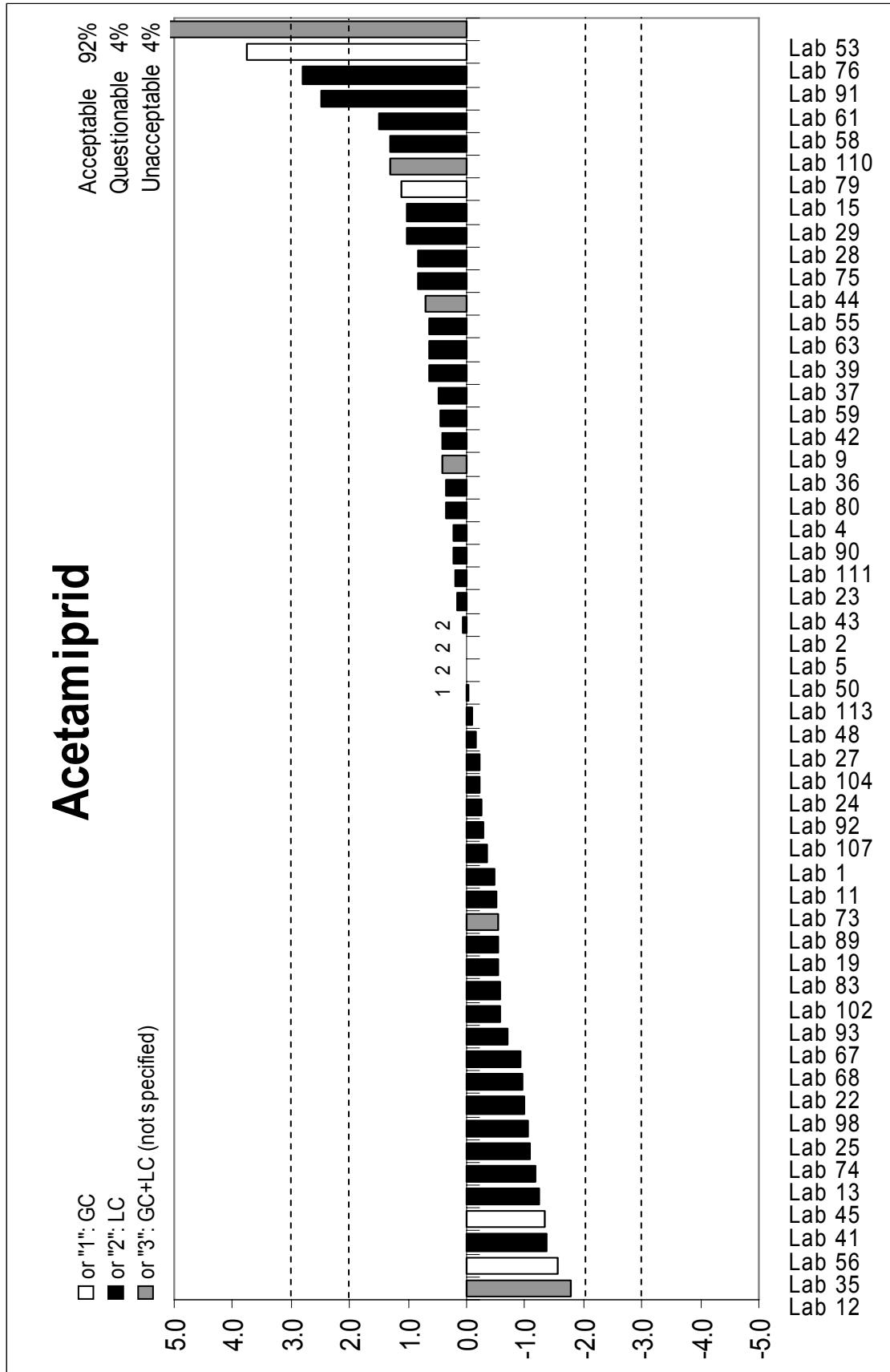
**APPENDIX 3. Results (mg/kg) and z-scores for FFP**

Lab Code	Procymidone	z-Score (FFP 25%)	Pyrimethanil	z-Score (FFP 25%)	Tetraconazole	z-Score (FFP 25%)	Thiabendazole	z-Score (FFP 25%)
MRPL	0.02							
Median (mg/kg)	1.90							
47	2.12	0.5	0.160	0.3	0.080	1.0	1.30	3.9
48	2.32	0.9	0.164	0.4	0.066	0.1	0.735	0.5
49	1.19	-1.5	0.070	-2.1	NA		0.280	-2.3
50	1.75	-0.3	0.109	-1.1	0.067	0.2	0.654	0.0
51	NA		NA		NA		NA	
52	1.95	0.1	0.155	0.2	0.058	-0.4	0.340	-1.9
53	2.16	0.5	0.211	1.7	0.081	1.1	0.653	0.0
54	1.93	0.1	0.146	-0.1	NA		0.928	1.7
55	2.24	0.7	0.173	0.7	0.087	1.4	0.508	-0.9
56	1.65	-0.5	0.175	0.7	0.036	-1.8	0.474	-1.1
57	1.78	-0.3	0.165	0.4	NA		0.750	0.6
58	2.12	0.5	0.194	1.2	0.073	0.6	0.895	1.5
59	2.06	0.3	0.130	-0.5	0.061	-0.2	0.736	0.5
60	2.18	0.6	0.110	-1.0	ND (0,05)	-0.9	0.740	0.5
61	2.33	0.9	0.192	1.2	0.152	5.5	0.991	2.0
62	1.78	-0.3	NA		NA		1.40	4.5
63	2.17	0.6	0.106	-1.1	0.039	-1.6	0.727	0.4
64	1.74	-0.3	0.176	0.7	0.084	1.3	0.826	1.0
65	1.49	-0.9	0.172	0.6	0.028	-2.3	NA	
66	1.23	-1.4	0.130	-0.5	NA		NA	
67	2.60	1.5	0.148	0.0	0.078	0.9	0.668	0.1
68	1.96	0.1	0.142	-0.2	0.073	0.6	0.665	0.1
69	2.16	0.5	0.271	3.3	0.092	1.8	1.03	2.3
70	0.564	-2.8	NA		NA		NA	
71	2.16	0.5	0.174	0.7	NA		NA	
72	1.50	-0.8	0.134	-0.4	0.042	-1.4	0.501	-0.9
73	1.95	0.1	0.132	-0.4	0.058	-0.4	0.655	0.0
74	1.80	-0.2	0.147	0.0	0.051	-0.8	0.605	-0.3
75	2.42	1.1	0.180	0.8	0.077	0.8	0.778	0.7
76	1.11	-1.7	0.150	0.0	ND	-0.9	1.23	3.5
77								
78	1.91	0.0	0.140	-0.2	ND (0,05)	-0.9	ND (0,05)	-3.7
79	1.73	-0.4	0.191	1.1	0.100	2.3	0.712	0.3
80	1.80	-0.2	0.160	0.3	0.055	-0.6	0.620	-0.2
81						-4.0		-4.0
82	2.15	0.5	0.185	1.0	0.104	2.5	0.707	0.3
83	1.80	-0.2	0.141	-0.2	0.059	-0.3	0.547	-0.7
84	2.31	0.9	0.130	-0.5	0.050	-0.9	0.631	-0.1
85	1.70	-0.4	NA		NA		0.207	-2.7
86	2.13	0.5	0.177	0.8	NA		0.653	0.0
87	2.63	1.5	0.145	-0.1	NA		1.27	3.7
88	1.79	-0.2	0.119	-0.8	NA		0.656	0.0
89	1.66	-0.5	0.150	0.0	0.060	-0.3	0.390	-1.6
90	1.99	0.2	0.151	0.1	0.074	0.6	0.760	0.6
91	2.25	0.7	ND (0,01)	-2.7	0.033	-1.9	0.400	-1.6
92	2.22	0.7	0.143	-0.1	0.074	0.6	0.584	-0.4
93	1.82	-0.2	0.174	0.7	0.079	0.9	0.408	-1.5
94	1.86	-0.1	0.156	0.2	0.060	-0.3	0.690	0.2
95	1.61	-0.6	0.125	-0.6	0.097	2.1	0.250	-2.5

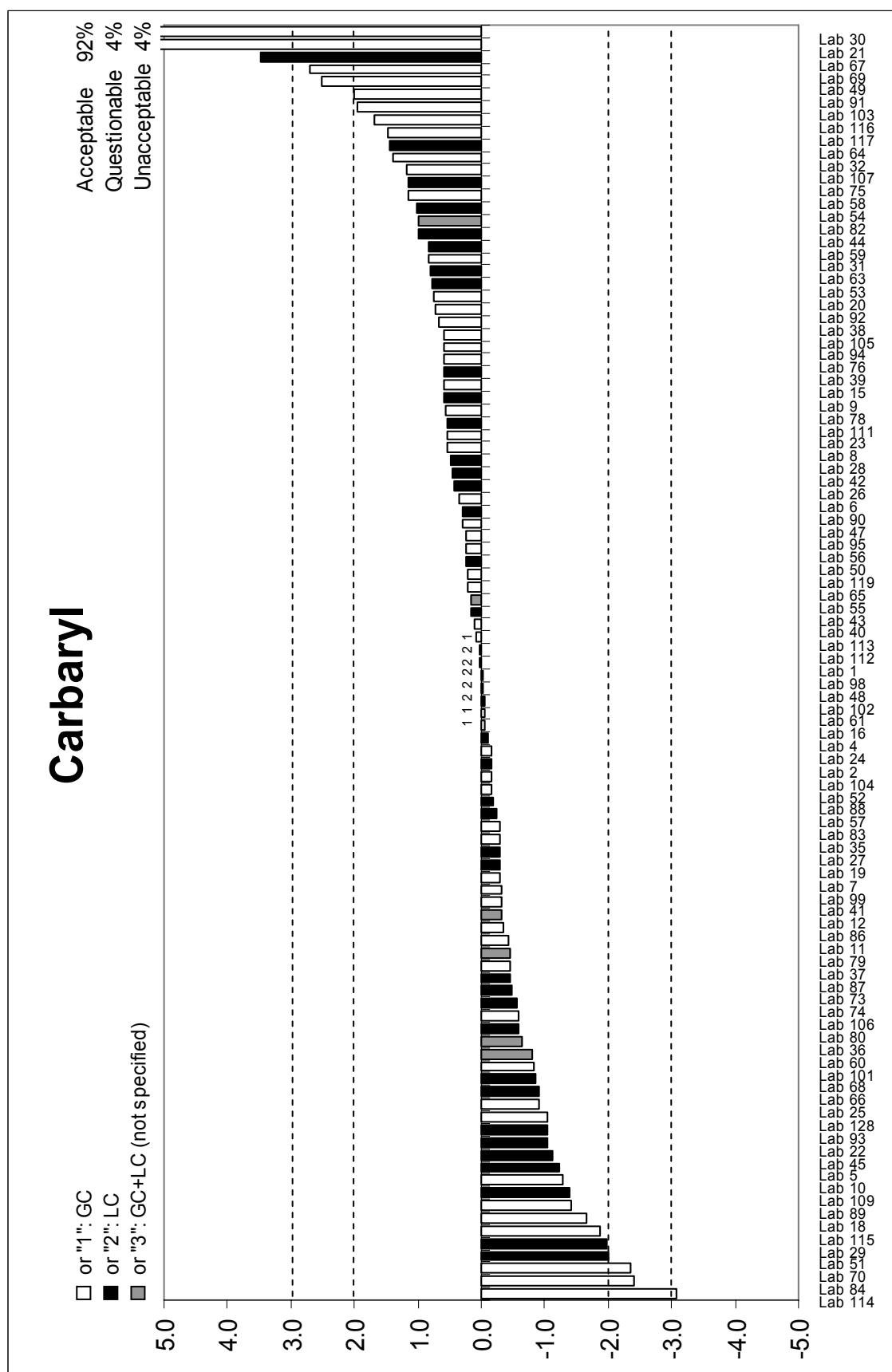
**APPENDIX 3. Results (mg/Kg) and z-scores for FFP**

Lab Code	Procymidone	z-Score (FFP 25%)	Pyrimethanil	z-Score (FFP 25%)	Tetraconazole	z-Score (FFP 25%)	Thiabendazole	z-Score (FFP 25%)
MRPL	0.02			0.05		0.05		
Median (mg/kg)	1.90			0.149				
96	1.75	-0.3	NA		NA		NA	
97	NA		NA		NA		NA	
98	1.63	-0.6	0.123	-0.7	0.059	-0.3	0.618	-0.2
99	1.83	-0.1	NA		NA		1.01	2.2
100	1.64	-0.5	NA		NA		NA	
101	1.22	-1.4	0.120	-0.8	0.050	-0.9	0.560	-0.6
102	2.26	0.8	0.148	0.0	0.063	-0.1	0.623	-0.2
103	1.76	-0.3	0.120	-0.8	NA		0.450	-1.3
104	1.86	-0.1	0.121	-0.7	0.119	3.4	0.743	0.5
105	2.17	0.6	0.127	-0.6	NA		ND (0,5)	-3.7
106	1.68	-0.5	0.155	0.2	ND (0,01)	-0.9	0.730	0.5
107	1.50	-0.8	0.170	0.6	0.070	0.4	0.860	1.2
108	NA		NA		NA		NA	
109	1.67	-0.5	0.151	0.1	0.062	-0.1	0.434	-1.4
110	2.14	0.5	ND (0,02)	-2.7	0.068	0.3	0.894	1.5
111	2.18	0.6	0.159	0.3	0.078	0.9	0.711	0.3
112	2.04	0.3	NA		NA		0.660	0.0
113	2.10	0.4	0.149	0.0	0.062	-0.1	0.595	-0.4
114	1.04	-1.8	0.095	-1.4	NA		0.755	0.6
115	2.34	0.9	0.108	-1.1	0.032	-2.0	0.730	0.5
116	2.21	0.7	0.390	6.5	NA		0.600	-0.3
117	2.45	1.2	NA		NA		0.550	-0.6
118	1.80	-0.2	0.140	-0.2	0.040	-1.5	0.420	-1.4
119	1.50	-0.8	NA		NA		D (no standard)	
120	1.18	-1.5	NA		NA		0.460	-1.2
121	0.638	-2.7	NA		NA		0.825	1.0
122	0.029	-3.9	NA		NA		0.486	-1.0
123	NA		NA		NA		NA	
124	1.62	-0.6	NA		NA		0.660	0.0
125	1.13	-1.6	NA		NA		0.514	-0.9
126	1.02	-1.9	NA		NA		0.410	-1.5
127								
128	1.93	0.1	NA		NA		NA	

**APPENDIX 4. Graphical Representation of z-scores using FFP (25%)**

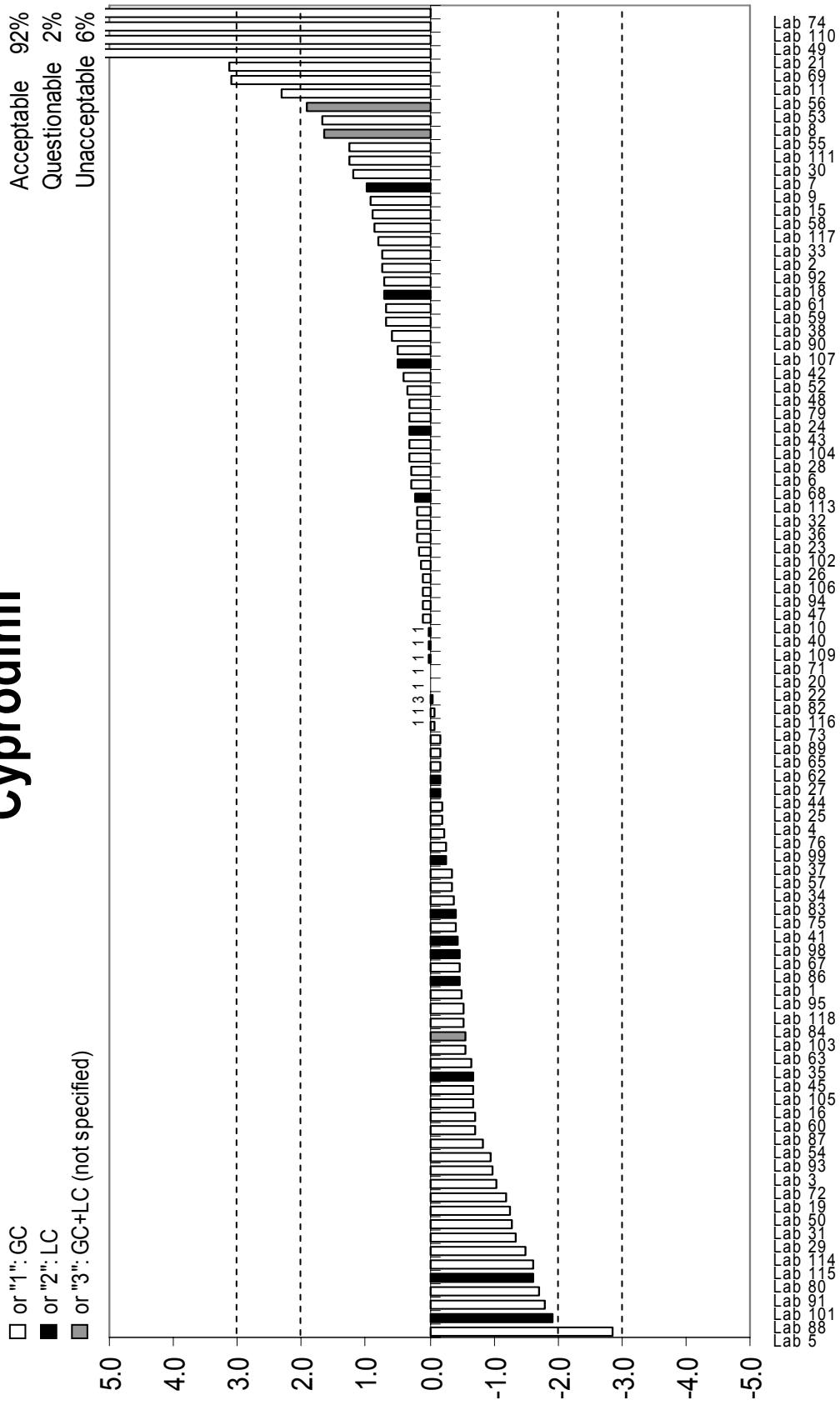


**APPENDIX 4. Graphical Representation of z-scores for FFP (25%)**

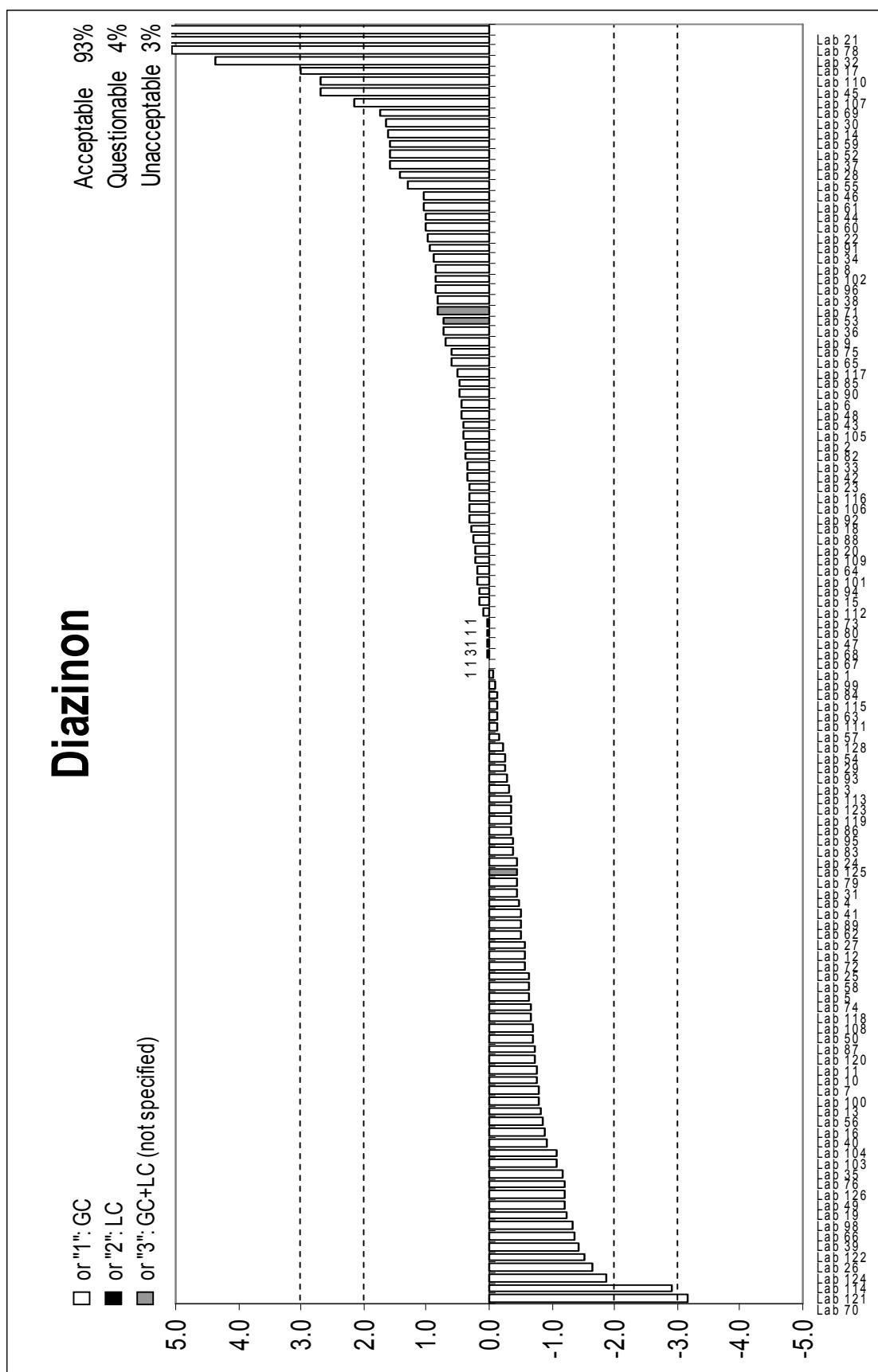


**APPENDIX 4. Graphical Representation of z-scores using FFP (25%)**

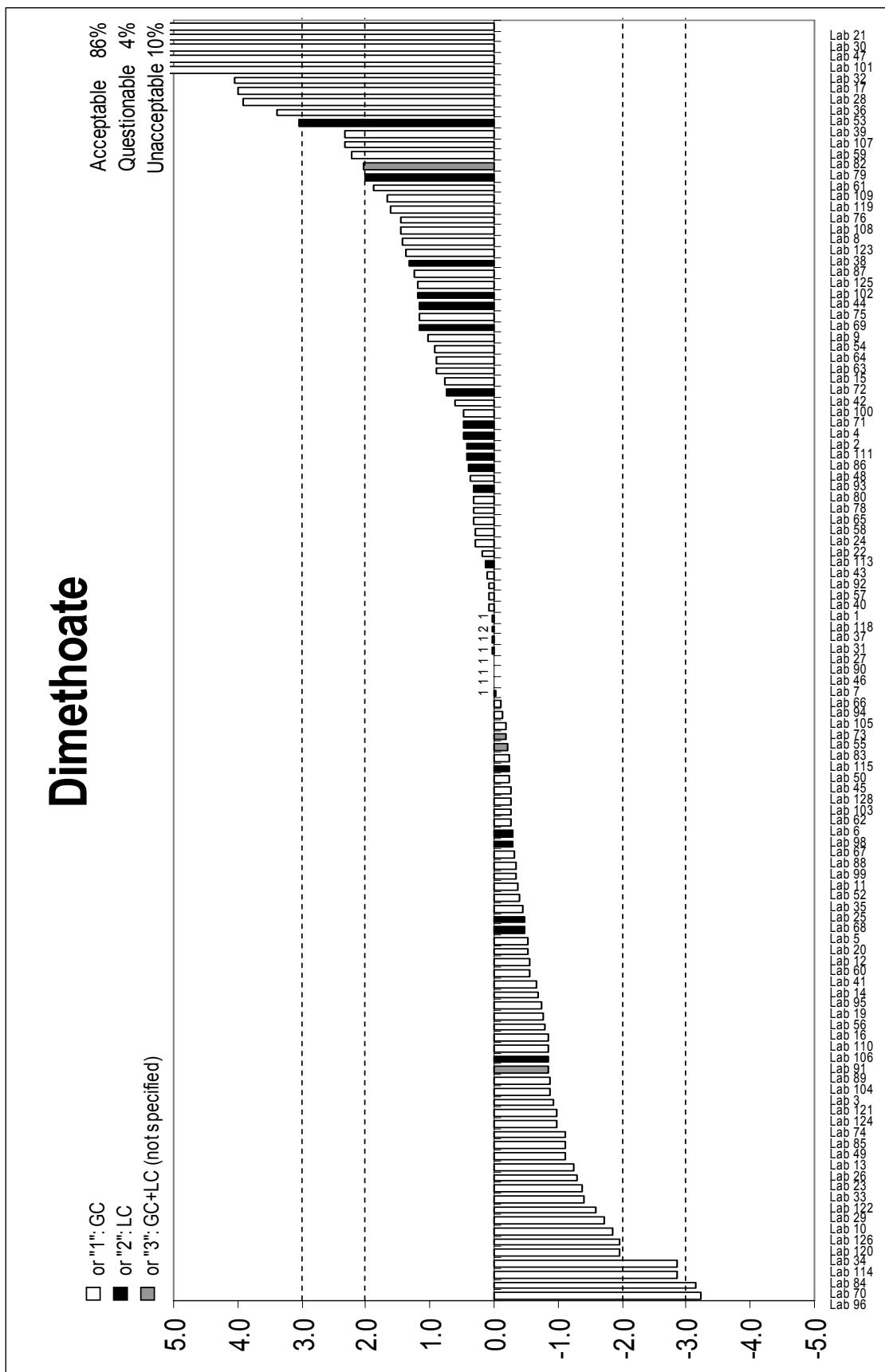
## Cyprodinil



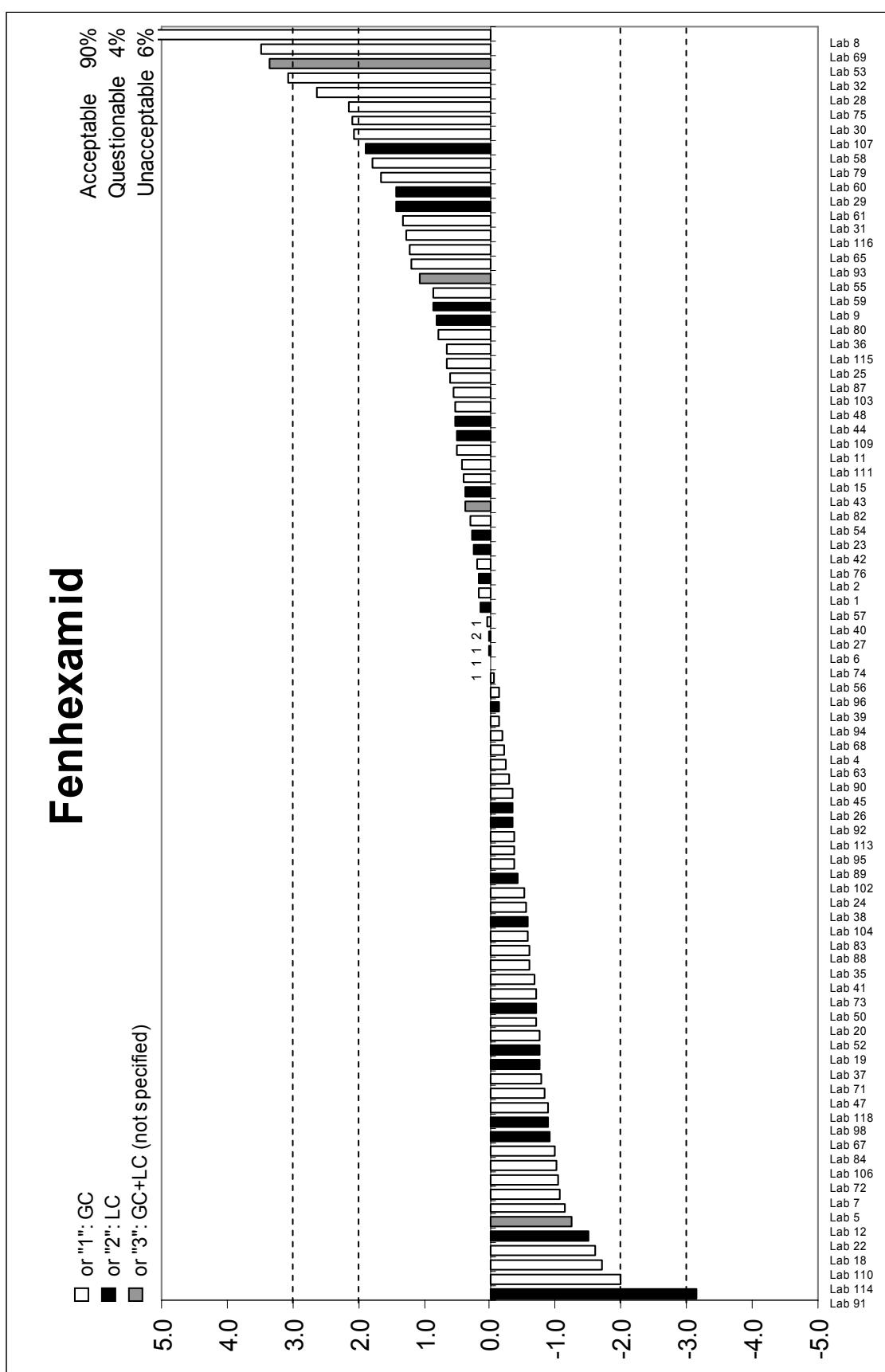
**APPENDIX 4. Graphical Representation of z-scores for FFP (25%)**



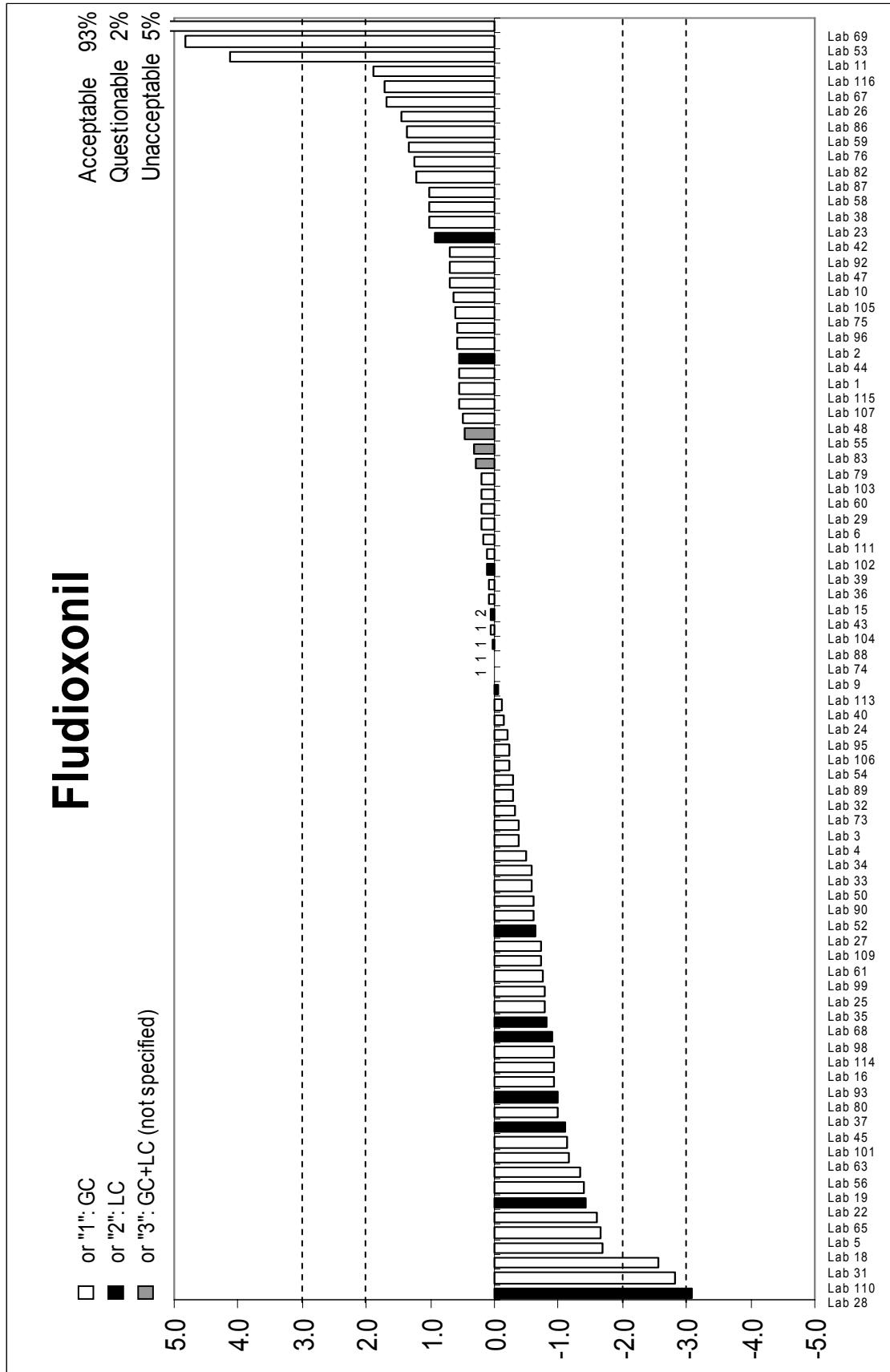
**APPENDIX 4. Graphical Representation of z-scores using FFP (25%)**



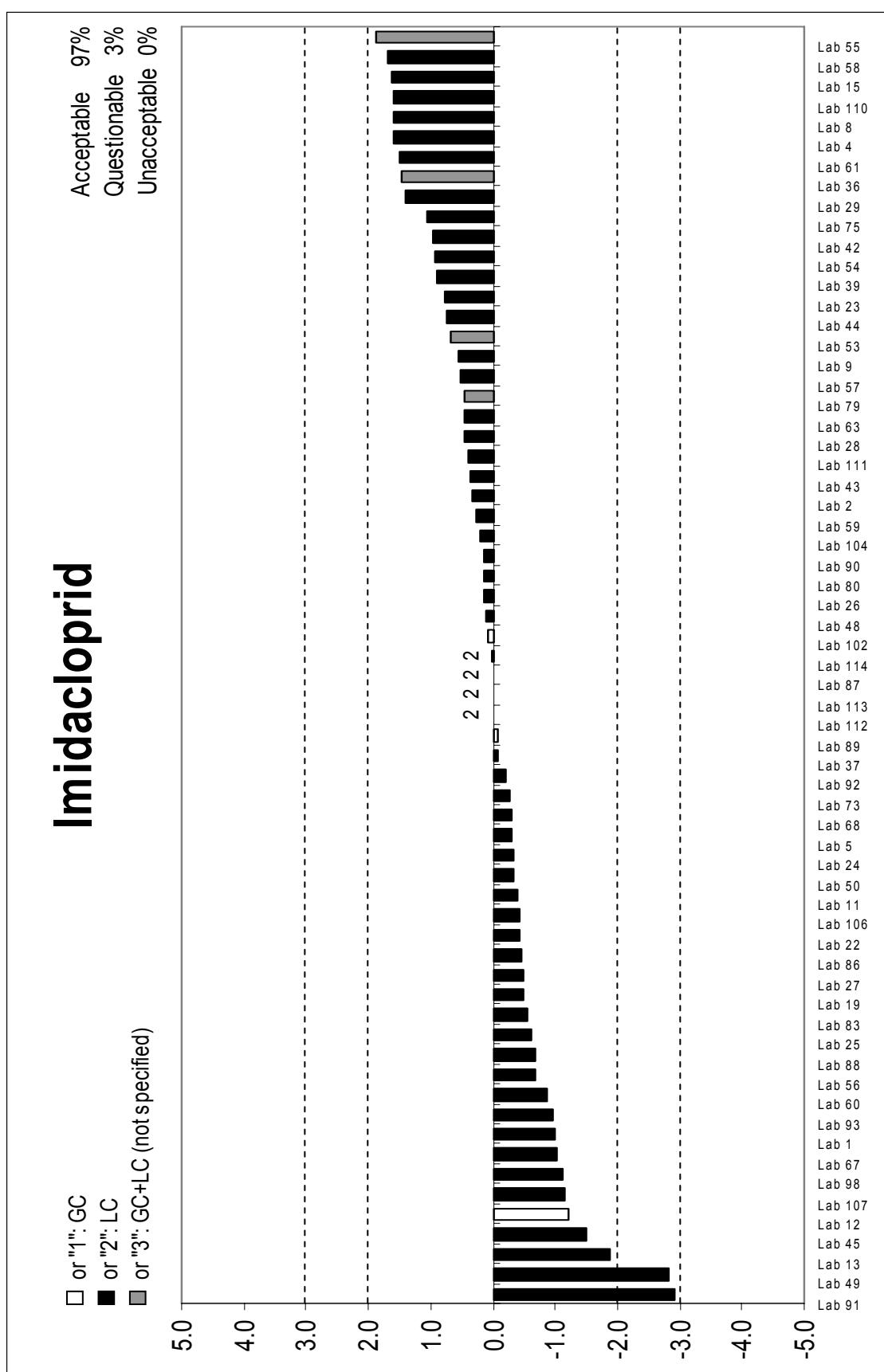
#### **APPENDIX 4. Graphical Representation of z-scores for FFP (25%)**



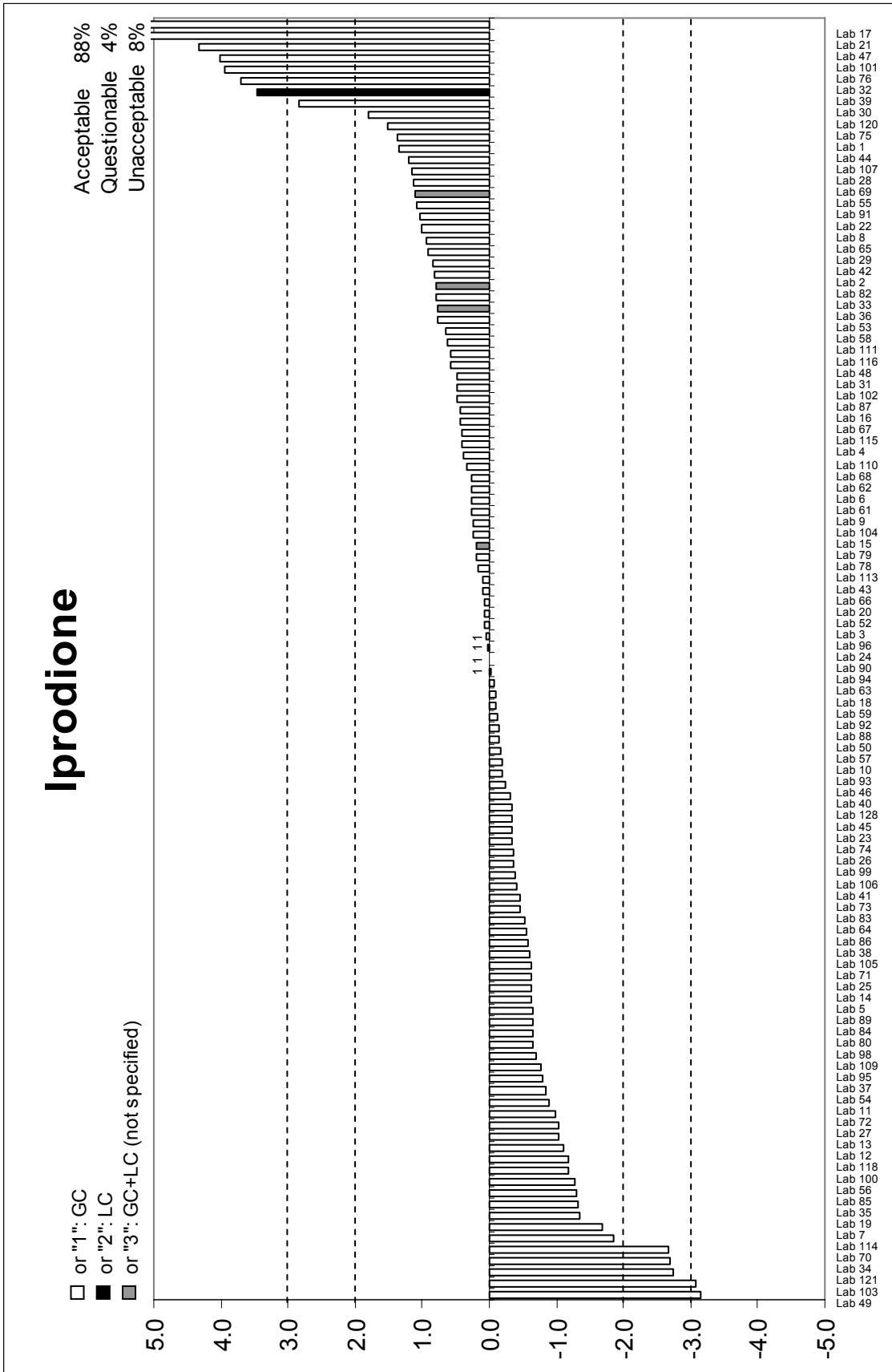
**APPENDIX 4. Graphical Representation of z-scores using FFP (25%)**



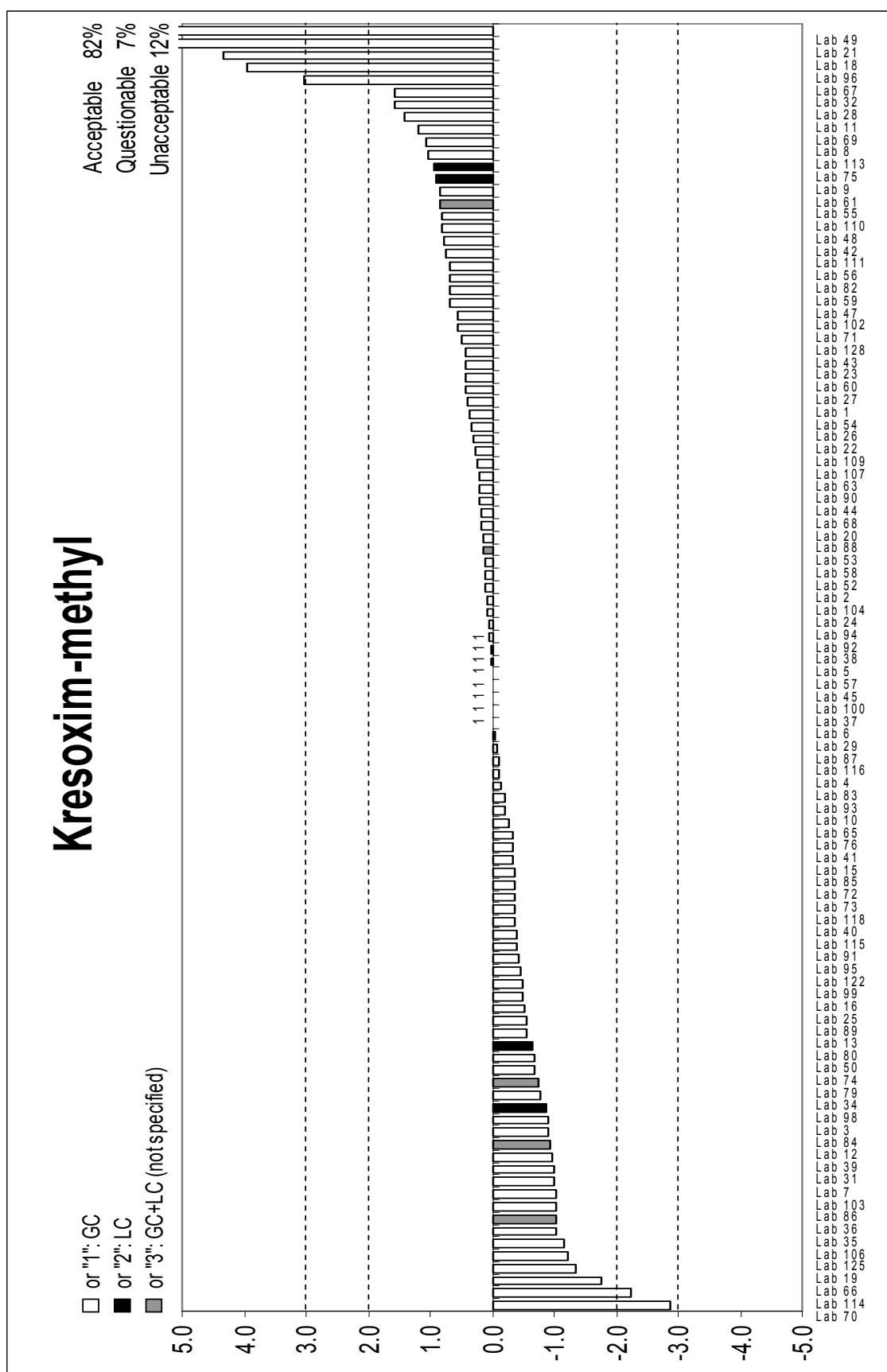
#### **APPENDIX 4. Graphical Representation of z-scores for FFP (25%)**



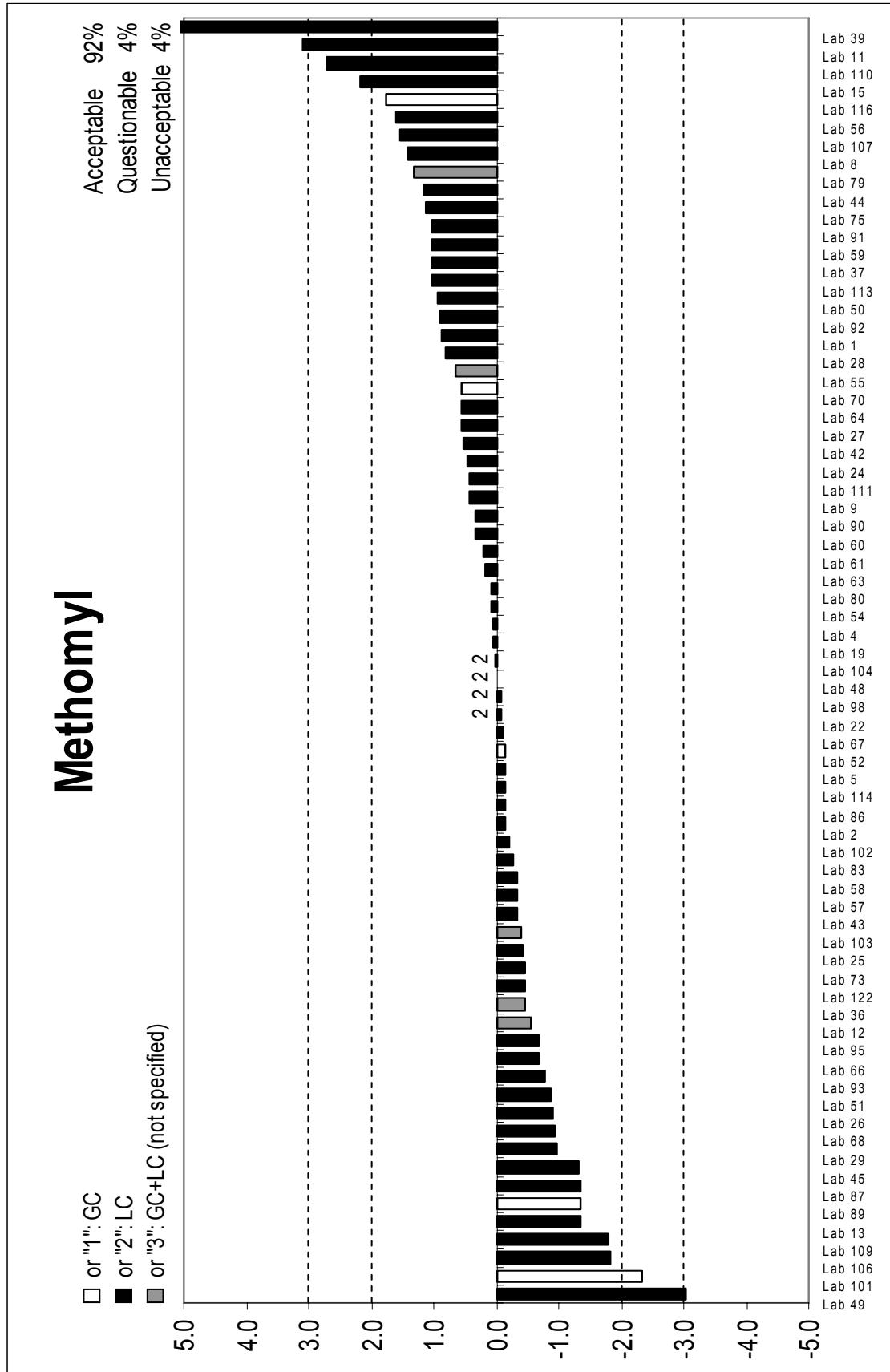
## **APPENDIX 4. Graphical Representation of z-scores using FFP (25%)**



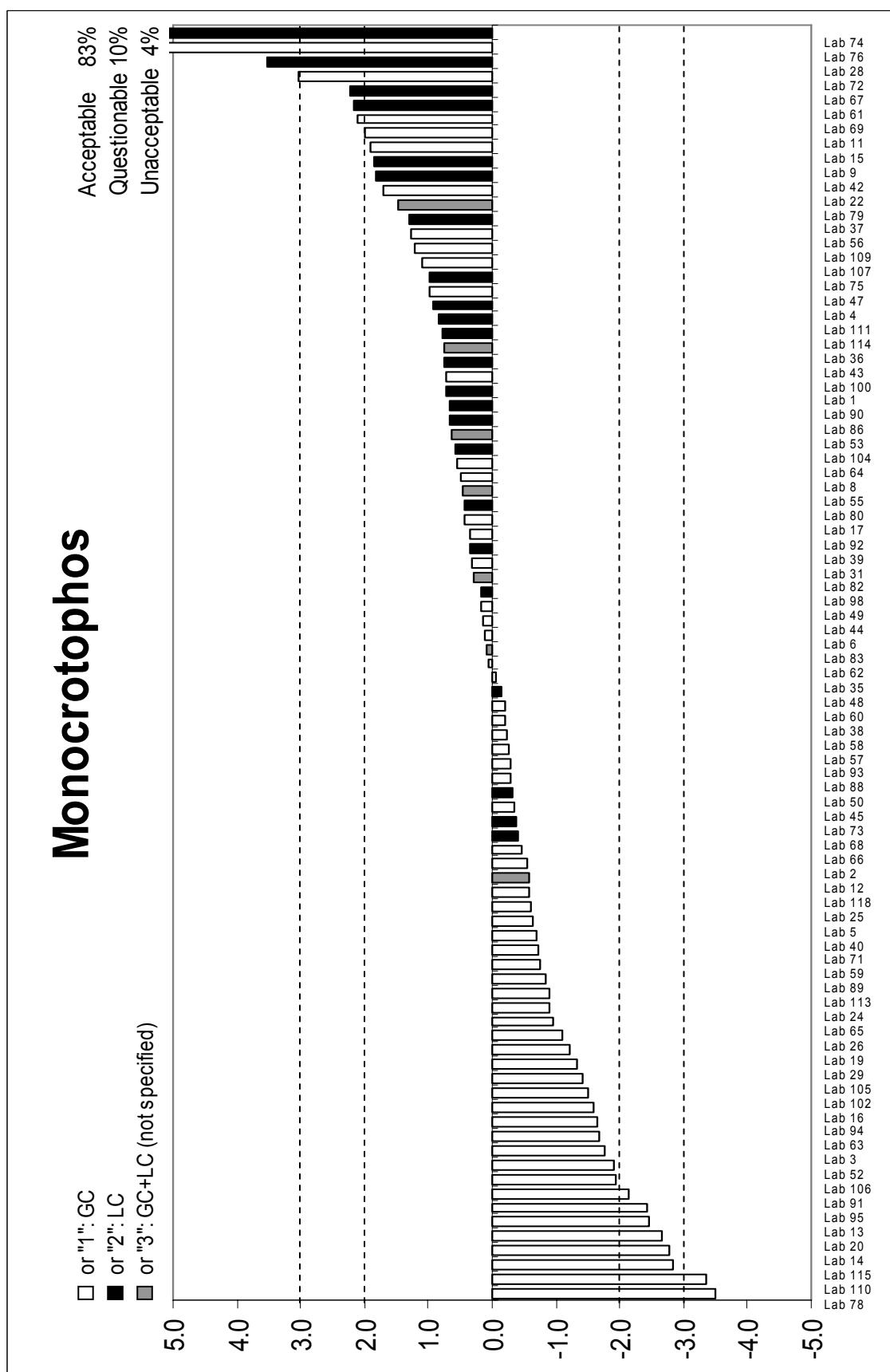
**APPENDIX 4. Graphical Representation of z-scores for FFP (25%)**



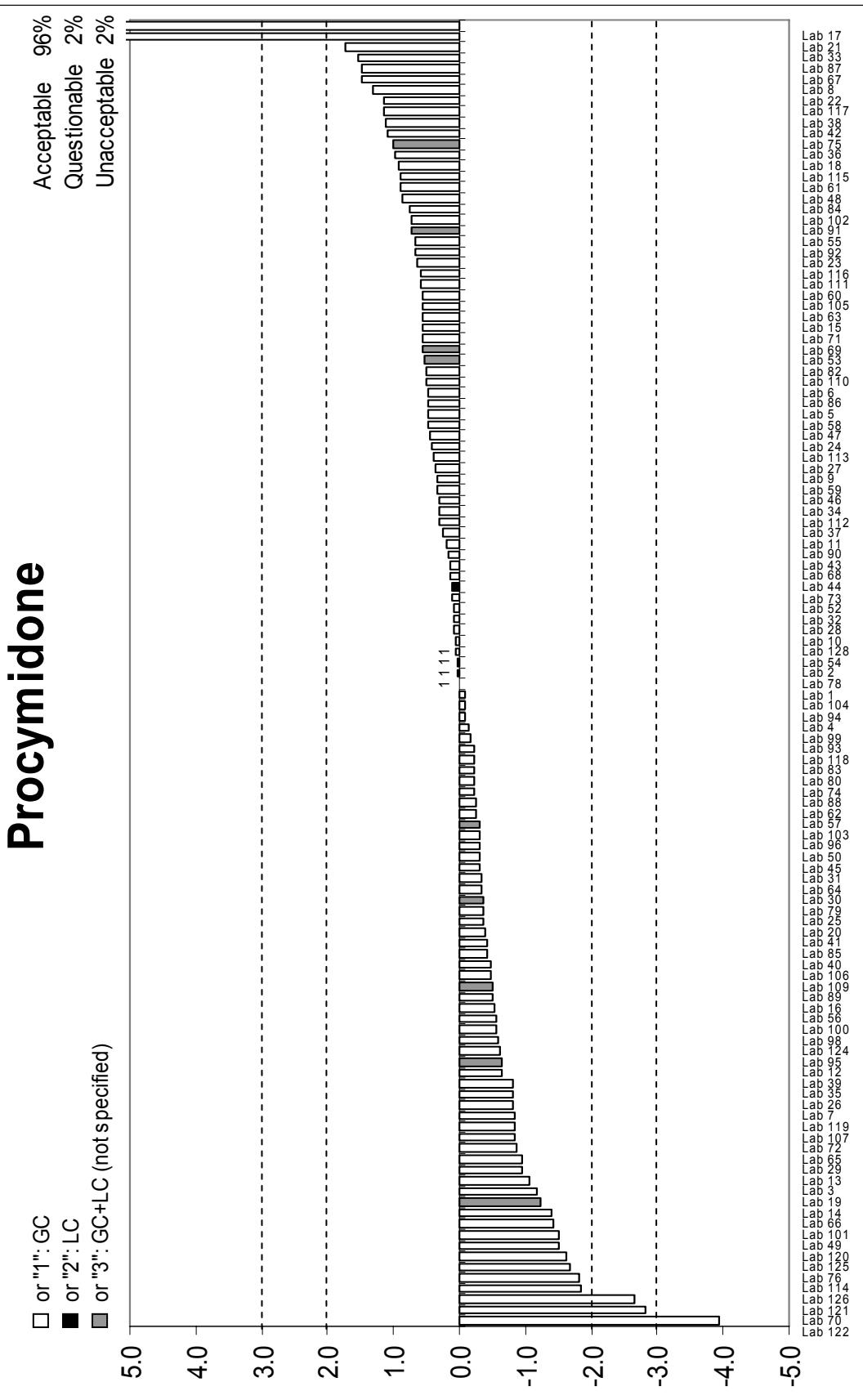
**APPENDIX 4. Graphical Representation of z-scores using FFP (25%)**



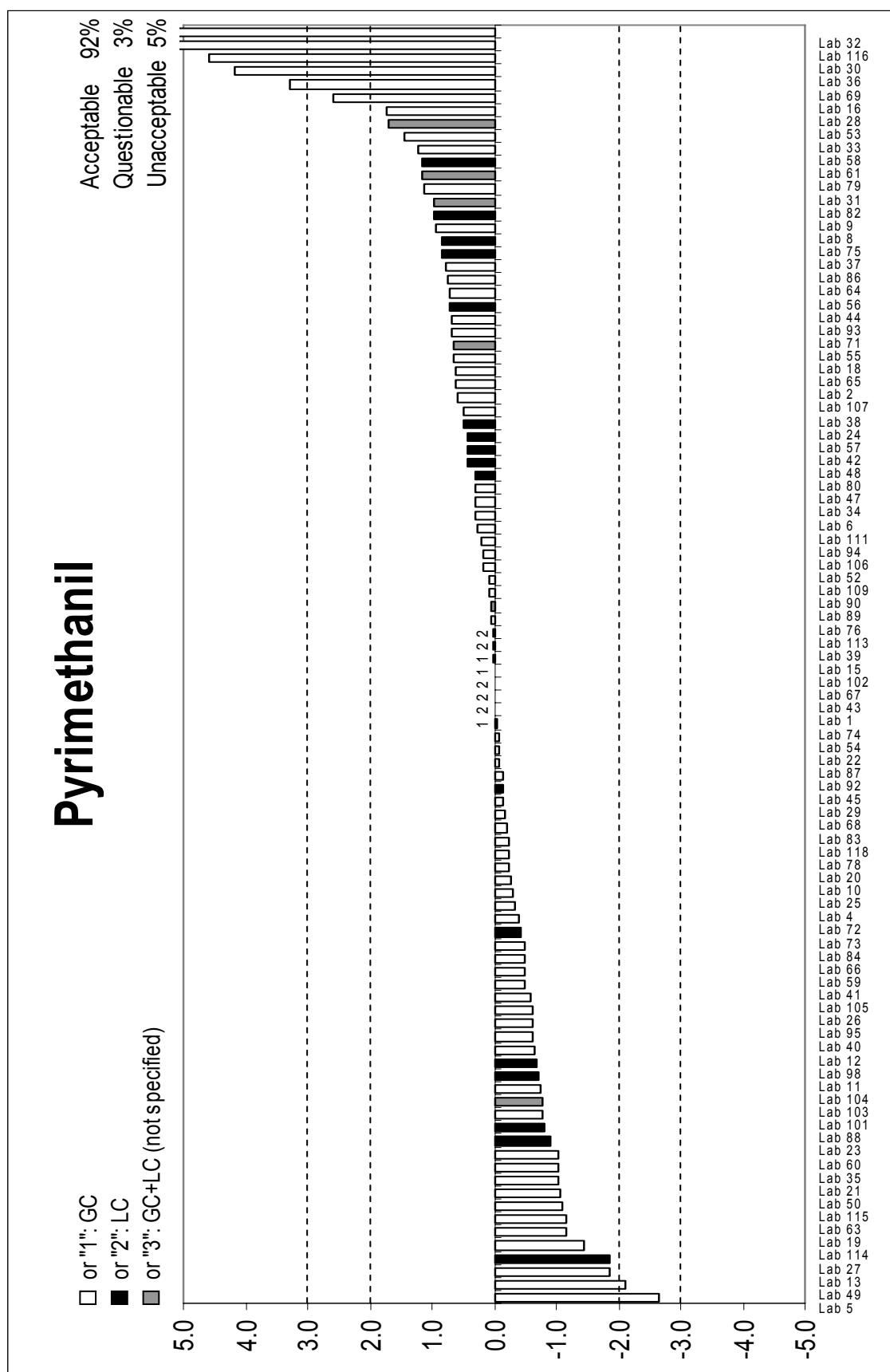
**APPENDIX 4. Graphical Representation of z-scores for FFP (25%)**



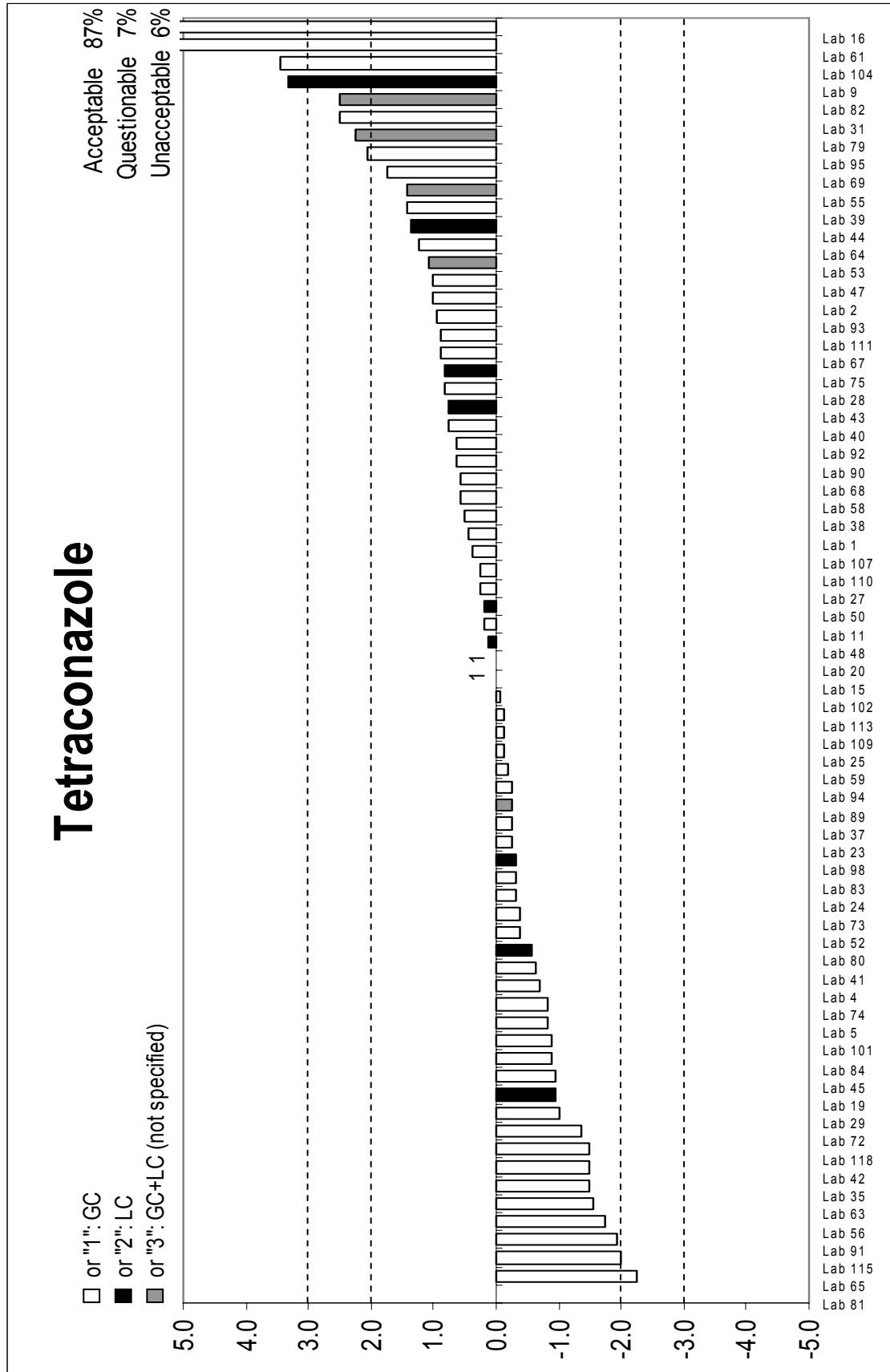
**APPENDIX 4. Graphical Representation of z-scores using FFP (25%)**



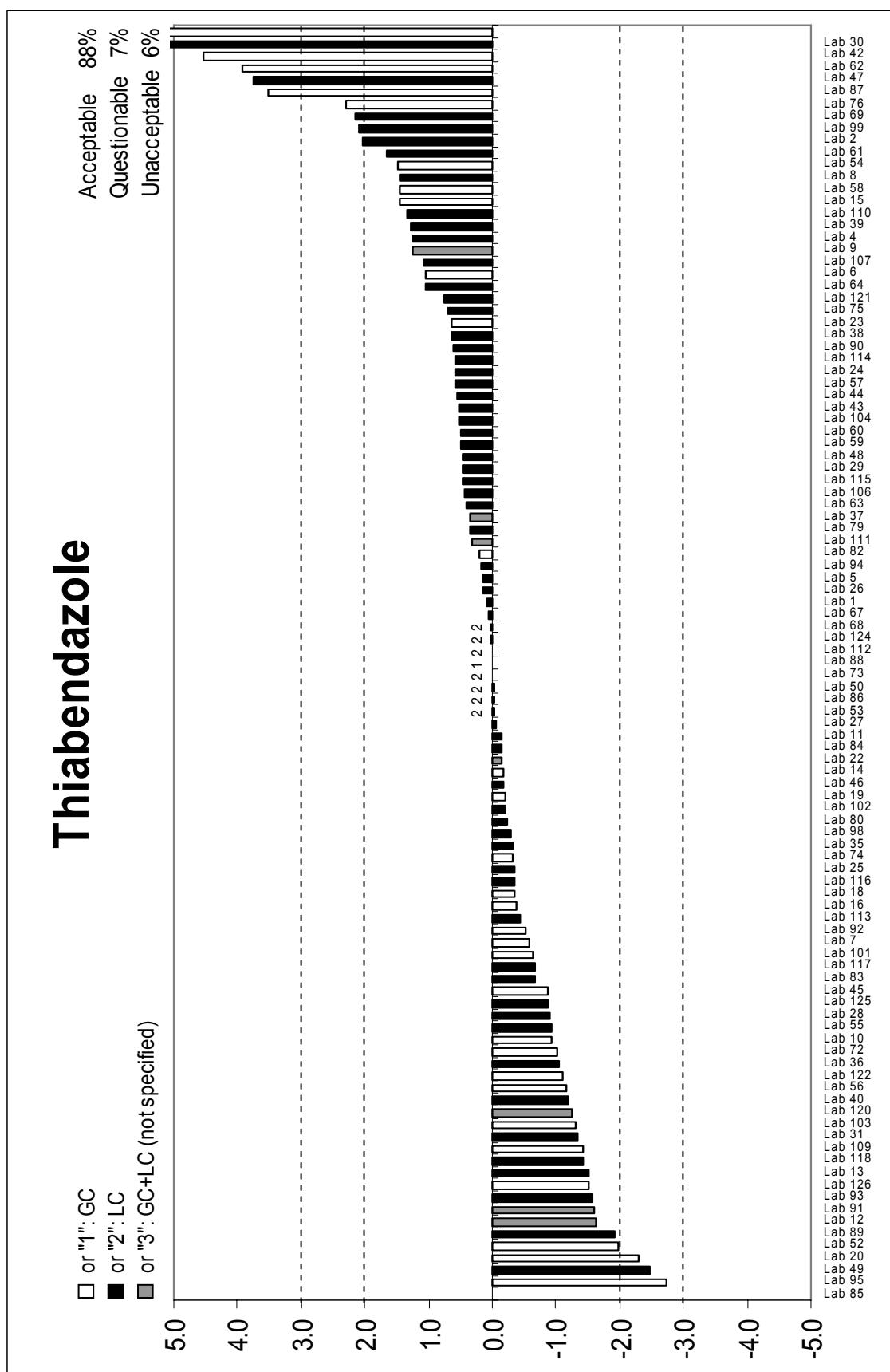
**APPENDIX 4. Graphical Representation of z-scores for FFP (25%)**



**APPENDIX 4. Graphical Representation of z-scores using FFP (25%)**



**APPENDIX 4. Graphical Representation of z-scores for FFP (25%)**



**APPENDIX 5. Combined scores RSZ and SSZ using FFP (25%)**

Lab Code	n	RSZ	SSZ
1	16	0.7	5.0
2	16	1.6	8.2
3	8	-2.2	7.0
4	16	0.6	6.6
5	16	-3.2	23.3
6	12	0.9	2.1
7	9	-1.7	8.1
8	13	6.3	93.8
9	16	3.6	21.4
10	10	-1.4	6.8
11	16	2.6	44.4
12	13	-3.3	13.2
13	11	-4.4	22.5
14	6	-1.6	12.9
15	16	2.9	16.7
16	12	7.9	961.6
17	5	12.5	233.7
18	13	-2.4	63.4
19	16	-3.5	15.5
20	12	-1.5	12.7
21	8	18.6	429.5
22	16	-0.3	14.1
23	16	-1.1	28.3
24	16	-0.1	2.8
25	16	-1.8	5.6
26	14	-1.1	10.4
27	15	-0.9	6.2
28	16	4.0	56.9
29	16	-1.1	19.3
30	11	10.2	348.7
31	13	-0.1	20.9
32	11	7.4	161.3
33	7	1.2	8.6
34	8	-1.7	13.2
35	15	-3.7	20.1
36	16	2.5	42.1
37	16	0.6	8.9
38	13	1.6	7.3
39	16	4.0	165.6
40	13	-1.0	4.0
41	11	-1.8	4.2
42	16	3.4	38.0
43	16	1.1	2.2
44	16	2.8	10.9
45	16	-1.9	17.8
46	5	0.5	1.8
47	15	3.4	113.4
48	16	1.3	3.2
49	12	1.9	332.2
50	16	-1.3	5.8
51	2	-2.0	4.8
52	14	-1.1	11.5

**APPENDIX 5. Combined scores RSZ and SSZ for FFP (25%)**

Lab Code	n	RSZ	SSZ
53	15	7.0	104.2
54	13	0.9	7.4
55	16	3.0	16.2
56	16	-0.8	22.1
57	13	0.0	1.3
58	16	2.9	17.0
59	16	2.3	14.9
60	14	-0.1	8.9
61	16	5.3	56.8
62	7	1.4	21.1
63	16	-0.6	11.0
64	10	1.8	6.8
65	12	-0.7	12.9
66	9	-2.4	8.7
67	16	2.4	34.1
68	16	-0.9	4.3
69	14	8.4	158.1
70	7	-6.2	49.1
71	9	0.3	3.6
72	12	-1.9	24.6
73	16	-1.2	2.4
74	15	5.8	498.7
75	16	3.8	18.3
76	14	4.7	100.3
77		No results	
78	13	-4.0	121.0
79	16	2.7	20.4
80	16	-0.8	6.4
81		No results	
82	13	3.1	15.9
83	16	-1.2	2.5
84	13	-4.5	42.9
85	6	-2.2	11.0
86	13	0.1	5.5
87	13	1.5	23.2
88	14	-2.2	17.2
89	16	-2.5	9.9
90	16	0.8	2.4
91	16	-3.0	65.6
92	16	0.9	3.7
93	16	-1.2	10.1
94	12	-0.3	3.4
95	14	-2.1	19.1
96	7	0.7	27.2
97	0		
98	16	-2.3	7.9
99	9	-0.2	6.0
100	6	-0.5	3.2
101	11	0.4	78.8
102	16	0.2	6.2
103	12	-1.8	18.6
104	16	0.4	14.4

**APPENDIX 5. Combined scores RSZ and SSZ using FFP (25%)**

Lab Code	n	RSZ	SSZ
105	10	-1.6	18.2
106	15	-2.3	12.6
107	16	3.3	27.8
108	2	0.6	2.6
109	14	-0.7	13.5
110	15	2.0	105.0
111	16	2.0	5.4
112	7	-2.6	26.6
113	16	0.2	3.6
114	14	-4.9	42.9
115	14	-2.7	29.5
116	13	1.9	80.5
117	7	-1.5	31.4
118	11	-2.3	7.9
119	5	-1.4	18.5
120	6	-2.6	19.2
121	5	-3.7	25.1
122	6	-3.6	21.0
123	2	0.8	2.2
124	4	-1.6	4.0
125	5	-1.3	6.7
126	5	-4.6	25.3
127		No results	
128	6	-0.5	1.6



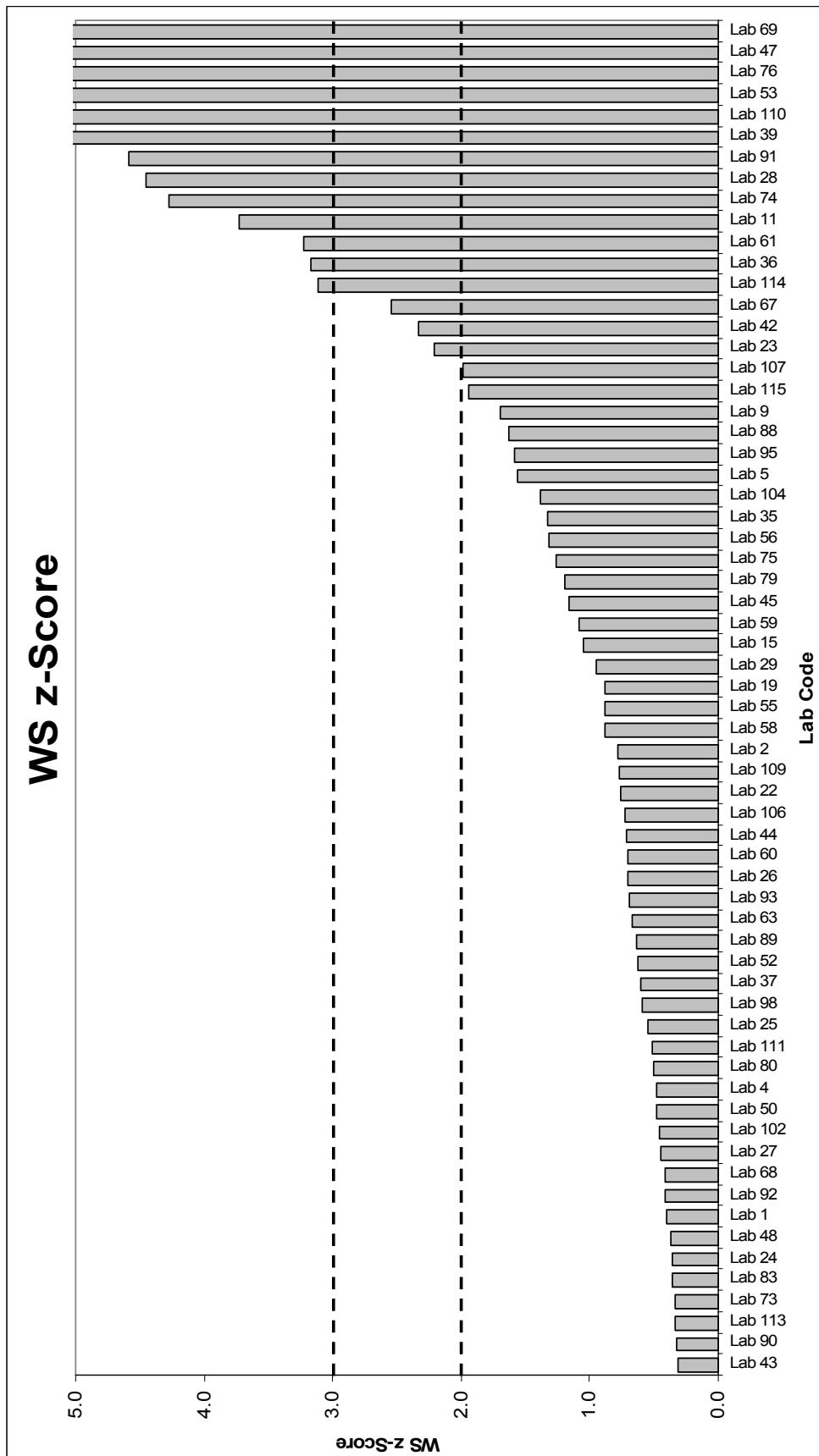
**APPENDIX 6. ‘Weighted Sum of z-score’ and graphical representation for laboratories in Category A.**

Lab Code	Acetamiprid	Carbaryl	Cyprodinil	Diazinon	DIMETHOATEe	Fenhexamid	Fludioxonil	Imidacloprid	Iprodione	Kresoxim-methyl	Methomyl	Monocrotophos	Procymidone	Pyrimethanil	Tetraconazole	N of Pesticides	WSZ
1	0.3	0.0	0.5	0.0	0.1	0.2	0.6	1.0	1.4	0.4	0.9	0.7	0.0	0.0	0.4	16	0.4
2	0.1	0.2	0.8	0.4	0.5	0.2	0.6	0.3	0.8	0.1	0.2	0.6	0.0	0.6	0.9	16	0.8
4	0.3	0.1	0.2	0.4	0.5	0.2	0.4	1.6	0.4	0.1	0.0	0.9	0.1	0.3	0.7	16	0.5
5	0.0	1.3	2.8	0.6	0.5	1.1	1.7	0.3	0.6	0.0	0.1	0.7	0.4	2.7	0.9	16	1.6
9	0.4	0.6	1.0	0.7	1.2	0.9	0.0	0.6	0.3	0.9	0.4	1.8	0.3	1.0	3.2	16	1.7
11	0.5	0.4	3.1	0.7	0.3	0.5	4.1	0.4	0.9	1.4	3.1	1.9	0.2	0.7	0.1	16	3.7
15	1.1	0.6	0.9	0.2	0.9	0.4	0.1	1.6	0.2	0.3	2.2	1.9	0.5	0.0	0.1	16	1.0
19	0.5	0.3	1.2	1.2	0.7	0.8	1.4	0.5	1.3	1.4	0.0	1.2	1.2	1.2	1.0	16	0.9
22	0.9	1.1	0.0	1.0	0.3	1.5	1.4	0.4	1.0	0.3	0.1	1.6	1.3	0.1	0.9	16	0.8
23	0.2	0.5	0.2	0.3	1.3	0.3	1.0	0.8	0.3	0.4	2.8	3.8	0.6	0.9	0.3	16	2.2
24	0.2	0.2	0.3	0.4	0.3	0.5	0.2	0.3	0.0	0.1	0.5	0.9	0.4	0.5	0.4	16	0.4
25	1.0	0.9	0.2	0.6	0.5	0.7	0.8	0.6	0.6	0.5	0.4	0.6	0.4	0.3	0.2	16	0.5
26		0.4	0.1	1.5	1.2	0.3	1.7	0.2	0.4	0.3	0.9	1.1	0.8	0.6		14	0.7
27	0.1	0.3	0.2	0.5	0.0	0.0	0.6	0.5	1.0	0.4	0.6		0.4	1.8	0.2	15	0.5
28	1.0	0.5	0.3	1.6	4.0	2.6	3.1	0.4	1.1	1.6	0.8	3.4	0.1	1.7	0.7	16	4.5
29	1.0	2.0	1.4	0.3	1.6	1.4	0.2	1.4	0.9	0.1	1.0	1.3	1.0	0.1	1.0	16	0.9
35	1.5	0.3	0.7	1.1	0.4	0.6	0.8		1.3	1.0	2.8	0.1	0.8	1.0	1.5	15	1.3
36	0.4	0.7	0.2	0.7	3.9	0.8	0.1	1.5	0.8	1.0	0.5	0.7	1.0	4.2	0.9	16	3.2
37	0.6	0.5	0.2	1.6	0.0	0.8	1.0	0.1	0.8	0.0	1.0	1.2	0.3	0.8	0.3	16	0.6
39	0.6	0.6	3.5	1.4	3.1	0.1	0.1	0.9	3.5	1.0	5.0	0.3	0.7	0.0	1.4	16	5.2
42	0.5	0.4	0.5	0.4	0.7	0.3	0.9	1.0	0.8	0.8	0.5	1.8	1.1	0.4	1.5	16	2.3
43	0.2	0.1	0.3	0.4	0.1	0.4	0.1	0.4	0.1	0.4	0.3	0.7	0.1	0.0	0.7	16	0.3
44	0.8	1.0	0.2	1.0	1.2	0.5	0.6	0.7	1.3	0.2	1.1	0.1	0.1	0.7	1.3	16	0.7
45	1.2	1.1	0.7	2.7	0.2	0.3	1.1	1.5	0.3	0.0	1.3	0.4	0.3	0.1	1.0	16	1.2
47	3.4	0.3	0.1	0.0	7.5	0.8	0.7		4.3	0.7	2.8	0.9	0.4	0.3	0.9	15	7.3
48	0.1	0.0	0.4	0.4	0.4	0.5	0.5	0.1	0.6	0.8	0.0	0.2	0.9	0.4	0.1	16	0.4
50	0.0	0.2	1.2	0.7	0.2	0.7	0.6	0.3	0.1	0.7	0.9	0.4	0.3	1.1	0.1	16	0.5
52		0.2	0.4	1.6	0.4	0.7	0.6		0.1	0.1	0.1	1.9	0.1	0.2	0.4	14	0.6
53	5.0	0.7	1.9	0.8	3.4	3.4	4.8	0.7	0.8	0.1		0.6	0.5	1.7	1.0	15	6.1
55	0.7	0.1	1.7	1.4	0.2	1.1	0.5	1.9	1.1	0.8	0.6	0.4	0.7	0.7	1.4	16	0.9
56	1.4	0.2	2.3	0.8	0.8	0.1	1.3	0.7	1.3	0.7	1.6	1.2	0.5	0.7	1.8	16	1.3
58	1.5	1.1	0.9	0.6	0.3	1.9	1.0	1.7	0.7	0.1	0.3	0.3	0.4	1.2	0.5	16	0.9
59	0.5	0.8	0.7	1.6	2.3	0.9	1.4	0.3	0.1	0.7	1.0	0.8	0.3	0.5	0.2	16	1.1
60		0.8	0.7	1.0	0.5	1.7	0.2	0.9		0.4	0.3	0.2	0.6	1.0	0.9	14	0.7
61	2.5	0.1	0.7	1.0	2.0	1.4	0.7	1.5	0.3	0.9	0.2	2.1	0.9	1.2	5.0	16	3.2
63	0.6	0.8	0.6	0.1	0.9	0.2	1.2	0.5	0.1	0.2	0.2	1.7	0.5	1.1	1.6	16	0.7
67	0.7	3.4	0.5	0.0	0.3	0.9	1.7	1.0	0.4	3.0	0.1	2.2	1.4	0.0	0.8	16	2.5
68	0.9	0.9	0.3	0.0	0.5	0.2	0.8	0.3	0.3	0.2	0.9	0.5	0.1	0.2	0.5	16	0.4

**APPENDIX 6. ‘Weighted Sum of z-score’ and graphical representation for laboratories in Category A.**

Lab Code	Acetamiprid	Carbaryl	Cyprodinil	Diazinon	DIMETHOATEe	Fenhexamid	Fludioxonil	Imidacloprid	Iprodione	Kresoxim-methyl	Methomyl	Monocrotophos	Procymidone	Pyrimethanil	Tetraconazole	N of Pesticides	WSZ
69		2.7	3.1	2.2	1.2	3.5	5.0		1.1	1.2	2.8	2.0	0.5	3.3	1.7	14	8.0
73	0.5	0.5	0.1	0.1	0.2	0.7	0.3	0.3	0.5	0.4	0.5	0.4	0.1	0.4	0.4	16	0.3
74	1.1	0.6	5.0	0.6	1.0	0.0	0.0		0.3	0.7	2.8	5.0	0.2	0.0	0.9	15	4.3
75	0.8	1.1	0.4	0.7	1.2	2.1	0.6	1.1	1.5	0.9	1.1	0.9	1.1	0.8	0.7	16	1.3
76	3.8	0.6	0.2	1.2	1.6	0.2	1.3		3.9	0.3		5.0	1.7	0.0	0.9	14	6.4
79	1.3	0.5	0.3	0.4	2.0	1.8	0.3	0.5	0.2	0.8	1.3	1.4	0.4	1.1	2.2	16	1.2
80	0.4	0.6	1.6	0.0	0.3	0.8	1.0	0.2	0.6	0.6	0.1	0.4	0.2	0.3	0.6	16	0.5
83	0.5	0.3	0.4	0.4	0.2	0.6	0.3	0.6	0.5	0.2	0.3	0.0	0.2	0.2	0.4	16	0.4
88	3.4	0.2	1.9	0.3	0.3	0.6	0.0	0.7	0.1	0.2		0.3	0.3	0.8		14	1.6
89	0.5	1.4	0.2	0.5	0.8	0.4	0.3	0.1	0.6	0.6	1.4	0.9	0.5	0.0	0.3	16	0.6
90	0.2	0.3	0.6	0.5	0.0	0.3	0.6	0.2	0.0	0.2	0.3	0.6	0.2	0.1	0.6	16	0.3
91	2.8	2.0	1.7	1.0	0.8	3.2	3.2	2.9	1.1	0.4	1.0	2.2	0.7	2.7	2.0	16	4.6
92	0.3	0.7	0.7	0.3	0.1	0.4	0.7	0.2	0.1	0.0	0.9	0.3	0.6	0.1	0.6	16	0.4
93	0.6	1.1	0.9	0.3	0.4	1.2	0.9	1.0	0.2	0.2	0.8	0.3	0.2	0.7	0.9	16	0.7
95		0.2	0.5	0.4	0.7	0.4	0.2		0.8	0.4	0.7	2.4	0.6	0.6	2.0	14	1.6
98	1.0	0.0	0.4	1.2	0.3	0.9	0.9	1.1	0.7	0.9	0.1	0.1	0.6	0.7	0.4	16	0.6
102	0.6	0.1	0.2	0.9	1.2	0.4	0.1	0.1	0.5	0.6	0.2	1.5	0.7	0.0	0.1	16	0.5
104	0.2	0.2	0.3	0.9	0.9	0.6	0.1	0.2	0.3	0.1	0.0	0.5	0.1	0.7	3.3	16	1.4
106		0.6	0.1	0.3	0.8	1.0	0.2	0.4	0.4	1.2	1.8	2.0	0.5	0.2	0.9	15	0.7
107	0.3	1.2	0.5	2.7	2.3	2.1	0.5	1.2	1.2	0.2	1.5	1.0	0.9	0.6	0.3	16	2.0
109		1.4	0.0	0.2	1.9	0.5	0.7		0.7	0.3	1.8	1.1	0.5	0.1	0.2	14	0.8
110	1.3		5.0	3.0	0.8	1.7	2.8	1.6	0.4	0.8	2.7	3.4	0.5	2.7	0.2	15	5.6
111	0.2	0.5	1.3	0.1	0.4	0.4	0.2	0.4	0.6	0.7	0.4	0.8	0.6	0.3	0.8	16	0.5
113	0.0	0.0	0.2	0.3	0.2	0.4	0.1	0.0	0.2	1.0	1.0	0.9	0.4	0.0	0.2	16	0.3
114		3.1	1.5	1.9	2.8	2.0	0.9	0.0	1.9	2.2	0.2	0.7	1.8	1.4		14	3.1
115		1.9	1.6	0.1	0.2	0.7	0.5		0.4	0.4	2.8	2.8	0.9	1.1	2.0	14	1.9

**APPENDIX 6. 'Weighted Sum of z-score' and graphical representation for laboratories in Category A.**





**APPENDIX 7. Methods used by participants for determining pesticides.**

ACETAMIPRID											
8	7	6	5	4	3	2	1	LAB CODE	SCOPE OF YOUR METHOD		
NA	NA	NA	D	D	NA	D	D		D		
			M	M		M			M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
			LC-MS/MS			LC-MS/MS		LC-MS/MS	CONFIRMATION METHOD		
			0.002	0.001		0.010		0.05	RL (mg/Kg)		
						98			RECOVERY (%)		
			10	10		10		10	SAMPLE WEIGHT (g)		
			5	5		6		1	EXTRACTION SOLVENT		
			SPE	SPE					CLEAN-UP STEP		
										INTERNAL STANDARD	
										YES	
										NOT APPLY (N/A)	
							5	20	INJECTION VOLUME (µL)		
							20	10	INJECTION TYPE		
										DETERMINATION	
			LC-MS/MS			LC-MS/MS		LC-MS/MS			
			M ANASTASSIAIDES ET AL. JAOAC 86, 412-431 (2003)	QUECHERS ANASTASSIADES		KLEIN, J., ALDER, L. (2003). APPLICABILITY OF GRADIENT LIQUID CHROMATOGRAPHY WITH TANDEM MASS SPECTROMETRY TO THE SIMULTANEOUS SCREENING FOR ABOUT 100 PESTICIDES IN CROPS, J. AOAC INT. 86 (5): 1015-1037				IN HOUSE	REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

ACETAMIPRID									
18	17	16	15	14	13	12	11	10	9
NA	NA	NA	D	NA	D	D	NA	D	LAB CODE
		S		M	M	S	S	S	SCOPE OF YOUR METHOD
									QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
									CONFIRMATION METHOD
									LC-MS/MS
									RL (mg/Kg)
									RECOVERY (%)
									SAMPLE WEIGHT (g)
									EXTRACTION SOLVENT
									LLE ACETONE FOLLOWED BY DICHLOROMETHANE AND PETROLEUM ETHER
									CLEAN-UP STEP
									INTERNAL STANDARD
									INJECTION VOLUME (µL)
									INJECTION TYPE
									DETERMINATION
									REFERENCE METHOD
									IN HOUSE SOP
									LOCAL SOP NO. MR405012

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

ACETAMIPRID										
37	36	35	34	33	32	31	30	29	28	LAB CODE
D	D	D	NA	NA	NA	NA	NA	D	D	SCOPE OF YOUR METHOD
M	S	M					M	S	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
LC-MS/MS	LC-MS	GC-MS					LC-MS/MS	LC-MS/MS	LC-MS/MS	CONFIRMATION METHOD
0.005	0.05	0.02					0.05	0.04	RL (mg/Kg)	
	88	97.5					110	80	RECOVERY (%)	
10	25	20					15	20	SAMPLE WEIGHT (g)	
5	3	3					5	3	EXTRACTION SOLVENT	
O	SPE							GPC	CLEAN-UP STEP	
DIMETHOATE D6									INTERNAL STANDARD	
20	1	2					50	20	INJECTION VOLUME (µL)	
LC-MS/MS (TRIPLE-QUAD)	SPLIT/SPLITLESS	SPLITLESS							INJECTION TYPE	
	GC-FPD GC-ECD GC-EC (SINGLE-QUAD.) LC-MS HPLC-MS HPLC-PICKERING	GC-ECD, NPD					LC-MS/MS	LC-MS/MS	DETERMINATION	
ANASTASSIADES, JOURNAL OF AOAC INT. VOLT 86 NO 2, 2003	EN 12393-1, 2, 3						QUECHERS	QUECHERS	REFERENCE METHOD	

**APPENDIX 7. Methods used by participants for determining pesticides.**

ACETAMIPRID									
44	43	42	41	40	39	38	LAB CODE	SCOPE OF YOUR METHOD	
D	D	D	D	NA	D	NA	NA	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
M	M	M	M	M	M	M	NA	CONFIRMATION METHOD	
GC-MS	LC-MS/MS	GC-NPD	LC/MS/MS	LC/MS/MS	RL (mg/Kg)	0.01	RL (mg/Kg)	RECOVERY (%)	
0.01	0.01	0.01	0.05	0.05	94	105	74.8	SAMPLE WEIGHT (g)	
50	10	10	20	20	50	10	10	EXTRACTION SOLVENT	
2	5	5	3	3	2	5	6 (METHANOL/WATER)	CLEAN-UP STEP	
LL	SPE	LL	SPE	SPE	LL	LL	SPE	INTERNAL STANDARD	
TRIPHENYLPHOSPHATE (TPP)								INJECTION VOLUME (µL)	
5	4	20	4	4	5	4	20	INJECTION TYPE	
LC-MS/MS	LC-MS/MS (ESI +)	LC-MS/MS	GC-ECD, GC-NPD	GC-ECD, GC-NPD	LC-MS/MS	LC-MS/MS	LC/MS/MS	DETERMINATION	
HOUSE METHOD	ANASTASSIAIDES ET AL., JAOAC INT. 86 412-431, MODIFIED	M. ANASTASSIAIDES ET AL., JAOAC INT. 86 (2), 412-431, MODIFIED					LC/MS/MS (BFR)	REFERENCE METHOD	

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

ACETAMIPRID										
62	61	60	59	58	57	56	55	54	LAB CODE	SCOPE OF YOUR METHOD
NA	D	NA	D	D	NA	D	D	NA	NA	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
M		M	M		S	M	M			CONFIRMATION METHOD
LC-MS/MS	HPLC-UV	LC-MS/MS		LC-MS/MS	–	LC-MS/MS	–	LC-MS/MS	RL (mg/Kg)	RECOVERY (%)
0.05		0.05	0.05	0.05	0.206	0.01	0.01	0.01		SAMPLE WEIGHT (g)
		83.5	113		88.9	102				EXTRACTION SOLVENT
20		50	10		30	100	100	100		CLEAN-UP STEP
METHANOL		5	6		1	1	2	2		INTERNAL STANDARD
		O	LLE ON CHEM ELUT		GPC	GPC			GC 1 µL HPLC 20 µL	INJECTION VOLUME (µL)
					3					INJECTION TYPE
					10	2	100	20	LOOP	GC-ECD, GC-NPD, GC-FPD, HPLC-UV, LC-MS/MS
										DETERMINATION
										MODULARE MULTIMETHODE S19
										REFERENCE METHOD
										APPL. OF LC-MS/MS (J. KLEIN, L. ALDERS)
										METOENVORSCHLAG EG-PROFICIENCY TEST 1996/97

**APPENDIX 7. Methods used by participants for determining pesticides.**

ACETAMIPRID											
73	72	71	70	69	68	67	66	65	64	63	LAB CODE
D	NA	NA	NA	NA	D	D	NA	NA	NA	D	SCOPE OF YOUR METHOD
S				S	M	M			M		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-NPD				LC-MS/MS				LC-MS/MS			CONFIRMATION METHOD
0.05				0.01	0.02			0.05			RL (mg/Kg)
78				78.1	80			75			RECOVERY (%)
30				10	10			10			SAMPLE WEIGHT (g)
1				4	5			METHANOL			EXTRACTION SOLVENT
											CLEAN-UP STEP
				LLE + SPE							
											INTERNAL STANDARD
					TRIPHENYLPHOSPHAT						
10					5	10			20		INJECTION VOLUME (µL)
											INJECTION TYPE
					LOOP						
								LC-MS/MS			DETERMINATION
											REFERENCE METHOD
											MULTI RESIDUE METHOD DRAFT BFR
											ANASTASSIADES, M. ET AL.. J. AOAC INT., 86 (2003), 412 - 431 QUECHERS - METHOD
											LOCAL SOP

**APPENDIX 7. Methods used by participants for determining pesticides.**

ACETAMIPRID									
								LAB CODE	
								SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
D	NA	D	D	NA	D	D	D	M	M
M		M	M		S	M	M		
LC-MS/MS		LC-MS			GC-MS		HPLC-DAD		
0.05		0.05	0.05		0.05	0.01	RL (mg/Kg)		
112		89			89	116	RECOVERY (%)		
10		15			10	10	SAMPLE WEIGHT (g)		
5		6	6		DICHLOROMETHANE	METHANOL	3		
					LLE (CHEM ELUT)	SPE	CLEAN-UP STEP		
				O	SPE				
							INTERNAL STANDARD		
							INJECTION VOLUME (µL)		
						1	20	50	
				PARTIAL	SPLITLESS	LC-MS/MS	INJECTION TYPE		
					GC-ECD; GC-NPD; GC-MS; LC-MS/MS; HPLC-FL	JAOAC INTERN., 86, 1015FF (2003)	DETERMINATION		
				LC-MS/MS	GC-MS				
				KLEIN J., ALDERLL (2003) JAOAC INT. 86, 1015-103	INTERNAL METHOD		REFERENCE METHOD		
NO RESULTS									
NO RESULTS									

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>ACETAMIPRID</b>									
								LAB CODE	
								SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
								CONFIRMATION METHOD	
92	91	90	89	88	87	86	85	84	LC-MS/MS
D	D	D	D	ND	NA	NA	NA	NA	LC-MS/MS
M	M	S	M						LC-MS/MS
				HPLC-DAD					HPLC-DAD
0.01	0.01	0.05	0.03	0.01					RL (mg/Kg)
84			80	89					RECOVERY (%)
50	10	50	15	5					SAMPLE WEIGHT (g)
4	METHANOL	3	6	5					EXTRACTION SOLVENT
									CLEAN-UP STEP
LLE	SPE	LLE	LL						
									INTERNAL STANDARD
10	20	OCT-25	1	10					INJECTION VOLUME (µL)
									INJECTION TYPE
				SPLIT/SPLITLESS					
				LOOP					
LC-MS/MS	LC-MS/MS	HPLC-DAD	GC-ECD, FPD, MS HPLC-F-DAD	LC-MS/MS					DETERMINATION
LC-MULTIRESIDUE	DRAFT § 35 LMBG-METHOD			IN-HOUSE					REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

ACETAMIPRID									
101	100	99	98	97	96	95	94	93	LAB CODE
NA	NA	NA	D	NA	NA	NA	NA	D	SCOPE OF YOUR METHOD
			M					M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
			LC-MS/MS					LC/MS/MS	CONFIRMATION METHOD
			0.05				0.01	RL (mg/Kg)	
			89					RECOVERY (%)	
			10				10	SAMPLE WEIGHT (g)	
			1					METHANOL	EXTRACTION SOLVENT
								LLE	CLEAN-UP STEP
									INTERNAL STANDARD
									CARBENDAZIM D4, METHOMYL D3, PENDIMETHALIN D5
								20	INJECTION VOLUME (µL)
									SAMPLE-LOOP
									INJECTION TYPE
									LC/MS/MS
									DETERMINATION
									MULTI-METHOD
									REFERENCE METHOD
									LC-MS MULTI RESIDUE

**APPENDIX 7. Methods used by participants for determining pesticides.**

ACETAMIPRID									
110	109	108	107	106	105	104	103	102	LAB CODE
D	NA	NA	D	NA	NA	D	NA	D	SCOPE OF YOUR METHOD
S		M			S			S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
LC-MS		LC-MS			LC-MS/MS			GC-ECD	CONFIRMATION METHOD
-		0.01			0.006			0.05	RL (mg/Kg)
		91						89	RECOVERY (%)
10		10			10			30	SAMPLE WEIGHT (g)
METHANOL		5			METHANOL			1	EXTRACTION SOLVENT
O		O			DIATOMACEOUS EARTH				CLEAN-UP STEP
O		O							INTERNAL STANDARD
20		1			25			50	INJECTION VOLUME (µL)
HPLC-MS		HPLC - MS (SINGLE-QUAD)			AUTOSAMPLER			AUTOSAMPLER	INJECTION TYPE
MULTI-RESIDUE-METHOD HPLC		QUECHERS ANASTASSIADES			LC-MS/MS			HPLC-UV	DETERMINATION
MULTI-RESIDUE-METHOD HPLC		QUECHERS ANASTASSIADES			APPLICATION NOTE BY WATERS			SOP MR 405012; EXTRACTION OF RESIDUES OF PLANT-PROTECTING MATERIAL FROM FRUIT AND VEGETABLE SAMPLES BY ETHYLACETATE; SOP 309M2; CLEAN UP OF EXTRACTS BY GEL PERMEATION CHROMATOGRAPHY	REFERENCE METHOD

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>ACETAMIPRID</b>									
128	127	126	125	124	123	122	LAB CODE	SCOPE OF YOUR METHOD	CONFIRMATION METHOD
NA		NA	NA	NA	NA	NA	NA	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	RL (mg/Kg)
								RECOVERY (%)	SAMPLE WEIGHT (g)
								EXTRACTION SOLVENT	CLEAN-UP STEP
								INTERNAL STANDARD	INJECTION VOLUME (µL)
								INJECTION TYPE	DETERMINATION
								REFERENCE METHOD	
NO RESULTS									

**APPENDIX 7. Methods used by participants for determining pesticides.**

CARBARYL											
6	5	4	3	2	1	LAB CODE	SCOPE OF YOUR METHOD				
D	D	D	NA	D	D		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX				
M	M	M		S		M	CONFIRMATION METHOD				
GC-MS	LC-MS/MS	GC-MS		GC-MS		LC-MS/MS	RL (mg/Kg)				
0.02	0.001	0.001		0.010		0.05	RECOVERY (%)				
99.5				99			SAMPLE WEIGHT (g)				
15	10	10	-10	10	10	10	EXTRACTION SOLVENT				
4	5	5	5	6	6	1	CLEAN-UP STEP				
GPC	SPE	SPE				SPE	INTERNAL STANDARD				
							YES	INJECTION VOLUME (µL)			
2	20	20	-10	5	5	20	INJECTION TYPE				
SPLITLESS						N/A	DETERMINATION				
GC-MS	LC-MS/MS	LC-MS/MS				LC-MS/MS	REFERENCE METHOD				
ANALYTICAL METHODS FOR PESTICIDE RESIDUES MINISTRY OF HEALTH NETHERLANDS 6TH ED	MANASTASSIADES ET AL. JAOAC 86, 412-431 (2003)	QUECHERS, ANASTASSIADES				KLEIN, J., ALDER, L. (2003): APPLICABILITY OF GRADIENT LIQUID CHROMATOGRAPHY WITH TANDEM MASS SPECTROMETRY TO THE SIMULTANEOUS SCREENING FOR ABOUT 100 PESTICIDES IN CROPS. J. AOAC INT. 86 (5): 1015-1037	IN HOUSE				

**APPENDIX 7. Methods used by participants for determining pesticides.**

CARBARYL									
								LAB CODE	
								SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
								CONFIRMATION METHOD	
15	14	13	12	11	10	9	8	7	
D	NA	NA	D	D	D	D	D	D	
S		M	M	M	S	M	S		
GC-MS		MS-ITD		GC-MS(ITD)	GC-MS	GC-NPD (80)			
0.05		0.02	0.05	0.006	0.01	0.01	0.02		
118		81	78.5	76	91	116			
25		50	30	25	15	30	10		
1		1	1	2	4	1	6 (DICHLOROMETHANE)		
				LLE ACETONE FOLLOWED BY DICHLOROMETHANE AND PETROLEUM ETHER	O				
				GPC	GPC				CLEAN-UP STEP
									INTERNAL STANDARD
									INJECTION VOLUME (µL)
2		1, 2, 5	1	2	5	2	4		
SPLIT/ SPLITLESS		SPLITLESS	SPLIT/ SPLITLESS	SSU/PTV	LOOP	SPLITLESS	SPLITLESS		INJECTION TYPE
GC-NPD			MS-ITD, LC-MS, GC-FPD, GC-ECD	GC-MS(ON TRAP)	GC-MS/MS	GC-ECD, GC-NPD (80)			DETERMINATION
LOCAL SOP NO. MR405012					IN-HOUSE SOP	RAPPORT IISTISAN 97/23			REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>CARBARYL</b>											
<b>25</b>	<b>24</b>	<b>23</b>	<b>22</b>	<b>21</b>	<b>20</b>	<b>19</b>	<b>18</b>	<b>17</b>	<b>16</b>	<b>LAB CODE</b>	
D	D	D	D	D	D	D	D	NA	D	SCOPE OF YOUR METHOD	
M	M	M	M	S	M	S	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
GC-MS	GC-MS	GC-MS	LC-MS/MS	GC-MS/MS	LC-MS/MS	GC-MS	GC-MS/MS	GC-MS/MS	GC-MS/MS	CONFIRMATION METHOD	
<0.02	0.05	0.05	0.05	0.01	0.05	0.01	0.03	0.03	0.05	RL (mg/Kg)	
92	88.7		86.3		102	97	118			RECOVERY (%)	
10	30	5	10	10	100	75	75	15	15	SAMPLE WEIGHT (g)	
6	1	5	METHANOL	1	3	1	1	4	4	EXTRACTION SOLVENT	
SPE	GPC			O						CLEAN-UP STEP	
YES	FENCHLORPHOS									INTERNAL STANDARD	
2	1	50	5	10	2	5	2	10	10	INJECTION VOLUME (µL)	
SPLITLESS	TPOCI	AUTOMATIC TDS SAMPLER	LVI	MANUAL			PULSED SPLITLESS			INJECTION TYPE	
GC-MS	GC-NPD	TDS-IN LINE WITH GC-MS (SINGLE-QUAD) TOTAL ION DETECTION	LC-MS/MS	GC-NPD	LC-MS/MS	GC-MS	GC-MS/MS	GC-MS/MS	GC-MS/MS	DETERMINATION	
	SOP NO.: MR 405012 M1: 308 M2	INTERNAL METHOD								REFERENCE METHOD	

## **APPENDIX 7. Methods used by participants for determining pesticides.**

CARBARYL									
LAB CODE		SCOPE OF YOUR METHOD		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX				CONFIRMATION METHOD	
32	31	30	29	28	27	26	26	LC-MS/MS	LC-MS/MS
D	D	D	D	D	D	D	D	RL (mg/kg)	RL (mg/kg)
S	S	S	S	S	S	S	M	RECOVERY (%)	RECOVERY (%)
GC-MS	MS	DIFFERENT COLUMN	LC-FL	GC-MS	GC-MS	GC-MS		SAMPLE WEIGHT (g)	SAMPLE WEIGHT (g)
0.1	0.05	0.02	0.05	0.02	0.05	0.05	0.01	EXTRACTION SOLVENT	EXTRACTION SOLVENT
104	105	105	87	87	87	87	95		
25	10	10	15	20	20	30	10		
2	6	1	4	3	1	1	6 METHANOL 95%-AMMONIAC ETATE 5%, 20 mM ACETIC ACID		
GPC	O	GPC	SPE (NH <sub>2</sub> )	GPC	GPC	GPC	CLEAN-UP STEP	INTERNAL STANDARD	INTERNAL STANDARD
							CARBARYL C13		
			DICHLOFENTHION						
1	1	1	1	400	20	20	10	INJECTION VOLUME (µL)	INJECTION VOLUME (µL)
SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS		RHEODYNE INJECTOR	SPLITLESS		INJECTION TYPE	INJECTION TYPE
GC-MSD	GC-ECD GC-NPD	GC-ECD GC-NPD	HPLC-FL	HPLC-FL	HPLC-UV	HPLC-UV	LC-MS/MS	DETERMINATION	DETERMINATION
\$35 LMBG 00.00-34	ISTISAN 97/23	RAPP ISTISAN 97/23	MINI LUKE		HUNGARIAN CENTRAL SOP NO.: 405012 M1			REFERENCE METHOD	REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>CARBARYL</b>									
40	39	38	37	36	35	34	33	LAB CODE	SCOPE OF YOUR METHOD
D	D	D	D	D	D	D	NA	NA	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
M	M	M	M	S	M				CONFIRMATION METHOD
GC-MS	LC/MS/MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS			RL (mg/Kg)
0.10	0.01	0.05	0.02	0.05	0.02	0.02			RECOVERY (%)
	117				92	120			
15	10	10	10	25	25	20			SAMPLE WEIGHT (g)
4	6	(METHANOL/WATER)	5	5	3	3			EXTRACTION SOLVENT
									CLEAN-UP STEP
				O					INTERNAL STANDARD
					TPP				INJECTION VOLUME (µL)
1	20		2	1	1	2			INJECTION TYPE
									DETERMINATION
GC-NPD, GC-ECD, GC-MS(IRD)	LC/MS/MS	GC-ECD GC-NPD GC-MS	GC-MS (SINGLE QUAD)	GC-FPD GC-ECD GC-MS (SINGLE QUAD.) LC-MS HP C-MS HPLC-PICKERING	GC-ECD, NPD				REFERENCE METHOD
ANALYTICAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS 6TH ED., 1996. GENERAL INSPECTORATE FOR HEALTH PROTECTION MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS	LC/MS/MS (BFR)		ANASTASSIADES ET AL. JAOAC INT. VOL. T 86 NO. 2, 2003	EN 12393-1, 2, 3					

**APPENDIX 7. Methods used by participants for determining pesticides.**

CARBARYL									
48	47	46	45	44	43	42	41	LAB CODE	SCOPE OF YOUR METHOD
D	D	NA	D	D	D	D	D	D	D
M	S		S	M	M	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
LC-MS/MS	GC-NPD	GC-MS	GC-MS	GC-MS	GC-MS/MS	GC-MS/MS	GC-MS	GC-MS	CONFIRMATION METHOD
0.01	0.05	0.01	0.01	0.01	0.01	0.01	0.05	RL (mg/Kg)	RECOVERY (%)
10	10	86	25	50	95	103	92.1		
6	5	1	2	5	10	10	20	SAMPLE WEIGHT (g)	
					5	5	3	EXTRACTION SOLVENT	
								CLEAN-UP STEP	
								LLE	
								SPE	
								GPC	
								LLE	
									TRIPHENYLPHOSPHATE (TPP)
									INTERNAL STANDARD
20	4			200	5	4	20		INJECTION VOLUME (µL)
								RHEODYNE	
									SPLITLESS
LC-MS/MS	GC-ECD, GC-NPD, GC-MS (SINGLE QUAD)	HPLC -ON LINE DERIVATIZATION - FD	LC-MS/MS	LC-MS/MS (ESi+)	LC-MS/MS				DETERMINATION
KLEIN, J.; ALDER, L. JOURNAL OF AOAC INT., 86, 1015- 1037	M. ANASTASSIADES, S. J. LEHOTAY, D. STAIBAHER, F. J. SCHENCK; J. AOAC INT., 86 (2003) 412-431	EXTRACTION AND CLEAN UP AS IN EN 12393 P METHOD	HOUSE METHOD	ANASTASSIADES ET AL. JAOAC INT. 86 82003) 412-431, MODIFIED	M. ANASTASSIADES ET AL. JAOAC INT. 86 82003) 412-431, MODIFIED				REFERENCE METHOD

## **APPENDIX 7. Methods used by participants for determining pesticides.**

CARBARYL										
	57	56	55	54	53	52	51	50	49	LAB CODE
D	D	D	D	D	D	D	D	D	D	SCOPE OF YOUR METHOD
M	S	M	M	S	M	S	S	S	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
LC-MS/MS	GC-MS	HPLC-UV	HPLC-DAD	GC-MS	GC-MS	HPLC-FLD	LMS	GC-NPD	CONFIRMATION METHOD	
0.01	1.72	0.01	0.01	0.05	0.05	0.01	0.05	0.05	RL (mg/Kg)	
	118.4	64.1	67			84	103	RECOVERY (%)		
25	30	100	37.5	100	10	50	50	SAMPLE WEIGHT (g)		
5	1	2	1	3	5	6	4	EXTRACTION SOLVENT		
								CLEAN-UP STEP		
								LLE		
									INTERNAL STANDARD	
									INJECTION VOLUME (µL)	
5	1	GC 1 µL HPLC 20 µL	20	10	1.5	10	50	2	INJECTION VOLUME (µL)	
									INJECTION TYPE	
LC-MS/MS	GC-MS/ITD	GC-ECD, GC-NPD, GC-FPD, HPLC-UV, LC-MS/MS	HPLC-DAD, FLD	POST COLUMN DERIVATIZATION HPLC-FL	GC-ECD, GC-NPD, GC-MS	HPLC-FLD	HPLC-FLD	GC-FPD, GC-NPD, GC-MS(ITD)/MS	DETERMINATION	
									REFERENCE METHOD	
									METHOD R <sup>®</sup> EN 12333-2:1999	

**APPENDIX 7. Methods used by participants for determining pesticides.**

CARBARYL									
								SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
65	64	63	62	61	60	59	58	LAB CODE	
D	D	D	NA	D	D	D	D	SCOPE OF YOUR METHOD	
M	S	M	M	M	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
GC-MS	GC-MS	LC-MS/MS	GC-MS	LC-MS/MS	GC/MSD	GC-ECD/NPD	GC-ECD/NPD	CONFIRMATION METHOD	
0.05	0.01	0.05	0.1	0.05	0.05	0.1	0.1	RL (mg/Kg)	
91	100	71			110	105		RECOVERY (%)	
5	5	10	50	15	10	15		SAMPLE WEIGHT (g)	
1	6	METHANOL	3	1	5	4		EXTRACTION SOLVENT	
								CLEAN-UP STEP	
GPC			GPC		O				
AZOBENZENE+RONNELL+TRIPHENYLPHOSPHATE				ISODRIN	1	PCB119 (ECD) TRIFLURALIN (GC-NPD)		INTERNAL STANDARD	
2	10	20	2	2	1	20	1	INJECTION VOLUME (µL)	
ON-COLUMN			PTV	SPLIT, SPLITLESS				INJECTION TYPE	
GC-(ECD/FPD/NPD)	HPLC - FLD DERIVATIZED POST-COLUMN	LC-MS/MS	GC-MS	GC-ECD/NPD/MS LC-MS/MS	HPLC FLD WITH ND	GC-ECD/NPD		DETERMINATION	
INTERNAL METHOD	MULTIRESIDUE WITH EXTRELUT	MULTI RESIDUE METHOD DRAFT BFR	DFG S19 (ASU \$35 LMBGL 00.00-34)				ANALMETHODS FOR PESTICIDES RESIDUES IN FOODSTUFFS (NL)	REFERENCE METHOD	

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>CARBARYL</b>									
73	72	71	70	69	68	67	66	LAB CODE	SCOPE OF YOUR METHOD
D	NA	NA	D	D	D	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
S		M	M	S	M	S	S	S	CONFIRMATION METHOD
GC-MS		GC-MS/MS	GC-MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	RL (mg/Kg)
0.05		0.05	0.05	0.01	0.02	0.05	0.05	0.05	RECOVERY (%)
86			105	77.8	80	115			SAMPLE WEIGHT (g)
30		15	25	20	10	15			EXTRACTION SOLVENT
1		5	1	4	5	5			CLEAN-UP STEP
GPC; O		O	GPC	LLE + SPE	SPE	SPE	SPE	SPE	INTERNAL STANDARD
				TRIPHENYLPHOSPHATE					INJECTION VOLUME (µL)
RHEODYNE									INJECTION TYPE
HPLC-UV			GC-ECD/TSD/PFPD GC-MS/MS	GC-NPD, GC-ECD	LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	DETERMINATION
LOCAL SOP			QUECHERS	MULTIRESIDUE METHOD 1, ANALYTICAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS, SIXTH EDITION	MINI-LUKE EXTRACTION	ANASTASSIADES, M. ET AL., J. AGAC INT., '86 (2003) 412 - 431 QUECHERS - METHOD			REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

CARBARYL									
82	81	80	79	78	77	76	75	74	LAB CODE
D	D	D	D	D	D	D	D	D	SCOPE OF YOUR METHOD
M	M	M	M	M	S	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-ECD	LC-MS/MS	GC/MS	GC/MS	GC/MS	GC/MS	GC/MS	GC/MS	GC/MS	CONFIRMATION METHOD
0.03	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	RL (mg/Kg)
80	110	110	80	80	136	116	84.8	84.8	RECOVERY (%)
10	10	15	15	15	10	10	25	25	SAMPLE WEIGHT (g)
5	6	6	1	1	DICHLOROMETHANE	6 METHANOL	3	3	EXTRACTION SOLVENT
NO RESULTS									
O (PSA)									
NO RESULTS									
SPLITLESS AND ON COLUMN	GC/ECD; GC/NPD; GC/FID; GC/MS; HPLC-FL, HPLC-DAD	LC-MS/MS	SPLITLESS	SPLITLESS	GC/MS	GC/MS (SINGLE-QUAD)	LC-MS/MS	20	INJECTION VOLUME (µL)
QUECHERS, M. ANASTASSIADES, S. J. LEHOTAY, D. STAIBNAHNER, F. J. SCHENK, J. AOAC INT., 86 (2003), 412-431	KLEIN,J., ALDER,L (2003)	KLEIN,J., ALDER,L (2003)	INTERNAL METHOD	INTERNAL METHOD	JAOAC INT., 86, 1015-103	JAOAC INT., 86, 1015FF (2003)	HPLC-DAD	1	INTERNAL STANDARD
REFERENCE METHOD									

**APPENDIX 7. Methods used by participants for determining pesticides.**

CARBARYL									
								LAB CODE	
								SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
								CONFIRMATION METHOD	
90	D	89	D	88	D	87	D	86	85
S	M	M	M	M	M	D	NA	D	D
GC-MS	HPLC-FID				GC-MS(TD)		GC-MS	GC-MS	
0.01	0.03	0.008	0.008		0.025		0.02	0.05	RL (mg/Kg)
95	105	91	91		92		80	90	RECOVERY (%)
50	15	5	10		15		50	30	SAMPLE WEIGHT (g)
3	6	5	6 METHANOL		4		2	1	EXTRACTION SOLVENT
LL	LL	LL	LL						CLEAN-UP STEP
10-25	1	10	20		1		1	3	INJECTION VOLUME (µL)
HPLC-DAD	GC-ECD FPD MS HPLC-FID DAD			SPLIT/ SPLITLESS		SPLIT/ SPLITLESS		SPLITLESS	INJECTION TYPE
	LC-MS/MS	LC-MS/MS	GC-MS(TD)		GC-MS (SINGLE-QUAD)		GC-MS		DETERMINATION
			JAOAC 86, 5, 1015-1037						REFERENCE METHOD
			IN-HOUSE						EN NF 12393

**APPENDIX 7. Methods used by participants for determining pesticides.**

CARBARYL									
								SCOPE OF YOUR METHOD	
		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX							
LAB CODE									
98	97	96	95	94	93	92	91		LAB CODE
D	NA	NA	D	D	D	D	D		SCOPE OF YOUR METHOD
M		S	M	M	M	M	S		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
LC-MS/MS	GC-MS	GC-NPD	LC-MS/MS	GC-MS	GC-MS	GC-MS	GC-MS		CONFIRMATION METHOD
0.05	0.2	0.05	0.02	0.02	0.02	0.02	0.02		RL (mg/Kg)
127	89	100.6			107				RECOVERY (%)
10	25	10	10	50	50	50	50		SAMPLE WEIGHT (g)
1	5	4	6. METHANOL	4	4	3	3		EXTRACTION SOLVENT
									CLEAN-UP STEP
		SPE	SPE	LLE	LLE	LLE	LLE		
CARBENDAZIM D4, METHOMYL D3, PENDIMETHALIN D5		YES			ALDRIN, DITALIMPHOS	YES			INTERNAL STANDARD
10		3	2	20	1	1	1		INJECTION VOLUME (µL)
		PTV	SPLITLESS	SAMPLE LOOP	SPLITLESS	SPLITLESS	SPLITLESS		INJECTION TYPE
LC-MS/MS	GC-ECD, GC-NPD, GC-MS	GC-ECD	LC-MS/MS	GC-ECD, GC-NPD, GC-MS	GC-NPD	GC-NPD	GC-NPD		DETERMINATION
LC-MS MULTI RESIDUE	FILLION ET AL. JOURNAL OF AOAC INTERNATIONAL 78-5-1995	MULTI-METHOD	OFFICIAL METHOD OF ANALYSIS (1990) 15TH ED., 985.22 AOAC ARLINGTON VA	DFG-S8	REFERENCE METHOD				

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>CARBARYL</b>									
105	104	103	102	101	100	99	LAB CODE	SCOPE OF YOUR METHOD	
D	D	D	D	D	NA	D		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
M	S	S	S	M		M		CONFIRMATION METHOD	
GC-NPD	GC-MS	GC-MS	HPLC-DAD	GC-MS		GC-MS		RL (mg/Kg)	
0.5	0.005	0.020	0.05	0.05		0.05		RECOVERY (%)	
		80	101		90			SAMPLE WEIGHT (g)	
100	100	50	30	10	10	20		EXTRACTION SOLVENT	
4	2	1	1	2	2	4		CLEAN-UP STEP	
								INTERNAL STANDARD	
								INTERNAL STANDARD	
								INJECTION VOLUME (µL)	
						1		PULSED SPLITLESS PTV	
								DETERMINATION	
								REFERENCE METHOD	
MULTIRESIDUE METHOD: AOAC 985.22/90 MODIFIED	LMBG §35 L-0.00.-34	ANN.FALS.EXP.CH M. SEPT-OCT 1974-87 NJ721-722 PPF513-524	SOP MR 405012; EXTRACTION OF RESIDUES OF PLANT-PROTECTING MATERIAL FROM FRUIT AND VEGETABLE SAMPLES BY ETHYL-ACETATE; SOP 308W2; CLEAN UP OF EXTRACTS BY GEL PERMEATION CHROMATOGRAPHY						

## **APPENDIX 7. Methods used by participants for determining pesticides.**

CARBARYL									
LAB CODE		SCOPE OF YOUR METHOD		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		CONFIRMATION METHOD		REFERENCE METHOD	
112	111	110	109	108	107	106			
D	D	NA	D	NA	D	D			
S	S	S	S	M	S	S			
GC/MS	LC-MS/MS	LC-MS/MS	GC/MS	GC-NPD	GC-NPD	RL (mg/Kg)	RL (mg/Kg)		
0.05	0.05	0.01	0.02	0.02	0.02				
90	99.3					81			
10	50	10	100	100	25				
6	6 METHANOL	1	2	6 ETHYLACETATE/DICHLOROMETHANE	EXTRACTION SOLVENT				
				CLEAN-UP STEP					
				GPC					
				SPE					
					INTERNAL STANDARD				
						INJECTION VOLUME (µL)			
						1			
					SPLITLESS		INJECTION TYPE		
HPLC/DAD	LC-MS/MS	LC-MS/MS	GC-ECD-ECD; GC-NPD; GC-MS (SINGLE-QUAD)	GC-PFPD GC-NPD GC-MS	DETERMINATION				
ISTISAN 97/23	VALIDATED IN HOUSE METHOD NACH MULTI- METHOD, KLEIN UND ALDER - DFG WORKSHOP PRESENTED	S19-DFG							

**APPENDIX 7. Methods used by participants for determining pesticides.**

CARBARYL											
123	122	121	120	119	118	117	116	115	114	113	LAB CODE
NA	NA	NA	D	NA	D	M	S	S	D	D	SCOPE OF YOUR METHOD
			S			M	S	S	S	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
			GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-NPD	GC-NPD	CONFIRMATION METHOD
			0.03	0.03	0.01	0.01	0.01	0.01	0.05	0.05	RL (mg/Kg)
			83.8		57			71.1	95		RECOVERY (%)
			10	40	15	50	50	50	30		SAMPLE WEIGHT (g)
			3	1	6	3	6	6	1		EXTRACTION SOLVENT
			O		GPC	SPE	SPE				CLEAN-UP STEP
											INTERNAL STANDARD
						YES					INJECTION VOLUME (µL)
								1	1	1	
			AUTO	SPLITLESS	SPLITLESS	ON COLUMN	SPLITLESS				INJECTION TYPE
			GC-ECD GC-MS	GC-MS	GC-ECD, GC-NPD, GC-MS(TD)	GC-EC/ GC-PFPD GC-MS	GC-MS				DETERMINATION
			ISS B6		RAPPORTO ISTISAN 97/23						SOP MR 405012; EXTRACTION OF RESIDUES OF PLANT-PROTECTING MATERIAL FROM FRUIT AND VEGETABLE SAMPLES BY ETHYL-ACETATE NFEN12393
											REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

CARBARYL						
128	127	126	125	124	LAB CODE	SCOPE OF YOUR METHOD
D		NA	NA	NA		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
M						CONFIRMATION METHOD
GC-MS						RL (mg/Kg)
0.05						RECOVERY (%)
20 / 5						SAMPLE WEIGHT (g)
1						EXTRACTION SOLVENT
CLEAN-UP STEP						
NO RESULTS						
INTERNAL STANDARD						
2						
INJECTION VOLUME (µL)						
SPLITLESS						
DETERMINATION						
GC-ECD, GC-FPD, GC-MSD, GC-MS(TD)						
MODIFIED LUKE						
REFERENCE METHOD						

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>CYPRODINIL</b>								
5	4	3	2	1	LAB CODE	SCOPE OF YOUR METHOD		
D	D	D	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		
M	M	M	M	M	M	CONFIRMATION METHOD		
GC-MS	GC-MS/MS	GC-MS	GC-MS	LC-MS/MS	LC-MS/MS	CONFIRMATION METHOD		
0.05	0.021	0.02	0.010	0.05	RL (mg/Kg)	RECOVERY (%)		
10	10	85.0	92	10	10	SAMPLE WEIGHT (g)		
5	5	15	15	1	1	EXTRACTION SOLVENT		
SPE	SPE	NO	NO	SPE	SPE	CLEAN-UP STEP		
(MIREX)TPP	TRIPHENYL PHOSPHATE ANTHRACENE			YES	YES	INTERNAL STANDARD		
1	2	10	1	1	20	INJECTION VOLUME ( $\mu$ L)		
SPLIT/ SPLITLESS	SPLIT/ SPLITLESS	SPLIT	SPLITLESS	N/A	N/A	INJECTION TYPE		
GC-MS	GC-MS	GC-MS/MS	GC-NPD	LC-MS/MS	LC-MS/MS	DETERMINATION		
MANASTASSIADES ET AL. JAOAC 86: 412-431 (2003)	QUECHERS, ANASTASSIADES			LUKE, M.A., FROBERG, J.E., DOOSE, G.M., MASUMOTO, H.T. (1981); IMPROVED MULTIRESIDUE GAS CHROMATOGRAPHIC DETERMINATION OF ORGANOPHOSPHORUS, ORGANONITROGEN, AND ORGANOHALOGEN PESTICIDES IN PRODUCE, USING FLAME PHOTOMETRIC AND ELECTROLYTIC CONDUCTIVITY DETECTORS. J. ASSOC. OFF. ANAL. CHEM. 64(5): 1187-1195	IN HOUSE	REFERENCE METHOD		

**APPENDIX 7. Methods used by participants for determining pesticides.**

CYPRODINIL									
									LAB CODE
									SCOPE OF YOUR METHOD
14	13	12	11	10	9	8	7	6	
NA	NA	D	D	D	M	M	S	M	D
									QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
									CONFIRMATION METHOD
									GC-MS
									RL (mg/Kg)
									RECOVERY (%)
									SAMPLE WEIGHT (g)
									EXTRACTION SOLVENT
									CLEAN-UP STEP
									GPC
									INTERNAL STANDARD
									INJECTION VOLUME (µL)
									INJECTION TYPE
									DETERMINATION
									ANALYTICAL METHODS FOR PESTICIDE RESIDUES MINISTRY OF HEALTH NETHERLANDS 6TH ED
									REFERENCE METHOD
									RAPPORT ISTITISAN 97/23
									IN-HOUSE SOP

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>CYPRODINIL</b>									
23	22	21	20	19	18	17	16	15	LAB CODE
D	D	D	D	D	D	NA	D	D	SCOPE OF YOUR METHOD
M	M	M	S	M	S	M	M	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-MS	GC-MS	GC-MS/MS	GC-NPD	GC-MS(FTD)	GC-MS	GC-MS/MS	GC-MS/MS	GC-MS	CONFIRMATION METHOD
0.05	0.06	0.01	0.05	0.02	0.03	0.02	0.02	0.05	RL (mg/Kg)
79.8			92	77	92			105	RECOVERY (%)
5	50	10	100	75	75	15	15	25	SAMPLE WEIGHT (g)
5	6	1	3	1	1	4	4	1	EXTRACTION SOLVENT
LLE (TOLUENE)	O								CLEAN-UP STEP
FENCHLORPHOS				DITALIMPHOS	ALDRIN				INTERNAL STANDARD
50	1,5	10	2	2	2	10	10	2	INJECTION VOLUME (µL)
AUTOMATIC TDS SAMPLER	SPLITLESS	LVI	MANUAL	SPLITLESS	PULSED SPLITLESS	LVI SPLITLESS	LVI SPLITLESS	SPLIT/ SPLITLESS	INJECTION TYPE
TDS-IN LINE WITH GC-MS (SINGLE-QUAD) TOTAL ION DETECTION	GC-NPD	GC-MS/MS	GC-ECD	GC-MS	GC-MS/MS	GC-NPD	GC-NPD	GC-NPD	DETERMINATION
INTERNAL METHOD									LOCAL SOP NO. MR405012
									REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

CYPRODINIL									
31	30	29	28	27	26	25	24	LAB CODE	SCOPE OF YOUR METHOD
D	D	D	D	D	D	D	D	D	D
S	S	M	S	S	M	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
MS	DIFFERENT COLUMN	GC/MS/MS	GC-MS	GC-MS/ITD)	GC-MS	GC-MS	GC-MS	GC-MS	CONFIRMATION METHOD
0.05	0.01	0.05	0.05	0.05	0.14	<0.02	0.05	RL (mg/Kg)	RL (mg/Kg)
80		93	85		63	94	92.1	RECOVERY (%)	RECOVERY (%)
10	10	15	50	30	25	10	30	SAMPLE WEIGHT (g)	SAMPLE WEIGHT (g)
6	1	4	6	1	2	6	1	EXTRACTION SOLVENT	EXTRACTION SOLVENT
O	GPC	SPE	GPC	GPC	SPE	GPC	GPC	CLEAN-UP STEP	CLEAN-UP STEP
DICHLORFENTHION		YES		DITALIMPHOS	YES			INTERNAL STANDARD	INTERNAL STANDARD
1	1	10	1	20	2	2	1	INJECTION VOLUME (µL)	INJECTION VOLUME (µL)
SPLITLESS	SPLITLESS	ON-COLUMN	RHEODYNE INJECTOR	SPLITLESS	SPLITLESS	TPOCI		INJECTION TYPE	INJECTION TYPE
GC-ECD GC-NPD	GC-MS/MS	GC-MS/ITD)	HPLC-UV	GC-ECD, GC-NPD, GC-MS/ITD)	GC-MS	GC-NPD		DETERMINATION	DETERMINATION
ISTISAN 9/7/23	RAPP ISTISAN 9/7/23	MINILUKE	HUNGARIAN CENTRAL SOP NO.: 405012 M1			SOP NO.: MR 405012 M1; 308 M2		REFERENCE METHOD	REFERENCE METHOD

## **APPENDIX 7. Methods used by participants for determining pesticides.**

## **APPENDIX 7. Methods used by participants for determining pesticides.**

CYPRODINIL												
46		45		44		43		42		41	40	LAB CODE
NA	D		D		D		D		D	D	D	SCOPE OF YOUR METHOD
	S		M		M		M		M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-MS		GC-MS		LC-MS/MS		GC-MS		GC-MS		GC-MS		CONFIRMATION METHOD
0.04		0.01		0.01		0.01		0.03		0.1		RL (mg/Kg)
98		94		106		95.1						RECOVERY (%)
25		50		10		10		20		15		SAMPLE WEIGHT (g)
1 (IN PRESENCE OF NaOH)		2		5		5		3		4		EXTRACTION SOLVENT
	GPC	LL		SPE	LL							CLEAN-UP STEP
												INTERNAL STANDARD
				TRIPHENYLPHOSPHATE (TPP)								INTERNAL STANDARD
				10		5		4		20		INJECTION VOLUME (µL)
				RHEODYNE								SPITLESS
	HPLC - DAD	LC-MS/MS		LC/MS-MS ESI (+)								INJECTION TYPE
					LC-MS/MS							DETERMINATION
						GC-ECD, GC-NPD						ANALYTICAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS, 6TH ED., 1996. GENERAL INSPECTORATE FOR HEALTH PROTECTION, MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS
	CLEAN UP AS IN EN 12393 P METHOD	HOUSE METHOD		ANASTASSIADES ET AL, JAAC INT, 86 82(2003), 412-431, MODIFIED		M. ANASTASSIADES ET AL, JAAC INT, 86 82(2003), 412-431, MODIFIED						REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

CYPRODINIL									
									LAB CODE
54	53	52	51	50	49	48	47		
D	D	D	NA	D	D	D	D		
M	S	M		S	M	S			
GC-MS	GC-MS	GC-MS		GC-MS	GC-MS(ITD)	GC-MS	GC-NPD	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
0.05	0.06	0.05		0.05	0.01	0.01	0.05	CONFIRMATION METHOD	RL (mg/Kg)
81				82				RECOVERY (%)	
18	15	10		20	50	25	10	SAMPLE WEIGHT (g)	
1	6	5		6	1	2	5	EXTRACTION SOLVENT	
GPC	GPC	O		LLE	GPC	SPE		CLEAN-UP STEP	
				DICLOBENIL				INTERNAL STANDARD	
1	1	1,5			2	2	3	INJECTION VOLUME (µL)	
SPLITLESS, ON-COLUMN	SPLIT/SPLITLESS	SPLIT/ SPLITLESS	ON COLUMN	SPLITLESS	PTV	SPLIT/SPLITLESS		INJECTION TYPE	
GC-ECD, FPD,NPD	GC-ECD, GC-NPD, HPLC-FID, HPLC-UV	GC-ECD, GC-NPD, GC-MS,	GC-NPD GC-MS(ITD)/MS	GC-FPD, GC-NPD, GC-MS(ITD)/MS	GC-MS	GC-ECD, GC-NPD, GC-MS (SINGLE QUAD)		DETERMINATION	
EN 12393-1,2,3	INTERNAL METHOD	QUECHERS		METHOD R'EN 12393-2:1999	DFG S 19	M. ANASTASSIADES, S. J. LEHOTAY, D. STAIBNBAHER, F. J. SCHENCK : J. AOAC INT. 86 (2003) 412-431		REFERENCE METHOD	

**APPENDIX 7. Methods used by participants for determining pesticides.**

CYPRODINIL									
61	60	59	58	57	56	55	LAB CODE		
D	D	D	D	D	D	D	SCOPE OF YOUR METHOD		
M	M	M	M	M	S	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		
LC-MS/MS	GC-MS	GC/MSD	GC-ECD-NPD	GC-MS	GC-MS	GC-NPD	CONFIRMATION METHOD		
0.02	0.05	0.02	0.1	0.01	0.69	0.015	RL (mg/Kg)		
		108	94		91.3	91.3	RECOVERY (%)		
20	15	10	15	25	30	100	SAMPLE WEIGHT (g)		
METHANOL	1	5	4	1	1	2	EXTRACTION SOLVENT		
							CLEAN-UP STEP		
		GPC	O	GPC	GPC	GPC			
		ISODRIN	3	PCB119 (ECD) TRIFLURALIN (GC-NPD)			INTERNAL STANDARD		
10	1	2	1	1	1	GC 1 µL HPLC 20 µL	INJECTION VOLUME (µL)		
		SPLIT/SPLITLESS	SPLIT/SPLITLESS	PULSED SPLITLESS	DIRECT	SPLIT/SPLITLESS	INJECTION TYPE		
		GC-ECDD/NPD/MS	GC-MS	GC-ECDD/NPD	GC-NPD	GC-ECD, GC-NPD, GC-FPD, HPLC-UV, LC-MS/MS	DETERMINATION		
				ANAL METHODS FOR PESTICIDES RESIDUES IN FOODSTUFFS (NL)	GC-MSD	MODULARE MULTIMETHODE S19	REFERENCE METHOD		
			§35 L 000034						

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>CYPRODINIL</b>									
68	67	66	65	64	63	62	LAB CODE	SCOPE OF YOUR METHOD	
D	D	NA	D	D	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
M	M	M	M	M	M	M	M	CONFIRMATION METHOD	
GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	RL (mg/Kg)	
0.02	0.02	0.02	0.02	0.02	0.05	0.05	0.02	RECOVERY (%)	
97,6	80	86	82	82	74	74	74	SAMPLE WEIGHT (g)	
20	10	5	5	5	75	75	10	EXTRACTION SOLVENT	
4	5	1	6	6	ACETONE FOLLOWED BY CYCLOHEXANE AND ETHYLACETATE		1	CLEAN-UP STEP	
LLE + SPE					GPC	GPC			
TRIPHENYLPHOSPHATE	AZOBENZENE+RONNELL+ TRIPHENYLPHOSPHATE	TPP	TPP	TPP	TPP	TPP	TPP	INTERNAL STANDARD	
2	10	2	0,5	0,5	1	1	2	INJECTION VOLUME (µL)	
SPLITLESS	ON-COLUMN	SPLITLESS						INJECTION TYPE	
GC-ECD/NPD	LC-MS/MS	GC-(ECD+FPD+NPD)	GC-MS	GC-NPD	GC-MS	GC-MS	GC-MS	DETERMINATION	
MINI-LUKE EXTRACTION	ANASTASSIADES, M. ET AL., J. AOAC INT., 86 (2003), 412 - 431 QUECHERS - METHOD	INTERNAL METHOD	MULTIRESIDUE WITH EXTRELUT	§ 35 LMBG L.00.00-34				REFERENCE METHOD	

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>CYPRODINIL</b>									
75	74	73	72	71	70	69	LAB CODE		
D	D	D	D	D	NA	D	SCOPE OF YOUR METHOD		
M	M	M	S	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		
							CONFIRMATION METHOD	CONFIRMATION METHOD	
GC-MS	GC-MS	GC-NP	GC-TSD	GC-MS	GC-MS	GC-MS	RL (mg/Kg)	RL (mg/Kg)	
0.01	0.005	0.05	0.02	0.05		0.05			
92	103	79	98	99		79	RECOVERY (%)	RECOVERY (%)	
10	25	30	100	25		25	SAMPLE WEIGHT (g)	SAMPLE WEIGHT (g)	
6 METHANOL	3	1	3	1		6. METHANOL	EXTRACTION SOLVENT	EXTRACTION SOLVENT	
LLE(CHEM ELUT)	HEXBROMOBENZENE	O					CLEAN-UP STEP	CLEAN-UP STEP	
							INTERNAL STANDARD	INTERNAL STANDARD	
20	1	1	1	5			1.0-2.0ML	INJECTION VOLUME (µL)	
LC-MS/MS	WIDE BORE	SPLITLESS	SPLITLESS	SPLITLESS			GC-NPD	INJECTION TYPE	
JAOAC INTERN, 86.1015FF (2003)	GC-MS	GC-NPD	GC-EC, GC-NP	GC-TSD			GC-NPD	DETERMINATION	
	LOCAL SOP						IN-HOUSE METHOD	REFERENCE METHOD	

**APPENDIX 7. Methods used by participants for determining pesticides.**

CYPRODINIL																	
								LAB CODE									
								SCOPE OF YOUR METHOD									
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX									
								S									
								GC/MS									
								CONFIRMATION METHOD									
								RL (mg/Kg)									
								0.05									
								RECOVERY (%)									
								87									
								SAMPLE WEIGHT (g)									
								10									
								DICHLOROMETHANE									
								EXTRACTION SOLVENT									
CLEAN-UP STEP																	
NO RESULTS																	
								INTERNAL STANDARD									
								INJECTION VOLUME (µL)									
								1									
								SPLITLESS									
								INJECTION TYPE									
								DETERMINATION									
								GC/MS									
NO RESULTS																	
								REFERENCE METHOD									
QUECHERS, M. ANASTASSIADES, S. J. LEHOTAY, D. STAIBNBAUER, F. J. SCHEINK J. AOAC INT., 86 (2003) 412-431																	
KLEIN,J., ALDER,L. J.AOAC INT. 86, 1015-103																	

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>CYPRODINIL</b>									
								LAB CODE	
								SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
								CONFIRMATION METHOD	
90	89	88	87			86	85	84	
D	D	D	D			D	NA	D	
M	M	M	S			M	M		
GC-MS	GC-MS	LC-MS/MS	GC-MS/ITD			GC-MS			
0.04	0.02	0.005	0.05			0.01			
	89	95	88			80			
50	15	5	50			50			
2	4	5	6 ETHYLACETATE/DICHLOROMETHANE (80/20)	4		2			
CLEAN-UP STEP									
GPC									
LLE									
TRICHLORONAT									
1	1	10	1			1			
SPLITLESS	SPLITLESS	ON-COLUMN	SSL			SPLITLESS			
GC-NPD	GC-ECD, FPD, MS HPLC-F-DAD	LC-MS/MS	GC-TSD			GC-MS (SINGLE QUAD)			
ACCORDING TO EN 12396-1: 1999									
IN-HOUSE									
EN NF 12393									
REFERENCE METHOD									

## **APPENDIX 7. Methods used by participants for determining pesticides.**

## **APPENDIX 7. Methods used by participants for determining pesticides.**

CYPRODINIL									
105		104		103		102		101	
D	D	D	D	D	D	D	D	NA	D
M	S	S	M	M	M	M	M	M	M
GC-NPD	GC-MS	GC-MS	HPLC-DAD	HPLC-DAD	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS
0.5	0.005	0.020	0.05	0.05	0.05	0.05	0.05	0.05	RL (mg/Kg)
100	100	80	91	91	90	90	90	90	RECOVERY (%)
4	2	50	30	30	10	10	10	10	SAMPLE WEIGHT (g)
		1	1	1	2	2	2	2	EXTRACTION SOLVENT
ALDRIN		TRIPHENYLPHOSPHAT		SPE		SPE		CLEAN-UP STEP	
GC-NPD, GC-ECD		PTV		DIRECT		SPLITLESS		GPC	
GC-MS		PTV		SPLITLESS/PTV		SPLIT-SPLITLESS/PTV		INTERNAL STANDARD	
GC-NPD, GC-ECD		GC-FPD		GC-FPD		GC-ECD, NPD, MS		PULSED SPLITLESS	
MULTIRESIDUE METHOD: AOAC 985.22/90 MODIFIED		LMBG §35 L-00.00 .34		ANNFA/L, EXP CHIM. SEPT-OCT 1974/67, N/721-722 PP513-524		GC-NPD		GC-MS	
REFERENCE METHOD		SOFT MR 405012; EXTRACTION OF RESIDUES OF PLANT- PROTECTING MATERIAL FROM FRUIT AND VEGETABLE SAMPLES BY ETHYL ACETATE; SOFT 30BW2; CLEAN UP OF EXTRACTS BY GEL PERMEATION CHROMATOGRAPHY							

**APPENDIX 7. Methods used by participants for determining pesticides.**

CYPRODINIL									
112	111	110	109	108	107	106	LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
NA	D	D	D	NA	D	D			S
M	M	M	M	M	M	M			
GC-MS	GC-NPD	GC-MS	GC-MS	GC-MS	GC-MS	GC-NPD	GC-NPD	CONFIRMATION METHOD	
0.05	0.05	0.05	0.02	0.02	0.02	0.01	RL (mg/Kg)		
102.2						79	RECOVERY (%)		
50	50	50	10	10	10	25	SAMPLE WEIGHT (g)		
2	2	6	2	2	2	5	6 ETHYLACETATE/DICHLOROMETHANE	EXTRACTION SOLVENT	
								CLEAN-UP STEP	
								INTERNAL STANDARD	
								INJECTION VOLUME (µL)	
								INJECTION TYPE	
								DETERMINATION	
								REFERENCE METHOD	
§35 LMBG; L 00, 00-34, L 00,00-37	MULTI-RESIDUE-METHOD DFG S 19						QUECHERS ANASTASSIADES S19 -DFG		

## **APPENDIX 7. Methods used by participants for determining pesticides.**

## **APPENDIX 7. Methods used by participants for determining pesticides.**

CYPRODINIL						
128	127	126	125	124	123	LAB CODE
NA	NA	NA	NA	NA	NA	SCOPE OF YOUR METHOD
						QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
						CONFIRMATION METHOD
						RL (mg/Kg)
						RECOVERY (%)
						SAMPLE WEIGHT (g)
						EXTRACTION SOLVENT
						CLEAN-UP STEP
						INTERNAL STANDARD
						INJECTION VOLUME ( $\mu$ L)
						INJECTION TYPE
						DETERMINATION
						REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

DIAZINON											
									LAB CODE		
									SCOPE OF YOUR METHOD		
M	M	M	M	M	M	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		
GC-MS	GC-FPD/NPD	GC-MS/MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	CONFIRMATION METHOD		
0.02	0.013	0.02	0.010	0.010	0.02	0.02	0.02	0.02	RL (mg/Kg)		
10	10	83.9	95	95	95	95	95	95	RECOVERY (%)		
5	5	1	15	15	15	15	15	15	SAMPLE WEIGHT (g)		
			4	4	4	4	4	4	EXTRACTION SOLVENT		
									CLEAN-UP STEP		
	SPE	SPE	NO	NO	SPE	SPE	SPE	SPE			
	MIREX/TPP	TRIPHENYL PHOSPHATE ANTHRACENE							INTERNAL STANDARD		
2	2	10	1	1	2	2	2	2	INJECTION VOLUME ( $\mu$ L)		
	SPLIT/ SPLITLESS	SPLIT/ SPLITLESS	SPLIT	SPLITLESS	SPLITLESS	SPLIT/ SPLITLESS	SPLIT/ SPLITLESS	SPLIT/ SPLITLESS	INJECTION TYPE		
	GC-ECD/ GC-NPD	GC-MS	GC-MS/MS	GC-NPD	GC-NPD	GC-MS	GC-MS	GC-MS	DETERMINATION		
MANASTASSIADES ET AL. JAOOC 86, 412-431 (2003)	QUECHERS; ANASTASSIADES					LUKE M.A., FROBERG, J.E., DOOSE, G.M., MASUMOTO, H.T. (1981); IMPROVED MULTIRESIDUE GAS CHROMATOGRAPHIC DETERMINATION OF ORGANOPHOSPHORUS, ORGANONITROGEN, AND ORGANOHALOGEN PESTICIDES IN PRODUCE, USING FLAME PHOTOMETRIC AND ELECTROLYTIC CONDUCTIVITY DETECTORS. J. ASSOC. OFF. ANAL. CHEM. 64(5): 1187-1195					REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

DIAZINON											
12	11	10	9	8	7	6	LAB CODE				SCOPE OF YOUR METHOD
D	D	D	D	D	D	D	M	S	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
M	S	M	M	M	M	M	GC-MS	GC-ECD	GC-MS	GC-MS	CONFIRMATION METHOD
MS(TD), GC-FPD	GC-MS	GC-MS(TD)	GC-MS	GC-MS	GC-MS	GC-MS	RL (mg/Kg)	RL (mg/Kg)	RL (mg/Kg)	RL (mg/Kg)	RECOVERY (%)
0.01	0.02	0.01	0.025	0.01	0.01	0.01	94	84	95	89	94.1
94	100.5	100.5	95	95	95	95	50	30	25	15	10
50	30	30	15	15	15	15	1	1	1	1	15
1	1	2	4	4	4	4	6 (DICHLOROMETHANE)	6 (DICHLOROMETHANE)	6 (DICHLOROMETHANE)	6 (DICHLOROMETHANE)	4
EXTRACTION SOLVENT											
CLEAN-UP STEP											
INTERNAL STANDARD											
GPC											
LLE ACETONE FOLLOWED BY DICHLOROMETHANE AND PETROLEUM ETHER											
O											
INJECTION VOLUME ( $\mu$ L)											
1, 2, 5											
1											
SPLITLESS											
ON COLUMN											
SSU/PTV											
PTV											
SPLITLESS											
GC-ECD, GC-NPD, GC-MS(TD)											
MS-(TD), LC-MS, GC-FPD, GC-ECD											
ANALYTICAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS (1986), MINISTRY OF HEALTH, WELFARE AND SPORTS, THE HAGUE (RUISWIJK), THE NETHERLANDS											
RAPPORT ISTIAN 97/23											
ANALYTICAL METHODS FOR PESTICIDE RESIDUES MINISTRY OF HEALTH, WELFARE AND SPORTS, THE HAGUE (RUISWIJK), THE NETHERLANDS											
REFERENCE METHOD											
ANALYTICAL METHODS FOR PESTICIDE RESIDUES MINISTRY OF HEALTH, WELFARE AND SPORTS, THE HAGUE (RUISWIJK), THE NETHERLANDS											

**APPENDIX 7. Methods used by participants for determining pesticides.**

DIAZINON									
20	19	18	17	16	15	14	13	LAB CODE	SCOPE OF YOUR METHOD
S	M	S	M	M	S	S	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	CONFIRMATION METHOD
GC-ECD	GC-MS(ITD)	GC-MS	GC/MS	GC-MS/MS	GC-MS	GC-MS	GC-MS/MS	GC/MS/MS	GC/MS/MS
0.02	0.02	0.02	0.01	0.05	0.02	0.02	0.02	0.02	RL (mg/Kg)
98	80	108			97				RECOVERY (%)
100	75	75	15	15	25	15	15	15	SAMPLE WEIGHT (g)
3	1	1	4	4	1	4	1	1	EXTRACTION SOLVENT
CLEAN-UP STEP									
O	GPC	GPC	LLE						
INTERNAL STANDARD									
DITALIMPHOS		ALDRIN					TPP (QC)	INTERNAL STANDARD	
2	2	2	10	10	2	2	10	INJECTION VOLUME (µL)	
MANUAL	SPLITLESS	PULSED SPLITLESS	LVI AUTOSAMPLER	LVI SPLITLESS	SPLIT/SPLITLESS			INJECTION TYPE	
GC-NPD	GC-TSD/PFPD	GC-MS	GC-MS	GC-MS/MS	GC-NPD		GC-ECD GC-TSD GC-MS HPLC-UV	DETERMINATION	
REFERENCE METHOD									
LOCAL SOP NO. MR405012									
MULTIRESIDUES									

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>DIAZINON</b>									
<b>28</b>	<b>27</b>	<b>26</b>	<b>25</b>	<b>24</b>	<b>23</b>	<b>22</b>	<b>21</b>	<b>LAB CODE</b>	<b>SCOPE OF YOUR METHOD</b>
D	D	D	D	D	D	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
S	S	M	M	M	M	M	M	M	CONFIRMATION METHOD
GC-MS	GC-MS(ITT)	GC-MS	GC-NPD	GC-MS	GC-MS	GC-MS	GC-MS/MS	GC-MS/MS	RL (mg/Kg)
0.02	0.02	0.02	<0.02	0.02	0.01	0.01	0.01	0.01	RECOVERY (%)
73	74	90	101,3				95,2		
50	30	25	10	30	5	5	50	10	SAMPLE WEIGHT (g)
6	1	2	6	1	5	5	6	1	EXTRACTION SOLVENT
SPE	GPC	GPC	SPE	GPC	LLE (ISOPROPYL ETHER)				CLEAN-UP STEP
YES	DITALIMPHOS	YES		FENCHLORPHOS					INTERNAL STANDARD
1	1	2	2	1 mg SAMPLE	50	2	10		INJECTION VOLUME (µL)
ON-COLUMN	SPI	SPLITLESS	SPLITLESS	TPOCI	AUTOMATIC TDS SAMPLER	SPLITLESS	LV		INJECTION TYPE
GC-MS(ITT)	GC-ECD	GC-ECD, GC-NPD, GC-MS(ITT)	GC-MS	GC-ECD	TDS-IN LINE WITH GC-MS (SINGLE-QUAD) TOTAL ION DETECTION	GC-ECD AND GC-NPD	GC-MS/MS		DETERMINATION
	HUNGARIAN CENTRAL SOP NO.: 405012 M1			SOP NO.: MR 405012 M1; 308 M2	INTERNAL METHOD				REFERENCE METHOD

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>DIAZINON</b>							
40	39	38	37	36	LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
D	D	D	D	D	D	GC-MS	GC-FPD, GC-NPD, GC-MS
M	S	M	M	S	S	GC/MS	GC-ECD, GC-NPD, GC-MS
0.02	0.01	0.02	0.02	0.02	RL (mg/Kg)	GC-MS	GC-FPD
15	100	10	10	10	RECOVERY (%)	GC/MS	GC-MS
4	2	5	5	5	SAMPLE WEIGHT (g)	GC-FPD	GC-FPD
					EXTRACTION SOLVENT	GC-FPD	GC-FPD
					CLEAN-UP STEP	GC-FPD	GC-FPD
					INTERNAL STANDARD	GC-FPD	GC-FPD
1	1	2	2	1	INJECTION VOLUME (µL)	GC-FPD	GC-FPD
SPITLESS	SPLIT	SPLITLESS	SPLITLESS	SPLIT/SPLITLESS	INJECTION TYPE	GC-FPD	GC-FPD
GC-NPD, GC-ECD, GC-MS(1D)	GC-ECD	GC-ECD GC-NPD GC-MS	GC-ECD GC-NPD GC-MS	GC-FPD	DETERMINATION	GC-FPD GC-ECD GC-MS (SINGLE-QUAD) LC-MS HPLC-MS HPLC-PICKERING	ANASTASSIADES, JOURNAL OF AOAC INT. VOLT 86 NO 2, 2003
ANALYTICAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS, 6TH ED., 1996. GENERAL INSPECTORATE FOR HEALTH PROTECTION, MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS	DFG S 19, L 00-00-54 (§ 35 LMBO)				REFERENCE METHOD		

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>DIAZINON</b>						
45	44	43	42	41	LAB CODE	
D	D	D	D	D	SCOPE OF YOUR METHOD	
M	M	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
GC-MS	LC-MS/MS	GC-MS	GC-MS	GC-MS	CONFIRMATION METHOD	
0.02	0.01	0.01	0.01	0.01	RL (mg/Kg)	
95	97	97	96.5	96.5	RECOVERY (%)	
25	50	10	10	20	SAMPLE WEIGHT (g)	
1	2	5	5	3	EXTRACTION SOLVENT	
GPC	GPC	SPE	LLE	LLE	CLEAN-UP STEP	
					INTERNAL STANDARD	
		PCB 138, TRIPHENYLPHOSPHATE			INJECTION VOLUME (µL)	
1	2	4	1	4	INJECTION TYPE	
SPLIT/SPLITLESS (SPLITLESS MODE)	PTV	PTV	PTV SOLVENT VENT	SPLITLESS		
GC-ECD OR GC-NPD OR GC-FPD	GC-MS (SINGLE QUAD)	GC-MS EI	GC-ECD, GC-MS	GC-ECD, GC-NPD	DETERMINATION	
EN 12393 P METHOD	§ 35 LIMBG 00.00-34	ANASTASSIAIDES ET AL- JAOAC INT. 86 82(2003) 412-431, MODIFIED	M. ANASTASSIAIDES ET AL., J. AOAC INT. 86 (2), 412-431, (2003)		REFERENCE METHOD	

**APPENDIX 7. Methods used by participants for determining pesticides.**

DIAZINON									
51	50	49	48	47	46	LAB CODE	SCOPE OF YOUR METHOD		
NA	D	D	D	D	D		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		
	S	S	M	S	M		CONFIRMATION METHOD		
	GC-MS	GC-FPD	GC-MS	GC-ECD	GC-NPD		RL (mg/Kg)		
	0.02	0.01	0.01	0.02	0.02		RECOVERY (%)		
96					87		SAMPLE WEIGHT (g)		
20	50	50	25	10	15		EXTRACTION SOLVENT		
6	1	1	2	5	4		CLEAN-UP STEP		
							INTERNAL STANDARD		
	LLE	GPC	SPE				INJECTION VOLUME (µL)		
	COUMAFOS ETHION						(PULSED) SPLITLESS/ON-COLUMN		
	2	2	3	4	3µl / 1µl		INJECTION TYPE		
ON COLUMN	SPLITLESS	PTV	SPLIT/SPLITLESS				DETERMINATION		
	GC-FPD, GC-NPD, GC-MS(ITD)/MS	GC-MS	GC-ECD, GC-NPD, GC-MS (SINGLE QUAD)				MULTI-RESIDUE METHOD/PESTICIDES AMENABLE TO GAS CHROMATOGRAPHY (3:1:2 ACETONE:PARTITION FLUID: VEGETABLES AND POTATOES) 6TH EDITION MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORT, THE NETHERLANDS		
	METHOD R'EN 12393-2:1999	DFG S 19	M. ANASTASSIADES, S. J. LEHOTAY, D. STAINBAHER, F. J. SCHENCK; J. AOAC INT. 86 (2003) 412-431				REFERENCE METHOD		

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>DIAZINON</b>							
							LAB CODE
65	64	63	62	61	60	59	
D	D	D	D	D	D	D	SCOPE OF YOUR METHOD
M	M	M	M	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	CONFIRMATION METHOD
0.01	0.02	0.02	0.02	0.02	0.02	0.01	RL (mg/Kg)
94	82	97				102	RECOVERY (%)
5	5	75	10	50	15	10	SAMPLE WEIGHT (g)
1	6	ACETONE FOLLOWED BY CYCLOHEXANE AND ETHYLACETATE	1	3	1	5	EXTRACTION SOLVENT
							CLEAN-UP STEP
AZOBENZENE+RONNEL+ TPP	TPP	TPP					INTERNAL STANDARD
2	0,5	1	2	2	1	2	INJECTION VOLUME ( $\mu$ L)
ON-COLUMN	SPLITLESS		PTV	SPLIT/ SPLITLESS	SPLITLESS		INJECTION TYPE
GC-(ECD+FID+NPD)	GC-MS	GC-NPD					DETERMINATION
INTERNAL METHOD	MULTIRESIDUE WITH EXTRELUT						REFERENCE METHOD
							\$35 L 000034
							DFG S19 (ASU \$35 LMBG L 00.00-34)

**APPENDIX 7. Methods used by participants for determining pesticides.**

DIAZINON									
									LAB CODE
									SCOPE OF YOUR METHOD
71	70	69	68	67	66				
D	D	D	D	D	D				
M	M	M	M	M	M				
GC-TSD	GC-MS/MS	GC-MS	GC-MS	GC-MS/MS	GC-MS/MS				CONFIRMATION METHOD
0.01	0.02	0.02	0.02	0.02	0.02				RL (mg/Kg)
106		90	92.4	80	100				RECOVERY (%)
25	15	25	20	10	15				SAMPLE WEIGHT (g)
6	5	1	4	5	5				EXTRACTION SOLVENT
O	O	GPC	LLE + SPE	SPE	CLEAN-UP STEP				
2	10	1.0-2.0	2	10	10				INTERNAL STANDARD
SPLITLESS	LVI	SPLITLESS	SPLITLESS						INJECTION VOLUME (µL)
GC-ECD	GC-ECD/TSD/PFPD GC-MS/MS	GC-NPD, GC-ECD	GC-ECD/NPD	LC-MS/MS	GC-MS(HTD)				DETERMINATION
MULTIRESIDUE METHOD FOR ANALYSIS OF PESTICIDES IN FOODSTUFFS P.A. GREVE ED, 1988 MINISTRY OF WELFARE, HEALTH AND CULTURAL AFFAIR, RUSWICK THE NETHERLANDS	QUECHERS	MULTIRESIDUE METHOD 1, ANALYTICAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS; SIXTH EDITION	MINI LUKE EXTRACTION	ANASTASSIADES, M. ET AL., J. AOAC INT., 86 (2003), 412 – 431 QUECHERS - METHOD	QUECHERS (LEOTHAY, S. ET AL., JAOAC, INT. (2005) 88, 615-629				REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

DIAZINON									
								LAB CODE	
								SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
								CONFIRMATION METHOD	
80	79	78	77	76	75	74	73	72	D
D	D	D	D	D	D	D	D	D	D
M	M	M	S	S	M	M	S	S	S
GC-MS	GC-MS	GC-MS	LC/MS/MS	GC-MS	GC-MS	GC-NPD	GC-NPD	GC-NPD	GC-NPD
0.02	0.02	0.01	0.01	0.005	0.01	0.02	0.02	0.02	0.02
97	80	94	91	72.4	89	108	108	108	108
50	15	15	10	50	25	30	100	100	100
2	6	1	DICHLOROMETHANE	2	3	1	3	3	EXTRACTION SOLVENT
CLEAN-UP STEP									
HEXABROMOBENZENE									
GPC									
NO RESULTS									
INTERNAL STANDARD									
TPP									
INJECTION VOLUME (µL)									
1									
INJECTION TYPE									
WIDE BORE									
SPLITLESS									
SPLIT/ SPLITLESS									
GC-NPD									
GC-MS									
GC-ECD; GC-NPD; GC-MS; LC-MS/MS; HPLC/FID									
GC-ECO; GC-NPD; GC-MS; (SINGLE-QUAD)									
§35 LMBGL 00.00-34 (1999)									
LOCAL SOP									
REFERENCE METHOD									
INTERNAL METHOD									
L-00.00-34									

**APPENDIX 7. Methods used by participants for determining pesticides.**

DIAZINON									
87	86	85	84	83	82	81	LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
D	D	D	D	D	D	D			
S	M	M	M	M	M	M			
LC-MS/MS	GC-MS(ITD)	GC-MS	GC-MS	GC-MS	GC-ECD				
0.01	0.025	0.02	0.01	0.02	0.02	0.02	RL (mg/Kg)		
	97		80	108		96	RECOVERY (%)		
50	15	25	50	30	30	10	SAMPLE WEIGHT (g)		
6 ETHYLACETATE/ DICHLOROMETHANE (80/20)	4	4	2	1	1	5	EXTRACTION SOLVENT		
							CLEAN-UP STEP		
							0 (PSA)		
							SPE GPC		
								INTERNAL STANDARD	
								INJECTION VOLUME (µL)	
								INJECTION TYPE	
								DETERMINATION	
								REFERENCE METHOD	
TRICHLORONAT	HCB			TRIPHENYL ETHERENE TRIPHENYL ETHYLENE					
1	1	1	1	3	3	1			
ON-COLUMN	SSL	SPLITLESS	SPLITLESS	SPLITLESS AND ON COLUMN					
GC-FPD	GC-MS(ITD)	GC-NPD	(SINGLE-QUAD)	GC-MS GC-MS	GC-ECD, GC-NPD, GC-FPD, GC- MS, HPLC-FL, HPLC-DAD				
ACCORDING TO EN 12396-1: 1999		SAR 1-04	EN NF 12393		QUECHERS, M. ANASTASSIADES, S. LEHOTAY, D. STAUBHANNER, F. J. SCHENK: J. AOAC INT., 86 (2003), 412-431				

**APPENDIX 7. Methods used by participants for determining pesticides.**

DIAZINON									
95	94	93	92	91	90	89	88	LAB CODE	
D	D	D	D	D	D	D	D	SCOPE OF YOUR METHOD	SCOPE OF YOUR METHOD
S	S	M	M	S	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-MS	GC-ECD	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	CONFIRMATION METHOD	CONFIRMATION METHOD
0.05	0.02	0.005	0.01	0.01	0.01	0.02	0.02	RL (mg/Kg)	RL (mg/Kg)
92	96.8		100			92	90	RECOVERY (%)	RECOVERY (%)
25	10	80	50	50	50	15	25	SAMPLE WEIGHT (g)	SAMPLE WEIGHT (g)
5	4	2	4	3	2	4	1	EXTRACTION SOLVENT	EXTRACTION SOLVENT
SPE	SPE	GPC	LLE	LLE	GPC	GPC	GPC	CLEAN-UP STEP	CLEAN-UP STEP
YES			ALDRIN, DITALIMPHOS	YES				INTERNAL STANDARD	INTERNAL STANDARD
3	2	1µL PER COLUMN	1	1	1	1	2	INJECTION VOLUME (µL)	INJECTION VOLUME (µL)
PTV	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	SPLIT/SPLITLESS	SPLIT/SPLITLESS	SPLITLESS	INJECTION TYPE	INJECTION TYPE
GC-ECD, GC-NPD, GC-MS	GC-ECD AND GC-NPD OR GC-ECD OR GC-NPD	GC-ECD, GC-NPD, GC-MS	GC-NPD	GC-NPD	GC-ECD, FPD, MS HPLC-F-DAD	GC-NPD	GC-NPD	DETERMINATION	DETERMINATION
FILLION ET AL. JOURNAL OG AOAC INTERNATIONAL 78-5-1995	GC-MS (SINGLE-QUAD)	OFFICIAL METHOD OF ANALYSIS (1990) 15TH ED., 985.22, AOAC ARLINGTON VA	DFG-S8					IN-HOUSE	REFERENCE METHOD

## **APPENDIX 7. Methods used by participants for determining pesticides.**

## **APPENDIX 7. Methods used by participants for determining pesticides.**

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

DIAZINON									
121	120	119	118	117	116	115	114	LAB CODE	SCOPE OF YOUR METHOD
D	D	D	D	D	D	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
S	S	S	S	M	S	S	S	S	CONFIRMATION METHOD
GC-ECD	GC-MS	GC-MS	GC-NPD	GC-MS	GC-MS	GC-MS GC-PFPD	GC-MS	GC-MS	RL (mg/Kg)
<0.020	0.002	0.02	0.02	0.03	0.06	0.01	0.01	0.01	RECOVERY (%)
60		79.1	108				97.1		
100	100	10	5	40	15	50	50	50	SAMPLE WEIGHT (g)
3, 1, CYCLOHEXANE, N-HEXANE, IZO-OCTANE, TOLUENE	3	3	3	1	6	3	3	6	EXTRACTION SOLVENT
GPC, SPE	GPC	O							CLEAN-UP STEP
									INTERNAL STANDARD
						YES			
2	1	1	2	1	1	1	1	1	INJECTION VOLUME (µL)
SPLITLESS	SPLIT	AUTO	SPLITLESS	SPLITLESS	SPLITLESS	ON COLUMN	SPLITLESS		INJECTION TYPE
GC-ECD	GC-ECD GC-NPD	GC-ECD GC-MS	GC-NPD	GC-MS	GC-ECD GC-NPD GC-MS(TD)	GC-MS	GC-MS	GC-MS	DETERMINATION
PN-EN 12393-1,2,3:2000	PN-EN 12393-1,2,3	ISS B6			RAPPORTO ISTISAN 9/7/23				REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

DIAZINON									
128	127	126	125	124	123	122	LAB CODE	SCOPE OF YOUR METHOD	
D	NO RESULTS	D	D	D	D	D	D	D	
M		S	S	S	S	S	S	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-MS	GC-ECD	GC-NPD	GC-NPD	GC-NPD	GC-NPD	GC-NPD	GC-NPD	GC-NPD	CONFIRMATION METHOD
0.02	0.001	0.0012	0.0025	0.001	0.001	0.06	0.06	RL (mg/Kg)	RL (mg/Kg)
20 / 5	100	62.63	59.3	123	87			RECOVERY (%)	RECOVERY (%)
1	3	25	20	50	50			SAMPLE WEIGHT (g)	SAMPLE WEIGHT (g)
		3	6	1	3			EXTRACTION SOLVENT	EXTRACTION SOLVENT
		LLE	GPC	LLE	GPC	O	O	CLEAN-UP STEP	CLEAN-UP STEP
								INTERNAL STANDARD	INTERNAL STANDARD
2		1	2	10	1	1	1	INJECTION VOLUME (µL)	INJECTION VOLUME (µL)
SPLITLESS	AUTOSAMPLER	SPLITLESS	PTV SOLVENT VENT	PTV PULSED SPLITLESS	PTV PULSED SPLITLESS			INJECTION TYPE	INJECTION TYPE
GC-ECD, GC-FPD, GC-MSD, GC-MS(TD)	GC-ECD	GC-ECD	GC-ECD	GC-NPD	GC-NPD			DETERMINATION	DETERMINATION
MODIFIED LUKE	PN-EN 12393: 2000	PN-EN 12393	GCMS PN-EN 12393:2000 (A)	GC-MS	GC-MS			REFERENCE METHOD	REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>DIMETHOATE</b>							
<b>6</b>	<b>5</b>	<b>4</b>	<b>3</b>	<b>2</b>	<b>1</b>	<b>LAB CODE</b>	<b>SCOPE OF YOUR METHOD</b>
D	D	D	D	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
M	M	M	M	S	S	M	CONFIRMATION METHOD
GC-PFPD	LC-MS/MS	GC-MS	GC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	RECOVERY (%)
0.02	0.005	0.001	0.02	0.010	0.02	RL (mg/Kg)	SAMPLE WEIGHT (g)
15	10	10	15	10	10	10	EXTRACTION SOLVENT
4	5	5	1	6	6	1	CLEAN-UP STEP
GPC	GPC	SPE	SPE	NO	SPE	SPE	INTERNAL STANDARD
				TRIPHENYL PHOSPHATE ANTHRACENTE		YES	INJECTION VOLUME (µL)
1	20	10	10	10	5	20	INJECTION TYPE
SPLITLESS				SPLIT		N/A	DETERMINATION
GC-PFPD	LC-MS/MS	LC-MS/MS	GC-MS/MS	GC-MS/MS	LC-MS/MS	LC-MS/MS	REFERENCE METHOD
ANALYTICAL METHODS FOR PESTICIDE RESIDUES MINISTRY OF HEALTH NETHERLANDS 6TH ED	M. ANASTASSIADES ET AL. J. AOAC 86, 412-431 (2003)	QUECHERS; ANASTASSIADES	KLEIN, J., ALDER, L. (2003); APPLICABILITY OF GRADIENT LIQUID CHROMATOGRAPHY WITH TANDEM MASS SPECTROMETRY TO THE SIMULTANEOUS SCREENING FOR ABOUT 100 PESTICIDES IN CROPS. J. AOAC INT. 86 (5): 1016-1037				IN HOUSE

**APPENDIX 7. Methods used by participants for determining pesticides.**

DIMETHOATE									
								LAB CODE	
								SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
								CONFIRMATION METHOD	
14	13	12	11	10	9	8	7	GC-NPD(80)	LAB CODE
D	D	D	D	D	D	D	D	RL (mg/Kg)	SCOPE OF YOUR METHOD
S	M	M	S	M	S	M	S	RECOVERY (%)	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-MS	GC-MS/MS	MS-ITD, GC-FPD	GC-MS	GC-MS/ITD	GC-MS	GC-MS	GC-MS	GC-ECD; GC-NPD(80)	CONFIRMATION METHOD
0.02	0.02	0.01	0.02	0.008	0.01	0.01	0.01	0.01	RL (mg/Kg)
90.2	99	115	75	101	101	101	101	101	RECOVERY (%)
15	15	50	30	25	7.5	30	10	10	SAMPLE WEIGHT (g)
4	1	1	1	2	4 (+Na <sub>2</sub> SO <sub>4</sub> )	1	6 (DICHLOROMETHANE)	6 (DICHLOROMETHANE)	EXTRACTION SOLVENT
					LLE ACETONE FOLLOWED BY DICHLOROMETHANE AND PETROLEUM ETHER	O	O	O	CLEAN-UP STEP
					GPC	GPC	GPC	GPC	INTERNAL STANDARD
									INJECTION VOLUME (µL)
2	10	1, 2, 5	1	2	5	2	4	4	INJECTION VOLUME (µL)
					SPLITLESS	SSL/PTV	SPLITLESS	SPLITLESS	INJECTION TYPE
					ON COLUMN	SSL/PTV	LOOP	LOOP	INJECTION TYPE
GC-ECD GC-TSD GC-MS HPLC-UV	GC-MS/ITD GC-FPD GC-ECD	MS-ITD, LC-MS, GC-FPD, GC-ECD	GC-FPD	GC-ECD, GC-NPD, GC-MS/ITD	GC-MS/MS	GC-MS	GC-ECD; GC-NPD(80)	GC-ECD; GC-NPD(80)	DETERMINATION
MULTIRESIDUES					IN-HOUSE SOP		RAPPORT IIST SAN 97/23	RAPPORT IIST SAN 97/23	REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

DIMETHOATE									
22	21	20	19	18	17	16	15	LAB CODE	SCOPE OF YOUR METHOD
D	D	D	D	ND	D	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
M	M	S	M	S	M	M	S	S	CONFIRMATION METHOD
GC-MS	GC-MS/MS	GC-ECD	GC-MS(I/TD)	GC-MS	GC/MS	GC-MS/MS	GC-MS	GC-NPD	RL (mg/Kg)
0.009	0.01	0.02	0.02	0.02	0.03	0.05	0.05	0.02	RECOVERY (%)
102	95	83						115	SAMPLE WEIGHT (g)
20	10	100	75	75	15	15	25		EXTRACTION SOLVENT
1	1	3	1	1	4	4	1		CLEAN-UP STEP
SPE		O	GPC	GPC	LL				INTERNAL STANDARD
			DITALIMPHOS	ALDRIN					INJECTION VOLUME (µL)
1.5	10	2	2	2	10	10	2		INJECTION TYPE
SPLITLESS	LVI	MANUAL	SPLITLESS	PULSED SPLITLESS	LVI AUTOSAMPLER	LVI SPLITLESS	SPLIT/ SPLITLESS		DETERMINATION
GC-NPD	GC-MS/MS	GC-NPD	GC-TSD/PFPD	GC-MS	GC-MS	GC-MS/MS	GC-NPD	LOCAL SOP NO. MR405012	REFERENCE METHOD
								MINI-LUKE	

## **APPENDIX 7. Methods used by participants for determining pesticides.**

DIMETHOATE									
30	29	28	27	26	25	24	23	LAB CODE	
D	D	D	D	D	D	D	D	SCOPE OF YOUR METHOD	
S	M	S	S	M	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
DIFFERENT COLUMN	GC-MS/MS	GC-MS	GC-NPD	GC-NPD	GC-MS	GC-ECD	GC-MS	CONFIRMATION METHOD	
0.01	0.02	0.05	0.02	0.01	<0.05	0.02	0.02	RL (mg/Kg)	
95	111	91	84	93	109.3			RECOVERY (%)	
10	15	50	30	25	10	30	5	SAMPLE WEIGHT (g)	
1	4	6	1	2	6	1	5	EXTRACTION SOLVENT	
								CLEAN-UP STEP	
GPC		SP <sup>E</sup>	GPC	GPC	SPE	GPC			
DICHLORFENTHION								INTERNAL STANDARD	
	YES		DITALIMPHOS	YES		FENCHLORPHOS			
1	10	1	1	2	2	1	50	INJECTION VOLUME (µL)	
SPLITLESS		ON COLUMN	SPI	SPLITLESS	SPLITLESS	TPOCI		INJECTION TYPE	
GC-ECD GC-NPD	GC-MS/MS	GC-MS(ITT)	GC-ECD	GC-ECD, GC-NPD, GC-MS(ITT)	GC-MS	GC-NPD	TDS-IN LINE WITH GC-MS (SINGLE-QUAD) TOTAL ION DETECTION	DETERMINATION	
RAPP ISTISAN 97/23	MINI LUKE		HUNGARIAN CENTRAL SOP NO.: 405012 M1				SOP NO.: MR 405012 M1, 308 M2	INTERNAL METHOD	REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

DIMETHOATE									
38	37	36	35	34	33	32	31	LAB CODE	SCOPE OF YOUR METHOD
D	D	D	D	D	D	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
M	M	S	M	M	M	S	S	S	CONFIRMATION METHOD
GC-MS	LC-MS/MS	GC-MS	GC-MS	GC-MS/MS	GC-MS/MS	GC-MS	MS	MS	RL (mg/Kg)
0.02	0.005	0.02	0.02	0.02	0.01	0.05	0.02	0.02	RECOVERY (%)
		85	90			101	94		
10	10	50	20	5	15	25	10		SAMPLE WEIGHT (g)
5	5	1	3	4	1	2	6		EXTRACTION SOLVENT
O	O								CLEAN-UP STEP
DIMETHOATE D6									
2	20	1	2	10	10	1	1		INTERNAL STANDARD
SPLITLESS		SPLIT/SPLITLESS	SPLITLESS	AUTO	LVI	SPLITLESS	SPLITLESS		INJECTION VOLUME (µL)
GC-ECD GC-NPD GC-MS	LC-MS/MS (TRIPLE-QUAD)	GC-NPD GC-FPD GC-MS (SINGLE-QUAD.)	GC-ECD, NPD	GC-MS/MS	GC-MS/MS (ION TRAP)	GC-MSD	GC-ECD GC-NPD	GC-ECD GC-NPD	DETERMINATION
	ANASTASSIADES, JOURNAL OF AOAC INT. VOLT 86 NO 2, 2003	EN 12393-1, 2, 3		PESTICIDE S MS/MS EI-Cl	\$35 LMBG 00-00-34	ISTISAN 97/23			REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

DIMETHOATE									
									LAB CODE
									SCOPE OF YOUR METHOD
44	43	42	41	40					39
D	D	D	D	D					D
M	M	M	M	M					M
	NONE	LC-MS/MS	GC-MS	GC-MS					QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
0.01	0.01	0.01	0.02	0.02					CONFIRMATION METHOD
93	110	90.8							RL (mg/Kg)
50	10	20							RECOVERY (%)
2	5	3	4	6 (METHANOL/WATER)					SAMPLE WEIGHT (g)
									EXTRACTION SOLVENT
									CLEAN-UP STEP
LLE	SPE	LLE							SPE
									INTERNAL STANDARD
	TRIPHENYLPHOSPHATE (TPP)								INJECTION VOLUME (µL)
5	4	20	4	1					INJECTION TYPE
									DETERMINATION
LC-MS/MS	LC-MS/MS (ESI +)	LC-MS/MS	GC-ECD, GC-NPD	GC-NPD, GC-ECD, GC-MS(ITD)					REFERENCE METHOD
									ANALYTICAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS, 6TH ED., 1996. GENERAL INSPECTORATE FOR HEALTH PROTECTION, MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS
HOUSE METHOD	ANASTASSIADES ET AL. JAOAC INT. 86 820(2003) 412-431, MODIFIED								LC-MS/MS (BFR)

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>DIMETHOATE</b>							
49	48	47	46	45	LAB CODE	SCOPE OF YOUR METHOD	
D	D	D	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
S	M	S	M	M	M	CONFIRMATION METHOD	
GC-FPD	LC-MS/MS	GC-ECD	GC-NPD	GC-MS	GC-FPD OR GC-MS	RL (mg/Kg)	
0.02	0.01	0.02	0.02	0.02	0.02	RECOVERY (%)	
50	10	10	15	25	SAMPLE WEIGHT (g)	EXTRACTION SOLVENT	
1	6	5	4	1	CLEAN-UP STEP	INTERNAL STANDARD	
LLE	SPE	SPE	GPC	1	INJECTION VOLUME ( $\mu$ L)	INJECTION TYPE	
SPLITLESS	SPLIT/SPLITLESS	(PULSED) SPLITLESS/(TRACK OVEN) ON-COLUMN	SPLIT/SPLITLESS (SPLITLESS MODE)	GC-ECD OR GC-NPD OR GC-FPD	DETERMINATION	REFERENCE METHOD	
GC-FPD, GC-NPD, GC-MS(TD)	LC-MS/MS	GC-NPD / GC-ECD	MULTI-RESIDUE METHOD/PESTICIDES AMENABLE TO GAS CHROMATOGRAPHY (3,1,2 ACETONE-PARITION FOR FRUITS, VEGETABLES AND POTATOES) 6TH EDITION, MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORT. THE NETHERLANDS	EN 12393 P METHOD		METHOD R' EN 12393-2:1999	
KLEIN, J.; ALDER, L. JOURNAL OF AOAC INT. 2003, 86, 1015-1037	M. ANASTASSIADES, S. J. LEHOTAY, D. STANBAHER, F. J. SCHENCK ; J. AOAC INT. 86 (2003) 412-431						

**APPENDIX 7. Methods used by participants for determining pesticides.**

DIMETHOATE											
57	56	55	54	53	52	51	50	LAB CODE			
D	D	D	D	D	D	NA	D	SCOPE OF YOUR METHOD			
M	S	M	M	S	M	S	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX			
GC-MS	GC-MS	GC-NPD	GC-MS/MS	GC-MS	GC-ECD	LC-MS	CONFIRMATION METHOD				
0.01	0.112	0.005	0.02	0.02	0.02	0.02	RL (mg/Kg)				
	76.9	96.2	75			96	RECOVERY (%)				
25	30	100	18	15	10	10	SAMPLE WEIGHT (g)				
1	1	2	1	6	5	4	EXTRACTION SOLVENT				
							CLEAN-UP STEP				
							O				
							INTERNAL STANDARD				
								INJECTION VOLUME (µL)			
								5			
							INJECTION TYPE				
							SPLIT/ SPLITLESS				
1	1	GC 1 µL HPLC 20 µL	1	1	1	1.5					
PULSED SPLITLESS	DIRECT	SPLIT/SPLITLESS	SPLITLESS, ON COLUMN	SPLIT/ SPLITLESS			DETERMINATION				
GC-ECD/NPD	GC-NPD	GC-ECD, GC-NPD, GC-FPD, HPLC-UV, LC-MS/MS	GC-ECD, FPD, NPD	GC-ECD, GC-NPD, GC-FPD, GC-MS, HPLC-FL, HPLC-UV	GC-ECD, GC-NPD, GC-FPD, GC-MS, HPLC-FL, HPLC-UV	LC-MS/MS					
		MODULARE MULTIMETHODE S19	EN 12393-1,2,3	INTERNAL METHOD	QUECHERS		REFERENCE METHOD				

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>DIMETHOATE</b>							
							LAB CODE
63	62	61	60	59	58		
D	D	D	D	D	D		SCOPE OF YOUR METHOD
M	M	M	M	M	M		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-MS	GC-MS	LC-MS/MS	GC-MS	GC-MS	GC-FPD		CONFIRMATION METHOD
0.02	0.02	0.01	0.02	0.02	0.02		RL (mg/Kg)
112				90.3	88		RECOVERY (%)
75	10	20	15	10	25		SAMPLE WEIGHT (g)
ACETONE FOLLOWED BY CYCLOHEXANE AND ETHYLACETATE	1	METHANOL	1	5	4		EXTRACTION SOLVENT
GPC							CLEAN-UP STEP
TPP		ISODRIN					INTERNAL STANDARD
1	2	10	1	2	1		INJECTION VOLUME ( $\mu$ L)
GC-NPD		SPLIT/SPLITLESS					INJECTION TYPE
GC-MS	LC-MS/MS	GC-ECD/NPD/MS	GC-MS	GC-FPD			DETERMINATION
							ANAL METHODS FOR PESTICIDES RESIDUES IN FOODSTUFFS (NL)
				\$35 L 000034			REFERENCE METHOD
							§ 35 LMBG L.00.00-34

**APPENDIX 7. Methods used by participants for determining pesticides.**

DIMETHOATE						
LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	CONFIRMATION METHOD	GC-MS	GC-MS/MS	LAB CODE
68	67	66	65	64		
D	D	D	D	D		
S	M	M	M	M		
LC-MS/MS						
0.01	0.02	0.02	0.02	0.02	RL (mg/Kg)	
90	80	112	97	96	RECOVERY (%)	
20	10	15	5	5	SAMPLE WEIGHT (g)	
4	5	5	1	6	EXTRACTION SOLVENT	
					CLEAN-UP STEP	
	LLE + SPE	SPE	GPC			
	TRIPHENYLPHOSPHATE		AZOBENZENE+RONNELL+ TPP	TPP	INTERNAL STANDARD	
5	10	10	2	0.5	INJECTION VOLUME (µL)	
LOOP			ON COLUMN	SPLITLESS	INJECTION TYPE	
LC-MS/MS	LC-MS/MS	GC-NPD-ECD-FPD(P)	GC-(ECD+FPD+NPD)	GC-MS	DETERMINATION	
MINI LUKE EXTRACTION	ANASTASSADES, M. ET AL., J. AOAC INT., 86 (2003), 412 – 43; QUECHERS - METHOD	QUECHERSILEOTHAY, S. ET AL. J.A.O.A.C. INT. (2005) 88, 615-629	INTERNAL METHOD	MULTIRESIDUE WITH EXTRELUT	REFERENCE METHOD	

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>DIMETHOATE</b>							
							LAB CODE
73	72	71	70	69			
D	D	D	D	D	D	D	SCOPE OF YOUR METHOD
M	S	M	M	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-MS	GC-NPD	GC-TSD	GC-MS/MS	GC-MS			CONFIRMATION METHOD
0.01	0.02	0.02	0.02	0.02			RL (mg/Kg)
100	112	118			86		RECOVERY (%)
30	100	25	15	25			SAMPLE WEIGHT (g)
1	3	1	5	1			EXTRACTION SOLVENT
		O	O				CLEAN-UP STEP
			O				INTERNAL STANDARD
1	1	5	10	1,0-2,0			INJECTION VOLUME ( $\mu$ L)
WIDE BORE	SPLITLESS	SPLITLESS	LVI	SPLITLESS			INJECTION TYPE
GC-NPD	GC-ECD, GC-NPD	GC-TSD	GC-ECD/TSI/PFPD GC-MS/MS	GC-NPD, GC-ECD			DETERMINATION
LOCAL SOP					MULTIRESIDUE METHODS FOR ANALYTICAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS P.A. GREVE ED. 1988, MINISTRY OF WELFARE, HEALTH AND CULTURAL AFFAIR, RUISWICK NETHERLANDS	QUECHERS	REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

DIMETHOATE									
79	78	77	76	75	74	LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	CONFIRMATION METHOD
D	D	NO RESULTS	D	D	D	D	D	M	GC-MS
M	M	S	S	M	M	M	M	M	GC-MS
GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS
0.02			0.01	0.01	0.01	0.01	0.01	0.01	RL (mg/Kg)
91	80		112	109	109	109	109	109	RECOVERY (%)
15	15		10	10	10	10	10	10	SAMPLE WEIGHT (g)
6	1	DICHLOROMETHANE	6 METHANOL	6 METHANOL	6 METHANOL	6 METHANOL	6 METHANOL	6 METHANOL	EXTRACTION SOLVENT
SPE	GPC	LLE (CHEM ELUT)	HEXBROMOBENZENE	CLEAN-UP STEP	HEXBROMOBENZENE	CLEAN-UP STEP	HEXBROMOBENZENE	HEXBROMOBENZENE	CONFIRMATION METHOD
2	1	1	20	INTERNAL STANDARD	1	INTERNAL STANDARD	1	INTERNAL STANDARD	INTERNAL STANDARD
SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	INJECTION VOLUME (µL)	1	INJECTION VOLUME (µL)	1	INJECTION VOLUME (µL)	INJECTION VOLUME (µL)
GC-ECD GC-NPD GC-MS LC-MS/MS HPLC-FLD	GC-MS (SINGLE-QUAD)	GC-MS	GC-MS	DETERMINATION	GC-MS	DETERMINATION	GC-MS	DETERMINATION	DETERMINATION
INTERNAL METHOD	JAOAC INTERN. 86. 1015FF (2003)	INTERNAL METHOD	INTERNAL METHOD	REFERENCE METHOD	INTERNAL METHOD	REFERENCE METHOD	INTERNAL METHOD	REFERENCE METHOD	REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

DIMETHOATE									
85	84	83	82	81	80	LAB CODE			SCOPE OF YOUR METHOD
D	D	D	D	D	D				Q <sup>UANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX</sup>
M	M	M	M	M	M				M
GC-MS	GC-MS	LC-MS/MS GC-MS	GC-ECD	LC-MS/MS	LC-MS/MS	CONFIRMATION METHOD			RL (mg/Kg)
0.02	0.02	0.02	0.01	0.05	0.05				RECOVERY (%)
80	80	101	92	20	20				SAMPLE WEIGHT (g)
25	50	10 30	10 30	10	10				EXTRACTION SOLVENT
4	2	5 1	5 1	6	6				CLEAN-UP STEP
		SPE	O (PSA)	O	O				INTERNAL STANDARD
		TRIPHENYL ETHYLENE							INJECTION VOLUME (µL)
1	1	7 3	1	20	20				INJECTION TYPE
SPLITLESS	SPLITLESS	PARTIAL SPLITLESS	SPLITLESS AND ON COLUMN						DETERMINATION
GC-NPD	GC-MS (SINGLE QUAD)	LC-MS/MS GC-MS	GC-ECD, GC-NPD, GC-FPD, GC-MS, HPLC-FLD, HPLC-DAD	LC-MS/MS	KLEIN J, ALDER L (2003) JAOC INT. 86, 1015-103				REFERENCE METHOD
SAR-1-04	EN NF 12393		QUECHERS, M. ANASTASSIADES, S. J. LEHOTAY, D. STAUNBAHNER, F. J. SCHENK: J. AOAC INT., 86 (2003) 412-431						

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>DIMETHOATE</b>							
							LAB CODE
98	97	96	95	94	93		
D	NA	D	D	D	D		SCOPE OF YOUR METHOD
M		S	S	M	M		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
LC-MS/MS	GC-MS	GC-MS	GC-ECD	GC-ECD	GC-MS		CONFIRMATION METHOD
0.02	0.1	0.05	0.02	0.005	RL (mg/Kg)		
129	98	99	100.1		RECOVERY (%)		
10		25	10	80	SAMPLE WEIGHT (g)		
1	2	5	4	2	EXTRACTION SOLVENT		
					CLEAN-UP STEP		
					GPC	GPC	
					SPE	SPE	
CARBENDAZIM D4, METHOMYL D3, PENDIMETHALIN D5	ISODRIN, CHINOMETHIONAT	YES					INTERNAL STANDARD
10	1	3	2	1µL PER COLUMN			INJECTION VOLUME (µL)
	SPLIT/SPLIFLES	PTV	SPLITLESS	SPLITLESS			INJECTION TYPE
LC-MS/MS	GC-ECD GC-NPD; GC-MS (SINGLE-GUAD)	GC-ECD, GC-NPD, GC-MS	GC-ECD	GC-ECD AND GC-NPD OR GC-ECD OR GC-NPD			DETERMINATION
	SOP 5.4 CH.1 (ACCORDING TO EN 12993/1-3, AND 35 LMBG L00.0034	FILLION ET AL. JOURNAL OF AOAC INTERNATIONAL 78-5- 1995		GC-MS (SINGLE-QUAD)			REFERENCE METHOD
LC-MS MULTI RESIDUE							

**APPENDIX 7. Methods used by participants for determining pesticides.**

DIMETHOATE									
LAB CODE	99	100	101	102	103	SCOPE OF YOUR METHOD	CONFIRMATION METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	LAB CODE
D	D	D	D	D	D	D	GC-NPD	M	
S	M	M	M	M	M	M	GC-MS	M	
GC-MS	GC-NPD	GC-MS	GC-NPD	GC-NPD	GC-NPD	GC-NPD	GC-MS	GC-MS	
0.020	0.02	0.05	0.05	0.02	0.02	0.05	RL (mg/Kg)	RL (mg/Kg)	
70	111	95	94	111	111	95	RECOVERY (%)	RECOVERY (%)	
50	30	10	25	30	30	10	SAMPLE WEIGHT (g)	SAMPLE WEIGHT (g)	
1	1	2	1	1	1	2	EXTRACTION SOLVENT	EXTRACTION SOLVENT	
SPE	GPC	SPE	GPC	SPE	SPE	SPE	CLEAN-UP STEP	CLEAN-UP STEP	
DIRECT	SPLITLESS	SPLITLESS	SPLIT/SPLITLESS PTV	SPLITLESS	SPLITLESS	SPLIT/SPLITLESS PTV	INTERNAL STANDARD	INTERNAL STANDARD	
GC-ECD GC-NPD GC-FPD GC-MS LC-MS	GC-ECD	GC-ECD	GC-ECD, NPD, MS	GC-ECD	GC-ECD	GC-ECD, NPD, MS	PULSED SPLITLESS	PULSED SPLITLESS	
ANN FA/SEXP CHIM. SEPT-OCT 1974-67 N/721-722 PP513-524	SOP MR 405012: EXTRACTION OF RESIDUES OF PLANT-PROTECTING MATERIAL FROM FRUIT AND VEGETABLE SAMPLES BY ETHYL-ACETATE; SOP 308M2: CLEAN UP OF EXTRACTS BY GEL PERMEATION CHROMATOGRAPHY	SOP 308M2: CLEAN UP OF EXTRACTS BY GEL PERMEATION CHROMATOGRAPHY	MINISTRY OF WELFARE, HEALTH AND CULTURAL AFFAIRS, NETHERLANDS. ANALYTICAL METHODS FOR RESIDUES OF PESTICIDES IN FOODSTUFFS, MULTIRESIDUE METHOD 1, 3.1-1, 6TH EDITION, 1996	MINISTRY OF WELFARE, HEALTH AND CULTURAL AFFAIRS, NETHERLANDS. ANALYTICAL METHODS FOR RESIDUES OF PESTICIDES IN FOODSTUFFS, MULTIRESIDUE METHOD 1, 3.1-1, 6TH EDITION, 1996	REFERENCE METHOD	REFERENCE METHOD	DETERMINATION	DETERMINATION	

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>DIMETHOATE</b>							
109	108	107	106	105	104	LAB CODE	SCOPE OF YOUR METHOD
D	D	D	D	D	D		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
M	M	M	S	M	S		CONFIRMATION METHOD
GC-MS	GC-NPD	GC-MS	GC-PFPD	GC-NPD	GC-MS		RL (mg/Kg)
0.02	0.02	0.005	0.01	0.05	0.005		RECOVERY (%)
110			80				
10	50	100	25	100	100		SAMPLE WEIGHT (g)
2	1	2	6 ETHYLACETATE/DICHLOROMETHANE	4	2		EXTRACTION SOLVENT
							CLEAN-UP STEP
			GPC		GPC		
							INTERNAL STANDARD
				ALDRIN	TRIPHENYLPHOSPHATE		
2	2	1		1	1		INJECTION VOLUME (µL)
	ON COLUMN	SPLITLESS					INJECTION TYPE
	GC-ECD GC-NPD GC-FPD GC-MS	GC NPD	GC-ECD; GC-NPD; GC-MS (SINGLE-QUAD)	GC-PFPD NPD GC-MS	GC-FPD GC-ECD		DETERMINATION
						MULTIRESIDUE METHOD 5, ORGANOPHOSPHOROUS COMPOUNDS MIN. WELFARE NETHERLANDS	LMBG §35 L-00.00.-34 AOAC 985.22/90 MODIFIED
							REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

DIMETHOATE						
						LAB CODE
					SCOPE OF YOUR METHOD	
					QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
114	113	112	111	110		
D	D	ND	D	D	D	
S	S		S	M	M	
GC-MS	GC-NPD	LC-MS/MS	GC-NPD	GC-NPD	CONFIRMATION METHOD	
0.02	0.02	0.01	0.02	0.02	RL (mg/Kg)	
70.5	105	86	100.3		RECOVERY (%)	
50	30	50	50	50	SAMPLE WEIGHT (g)	
6	1	6	6 METHANOL	6	EXTRACTION SOLVENT	
					CLEAN-UP STEP	
		SPE	SPE	GPC		
					INTERNAL STANDARD	
			YES	BROMOPHOS-ETHYL		
1	1	1	25	2	INJECTION VOLUME (µL)	
	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	INJECTION TYPE	
GC-MS	GC-NPD	GC-MS/MS	GC-ECD AND GC-NPD	GC-ECD AND GC-NPD	DETERMINATION	
	SOP MR 405012; EXTRACTION OF RESIDUES OF PLANT-PROTECTING MATERIAL FROM FRUIT AND VEGETABLE SAMPLES BY ETHYL-ACETATE	VALIDATED IN HOUSE METHOD NACH MULTIMETHOD, KLEIN UND ALDER - DFG WORKSHOP PRESENTED	MULTI-RESIDUE-METHOD DFG S 19	MULTI-RESIDUE-METHOD DFG S 19	REFERENCE METHOD	
	ISTISAN 97/23					

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>DIMETHOATE</b>							
							LAB CODE
121	120	119	118	117	116	115	
D	D	D	D	ND	D	D	SCOPE OF YOUR METHOD
S	S	S	S			S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-ECD	GC-MS	GC-ECD	GC-ECD			GC-PFPD	CONFIRMATION METHOD
<0.020	0.003	0.02	0.02	0.03	0.01	0.01	RL (mg/Kg)
75		106.5	102		120		RECOVERY (%)
100	100	10	5	40	15	50	SAMPLE WEIGHT (g)
3, 1, CYCLOHEXANE, N-HEXANE, IZO-OCTANE, TOLUENE	3	3	3	1	6	3	EXTRACTION SOLVENT
GPC, SPE	GPC	O			GPC	SPE	CLEAN-UP STEP
							INTERNAL STANDARD
					YES		
2	1	1	2	1	1	1	INJECTION VOLUME (µL)
SPLITLESS	SPLIT	AUTO	SPLITLESS	SPLITLESS	SPLITLESS	ON COLUMN	INJECTION TYPE
GC-ECD	GC-ECD GC-NPD	GC-ECD, GC-NPD GC-MS	GC-MS	GC-ECD, GC-NPD, GC-MS(TD)	GC-ECD GC-PFPD GC-MS		DETERMINATION
PN-EN 12393-1,2,3:2000	PN-EN 12393-1,2,3	ISS B6		RAPPORTO ISTISAN 37/23	NFEN12393		REFERENCE METHOD

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>FENHEXAMID</b>							
6	5	4	3	2	1	LAB CODE	SCOPE OF YOUR METHOD
D	D	D	NA	D	D		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
M	M	M		M	M		GC-MS
GC-MS	GC-MS	GC-ECD		LC-MS/MS	GC-MS		CONFIRMATION METHOD
0.02	0.05	0.021		0.010	0.05		RL (mg/Kg)
				98			RECOVERY (%)
15	10	10	10	10	10		SAMPLE WEIGHT (g)
4	5	5	5	6	6		EXTRACTION SOLVENT
GPC	SPE	SPE	SPE	SPE	SPE		CLEAN-UP STEP
	MIREX/TPP						INTERNAL STANDARD
2	2	2	2	5	5		INJECTION VOLUME ( $\mu$ L)
SPLITLESS	SPLIT/SPLITLESS	SPLIT/SPLITLESS	SPLIT/SPLITLESS				INJECTION TYPE
GC-MS	GC-ECD/ GC-NPD	GC-MS		LC-MS/MS	GC-MS		DETERMINATION
ANALYTICAL METHODS FOR PESTICIDE RESIDUES MINISTRY OF HEALTH NETHERLANDS 6TH ED	MANASTASSIADES ET AL. JAOAC 86, 412-431 (2003)	QUECHERS; ANASTASSIADES		KLEIN, J., ALDER, L. (2003); APPLICABILITY OF GRADIENT LIQUID CHROMATOGRAPHY WITH TANDEM MASS SPECTROMETRY TO THE SIMULTANEOUS SCREENING FOR ABOUT 100 PESTICIDES IN CROPS. J. AOAC INT. 86 (5): 1015-1037			REFERENCE METHOD IN HOUSE

**APPENDIX 7. Methods used by participants for determining pesticides.**

FENHEXAMID									
14	13	12	11	10	9	8	7	LAB CODE	
NA	NA	D	NA	D	D	D	D	SCOPE OF YOUR METHOD	
M	S	S	S	S	M	S	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
LC-MS	GC-MS/ITD	GC-MS/ITD	GC-MS/ITD	GC-MS/ITD	GC-MS	GC-ECD	GC-ECD	CONFIRMATION METHOD	
0.02	0.05	0.01	0.01	0.05	0.05	0.02	0.02	RL (mg/Kg)	
98	96	96	96	101	101	101	101	RECOVERY (%)	
50	30	15	15	30	30	10	10	SAMPLE WEIGHT (g)	
1	1	4	4	1	1	6 (DICHLOROMETHANE)	6 (DICHLOROMETHANE)	EXTRACTION SOLVENT	
GPC	LLE ACETONE FOLLOWED BY DICHLOROMETHANE AND PETROLEUM ETHER	O	O	O	O	O	O	CLEAN-UP STEP	
1,2,5	1	5	5	2	2	4	4	INTERNAL STANDARD	
SPLITLESS	SPLIT/SPURITLESS	LOOP	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	INJECTION VOLUME (µL)	
MS-ITD, LC-MS, GC-FPD, GC-ECD	GC-ECD	GC-MS/MS	GC-MS/MS	GC-ECD; GC-NPD(80)	GC-ECD; GC-NPD(80)	GC-ECD; GC-NPD(80)	GC-ECD; GC-NPD(80)	DETERMINATION	
		IN-HOUSE SOP	IN-HOUSE SOP	RAPPORT ISTIT SAN 97/23	REFERENCE METHOD				

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>FENHEXAMID</b>										
	<b>23</b>	<b>22</b>	<b>21</b>	<b>20</b>	<b>19</b>	<b>18</b>	<b>17</b>	<b>16</b>	<b>15</b>	<b>LAB CODE</b>
D	D	NA	D	D	D	NA	NA	NA	D	SCOPE OF YOUR METHOD
M	M	S	M	S	S				S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
HPLC-DAD	LC-MS/MS	GC-NPD	LC-MS/MS	GC-MS	GC-MS				GC-MS	CONFIRMATION METHOD
0.05	0.05	0.05	0.01	0.03	RL (mg/Kg)				0.05	RECOVERY (%)
	84.9	82	85	98					114	
5	10	100	75	75					25	SAMPLE WEIGHT (g)
5	METHANOL	3	1	1					1	EXTRACTION SOLVENT
SPE FLORISIL	O									CLEAN-UP STEP
										GPC
										INTERNAL STANDARD
						ALDRIN				
20	5	2	5	2					2	INJECTION VOLUME ( $\mu$ L)
AUTOMATIC		MANUAL			PULSED SPLITLESS					INJECTION TYPE
HPLC-DAD	LC-MS/MS	GC-NPD	LC-MS/MS	GC-MS					GC-ECD	DETERMINATION
										LOCAL SOP NO. MR405012
										REFERENCE METHOD
										INTERNAL METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

FENHEXAMID									
29	28	27	26	25	24	LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	CONFIRMATION METHOD
D	D	D	D	D	D	D	D	M	GC-MS
M	S	S	M	M	M	M	M	M	GC-MS
LC-MS/MS	GC-MS	LC-MS/MS	LC-MS/MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS
0.05	0.05	0.05	0.01	<0.02	0.05	RL (mg/Kg)	RL (mg/Kg)	RECOVERY (%)	RECOVERY (%)
117	84	91	91	97	98.2				
15	50	30	10	10	30	SAMPLE WEIGHT (g)	SAMPLE WEIGHT (g)		
5	6	1	6 METHANOL 95%-AMMONIACETATE 5%, 20 mM ACETIC ACID	6	1	EXTRACTION SOLVENT	EXTRACTION SOLVENT		
						CLEAN-UP STEP	CLEAN-UP STEP		
						GPC	GPC		
						SPE	SPE		
						CARBARYL C13	YES	INTERNAL STANDARD	INTERNAL STANDARD
						YES	YES		
50	1	20	10	2	1 mg SAMPLE			INJECTION VOLUME (µL)	INJECTION VOLUME (µL)
		RHEODYNE INJECTOR	SPLITLESS	SPLITLESS	TPOCI			INJECTION TYPE	INJECTION TYPE
LC-MS/MS	GC-MS(ITD)	HPLC-UV	LC-MS/MS	GC-MS	GC-ECD			DETERMINATION	DETERMINATION
						HUNGARIAN CENTRAL SOP NO.: 405012 M1	SOP NO.: MR 405012 M1; 308 M2	REFERENCE METHOD	REFERENCE METHOD
QUECHERS									

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>FENHEXAMID</b>							
						LAB CODE	
						SCOPE OF YOUR METHOD	
				QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX			
				CONFIRMATION METHOD			
				MS		DIFFERENT COLUMN	
				GC-MS		GC-MS	
36	D	35	NA	34	NA	32	D
S	M			S	D	S	D
0.05	0.02			0.05	0.05	0.05	
85	102			87	107		
50	20			25	10	10	
1	3			2	6	1	
EXTRACTION SOLVENT							
CLEAN-UP STEP							
INTERNAL STANDARD							
DICHLOFENTHION							
1	2			1	1	1	
INJECTION VOLUME (µL)							
INJECTION TYPE							
SPLITLESS							
SPLITLESS							
GC-ECD, GC-NPD							
GC-MS (SINGLE-QUAD.)							
DETERMINATION							
RAPP ISTISAN 97/23							
REFERENCE METHOD							
EN 12393-1, 2, 3							
\$35 LMBG 00,00-34							

**APPENDIX 7. Methods used by participants for determining pesticides.**

FENHEXAMID									
									LAB CODE
									SCOPE OF YOUR METHOD
42	41	40	39	38	37				
D	D	D	D	D	D				
M	M	M	M	M	M				
LC-MS/MS	GC-MS	GC-CMS	LC-MS/MS	GC-MS	LC-MS/MS	CONFIRMATION METHOD			
0.01	0.02	0.05	0.01	0.05	0.005	RL (mg/Kg)			
93	96.5		74			RECOVERY (%)			
10	20	15	10	10	10	SAMPLE WEIGHT (g)			
5	3	4	6 (METHANOL/WATER)	5	5	EXTRACTION SOLVENT			
						CLEAN-UP STEP			
			SPE	O	O				
						INTERNAL STANDARD			
						DIMETHOATE D6			
20	4	1		20	2	INJECTION VOLUME (µL)			
			SPLITLESS			SPLITLESS			
						INJECTION TYPE			
							GC-ECD		
							GC-NPD	LC-MS/MS (TRIPLE-QUAD)	
							GC-MS	DETERMINATION	
LC-MS/MS	GC-ECD, GC-NPD	GC-NPD, GC-ECD, GC-MS(IRD)	LC-MS/MS						
M. ANASTASSIADES ET AL., J. AOAC INT. 86 (2), 412-431, (2003)		ANALYTICAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS, 6TH ED., 1996 GENERAL INSPECTORATE FOR HEALTH PROTECTION, MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS	LC-MS/MS (BFR)	ANASTASSIADES, JOURNAL OF AOAC, INT. VOL. T 86 NO 2, 2003	REFERENCE METHOD				

**APPENDIX 7. Methods used by participants for determining pesticides.**

FENHEXAMID										
51	50	49	48	47	46	45	44	43	LAB CODE	SCOPE OF YOUR METHOD
NA	D	NA	D	D	NA	D	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
S		M		S		M	M	M	M	CONFIRMATION METHOD
LC-MS		GC-MS		GC-ECD		GC-MS		GC-MS		RL (mg/Kg)
0.05		0.02		0.01		0.05	0.03	0.01	0.01	RECOVERY (%)
100						66		94		SAMPLE WEIGHT (g)
10		25		10		25	50	10		EXTRACTION SOLVENT
4		2		5		1	2	5		CLEAN-UP STEP
										INTERNAL STANDARD
										NICARBAZIN
										INJECTION VOLUME (µL)
										INJECTION TYPE
LC-MS/MS		GC-MS								DETERMINATION
										ANASTASSIADES ET AL. JAOAC INT. 86 82003 412-431, MODIFIED
										REFERENCE METHOD
										DFG S 19 EN 12393 P METHOD 412-431

**APPENDIX 7. Methods used by participants for determining pesticides.**

FENHEXAMID									
59	58	57	56	55	54	53	52	LAB CODE	SCOPE OF YOUR METHOD
D	D	D	D	D	D	D	D		
M	M	M	S	M	M	S	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
GC-MS	LC-MS/MS	LC-MS/MS	GC-MS	GC-ECD	GC-MS	GC-MS	GC-ECD	CONFIRMATION METHOD	
0.02	0.05	0.01	0.693	0.02	0.05	0.05	0.05	RL (mg/Kg)	
91.3	110		91.6	90.1	80			RECOVERY (%)	
10	10	25	30	100	18	15	10	SAMPLE WEIGHT (g)	
5	6	5	1	2	1	6	5	EXTRACTION SOLVENT	
O	LLE ON CHEM ELUT		GPC	GPC	GPC	GPC	O	CLEAN-UP STEP	
3								INTERNAL STANDARD	
2	100	5	1	GC 1 µL HPLC 20 µL	1	1	1.5	INJECTION VOLUME (µL)	
SPLITLESS			SPLIT	SPLIT/SPLITLESS	SPLITLESS, ON COLUMN	SPLIT/ SPLITLESS	SPLIT/ SPLITLESS	INJECTION TYPE	
GC-MS	LC-MS/MS	GC-ECD	GC-ECD, GC-NPD, GC-FPD, HPLC-UV, LC-MS/MS	GC-ECD, FPD,NPD	GC-ECD, GC-NPD, GC-FPD, HPLC-FID, HPLC-UV	GC-ECD, GC- NPD, GC-FPD, GC-MS, HPLC-FID, HPLC-UV	GC-ECD, GC-NPD, GC-FPD, GC-MS	DETERMINATION	
§35 L 000034	APPL OF LC-MS/MS (J. KLEIN, L. ALDERS)			MODULARE MULTIMETHODE S19	EN 12393-1,2,3	INTERNAL METHOD	QUEOHERS	REFERENCE METHOD	

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>FENHEXAMID</b>							
<b>66</b>	<b>65</b>	<b>64</b>	<b>63</b>	<b>62</b>	<b>61</b>	<b>60</b>	<b>LAB CODE</b>
NA	D	NA	D	NA	D	D	<b>SCOPE OF YOUR METHOD</b>
M		M		M	M	M	<b>QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX</b>
GC-MS		GC-MS		LC-MS/MS	GC-MS	GC-MS	<b>CONFIRMATION METHOD</b>
0.05		0.05		0.05	RL (mg/Kg)	0.05	<b>RECOVERY (%)</b>
83		88					
5		75		20	15	15	<b>SAMPLE WEIGHT (g)</b>
1		AACETONE FOLLOWED BY CYCLOHEXANE AND ETHYLACETATE		METHANOL	1	1	<b>EXTRACTION SOLVENT</b>
					GPC	GPC	<b>CLEAN-UP STEP</b>
							<b>INTERNAL STANDARD</b>
		AZORENENE <sup>+</sup> RONNEL+TRIPHENYLPHOSPHATE				ISODRIN	<b>INTERNAL STANDARD</b>
		2		1	10	1	<b>INJECTION VOLUME (µL)</b>
		ON COLUMN					<b>INJECTION TYPE</b>
							<b>DETERMINATION</b>
		GC-(ECD+FPD+NPD)		GC-MS/MS	GC-ECD/NPD/MS	GC-ECD/NPD/MS	<b>REFERENCE METHOD</b>
		INTERNAL METHOD					§ 35 LMBG L.00.00-34

**APPENDIX 7. Methods used by participants for determining pesticides.**

FENHEXAMID									
72	71	70	69	68	67	LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	CONFIRMATION METHOD
D	D	NA	D	D	D	D	D	M	GC-MS
S	M	M	M	M	M	M	M	M	RL (mg/Kg)
GC-ECD	GC-TSD	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	RL (mg/Kg)
0.02	0.1	0.05	0.05	0.02	0.02	0.02	0.02	0.02	RECOVERY (%)
106	77	113	98.6	65	65	65	65	65	SAMPLE WEIGHT (g)
100	25	25	20	10	10	10	10	10	EXTRACTION SOLVENT
3	1	1	4	5	5	5	5	5	CLEAN-UP STEP
		O	GPC	LLE + SPE	LLE + SPE	LLE + SPE	LLE + SPE	LLE + SPE	
				TRIPHENYLPHOSPHATE	TRIPHENYLPHOSPHATE	TRIPHENYLPHOSPHATE	TRIPHENYLPHOSPHATE	TRIPHENYLPHOSPHATE	INTERNAL STANDARD
1	5	10-20	2	10	10	10	10	10	INJECTION VOLUME (µL)
	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	INJECTION TYPE
	GC-ECD, GC-NPD	GC-TSD	GC-NPD, GC-ECD	GC-ECD/NPD	GC-ECD/NPD	GC-ECD/NPD	GC-ECD/NPD	GC-ECD/NPD	DETERMINATION
			MULTIRESIDUE METHOD FOR ANALYSIS OF PESTICIDES IN FOODSTUFFS P.A. GREVE ED., 1988, MINISTRY OF WELFARE, HEALTH AND CULTURAL AFFAIR, RUSWICK NETHERLANDS	MULTIRESIDUE METHOD 1, ANALYTICAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS, SIXTH EDITION	MINI LUKE EXTRACTION	ANASTASSIADES, M. ET AL. J. AOAC INT., 86 (2003), 412 – 431 QUECHERS - METHOD	ANASTASSIADES, M. ET AL. J. AOAC INT., 86 (2003), 412 – 431 QUECHERS - METHOD	ANASTASSIADES, M. ET AL. J. AOAC INT., 86 (2003), 412 – 431 QUECHERS - METHOD	REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

FENHEXAMID									
								LAB CODE	
								SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
								M	
80	D	79	78	77	76	75	74	73	73
M	M	NA	D	D	D	D	D	D	D
LC-MS/MS	GC-MS	S	S	S	M	M	M	M	M
0.05	0.05	0.01	0.01	0.01	0.05	0.05	0.05	0.05	RL (mg/Kg)
98	98	130	130	103	103.8	103.8	78	78	RECOVERY (%)
10	15	10	50	50	25	25	30	30	SAMPLE WEIGHT (g)
6	6	DICHLOROMETHANE	DICHLOROMETHANE	DICHLOROMETHANE	DICHLOROMETHANE	DICHLOROMETHANE	DICHLOROMETHANE	DICHLOROMETHANE	EXTRACTION SOLVENT
O	O	SPE	SPE	SPE	GPC	HEXBROMOBENZENE	GPC	GPC	CLEAN-UP STEP
20	20	2	2	1	2	1	1	1	INTERNAL STANDARD
LC-MS/MS	GC-ECD	SPLITLESS	SPLITLESS	SPLITLESS	SPLIT/ SPLITLESS	SPLIT/ SPLITLESS	GC-MS	GC-MS	INJECTION VOLUME ( $\mu$ L)
KLEIN J., ALDERL (2003) JAOAC INT. 86, 1015-103	INTERNAL METHOD	GC-ECD; GC-NPD; GC-MS; LC-MS/MS; HPLC-FLD	GC-MS	GC-ECD	GC-MS	GC-MS	GC-MS (ON TRAP)	DETERMINATION	REFERENCE METHOD
§35 LMBG L 00.00-34 (1999)									
LOCAL SOP									
NO RESULTS									

## **APPENDIX 7. Methods used by participants for determining pesticides.**

FENHEXAMID									
									LAB CODE
87	86	85	84	83	82	81			
D	NA	NA	D	D	D				SCOPE OF YOUR METHOD
S		M		M	M				QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
LC-MS/MS	GC-MS	GC-MS	GC-ECD	GC-ECD	GC-FPD				CONFIRMATION METHOD
	0.05	0.05	0.02						RL (mg/Kg)
	80	109	108						RECOVERY (%)
50	50	30	10						SAMPLE WEIGHT (g)
6 ETHYLACETATEDICHLOROMETHANE (80/20)	2	1	5						EXTRACTION SOLVENT
									CLEAN-UP STEP
			0 (PSA)						NO RESULTS
TRICHLORONATE	TRIPHENYL ETHER TRIPHENYL ETHER								INTERNAL STANDARD
1	1	3	1						INJECTION VOLUME (µL)
ON COLUMN	SPLITLESS	SPLITLESS	SPLITLESS AND ON COLUMN						INJECTION TYPE
GC-TSD	GC-MS (SINGLE-QUAD)	GC-MS GC-MS	GC-ECD, GC-NPD, GC-FPD, GC-MS, HPLC-FLD, HPLC-DAD						DETERMINATION
									REFERENCE METHOD
									QUECHERS, M. ANASTASSIDES, S. J. LEHOTAY, D. STAINBACHER, F. J. SCHENK; J. AOAC INT., 86 (2003) 412-431
									EN NF 1293
									ACCORDING TO EN 12396-1: 1999

**APPENDIX 7. Methods used by participants for determining pesticides.**

FENHEXAMID									
								SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
								CONFIRMATION METHOD	
								GC-MS	
95	94	93	92	91	90	89	88	GC-MS	GC-MS
D	D	D	D	D	D	D	D	GC-MS	GC-MS
S	M	M	M	M	M	M	M	GC-MS	GC-MS
GC-MS	GC-NPD	GC-MS	LC-MS/MS	LC-MS/MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS
0.05	0.05	0.02	0.01	0.006	0.01	0.03	0.05	RL (mg/Kg)	RL (mg/Kg)
100	108.5		76			90	86	RECOVERY (%)	RECOVERY (%)
25	10	80	50	10	50	15	25	SAMPLE WEIGHT (g)	SAMPLE WEIGHT (g)
5	6	2	4	6 METHANOL	2	6	1	EXTRACTION SOLVENT	EXTRACTION SOLVENT
SPE	SPE	GPC	LLE	SPE	GPC	GPC	GPC	CLEAN-UP STEP	CLEAN-UP STEP
YES								INTERNAL STANDARD	INTERNAL STANDARD
3	2	1µL PER COLUMN	10	20	1	1	1	INJECTION VOLUME (µL)	INJECTION VOLUME (µL)
PTV	SPLITLESS	SPLITLESS	LOOP	SPLITLESS	SPLIT/SPLITLESS	SPLITLESS	SPLITLESS	INJECTION TYPE	INJECTION TYPE
GC-ECD, GC-NPD, GC-MS	GC-NPD	GC-ECD AND GC-NPD OR GC-ECD OR GC-NPD	LC-MS/MS	GC-NPD	GC-ECD, FPD, MS HPLC-F-DAD	GC-ECD	GC-ECD	DETERMINATION	DETERMINATION
FILLION ET AL. JOURNAL OF AOAC INTERNATIO NAL 78-5- 1995		GC-MS (SINGLE-QUAD)	LC-MULTIRESIDUE DRAFT § 35 LMBG-METHOD					IN-HOUSE	REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

FENHEXAMID											
								LAB CODE			
								SCOPE OF YOUR METHOD			
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX			
								CONFIRMATION METHOD			
103	102	101	100	99	98	97	96	GC-MS			
D	D	NA	NA	NA	D	NA	D	SCOPE OF YOUR METHOD			
S	S			M		S		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX			
GC-MS	HPLC-DAD			LC-MS/MS		GC-MS		CONFIRMATION METHOD			
0.020	0.05			0.05		0.05		RL (mg/Kg)			
70	89			105		102		RECOVERY (%)			
50	30			10		25		SAMPLE WEIGHT (g)			
1	1			1		2		EXTRACTION SOLVENT			
CLEAN-UP STEP											
SPE											
GPC											
CARBENDAZIM D4, METHOMYL D3, PENDEMETHALIN D5											
ISODRIN, CHINOMETHIONAT											
1	50			10		1		INTERNAL STANDARD			
INJECTION VOLUME (µL)											
DIRECT											
AUTOSAMPLER											
GC-ECD GC-NPD GC-FPD GC-MS LC-MS											
HPLC-UV											
LC-MS/MS											
SOP MR 405012; EXTRACTION OF RESIDUES OF PLANT- PROTECTING MATERIAL FROM FRUIT AND VEGETABLE SAMPLES BY ETHYL ACETATE; SOP 308M2; CLEAN UP OF EXTRACTS BY GEL PERMEATION CHROMATOGRAPHY											
ANN.FALS.EXP.CHI M. SEFT-CCT 1974- 67 NT721-722 PP513-524											
SOP 5.4, CH.1 (ACCORDING TO EN 12393/1-3; AND 35 LMBG L00.0034											
LC-MS MULTI RESIDUE											
REFERENCE METHOD											

**APPENDIX 7. Methods used by participants for determining pesticides.**

FENHEXAMID									
110	109	108	107	106	105	104	LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
D	D	NA	D	D	NA	D			
M	S		M	S		S			
GC-ECD	LC-MS/MS		GC-MS	GC-NPD		LC-MS/MS	CONFIRMATION METHOD		
0.05	0.01		0.01	0.01		0.005	RL (mg/Kg)		
50	10		100	25			RECOVERY (%)		
6	1		2	6 ETHYLACETATE/DICHLOROMETHANE		10	SAMPLE WEIGHT (g)		
EXTRACTION SOLVENT									
DIATOMACEOUS EARTH									
CLEAN-UP STEP									
INTERNAL STANDARD									
INJECTION VOLUME (µL)									
INJECTION TYPE									
DETERMINATION									
APPLICATION NOTE BY WATERS									
REFERENCE METHOD									
MULTI-RESIDUE-METHOD DFG S 19									
S19 - DFG									
GC-ECD AND GC-NPD									
GC-ECD-ECD; GC-NPD; GC-MS (SINGLE-QUAD)									
GC-PPD									
GC-NPD									
GC-MS									
SPLITLESS									
BROMOPHOS-ETHYL									
GPC									
SPLITLESS									
GC-EC									
GC-MS/MS									
S19 - DFG									

## **APPENDIX 7. Methods used by participants for determining pesticides**

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>FENHEXAMID</b>											
128	127	126	125	124	123	122	121	120	119	LAB CODE	SCOPE OF YOUR METHOD
NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
											CONFIRMATION METHOD
											RL (mg/Kg)
											RECOVERY (%)
											SAMPLE WEIGHT (g)
											EXTRACTION SOLVENT
											CLEAN-UP STEP
											INTERNAL STANDARD
											INJECTION VOLUME ( $\mu$ L)
											INJECTION TYPE
											DETERMINATION
											REFERENCE METHOD

NO RESULTS

**APPENDIX 7. Methods used by participants for determining pesticides.**

FLUDIOXONIL									
LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	CONFIRMATION METHOD	GC-MSS	GC-MSS	GC-MSS	GC-NPD	M	M
5	4	3		2		1		1	1
D	D	D		D		D		D	D
M	M	M		M		M		M	M
GC-MS	GCNPD	GC-MS/MS		GC-MS		GC-MS		GC-MS	
0.05	0.05	0.01		0.050		0.05		RL (mg/Kg)	
				74.0	76			RECOVERY (%)	
-10	10	15		15	15			SAMPLE WEIGHT (g)	
5	5	1		4	4			EXTRACTION SOLVENT	
								CLEAN-UP STEP	
	SPE	NO						SPE	
(MIREX)/TPP		TRIPHENYL PHOSPHATE ANTHRAZENE						INTERNAL STANDARD	
1	2	10		1		2		INJECTION VOLUME ( $\mu$ L)	
SPLIT/SPLITLESS		SPLIT/SPLITLESS		SPLITLESS		SPLIT/SPLITLESS		INJECTION TYPE	
GC-MS		GC-MS/MS						DETERMINATION	
MANASTASSIADES ET AL. JAOAC 86, 412-431 (2003)	QUECHERS; ANASTASSIADES			GC-NPD		GC-MS		REFERENCE METHOD	
				LUKE M.A. FROBERG, J.E., DOOSE, G.M., MASUMOTO, H.T. (1981); IMPROVED MULTIRESIDUE GAS CHROMATOGRAPHIC DETERMINATION OF ORGANOPHOSPHORUS, ORGANONITROGEN, AND ORGANOHALOGEN PESTICIDES IN PRODUCE, USING FLAME PHOTOMETRIC AND ELECTROLYTIC CONDUCTIVITY DETECTORS, J. ASSOC. OFF. ANAL. CHEM. 64(5): 1187-1195					

## **APPENDIX 7. Methods used by participants for determining pesticides.**

## **APPENDIX 7. Methods used by participants for determining pesticides.**

FLUDIOXONIL									
LAB CODE		SCOPE OF YOUR METHOD		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		CONFIRMATION METHOD			
23	22	21	20	19	18	17	16	15	
D	D	NA	NA	D	D	NA	D	D	
M	M			M	S		M	S	
GC-MS	LC-MS/MS	GC-MS/ITD	GC-MS	GC-MS/MS	GC-MS/MS	GC-MS	GC-MS	GC-MS	
0.01	0.05	0.05	0.04	0.05	0.04	0.05	0.05	0.05	RL (mg/Kg)
	81.2	69							RECOVERY (%)
5	10	75	75	15	15	25	25	25	SAMPLE WEIGHT (g)
5	METHANOL	1	1	4	4	1	1	1	EXTRACTION SOLVENT
									CLEAN-UP STEP
									GPC
FENCHLORPHOS		DITALIMPHOS	ALDRIN						INTERNAL STANDARD
50	5	2	2	10	10	2	2	2	INJECTION VOLUME (µL)
AUTOMATIC TDS SAMPLER		SPLITLESS	PULSED SPLITLESS	LVI SPLITLESS	SPLIT/SPLITLESS				INJECTION TYPE
TDS-IN LINE WITH GC-MS (SINGLE-QUAD) TOTAL ION DETECTION	LC-MS/MS	GC-ECD	GC-MS	GC-MS/MS	GC-ECD				DETERMINATION
INTERNAL METHOD									REFERENCE METHOD
									LOCAL SOP NO. MR405012

**APPENDIX 7. Methods used by participants for determining pesticides.**

FLUDIOXONIL									
31	30	29	28	27	26	25	24	LAB CODE	SCOPE OF YOUR METHOD
D	ND	D	D	D	D	D	D		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
S	S	M	S	S	M	M	M		CONFIRMATION METHOD
MS	DIFFERENT COLUMN	GC-MS/MS	GC-MS	GC-MS/ITD)	GC-MS	GC-MS	GC-MS		RL (mg/Kg)
0.05	0.05	0.05	0.03	0.05	0.04	<0.02	0.05		RECOVERY (%)
70		84	90		83	94	70.4		
10	10	15	20	30	25	10	30		SAMPLE WEIGHT (g)
6	1	4	3	1	2	6	1		EXTRACTION SOLVENT
O	GPC		GPC	GPC	GPC	SPE			CLEAN-UP STEP
	DICHOLOFENTHION				DITALIMPHOS	YES			INTERNAL STANDARD
1	1	10	20	20	2	2	1		INJECTION VOLUME (µL)
SPLITLESS	SPLITLESS		RHEODYNIE INJECTOR	SPLITLESS	SPLITLESS	TPOCI			INJECTION TYPE
GC-ECD GC-NPD	GC-MS/MS GC-NPD		LC-MS/MS	HPLC-UV	GC-ECD, GC-NPD, GC-MS/ITD)	GC-MS	GC-NPD		DETERMINATION
ISTISAN 97/23	RAPP ISTISAN 97/23	MINI LUKE			HUNGARIAN CENTRAL SOP NO.: 405012 M1			SOP NO.: MR 405012 M1	REFERENCE METHOD

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>FLUDIOXONIL</b>							
44	43	42	41	40	39	LAB CODE	SCOPE OF YOUR METHOD
D	D	D	NA	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
M	M	M	M	M	M	M	CONFIRMATION METHOD
	GC-MS	LC-MS/MS	GC-MS	GC-MS	LC-MS/MS	LC-MS/MS	RL (mg/Kg)
0.01	0.01	0.01	0.01	0.05	0.01	0.01	RECOVERY (%)
	94	94	94			80	SAMPLE WEIGHT (g)
50	10	10	10	15	10	10	EXTRACTION SOLVENT
2	5	5	5	4	6 (METHANOL/WATER)		CLEAN-UP STEP
LLE	SPE	LLE			SPE		
	NICARBAZIN						INTERNAL STANDARD
							INJECTION VOLUME (µL)
5	4	20		1	20		INJECTION TYPE
LC-MS/MS	LC-MS/MS(ESI-)	LC-MS/MS			GC-NPD, GC-ECD, GC-MS(TD)	LC-MS/MS	DETERMINATION
HOUSE METHOD	ANASTASSIADES ET AL., JAOAC INT. 86(2), 412-431, (2003) MODIFIED	M. ANASTASSIADES ET AL., J. AOAC INT. 86 (2), 412-431, (2003)			ANALYTICAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS, 6TH ED., 1996, GENERAL INSPECTORATE FOR PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS	LC-MS/MS (BFR)	REFERENCE METHOD

## **APPENDIX 7. Methods used by participants for determining pesticides.**

FLUDIOXONIL									
TESTS		TESTS		TESTS		TESTS		TESTS	
ITEM	TEST	TEST	TEST	TEST	TEST	TEST	TEST	TEST	TEST
52	51	50	49	48	47	46	45	44	43
D	NA	D	NA	D	D	NA	D	NA	D
M	S	M	M	S	S	S	S	S	D
GC-ECD	GC-MS	GC-MSS	GC-NPD	GC-NPD	GC-NPD	GC-MS	GC-MS	GC-MS	GC-MS
0.05	0.05	0.05	0.01	0.05	0.05	0.04	0.04	0.04	0.04
117	117	117	117	117	117	117	117	117	117
10	20	25	10	10	10	25	25	25	25
5	6	2	5	5	5	1 (IN PRESENCE OF NaOH)			
O	O	O	O	O	O	O	O	O	O
DICHLOBENIL	DICHLOBENIL	DICHLOBENIL	DICHLOBENIL	DICHLOBENIL	DICHLOBENIL	DICHLOBENIL	DICHLOBENIL	DICHLOBENIL	DICHLOBENIL
1.5	2	3	4	4	4	10	10	10	10
SPLIT/SPLITLESS	ON COLUMN	PTV	SPLIT/SPLITLESS	SPLIT/SPLITLESS	SPLIT/SPLITLESS	RHEODYNE	RHEODYNE	RHEODYNE	RHEODYNE
GC-ECD, GC-NPD, GC-MS	GC-NPD	GC-MS	GC-ECD, GC-NPD, GC-MS (SINGLE QUAD)	GC-ECD, GC-NPD, GC-MS (SINGLE QUAD)	GC-ECD, GC-NPD, GC-MS (SINGLE QUAD)	HPLC - DAD	HPLC - DAD	HPLC - DAD	HPLC - DAD
QUECHERS	DFG S 19	M. ANASTASSIADES, S. J. LEHOTAY, D. STANBAHER, F. J. SCHENCK J. AOAC INT. 86 (2003) 412-431	M. ANASTASSIADES, S. J. LEHOTAY, D. STANBAHER, F. J. SCHENCK J. AOAC INT. 86 (2003) 412-431	CLEAN UP AS IN EN 12393 P METHOD	CLEAN UP AS IN EN 12393 P METHOD	REFERENCE METHOD	REFERENCE METHOD	REFERENCE METHOD	REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

FLUDIOXONIL									
60	59	58	57	56	55	54	53	LAB CODE	SCOPE OF YOUR METHOD
D	D	D	NA	D	D	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
M	M	M	S	S	M	M	S	S	CONFIRMATION METHOD
GC-MS	GC-MS	GC-ECD-NPD	GC-MS	GC-NPD	GC-MS/MS	GC-MS/MS	GC-MS	GC-MS	RL (mg/Kg)
0.05	0.02	0.05	0.159	0.03	0.02	0.02	0.05	0.05	RECOVERY (%)
73.5	94	94	104.5	96.7	96	96	96	96	SAMPLE WEIGHT (g)
15	10	15	30	100	10	10	15	15	EXTRACTION SOLVENT
1	5	4	1	2	5	5	6	6	CLEAN-UP STEP
GPC	O			GPC	O	GPC	GPC	GPC	INTERNAL STANDARD
ISODRIN	3	PCB119 (ECD) TRIFLURALIN (GC-NPD)							INTERNAL STANDARD
1	2	1	1	GC 1 µL HPLC 20 µL	5	5	1	1	INJECTION VOLUME (µL)
SPLIT/SPLITLESS	SPLIT/SPLITLESS	SPLITLESS	SPLITLESS	SPLIT/ SPLITLESS	LVI	SPLIT/ SPLITLESS	SPLIT/ SPLITLESS	SPLIT/ SPLITLESS	INJECTION TYPE
GC-ECD/NPD/MS	GC-MS	GC-ECD-NPD	GC-MS(IRD)	GC-ECD, GC-NPD, GC-FPD, HPLC-LV LC-MS/MS	GC-MS/MS	GC-MS/MS	GC-ECD, GC-NPD, GC-FPD, HPLC-FLD, HPLC-UV	DETERMINATION	
§35 L 000034		ANAL.METHODS FOR PESTICIDES RESIDUES IN FOODSTUFFS (NL)		MODULARE MULTIMETHODE S19	QUECHERS	QUECHERS	INTERNAL METHOD	INTERNAL METHOD	REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

FLUDIOXONIL									
67	66	65	64	63	62	61	LAB CODE		
D	NA	D	NA	D	NA	D	SCOPE OF YOUR METHOD		
M	M	M	M	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		
							CONFIRMATION METHOD		
0.05			GC-MS	LC/MS/MS	GC-MS		RL (mg/Kg)		
			0.03	0.05	0.05		RECOVERY (%)		
			91	65			SAMPLE WEIGHT (g)		
90			5	75		50	EXTRACTION SOLVENT		
3			1	ACETONE FOLLOWED BY CYCLOHEXANE AND ETHYLACETATE					
							CLEAN-UP STEP		
			GPC	GPC	GPC				
							INTERNAL STANDARD		
			AZOBENZENE+RONNELL TRIPHENYLPHOSPHATE						
1			2	1		2	INJECTION VOLUME (µL)		
			SPLIT/SPLITLESS		ON COLUMN	PTV	INJECTION TYPE		
			GC-MS (AGILENT MSD)	GC-(ECD+FPD+NPD)	GC-NPD	GC-MS	DETERMINATION		
			OFFICIAL METHOD L 00.00-34; § 35 LMBG	INTERNAL METHOD	§ 35 LMBG L 00.00-34	DFG S19 (ASU \$35 LMBG L 00.00-34)	REFERENCE METHOD		

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>FLUDIOXONIL</b>									
75		74		73		72		71	
D	D	D	NA	NA	NA	D	D	D	LAB CODE
S	M	M				M	S	S	SCOPE OF YOUR METHOD
LC-MS/MS	GC-MS	GC-MS				GC-MS	LC-MS/MS	LC-MS/MS	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
0.01	0.05	0.05	0.05			0.02	0.01	RL (mg/Kg)	CONFIRMATION METHOD
91	98.7	84				97	83.1	RECOVERY (%)	
50	25	30				25	20	SAMPLE WEIGHT (g)	
2	3	1				1	4	EXTRACTION SOLVENT	
GPC	HEXBROMOBENZENE							CLEAN-UP STEP	
TPP								INTERNAL STANDARD	
2	1	1				1,0-2,0	5	INJECTION VOLUME (µL)	
SPLIT/SPLITLESS		WIDE BORE				SPLITLESS	LOOP	INJECTION TYPE	
GC-NPD	GC-MS	GC-NPD				GC-NPD, GC-ECD	LC-MS/MS	DETERMINATION	
§35 LMBG L 00-34 (1999)		LOCAL SOP				MULTIRESIDUE METHOD 1, ANALYTICAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS, SIXTH EDITION	MINI LUKE EXTRACTION	REFERENCE METHOD	

**APPENDIX 7. Methods used by participants for determining pesticides.**

FLUDIOXONIL									
LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	CONFIRMATION METHOD	RL (mg/Kg)	RECOVERY (%)	SAMPLE WEIGHT (g)	EXTRACTION SOLVENT	CLEAN-UP STEP	INTERNAL STANDARD
82	81	80	79	78	77	76	DICHLOROMETHANE	GC-MS	0.01
D	D	D	D	ND					D
M	M	M	M	M					S
GC-ECD	LC-MS/MS	GC-MS	GC-MS						
0.02	0.05	0.05							
83		88			91				
10	10	15	15			10			
5	6	6	1				DICHLOROMETHANE		
NO RESULTS									
0 (PSA)	O	SPE	GPC						
1	20	2	1			1			
NO RESULTS									
SPLITLESS AND ON COLUMN									
GC-ECD, GC-NPD, GC-FPD, GC-MS, HPLC-FLD, HPLC-DAD									
LC-MS/MS									
GC-ECD GC-NPD GC-MS (SINGLE-QUAD) LC-MS/MS HPLC-FLD									
QUECHERS, M. ANASTASIADIS, S. J. LEHOTAY, D. STAIBANNER, F. J. SCHENK: J. AOAC INT., 86, (2003) 1015-103 QUECHERS, M. ANASTASIADIS, S. J. LEHOTAY, D. STAIBANNER, F. J. SCHENK: J. AOAC INT., 86, (2003) 412-431									
KLEIN J., ALDER L (2003) JAOAC INT.: 86, 1015-103 INTERNAL METHOD									
REFERENCE METHOD									

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

FLUDIOXONIL									
96	95	94	93	92	91	90	LAB CODE	SCOPE OF YOUR METHOD	
D	D	NA	D	D	ND	D			
S	S	M	M	M	S		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		
GC-MS	GC-MS	GC-MS	GC-MS	GC-MS			CONFIRMATION METHOD		
0.05	0.15	0.03	0.05	0.02	0.02	0.02	RL (mg/Kg)		
93			102				RECOVERY (%)		
25	25	80	50	50	50	50	SAMPLE WEIGHT (g)		
2	5	2	4	3	3	2	EXTRACTION SOLVENT		
GPC	SPE	GPC	LLE	LLE	GPC	GPC	CLEAN-UP STEP		
ISODRIN, CHINOMETHIONAT	YES						INTERNAL STANDARD		
1	3	1 $\mu$ L PER COLUMN		ALDRIN, DITALIMPHOS	YES		INJECTION VOLUME ( $\mu$ L)		
SPLIT/SPLITLESS	PTV	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	INJECTION TYPE		
GC-ECD GC-NPD; GC-MD (SINGLE-QUAD)	GC-ECD, GC-NPD, GC-MS	GC-ECD AND GC-NPD OR GC-ECD OR GC-NPD	GC-ECD, GC-NPD, GC-MS	GC-NPD	GC-NPD	GC-NPD	DETERMINATION		
SOP 5.4.CH.1 (ACCORDING TO EN 12393/1-3; AND 35 LMBG L00.0034)	FILLION ET AL JOURNAL OG AOAC INTERNATIONAL 78-5- 1995	GC-MS (SINGLE-QUAD)	OFFICIAL METHOD OF ANALYSIS (1990) 15TH ED. 985.22, AOAC ARLINGTON VA	DFG-S8			REFERENCE METHOD		

## **APPENDIX 7. Methods used by participants for determining pesticides.**

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

FLUDIOXONIL									
116	115	114	113	112	111	110	LAB CODE		
D	D	D	D	NA	D	D	SCOPE OF YOUR METHOD		
S	S	S	S		M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		
GC-MS	GC-MS	GC-MS	HPLC-UV		GC-MS	GC-MS	CONFIRMATION METHOD		
0.05	0.01	0.02	0.05		0.05	0.05	RL (mg/Kg)		
		98.2	102			128.2	RECOVERY (%)		
15	50	50	30		50	50	SAMPLE WEIGHT (g)		
6	3	6	1		2	6	EXTRACTION SOLVENT		
GPC	SPE	SPE	GPC		GPC	GPC	CLEAN-UP STEP		
YES							BROMOPHOS-ETHYL	INTERNAL STANDARD	
1	1	1	20 mL/20 mg		1	2	INJECTION VOLUME (µL)		
SPLITLESS	ON COLUMN	SPLITLESS	MANUAL		OC; SS	SPLITLESS	INJECTION TYPE		
GC-ECD, GC-NPD, GC-MS(TID)	GC-ECD GC-PFPD GC-MS		HPLC-UV		GC-ECD, GC-NPD	GC-ECD AND GC-NPD	DETERMINATION		
RAPPORTO ISTISAN 97/23	NFEN12398			SOP MR 406012; EXTRACTION OF RESIDUES OF PLANT-PROTECTING MATERIAL FROM FRUIT AND VEGETABLE SAMPLES BY ETHYL-ACETATE; SOP 308M2; CLEAN UP OF EXTRACTS BY GEL PERMEATION CHROMATOGRAPHY	\$35 LMBG; L 00. 00-34, L 00. 00-37	MULTI-RESIDUE-METHOD DFG S 19	REFERENCE METHOD		

**APPENDIX 7. Methods used by participants for determining pesticides.**

FLUDIOXONIL												
128	127	126	125	124	123	122	121	120	119	118	117	LAB CODE
NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	SCOPE OF YOUR METHOD
												CONFIRMATION METHOD
												RL (mg/Kg)
												RECOVERY (%)
												SAMPLE WEIGHT (g)
												EXTRACTION SOLVENT
												CLEAN-UP STEP
												INTERNAL STANDARD
												INJECTION VOLUME (µL)
												INJECTION TYPE
												DETERMINATION
												REFERENCE METHOD

NO RESULTS

**APPENDIX 7. Methods used by participants for determining pesticides.**

IMIDACLOPRID										
								LAB CODE		
								SCOPE OF YOUR METHOD		
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		
								M		
D	NA	NA	D	D	NA	D	D	D	D	
M		M		M		M		M		
LC-MS/MS		LC-MS/MS			LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	CONFIRMATION METHOD	
0.01		0.005		0.003		0.010		0.05	RL (mg/Kg)	
104						97			RECOVERY (%)	
20		10		10		10		10	SAMPLE WEIGHT (g)	
6		5		5		6		1	EXTRACTION SOLVENT	
LLE		SPE		SPE		LLE			CLEAN-UP STEP	
								YES	INTERNAL STANDARD	
10		20		10		5		20	INJECTION VOLUME (µL)	
								N/A	INJECTION TYPE	
LC-MS/MS		LC-MS/MS			LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	DETERMINATION	
					KLEIN, J., ALDER, L. (2003); APPLICABILITY OF GRADIENT LIQUID CHROMATOGRAPHY WITH TANDEM MASS SPECTROMETRY TO THE SIMULTANEOUS SCREENING FOR ABOUT 100 PESTICIDES IN CROPS. J. AOAC INT. 86 (5): 1015-1037					REFERENCE METHOD
				MANASTASSIADES ET AL. J.AOAC 86, 412-431 (2003)	QUECHERS; ANASTASSIADES				IN HOUSE	

**APPENDIX 7. Methods used by participants for determining pesticides.**

IMIDACLOPRID									
18	17	16	15	14	13	12	11	10	9
NA	NA	D	NA	D	M	M	S	S	LAB CODE
		S							SCOPE OF YOUR METHOD
									QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
									CONFIRMATION METHOD
									RL (mg/Kg)
									RECOVERY (%)
									SAMPLE WEIGHT (g)
									EXTRACTION SOLVENT
									LLE ACETONE FOLLOWED BY DICHLOROMETHANE AND PETROLEUM ETHER
									CLEAN-UP STEP
									INTERNAL STANDARD
									INJECTION VOLUME (µL)
									INJECTION TYPE
									DETERMINATION
									REFERENCE METHOD
									IN HOUSE SOP
									LOCAL SOP
									NO. MR405012

**APPENDIX 7. Methods used by participants for determining pesticides.**

IMIDACLOPRID									
26	25	24	23	22	21	20	19	LAB CODE	SCOPE OF YOUR METHOD
D	D	D	D	D	NA	NA	NA	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
M	M	S	M	M				M	
LC-MS/MS	LC-MS/MS	LC-MS/MS	HPLC-DAD	LC-MS/MS				LC-MS/MS	CONFIRMATION METHOD
0.01	<0.01	0.05	0.02	0.05				0.01	RL (mg/Kg)
84	85	90		98					RECOVERY (%)
10	5	30	5	10				75	SAMPLE WEIGHT (g)
6 METHANOL 95% AMMONIACETATE 5%, 20 mM ACETIC ACID	5	1	5	METHANOL					EXTRACTION SOLVENT
									CLEAN-UP STEP
				SPE FLORISIL					
CARBARYL C13									INTERNAL STANDARD
10	10	1 mg SAMPLE	20	5				5	INJECTION VOLUME (µL)
SPLITLESS		AUTOSAMPLER	AUTOMATIC						INJECTION TYPE
LC-MS/MS	LC-MS/MS	HPLC-DAD	LC-MS/MS					LC-MS/MS	DETERMINATION
									REFERENCE METHOD
				SOP NO.: MR 405012.M1	INTERNAL METHOD				

**APPENDIX 7. Methods used by participants for determining pesticides.**

IMIDACLOPRID									
									LAB CODE
									SCOPE OF YOUR METHOD
37	36	35	34	33	32	31	30	29	28
D	D	NA	NA	NA	NA	NA	D	D	D
M	S					M	S	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
LC-MS/MS	LC-MS					LC-MS/MS	LC-MS/MS		CONFIRMATION METHOD
0.005	0.05					0.05	0.04	0.05	RL (mg/Kg)
	87					122	89		RECOVERY (%)
10	25					15	20	30	SAMPLE WEIGHT (g)
5	3					5	3	1	EXTRACTION SOLVENT
O	SPE								CLEAN-UP STEP
DIMETHOATE D6									INTERNAL STANDARD
	20					50	20	20	INJECTION VOLUME (µL)
		SPLIT/SPLITLESS							INJECTION TYPE
LC-MS/MS (TRIPLE-QUAD)									RHEODYNE INJECTOR
ANASTASSIADES, JOURNAL OF AOAC INT. VOLT 86 NO 2, 2003									DETERMINATION
									HUNGARIAN CENTRAL SOP NO.: 405012 M1
									REFERENCE METHOD
									QUECHERS

**APPENDIX 7. Methods used by participants for determining pesticides.**

IMIDACLOPRID									
45	44	43	42	41	40	39	38	LAB CODE	SCOPE OF YOUR METHOD
D	D	D	D	NA	NA	D	NA		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
S	M	M	M			M			CONFIRMATION METHOD
		NONE	LC-MS/MS		LC-MS/MS				RL (mg/Kg)
0.04	0.01	0.01	0.01	0.01	0.01	0.01			RECOVERY (%)
77		91		111		87			SAMPLE WEIGHT (g)
25	50	10	10	10	10	10			EXTRACTION SOLVENT
1 (IN PRESENCE OF NaOH)	2	5	5	5	5	6 (METHANOL/WATER)			CLEAN-UP STEP
GPC	LL	SPE	LL			SPE			INTERNAL STANDARD
									INJECTION VOLUME (µL)
RHEODYNE									INJECTION TYPE
HPLC - DAD	LC-MS/MS	LC-MS/MS(ESI+)		LC-MS/MS		LC-MS/MS			DETERMINATION
CLEAN UP AS IN EN 12393 P METHOD	HOUSE METHOD	ANASTASSIADES ET AL., J. AOAC INT. 86(2), 412-431, MODIFIED		M. ANASTASSIADES ET AL., J. AOAC INT. 86(2), 412-431, (2003)		LC-MS/MS (BFR)			REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

IMIDACLOPRID									
									SCOPE OF YOUR METHOD
									QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
54	53	52	51	50	49	48	47	46	LAB CODE
D	D	NA	NA	D	D	D	NA	NA	SCOPE OF YOUR METHOD
S	S			S	S	M			QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
LC-MS/MS	HPLC-DAD	LC-MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS			CONFIRMATION METHOD
0.005	0.05		0.05	0.01	0.01	0.01			RL (mg/Kg)
118			95						RECOVERY (%)
5	15		10	10	10	10			SAMPLE WEIGHT (g)
1	6		4	6	6	6			EXTRACTION SOLVENT
									CLEAN-UP STEP
				LLE	SPE				
		GPC							
									INTERNAL STANDARD
									INJECTION VOLUME (µL)
					5	20	20		
5	1								INJECTION TYPE
			SPLIT/ SPLITLESS			FULL LOOP			
									DETERMINATION
				GC-ECD, GC-NPD, GC-FPD, GC-MS, HPLC-FLD, HPLC-UV	LC-MS/MS	LC-MS/MS			
JOURNAL OF CHROMATOGRAPHY A,1023(2004)93-104	INTERNAL METHOD						J. KLEIN, L.ALDER, JOURNAL OF AOAC INTERNATIONAL VOL 86, NO 5, 2003	KLEIN, J.; ALDER, L; JOURNAL OF AOAC INT, 2003, 86, 1015- 1037	REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

IMIDACLOPRID									
								SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
								CONFIRMATION METHOD	
								LC-MS/MS	
D	NA	D	D	D	D	D	D	RL (mg/Kg)	0.01
M	M	M	M	M	M	S	M	RECOVERY (%)	74
LC-MS/MS	GC-MS	HPLC-UV	LC-MS/MS	LC-MS/MS	LC-MS/MS	—	LC-MS/MS	SAMPLE WEIGHT (g)	100
0.05	0.01	0.05	0.05	0.05	0.01	0.454	0.01	EXTRACTION SOLVENT	
72			869	107		89.2		CLEAN-UP STEP	
10	20	5	50	10	25	30	GPC	INTERNAL STANDARD	
METHANOL	METHANOL	1	5	6	5	1	GC 1 µL HPLC 20 µL	INJECTION VOLUME (µL)	
			O	LLE ON CHEM ELUT			LOOP	SPLIT/ SPLITLESS	
				3				INJECTION TYPE	
20	10	20	2	100	5	20			
LC-MS/MS	LC-MS/MS	HPLC-FLD OR DAD	LC-MS/MS	LC-MS/MS	HPLC-UV		GC-ECD, GC-NPD, GC-FPD, HPI C-UV LC-MS/MS	DETERMINATION	
			METODENVORSC HLAG EG- PROFICIENCY TEST 1996/97	APPL. OF LC-MS/MS (J. KLEIN, L. ALDERS)			MODULARE MULTIMETHOD E S19	REFERENCE METHOD	
MULTI RESIDUE METHOD DRAFT BFR									

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>IMIDACLOPRID</b>									
74	73	72	71	70	69	68	67	66	65
NA	D	NA	NA	NA	D	D	NA	NA	NA
S					S	M			
HPLC-UV					LC-MS/MS				
0.05					0.01	0.02			
84					88.8	80			
30					20	10			
1					4	5			
GPC; O					LLE + SPE				
RHEODYNE					TRIPHENYLPHOSPHATE				
HPLC-UV					5	10			
LOCAL SOP					LOOP				
					LC-MS/MS				
					MINI LUKE EXTRACTION				
					ANASTASSIADES M. ET AL., J. AOAC INT., 86 (2003), 412 – 431 QUECHERS - METHOD				
									REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

IMIDACLOPRID											
85	84	83	82	81	80	79	78	77	76	75	LAB CODE
NA	NA	D	NA		D	D	ND		NA	D	SCOPE OF YOUR METHOD
		M		M	M	M	M		M		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
		LC-MS/MS		LC-MS	GC-MS						CONFIRMATION METHOD
		0.05		0.05					0.01		RL (mg/Kg)
		101			77				96		RECOVERY (%)
		10		10	15	15			10		SAMPLE WEIGHT (g)
		5		6	6	1			6		EXTRACTION SOLVENT
		O									CLEAN-UP STEP
											LLE (CHEMELUT)
											INTERNAL STANDARD
											INJECTION VOLUME (µL)
											INJECTION TYPE
											DETERMINATION
											REFERENCE METHOD

NO RESULTS

NO RESULTS

**APPENDIX 7. Methods used by participants for determining pesticides.**

IMIDACLOPRID									
								LAB CODE	
								SCOPE OF YOUR METHOD	
		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX							
CONFIRMATION METHOD									
93	92	91	90	89	88	87	86	LAB CODE	
D	D	D	D	D	D	D	D	SCOPE OF YOUR METHOD	
M	M	M	S	M	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	HPLC-DAD			LC-MS/MS	CONFIRMATION METHOD	
0.01	0.01	0.006	0.05	0.04	0.01		0.025	RL (mg/Kg)	
	87			85	116	93	95	RECOVERY (%)	
10	50	10	50	15	5	10	7.5	SAMPLE WEIGHT (g)	
6. METHANOL	4	6 METHANOL	3	6	5	6 METHANOL	4	EXTRACTION SOLVENT	
								CLEAN-UP STEP	
LLE	LLE	SPE	LLE	LLE	LLE	LLE		INTERNAL STANDARD	
								INJECTION VOLUME (µL)	
								INJECTION TYPE	
20	10	20	OCT-25	1	10	20	10	FULL LOOP	
SAMPLE-LOOP	LOOP			SPLIT/SPLITLESS				DETERMINATION	
LC-MS/MS	LC-MS/MS	LC-MS/MS	HPLC-DAD	GC-ECD FPD, MS HPLC-F-DAD	LC-MS/MS	LC-MS/MS	LC-MS/MS	DETERMINATION	
MULTI-METHOD	LC-MULTIRESIDUE	DRAFT § 35 LMBG-METHOD				IN-HOUSE	JAOAC 86, 5, 1015-1037	REFERENCE METHOD	

**APPENDIX 7. Methods used by participants for determining pesticides.**

IMIDACLOPRID										
103	102	101	100	99	98	97	96	95	94	LAB CODE
NA	D	NA	NA	NA	D	NA	NA	NA	NA	SCOPE OF YOUR METHOD
	S				M					QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
	HPLC-DAD				LC-MS/MS					CONFIRMATION METHOD
		0.05			0.05					RL (mg/Kg)
		99			109					RECOVERY (%)
		30			10					SAMPLE WEIGHT (g)
		1			1					EXTRACTION SOLVENT
										CLEAN-UP STEP
										INTERNAL STANDARD
						CARBENDAZIM D4, METHOMYL D3, PENDIMETHALIN D5				INJECTION VOLUME (µL)
							2	10		INJECTION TYPE
							SPLITLESS			DETERMINATION
										REFERENCE METHOD
										SOP MR 405012; EXTRACTION OF RESIDUES OF PLANT-PROTECTING MATERIAL FROM FRUIT AND VEGETABLE SAMPLES BY ETHYL-ACETATE
										LC-MS MULTI RESIDUE

## **APPENDIX 7. Methods used by participants for determining pesticides.**

IMIDACLOPRID													
111		110		109		108		107		106	105	104	LAB CODE
D	D	NA	NA	M	M	D	NA	D	NA	D	D	D	SCOPE OF YOUR METHOD
S	S									S	S	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
LC-MS/MS	LC-MS/MS			LC-MS	LC-MS/MS			LC-MS/MS	LC-MS/MS				CONFIRMATION METHOD
0.05	0.04			0.02	0.02			0.02	0.005				RL (mg/Kg)
99.3				78	77			77					RECOVERY (%)
50	10			10	15			15	10				SAMPLE WEIGHT (g)
6 METHANOL	6 METHANOL			5	4			4	6 (METHANOL)				EXTRACTION SOLVENT
SPE; LLE	O			O					DIATOMACEOUS EARTH				CLEAN-UP STEP
50	20			1	10			10					INTERNAL STANDARD
HPCL-UV	HPCL-UV			AUTOSAMPLER						25			INJECTION VOLUME (µL)
				HPLC-MS (SINGLE-QUAD)	LC-MS/MS								INJECTION TYPE
													DETERMINATION
				QUECHERS ANASTASSIADES	DE KOK								REFERENCE METHOD
													APPLICATION NOTE BY WATERS
VALIDATED INHOUSE METHOD NACH PFLANZENSCHUTZ NACHICHTEN BAYER BD. 46, 1993, 2 PLÄCKE U. WEBER		MULTI-RESIDUE-METHOD HPLC											

## **APPENDIX 7. Methods used by participants for determining pesticides.**

IMIDACLOPRID									
120	119	118	117	116	115	114	113	112	LAB CODE
NA	NA	NA	NA	NA	NA	D	D	D	SCOPE OF YOUR METHOD
					S	S	S	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
						HPLC-UV			CONFIRMATION METHOD
					0.01	0.05	0.05	RL (mg/Kg)	
					94.5	88	100	RECOVERY (%)	
					20	30	10	SAMPLE WEIGHT (g)	
					6	1	6	EXTRACTION SOLVENT	
								CLEAN-UP STEP	
						SPE	SPE		
						GPC			
								INTERNAL STANDARD	
							20 mL/20 mg	INJECTION VOLUME (µL)	
							30		
								MANUAL	INJECTION TYPE
									DETERMINATION
									REFERENCE METHOD
									ISTISAN 97/23
									SOP MR 405012; EXTRACTION OF RESIDUES OF PLANT-PROTECTING MATERIAL FROM FRUIT AND VEGETABLE SAMPLES BY ETHYL-ACETATE; SOP 308M2; CLEAN UP OF EXTRACTS BY GEL PERMEATION CHROMATOGRAPHY
									WATERS A MULTRESIDUE LC-MS/MS METHOD FOR PESTICIDES

**APPENDIX 7. Methods used by participants for determining pesticides.**

IMIDACLOPRID									
128	127	126	125	124	123	122	121	LAB CODE	SCOPE OF YOUR METHOD
NA		NA	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX						
									CONFIRMATION METHOD
									RL (mg/Kg)
									RECOVERY (%)
									SAMPLE WEIGHT (g)
									EXTRACTION SOLVENT
									CLEAN-UP STEP
									INTERNAL STANDARD
									INJECTION VOLUME (µL)
									INJECTION TYPE
									DETERMINATION
									REFERENCE METHOD

NO RESULTS

**APPENDIX 7. Methods used by participants for determining pesticides.**

IPRODIONE									
5	4	3	2	1	LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	CONFIRMATION METHOD	REFERENCE METHOD
D	D	D	D	D		D	M	GC-MS	
M	M	M	S	S		M			
GC-MS	GC-ECD	GC-MS/MS	GC-MS	GC-MS		GC-MS			
0.02	0.013	0.01	0.020	0.02		RL (mg/Kg)			
90.9		74.0	94			RECOVERY (%)			
10	10	15	15	10		SAMPLE WEIGHT (g)			
5	5	1	4	1		EXTRACTION SOLVENT			
SPE	SPE	NO				CLEAN-UP STEP			
MIREX/TPP		TRIPHENYLPHOSPHATE ANTHRACENTE				INTERNAL STANDARD	YES		
2	2	10	1	1		INJECTION VOLUME (µL)	2		
SPLIT/SPLITLESS	SPLIT/SPLITLESS	SPLIT	SPLITLESS	SPLITLESS		INJECTION TYPE			
GC-ECD GC-NPD	GC-MS	GC-MS/MS	GC-NPD	GC-MS		DETERMINATION			
MANASTASIADES ET AL. JAOAC 86, 412-431 (2003)	QUECHERS; ANASTASIADES		LUKE, M.A., FROBERG, J.E., DOOSE, G.M., MASUMOTO, H.T. (1981). IMPROVED MULTIRESIDUE GAS CHROMATOGRAPHIC DETERMINATION OF ORGANOPHOSPHORUS, ORGANONITROGEN, AND ORGANOHALOGEN PESTICIDES IN PRODUCE USING FLAME PHOTOMETRIC AND ELECTROLYTIC CONDUCTIVITY DETECTORS. J. ASSOC. OFF. ANAL. CHEM. 64(5): 1187-1195			IN HOUSE			

## **APPENDIX 7. Methods used by participants for determining pesticides.**

IPRODIONE									
LAB CODE		SCOPE OF YOUR METHOD		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		CONFIRMATION METHOD		REFERENCE METHOD	
12	11	10	9	8	7	6			
D	D	D	D	D	D	D	D	D	D
M	S	M	M	M	S	M	M	M	M
MS-ITD, GC-ECD	GC-NPD	GC-MS(ITT)	GC-MS	GC-ECD	GC-MS	GC-ECD	GC-MS	ANALYTICAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS (1996), MINISTRY OF HEALTH, WELFARE AND SPORTS, THE HAGUE (RJSWJK), THE NETHERLANDS	ANALYTICAL METHODS FOR PESTICIDE RESIDUES MINISTRY OF HEALTH NETHERLANDS 6TH ED
0.01	0.02	0.02	0.025	0.01	0.01	0.01	0.02	RL (mg/Kg)	RL (mg/Kg)
88	80.6	88	103	81	81	81	81	RECOVERY (%)	RECOVERY (%)
50	30	25	15	30	10	15	15	SAMPLE WEIGHT (g)	SAMPLE WEIGHT (g)
1	1	2	4	1	6 (DICHLORMETHANE)	4	4	EXTRACTION SOLVENT	EXTRACTION SOLVENT
GPC		LLE ACETONE FOLLOWED BY DICHLORMETHANE AND PETROLEUM ETHER		O		GPC		CLEAN-UP STEP	
SPLITLESS		SSU/PTV		PTV		SPLITLESS		SPLITLESS	
1,2,5	1	2	2	2	2	4	4	2	2
SPLITLESS		SPLIT/ SPLITLESS		SPLITLESS		SPLITLESS		INJECTION TYPE	
MS-ITD, LC-MS, GC-FPD, GC-ECD		GC-MS(ITT)		GC-MS		GC-MS		DETERMINATION	
GC-MS (ION TRAP)		GC-ECD, GC-NPD, GC-MS(ITT)		GC-MS(ITT)		GC-MS		RAPPORT ISTITUSAN 97/23	

**APPENDIX 7. Methods used by participants for determining pesticides.**

IPRODIONE									
19	18	17	16	15	14	13	LAB CODE	SCOPE OF YOUR METHOD	
D	D	D	D	D	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
M	S	M	M	S	S	M	M	CONFIRMATION METHOD	
GC-MS(FTD)	GC-MS	GC-MS	GC-MS/MS	GC-MS	GC-MS	GC-MS/MS	GC-MS/MS	RL (mg/Kg)	
0.02	0.03	0.01	0.02	0.02	0.02	0.02	0.02	RECOVERY (%)	
73	94			107		83.5		SAMPLE WEIGHT (g)	
75	75	15	15	25	15	15	15	EXTRACTION SOLVENT	
1	1	4	4	1	4	1	1	CLEAN-UP STEP	
GPC	GPC	LLE		GPC				INTERNAL STANDARD	
DITALIMPHOS	ALDRIN						TPP (QC)	INJECTION VOLUME (µL)	
2	2	10	10	2	2	2	10	INJECTION TYPE	
SPLITLESS	PULSED SPLITLESS	LVI AUTOSAMPLER	LVI SPLITLESS	SPLIT/ SPLITLESS				GC-ECD GC-TSD GC-MS HPLC-UV	
GC-ECD	GC-MS	GC-MS	GC-MS/MS	GC-ECD			GC-MS(ITD)	DETERMINATION	
								REFERENCE METHOD	
								LOCAL SOP NO. MR405012	
								MULTIRESIDUES	
								MINI LUKE	

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>IPRODIONE</b>									
26	25	24	23	22	21	20	LAB CODE		
D	D	D	D	D	D	D	SCOPE OF YOUR METHOD		
M	M	M	M	M	M	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		
GC-ECD	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS/MS	GC-NPD	CONFIRMATION METHOD		
0.01	<0.02	0.05	0.03	0.1	0.01	0.02	RL (mg/Kg)		
79	96	90.2		101.6		93	RECOVERY (%)		
25	10	30	5	50	10	100	SAMPLE WEIGHT (g)		
2	6	1	5	6	1	3	EXTRACTION SOLVENT		
				LLE (ISOPROPYL ETHER)		O	CLEAN-UP STEP		
GPC	SPE	GPC					INTERNAL STANDARD		
DITALIMPHOS	YES		FENCHLORPHOS				INJECTION VOLUME (µL)		
2	2	1 mg SAMPLE	50	2	10	1	AUTOSAMPLER		
SPLITLESS	SPLITLESS	TPOCI	AUTOMATIC TDS SAMPLER	SPLITLESS	LVI		INJECTION TYPE		
GC-ECD, GC-NPD, GC-MS(TD)	GC-MS	GC-ECD	TDS-IN LINE WITH GC-MS (SINGLE-QUAD) TOTAL ION DETECTION	GC-ECD AND GC-NPD	GC-MS/MS	GC-ECD	DETERMINATION		
			SOP NO : MR 405012 M1; 308 M2	INTERNAL METHOD			REFERENCE METHOD		

**APPENDIX 7. Methods used by participants for determining pesticides.**

IPRODIONE									
								LAB CODE	
								SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
								S	
34	D	33	32	31	30	29	28	27	LAB CODE
M	M	D	D	D	D	D	D	D	SCOPE OF YOUR METHOD
GC-MS/MS	GC-MS/MS	GC-MS	MS	S	M	S	S	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
0.02	0.01	0.02	0.02	0.01	0.02	0.02	0.02	0.02	CONFIRMATION METHOD
5	15	101	92	95	114	99	99	99	RL (mg/Kg)
4	1	25	10	10	15	50	30	30	RECOVERY (%)
									SAMPLE WEIGHT (g)
									EXTRACTION SOLVENT
									CLEAN-UP STEP
									INTERNAL STANDARD
									INJECTION VOLUME (µL)
									INJECTION TYPE
									DETERMINATION
									REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

IPRODIONE									
								LAB CODE	
								SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
								CONFIRMATION METHOD	
40	D	39	38	37		36		35	
M	M	M	M	M	S	S	M	D	D
GC-MS	LC-MS/MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	RL (mg/Kg)	SCOPE OF YOUR METHOD
0.02	0.01	0.02	0.02	0.02	0.02	0.02	0.02	0.02	GENERAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS. 6TH ED. 1996, GENERAL INSPECTORATE FOR HEALTH PROTECTION, MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS
15	10	82	10	10	90	90	95	RECOVERY (%)	GENERAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS. 6TH ED. 1996, GENERAL INSPECTORATE FOR HEALTH PROTECTION, MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS
4	6 (METHANOL/WATER)	5	5	5	25	25	20	SAMPLE WEIGHT (g)	GENERAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS. 6TH ED. 1996, GENERAL INSPECTORATE FOR HEALTH PROTECTION, MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS
					3	3	3	EXTRACTION SOLVENT	GENERAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS. 6TH ED. 1996, GENERAL INSPECTORATE FOR HEALTH PROTECTION, MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS
								CLEAN-UP STEP	GENERAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS. 6TH ED. 1996, GENERAL INSPECTORATE FOR HEALTH PROTECTION, MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS
								INTERNAL STANDARD	GENERAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS. 6TH ED. 1996, GENERAL INSPECTORATE FOR HEALTH PROTECTION, MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS
								INJECTION VOLUME (µL)	GENERAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS. 6TH ED. 1996, GENERAL INSPECTORATE FOR HEALTH PROTECTION, MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS
								INJECTION TYPE	GENERAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS. 6TH ED. 1996, GENERAL INSPECTORATE FOR HEALTH PROTECTION, MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS
								DETERMINATION	GENERAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS. 6TH ED. 1996, GENERAL INSPECTORATE FOR HEALTH PROTECTION, MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS
								REFERENCE METHOD	GENERAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS. 6TH ED. 1996, GENERAL INSPECTORATE FOR HEALTH PROTECTION, MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS
									GENERAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS. 6TH ED. 1996, GENERAL INSPECTORATE FOR HEALTH PROTECTION, MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>IPRODIONE</b>									
<b>46</b>	<b>45</b>	<b>44</b>	<b>43</b>	<b>42</b>	<b>41</b>	<b>LAB CODE</b>			
D	D	D	D	D	D	D	D	D	<b>SCOPE OF YOUR METHOD</b>
M	M	M	M	M	M	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-NPD	GC-MS		LC-MS/MS		GC-MS	GC-MS			CONFIRMATION METHOD
0.02	0.07	0.01	0.02	0.01	0.02	RL (mg/Kg)			RECOVERY (%)
76	120		95		95.9				SAMPLE WEIGHT (g)
15	25	50	10	10	10	20			EXTRACTION SOLVENT
4	1	2	5	5	5	3			CLEAN-UP STEP
(PULSED) SPLITLESS/(TRACK OVEN) ON COLUMN	GPC	GPC	SPE	LLE					INTERNAL STANDARD
			PCB 138, TRIPHENYLPHOSPHATE (TPP)						
3 / 1	1	2	4	1	1	4			INJECTION VOLUME (µL)
	SPLIT/SPLITLESS (SPLITLESS MODE)	PTV	PTV	PTV	PTV	SPLITLESS			INJECTION TYPE
GC-NPD / GC-ECD	GC-ECD OR GC-NPD OR GC-FPD	GC-MS (SINGLE QUAD)	GC-MS EI		GC-ECD, GC-MS	GC-ECD, GC-NPD			DETERMINATION
MULTI-RESIDUE METHOD PESTICIDES AMENABLE TO GAS CHROMATOGRAPHY (3:12 ACETONE-PARTITION FOR FRUITS, VEGETABLES AND POTATOES) 6TH EDITION MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORT, THE NETHERLANDS	EN 12393 P METHOD	§ 35 LMBG 00/00-34	ANASTASSIADES ET AL., JAOAC INT. 86 (2), 412-431, (2003)						REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

IPRODIONE									
								LAB CODE	
								SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
								CONFIRMATION METHOD	
54	D	53	D	52	51	50	49	48	47
M	M	S	M	S	S	M	S	D	D
GC-MS	GC-MS	GC-ECD	GC-MS	GC-ECD	GC-MS	GC-ECD	GC-ECD	RL (mg/Kg)	RECOVERY (%)
0.03	0.02	0.02	0.1	0.01	0.01	0.01	0.02	0.02	0.02
87	18	15	10	113	113	113	113	113	113
1	1	6	5	20	20	50	25	10	10
				6	1	2	2	5	5
GPC	GPC	O		LLE	GPC	GPC	SPE	CLEAN-UP STEP	
				NITROFEN				INTERNAL STANDARD	
1	1	1	1.5	2	2	2	3	INJECTION VOLUME (µL)	
SPLITLESS, ON COLUMN	SPLIT/ SPLITLESS	SPLIT/ SPLITLESS	ON COLUMN	SPLITLESS	PTV	SPLIT/SPLITLESS	PTV	INJECTION TYPE	
GC-ECD, FPD,NPD	-ECD, GC-NPD, GC-FPD, GC-MS, HPLC-FL, HPLC-UV	GC-ECD, GC-NPD, GC-MS		GC-FPD, GC-NPD, GC-MS(UD)	GC-MS	GC-ECD, GC-NPD, GC-MS (SINGLE QUAD)		DETERMINATION	
EN 12393-1,2,3	INTERNAL METHOD	QUECHERS	METHOD I <sup>R</sup> EN 12393-2:1999	DFG S 19	M. ANASTASSIADES, S. J. LEHOTAY, D. STAIBNAIER, F. J. SCHENCK-J. AOAC INT. 86 (2003) 412-431	M. ANASTASSIADES, S. J. LEHOTAY, D. STAIBNAIER, F. J. SCHENCK-J. AOAC INT. 86 (2003) 412-431		REFERENCE METHOD	

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>IPRODIONE</b>									
									LAB CODE
62	61	60	59	58	57	56	55		
D	D	NA	D	D	D	D	D	D	SCOPE OF YOUR METHOD
M	M	M	M	M	S	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-MS	GC-MS	GC-MS	GC-ECD-NPD	GC-MS	GC-ECD	GC-ECD	GC-ECD	GC-ECD	CONFIRMATION METHOD
0.1	0.02	0.02	0.02	0.01	0.356	0.01	0.01	0.01	RL (mg/Kg)
10	50	95.1	113		80	83.8			RECOVERY (%)
1	3	10	15	25	30	100			SAMPLE WEIGHT (g)
		5	4	1	1	2			EXTRACTION SOLVENT
		GPC	O	GPC	GPC	GPC	GPC	GPC	CLEAN-UP STEP
TPP		3	PCB119 (ECD) TRIFLURALIN (GC-NPD)						INTERNAL STANDARD
2	2	2	2	1	1	1	GC 1 µL HPLC 20 µL		INJECTION VOLUME (µL)
PTV		SPLITLESS	SPLIT/SPLITLESS	PULSED SPLITLESS	DIRECT	SPLIT/SPLITLESS			INJECTION TYPE
GC-MS	GC-MS	GC-MS	GC-ECD/NPD	GC-ECD/NPD	GC-NPD	GC-ECD, GC-NPD, GC-FPD, HPLC-UV LC-MS/MS	GC-ECD, GC-NPD, GC-FPD, HPLC-UV LC-MS/MS	DETERMINATION	
	DFG S19 (ASU §35 LMBGL 00.00-34)		ANAL METHODS FOR PESTICIDES RESIDUES IN FOODSTUFFS (NL)			MODULARE MULTIMETHODE S19		REFERENCE METHOD	

## **APPENDIX 7. Methods used by participants for determining pesticides.**

IPRODIONE									
LAB CODE		SCOPE OF YOUR METHOD		CONFIRMATION METHOD		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		REFERENCE METHOD	
67	D	66	D	65	D	64	D	63	D
M	M	M	M	M	M	S	S	S	S
GC-MS	GC-MS/MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-NPD GC-ECD	GC-NPD GC-ECD
0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	RL (mg/kg)	RL (mg/kg)
110	110	81	90	90	90	92	92	RECOVERY (%)	RECOVERY (%)
90	15	5	5	5	5	75	75	SAMPLE WEIGHT (g)	SAMPLE WEIGHT (g)
3	5	1	6	6	6	ACETONE FOLLOWED BY CYCLOHEXANE AND ETHYLACETATE ACETONE FOLLOWED BY CYCLOHEXANE AND ETHYLACETATE	ACETONE FOLLOWED BY CYCLOHEXANE AND ETHYLACETATE ACETONE FOLLOWED BY CYCLOHEXANE AND ETHYLACETATE	EXTRACTION SOLVENT	EXTRACTION SOLVENT
GPC	SPE	GPC	GPC	GPC	GPC	GPC	GPC	CLEAN-UP STEP	CLEAN-UP STEP
PARATHION	AZOBENZENE+ RONNEL+ TRIPHENYLPHOSPHATE	TPP	TPP	TPP	TPP	TPP	TPP	INTERNAL STANDARD	INTERNAL STANDARD
1	10	2	2	0.5	0.5	1	1	INJECTION VOLUME (µL)	INJECTION VOLUME (µL)
SPLIT/SPLITLESS	ON COLUMN	SPLITLESS	SPLITLESS	ON COLUMN	ON COLUMN	SPLITLESS	SPLITLESS	INJECTION TYPE	INJECTION TYPE
GC - NPD	GC-MS(TD)	GC-(ECD+FPD+NPD)	GC-MS	GC-MS	GC-MS	GC-NPD GC-ECD	GC-NPD GC-ECD	DETERMINATION	DETERMINATION
OFFICIAL METHOD L 00.00-34; § 35 LMBG 615-629	QUECHERSLEOTHAY, S. ET AL. JAOAC INT. (2005) 88, 615-629	INTERNAL METHOD	INTERNAL METHOD	MULTIRESIDUE WITH EXTRELTU	MULTIRESIDUE WITH EXTRELTU	§ 35 LMBG L 00.00-34	§ 35 LMBG L 00.00-34	REFERENCE METHOD	REFERENCE METHOD

## **APPENDIX 7. Methods used by participants for determining pesticides.**

IPRODIONE													
73		72		71		70		69		68	LAB CODE		
D		D		D		D		D		SCOPE OF YOUR METHOD			
S		S		M		M		M		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX			
GC-MS	GC-ECD	GC-ECD	GC-ECD	GC-MS/MS	GC-MS/MS	GC-M/SIMS	GC-M/SIMS	GC-MS	GC-MS	CONFIRMATION METHOD			
0.05	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	RL (mg/Kg)			
89	94	83	83	97	97	97	97	94.4	94.4	RECOVERY (%)			
30	100	25	25	15	15	25	25	20	20	SAMPLE WEIGHT (g)			
1	3	6	6	5	5	1	1	4	4	EXTRACTION SOLVENT			
CLEAN-UP STEP													
GPC													
INTERNAL STANDARD													
1	1	2	2	10	10	1,0-2,0	1,0-2,0	2	2	INJECTION VOLUME (µL)			
INJECTION TYPE													
SPLIT/SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	LVI	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	DETERMINATION			
GC-MS (ION TRAP)	GC-ECD GC-NPD	GC-ECD	GC-ECD	GC-ECD/TSD/PFPD GC-M/SIMS	GC-NPD, GC-ECD	GC-ECD/NPD	GC-ECD/NPD	GC-ECD/NPD	GC-ECD/NPD	REFERENCE METHOD			
LOCAL SOP	MULTIRESIDUE METHOD FOR ANALYSIS OF PESTICIDES IN FOODSTUFFS P.A. GREVE ED, 1988, MINISTRY OF WELFARE, HEALTH AND CULTURAL AFFAIR, RUISWICK NETHERLANDS										MINI-LUKE EXTRACITION		

**APPENDIX 7. Methods used by participants for determining pesticides.**

IPRODIONE									
81	80	79	78	77	76	75	74	LAB CODE	SCOPE OF YOUR METHOD
M	D	D	D	D	D	D	D		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	LC-MS/MS	GC-MS	GC-MS		CONFIRMATION METHOD
0.03	0.02			0.05	0.01	0.005			RL (mg/Kg)
50	70	90	95	96	96	90.1			RECOVERY (%)
2	15	15	10	50	50	25			SAMPLE WEIGHT (g)
	DICHLOROMETHANE		2	3					EXTRACTION SOLVENT
NO RESULTS									
8	SPE	GPC			HEXBROMOBENZENE				CLEAN-UP STEP
5	2	1			TPP				INTERNAL STANDARD
LVI	SPLITLESS	SPLITLESS							INJECTION VOLUME (µL)
GC-MS	GC-ECID GC-NPD GC-MS LC-MS/MS HPLC-FLD	GC-MS (SINGLE-QUAD)	GC-NPD	GC-MS	GC-MS	GC-MS			DETERMINATION
NO RESULTS									
L-00-00-34	INTERNAL METHOD								REFERENCE METHOD
§35 LMBGL 00.00-34 (1999)									

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>IIPRODIONE</b>							
							LAB CODE
87	D	86	D	85	D	84	83
S	M	M	M	M	M	M	D
LC-MS/MS	GC-MS(TD)	GC-MS	GC-MS	GC-MS	GC-ECD	GC-ECD	SCOPE OF YOUR METHOD
0.025	0.05	0.04	0.04	0.02	0.006	RL (mg/Kg)	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
78	80	80	80	107	115	RECOVERY (%)	CONFIRMATION METHOD
50	15	25	50	30	10	SAMPLE WEIGHT (g)	
6 ETHYLACETATE/DICHLOROMETHANE (80/20)	4	4	2	1	5	EXTRACTION SOLVENT	
SPE-FLORISIL						CLEAN-UP STEP	
HCB						TRIPHENYL ETHYLENE	INTERNAL STANDARD
1	1	1	1	3	1	INJECTION VOLUME (µL)	
SPLIT/ SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS AND ON COLUMN		INJECTION TYPE	
GC-ECD	GC-MS(TD)	GC-ECD	GC-MS (SINGLE QUAD)	GC-MS	GC-ECD, GC-NPD, GC-FPD, GC-MS, HPLC-FID, HPLC-DAD	DETERMINATION	
ACCORDING TO EN 12396-1:1999	SAR-2-04OC	EN NF 12393			QUECHERS, M., ANASTASSIADES, S., J. LEHOTAY, D. STAUBBAUER, F. J. SCHENK, J. AOAC INT., 86 (2003) 412-431	REFERENCE METHOD	

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>IPRODIONE</b>									
								LAB CODE	
								SCOPE OF YOUR METHOD	
						QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX			
						CONFIRMATION METHOD			
94	93	92	91	90	89	88			
D	D	D	D	D	D	D			
M	M	M	S	M	M	M			
GC-NPD	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS			
0.05	0.005	0.02	0.02	0.02	0.02	0.05	RL (mg/Kg)		
102.7	96				83	87	RECOVERY (%)		
10	80	50	50	50	15	25	SAMPLE WEIGHT (g)		
4	2	4	3	2	4	1	EXTRACTION SOLVENT		
SPE	GPC	LLE	LLE	GPC	GPC	GPC	CLEAN-UP STEP		
							INTERNAL STANDARD		
		ALDRIN, DITALIMPHOS					INJECTION VOLUME (µL)		
2	1µL PER COLUMN	1	1	1	1	1			
SPLITLESS	SPLITLESS	SPLIT	SPLITLESS	SPLITLESS	SPLIT/SPLITLESS	SPLITLESS	INJECTION TYPE		
GC-ECD	GC-ECD AND GC-NPD OR GC-ECD OR GC-NPD	GC-ECD	GC-ECD	GC-NPD	GC-ECD, FPD, MS, HPLC-F-DAD	GC-GC-TOF	DETERMINATION		
	GC-MS (SINGLE-QUAD)	OFFICIAL METHOD OF ANALYSIS (1990) 15TH ED., 985-22 AOAC ARLINGTON VA	DFG-419				REFERENCE METHOD		
							IN HOUSE		

## **APPENDIX 7. Methods used by participants for determining pesticides.**

IPRODIONE							
100		99	98	97	96	95	LAB CODE
D	D	D	NA	D	D	D	SCOPE OF YOUR METHOD
M	M	M		S	S	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-ECD	GC-MS	GC-MS		GC-MS	GC-MS	GC-MS	CONFIRMATION METHOD
0.04	0.02	0.02		0.02	0.05	RL (mg/Kg)	
82		115		102	103	RECOVERY (%)	
25		20	10	25	25	SAMPLE WEIGHT (g)	
6		4	1	2	5	EXTRACTION SOLVENT	
O	GPC	GPC		SPE	CLEAN-UP STEP		
2	PULSED SPLITLESS	ON-COLUMN		SPLIT/SPLITLESS	INTERNAL STANDARD		
GC-ECD	GC-MS	GC-MS		GC-ECD GC-NPD; GC-MD (SINGLE-GUAD)	PTV	INJECTION TYPE	
MINISTRY OF WELFARE, HEALTH AND CULTURAL AFFAIRS, NETHERLANDS, ANALYTICAL METHODS FOR RESIDUES OF PESTICIDES IN FOODSTUFFS MULTIRESIDUE METHOD 1, SUBMETHOD 1, PART I-3, 5TH EDITION, 1988	GC-MS MULTI RESIDUE			SOP 5.4.CH.1 (ACCORDING TO EN 12393/-3; AND 35 LMBG L00.0034	FILLION ET AL, JOURNAL OG AOAC INTERNATIONAL 78-1995	REFERENCE METHOD	

**APPENDIX 7. Methods used by participants for determining pesticides.**

IPRODIONE									
105	104	103	102	101	LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	CONFIRMATION METHOD	
D	D	D	D	D		D	M	GC-MS	
M	S	S	M	M			M		
GC-ECD	GC-MS	GC-MS	GC-NPD	GC-NPD				GC-MS	
0.05	0.005	0.020	0.05	0.05				RL (mg/Kg)	
			70	86				RECOVERY (%)	
100	100	50	30	30				SAMPLE WEIGHT (g)	
4	2	1	1	1				EXTRACTION SOLVENT	
								CLEAN-UP STEP	
								SPE	
								GPC	
ALDRIN	TRIPHENYLPHOSPHATE							INTERNAL STANDARD	
1	5	1	1	1				1	
								INJECTION VOLUME (µL)	
	PTV	DIRECT	SPLITLESS	SPLIT/SPLITLESS				INJECTION TYPE	
			PTV	PTV					
GC-NPD, GC-ECD	GC-MS		GC-ECD	GC-ECD				DETERMINATION	
MULTIRESIDUE METHOD: AOAC 985:22/90 MODIFIED	ANN.FALS.EXP.CH IM. SEPT-OCT 1974-67 NY721-722 PP513-524	LMBG §35 L-00.00 .34	SOP MR 405012; EXTRACTION OF RESIDUES OF PLANT-PROTECTING MATERIAL FROM FRUIT AND VEGETABLE SAMPLES BY ETHYL-ACETATE; SOP 308M2; CLEAN UP OF EXTRACTS BY GEL PERMEATION CHROMATOGRAPHY					REFERENCE METHOD	

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>IIPRODIONE</b>									
112	111	110	109	108	107	106	LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
ND	D	D	D	NA	D	D			
M	M	M	M	M	M	S			
GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-NPD	CONFIRMATION METHOD		
0.05	0.02	0.02	0.02	0.02	0.006	0.01	RL (mg/Kg)		
95	105.5					78	RECOVERY (%)		
50	50	50	10	10	100	25	SAMPLE WEIGHT (g)		
6	2	6	2	2	2	6	6 ETHYLACETATE/DICHLOROMETHANE	EXTRACTION SOLVENT	
SPE	GPC	GPC	GPC	GPC	GPC		CLEAN-UP STEP		
							INTERNAL STANDARD		
			BROMOPHOS-ETHYL				INJECTION VOLUME (µL)		
1	1	2	2	2	1	1			
SPLITLESS	OC; SS	SPLITLESS	SPLITLESS				INJECTION TYPE		
GC-ECD	GC-ECD, GC-NPD	GC-ECD AND GC-NPD	GC-ECD GC-NPD GC-FPD GC-MS	GC-ECD-ECOD; GC-NPD; GC-MS (SINGLE-QUAD)	GC-PPPD NPD GC-MS		DETERMINATION		
ISTISAN 97/23	\$35 LMBG; L 00. 00-34, L 00.00-37	MULTI-RESIDUE- METHOD DFG S 19					REFERENCE METHOD		

**APPENDIX 7. Methods used by participants for determining pesticides.**

IPRODIONE									
119	118	117	116	115	114	113	LAB CODE		
D	D	ND	D	D	D	D	SCOPE OF YOUR METHOD		
S	S		S	S	S	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		
GC-MS	GC-ECD		GC-MS	GC-ECD	GC-MS	GC-ECD	CONFIRMATION METHOD		
0.02	0.02	0.03	0.01	0.01	0.02	0.05	RL (mg/Kg)		
109.6	84		64		93.2	86	RECOVERY (%)		
10	5	40	15	50	50	30	SAMPLE WEIGHT (g)		
3	3	1	6	3	6	1	EXTRACTION SOLVENT		
CLEAN-UP STEP									
O			GPC	SPE	SPE	GPC			
							INTERNAL STANDARD		
1	2	1	1	1	1	1	INJECTION VOLUME (µL)		
AUTO	SPLITLESS	SPLITLESS	SPLITLESS	ON COLUMN	SPLITLESS	SPLITLESS	INJECTION TYPE		
GC-ECD GC-MS	GC-ECD, GC-NPD	GC-MS	GC-ECD, GC-NPD, GC-MS(ITT)	GC-ECD GC-PFPD GC-MS	GC-MS	GC-ECD	DETERMINATION		
ISS B6			RAPPORTO ISTISAN 97/23	NFEN12393			REFERENCE METHOD		
SOP MR 405012; EXTRACTION OF RESIDUES OF PLANT-PROTECTING MATERIAL FROM FRUIT AND VEGETABLE SAMPLES BY ETHYL-ACETATE; SOP 308M2; CLEAN UP OF EXTRACTS BY GEL PERMEATION CHROMATOGRAPHY									

**APPENDIX 7. Methods used by participants for determining pesticides.**

IPRODIONE									
128	127	126	125	124	123	122	121	120	LAB CODE
D	ND	NA	NA	NA	NA	D	D	SCOPE OF YOUR METHOD	
M	S					S	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
GC-MS	GC-ECD					GC-ECD	GC-MS	CONFIRMATION METHOD	
0.02	0.01					<0.020	RL (mg/Kg)		
20 / 5						50		RECOVERY (%)	
1	100					100	100	SAMPLE WEIGHT (g)	
	3					3,1',CYCLOHEXANE, N-HEXANE, IZO-OCTANE, TOLUENE	3	EXTRACTION SOLVENT	
								CLEAN-UP STEP	
		LLE					GPC	GPC, SPE	
								INTERNAL STANDARD	
								INJECTION VOLUME (µL)	
							1		
							2		
								INJECTION TYPE	
								SPLIT	
								GC-ECD	DETERMINATION
								GC-FPD, GC-MSD, GC-MS(TD)	
								PN-EN 12393:2000	PN-EN 12393-1,2,3
									REFERENCE METHOD

NO RESULTS

## **APPENDIX 7. Methods used by participants for determining pesticides.**

KRESOXIM-METHYL										
LAB CODE		SCOPE OF YOUR METHOD		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		CONFIRMATION METHOD		REFERENCE METHOD		
5	4	3	2	M	S	GC-MS	GC-MS	GC-MS	GC-MS	
D	D	D	D	M	M	GC-NPD	GC-MS/MS	GC-MS	GC-MS	
0.05	0.008	0.05	0.010	0.05	0.05	RECOVERY (%)	RL (mg/kg)	RL (mg/kg)	RL (mg/kg)	
10	10	15	15	15	15	92	92	92	92	
5	5	1	4	1	4	EXTRACTION SOLVENT	SAMPLE WEIGHT (g)	SAMPLE WEIGHT (g)	SAMPLE WEIGHT (g)	
SPE	SPE	NO	SPE	SPE	SPE	CLEAN-UP STEP	INJECTION VOLUME (µL)	INJECTION VOLUME (µL)	INJECTION VOLUME (µL)	
MIREX/TPP		TRIPHENYL PHOSPHATE ANTHRACENE				INTERNAL STANDARD	GC-ECD	GC-ECD	GC-ECD	
2	2	10	1	1	1	YES	GC-MS	GC-MS	GC-MS	
SPLIT/SPLITLESS	SPLIT/SPLITLESS	SPLIT	SPLITLESS	SPLITLESS	SPLITLESS	INJECTION TYPE	DETERMINATION	DETERMINATION	DETERMINATION	
GC-ECD/ GC-NPD	GC-MS	GC-MS/MS	GC-ECO	GC-ECO	GC-ECO	IN HOUSE	IN HOUSE	IN HOUSE	IN HOUSE	
MANASTASSIADES ET AL. JAOAC 86, 412-431 (2003)		QUECHERS; ANASTASSIADES	LUKE, M.A., FROBERG, J.E., DOOSE, G.M., MASUMOTO, H.T. (1981) IMPROVED MULTIRESIDUE GAS CHROMATOGRAPHIC DETERMINATION OF ORGANOHALOGEN PESTICIDES IN PRODUCE, USING FLAME PHOTOMETRIC AND ELECTROLYTIC CONDUCTIVITY DETECTORS. J. ASSOC. OFF. ANAL. CHEM. 64(5): 1187-1195		REFERENCE METHOD					

## **APPENDIX 7. Methods used by participants for determining pesticides.**

KRESOXIM-METHYL							
QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX				CONFIRMATION METHOD			
LAB CODE	SCOPE OF YOUR METHOD	STANDARDS IN SOLVENT OR IN MATRIX	CONFIRMATION METHOD	LAB CODE	SCOPE OF YOUR METHOD	STANDARDS IN SOLVENT OR IN MATRIX	CONFIRMATION METHOD
11	10	9	8	7	6		
D	D	D	D	D	D		
S	M	S	M	S	M		
GC-MS(1TD)	GC-MS	GC-ECD	GC-MS	GC-MS	GC-MS		
0.05	0.007	0.01	0.01	0.01	0.02		
122	93		100		86.9		
30	25	15	30	10	15		
1	2	4	1	6 (DICHLOROMETHANE)	4		
EXTRACTION SOLVENT				CLEAN-UP STEP			
LLE ACETONE FOLLOWED BY DICHLOROMETHANE AND PETROLEUM ETHER				GPC			
GPC				O			
INTERNAL STANDARD							
1	2	5	2	4	2		
SPLIT/SPLITLESS	SPLIT/ SPLITLESS PTV	LOOP	SPLITLESS	SPLITLESS	SPLITLESS		
GC-MS (ION TRAP)	GC-ECD, GC-NPD, GC-MS(1TD)	LC-MS/MS	GC-MS	GC-ECD; GC-NPD(80)	GC-MS		
IN HOUSE SOP				RAPPORT IISTISAN 97/23			
ANALYTICAL METHODS FOR PESTICIDE RESIDUES MINISTRY OF HEALTH NETHERLANDS 6TH ED				REFERENCE METHOD			

**APPENDIX 7. Methods used by participants for determining pesticides.**

KRESOXIM-METHYL										
19	18	17	16	15	14	13	12	LAB CODE		
D	D	NA	D	D	NA	D	D	SCOPE OF YOUR METHOD		
M	S	M	S	S	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		
GC-MS(1TD)	GC-MS	GC-MS/MS	GC-MS	GC-MS/MS	GC-MS/MS	GC-MS/MS	MS-1TD, GC-ECD	CONFIRMATION METHOD		
0.02	0.02	0.05	0.05			0.05	0.01	RL (mg/Kg)		
74	147		50			90.2	90	RECOVERY (%)		
75	75		15	104		15	50	SAMPLE WEIGHT (g)		
1	1		4	1		1	1	EXTRACTION SOLVENT		
								CLEAN-UP STEP		
								GPC		
DITALIMPHOS	ALDRIN					TPP (QC)		INTERNAL STANDARD		
2	2		10	2		10	1, 2, 5	INJECTION VOLUME (µL)		
								INJECTION TYPE		
								SPLITLESS		
								SPLIT/SPLITLESS		
GC-ECD	GC-MS					GC-ECD	GC-MS(1TD)	DETERMINATION		
								LOCAL SOP NO.		
								MR405012		
								REFERENCE METHOD		

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>KRESOXIM-METHYL</b>									
<b>27</b>	<b>26</b>	<b>25</b>	<b>24</b>	<b>23</b>	<b>22</b>	<b>21</b>	<b>20</b>	<b>LAB CODE</b>	
D	D	D	D	D	D	D	D	SCOPE OF YOUR METHOD	
S	M	M	M	M	M	M	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
	GC-MS(FTD)	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-NPD	CONFIRMATION METHOD	
0.05	0.02	<0.02	0.02	0.05	0.06	0.01	0.05	RL (mg/Kg)	
	67	95	106.6		98.1		97	RECOVERY (%)	
30	25	10	30	5	50	10	100	SAMPLE WEIGHT (g)	
1	2	6	1	5	6	1	3	EXTRACTION SOLVENT	
	GPC	SPE	GPC		LLE (ISOPROPYL ETHER)		O	CLEAN-UP STEP	
	DITALIMPHOS	YES		FENCHLORPHOS				INTERNAL STANDARD	
1	2	2	1 ng SAMPLE	50	2	10	1	INJECTION VOLUME (µL)	
	SPLITLESS	SPLITLESS	TPOCI	AUTOMATIC TDS SAMPLER	SPLITLESS	LVI	AUTOSAMPLER	INJECTION TYPE	
	GC-NPD	GC-ECD, GC-NPD, GC-MS(FTD)	GC-MS	GC-ECD	TDS-IN-LINE WITH GC-MS (SINGLE-QUAD) TOTALION DETECTION	GC-ECD AND GC-NPD	GC-MS/MS	GC-ECD	DETERMINATION
	HUNGARIAN CENTRAL SPP NO.: 405012 M1			SOF NO.: MR 405012 M1; 308 M2	INTERNAL METHOD				REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

KRESOXIM-METHYL									
								LAB CODE	
								SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
								S	
35	34	33	32	31	30	29	28	CONFIRMATION METHOD	
D	D	NA	D	D	NA	D	D	RL (mg/Kg)	
M	M	S	S	S	M	M	S	RECOVERY (%)	
GC-MS	GC-MS/MS	GC-MS	MS	GCMS2	GC-MS	GC-MS	GC-MS	SAMPLE WEIGHT (g)	
0.02	0.05	0.05	0.05	0.05	0.05	0.05	0.05	EXTRACTION SOLVENT	
96	98	98	94	100	100	100	88		
20	1	25	10	15	15	15	50		
3	4	2	6	4	4	4	6		
								CLEAN-UP STEP	
								SPE	
								INTERNAL STANDARD	
								INJECTION VOLUME (µL)	
								ON COLUMN	
								INJECTION TYPE	
								DETERMINATION	
								REFERENCE METHOD	
EN 12393-1, 2, 3		PESTICIDES MS/MS EI-Cl		\$35 LM BG 00,00-34		ISTISAN 97/23		MINI LUKE	

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>KRESOXIM-METHYL</b>							
							LAB CODE
41	40	39	38	37	36	D	SCOPE OF YOUR METHOD
D	D	D	D	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
M	M	S	M	M	S	S	CONFIRMATION METHOD
GC-MS	GC-MS	GC/MS	GC-ECD	GC-MS	GC-MS	GC-MS	RL (mg/Kg)
0.02	0.05	0.01	0.06	0.02	0.05	0.05	RECOVERY (%)
92.7						94	
20	15	100	10	10	25	25	SAMPLE WEIGHT (g)
3	4	2	5	5	3	3	EXTRACTION SOLVENT
							CLEAN-UP STEP
							INTERNAL STANDARD
							INJECTION VOLUME (µL)
							INJECTION TYPE
							GC-FPD GC-ECD GC-MS (SINGLE QUAD) LC-MS HPLC-MS HPLC-PICKERING DETERMINATION
							REFERENCE METHOD
GC-ECD, GC-NPD, GC-NPD	GC-NPD, GC-ECD, GC-MS(1D)	GC-ECD	GC-ECD GC-NPD GC-MS	GC-MS (SINGLE QUAD)	Anastassiades et al. JAOAC INT. VOL T 86 NO. 2, 2003		
	ANALYTICAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS, 6TH ED., 1996, GENERAL INSPECTORATE FOR HEALTH PROTECTION, MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS	DFG S 19, L 00.00-34 (§ 35 LMBG)					

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

KRESOXIM-METHYL									
								LAB CODE	
								SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
								CONFIRMATION METHOD	
D	55	54	53	52	51	50	49	48	LAB CODE
M		D	D	D	NA	D	D	D	SCOPE OF YOUR METHOD
GC-ECD	GC-MS	GC-MS	S	S	S	S	S	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
0.01	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.01	CONFIRMATION METHOD
83.3	90					96			RL (mg/Kg)
100	18		15	10		20	50	25	RECOVERY (%)
2	1	6	5	5	6	1	2		SAMPLE WEIGHT (g)
GPC	GPC	GPC	O						EXTRACTION SOLVENT
									CLEAN-UP STEP
GC 1 µL HPLC 20 µL						NITROFEN			INTERNAL STANDARD
SPLIT/SPLITLESS	SPLITLESS ON COLUMN	SPLIT/SPLITLESS							INJECTION VOLUME (µL)
GC-ECD, GC-NPD, GC-FPD, HPLC-UV, LC-MS/MS	GC-ECD, FPD,NPD	GC-ECD, GC-NPD, GC-FPD, GC-MS, HPLC-FLD, HPLC-UV	1.5	1	1.5	2	2	3	INJECTION TYPE
MODULARE MULTIMETHOD S19	EN 12393-1,2,3	INTERNAL METHOD	QUECHERS						DETERMINATION
									REFERENCE METHOD
									DFG S 19
									METHOD R EN 12393-2:1999

**APPENDIX 7. Methods used by participants for determining pesticides.**

KRESOXIM-METHYL									
62	61	60	59	58	57	56	LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
NA	D	D	D	D	D	D			
M	M	M	M	M	M	S			
GC-MS	GC-MS	GC-MS	GC-ECD-NPD	GC-MS	GC-MS	GC-MS	CONFIRMATION METHOD		
0.02	0.05	0.02	0.01	0.01	0.01	0.532	RL (mg/Kg)		
		97.4	109			99.1	RECOVERY (%)		
50	15	10	15	15	25	30	SAMPLE WEIGHT (g)		
3	1	5	4	4	1	1	EXTRACTION SOLVENT		
							CLEAN-UP STEP		
	GPC	GPC	O		GPC	GPC			
							INTERNAL STANDARD		
		ISODRIN	3	PCB119 (ECD) TRIFLURALIN (GC-NPD)					
	2	1	2	1	1	1	INJECTION VOLUME (µL)		
	PTV	SPLIT/SPLITLESS	SPLITLESS	SPLIT/SPLITLESS	PULSED SPLITLESS	SPLIT	INJECTION TYPE		
	GC-MS	GC-ECD/NPD/MS	GC-MS	GC-ECD-NPD	GC-ECD/NPD	GC-ECD	DETERMINATION		
	DFG S19 (ASU §35 LM BG L 00 00-34)			ANAL.METHODS FOR PESTICIDES RESIDUES IN FOODSTUFFS (NL)			REFERENCE METHOD		

## **APPENDIX 7. Methods used by participants for determining pesticides.**

KRESOXIM-METHYL									
LAB CODE		SCOPE OF YOUR METHOD		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX				CONFIRMATION METHOD	
68	67	66	65	64	63				
D	D	D	D	NA	D				
M	S	M	M	M	M				
GC-MS	GC-MS	GC-MS/MS	GC-MS	GC-MS	GC-MS			GC-MS	
0.05	0.05	0.05	0.03	0.03	0.05			RL (mg/Kg)	
94.8		88	104					RECOVERY (%)	
20	90	15	5	5				SAMPLE WEIGHT (g)	
4	3	5	1	1				EXTRACTION SOLVENT	
								CLEAN-UP STEP	
								GPC	
								INTERNAL STANDARD	
								INJECTION VOLUME ( $\mu$ L)	
								INJECTION TYPE	
								DETERMINATION	
									REFERENCE METHOD
MINI LUKE EXTRACTION	OFFICIAL METHOD L	QUECHERS LEO THAY, S. ET AL. JAOA.C. INT. (2005) 88, 615-629	INTERNAL METHOD	INTERNAL METHOD	INTERNAL METHOD				\$ 351 LMBG L 00 00-34

## **APPENDIX 7. Methods used by participants for determining pesticides.**

## **APPENDIX 7. Methods used by participants for determining pesticides.**

KRESOXIM-METHYL									
QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX					CONFIRMATION METHOD				
LAB CODE		SCOPE OF YOUR METHOD		M	GC-MS		RL (mg/Kg)		
D	D	D	ND	M	S	M	0.01	0.01	RECOVERY (%)
M	M	M	M	M	S	M	88	114	SAMPLE WEIGHT (g)
82	81	80	79	78	77	76	75	75	EXTRACTION SOLVENT
D	D	D	ND	M	S	M	0.05	0.05	DICHLOROMETHANE
105	105	105	105	105	105	105	10	10	6 METHANOL
10	10	10	10	10	10	10	15	15	
5	5	5	5	5	5	5	6	6	
NO RESULTS									
0 (PSA)	0	SPE	GPC		LLE (CHEM ELUT)				CLEAN-UP STEP
1	20	20	20	20	1	1	20	20	INTERNAL STANDARD
SPLITLESS AND ON COLUMN					SPLITLESS	SPLITLESS	LC-MS/MS	LC-MS/MS	INJECTION VOLUME ( $\mu$ L)
GC-ECD, GC-NPD, GC-FPD, GC-MS, HPLC-FLD, HPLC-DAD	LC-MS/MS	GC-EC	GC-MS (SINGLE-QUAD)	GC-MS	GC-MS	JAOAC INTERN., 86, 1015FF (2003),			DETERMINATION
QUECHERS, M. ANASTASSIADES, S. J. LEHOTAY D. STAINBAUER F. J. SCHENK; J. AOAC INT., 86 (2003)412-431	KLEIN J., ALDERL (2003) JAOAC INT. 86, 1015-103	INTERNAL METHOD							REFERENCE METHOD

## **APPENDIX 7. Methods used by participants for determining pesticides.**

KRESOXIM-METHYL							
LAB CODE				SCOPE OF YOUR METHOD			
QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX				CONFIRMATION METHOD			
88	87	86	85	84	83	83	LAB CODE
D	D	D	D	D	D	D	SCOPE OF YOUR METHOD
M	S	M	M	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-MS	GC-MS	GC-MS(TD)	GC-MS	GC-MS	GC-MS	GC-MS	CONFIRMATION METHOD
0.16		0.025	0.05	0.01	0.05	0.05	RL (mg/kg)
85		93		80		109	RECOVERY (%)
25	50	15	25	50	30	30	SAMPLE WEIGHT (g)
1	6 ETHYLACETATE/DICHLOROMETHANE (80/20)	4	4	2	1	1	EXTRACTION SOLVENT
GPC	SPE-FLORISIL				SPE GPC		CLEAN-UP STEP
							INTERNAL STANDARD
		HCB			TRIPHENYL ETHER TRIPHENYL ETHYLENE		INTERNAL STANDARD
2	1	1	1	1	1	3	INJECTION VOLUME (µL)
SPLITLESS		SSL	SPLITLESS	SPLITLESS	SPLITLESS SPLITLESS		INJECTION TYPE
GC-NPD	GC-ECD	GC-MS(TD)	GC-ECD	GC-MS (SINGLE-QUAD)	GC-MS GC-MS		DETERMINATION
IN HOUSE							REFERENCE METHOD
							EN NF 12393
							ACCORDING TO EN 12396-1:1999

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>KRESOXIM-METHYL</b>							
				SCOPE OF YOUR METHOD			
				QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX			
				GC-MS	GC-MS	GC-MS	CONFIRMATION METHOD
D	D	D	D	S	M	M	RL (mg/Kg)
S	M	M	M	S	M	M	RECOVERY (%)
GC-MS	GC-NPD	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	SAMPLE WEIGHT (g)
0.05	0.03	0.005	0.03	0.02	0.01	0.02	EXTRACTION SOLVENT
95	104		105			89	CLEAN-UP STEP
25	10	80	50	50	50	15	INTERNAL STANDARD
5	6	2	4	3	2	4	INJECTION VOLUME (µL)
SPE	SPE	GPC	LL	LL	GPC	GC-NPD	INJECTION TYPE
YES			ALDRIN, DITALIMPHOS	YES		GC-ECD	DETERMINATION
3	2	1µL PER COLUMN	1	1	1	FPD	
PTV	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	MS	
GC-ECD, GC-NPD, GC-MS	GC-NPD	GC-ECD AND GC-NPD OR GC-ECD OR GC-NPD	GC-MS	GC-NPD	GC-NPD	HPLC-F-DAD	
FILLION ET AL., JOURNAL OG AOAC INTERNATIONAL 78-1995		(SINGLE-QUAD)		OFFICIAL METHOD OF ANALYSIS (1980) 15TH ED. 985/22, AOAC ARLINGTON VA	DFG-S8		REFERENCE METHOD

## **APPENDIX 7. Methods used by participants for determining pesticides.**

## **APPENDIX 7. Methods used by participants for determining pesticides.**

KRESOXIM-METHYL						
LAB CODE	SCOPE OF YOUR METHOD			QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		
	S	S	S	M	M	M
106	105	104	103	102	102	102
D	NA	D	D	D	D	D
S	S	S	S	M	M	M
GC-ECD	GC-MS	GC-MS	HPLC-DAD	HPLC-DAD	CONFIRMATION METHOD	
0.01	0.005	0.010	0.02	0.02	RL (mg/Kg)	
80		90	103	103	RECOVERY (%)	
25	100	50	30	30	SAMPLE WEIGHT (g)	
6 ETHYLACETATE/DICHLOROMETHANE	2	1	1	1	EXTRACTION SOLVENT	
					CLEAN-UP STEP	
SPE	GPC	SPE	GPC	GPC		
					INTERNAL STANDARD	
					INJECTION VOLUME ( $\mu$ L)	
					INJECTION TYPE	
					DETERMINATION	
					SOP MR 405012; EXTRACTION OF RESIDUES OF PLANT-PROTECTING MATERIAL FROM FRUIT AND VEGETABLE SAMPLES BY ETHYLACETATE; SOP 303M2; CLEAN UP OF EXTRACTS BY GEL PERMEATION CHROMATOGRAPHY	REFERENCE METHOD
					ANN FALS EXP. CHIM. SEPT-OCT 1974-87 N)721-722 PP513-524	
					LMBG §35 L-00.00-34	MESTRES

## **APPENDIX 7. Methods used by participants for determining pesticides.**

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

KRESOXIM-METHYL							
128	127	126	125	LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	CONFIRMATION METHOD
D	NA	D					
M	GC/MS	S					
0.05	RL (mg/Kg)	0.0006	RECOVERY (%)	76.13			
20 G / 5G	SAMPLE WEIGHT (g)	25	EXTRACTION SOLVENT	3			
	CLEAN-UP STEP	GPC					
	INTERNAL STANDARD						
2	INJECTION VOLUME (µL)	2					
SPLITLESS	INJECTION TYPE						
GC-ECD, GC-FPD, GC-MSD, GC-MS(TID)	DETERMINATION	GC-ECD					
MODIFIED LUKE	REFERENCE METHOD						

NO RESULTS

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>METHOMYL</b>									
<b>8</b>	<b>7</b>	<b>6</b>	<b>5</b>	<b>4</b>	<b>3</b>	<b>2</b>	<b>1</b>	<b>LAB CODE</b>	<b>SCOPE OF YOUR METHOD</b>
D	NA	NA	D	D	NA	D	D		
M		M	M		M	M	M		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-MS		LC-MS/MS		LC-MS/MS		LC-MS/MS		LC-MS/MS	CONFIRMATION METHOD
0.02		0.002	0.006		0.010	0.05	0.05	LC-MS/MS	RL (mg/Kg)
105					93				RECOVERY (%)
10		10	10		10	10	10		SAMPLE WEIGHT (g)
6		5	5		6	6	1		EXTRACTION SOLVENT
SPE		SPE	SPE					LC-MS/MS	CLEAN-UP STEP
									INTERNAL STANDARD
10		20	10		5	5	20		INJECTION VOLUME ( $\mu$ L)
								LC-MS/MS	INJECTION TYPE
LC-MS/MS			LC-MS/MS					LC-MS/MS	DETERMINATION
								KLEIN, J., ALDER, L. (2003); APPLICABILITY OF GRADIENT LIQUID CHROMATOGRAPHY WITH TANDEM MASS SPECTROMETRY TO THE SIMULTANEOUS SCREENING FOR ABOUT 100 PESTICIDES IN CROPS. J. AOAC INT. 86 (5): 1015-1037	REFERENCE METHOD
			M. ANASTASSIADES ET AL. JAOAC 86, 412-431 (2003)		QUECHERS; ANASTASSIADES				IN HOUSE

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>METHOMYL</b>									
									LAB CODE
									SCOPE OF YOUR METHOD
S		S	M	M	S	S	S	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-MS	HPLC-UV	LC-MS/MS	LC-MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	CONFIRMATION METHOD
0.05	0.05	0.05	0.02	0.05	0.05	0.01	0.01	0.01	RL (mg/Kg)
75	86	93.5	73	80	80	97	97	97	RECOVERY (%)
1	25	15	50	30	30	15	15	15	SAMPLE WEIGHT (g)
		1	1	1	1	4	4	4	EXTRACTION SOLVENT
GPC	O	GPC	O	GPC	O	LLE ACETONE FOLLOWED BY DICHLOROMETHANE AND PETROLEUM ETHER	CLEAN-UP STEP	CLEAN-UP STEP	
ALDRIN		TPP (QC)					INTERNAL STANDARD	INTERNAL STANDARD	
2	25	10	1,2,5	20	20	5	INJECTION VOLUME (µL)	INJECTION VOLUME (µL)	
PULSED SPLITLESS			SPLITLESS			LOOP	INJECTION TYPE	INJECTION TYPE	
GC-MS	HPLC-DAD	LC-MS/MS	MS-TID, LC-MS, GC-FPD, GC-ECD	HPLC-UV	LC-MS/MS	LC-MS/MS	DETERMINATION	DETERMINATION	
		LOCAL SOP NO. MR405012				IN HOUSE SOP	REFERENCE METHOD	REFERENCE METHOD	

## **APPENDIX 7. Methods used by participants for determining pesticides.**

METHOMYL									
LAB CODE		SCOPE OF YOUR METHOD		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		CONFIRMATION METHOD			
26	25	24	23	22	21	20	19		
D	D	D	ND	D	NA	NA	D		
M	M	S	M	M	M	M			
LC-MS/MS	LC-MS/MS	GC-MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS			
0.01	<0.05	0.05	0.1	0.05			0.01	RL (mg/Kg)	
114	94	82.0		118.5			98	RECOVERY (%)	
10	5	30	5	10			75	SAMPLE WEIGHT (g)	
6 METHANOL 95%-AMMONIACETATE 5%, 20 mM ACETIC ACID	5	1	5	METHANOL			1	EXTRACTION SOLVENT	
CLEAN-UP STEP									
CARBARYL C13		FENCHLORPHOS						INTERNAL STANDARD	
10	10	1 mg SAMPLE	50	5			5	INJECTION VOLUME (µL)	
SPLITLESS	AUTOSAMPLER	AUTOMATIC TDS SAMPLER						INJECTION TYPE	
LC-MS/MS	LC-MS/MS	TDS-IN LINE WITH GC-MS (SINGLE-QUAD) TOTAL ION DETECTION	LC-MS/MS					DETERMINATION	
	SOP NO.: MR 405012 M	INTERNAL METHOD						REFERENCE METHOD	

## **APPENDIX 7. Methods used by participants for determining pesticides.**

METHOMYL									
								LAB CODE	
		SCOPE OF YOUR METHOD		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX					
		CONFIRMATION METHOD							
36	35	34	33	32	31	30	29	28	27
D	ND	NA	NA	NA	NA	NA	D	D	D
S	M					S	S	S	S
GC-MSS		HPLC-FLD		HPLC-FLD		HPLC-FLD		HPLC-FLD	
0.05	0.05			0.05		0.03		0.05	
90				85		90			
25	20			15		20		30	
3	3			4		3		1	
SPE		CLEAN-UP STEP		INTERNAL STANDARD		INJECTION VOLUME (µL)		INJECTION TYPE	
		SPE (NH <sub>2</sub> )		GPC		GPC		RHEODYNE INJECTOR	
1	2			400		20			
SPLIT/SPLITLESS		SPLITLESS							
GC-FPD GC-ECD GC-MS (SINGLE-QUAD) LC-MS HPLC-MS HPLC-PICKERING		HPLC-FLD		HPLC-FLD		HPLC-UV		DETERMINATION	
EN 12393-1, 2, 3		MINI LUKE		HUNGARIAN CENTRAL SOP NO.: 405012 M1		REFERENCE METHOD			

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>METHOMYL</b>									
44	43	42	41	40	39	38	37	LAB CODE	SCOPE OF YOUR METHOD
D	D	D	NA	NA	D	NA	D		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
M	M	M			M		M		CONFIRMATION METHOD
	NONE	LC-MS/MS			LC-MS/MS		LC-MS/MS		RL (mg/Kg)
0.01	0.01	0.01			0.01		0.005		RECOVERY (%)
	85	111			95				SAMPLE WEIGHT (g)
50	10	10			10		10		EXTRACTION SOLVENT
2	5	5			6 (METHANOL/WATER)		5		CLEAN-UP STEP
LLE	SPE	LLE			SPE		O		INTERNAL STANDARD
							DIMETHOATE D6		INJECTION VOLUME (µL)
									INJECTION TYPE
LC-MS/MS	LC-MS/MS(ESI+)				LC-MS/MS		LC-MS/MS (TRIPLE-QUAD)		DETERMINATION
HOUSE METHOD	ANASTASSIAIDES ET AL., JAOAC INT. 86 82003 (2003) 412-431, MODIFIED						ANASTASSIAIDES, JOURNAL OF AOAC INTERNATIONAL, VOL 86 NO 2, 2003		REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>METHOMYL</b>									
53	52	51	50	49	48	47	46	45	LAB CODE
NA	D	D	D	D	D	ND	NA	D	SCOPE OF YOUR METHOD
M	S	S	S	S	M		S		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-MS	HPLC-FLD	LC-MS	LC-MS/MS	LC-MS/MS	LC-MS/MS				CONFIRMATION METHOD
0.05	0.01	0.05	0.01	0.01	0.05				RL (mg/Kg)
	99	101							RECOVERY (%)
10	50	50	10	10					SAMPLE WEIGHT (g)
5	6	4	6	6					EXTRACTION SOLVENT
	O	SPE	LLE	SPE					CLEAN-UP STEP
									GPC
									INTERNAL STANDARD
									TRIMETACARB
									INJECTION VOLUME (µL)
									200
									RHEODYNE
									INJECTION TYPE
									HPLC - ON LINE DERIVATIZATION - FD
									DETERMINATION
									KLEIN, J.; ALDER, L. JOURNAL OF AOAC INTERNATIONAL VOL 86, NO 5, 2003
									EXTRACTION AND CLEAN UP AS IN EN 12393 P METHOD
									REFERENCE METHOD
									QUECHERS EPA 531.1

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>METHOMYL</b>									
									LAB CODE
60	59	58	57	56	55	54			
D	D	D	D	D	D	D			SCOPE OF YOUR METHOD
M	M	S	M	S	M	S			QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
LC-MS/MS	HPLC-FLD	LC-FLD-PC	LC-MS/MS	—	HPLC-UV	LC-MS/MS			CONFIRMATION METHOD
0.05	0.05	0.03	0.01	0.233	0.01	0.025			RL (mg/Kg)
		98.5	93	47.7	88.9	104			RECOVERY (%)
5	10	15	25	30	100	5			SAMPLE WEIGHT (g)
1	5	4	5	1	2	1			EXTRACTION SOLVENT
									CLEAN-UP STEP
	O	SPE		GPC					
	1	BENZIMIDAZOLE							INTERNAL STANDARD
20	20	100	5	20	GC 1 µL HPLC 20 µL	5			INJECTION VOLUME (µL)
				LOOP	SPLIT/SPLITLESS				INJECTION TYPE
LC-MS/MS	HPLC-FLD WITH ND	LC-DAD-FLD	LC-MS/MS	HPLC-UV	GC-ECD, GC-NPD, GC-FPD, HPLC-UV LC-MS/MS	LC-MS/MS			DETERMINATION
§ 35 L 2900-6			ANAL.METHODS FOR PESTICIDES RESIDUES IN FOODSTUFFS (NL)		MODULARE MULTIMETHODE S19	JOURNAL OF CHROMATOGRAPHY A, 1023(2004),93-104			REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>METHOMYL</b>									
									LAB CODE
									SCOPE OF YOUR METHOD
S	M	S	S	S	M	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	CONFIRMATION METHOD
0.01	0.05	0.05	0.01	0.05	0.05	0.01	0.01	0.01	RL (mg/Kg)
77.1	80	88	100	79	79				RECOVERY (%)
10	10	15	5	10	10	20	20	20	SAMPLE WEIGHT (g)
4	5	5	6	METHANOL	METHANOL	METHANOL	METHANOL	METHANOL	EXTRACTION SOLVENT
									CLEAN-UP STEP
		LLE + SPE	SPE						
		TRIPHENYLPHOSPHATE							INTERNAL STANDARD
5	10	20	10	20	10	20	10	10	INJECTION VOLUME (µL)
		LOOP							INJECTION TYPE
LC-MS/MS	LC-MS/MS	LC-MS/MS	HPLC - FLD DERIVATIZED POST-COLUMN	LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	DETERMINATION
MINI LUKE EXTRACTION (SODIUM SULPHATE ADDED)	ANASTASSIADES, M. ET AL., J. AOAC INT., 86 (2003), 412 – 431 QUECHERS - METHOD		MULTIRESIDUE WITH EXTRELT	MULTIRESIDUE METHOD DRAFT BFR					REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

METHOMYL									
77	76	75	74	73	72	71	70	69	LAB CODE
NA	D	ND	D	ND	NA	D	ND	ND	SCOPE OF YOUR METHOD
M	M	S	S			M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
HPLC-DAD	HPLC-UV	GC-NPD		HPLC-FID		GC-MS		GC-MS	CONFIRMATION METHOD
0.01	0.01	0.05	0.05		0.05	RL (mg/Kg)	0.05	0.05	RECOVERY (%)
114	99.8	71							SAMPLE WEIGHT (g)
10	25	30			15		25		EXTRACTION SOLVENT
6 METHANOL	3	1	100		5		1		CLEAN-UP STEP
LLE (CHEMELUT)	SPE	GPC; O		3	O	SPE			INTERNAL STANDARD
									INJECTION VOLUME (µL)
							10	2	
LC-MS/MS	RHEODYNE			1	LVI	SPLITLESS			INJECTION TYPE
JAOAC INTERN. 86-1015FF (2003)	HPLC-DAD	HPLC-UV	SPLITLESS		GC-ECD/TSD/PFPD GC-MS/MS	GC-NPD			DETERMINATION
								METHYL CARBAMATES S25	REFERENCE METHOD
				LOCAL SOP	GC-EC, GC-NPD	QUECHERS			
NO RESULTS									

**APPENDIX 7. Methods used by participants for determining pesticides.**

METHOMYL											
88	87	86	85	84	83	82	81	80	79	78	LAB CODE
NA	D	D	NA	NA	D	NA	D	D	D	NA	SCOPE OF YOUR METHOD
M	M	M	M	M	M	M	M	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
6 METHANOL	4	4	5	5	5	5	6	6	6	6	CONFIRMATION METHOD
LLE							O	SPE			CLEAN-UP STEP
LC-MS/MS	0.025	0.025	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	RL (mg/Kg)
87	70	70	97	97	97	97	69	69	69	69	RECOVERY (%)
10	7.5	7.5	10	10	10	10	15	15	15	15	SAMPLE WEIGHT (g)
							6	6	6	6	EXTRACTION SOLVENT
											NO RESULTS
											INTERNAL STANDARD
									20	2	INJECTION VOLUME (µL)
											INJECTION TYPE
											DETERMINATION
											REFERENCE METHOD
							KLEIN, J., ALDERL (2003) JAOAC INT. 86, 1015-103	INTERNAL METHOD			
							JAOAC 86, 5, 1015- 1037				

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>METHOMYL</b>									
97	96	95	94	93	92	91	90	89	LAB CODE
NA	NA	D	NA	D	D	D	D	D	SCOPE OF YOUR METHOD
		S		M	M	S	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
				LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	HPLC-DAD	CONFIRMATION METHOD
0.05		0.01	0.01	0.006	0.006	0.05	0.05	0.04	RL (mg/Kg)
110				98				78	RECOVERY (%)
25		10	50	10	50	50	50	15	SAMPLE WEIGHT (g)
5	6, METHANOL	4	6 METHANOL	6 METHANOL	3	3	6	6	EXTRACTION SOLVENT
									CLEAN-UP STEP
				LLE	SPE	LLE			
									INTERNAL STANDARD
		YES							
				20	10	20	OCT-25	1	INJECTION VOLUME (µL)
				SAMPLE-LOOP	LOOP			SPLIT/SPLITLESS	INJECTION TYPE
				HPLC-MS/MS	LC-MS/MS	HPLC-DAD	GC-ECD, FPD, MS HPLC-F-DAD	DETERMINATION	
									REFERENCE METHOD
				MULTI-METHOD	LC-MULTIRESIDUE	DRAFT § 35 LMBG-METHOD			

## **APPENDIX 7. Methods used by participants for determining pesticides.**

## **APPENDIX 7. Methods used by participants for determining pesticides.**

METHOMYL									
VALIDATED IN HOUSE		METHOD NACH MULTI-RESIDUE-METHOD KLEIN UND ALDER - DFG WORKSHOP PRESENTED		APPLICATION NOTE BY		REFERENCE METHOD			
SCOPE OF YOUR METHOD		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		CONFIRMATION METHOD		CONFIRMATION METHOD			
LC-MS/MS	LC-MS	LC-MS/MS	LC-MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	
D	D	D	NA	D	D	NA	D	D	SCOPE OF YOUR METHOD
S	S	S	M	S	S	S	S	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
111	110	109	108	107	106	105	104	104	LAB CODE
0.05	-	0.01	0.01	0.02	0.02	0.005	0.005	0.005	CONFIRMATION METHOD
102.3				79					RECOVERY (%)
50	10	10	10	15	15	10	10	10	SAMPLE WEIGHT (g)
6 METHANOL	6 METHANOL	1	5	4	6 (METHANOL)				EXTRACTION SOLVENT
SPE	O	O	O						CLEAN-UP STEP
25	20	25	1	10			25		INTERNAL STANDARD
LC- MS/MS	HPLC-MS	LC-MS/MS	AUTOSAMPLER						INJECTION VOLUME ( $\mu$ L)
			HPLC - MS (SINGLE-QUAD)						INJECTION TYPE
									DETERMINATION
				QUECHERS ANASTASSIADES	DE KOK				

**APPENDIX 7. Methods used by participants for determining pesticides.**

120	119	118	117	116	115	114	113	112	LAB CODE
ND	NA	NA	NA	D	ND	D	D	NA	SCOPE OF YOUR METHOD
S				S	S	S	S		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC/MS				GC-MS	GC-MS	LC-MS/MS	HPLC-UV		CONFIRMATION METHOD
0.006				0.1	0.01	0.01	0.05		RL (mg/Kg)
100					94.7	94.7	87		RECOVERY (%)
3				15	50	20	30		SAMPLE WEIGHT (g)
				6	3	6	1		EXTRACTION SOLVENT
GPC									CLEAN-UP STEP
				GPC	SPE	SPE	GPC		
									INTERNAL STANDARD
					YES				
1				1	1	1	30	20 mL/20 mg	INJECTION VOLUME (µL)
SPLIT					SPLITLESS	ON COLUMN		MANUAL	INJECTION TYPE
GC-ECD ,GC-NPD				GC-ECD, GC-NPD, GC-MS(FTD)	GC-ECD GC-PFPD GC-MS	LC-MS/MS	HPLC-UV		DETERMINATION
PN-EN 12393-1,2,3				RAPPORTO ISTISAN 97/23	NFEN12393	WATERS A MULTIRESIDUE LC- MS/MS METHOD FOR PESTICIDES			REFERENCE METHOD
									SOP MR 405012; EXTRACTION OF RESIDUES OF PLANT- PROTECTING MATERIAL FROM FRUIT AND VEGETABLE SAMPLES BY ETHYL ACETATE; SOP 308M2; CLEAN UP OF EXTRACTS BY GEL PERMEATION CHROMATOGRAPHY

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>METHOMYL</b>										
128	127	126	125	124	123	122	121	LAB CODE	SCOPE OF YOUR METHOD	
NA	NA	NA	NA	NA	D	NA	NA	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		
					S					
					HPLC-FID			CONFIRMATION METHOD		
					0.009			RL (mg/Kg)		
					90			RECOVERY (%)		
					10			SAMPLE WEIGHT (g)		
					4			EXTRACTION SOLVENT		
								CLEAN-UP STEP		
								SPE		
								1, 2, 3-TRIMETHACARB	INTERNAL STANDARD	
								100	INJECTION VOLUME ( $\mu$ L)	
									INJECTION TYPE	
									DETERMINATION	
										REFERENCE METHOD
NO RESULTS										

**APPENDIX 7. Methods used by participants for determining pesticides.**

MONOCROTOPHOS										
5	4	3	2	1	LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	CONFIRMATION METHOD		
D	D	D	D	D		D	M	LC-MS/MS		
M	M	M	M	M		D	M			
GC-MS	GC-ECD	GC-MS/MS	GC-MS	GC-MS/MS						
0.03	0.001	0.02	0.030	0.030		0.03		RL (mg/Kg)		
		93.4	91					RECOVERY (%)		
10	10	15	15	10			10	SAMPLE WEIGHT (g)		
5	5	1	4	1			1	EXTRACTION SOLVENT		
								CLEAN-UP STEP		
	SPE	NO						SPE		
	MIREX/TPP	TRIPHENYL PHOSPHATE ANTHRACENE						INTERNAL STANDARD		
2	10	10	1	1			20	INJECTION VOLUME (µL)		
	SPLIT/SPLITLESS	SPLIT	SPLITLESS				N/A	INJECTION TYPE		
	GC-ECD/ GC-NPD	LC-MS/MS	GC-MS/MS	GC-NPD			LC-MS/MS	DETERMINATION		
MANASTASSIADES ET AL. JAOAC 86, 412-431 (2003)	QUECHERS; ANASTASSIADES				LUKE, M.A., FROBERG, J.E., DOOSE, G.M., MASUMOTO, H.T. (1981); IMPROVED MULTIRESIDUE GAS CHROMATOGRAPHIC DETERMINATION OF ORGANOPHOSPHORUS, ORGANONITROGEN, AND ORGANOHALOGEN PESTICIDES IN PRODUCE, USING FLAME PHOTOMETRIC AND ELECTROLYTIC CONDUCTIVITY DETECTORS, J. ASSOC. OFF. ANAL. CHEM. 64(5): 1187-1195				REFERENCE METHOD	

**APPENDIX 7. Methods used by participants for determining pesticides.**

MONOCROTOPHOS									
13	12	11	10	9	8	7	6	LAB CODE	SCOPE OF YOUR METHOD
D	D	D	NA	D	D	NA	D	GC-MS	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
M	M	S		S	M	M	M	GC-MS	CONFIRMATION METHOD
GC-MS	GC-FPD, MS-ITD	GC-NPD		GC-MS(ITD)	GC-MS			GC-MS	RL (mg/Kg)
0.02	0.03	0.03		0.01	0.01		0.02		RECOVERY (%)
70.2	92	116		93	83		93.6		
15	50	30		7.5	30		15		SAMPLE WEIGHT (g)
1	1	1		4 (+Na <sub>2</sub> SO <sub>4</sub> )	1		4		EXTRACTION SOLVENT
				LLE ACETONE FOLLOWED BY DICHLOROMETHANE AND PETROLEUM ETHER	O				CLEAN-UP STEP
TPP (QC)				GPC				GPC	INTERNAL STANDARD
1	1,2,5	1		5	2		2		INJECTION VOLUME (µL)
	SPLITLESS	ON COLUMN		LOOP	SPLITLESS				INJECTION TYPE
GC-ECD/TSD	MS-ITD, LC-MS, GC-FPD, GC-ECD	GC-FPD		LC-MS/MS	GC-MS			GC-MS	DETERMINATION
									ANALYTICAL METHODS FOR PESTICIDE RESIDUES MINISTRY OF HEALTH NETHERLANDS 6TH ED
									REFERENCE METHOD
									IN HOUSE SOP

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>MONOCROTOPHOS</b>									
<b>29</b>	<b>28</b>	<b>27</b>	<b>26</b>	<b>25</b>	<b>24</b>	<b>23</b>	<b>22</b>	<b>LAB CODE</b>	
D	D	NA	D	D	D	ND	ND	D	<b>SCOPE OF YOUR METHOD</b>
M	S		M	M	M	M	M	M	<b>QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX</b>
GC-MS/MS	LC-MS/MS	GC-NPD	GC-MS	LC-MS/MS	HPLC-DAD	GC-MS	GC-MS	GC-NPD	<b>CONFIRMATION METHOD</b>
0.03	0.03	0.02	<0.02	0.03	0.05	0.05	0.02	RL (mg/Kg)	<b>RECOVERY (%)</b>
40	89	75	99				92.7		
15	20	25	10	30	5	5	20	SAMPLE WEIGHT (g)	
4	3	2	6	1	1	5	1	1	<b>EXTRACTION SOLVENT</b>
		GPC	SPE	GPC	SPE FLORISIL	SPE	SPE		<b>CLEAN-UP STEP</b>
		DITALIMPHOS	YES						<b>INTERNAL STANDARD</b>
10	20	2	2	1	1	20	15		<b>INJECTION VOLUME (µL)</b>
		SPLITLESS	SPLITLESS	TPOCI	AUTOMATIC	SPLITLESS			<b>INJECTION TYPE</b>
GC-MS/MS	LC-MS/MS	GC-ECD, GC-NPD, GC-MS/ITD)	GC-MS	GC-NPD	HPLC-DAD	GC-NPD	GC-NPD	GC-NPD	<b>DETERMINATION</b>
						SOP NO.: MR 405012 M1, 308 M2	INTERNAL METHOD		<b>REFERENCE METHOD</b>
		MINI LUKE							

**APPENDIX 7. Methods used by participants for determining pesticides.**

MONOCROTOPHOS									
									LAB CODE
									SCOPE OF YOUR METHOD
37		36		35	34	33	32	31	30
D	D	D	NA	NA	NA	NA	D	ND	
M	S	M				S	S	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
LC-MS/MS	GC-MS	GC-MS				MS	DIFFERENT COLUMN		CONFIRMATION METHOD
0.005	0.02	0.02				0.05	0.05		RL (mg/Kg)
	90	100				101			RECOVERY (%)
10	25	20				10	10		SAMPLE WEIGHT (g)
5	3	3				6	1		EXTRACTION SOLVENT
O	SPE					O	GPC		CLEAN-UP STEP
DIMETHOATE D6							DICHLOFENTHION		INTERNAL STANDARD
20	1	2				1	1		INJECTION VOLUME (µL)
	SPLIT/SPLITLESS	SPLITLESS				SPLITLESS	SPLITLESS		INJECTION TYPE
LC-MS/MS (TRIPLE-QUAD)	GC-FPD GC-ECD GC-MS (SINGLE-QUAD) LC-MS HPLC-MS HPLC-PICKERING	GC-ECD, NPD				GC-ECD GC-NPD	GC-ECD GC-NPD		DETERMINATION
ANASTASSIADES, JOURNAL OF AOAC INT. VOL 86 NO 2, 2003	EN 12393-1, 2, 3					ISTISAN 97/23	RAPP/ISTISAN 97/23		REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>MONOCROTOPHOS</b>							
							LAB CODE
43	42	41	40	39	38		
D	D	NA	D	D	D		SCOPE OF YOUR METHOD
M	M	M	M	M	M		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-MS	LC-MS/MS	GC-MS	LC-MS/MS	GC-ECD	GC-ECD		CONFIRMATION METHOD
0.01	0.01	0.03	0.01	0.03	RL (mg/Kg)		
89	115			97			RECOVERY (%)
10	10	15	10	10	10		SAMPLE WEIGHT (g)
5	5	4	6 (METHANOL/WATER)	5			EXTRACTION SOLVENT
SPE	LL			O			CLEAN-UP STEP
TRIPHENYLPHOSPHATE (TPP)							INTERNAL STANDARD
4	20	1	20	2			INJECTION VOLUME (µL)
							INJECTION TYPE
LC-MS/MS(ESI+)	LC-MS/MS	GC-NPD, GC-ECD, GC-MS(ID)	LC-MS/MS	GC-ECD GC-NPD GC-MS			DETERMINATION
ANASTASSIADES ET AL. AOAC INT. 86 82003) 412-431, MODIFIED	M. ANASTASSIADES ET AL. J. AOAC INT. 86 (2), 412-431, (2003)	ANALYTICAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS. 6TH ED. 1996. GENERAL INSPECTORATE FOR HEALTH PROTECTION, MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS	LC-MS/MS (BFR)				REFERENCE METHOD

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>MONOCROTOPHOS</b>										
<b>59</b>	<b>58</b>	<b>57</b>	<b>56</b>	<b>55</b>	<b>54</b>	<b>53</b>	<b>52</b>	<b>LAB CODE</b>		
D	D	D	D	D	NA	D	D	SCOPE OF YOUR METHOD		
M	M	M	S	M	S	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		
GC-MS	GC-FPD	GC-MS	GC-NPD	GC-FPD	GC-MS	GC-MS	GC-MS	CONFIRMATION METHOD		
0.02	0.02	0.01	0.845	0.01	0.03	0.03	0.03	RL (mg/Kg)		
68.2	70		75	65.7				RECOVERY (%)		
10	25	25	30	100	15	15	10	SAMPLE WEIGHT (g)		
5	4	1	1	2	6	6	5	EXTRACTION SOLVENT		
O			GPC		GPC		O	CLEAN-UP STEP		
3	TRIBUTYLPHOSPHATE							INTERNAL STANDARD		
2	1	1	1	GC 1 µL HPLC 20 µL	1	1	1.5	INJECTION VOLUME (µL)		
SPLITLESS	SPLIT/SPLITLESS	PULSED SPLITLESS	DIRECT	SPLIT/SPLITLESS	SPLIT/ SPLITLESS	SPLIT/ SPLITLESS	SPLIT/ SPLITLESS	INJECTION TYPE		
GC-MS	GC-FPD	GC-ECD/NPD	GC-NPD	GC-ECD, GC-NPD, GC-FPD, HPLC-UV, LC-MS/MS	GC-ECD, GC-NPD, GC-FPD, HPLC-FID, HPLC-UV	GC-ECD, GC-NPD, GC-FPD, HPLC-UV	GC-ECD DETERMINATION			
§35 L 000034	ANALMETHODS FOR PESTICIDES RESIDUES IN FOODSTUFFS (NL)	S19		MODULARE MULTIMETHODE	INTERNAL METHOD	QUECHERS	REFERENCE METHOD			

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>MONOCROTOPHOS</b>									
LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	CONFIRMATION METHOD	RL (mg/Kg)	RECOVERY (%)	SAMPLE WEIGHT (g)	EXTRACTION SOLVENT	CLEAN-UP STEP	REFERENCE METHOD
65	64	63	62	61	60				
D	D	D	D	D	D				
M	M	M	M	M	M				
GC-MS	GC-MS	GC-MS	GC-MS/MS	GC-MS	GC-MS				
0.02	0.03	0.03	0.02	0.01	0.03				
84	100	73							
5	5	75	10	20	15				
1	6	ACETONE FOLLOWED BY CYCLOHEXANE AND ETHYLACETATE	1	METHANOL	1				
	GPC	GPC	GPC	GPC	GPC				
AZOBENZENE+RONNELL+TRIPHENYLPHOSPHATE	TPP	TPP	TPP	ISODRIN	INTERNAL STANDARD				
2	0.5	1	2	10	1				
ON COLUMN	SPLITLESS				SPLIT/SPLITLESS				
GC-(ECD+FID+NPD)	GC-MS	GC-NPD	GC-MS	GC-ECD/NPD/MS	DETERMINATION				
INTERNAL METHOD	MULTIRESIDUE WITH EXTRELUT	§ 35 LMBG L.00.00-34			REFERENCE METHOD				

**APPENDIX 7. Methods used by participants for determining pesticides.**

MONOCROTOPHOS									
									LAB CODE
D	72	71	70	69	68	67	66	D	SCOPE OF YOUR METHOD
S	D	NA	D	D	D	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-NPD	GC-TSD	GC-MS	LC-MS/MS	GC-MS/MS	GC-MS/MS	GC-MS/MS	GC-MS/MS	GC-MS/MS	CONFIRMATION METHOD
0.03	0.02	0.02	0.01	0.02	0.01	0.02	0.03	RL (mg/Kg)	RECOVERY (%)
76	129	71	77.1	80	80	93	93		SAMPLE WEIGHT (g)
100	25	25	10	10	10	15	15		EXTRACTION SOLVENT
3	1	6. METHANOL	4	5	5	5	5		CLEAN-UP STEP
		O	SPE	LLE + SPE	SPE	SPE	SPE		INTERNAL STANDARD
				TRIPHENYLPHOSPHATE					INJECTION VOLUME (µL)
1	5	1,0:2,0	5	5	10	10	10		INJECTION TYPE
SPLITLESS	SPLITLESS	SPLITLESS	LOOP	LOOP	LOOP	LOOP	LOOP		DETERMINATION
GC-ECD GC-NPD	GC-TSD	GC-NPD	LC-MS/MS	LC-MS/MS	GC-NPD-ECD-FPD(P)	GC-NPD-ECD-FPD(P)	GC-NPD-ECD-FPD(P)	GC-NPD-ECD-FPD(P)	REFERENCE METHOD
	MULTIRESIDUE METHOD FOR ANALYSIS OF PESTICIDES IN FOODSTUFFS P.A. GREVE ED, 1988, MINISTRY OF WELFARE, HEALTH AND CULTURAL AFFAIR, RIJSWICK NETHERLANDS	IN-HOUSE METHOD	MINI-LUKE EXTRACTION	ANASTASSIADES, M. ET AL, J. AOAC INT., 86 (2003), 412 - 431 QUECHERS - METHOD	QUECHERS(LEOTHAY S. ET AL, J. AOAC INT. (2005) 88, 615-629				

**APPENDIX 7. Methods used by participants for determining pesticides.**

MONOCROTOPHOS									
80	79	78	77	76	75	74	73	LAB CODE	SCOPE OF YOUR METHOD
D	D	D		D	D	D	D		
M	M	M	S	M	M	S		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
LC-MS/MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS		CONFIRMATION METHOD	
0.03	0.03		0.01	0.01	0.02	0.01		RL (mg/Kg)	
95	80		119	96	87.5	75		RECOVERY (%)	
10	15		10	10	25	30		SAMPLE WEIGHT (g)	
6	6		DICHLOROMETHANE	6 METHANOL	3	1		EXTRACTION SOLVENT	
O	SPE	GPC	LLE (CHEM ELUT)	SPE	GPC; O	CLEAN-UP STEP			
20	2		1	1	20	50	10	INTERNAL STANDARD	
SPLITLESS	SPLITLESS		SPLITLESS	LC-MS/MS	RHEODYNE	INJECTION VOLUME (µL)		INJECTION TYPE	
LC-MS/MS	GC-ECD GC-NPD GC-MS LC-MS/MS HPLC-FLD		GC-MS (SINGLE-QUAD)	JAOAC INTERN., 86, 1015FF (2003)	HPLC-DAD	HPLC-UV		DETERMINATION	
KLEIN, J., ALDERL (2003) JAOAC INT, 86, 1015-103	INTERNAL METHOD						LOCAL SOP	REFERENCE METHOD	

NO RESULTS

**APPENDIX 7. Methods used by participants for determining pesticides.**

MONOCROTOPHOS										
88	87	86	85	84	83	82	81	LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
D	NA	D	NA	ND	D	D	D		CONFIRMATION METHOD	RL (mg/Kg)
M		M		M	M	M	M		RECOVERY (%)	
GC-MS	LC-MS/MS	GC-MS	LC-MS/MS GC-MS	GC-MS	LC-MS/MS GC-NPD	GC-NPD			SAMPLE WEIGHT (g)	
0.1	0.025	0.025	0.1	0.03	0.03	0.02			EXTRACTION SOLVENT	
87	78	78	80	100	100	87			CLEAN-UP STEP	
25	7.5	7.5	50	10	10	10			INTERNAL STANDARD	
1	4	4	2	5	5	5			INJECTION VOLUME (µL)	
									INJECTION TYPE	
									DETERMINATION	
									REFERENCE METHOD	
										QUECHERS, M. ANASTASSIADES, S. J. LEHOTAY, D. STAUBBAUER, F. J. SCHEIK: J. AOAC INT., 86 (2003) 412-431
										EN NF 12393 IN HOUSE

NO RESULTS

**APPENDIX 7. Methods used by participants for determining pesticides.**

MONOCROTOPHOS									
								SCOPE OF YOUR METHOD	
		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		CONFIRMATION METHOD					
GC-MS	GC-NPD	LC-MS/MS	GC-MS	GC-MS	LC-MS/MS	GC-MS	GC-MS	GC-MS	LAB CODE
D	D	D	D	D	D	D	D	D	89
S	M	M	M	S	S	S	M	M	
GC-MS	GC-NPD	LC-MS/MS	GC-MS	GC-MS	LC-MS/MS	GC-MS	GC-MS	GC-MS	
0.1	0.03	0.005	0.1	0.02	0.01	0.01	0.02	RL (mg/Kg)	
115	72.4		101					RECOVERY (%)	
25	10	80	50	50	50	50	50	SAMPLE WEIGHT (g)	
5	6	2	4	3	3	3	6	EXTRACTION SOLVENT	
CLEAN-UP STEP									
SPE	SPE	GPC	LL	LL	LL	LL	LL		
YES			ALDRIN, DITALIMPHOS	YES				INTERNAL STANDARD	
3	2	1 $\mu$ L PER COLUMN	1	1	1	OCT-25	1	INJECTION VOLUME ( $\mu$ L)	
PTV	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	SPLIT/SPLITLESS	SPLIT/SPLITLESS	INJECTION TYPE	
GC-ECD, GC-NPD, GC-MS	GC-NPD	GC-ECD AND GC-NPD OR GC-ECD ONLY OR GC-NPD ONLY	GC-ECD, GC-NPD, GC-MS	GC-NPD	HPLC-DAD	GC-ECD, FPD, MS HPLC-F-DAD	GC-ECD, FPD, MS HPLC-F-DAD	DETERMINATION	
FILLION ET AL. JOURNAL OG AOAC INTERNATIONAL 78- 5-1995		LC-MS/MS (MULTIMETHOD, 2 COLUMNS)	OFFICIAL METHOD OF ANALYSIS (1990 15TH ED., 985:22, AOAC ARLINGTON VA)	DFG-S8				REFERENCE METHOD	

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>MONOCROTOPHOS</b>										
103	102	101	100	99	98	97	96	LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
NA	D	NA	D	NA	D	NA	NA	NA	CONFIRMATION METHOD	RL (mg/Kg)
	M		M		M				RECOVERY (%)	
	GC-NPD		GC-NPD		LC-MS/MS				SAMPLE WEIGHT (g)	
0.05		0.1		0.02					EXTRACTION SOLVENT	
89		81		121					CLEAN-UP STEP	
30		25		10					INTERNAL STANDARD	CARBENDAZIM D4, METHOMYL D3, PENDIMETHALIN D5
1		1		1					INJECTION VOLUME (µL)	
									INJECTION TYPE	
									DETERMINATION	
					GC-NPD				REFERENCE METHOD	
	SOP MR 405012; EXTRACTION OF RESIDUES OF PLANT-PROTECTING MATERIAL FROM FRUIT AND VEGETABLE SAMPLES BY ETHYL-ACETATE; SOP 308012; CLEAN UP OF EXTRACTS BY GEL PERMEATION CHROMATOGRAPHY				MINISTRY OF WELFARE, HEALTH AND CULTURAL AFFAIRS, NETHERLANDS, ANALYTICAL METHODS FOR RESIDUES OF PESTICIDES IN FOODSTUFFS. MULTIRESIDUE METHOD 1, 3.1. 6TH EDITION, 1996					
										LC-MS MULTI RESIDUE

## **APPENDIX 7. Methods used by participants for determining pesticides.**

MONOCROTOPHOS									
LAB CODE		SCOPE OF YOUR METHOD		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		CONFIRMATION METHOD			
109	108	107	106	D	D	D	D	104	
D	NA	D	D	M	S	M	S		
GC-MS	GC-MS	GC-PFPD	GC-NPD	LC-MS/MS					
0.02	0.01	0.01	0.1	0.005	RL (mg/Kg)				
10	100	25	100	10	RECOVERY (%)				
2	2	6 ETHYLACETATE/DICHLOROMETHANE	4	6 (METHANOL)	SAMPLE WEIGHT (g)				
				6 (METHANOL)	EXTRACTION SOLVENT				
					CLEAN-UP STEP				
					DIATOMACEOUS EARTH				
					ALDRIN	INTERNAL STANDARD			
2	1	1	1	1	25	INJECTION VOLUME (µL)			
					SPLITLESS	INJECTION TYPE			
GC-ECD GC-NPD GC-FPD GC-MS	GC-ECD-FCD; GC-NPD; GC-MS(SINGLE-QUAD)	GC-PFPD NPD GC-MS	GC-NPD GC-ECD	LC-MS/MS	DETERMINATION				
		MULTIRESIDUE METHOD: AOAC 985.22/90 MODIFIED	APPLICATION NOTE BY WATERS	REFERENCE METHOD					
		S19 - DFG							

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>MONOCROTOPHOS</b>							
115	114	113	112	111	110	LAB CODE	SCOPE OF YOUR METHOD
D	D	D	NA	D	D		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
S	S	S		S	M		CONFIRMATION METHOD
GC-MS	LC/MS/MS	GC-NPD	LC-MS/MS	GC-NPD	RL (mg/Kg)		RECOVERY (%)
0.01	0.01	0.05	0.03	0.01			SAMPLE WEIGHT (g)
	96.5	89	99.7				EXTRACTION SOLVENT
50	20	30	50	50	6		CLEAN-UP STEP
3	6	1	6 METHANOL				INTERNAL STANDARD
				SPE	GPC		BROMOPHOS-ETHYL
							INJECTION VOLUME ( $\mu$ L)
							INJECTION TYPE
ON COLUMN							
GC-ECD GC-PFPD GC-MS	LC-MS/MS	GC-NPD	LC- MS/MS	GC-ECD AND GC-NPD		DETERMINATION	
NFEN12393	WATERS A MULTIRESIDUE LC-MS/MS METHOD FOR PESTICIDES	SOP MR 405012: EXTRACTION OF RESIDUES OF PLANT-PROTECTING MATERIAL FROM FRUIT AND VEGETABLE SAMPLES BY ETHYL-ACETATE	VALIDATED IN HOUSE METHOD NACH MULTI-METHOD, KLEIN UND ALDER -DFG WORKSHOP PRESENTED	MULTI-RESIDUE-METHOD DFG S 19		REFERENCE METHOD	

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>MONOCROTOPHOS</b>													
128	127	126	125	124	123	122	121	120	119	118	117	116	LAB CODE
NA	NA	NA	NA	NA	NA	NA	NA	D	NA	NA	ND	SCOPE OF YOUR METHOD	
								S	S			QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
								GC-MS	GC-NPD			CONFIRMATION METHOD	
									0.03		0.03	RL (mg/Kg)	
												RECOVERY (%)	
									78			SAMPLE WEIGHT (g)	
								10	5		15	EXTRACTION SOLVENT	
								3	3		6	CLEAN-UP STEP	
								O				GPC	
												INTERNAL STANDARD	
												INJECTION VOLUME (µL)	
									1	2	1	INJECTION TYPE	
								AUTO	SPLITLESS			GC-ECD GC-NPD GC-MS(TD)	
												DETERMINATION	
												RAPPORTO ISTISAN 97/23	
												REFERENCE METHOD	
												ISS B6	

NO RESULTS

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>PROCYMIDONE</b>							
<b>5</b>	<b>4</b>	<b>3</b>	<b>2</b>	<b>1</b>	<b>LAB CODE</b>	<b>SCOPE OF YOUR METHOD</b>	
D	D	D	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
M	M	M	S	M	GC-MS	CONFIRMATION METHOD	
GC-MS	GC-NPD	GC-MS/MS	GC-MS	GC-MS	GC-MS	RL (mg/Kg)	
0.02	0.015	0.02	0.010	0.02		RECOVERY (%)	
		84.5	91			SAMPLE WEIGHT (g)	
10	10	15	15	10		EXTRACTION SOLVENT	
5	5	1	4	1		CLEAN-UP STEP	
SPE	SPE	NO		SPE		INTERNAL STANDARD	
MIREX/TPP		TRIPHENYLPHOSPHATE ANTHRACENTE			YES	INJECTION VOLUME ( $\mu$ L)	
2	2	10	1	2		INJECTION TYPE	
SPLIT/SPLITLESS	SPLIT/SPLITLESS	SPLIT	SPLITLESS	SPLIT/SPLITLESS		DETERMINATION	
GC-ECD/ GC-NPD	GC-MS	GC-MS/MS	GC-ECD	GC-MS		REFERENCE METHOD	
M. ANASTASSIADES ET AL. JAOAC 86, 412-431 (2003)	QUECHERS; ANASTASSIADES					LUKE, M.A., FROBERG, J.E., DOOSE, G.M., MASUMOTO, H.T. (1981): IMPROVED MULTIRESIDUE GAS CHROMATOGRAPHIC DETERMINATION OF ORGANOPHOSPHORUS, ORGANONITROGEN, AND ORGANOHALOGEN PESTICIDES IN PRODUCE, USING FLAME PHOTOMETRIC AND ELECTROLYTIC CONDUCTIVITY DETECTORS. J. ASSOC. OFF. ANAL. CHEM. 64(5): 1187-1195	
						IN HOUSE	

**APPENDIX 7. Methods used by participants for determining pesticides.**

PROCYMICONE									
								LAB CODE	
								SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
								CONFIRMATION METHOD	
12	11	10	9	8	7	6			
D	D	D	D	D	D	D			
M	S	M	M	M	S	M			
MS-TD, GC-ECD	GC-MS	GC-MS(TD)	GC-MS	GC-NPD80	GC-MS	GC-MS			
0.02	0.01	0.02	0.025	0.01	0.01	0.02	RL (mg/Kg)		
77	96	83	98	105			RECOVERY (%)		
50	30	25	15	30	10	15	SAMPLE WEIGHT (g)		
1	1	2	4	1	6 (DICHLOROMETHANE)	4	EXTRACTION SOLVENT		
								CLEAN-UP STEP	
								GPC	
								INTERNAL STANDARD	
								INJECTION VOLUME (µL)	
1, 2, 5	1	2	2	2	4	2			
								INJECTION TYPE	
								SPLITLESS	
								GC-MS	
MS-TD, LC-MS, GC-FPD, GC-ECD	GC-ECD	GC-ECD, GC-NPD, GC-MS(TD)	GC-MS(TD)	GC-MS	GC-ECD; GC-NPD(80)	GC-MS		DETERMINATION	
								ANALYTICAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS (1996), MINISTRY OF HEALTH, WELFARE AND SPORTS, THE HAGUE (RUSWILK), THE NETHERLANDS	
								RAPPORI ISTISAN 97/23	
								REFERENCE METHOD	

**APPENDIX 7. Methods used by participants for determining pesticides.**

PROCYMICONE									
20	19	18	17	16	15	14	13	LAB CODE	SCOPE OF YOUR METHOD
D	D	D	D	D	D	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
S	M	S	M	M	S	S	M	M	CONFIRMATION METHOD
GC-NPD	GC-MS(ITD)	GC-MS	GC-MS	GC-MS/MS	GC-MS	GC-MS	GC-MS/MS	GC-MS/MS	RL (mg/Kg)
0.02	0.02	0.02	0.01	0.02	0.02	0.02	0.02	0.02	RECOVERY (%)
90	80	123			117		81.9		SAMPLE WEIGHT (g)
100	75	75	15	15	25	15	15		EXTRACTION SOLVENT
3	1	1	4	4	1	4	1		CLEAN-UP STEP
O	GPC	GPC	LL		GPC				INTERNAL STANDARD
	DITALIMPHOS	ALDRIN					TPP (QC)		INJECTION VOLUME (µL)
1	2	2	10	10	2	2	10		INJECTION TYPE
AUTOSAMPLER	SPLITLESS	PULSED SPLITLESS	LVI AUTOSAMPLER	LVI SPLITLESS	SPLIT/ SPLITLESS				
GC-ECD	GC-ECD	GC-MS	GC-MS	GC-MS/MS	GC-ECD	GC-ECD	GC-MS(ITD)	DETERMINATION	REFERENCE METHOD
					MINI LUKE	LOCAL SOP NO. MR405012	MULTIRESIDUES		

**APPENDIX 7. Methods used by participants for determining pesticides.**

27	26	25	24	23	22	21	LAB CODE
D	D	D	D	D	D	D	SCOPE OF YOUR METHOD
S	M	M	M	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
							CONFIRMATION METHOD
GC-ECD	GC-MS	GC-NPD	GC-MS	GC-MS	GC-MS/MS	GC-MS/MS	GC-MS/MS
0.02	0.01	<0.02	0.02	0.01	0.28	0.01	RL (mg/Kg)
77	94	101.7		88.4			RECOVERY (%)
30	25	10	30	5	50	10	SAMPLE WEIGHT (g)
1	2	6	1	5	6	1	EXTRACTION SOLVENT
							CLEAN-UP STEP
GPC	GPC	SPE	GPC	LLE (ISOPROPYL ETHER)			
				FENCHLORPHOS			INTERNAL STANDARD
DITALIMPHOS	YES						INJECTION VOLUME (µL)
1	2	2	1 mg SAMPLE	50	2	10	
SPI	SPLITLESS	SPLITLESS	TPOCI	AUTOMATIC TDS SAMPLER	SPLITLESS	LVI	INJECTION TYPE
GC-ECD	GC-ECD, GC-NPD, GC-MS(ITD)	GC-MS	GC-ECD	TDS-IN LINE WITH GC-MS (SINGLE-QUAD) TOTAL ION DETECTION	GC-ECD AND GC-NPD	GC-MS/MS	DETERMINATION
HUNGARIAN CENTRAL SOP NO.: 405012 M1				SOP NO.: MR 405012 M1; 308 M2	INTERNAL METHOD		REFERENCE METHOD

## **APPENDIX 7. Methods used by participants for determining pesticides.**

PROCYMICIDE									
TEST METHOD		TEST CONC. (µg/L)		TEST VOLUME (mL)		INSTRUMENTATION		TEST CONDITIONS	
TEST	CONC.	TEST	CONC.	TEST	CONC.	TEST	CONC.	TEST	CONC.
GC-MS	35	34	33	32	31	30	29	28	LAB CODE
D	D	D	D	D	D	D	D	D	SCOPE OF YOUR METHOD
M	M	M	S	S	S	M	S	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-MS/MS	GC-MS/MS	GC-MS/MS	GC-MS	MS	DIFFERENT COLUMN	GC-MS/MS	GC-MS	GC-MS	CONFIRMATION METHOD
0.02	0.02	0.02	0.02	0.02	0.01	0.02	0.02	0.02	RL (mg/Kg)
95			95	99	98	106	88		RECOVERY (%)
20	1	15	25	10	10	15	50		SAMPLE WEIGHT (g)
3	4	1	2	6	1	4	6		EXTRACTION SOLVENT
									CLEAN-UP STEP
				GPC	O	SPE	SPE	SPE	
									INTERNAL STANDARD
					DICHLOFENTHION		YES		
2	10	10	1	1	1	10	1	1	INJECTION VOLUME (µL)
SPLITLESS	AUTO	LVI	SPLITLESS	SPLITLESS	SPLITLESS	ON COLUMN	ON COLUMN	ON COLUMN	INJECTION TYPE
GC-ECD, NPD	GC-MS/MS	GC-MS/MS (ION TRAP)	GC-MSD	GC-ECD GC-NPD	GC-MS/MS	GC-MS/MS	GC-MS/MS	GC-MS/MS	DETERMINATION
EN 12393-1, 2, 3	PESTICIDES MS/MS EI-Cl	\$35 LMBG 00.00-34	ISTISAN 97/23	RAPP ISTISAN 97/23	MINI LUKE				REFERENCE METHOD

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

PROCYMICONE						
						LAB CODE
D	46	45	44	43	42	SCOPE OF YOUR METHOD
M	D	D	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-ECD	GC-MS	GC-MS	NONE	GC-MS	GC-MS	CONFIRMATION METHOD
0.02	0.03	0.01	0.01	0.01	0.01	RL (mg/Kg)
79	97		101			RECOVERY (%)
15	25	50	10	10	10	SAMPLE WEIGHT (g)
4	1	2	5	5	5	EXTRACTION SOLVENT
(PULSED) SPLITLESS/(TRACK OVEN) ON-COLUMN	GPC	GPC	SPE	LLE		CLEAN-UP STEP
						INTERNAL STANDARD
3 / 1	1	2	4	4	1	INJECTION VOLUME ( $\mu$ L)
MULTI-RESIDUE METHOD/PESTICIDES AMENABLE TO GAS CHROMATOGRAPHY (3,1,2 ACETONE-PARTITION FOR FRUITS, VEGETABLES AND POTATOES) 6TH EDITION MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORT. THE NETHERLANDS	GC-NPD / GC-ECD	GC-ECD OR GC-NPD OR GC-FFD	GC-MS (SINGLE QUAD)	GC-MS EI	GC-ECD GC-MS	DETERMINATION
		§ 35 LIMBG 00.00-34		ANASTASSIADES ET AL. JAOAC INT.: 86(82003) 412-431, MODIFIED	M. ANASTASSIADES ET AL., J. AOAC INT. 86 (2), 412-431, (2003)	REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

PROCYMICONE									
								LAB CODE	
								SCOPE OF YOUR METHOD	
						QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX			
						CONFIRMATION METHOD			
54	53	52	51	50	49	48	47		
D	D	D	NA	D	D	D	D		
M	S	S		S	S	M	S		
GC-MS	GC-ECD	GC-MS	GC-ECD	GC-MS	GC-ECD	GC-MS	GC-ECD		
0.02	0.02	0.02		0.02	0.01	0.01	0.02		
88				107					
18	15	10		20	50	25	10		
1	6	5		6	1	2	5		
GPC	GPC	O			LL	GPC	SPE		
					NITROFEN				
1	1	1.5			2	2	3		
SPLITLESS, ON COLUMN	SPLIT/SPLITLESS	SPLIT/SPLITLESS		ON COLUMN	SPLITLESS	PTV	SPLIT/SPLITLESS		
GC-ECD, FPD,NPD	GC-ECD, GC-NPD, GC-FFD, GC-MS, HPLC-FLD, HPLC-JV	GC-ECD, GC-NPD, GC-MS		GC-ECD	GC-FPD, GC-NPD, GC-MS(IRD)	GC-MS	GC-ECD, GC-NPD, GC-MS (SINGLE QUAD)		
EN 12393-1,2,3	INTERNAL METHOD	QUECHERS				METHOD R'EN 12393-2:1999	DFG S 19	M. ANASTASSIADES, S. J. LEHOTAY, D. STAIBBAHER, F. J. SCHEINCK : J. AOAC INT. 86 (2003) 412-431	REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>PROCYMIDONE</b>									
61	60	59	58	57	56	55	LAB CODE		
D	D	D	D	D	D	D	D	D	SCOPE OF YOUR METHOD
M	M	M	M	M	S	M	ANALYSTS STANDARDS IN SOLVENT OR IN MATRIX	ANALYSTS STANDARDS IN SOLVENT OR IN MATRIX	CONFIRMATION METHOD
GC-MS	GC-MS	GC-MS	GC-ECD-NPD	GC-MS	GC-ECD GC-MS	GC-ECD	GC-ECD	GC-ECD	CONFIRMATION METHOD
0.01	0.02	0.01	0.05	0.01	1.65	0.005	RL (mg/Kg)	RL (mg/Kg)	RECOVERY (%)
50	15	10	105	105	103	83.8	SAMPLE WEIGHT (g)	SAMPLE WEIGHT (g)	EXTRACTION SOLVENT
3	1	5	4	1	1	2	CLEAN-UP STEP	CLEAN-UP STEP	INTERNAL STANDARD
GPC	GPC	O	GPC	GPC	GPC	GPC	GPC	GPC	INTERNAL STANDARD
ISODRIN	3	PCB 119 (ECD) TRIFLURALIN (GC-NPD)					GC 1 µL HPLC 20 µL	GC 1 µL HPLC 20 µL	INJECTION VOLUME (µL)
2	1	2	1	1	1	1	SPLIT/SPLITLESS	SPLIT/SPLITLESS	INJECTION TYPE
PTV	SPLIT/SPLITLESS	SPLITLESS	SPLIT/SPLITLESS	PULSED SPLITLESS	DIRECT	SPLIT/SPLITLESS	GC-ECD/NPD	GC-NPD	DETERMINATION
GC-MS	GC-ECD/NPD/MS	GC-MS	GC-ECD-NPD	GC-ECD/NPD	GC-NPD	GC-ECD, GC-NPD, GC-FPD, HPLC-UV, LC-MS/MS	MODULARE MULTIMETHODE S19	MODULARE MULTIMETHODE S19	REFERENCE METHOD
DFG S19 (ASU §35 L 000034 00.00-34)	§35 L 000034	ANAL METHODS FOR PESTICIDES RESIDUES IN FOODSTUFFS (NL)							

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>PROCYMICONE</b>									
67	66	65	64	63	62	LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	CONFIRMATION METHOD
D	D	D	D	D	D		D	M	GC-MS
S	M	M	M	M	M			M	GC-MS/MS
GC-MS	GC-MS/MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS		GC-MS	GC-MS
0.02	0.05	0.02	0.02	0.02	0.02	0.02		RL (mg/Kg)	RL (mg/Kg)
85	85	81	81	81	101			RECOVERY (%)	RECOVERY (%)
90	15	5	5	5	75			SAMPLE WEIGHT (g)	SAMPLE WEIGHT (g)
3	5	1	6	ACETONE FOLLOWED BY CYCLOHEXANE AND ETHYLACETATE	1			EXTRACTION SOLVENT	EXTRACTION SOLVENT
GPC + SPE	SPE	GPC	GPC	GPC	GPC	CLEAN-UP STEP			
ALDRIN	AZOBENZENE+RONNELL TRIPHENYLPHOSPHATE	TPP	TPP	TPP	TPP	INTERNAL STANDARD			
1	10	2	0.5	1	2	INJECTION VOLUME (µL)			
SPLIT/SPLITLESS	ON COLUMN	SPLITLESS				INJECTION TYPE			
GC - ECD	GC-MS(IRD)	GC-(ECD+FID+NPD)	GC-NPD	GC-MS	GC-MS	DETERMINATION			
OFFICIAL METHOD L 00/00-34; § 35 LMBG	QUECHERS (LEOTHAY, S. ET AL., J.A.O.A.C. INT. (2005) 88, 615-629	INTERNAL METHOD	MULTIRESIDUE WITH EXTRELUT	§ 35 LMBG L 00/00-34		REFERENCE METHOD			

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>PROCYMIDONE</b>							
						LAB CODE	
						SCOPE OF YOUR METHOD	
				QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX			
						CONFIRMATION METHOD	
73	D	72	D	71	D	70	69
S	S	M	M	M	D	D	68
GC-MS	GC-ECD	GC-ECD	GC-MS/MS	GC-MS	GC-MS	GC-MS	GC-MS
0.05	0.02	0.01	0.02	0.02	0.02	0.02	RL (mg/Kg)
84	87	103			97	92.2	RECOVERY (%)
30	100	25	15	25	25	20	SAMPLE WEIGHT (g)
1	3	6	5	1	1	4	EXTRACTION SOLVENT
						CLEAN-UP STEP	
						GPC	
						INTERNAL STANDARD	
						INJECTION VOLUME ( $\mu$ L)	
				10		1.0-2.0	2
						INJECTION TYPE	
				SPLITLESS		SPLITLESS	SPLITLESS
						DETERMINATION	
				GC-ECD/ITSD/PFPD GC-MS/MS		GC-NPD, GC-ECD	GC-ECD/NPD
						MULTIRESIDUE METHOD 1. ANALYTICAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS, SIXTH EDITION	
						MINI-LUKE EXTRACTION	
						REFERENCE METHOD	

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>PROCYMICONE</b>							
87	86	85	84	83	82	81	LAB CODE
D	D	D	D	D	D	D	SCOPE OF YOUR METHOD
M	M	M	M	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-MS	GC-MS(TD)	GC-MS	GC-MS	GC-MS	GC-ECD	GC-ECD	CONFIRMATION METHOD
0.02	0.025	0.02	0.02	0.02	0.007	0.007	RL (mg/Kg)
96		80		108		105	RECOVERY (%)
50	15	25	50	30	30	10	SAMPLE WEIGHT (g)
6 ETHYLACETATE/ DICHLOROMETHANE (80/20)	4	4	2	1	1	5	EXTRACTION SOLVENT
SPE-FLORISIL							CLEAN-UP STEP
HCB							INTERNAL STANDARD
1	1	1	1	3	3	1	INJECTION VOLUME ( $\mu$ L)
SSL	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS AND ON COLUMN	INJECTION TYPE
GC-ECD	GC-ECD	GC-MS (SINGLE-QUAD)	GC-MS (SINGLE-QUAD)	GC-MS	GC-MS	GC-ECD, GC-NPD, GC-FPD, GC-MS, HPLC-FID, HPLC-DAD	DETERMINATION
ACCORDING TO EN 12399- 1:1999	SAR-2-04OC	EN NF 12393		QUECHERS, M. ANASTASSIADES, S. J. LEHOTAY, D. STAIBBAUER, F. J. SCHENK: J. AOAC INT., 86 (2003) 412-431			REFERENCE METHOD
NO RESULTS							

## **APPENDIX 7. Methods used by participants for determining pesticides.**

PROCYRIDONE									
94	93	92	91	90	89	88	LAB CODE		
D	D	D	D	D	D	D	SCOPE OF YOUR METHOD		
M	M	M	S	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		
GC-NPD	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	CONFIRMATION METHOD		
0.02	0.005	0.01	0.02	0.01	0.02	0.01	RL (mg/Kg)		
96.1		107			85	104	RECOVERY (%)		
10	80	50	50	50	45	25	SAMPLE WEIGHT (g)		
4	2	4	3	2	4	1	EXTRACTION SOLVENT		
SPE	GPC	LLE	LLE	GPC	GPC	GPC	CLEAN-UP STEP		
							INTERNAL STANDARD		
		Aldrin, Ditalimphos	YES				INTERNAL STANDARD		
2	1µL PER COLUMN	1	1	1	1	1	INJECTION VOLUME (µL)		
SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	SPLIT-SPLITLESS	SPLITLESS	SPLITLESS	INJECTION TYPE		
GC-ECD	GC-ECD AND GC-NPD OR GC-ECD OR GC-NPD	GC-ECD, GC-NPD, GC-MS	GC-NPD	GC-NPD	GC-ECD, FPD, MS HPLC-F-DAD	GC-ECD	DETERMINATION		
(SINGLE-QUAD)	OFFICIAL METHOD OF ANALYSIS (1990) 15TH ED., 985-22, AOAC ARLINGTON VA	DFG-88	IN-HOUSE	REFERENCE METHOD					

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>PROCYMIDONE</b>							
100	99	98	97	96	95	LAB CODE	SCOPE OF YOUR METHOD
D	D	D	NA	D	D		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
M	M	M		S	S		CONFIRMATION METHOD
GC-ECD	GC-MS	GC-MS		GC-MS	GC-MS		RL (mg/Kg)
0.04	0.02	0.02		0.02	0.02		RECOVERY (%)
84	95			80	87		SAMPLE WEIGHT (g)
25	20	10		25	25		EXTRACTION SOLVENT
6	4	1		2	5		CLEAN-UP STEP
O	GPC	GPC		GPC	SPE		INTERNAL STANDARD
				ISODRIN, CHINOMETHIONAT	YES		INJECTION VOLUME (µL)
2	1	1		1	3		INJECTION TYPE
SPLITLESS	PULSED SPLITLESS	ON COLUMN		SPLIT/SPLITLESS	PTV		
GC-ECD	GC-MS	GC-MS		GC-ECD GC-NPD; GC-MS (SINGLE-QUAD)	GC-ECD, GC-NPD, GC-MS	DETERMINATION	
MINISTRY OF WELFARE, HEALTH AND CULTURAL AFFAIRS, NETHERLANDS. ANALYTICAL METHODS FOR RESIDUES OF PESTICIDES IN FOODSTUFFS, MULTIRESIDUE METHOD , SUBMETHOD 1, PART I-3, 5TH EDITION, 1988	GC-MS MULTIRESIDUE			SOP 5.4.CH.1 (ACCORDING TO EN 12393/1-3; AND 35 LIMB L00.0034	FILLION ET AL, JOURNAL OF AOAC INTERNATIONAL 78-5-1995	REFERENCE METHOD	

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

PROCYMICIDONE									
113	112	111	110	109	108	107	LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
D	D	D	D	D	NA	D			
S	S	M	M	M		M			
GC-NPD	GC-MS	GC-ECD	GC-ECD	GC-MS		GC-MS		CONFIRMATION METHOD	
0.02	0.01	0.02	0.01	0.02		RL (mg/Kg)			
101	110	105.6				101		RECOVERY (%)	
30	50	50	50	10	10	100		SAMPLE WEIGHT (g)	
1	6	2	6	2	2	2		EXTRACTION SOLVENT	
GPC	SPE	GPC	GPC	GPC		GPC		CLEAN-UP STEP	
								INTERNAL STANDARD	
			BROMOPHOS-ETHYL						
1		1	1	2	2	1		INJECTION VOLUME ( $\mu$ L)	
SPLITLESS	SPLITLESS	OC; SS	SPLITLESS					INJECTION TYPE	
GC-ECD	GC/ECD	GC-ECD, GC-NPD	GC-ECD AND GC-NPD	GC-ECD GC-NPD GC-FPD GC-MS		GC-ECD-ECD; GC-NPD; GC-MS (SINGLE-QUAD)		DETERMINATION	
SOP MR 405012: EXTRACTION OF RESIDUES OF PLANT-PROTECTING MATERIAL FROM FRUIT AND VEGETABLE SAMPLES BY ETHYL ACETATE; SOP 308M2; CLEAN UP OF EXTRACTS BY GEL PERMEATION CHROMATOGRAPHY	ISTISAN 97/23	\$35 LMBG; L 00-00-34; L 00-00-37	MULTI-RESIDUE-METHOD DFG S 19					REFERENCE METHOD	

## **APPENDIX 7. Methods used by participants for determining pesticides.**

PROCYMICONE									
LAB CODE		SCOPE OF YOUR METHOD		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX					
121	120	119	118	117	116	115	114		
D	D	D	D	D	D	D	D		
S	S	S	S	M	S	S	S		
GC-ECD	GC-MS	GC-ECD	GC-MS	GC-MS	GC-MS/GC-ECD	GC-MS	GC-MS	CONFIRMATION METHOD	SCOPE OF YOUR METHOD
<0.020	0.002	0.02	0.02	0.03	0.01	0.01	0.01	RL (mg/Kg)	SCOPE OF YOUR METHOD
85	103.7	87	87	90	90	90	90	RECOVERY (%)	SCOPE OF YOUR METHOD
100	100	10	5	40	15	50	50	SAMPLE WEIGHT (g)	SCOPE OF YOUR METHOD
3, 1, CYCLOHEXANE, N-HEXANE, IZO-OCTANE, TOLUENE	3	3	3	1	6	3	6	EXTRACTION SOLVENT	SCOPE OF YOUR METHOD
GPC, SPE	GPC	O			GPC	SPE	SPE	CLEAN-UP STEP	SCOPE OF YOUR METHOD
								INTERNAL STANDARD	SCOPE OF YOUR METHOD
2	1	1	2	1	1	1	1	INJECTION VOLUME (µL)	SCOPE OF YOUR METHOD
SPLITLESS	SPLIT	AUTO	SPLITLESS	SPLITLESS	ON COLUMN	SPLITLESS	SPLITLESS	INJECTION TYPE	SCOPE OF YOUR METHOD
GC-ECD	GC-ECD GC-NPD	GC-ECD GC-NPD	GC-MS	GC-ECD / GC-NPD, GC-MS(HTD)	GC-ECD / GC-PFPD – GC-MS	GC-MS	GC-MS	DETERMINATION	SCOPE OF YOUR METHOD
PN-EN 12393-1,2,3:2000	PN-EN 12393-1,2,3:2000	ISS B6		RAPPORTO ISTISAN 97/23	NFEN12393			REFERENCE METHOD	SCOPE OF YOUR METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>PROCYMIDONE</b>							
							LAB CODE
128	127	126	125	124	123	122	
D	D	D	D	D	NA	D	SCOPE OF YOUR METHOD
M	S	S	S	S	S	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-MS	GC-ECD	GC-ECD	GC-ECD	GC-ECD	GC-ECD	GC-ECD	CONFIRMATION METHOD
0.02	0.01	0.0012	0.0025	0.02	0.02	0.02	RL (mg/Kg)
20 G / 5G	100	25	20	86	86	86	RECOVERY (%)
1	3	3	6	10	10	10	SAMPLE WEIGHT (g)
							EXTRACTION SOLVENT
LLE	LLE	GPC	LLE	O	O	O	CLEAN-UP STEP
							INTERNAL STANDARD
							INJECTION VOLUME ( $\mu$ L)
							INJECTION TYPE
AUTOSAMPLER	SPLITLESS	PTV-SOLVENT VENT	ON COLUMN				
							DETERMINATION
	GC-ECD	GC-NPD	GC-ECD				
GC-ECD, GC-FPD, GC-MSD, GC-MS(TD)							REFERENCE METHOD
MODIFIED LUKE	PN-EN 12393: 2000	PN-EN 12393	GC-MS				

NO RESULTS

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>PYRIMETHANIL</b>										
<b>15</b>	<b>14</b>	<b>13</b>	<b>12</b>	<b>11</b>	<b>10</b>	<b>9</b>	<b>8</b>	<b>7</b>	<b>LAB CODE</b>	<b>SCOPE OF YOUR METHOD</b>
D	NA	D	D	D	D	D	D	NA	NA	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
S	M	M	M	S	M	S	M			CONFIRMATION METHOD
GC-MS	GC-MS/MS	MS-ITD				GC-MS(ITD)	GC-MS			RL (mg/Kg)
0.05	0.02	0.05	0.05	0.005	0.005	0.01	0.01			RECOVERY (%)
97	77.9	87	80	84	97	97	86			SAMPLE WEIGHT (g)
25	15	50	30	25	15	15	30			EXTRACTION SOLVENT
1	1	1	1	2	2	4	1			CLEAN-UP STEP
				GPC	GPC	LLE ACETONE FOLLOWED BY DICHLOROMETHANE AND PETROLEUM ETHER	O			INTERNAL STANDARD
				O						INJECTION VOLUME ( $\mu$ L)
										INJECTION TYPE
										DETERMINATION
										REFERENCE METHOD
										IN HOUSE SOP
										LOCAL SOP NO. MR405012

## **APPENDIX 7. Methods used by participants for determining pesticides.**

PYRIMETHANIL											
LAB CODE		SCOPE OF YOUR METHOD		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX							
24	23	22	21	20	19	18	17	16	15	14	13
D	D	D	D	D	D	D	NA	D	NA	SC	SC
S	M	M	M	S	M	S	M	M	M	CONFIRMATION METHOD	
LC-MS/MS	HPLC-DAD	GC-MS	GC-MS/MS	GC-NPD	GC-MS/ITD	GC-MS	GC-MS/MS	GC-MS/MS	GC-MS/MS	CONFIRMATION METHOD	
0.05	0.01	0.02	0.01	0.05	0.02	0.03	0.05	0.05	0.05	RL (mg/Kg)	
120.0		71.6		89	75	82				RECOVERY (%)	
30	5	50	10	100	75	75	15	15	15	SAMPLE WEIGHT (g)	
1	5	6	1	3	1	1	4	4	4	EXTRACTION SOLVENT	
										CLEAN-UP STEP	
	SPE FLORISIL	LLE (TOLUENE)	O	GPC	GPC					INTERNAL STANDARD	
					DITALIMPHOS	ALDRIN				INTERNAL STANDARD	
1 mg SAMPLE	20	1.5	10	2	2	2	10	10	10	INJECTION VOLUME (µL)	
AUTOSAMPLER	AUTOMATIC	SPLITLESS	LVI	MANUAL	SPLITLESS	PULSED SPLITLESS	LVI SPLITLESS			INJECTION TYPE	
LC-MS/MS	HPLC-DAD	GC-NPD	GC-MS/MS	GC-NPD	GC-ECD	GC-MS	GC-MS/MS	GC-MS/MS	GC-MS/MS	DETERMINATION	
										REFERENCE METHOD	
										SOP NO.: MR 405012 M1 INTERNAL METHOD	

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>PYRIMETHANIL</b>									
<b>32</b>	<b>31</b>	<b>30</b>	<b>29</b>	<b>28</b>	<b>27</b>	<b>26</b>	<b>25</b>	<b>LAB CODE</b>	<b>SCOPE OF YOUR METHOD</b>
D	D	D	D	D	D	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
S	S	S	M		S	M	M	M	CONFIRMATION METHOD
GC-MS	MS	DIFFERENT COLUMN	GC-MS/MS	GC-MS	GC-MS/ITD	GC-MS	GC-MS	GC-MS	RL (mg/Kg)
0.1	0.05	0.01	0.05	0.05	0.05	0.01	<0.02		RECOVERY (%)
58	89		97	75		82	93		SAMPLE WEIGHT (g)
25	10	10	15	50	30	25	10		EXTRACTION SOLVENT
2	6	1	4	6	1	2	6		CLEAN-UP STEP
GPC	O	GPC		SPE	GPC	SPE			INTERNAL STANDARD
		DICHLORENTHION		YES	DITALIMPHOS	YES			INJECTION VOLUME (µL)
1	1	1	10	1	20	2	2		INJECTION TYPE
SPLITLESS	SPLITLESS	SPLITLESS	ON COLUMN	RHEODYNE INJECTOR	SPLITLESS	SPLITLESS			DETERMINATION
GC-MS	GC-ECD GC-NPD	GC-MS/MS GC-NPD	GC-MS/ITD	HPLC-UV	GC-ECD, GC-NPD, GC-MS/ITD	GC-MS			REFERENCE METHOD
\$35 LMBG 00.00-34	ISTISAN 97/23	RAPP ISTISAN 97/23	MINI LUKE		HUNGARIAN CENTRAL SOP NO.: 405012 M1				

**APPENDIX 7. Methods used by participants for determining pesticides.**

PYRIMETHANIL									
									LAB CODE
									SCOPE OF YOUR METHOD
39	38	37	36	35	34	33			
D	D	D	D	D	D	D			
M	M	M	S	M	M	M			QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
LC-MS/MS	GC-MS	LC-MS/MS	GC-M/S	GC-MS	GC-MS/MS	GC-MS/MS			CONFIRMATION METHOD
0.01	0.05	0.005	0.05	0.02	0.05	0.01			RL (mg/Kg)
71			88	87					RECOVERY (%)
10	10	10	50	20	5	15			SAMPLE WEIGHT (g)
6 (METHANOL/WATER)	5	5	1	3	4	1			EXTRACTION SOLVENT
									CLEAN-UP STEP
			O						
			DIMETHOATE D6						INTERNAL STANDARD
20	2	20	1	2	10	10			INJECTION VOLUME (µL)
			SPLITLESS	SPLITLESS	AUTO	LVI			INJECTION TYPE
LC-MS/MS	GC-ECD GC-NPD GC-MS	LC-MS/MS (TRIPLE-QUAD)	GC-NPD GC-FPD GC-M/S (SINGLE-QUAD.)	GC-ECD, NPD	GC-MS/MS	GC-MS/MS (ON TRAP)			DETERMINATION
LC-MS/MS (BFR)			ANASTASSIADES, JOURNAL OF AOAC INT. VOLT 86 NO 2, 2003		EN 12393-1, 2, 3	PESTICIDES MS/MS EI-Cl			REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>PYRIMETHANIL</b>							
							LAB CODE
D	45	44	43	42	41	40	
S	D	D	M	D	D	D	SCOPE OF YOUR METHOD
GC-MS	M	M	M	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
0.04	GC-MS	LC-MS/MS	GC-MS	GC-MS	GC-MS	GC-MS	CONFIRMATION METHOD
90	0.01	0.01	0.01	0.02	0.02	0.05	RL (mg/Kg)
25	50	10	10	10	20	15	RECOVERY (%)
1 (IN PRESENCE OF NaOH)	2	5	5	3	3	4	SAMPLE WEIGHT (g)
GPC	LL	SPE	LL				EXTRACTION SOLVENT
RHEODYNE							CLEAN-UP STEP
HPLC - DAD							INTERNAL STANDARD
CLEAN UP AS IN EN 12393 P METHOD							INJECTION VOLUME (µL)
ANASTASSIAIDES ET AL. JAOAC INT. 86(2), 412-431, MODIFIED	LC-MS/MS	LC-MS/MS(ESI+)	LC-MS/MS	GC-ECD, GC-NPD	GC-NPD, GC-ECD, GC-MS(ITD)	GC-NPD	DETERMINATION
M. ANASTASSIAIDES ET AL., J. AOAC INT. 86(2), 412-431, (2003)							ANALYTICAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS, 6TH ED, 1986, GENERAL INSPECTORATE FOR HEALTH PROTECTION, MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS
							REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>PYRIMETHANIL</b>									
								SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
CONFIRMATION METHOD									
GC-MS/MS	GC-MS	GC-MS	GC-CMS	GC-MS/ITD)	LC-MS/MS	GC-NPD			LAB CODE
0.02	0.05	0.05	0.05	0.02	0.01	0.05			46
100			84				RECOVERY (%)		
10	15	10	20	50	10	10	SAMPLE WEIGHT (g)		
5	6	5	6	1	6	5	EXTRACTION SOLVENT		
O	GPC	O					CLEAN-UP STEP		
			LL	SPE			INTERNAL STANDARD		
			DICHLOBENIL				INJECTION VOLUME (µL)		
5	1	1.5		2	2	20	4		
LVI	SPLIT/ SPLITLESS	SPLIT/ SPLITLESS	ON COLUMN	SPLITLESS		SPLIT/SPLITLESS	INJECTION TYPE		
GC-MS/MS	GC-ECD, GC-NPD, GC-FPD, GC-MS, HPLC-FID, HPLC-UV	GC-ECD, GC-NPD, GC-MS	GC-NPD	GC-FPD, GC-NPD, GC-MS(ITD)	LC-MS/MS	GC-ECD, GC-NPD, GC-MS (SINGLE QUAD)	DETERMINATION		
QUECHERS	INTERNAL METHOD	QUECHERS				KLEIN, J.; ALDER, R. L. JOURNAL OF AOAC INT. 2003, 86, 1015-1037	REFERENCE METHOD		
			METHOD R'EN 12393-2:1999			M. ANASTASSIADES, S. J. LEHOTAY, D. STAINEBAHER, F. J. SCHENCK; J. AOAC INT. 86 (2003) 412-431			

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>PYRIMETHANIL</b>							
							LAB CODE
61	60	59	58	57	56	55	
D	D	D	D	D	D	D	SCOPE OF YOUR METHOD
M	M	M	M	M	S	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
LC-MS/MS	GC-MS	GC-MS	GC-ECD-NPD	LC-MS/MS	GC-MS	GC-NPD	CONFIRMATION METHOD
0.01	0.06	0.02	0.05	0.01	0.175	0.01	RL (mg/Kg)
		98.6	107		86.8	102.9	RECOVERY (%)
20	15	10	15	25	30	100	SAMPLE WEIGHT (g)
METHANOL	1	5	4	5	1	2	EXTRACTION SOLVENT
							CLEAN-UP STEP
							GPC
			O				INTERNAL STANDARD
				PCB119 (ECD) TRIFLURALIN (GC-NPD)			INJECTION VOLUME (µL)
ISODRIN	3						
10	1	2	1	5	1	GC 1 µL HPLC 20 µL	
SPLIT/SPLITLESS	SPLITLESS	SPLIT/SPLITLESS		DIRECT	SPLIT/SPLITLESS		INJECTION TYPE
LC-MS/MS	GC-ECD/NPD/MS	GC-MS	GC-ECD-NPD	LC-MS/MS	GC-NPD	GC-ECD, GC-NPD, GC-FPD, HPLC-UV LC-MS/MS	DETERMINATION
							MODULARE MULTIMETHOD S19
			§35 L 000034	ANAL METHODS FOR PESTICIDES RESIDUES IN FOODSTUFFS (NL)			REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>PYRIMETHANIL</b>									
67	66	65	64	63	62	LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	CONFIRMATION METHOD
D	D	D	D	D	NA	NA	NA	NA	RL (mg/Kg)
M	M	M	M	M					RECOVERY (%)
									SAMPLE WEIGHT (g)
									EXTRACTION SOLVENT
									CLEAN-UP STEP
									GPC
									INTERNAL STANDARD
									INJECTION VOLUME (µL)
									INJECTION TYPE
									DETERMINATION
									REFERENCE METHOD
0.02	0.05	0.05	0.01	0.05					
80	96	83	91	89					
10	15	5	5	75					
5	5	1	6	AACETONE FOLLOWED BY CYCLOHEXANE AND ETHYLACETATE					
LLE + SPE	SPE	GPC							
TRIPHENYLPHOSPHATE		AZOBENZENE+RONNELL+ TPP							
10	10	2	0.5	1					
		ON COLUMN	SPLITLESS						
L-C-MS/MS	GC-MS(FTD)	GC-(ECD+FPD+NPD)	GC-MS	GC-NPD					
ANASTASSIADES, M. ET AL., J. ACAC INT., 86 (2003), 412 – 431 QUECHERS - METHOD	QUECHERS(LEOTHAY S. ET AL., J.A.O.C. INT. (2005) 88, 615-629	INTERNAL METHOD	MULTIRESIDUE WITH EXTRELLUT	§ 35 LMBG L.00.00-34					

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>PYRIMETHANIL</b>									
74		73		72		71		70	
D	D	D	D	D	NA	D	D	D	LAB CODE
M	S	S	S	M	M	M	M	M	SCOPE OF YOUR METHOD
GC-MS	GC-NPD	GC-TSD	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
0.005	0.05	0.02	0.06	0.05	0.05	0.05	0.05	0.02	CONFIRMATION METHOD
81.2	77	84	119	90	90	90	90	95	RECOVERY (%)
25	30	100	25	25	25	25	25	20	SAMPLE WEIGHT (g)
3	1	3	1	1	6, METHANOL	6, METHANOL	6, METHANOL	4	EXTRACTION SOLVENT
HEXABROMOBENZENE									
GPC, O	O	O	O	O	SPE	SPE	SPE	SPE	CLEAN-UP STEP
INTERNAL STANDARD									
1	10	1	5	5	1,0-2,0	1,0-2,0	1,0-2,0	2	INJECTION VOLUME (µL)
RHEODYNE	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	SPLITLESS	INJECTION TYPE
GC-MS	HPLC-UV	GC-ECD, GC-NPD	GC-TSD	GC-NPD	GC-ECD/NPD	GC-ECD/NPD	GC-ECD/NPD	GC-ECD/NPD	DETERMINATION
LOCAL SOP		MULTIRESIDUE METHOD FOR ANALYSIS OF PESTICIDES IN FOODSTUFFS P.A. GREVE ED, 1988, MINISTRY OF WELFARE, HEALTH AND CULTURAL AFFAIR RUISWICK NETHERLANDS			MINI-LUKE EXTRACTION	MINI-LUKE EXTRACTION	MINI-LUKE EXTRACTION	MINI-LUKE EXTRACTION	REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

PYRIMETHANIL									
82	81	80	79	78	77	76	75	LAB CODE	SCOPE OF YOUR METHOD
D		D	D	D		D	D		
M		M	M	M		S	M		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-NPD	LC-MS/MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	CONFIRMATION METHOD	
0.02	0.05	0.05	0.05	0.05	0.01	0.01	0.01	RL (mg/Kg)	
79		75	90	90	96	96	96	RECOVERY (%)	
10		10	15	15	10	10	10	SAMPLE WEIGHT (g)	
5		6	6	1	DICHLORMETHANE	6 METHANOL	6 METHANOL	EXTRACTION SOLVENT	
CLEAN-UP STEP									
LLE (CHEMELUT)									
INTERNAL STANDARD									
INJECTION VOLUME (µL)									
1									
INJECTION TYPE									
LC-MS/MS									
DETERMINATION									
GC-ECD, GC-NPD, GC-FPD, GC-MS, HPLC-FLD, HPLC-DAD									
GC-MS (SINGLE-QUAD)									
GC-MS 1015FF (2003)									
REFERENCE METHOD									
QUECHERS, M., ANASTASSIADES, S. J. LEHOTAY, D. STAIBNBAUER, F. J. SCHEINK, J. AOAC INT., 86, (2003) 412-431									
KLEIN, J., ALDER, L (2003) J. AOAC INT. 86, 1015-103									
INTERNAL METHOD									
NO RESULTS									
NO RESULTS									

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>PYRIMETHANIL</b>							
							LAB CODE
<b>89</b>	<b>88</b>	<b>87</b>	<b>86</b>	<b>85</b>	<b>84</b>	<b>83</b>	
D	D	D	D	NA	D	D	SCOPE OF YOUR METHOD
M	M	M	M	M	M	M	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-MS	LC-MS/MS	GC-MS/ITD	GC-MS	GC-MS	GC-MS	GC-MS	CONFIRMATION METHOD
0.02	0.005	0.04	0.025	0.01	0.05	RL (mg/Kg)	
85	98		92	80	109	RECOVERY (%)	
15	5	50	15	50	30	SAMPLE WEIGHT (g)	
4	5	6 ETHYLACETATE/DICHLOROMETHANE (80/20)	4	2	1	EXTRACTION SOLVENT	
		LLE				CLEAN-UP STEP	
						GPC	
			TRICLORONAT	HCB	TRIPHENYL ETHYLENE	INTERNAL STANDARD	
1	10		1	1	1	3	INJECTION VOLUME (µL)
SPLIT/SPLITLESS	ON COLUMN		SPLIT/SPLITLESS	SPLITLESS	SPLITLESS	INJECTION TYPE	
GC-ECD, FPD, MS HPLC-F-DAD	LC-MS/MS	GC-MS/ITD	GC-MS (SINGLE-QUAD)	GC-MS	DETERMINATION		
	IN-HOUSE	ACCORDING TO EN 12396-1; 1999	EN NF 12393		REFERENCE METHOD		

**APPENDIX 7. Methods used by participants for determining pesticides.**

PYRIMETHANIL									
97	96	95	94	93	92	91	90	LAB CODE	
NA	NA	D	D	M	M	S	M	SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
								CONFIRMATION METHOD	
GC-MS	GC-NPD	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	
0.05	0.03	0.005	0.01	0.01	0.01	0.01	0.02	RL (mg/Kg)	
87	93.6			103				RECOVERY (%)	
25	10	80		50		50	50	SAMPLE WEIGHT (g)	
5	4	2	4	4	3	3	2	EXTRACTION SOLVENT	
								CLEAN-UP STEP	
	SPE	SPE	GPC	LLE	LLE	LLE	GPC	CLEAN-UP STEP	
								INTERNAL STANDARD	
		YES		ALDRIN, DITALIMPHOS	YES			INTERNAL STANDARD	
		3	2	5	1	1	1	INJECTION VOLUME (µL)	
		PTV	SPLITLESS	PTV	SPLITLESS	SPLITLESS	SPLITLESS	INJECTION TYPE	
	GC-ECD, GC-NPD, GC-MS	GC-ECD	GC-MS (SINGLE-QUAD)	GC-ECD, GC-NPD, GC-MS	GC-NPD	GC-NPD	GC-NPD	DETERMINATION	
	FILLION ET AL. JOURNAL OG AOAC INTERNATIONAL 78- 5-1995		MULTIMETHOD GC-MS (SINGLE- QUAD)	OFFICIAL METHOD OF ANALYSIS (1990) 15TH ED., 98522, AOAC ARLINGTON VA	DFG-S8			REFERENCE METHOD	

## **APPENDIX 7. Methods used by participants for determining pesticides.**

PYRIMETHANIL										
103		102		101		100		99		LAB CODE
D		D		D		NA		NA		SCOPE OF YOUR METHOD
S		M		M		M		M		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
GC-MS		HPLC-DAD		GC-MS		LC-MS/MS		LC-MS/MS		CONFIRMATION METHOD
0.020		0.05		0.05		0.05		RL (mg/Kg)		RL (mg/Kg)
80		98		95		92		RECOVERY (%)		RECOVERY (%)
50		30		10		10		SAMPLE WEIGHT (g)		SAMPLE WEIGHT (g)
1		1		2		1		EXTRACTION SOLVENT		EXTRACTION SOLVENT
								CLEAN-UP STEP		CLEAN-UP STEP
SPE		GPC		SPE						SPE
						CARBENDAZIM D4, METHOMYL D3, PENDIMETHALIN D5		INTERNAL STANDARD		INTERNAL STANDARD
1		1		1		10		INJECTION VOLUME (µL)		INJECTION VOLUME (µL)
DIRECT		SPLITLESS		SPLIT/SPLITLESS PTV				INJECTION TYPE		INJECTION TYPE
GC-ECD GC-NPD GC-FPD GC-MS LC-MS		GC-NPD		GC-ECD,NPD,MS				DETERMINATION		DETERMINATION
ANN.FALS.EXP.CHIM. SEPT-OCT 1974-87 N721-722 PF13-524		SOP MR 405012; EXTRACTION OF RESIDUES OF PLANT-PROTECTING MATERIAL FROM FRUIT AND VEGETABLES BY ETHYL ACETATE; SOP 308M2; CLEAN UP OF EXTRACTS BY GEL PERMEATION CHROMATOGRAPHY		LC-MS MULTI RESIDUE		REFERENCE METHOD				

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

PYRIMETHANIL									
								LAB CODE	
								SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
								CONFIRMATION METHOD	
116	115	114		113		112		111	110
D	D	D	D	NA	NA	D	ND	ND	
S	S	S	S	M	M				
GC-MS	GC-MS	GC-MS	HPLC-UV			GC-NPD	GC-NPD		
0.01	0.01	0.01	0.05			RL (mg/Kg)	0.02		
		109	98			RECOVERY (%)			
15	50	50	30			50	50		
6	3	6	1			2	6		
GPC	SPE	SPE	GPC			GPC	GPC	CLEAN-UP STEP	
YES								BROMOPHOS-ETHYL	INTERNAL STANDARD
1	1	1	20 mL/20 mg			1	2	INJECTION VOLUME (µL)	
SPLITLESS	ON COLUMN	SPLITLESS	MANUAL			OC; SS	SPLITLESS	INJECTION TYPE	
GC-ECD, GC-NPD GC-MS(TD)	GC-ECD GC-PFPD GC-MS	GC-MS	HPLC-UV			GC-ECD, GC-NPD	GC-ECD AND GC-NPD	DETERMINATION	
RAPPORTO ISTISAN 97/23	NFEN12393		SOP MR 405012; EXTRACTION OF RESIDUES OF PLANT-PROTECTING MATERIAL FROM FRUIT AND VEGETABLE SAMPLES BY ETHYLACETATE; SOP 308W2; CLEAN UP OF EXTRACTS BY GEL PERMEATION CHROMATOGRAPHY					MULTIRESIDUE-METHOD DFG S 19	REFERENCE METHOD
								\$35 LMBG; L 00.00-34, L 00.00-37	

## **APPENDIX 7. Methods used by participants for determining pesticides.**

PYRIMETHANIL												
128	127	126	125	124	123	122	121	120	119	118	117	LAB CODE
NA	NA	NA	NA	NA	NA	NA	NA	NA	D	NA	NA	SCOPE OF YOUR METHOD
									S			QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
												CONFIRMATION METHOD
												GC-NPD
												RL (mg/Kg)
												RECOVERY (%)
												SAMPLE WEIGHT (g)
												EXTRACTION SOLVENT
												CLEAN-UP STEP
												INTERNAL STANDARD
												INJECTION VOLUME ( $\mu$ L)
												INJECTION TYPE
												DETERMINATION
												REFERENCE METHOD
NO RESULTS												
												GC-ECD, GC-NPD

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

TETRACONAZOLE										
18	17	16	15	14	13	12	11	10	9	LAB CODE
NA	NA	D	D	NA	NA	NA	D	NA	D	SCOPE OF YOUR METHOD
M	M	S	S	S	S	S	S	S	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
										GC-MS/ITD
										CONFIRMATION METHOD
										RL (mg/Kg)
										RECOVERY (%)
										SAMPLE WEIGHT (g)
										EXTRACTION SOLVENT
										LLE ACETONE FOLLOWED BY DICHLOROMETHANE AND PETROLEUM ETHER
										CLEAN-UP STEP
										INTERNAL STANDARD
										INJECTION VOLUME (µL)
										INJECTION TYPE
										DETERMINATION
										REFERENCE METHOD
										IN-HOUSE SOP
										LOCAL SOP NO. MR405012

**APPENDIX 7. Methods used by participants for determining pesticides.**

TETRACONAZOLE										
27	26	25	24	23	22	21	20	19	LAB CODE	SCOPE OF YOUR METHOD
D	NA	D	D	D	ND	NA	D	D		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
S	M	M	M	M		S	M	M		CONFIRMATION METHOD
	GC-MS	GC-ECD	GC-MS	GC-NPD		GC-NPD	LC-MS/MS	LC-MS/MS		RL (mg/Kg)
0.05	<0.02	0.05	0.05	0.05		0.05	0.01	0.01		RECOVERY (%)
	98	92.6				92	78	78		SAMPLE WEIGHT (g)
30	10	30	5	10		100	75	75		EXTRACTION SOLVENT
1	6	1	5	METHANOL		3	1	1		CLEAN-UP STEP
	GPC	SPE				O				INTERNAL STANDARD
		YES		FENCHLORPHOS						INJECTION VOLUME ( $\mu$ L)
1		2	1	50	5	1	5	5		AUTOSAMPLER
SPI		SPLITLESS	TPOCI	AUTOMATIC TDS SAMPLER						INJECTION TYPE
	GC-ECD	GC-MS	GC-NPD	TDS-IN LINE WITH GC-MS (SINGLE-QUAD) TOTAL ION DETECTION	LC-MS/MS	GC-ECD	LC-MS/MS	LC-MS/MS		DETERMINATION
HUNGARIAN CENTRAL SOP NO.: 405012 M1		SOP NO.: MR 405012 M1	INTERNAL METHOD							REFERENCE METHOD

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

TETRACONAZOLE									
43	42	41	40	39	38	37	LAB CODE	SCOPE OF YOUR METHOD	
D	D	D	D	D	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
M	M	M	M	S	M	M	M	CONFIRMATION METHOD	
GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-NPD	GC-MS	GC-MS	RECOVERY (%)	
0.01	0.02	0.02	0.01	0.01	0.05	0.05	0.02	SAMPLE WEIGHT (g)	
94		95.1						EXTRACTION SOLVENT	
10	10	20	15	100	10	10	10	CLEAN-UP STEP	
5	5	3	4	2	5	5	5	INTERNAL STANDARD	
SPE	LL			GPC, SPE	O	O	O	INJECTION VOLUME ( $\mu$ L)	
TRIPHENYLPHOSPHATE (TPP)								INJECTION TYPE	
4	1	4	1	1	2	2	1	GC-ECD GC-NPD GC-MS	
PTV SOLVENT VENT			SPLITLESS	SPLIT	SPLITLESS	SPLITLESS	SPLIT/SPLITLESS	DETERMINATION	
LC-MS/MS(ESI+)	GC-ECD, GC-MS	GC-ECD, GC-NPD	GC-NPD, GC-ECD, GC-MS(FTD)	GC-ECD	GC-ECD GC-NPD GC-MS	GC-MS (SINGLE QUAD)	GC-MS (SINGLE QUAD)	REFERENCE METHOD	
ANASTASSIADES ET AL. JAOAC INT. 86 82003 412-431, MODIFIED	M. ANASTASSIADES ET AL., J. AOAC INT. 86 (2), 412-431, (2003)		ANALYTICAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS, 6TH ED. 1996, GENERAL INSPECTORATE FOR HEALTH PROTECTION, MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS	DFG S 19, L 00-34 (\$ 35 LMBG)	ANASTASSIADES ET AL. JAOAC INT. VOL T 86 NO. 2, 2003				

## **APPENDIX 7. Methods used by participants for determining pesticides.**

**APPENDIX 7. Methods used by participants for determining pesticides.**

TETRACONAZOLE									
									LAB CODE
									SCOPE OF YOUR METHOD
59	58	57	56	55	54	53	52		GC-ECD
D	D	NA	D	D	NA	D	D		GC-MS
M	M	S	M	S	S	M	M		GC-NPD
GC-MS	GC-ECD-NPD	GC-MS	GC-NPD	GC-MS	GC-ECD	GC-ECD	GC-ECD		GC-MS
0.05	0.01	0.036	0.005	0.05	0.05	0.05	0.05		RL (mg/Kg)
118		90	96.2						RECOVERY (%)
10	15	30	100	15	10	10	10		SAMPLE WEIGHT (g)
5	4	1	2	2	6	6	5		EXTRACTION SOLVENT
O									CLEAN-UP STEP
3	PCB119 (ECD) TRIFLURALIN (GC-NPD)								INTERNAL STANDARD
2	1	1	GC 1 µL HPLC 20 µL	1	1	1	1.5		INJECTION VOLUME (µL)
SPLITLESS	SPLIT/SPLITLESS	SPLIT	SPLIT/SPLITLESS	SPLIT/SPLITLESS	SPLIT/SPLITLESS	SPLIT/SPLITLESS	SPLIT/SPLITLESS		INJECTION TYPE
GC-MS	GC-ECD-NPD	GC-ECD	GC-ECD, GC-NPD, GC-FPD, HPLC-UV, LC-MS/MS	GC-ECD, GC-NPD, GC-FPD, HPLC-FID, HPLC-UV	GC-ECD, GC-NPD, GC-FPD, GC-MS, HPLC-FID, HPLC-UV	GC-ECD, GC-NPD, GC-FPD, GC-MS, HPLC-FID, HPLC-UV	GC-ECD, GC-NPD, GC-FPD, GC-MS, HPLC-FID, HPLC-UV		DETERMINATION
§35 L 000034	ANAL.METHODS FOR PESTICIDES RESIDUES IN FOODSTUFFS (NL)		MODULARE MULTIMETHODE S19		INTERNAL METHOD	INTERNAL METHOD	QUECHERS		REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>TETRACONAZOLE</b>									
65	64	63	62	61	60	LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	CONFIRMATION METHOD
D	D	D	NA	D	ND				
M	M	M		M	M				
GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS	GC-MS			
0.01	0.02	0.03		0.01	0.05	RL (mg/Kg)			
88	83	62				RECOVERY (%)			
5	5	75		50	15	SAMPLE WEIGHT (g)			
1	6	ACETONE FOLLOWED BY CYCLOHEXANE AND ETHYLACETATE	3	1	1	EXTRACTION SOLVENT			
						CLEAN-UP STEP			
	GPC	GPC	GPC	GPC	GPC				
AZOBENZENE+RONNELL+TRIPHENYLPHOSPHATE	TPP					ISODRIN	INTERNAL STANDARD		
2	0.5	1		2	1		INJECTION VOLUME (µL)		
ON COLUMN	SPLITLESS			PTV			INJECTION TYPE		
GC-(ECD+FPD+NPD)	GC-MS	GC-NPD	GC-MS	GC-ECD/NPD/MS			DETERMINATION		
INTERNAL METHOD	MULTIRESIDUE WITH EXTRELLUT			DFG S19 (ASU \$35 LMBGL 00.00-34)			REFERENCE METHOD		

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>TETRACONAZOLE</b>									
<b>73</b>	<b>72</b>	<b>71</b>	<b>70</b>	<b>69</b>	<b>68</b>	<b>67</b>	<b>66</b>	<b>LAB CODE</b>	<b>SCOPE OF YOUR METHOD</b>
D	D	NA	NA	D	D	D	NA	NA	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
M	S			M	S	S			CONFIRMATION METHOD
GC-NPD	GC-ECD			GC-MS	GC-MS	GC-MS			RL (mg/Kg)
0.05	0.02			0.05	0.05	0.05			RECOVERY (%)
81	72			94	104				SAMPLE WEIGHT (g)
30	100			25	20	90			EXTRACTION SOLVENT
1	3			1	4	3			CLEAN-UP STEP
									GPC + SPE
									ALDRIN
									INTERNAL STANDARD
									INJECTION VOLUME (µL)
0.5	1			1.0-2.0	2	1			INJECTION TYPE
ON COLUMN	SPLITLESS			SPLITLESS	SPLITLESS	SPLIT/ SPLITLESS			DETERMINATION
GC-ECD	GC-ECD, GC-NPD			GC-NPD, GC-ECD	GC-MS (SINGLE-QUAD)	GC - ECD			OFFICIAL METHOD L 00/00-34; § 35 LMBG
LOCAL SOP				MULTIRESIDUE METHOD 1, ANALYTICAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS, SIXTH EDITION	MINI LUKE EXTRACTION				REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

TETRACONAZOLE									
81	80	79	78	77	76	75	74	LAB CODE	SCOPE OF YOUR METHOD
M	D	D	ND	ND	D	M	M	GC-MS	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
LC-MS/MS	GC-MS	GC-MS						CONFIRMATION METHOD	
0.05	0.05				0.01		0.02	RL (mg/Kg)	
82				106			89.5	RECOVERY (%)	
10	15	15		10		25		SAMPLE WEIGHT (g)	
6	6	1		6 METHANOL	3			EXTRACTION SOLVENT	
NO RESULTS									
O	SPE	GPC		LLE (CHEM ELUT)			HEXABROMOBENZENE	CLEAN-UP STEP	
								INTERNAL STANDARD	
20		2	1		20		1	INJECTION VOLUME (µL)	
								INJECTION TYPE	
LC-MS/MS		SPLITLESS					LC-MS/MS		DETERMINATION
									JAOAC INTERN., 86, 1015FF (2003)
									GC-MS
									(SINGLE-QUAD)
									HPLC-FID
KLEIN,J., ALDER,L.(2003) JAOAC INT. 86, 1015-103		INTERNAL METHOD							REFERENCE METHOD
NO RESULTS									

**APPENDIX 7. Methods used by participants for determining pesticides.**

TETRACONAZOLE									
								LAB CODE	
								SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
								GC-ECD	
89	88	87	86	85	84	83	82	D	D
D	NA	NA	NA	NA	D	D			
M				M	M	M			
GC-MS			GC-MS		LC-MS/MS, GC-MS, GC-MS				
0.02			0.05		0.05		0.05		
80			80		99		112		
15			50		10, 30, 30		10		
4			2		5, 1, 1		5		
EXTRACTION SOLVENT									
CLEAN-UP STEP									
SPE GPC									
0 (PSA)									
INTERNAL STANDARD									
TRIPHENYL ETHYLENE TRIPHENYL ETHYLENE									
1			1		7, 3, 3		1		
INJECTION VOLUME (µL)									
SPLIT/SPLITLESS									
PARTIAL, SPLITLESS, SPLITLESS									
SPLITLESS AND ON COLUMN									
GC-ECD, FPD, MS HPLC-F-DAD									
GC-MS (SINGLE-QUAD)									
LC-MS/MS GC-MS GC-MS									
DETERMINATION									
QUECHERS, M. ANASTASSIADES, S.J. LEHOTAY, D. STAIBNABER, F. J. SCHEIK, J AOAC INT., 86 (2003) 412-431									
REFERENCE METHOD									
EN NF 12393									

**APPENDIX 7. Methods used by participants for determining pesticides.**

TETRACONAZOLE									
97	96	95	94	93	92	91	90	LAB CODE	
NA	NA	D	D	D	D	D	D	SCOPE OF YOUR METHOD	
QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX									
								CONFIRMATION METHOD	
								GC-MS	
								RL (mg/Kg)	
								RECOVERY (%)	
								SAMPLE WEIGHT (g)	
								EXTRACTION SOLVENT	
								CLEAN-UP STEP	
								GPC	
								LLE	
								INTERNAL STANDARD	
								INJECTION VOLUME (µL)	
								INJECTION TYPE	
								DETERMINATION	
								REFERENCE METHOD	

**APPENDIX 7. Methods used by participants for determining pesticides.**

TETRACONAZOLE							
103	102	101	100	99	98	LAB CODE	SCOPE OF YOUR METHOD
NA	D	D	NA	NA	D		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
	M	M			M		CONFIRMATION METHOD
		GC-NPD	GC-MS		LC-MS/MS		RL (mg/Kg)
		0.01	0.05		0.05		RECOVERY (%)
		81	90		96		SAMPLE WEIGHT (g)
		30	10		10		EXTRACTION SOLVENT
		1	2		1		CLEAN-UP STEP
							GPC
							SPE
							CARBENDAZIM D4, METHOMYL D3, PENDIMETHALIN D5
							INTERNAL STANDARD
							10
							INJECTION VOLUME (µL)
							INJECTION TYPE
							DETERMINATION
							GC-ECD, NPD, MS
							LC-MS/MULTI RESIDUE
							REFERENCE METHOD
							SOP MR 405012; EXTRACTION OF RESIDUES OF PLANT-PROTECTING MATERIAL FROM FRUIT AND VEGETABLE SAMPLES BY ETHYL-ACETATE; SOP 308M2; CLEAN UP OF EXTRACTS BY GEL PERMEATION CHROMATOGRAPHY

**APPENDIX 7. Methods used by participants for determining pesticides.**

TETRACONAZOLE									
111	110	109	108	107	106	105	104	LAB CODE	SCOPE OF YOUR METHOD
D	D	D	NA	D	ND	NA	D		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
M	M	M		M			S		CONFIRMATION METHOD
GC-MS	GC-NPD	GC-MS		GC-MS			GC-MS		RL (mg/Kg)
0.05	0.01	0.01		0.02	0.01		0.005		RECOVERY (%)
100				102					SAMPLE WEIGHT (g)
50	50	10		100			100		EXTRACTION SOLVENT
2	6	2		2			2		
									CLEAN-UP STEP
	GPC			GPC			GPC		
									INTERNAL STANDARD
		BROMOPHOS-ETHYL					NITROFEN		INJECTION VOLUME (µL)
1		2		2	1		2		
OC; SS		SPLITLESS		SPLITLESS					INJECTION TYPE
	GC-ECD, GC-NPD	GC-ECD AND GC-NPD		GC-ECD GC-NPD GC-FPD GC-MS			GC-ECD		DETERMINATION
	§35 LMBG; L 00..00-34, L 00.00-37	MULTI-RESIDUE- METHOD DFG S 19		S19 - DFG			LMBG §35 L- 00.00..34		REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

<b>TETRACONAZOLE</b>											
122	121	120	119	118	117	116	115	114	113	112	LAB CODE
NA	NA	NA	NA	D	NA	NA	D	NA	D	NA	SCOPE OF YOUR METHOD
			S			S		S			QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
				GC-NPD			GC-MS		GC-ECD		CONFIRMATION METHOD
					0.05		0.01		0.01		RL (mg/Kg)
						73			80		RECOVERY (%)
						5	50		30		SAMPLE WEIGHT (g)
						3	3		1		EXTRACTION SOLVENT
											CLEAN-UP STEP
											INTERNAL STANDARD
											INJECTION VOLUME (µL)
											INJECTION TYPE
											DETERMINATION
											REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

TETRACONAZOLE									
128	127	126	125	124	123	LAB CODE	SCOPE OF YOUR METHOD	CONFIRMATION METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
NA	NA	NA	NA	NA	NA	RL (mg/Kg)	RECOVERY (%)	SAMPLE WEIGHT (g)	EXTRACTION SOLVENT
NO RESULTS									

**APPENDIX 7. Methods used by participants for determining pesticides.**

THIABENDAZOLE									
6	5	4	3	2	1	LAB CODE	SCOPE OF YOUR METHOD		
D	D	D	NA	D	D		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		
S	M	M		M	M		CONFIRMATION METHOD		
HPLC-DAD	LC-MS/MS	GC-MS		LC-MS/MS	LC-MS/MS		RL (mg/Kg)		
0.05	0.005	0.001		0.010	0.010		RECOVERY (%)		
95.7				103			SAMPLE WEIGHT (g)		
15	10	10	10	10	10		EXTRACTION SOLVENT		
4	5	5	5	6	6		CLEAN-UP STEP		
SPE	SPE	SPE					INTERNAL STANDARD		
							YES		
100	20	10		5	5		INJECTION VOLUME (µL)		
FULL LOOP							INJECTION TYPE		
HPLC-DAD	LC-MS/MS	LC-MS/MS					DETERMINATION		
ANALYTICAL METHODS FOR PESTICIDE RESIDUES MINISTRY OF HEALTH NETHERLANDS 6TH ED	M.ANASTASSIADES ET AL. JAOAC 86, 412431 (2003)	QUECHERS; ANASTASSIADES					KLEIN, J.; ALDER, L. (2003): APPLICABILITY OF GRADIENT LIQUID CHROMATOGRAPHY WITH TANDEM MASS SPECTROMETRY TO THE SIMULTANEOUS SCREENING FOR ABOUT 100 PESTICIDES IN CROPS. J. AOAC INT. 86 (5): 1015-1037		
							IN HOUSE		
							REFERENCE METHOD		

**APPENDIX 7. Methods used by participants for determining pesticides.**

THIABENDAZOLE									
13	12	11	10	9	8	7	LAB CODE		
D	D	D	D	D	D	D	SCOPE OF YOUR METHOD		
M	M	S	S	S	M	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		
LC-MS/MS	MS-TID, LC-MS			GC-MS/ITD)	GC-MS	GC-NPD(80)	CONFIRMATION METHOD		
0.05	0.2	0.05	0.05	0.01	0.05	0.03	RL (mg/Kg)		
96.9	75	98	89	91	102		RECOVERY (%)		
15	50	30	25	15	30	10	SAMPLE WEIGHT (g)		
1	1	1	6	4	1	6 (DICHLOROMETHANE)	EXTRACTION SOLVENT		
							CLEAN-UP STEP		
				LLE ACETONE FOLLOWED BY DICHLOROMETHANE AND PETROLEUM ETHER	O				
							INTERNAL STANDARD		
							INJECTION VOLUME (µL)		
							INJECTION TYPE		
							SPLITLESS		
					LOOP	SPLITLESS			
							DETERMINATION		
							GC-ECD; GC-NPD(80)		
							REFERENCE METHOD		
							RAPPORII ISTISAN 97/23		
							IN HOUSE SOP		

**APPENDIX 7. Methods used by participants for determining pesticides.**

THIABENDAZOLE										
	22	21	20	19	18	17	16	15	14	LAB CODE
D	NA	D	D	D	NA	D	D	D		SCOPE OF YOUR METHOD
S		S	M	S		M	S	S		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
LC-DAD	GC-NPD	LC-MS/MS	GC-MS	GC-MS/MS	GC-MS/MS	GC-MS	GC-MS	GC-MS		CONFIRMATION METHOD
0.05	0.05	0.01	0.02		0.05		0.05	0.05		RL (mg/Kg)
77.4	81	96					106			RECOVERY (%)
50	100	75	75		15		25	15		SAMPLE WEIGHT (g)
1	3	1	1 (pH10)		4		1	1	4	EXTRACTION SOLVENT
LLE	O			GPC						CLEAN-UP STEP
										INTERNAL STANDARD
20	2	5	20		10		2	2	2	INJECTION VOLUME (µL)
	MANUAL				LVI SPLITLESS					INJECTION TYPE
HPLC-DAD	GC-NPD	LC-MS/MS	HPLC-FID	GC-MS/MS	GC-NPD		GC-ECVD GC-TSD GC-MS HPLC-UV			DETERMINATION
								LOCAL SOP NO. MR405012	MULTIRESIDUES	REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

THIABENDAZOLE									
LAB CODE	SCOPE OF YOUR METHOD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	CONFIRMATION METHOD	RL (mg/Kg)	RECOVERY (%)	SAMPLE WEIGHT (g)	EXTRACTION SOLVENT	CLEAN-UP STEP	INTERNAL STANDARD
28	27	26	25	24	23				
D	D	D	D	D	D				
S	S	M	M	S	M				
HPLC-UV	LC-MS/MS	GC-MS	LC-MS/MS	HPLC-DAD					
0.04	0.05	0.01	<0.02	0.05	0.01				
90		83	90	104.0					
20	30	10	10	30	5				
1	1	6 METHANOL 95%-AMMONIACETATE 5%, 20 mM ACETIC ACID	6	1	5				
LLE	GPC	SPE	SPE FLORISIL						
		CARBARYL C13	YES						
20	20	10	2	1 mg SAMPLE	20				
RHEODYNE INJECTOR	SPLITLESS	SPLITLESS	AUTOSAMPLER	AUTOMATIC					
HPLC-UV	HPLC-UV	GC-MS	LC-MS/MS	HPLC-DAD					
		HUNGARIAN CENTRAL SOP NO.: 405012 M1	SOP NO.: MR 405012 M1	INTERNAL METHOD					

**APPENDIX 7. Methods used by participants for determining pesticides.**

THIABENDAZOLE									
36	35	34	33	32	31	30	29	LAB CODE	SCOPE OF YOUR METHOD
D	D	NA	NA	ND	D	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
S	S			S	S	S	S	S	CONFIRMATION METHOD
GC-MCS	HPLC-DAD			MS	DIFFERENT COLUMN	LC-DAD			RL (mg/Kg)
0.05	0.05	0.5	0.05	0.05	0.05	0.05	0.05		RECOVERY (%)
80	90.0			78			88		
50	50			10	10	10	15		SAMPLE WEIGHT (g)
1	6			6	1	1	4		EXTRACTION SOLVENT
LLE (ACID-BASE PARTITION)	SPE			O	GPC	SPE (2OH)			CLEAN-UP STEP
					DICHOFENTHION	BENZIMIDAZOLE			INTERNAL STANDARD
1	20			1	1	100			INJECTION VOLUME (µL)
SPLIT/SPLITLESS				SPLITLESS					INJECTION TYPE
GC-NPD SPECTROPHOTOMETRY -UV/VIS	HPLC-DAD			GC-MS	GC-ECD GC-NPD	HPLC-DAD			DETERMINATION
DANISH VETERINARY AND FOOD ADMINISTRATION, METHOD FP 018.1, 1996				ISTISAN 97/23	RAPP ISTISAN 97/23	MINI LUKE			REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

THIABENDAZOLE									
								LAB CODE	
								SCOPE OF YOUR METHOD	
						QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX			
LC-MS/MS	HPLC	LC-MS/MS	HPLC-DAD	LC-MS/MS	HPLC-DAD	LC-MS/MS	CONFIRMATION METHOD	LC-MS/MS	CONFIRMATION METHOD
42	41	40	39	38	37	37	RL (mg/Kg)	RL (mg/Kg)	SCOPE OF YOUR METHOD
D	NA	D	D	D	D	D	RECOVERY (%)	RECOVERY (%)	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
M	M	M	M	M	M	M	SAMPLE WEIGHT (g)	SAMPLE WEIGHT (g)	CONFIRMATION METHOD
LC-MS/MS	HPLC	LC-MS/MS	HPLC-DAD	LC-MS/MS	HPLC-DAD	LC-MS/MS	EXTRACTION SOLVENT	EXTRACTION SOLVENT	CONFIRMATION METHOD
0.01	0.2	0.01	0.2	0.01	0.2	0.01	6 (METHANOL/WATER)	6 (METHANOL/WATER)	CONFIRMATION METHOD
88		95		95			5	5	CONFIRMATION METHOD
10	15	10	10	10	10	10	5	5	CONFIRMATION METHOD
5	4	6 (METHANOL/WATER)	5	5	5	5			CONFIRMATION METHOD
LL		SPE	O	O	O	O	CLEAN-UP STEP	CLEAN-UP STEP	CONFIRMATION METHOD
							DIMETHOATE D6	DIMETHOATE D6	CONFIRMATION METHOD
							INTERNAL STANDARD	INTERNAL STANDARD	CONFIRMATION METHOD
20	1	20	2	20	2	20	INJECTION VOLUME (µL)	INJECTION VOLUME (µL)	CONFIRMATION METHOD
		SPITLESS	SPLITLESS	SPITLESS	SPLITLESS	SPLITLESS	INJECTION TYPE	INJECTION TYPE	CONFIRMATION METHOD
LC-MS/MS	GC-NPD, GC-ECD, GC-MS(IRD)	LC-MS/MS	GC-ECD GC-NPD GC-MS	LC-MS/MS	GC-ECD GC-NPD GC-MS	LC-MS/MS	DETERMINATION	DETERMINATION	CONFIRMATION METHOD
M. ANASTASSIADES ET AL., J. AOAC INT. 86 (2), 412-431, (2003)	ANALYTICAL METHODS FOR PESTICIDE RESIDUES IN FOODSTUFFS, 6TH ED., 1996. GENERAL INSPECTORATE FOR HEALTH PROTECTION, MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORTS, THE NETHERLANDS	ANASTASSIADES, JOURNAL OF AOAC INT. VOLT 86 NO 2, 2003	ANASTASSIADES, JOURNAL OF AOAC INT. VOLT 86 NO 2, 2003	ANASTASSIADES, JOURNAL OF AOAC INT. VOLT 86 NO 2, 2003	ANASTASSIADES, JOURNAL OF AOAC INT. VOLT 86 NO 2, 2003	ANASTASSIADES, JOURNAL OF AOAC INT. VOLT 86 NO 2, 2003	REFERENCE METHOD	REFERENCE METHOD	CONFIRMATION METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

THIABENDAZOLE							
							LAB CODE
47	D	46	D	45	D	44	43
S	M		S	M	M	D	D
GC-MS	GC-NPD	GC-NPD	GC-NPD	GC-NPD	GC-NPD	NONE	SCOPE OF YOUR METHOD
0.05	0.05	0.05	0.1	0.01	0.01	0.01	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
10	15	15	25	50	50	10	CONFIRMATION METHOD
5	4	4	1 (IN PRESENCE OF NaOH)	2	2	5	RECOVERY (%)
SPE			GPC	LLE	SPE		SAMPLE WEIGHT (g)
							EXTRACTION SOLVENT
							CLEAN-UP STEP
							INTERNAL STANDARD
4	3 / 1	3 / 1	10	5	5	4	TRIPHENYLPHOSPHATE (TPP)
SPLIT/SPLITLESS	(PULSED) SPLITLESS/(TRACK OVEN) ON COLUMN	RHEODYNE					INJECTION VOLUME ( $\mu$ L)
GC-ECD, GC-NPD, GC-MS (SINGLE QUAD)	GC-NPD / GC-ECD	HPLC - DAD	LC-MS/MS	LC-MS/MS(ESI+)			INJECTION TYPE
M. ANASTASSIADES, S. J. LEHOTAY, D. STAIBBAHER, F. J. SCHENCK : J. AOAC INT. 86 (2003) 412-431	MULTI-RESIDUE METHOD/PESTICIDES AMENABLE TO GAS CHROMATOGRAPHY (3:1:2 ACETONE-PARTITION FOR FRUITS, VEGETABLES AND POTATOES) 6TH EDITION MINISTRY OF PUBLIC HEALTH, WELFARE AND SPORT. THE NETHERLANDS		HOUSE METHOD				DETERMINATION
		CLEAN UP AS IN EN 12393 P METHOD					REFERENCE METHOD
							ANASTASSIADES ET AL. JAOAC INT. 86 8203 (2003) 412-431, MODIFIED

**APPENDIX 7. Methods used by participants for determining pesticides.**

THIABENDAZOLE									
									LAB CODE
									SCOPE OF YOUR METHOD
55	54	53	52	51	50	49	48		
D	D	D	D	NA	D	D	D		
M	M	S	S		S	S	M		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
HPLC:DAD	HPLC:FLD	HPLC:DAD	HPLC:DAD		LC:MS	GC:NPD	LC:MS/MS		CONFIRMATION METHOD
0.02	0.05	0.05	0.05		0.05	0.02	0.01		RL (mg/Kg)
75.3	101				87				RECOVERY (%)
50	37.5	50	30		10	50	10		SAMPLE WEIGHT (g)
2	1	1	6 (DICHLOROMETHANE)		4	1	6		EXTRACTION SOLVENT
LLE	GPC	LLE			LLE	SPE	CLEAN-UP STEP		
20	20	10	20		5	2	20		INTERNAL STANDARD
HPLC:DAD	HPLC:DAD, FID	HPLC:FLD, HPLC:UV	HPLC:DAD			SPLITLESS			INJECTION VOLUME (µL)
IN HOUSE METHOD	EN 12393-1,2,3	INTERNAL METHOD							INJECTION TYPE
									DETERMINATION
						GC:FPD, GC:NPD, GC-MS(ITD)/MS	LC:MS/MS		KLEIN, J.; ALDER, L.; JOURNAL OF AOAC INT, 2003, 86, 1015- 1037
									REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

THIABENDAZOLE									
62	61	60	59	58	57	56	LAB CODE	SCOPE OF YOUR METHOD	
D	D	D	D	D	D	D	GC-NPD	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
M	M	M	M	S	M	S	GC-MS/MS	CONFIRMATION METHOD	
GC-MS	LC-MS/MS	LC-MS/MS	HPLC-UV	LC-DAD-FLD	LC-MS/MS	RL (mg/Kg)	0.474	RECOVERY (%)	
0.05	0.01	0.05	0.05	0.1	0.01	69.4		SAMPLE WEIGHT (g)	
10	20	5	50	15	25	30		EXTRACTION SOLVENT	
1	METHANOL	1	5	4	5	1		CLEAN-UP STEP	
			O	SPPE				INTERNAL STANDARD	
TPP			3					INJECTION VOLUME (µL)	
2	10	20	2	200	5	1		INJECTION TYPE	
GC-MS	LC-MS/MS	LC-MS/MS	HPLC-FLD OR DAD	LC-FLD-PC (POSTCOLUMN DERIVATIZATION)	LC-MS/MS	GC-NPD	DETERMINATION	REFERENCE METHOD	
			METODENVOR SCHLAG EG- PROFICIENCY TEST 1996/97	ANAL.METHODS FOR PESTICIDES RESIDUES IN FOODSTUFFS (NL)					

## **APPENDIX 7. Methods used by participants for determining pesticides.**

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THIABENDAZOLE									
								LAB CODE	
								SCOPE OF YOUR METHOD	
								QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
								CONFIRMATION METHOD	
93	D	92	D	91	D	90	D	88	D
M	M	M	M	S	M	M	D	87	D
LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS	GC-MS	GC-TSD	GC-TSD	GC-TSD		
0.02	0.01	0.006	0.05	0.03	0.002	0.05	0.05		
67	67			70	98				
80	50	10	50	15	5	5	25		
2	4	6 METHANOL	3	6	5	5	6 ACETONE/DICHLOROMETHANE/PETROLEUM-ETHER (1/1/1)		
GPC	LLE	SPE	LLE	LLE	LLE	SPE DIOL	CLEAN-UP STEP		
						BENZIMIDAZOLE	INTERNAL STANDARD		
1µL PER COLUMN	10	20	OCT-25	1	10	100	INJECTION VOLUME (µL)		
SPLITLESS	LOOP			SPLIT/SPLITLESS		AUTOSAMPLER-FULL LOOP	INJECTION TYPE		
GC-ECD AND GC-NPD OR GC-ECD ONLY OR GC-NPD ONLY	LC-MS/MS	LC-MS/MS	HPLC-DAD	GC-ECD, FPD, MS HPLC-F-DAD	LC-MS/MS	HPLC-DAD	DETERMINATION		
LC-MS/MS (MULTIMETHOD, 2 COLUMNS)	LC-MULTIRESIDUE	DRAFT § 35 LMBG-METHOD			IN-HOUSE	EN 14333-1: 2004	REFERENCE METHOD		

**APPENDIX 7. Methods used by participants for determining pesticides.**

THIABENDAZOLE									
101	100	99	98	97	96	95	94	LAB CODE	SCOPE OF YOUR METHOD
D	NA	D	D	NA	NA	D	D		
M		S	M		S	M		QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX	
GC-MS	HPLC-FLD	LC-MS/MS						GC-NPD	CONFIRMATION METHOD
0.05	0.05	0.05			0.05		0.05	RL (mg/Kg)	
85		105			84		74.2	RECOVERY (%)	
10		8	10		25		10	SAMPLE WEIGHT (g)	
2		4	1		5		6	EXTRACTION SOLVENT	
				LLE				CLEAN-UP STEP	
		SPE						SPE	
	BENZIMIDAZOLE	CARBENDAZIM D4, METHOMYL D3, PENDIMETHALIN D5						INTERNAL STANDARD	
1		50	10			20	2	INJECTION VOLUME (µL)	
	SPLIT/SPLITLESS PTV							SPLITLESS	INJECTION TYPE
	GC-ECD,NPD,MS	HPLC-FLD	LC-MS/MS					LC-MS/MS	DETERMINATION
								LC-MS MULTI RESIDUE	REFERENCE METHOD

**APPENDIX 7. Methods used by participants for determining pesticides.**

THIABENDAZOLE							
						LAB CODE	
						SCOPE OF YOUR METHOD	
				QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX			
						CONFIRMATION METHOD	
D	ND	D	D	HPLC:DAD	HPLC:DAD	RL (mg/Kg)	
S	M	S	S	LC-MS/MS		RECOVERY (%)	
0.02	0.5	0.005	0.010			SAMPLE WEIGHT (g)	
85			80				
75	100	10	50				
1	4	6 (METHANOL)	1				
CLEAN-UP STEP							
DIATOMACEOUS EARTH SPE							
ALDRIN							
40	1	25	1			INJECTION VOLUME (µL)	
DIRECT SPLITLESS							
HPLC:DAD GC:NPD, GC:ECD LC-MS/MS							
GC:ECD GC:NPD GC:FPD GC-MS LS-MS							
ANN FALS EXP CHIM. SEPT-OCT 1974-67 NY721-722 PP513-524							
SOP MR 405012; EXTRACTION OF RESIDUES OF PLANT-PROTECTING MATERIAL FROM FRUIT AND VEGETABLE SAMPLES BY ETHYL ACETATE; SOP 308M2; CLEAN UP OF EXTRACTS BY GEL PERMEATION CHROMATOGRAPHY							
NF-EN-14333-3 MULTIRESIDUE METHOD: AOAC 985.22/90 MODIFIED APPLICATION NOTE BY WATERS							
REFERENCE METHOD							

## **APPENDIX 7. Methods used by participants for determining pesticides.**

THIABENDAZOLE									
ANALYSIS OF THIABENDAZOLE IN PLANT-PROTECTING MATERIAL FROM FRUIT AND VEGETABLE SAMPLES BY ETHYL-ACETATE					TEST METHODS				
TEST METHOD		TEST CONDITIONS		TEST DETAILS	TEST RESULTS		TEST CONCLUSIONS		TEST COMMENTS
GC-NPD		HPLC-DAD		HPLC-DAD	109		108		107
0.05		GC-MS		GC-MS	D		NA		D
98		LC-DAD		LC-DAD	S		S		M
30		SPE; LLE		SPE; LLE	M		M		M
1		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE		SPE; LLE		SPE; LLE	O		O		O
SPE		SPE		SPE	O		O		O
SPE; LLE									

**APPENDIX 7. Methods used by participants for determining pesticides.**

THIABENDAZOLE									
121	120	119	118	117	116	115	114	LAB CODE	SCOPE OF YOUR METHOD
D	D	D	D	D	D	D	D	D	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX
S	S	S	S	M	S	S	S	S	CONFIRMATION METHOD
HPLC-UV	-	GC-MS	GC-NPD	GC-MS	GC-MS	LC-DAD	LC-MS/MS	RL (mg/Kg)	RECOVERY (%)
<0.05	0.04	0.05	0.03	0.03	0.05	0.02	0.01	90	87
90			75		70				
25	10	5	40	10	10	10	20		SAMPLE WEIGHT (g)
1	3	3	1	1	6	6	6		EXTRACTION SOLVENT
			O						CLEAN-UP STEP
									INTERNAL STANDARD
									INJECTION VOLUME (µL)
									INJECTION TYPE
									DETERMINATION
									WATERS A MULTIRESIDUE LC-MS/MS METHOD FOR PESTICIDES
									REFERENCE METHOD
									NFEN1413-1
									ISS B6
									WYD. METODYCZNE PZH WARSZAWA 2002

## **APPENDIX 7. Methods used by participants for determining pesticides.**

THIABENDAZOLE									
128	127	126	125	124	123	122	LAB CODE		
NA		D	D	D	NA	D	SCOPE OF YOUR METHOD		
		S	S	S	S	S	QUANTIFICATION USING STANDARDS IN SOLVENT OR IN MATRIX		
	HPLC-DAD		HPLC-FLD	HPLC-UV			CONFIRMATION METHOD		
0.01	0.0095	0.04	0.04	0.02			RL (mg/Kg)		
		87	80.0	95			RECOVERY (%)		
25	25	25	50	50			SAMPLE WEIGHT (g)		
6	3	6	16	16			EXTRACTION SOLVENT		
	O	GPC	LLE				CLEAN-UP STEP		
							INTERNAL STANDARD		
							INJECTION VOLUME (µL)		
	AUTOSAMPLER	SPLITLESS	MANUAL	LOOP			INJECTION TYPE		
	HPLC-DAD	GC-NPD	HPLC-UV	HPLC-UV			DETERMINATION		
							REFERENCE METHOD		
							GILYDIS DM - WALTERS SM (1990) JAOA CHEM. 73, 753-761		
							WYDAWNICTWO METODYCZNE PZH WARSZAWA 2002: OZNACZANIE POZOSTALosci FUNGICYDOW Z GRUPY BENOMYL I THIABENDAZOLU		
							NO RESULTS		



# Protocol



## Instructions

Only laboratories that are involved in providing residue data for their national monitoring programmes, and/or the EU co-ordinated monitoring programme are invited to participate in the 7<sup>th</sup> European Proficiency Test.

To participate, each laboratory will have to send by e-mail the **Application Form** to the Organiser. They will then receive confirmation of acceptance of their participation by e-mail with a **web security code** to enable them to access the Protocol, the Forms and the Pesticide List. A **Laboratory Code** will also be sent and subsequently this code must always be used in communications with the Organiser. Any e-mail without this code will not be answered. This code will only be known by the participant, the Organiser, and the Commission. This will ensure confidentiality during the test. In the Final Report there will not be any correlation between the code and the laboratory name. However, some results may need to be presented on a country basis to the Standing Committee on the Food Chain and Animal Health, and a link between codes and laboratories is possible, especially if there are only a few laboratories in one country.

This **Protocol**, together with three **Forms (1-3)** will be uploaded onto the web page and access will be using the security code. Each form will have a deadline; please ensure you adhere strictly to these deadlines. The completed forms must be returned to the Organiser. On receipt of each form, the Organiser will respond with a confirmatory e-mail.

The **Pesticide List** will also be present at this site without using the security code. This list will include all the possible pesticides that could be present in the test material. This list will specify which compounds to look for. The list will be available from 9<sup>th</sup> March 2005, so that all participants know in good time before receipt of the test materials which pesticides might be present. Thus, participants will have time enough to buy any standards required and validate their methods. MPRL values (minimum performance reporting levels) for each pesticide will be given. These values are the levels that the laboratories should be able to attain.

The official language used in this Proficiency Test will be English.

Communications between participating laboratories during the test are not allowed.

Invoices to cover the cost of transporting the test materials will be available from the start of the test, so that once the shipping begins laboratories will be able to receive the test materials. Only laboratories that have paid the transport costs will receive the test materials. If laboratories need more time to pay, they must send by **fax** a justification to verify that the payment procedure has started.

# **General Characteristics**

## **Objectives**

The objective of this proficiency test is to obtain information about the quality, accuracy and comparability of the pesticide residue data sent to the European Commission in the framework of the EU and national pesticide monitoring programmes. Participating laboratories will be provided with an assessment of their own analytical performance and the reliability of their data compared to other laboratories.

## **Steps to Follow**

The Proficiency Test is made up of the following 8 steps that are essential for the generation of satisfactory results:

1. Invitation to the participating laboratories. Also supplying details of the web site & web page where they can download the Application Form.
2. Preparation of the test materials. Homogeneity and stability testing performed by the Organiser.
3. Confirmation of the receipt of the participants Application Form and supplying the Laboratory Codes and security code to access to the Pesticide List, the Forms and this Protocol.
4. Payment in advance for the shipment of the test materials, or receipt of a fax demonstrating that the payment procedure has started.
5. Shipment of the test material, together with the blank.
6. The participant laboratories will be responsible for reporting their data to the Organiser using the Forms supplied by the stipulated deadline.
7. The Organiser will evaluate the results at the end of the proficiency test, once the deadline for receipt of results has passed.
8. The Organiser will send a copy of the Final Report to each participant laboratory. This report will include information regarding the design of the test, the homogeneity and stability test results, a record of the shipped samples, a statistical evaluation of the participant's results, graphical displays of the results and conclusions. Any other relevant information considered of value will also be included.

## Evaluation of the Results

The statistics used for the treatment and assessment of the data will be described in detail in the Final Report. A short summary of how the results will be treated is given below.

The results will be grouped into:

- ***False Positives***

These are the results that show the presence of pesticides which are listed in the pesticide list and which are (i) not used in the sample treatment, (ii) and not detected by the organizer even in a repeated analysis. However, if a number of laboratories detect the same additional pesticide, or if the concentration is close to the MPRL, 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.

Nevertheless, any results reported that are lower than 0.01mg/Kg will be ignored by the Organiser and will not therefore be considered as false positives.

- ***False Negatives***

These are results for pesticides that were not reported by the laboratories although they were used by the Organiser to treat the test material and are detected at, or above, the MPRL.

- ***Establishing the true concentration ( $\mu$ )***

The true concentration in all cases will be determined by the median of all the results. Therefore there will be a median value for every pesticide present.

- ***Establishing the assigned value for the standard deviation***

The assigned value for the standard deviation ( $\delta$ ) will be fixed by the Organiser.

$$\text{Where } \delta = b_i * \mu_i \quad \text{being } b_i = \%FFP/100\%$$

An assigned value will be established based on the Fit-For-Purpose (FFP) Standard Deviation model. An average fixed value of 25% has already been chosen. However, the Organizer may increase this value for certain difficult pesticide-crop-concentration combinations, after consultation with the committee of experts, and based on experience gained from previous Proficiency Tests.

## ANNEX 1. Protocol and Instructions. List of pesticides to be sought.

### - z-Scores

This parameter is calculated using this formula:

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

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

Any z-score values of  $|z| > 5$  will be reported as '+5', or '-5'.

z-Score values will be interpreted in the following way:

- $|z| \leq 2$  acceptable
- $2 < |z| \leq 3$  questionable
- $|z| > 3$  unacceptable

For the values considered to be false negative results, z-scores will be calculated using the MPRL values as the value for  $x_i$ .

However, a z-score will not be assigned to any false positive results.

The organizer will consider whether, or not, these values should appear in the histograms.

### - Combined z-Score values

Taking into account all the pesticides analysed, two ways will be used to combine their z-scores: the re-scaled sum of z-scores (RSZ), and the sum of squared z-scores (SSZ).

The equations are:

$$RSZ = \sum z / (n)^{1/2}$$

$$SSZ = \sum z^2$$

$n$  = number of detected pesticides

These formulas will only have informative purposes and will not be used for laboratory evaluations.

In order to evaluate each laboratory's performance, only those laboratories that have reported at least 90% of the pesticides present, and have reported no false positive results, will be classified according to the Weighted Sum of z-Scores. A fixed maximum value of 5 will be used as a default z-score using the following formula:

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

$n$  = number of detected pesticides

So for each lab,

- The first factor is the sum of all their /z-scores/ between zero to two, multiplied by one.
- The second factor is the sum of all their /z-scores/ greater than two but less than or equal to, three, multiplied by three.
- The third factor is the sum of all their z-Scores greater than three, multiplied by five.

These weighted summed z-score results are considered to be less important than the individual z-scores. Therefore the Organizer retains the right to not use them if he feels they are not helpful.

## **Organisation Address**

The official postal address of the organizer, phone number, fax number and e-mail are as follows:

**Universidad de Almería**

**Edificio Químicas CITE I**

**Ctra. Sacramento s/n**

**04120 Almería - Spain**

**Phone Numbers: +34 950015034**

**+34 950015645**

**Fax Number: +34 950015645**

**E-mail: pmedina@ual.es or amadeo@ual.es**

## **On-Line News**

The latest information currently updated can be found at the web address:

**<http://www.ual.es/GruposInv/EUPT07/>**



# European Proficiency Test 07



## Introduction

This proficiency test is based on pesticide residues analysis in grapes. The grapes were grown in Almería, Spain.

The pesticide treatments will be carried out as a post harvest treatment using commercial formulation in micro spray solutions. The test material will be frozen (using liquid nitrogen), chopped, homogenized and sub-sampled into polyethylene bottles that have previously been coded.

Ten of these bottles, containing the test material, will be chosen randomly and analysed by an independent laboratory to check for homogeneity.

The test material will be stored frozen (-20°C) prior to shipment to participants.

Two bottles, again chosen randomly, will be analysed over a period of time to confirm the stability of the pesticides in the test material (firstly when the test materials are shipped, and then a few days after the deadline for receipt of results from the participants). These results will not be included in the statistical analysis of the proficiency test.

The aim is only to check the stability during the shipping process and the proficiency test.

## Calendar

The following table shows the programme for this EUPIT 07

Activity	Date
- Growing the grapes.	December 2004
- Selection of pesticides and design of the web page and protocol.	4 <sup>th</sup> February 2005
- Deadline for receiving the Application Form from invited laboratories.	17 <sup>th</sup> March 2005
- Sample Treatment, Homogenisation, and Storage/Stability Test.	March 2005

## ANNEX 1. Protocol and Instructions. List of pesticides to be sought.

Activity	Date
- Sample distribution.	4 <sup>th</sup> -14 <sup>th</sup> April 2005
- Deadline for receiving Form 1.	15 <sup>th</sup> April 2005
- Deadline for receiving results: Forms 2 and 3.	20 <sup>th</sup> May 2005
- Preliminary Report: <b>only results, no statistical treatment.</b>	7 <sup>th</sup> June 2005
- Final Report.	14 <sup>th</sup> October 2005

## Participant Laboratories

It is up to the contact points/authorities/organisations responsible for the official monitoring of pesticide residues in each country to select the laboratories that should participate, although it is a requirement that a laboratory must be active in contributing results to the national monitoring programme and/or the EU co-ordinated programme. It is up to the participants to fill in and return the Application Form so the Organiser has all their details before the deadline. The Organiser will not be responsible if a laboratory does not receive notice of the web page address necessary to take part in the test.

## Amount of Sample

Approximately 300g of grapes test material will be shipped together with 300g of 'blank' grapes surrounded with dry ice and packed in boxes. The courier costs are charged and must be paid by the participants before shipment of the samples. There will only be a limited amount of test material and laboratories should not ask for more than they require to perform the analysis.

## Application Form

Using the web page site: <http://www.ual.es/GruposInv/EUPT07/> the participating laboratories must complete the application form and return it by e-mail to the Organiser.

In the Application Form there is also information that must be provided in order to make an official invoice. The Application Form must be sent to the Organiser by 17<sup>th</sup> March 2005, at the latest.

## Shipping of Samples

The shipment of the test materials will be carried out during a two-week period because of the differences in the time that it takes to reach the different participant countries (from 2-5 days). A warning message will be sent out a the week before shipment, and laboratories must make arrangements for the reception of the test materials. They should let the Organiser know of any possible public holidays in their country/city during the delivery time mentioned in the calendar and make every effort to receive the shipment even if the laboratory is closed.

## Form 1

Once the laboratory has received the test materials they **must** complete Form 1, filling in the date of receipt, the condition of the test material, and its acceptance. Form 1 has a deadline, so if it is not returned by e-mail latest 15<sup>th</sup> April 2005, the Organiser will assume the laboratory has received and accepted the test material.

Please note that you must include the laboratory code assigned to you on this form.

## Analyses and Results Forms (Form 2)

### ***Significant Figures***

The results must be expressed in mg/kg in the following way:

- concentrations <0.100 mg/kg, to be expressed to two significant figures (three decimals places, i.e. 0.058 mg/kg).
- concentrations > 0.100 mg/kg, to be expressed to three significant figures, i.e. 0.156, 1.64, 10.3 mg/kg.

In cases where a pesticide was not detected, it should be recorded as ND. If it was not sought, it should be recorded as NA.

The results/concentrations must be reported as numbers. Any other form of data will not be considered.

## **Correction of Results (Form 2)**

The results must **not** be corrected using recovery factors. If the laboratory usually corrects the results for their recoveries, they should provide the correction factor used for each pesticide as informative data only. This information must be sent together with the results in Form 2.

## **Samples Material for Analysis (Form 2)**

The test material contains a certain number of pesticides from the Pesticide List. Please read carefully the list in Form 2 since the residue definitions are not given (see the Pesticide List).

It should **not** be assumed that only pesticides registered for use on grapes will be present.

Each laboratory must report only **one** result for each of the pesticides present in the test material, using their normal routine analytical procedure(s). This does not mean that more than one method has to be used to cover all the compounds present.

The analytical procedure used must be reported using Form 2. The results, expressed as concentration levels in mg/kg, must also be reported, together with the laboratories reporting level (RL) for each pesticide. This level will only be used for information.

Form 2 must be sent to the Organiser by 20<sup>th</sup> of May 2005, at the latest. Results received after this date will not be included in the statistical treatment, or in the final report. The laboratories are responsible for reporting their results to the Organiser. The Organiser will acknowledge receipt of the results by e-mail.

Please note that you must include the laboratory code assigned to you on this Form.

## **Analytical Procedures Used (Form 3)**

A brief summary of the analytical procedure(s) used is required from each laboratory on Form 3.

If more than one method has been used, please label them with different letters or codes in Form 2, and use as many copies of Form 3 as are needed (one for each method).

The organizer must receive Form 3 by mail by 20<sup>th</sup> May 2005, at the latest.

Please note that you must include the laboratory code assigned to you on this Form.

## **Sample Manipulation Advises**

Once received, the test material must be stored frozen until it is to be analysed.

- Allow the test material to defrost in the refrigerator the afternoon before the analysis is performed.

- Once defrosted, be sure to mix the contents of the bottle thoroughly, to ensure homogeneity of the test material, before taking the analytical portion(s).



# Form 1

Laboratory Code:

EUPT07-Lab-

Test material code:

(check the blank bottle  
and the test material)

Date of receipt:

/ /2005

EUPT07-blank-

EUPT07-sample-

Loses:

YES

NO

Frozen:

YES

NO

accept the test material. I do not need more.

Please, fill in this form and send it back by e-mail ([pmedina@ual.es](mailto:pmedina@ual.es)) as soon as you have received the test material, latest 15<sup>th</sup> April 2005.

If no form is received by the Organiser, it will be assumed that the test material has been accepted by the laboratory.

Signature:

Laboratories should fill in this form and send it to the following e-mail: [pmedina@ual.es](mailto:pmedina@ual.es)



# Form 2 (Results)

Laboratory Code:  Date:

Test material and blank code:

Pesticide	Scope of your Method (1)	Analytical Procedure (2)	Conc. (mg/kg) (3)	Quantification Using Standards in Solvents or Matrix (4)	Confirmation Method (5)	RL (mg/kg) (6)	Recovery (7)
Acephate							
Acetamiprid							
Acrinathrin							
Aldicarb							
Azinphos-methyl							
Azoxystrobin							
Bifenthrin							
Bromopropylate							
Bupirimate							
Captan							
Carbaryl							
Carbendazim							
Chlorothalonil							
Chlorpropham							
Chlorpyrifos							
Chlorpyrifos-methyl							
Cypermethrin							
Cyprodinil							
Deltamethrin							
Diazinon							
Dichlofuanid							
Dicofol							
DIMETHOATEe							
Dimethomorph							
Endosulfan							
Fenhexamid							
Fenitrothion							
Fludioxonil							
Flusilazole							
Imazalil							
Imidacloprid							
Iprodione							
Kresoxim-methyl							
Lambda-cyhalothrin							

**ANNEX 1. Protocol and Instructions. List of pesticides to be sought.**

Pesticide	Scope of your Method (1)	Analytical Procedure (2)	Conc. (mg/kg) (3)	Quantification Using Standards in Solvents or Matrix (4)	Confirmation Method (5)	RL (mg/kg) (6)	Recovery (7)
Malathion							
Metalaxyl							
Methamidophos							
Methidathion							
Methiocarb							
Methomyl							
Monocrotophos							
Myclobutanil							
Oxydemeton-methyl							
Parathion							
Parathion-methyl							
Penconazole							
Phosalone							
Pirimicarb							
Pirimiphos-methyl							
Procymidone							
Propargite							
Pyrimethanil							
Spiroxamine							
Tetraconazole							
Thiabendazole							
Tolcloflos-methyl							
Tolyfluanid							
Triadimefon							
Vinclozolin							

- (1) If the pesticide is not included in your analysis, fill **NA**. If the pesticide is detected, fill **D**. If the pesticide is NOT detected, fill **ND**.
- (2) Write the same code as you use in Form 3 for the analytical method used, e.g. A, B, C...
- (3) Concentration, report only one result. Record the concentrations for all pesticides according to the residue definition given in the Pesticide List.
- (4) Standards: **S** = standard/calibration in pure solvent, **M** = standard/calibration in matrix extract
- (5) Give the determination technique used e.g. **GC-FPD, HPLC-UV, GC-MS, LC-MS, LC-MS/MS**
- (6) **RL** Your Reporting Level, must be given for all pesticides. For pesticides with metabolites/degradation products included in the MRL definition, give the "Reporting Level" for the global compound (see residue definition in the pesticide list).
- (7) The concentration/results reported in (3) must not be corrected using recovery factors even if the laboratory usually corrects them. Nevertheless, you may give the correction factor for each pesticide as informative data.

**I agree to be responsible for completing and returning this form to the Organizer latest 20<sup>th</sup> May 2005. In case of no e-mail confirmation of reception of this document (in 3 or 4 days), I will contact the Organizer as soon as possible.**

Signature:

Laboratories should fill in this form and send it to the following e-mail: [pmedina@ual.es](mailto:pmedina@ual.es)



## Form 3 (Analytical Procedures Used)

Laboratory Code:

Date:

**Complete one of these forms for every different analytical procedure used**

Analytical Procedure (2): \_\_\_\_\_

Sample Weight (g): \_\_\_\_\_ Extraction solvent/s (7): \_\_\_\_\_

CLEAN-UP step (8): \_\_\_\_\_ Internal standard (if any): \_\_\_\_\_

Injection Volume: \_\_\_\_\_ Injection Type: \_\_\_\_\_

Determination (9): \_\_\_\_\_

Reference Method: \_\_\_\_\_

Signature:

I agree to be responsible for delivering this form to the Organizer. In case of no e-mail confirmation of receipt of this form (in 3 or 4 days), I will contact the Organizer as soon as possible.

**Please return this Form latest 20<sup>th</sup> May, 2005**

(2) Write the same code as you use in Form 2 for the analytical method used, e.g. A, B, C...

(7) Denoted as **1** = ethyl acetate, **2** = acetone followed by cyclohexane and ethyl acetate, **3** = acetone followed by dichloromethane, **4** = acetone followed by dichloromethane and petroleum ether, **5** = acetonitrile, **6** = others.

(8) **Clean-up:** GPC = gel permeation chromatography, SPE = solid phase extraction, LL = liquid-liquid partition, NO = no clean-up, O = other clean-up method

(9) **Determination Technique:** e.g. GC-ECD, GC-NPD, GC-FPD, GC-MS (single-quad), GC-MS(ITD), HPLC-FL, HPLC-UV, HPLC-DAD, LC-MS, LC-MS/MS

Laboratories should fill in this form and send it to the following e-mail: [pmedina@ual.es](mailto:pmedina@ual.es)



# Pesticide List

NAME	MPRL (mg/kg)
Acephate	0.02
Acetamiprid	0.05 #
Acrinathrin	0.05 #
Aldicarb (Aldicarb + Aldicarb Sulphone + Aldicarb Sulphoxide expressed as Aldicarb)	0.05
Azinphos-methyl	0.05 #
Azoxystrobin	0.05
Benomyl (See Carbendazim)	
Bifenthrin	0.05
Bromopropylate	0.05
Bupirimate	0.05 #
Captan (Captan + Folpet expressed as Captan)	0.05 #
Carbaryl	0.05 #
Carbendazim (Benomyl + Carbendazim + Thiophanate-methyl, expressed as Carbendazim)	0.1

**ANNEX 1. Protocol and Instructions. List of pesticides to be sought.**

NAME	MPRL (mg/kg)
Chlorothalonil	0.01
Chlorpropham	0.05 #
Chlorpyriphos	0.05
Chlorpyriphos-methyl	0.05
Cypermethrin	0.05
Cyprodinil	0.05 #
Deltamethrin	0.05
Diazinon	0.02
Dichlofluanid	0.05 #
Dicofol	0.02
DIMETHOATEe (DIMETHOATEe + Omethoate, expressed as DIMETHOATEe)	0.02
Dimethomorph	0.05 #
Endosulfan ( $\alpha+\beta$ Endosulfan + Endosulfan Sulphate, expressed as Endosulfan)	0.05
Fenhexamid	0.05 #
Fenitrothion	0.05 #
Fludioxonil	0.05 #

**ANNEX 1. Protocol and Instructions. List of pesticides to be sought.**

NAME	MPRL (mg/kg)
Flusilazole	0.05 #
Folpet (see Captan)	
Imazalil	0.02
Imidacloprid (only parent compound)	0.05 #
Iprodione	0.02
Kresoxim-methyl	0.05
Lambda-cyhalothrin	0.02
Malathion (Malathion + Malaoxon, expressed as Malathion)	0.05 #
Metalaxy	0.02
Methamidophos	0.01
Methidathion	0.02
Methiocarb (Methiocarb + Methiocarb sulphone + Methiocarb sulphoxide, expressed as Methiocarb)	0.05 #
Methomyl (Methomyl + Thiodicarb, expressed as Methomyl)	0.05
Monocrotophos	0.03 #
Myclobutanil	0.02
Omethoate (see DIMETHOATEe)	

**ANNEX 1. Protocol and Instructions. List of pesticides to be sought.**

NAME	MPRL (mg/kg)
Oxydemeton-methyl (Oxydemeton-methyl + Demeton-S-Methylsulfon, expressed as Oxydemeton-methyl)	0.02
Parathion	0.05
Parathion-methyl (Parathion-methyl + Paraoxon-methyl expressed as Parathion-methyl)	0.05 #
Penconazole	0.05 #
Phosalone	0.05 #
Pirimicarb	0.05 #
Pirimiphos-methyl	0.05
Procymidone	0.02
Propargite	0.05 #
Pyrimethanil	0.05 #
Spiroxamine	0.05
Tetraconazole	0.05 #
Thiabendazole	0.05
Thiodicarb (see Methomyl)	
Thiophanate-methyl (see Carbendazim)	
Tolclofos-methyl	0.05 #

**ANNEX 1. Protocol and Instructions. List of pesticides to be sought.**

NAME	MPRL (mg/kg)
Tolyfluanid	0.05 #
Triadimefon (Triadimefon + Triadimenol expressed as Triadimefon)	0.1
Triadimenol (see Triadimefon)	
Vinclozolin (only parent compound)	0.05

MPRL (Minimum Performance Reporting Level) is set at the same level as MRL\* in the EU Directives ("lower limit of analytical determination"). If no EU-MRL are set at the lower limit of analytical determination or yet fixed, the MPRL is set by the EUPT-7 organizing committee and the figure is followed by #.



**ANNEX 2. List of laboratories invited to participate in PT7.**

COUNTRY	CITY	Laboratory Name	Reported Results
AUSTRIA	Innsbruck	Austrian Agency for Health and Food Safety Joint services Competence Centre Plant Protection Products Residues	Yes
AUSTRIA	Vienna	Competence Centre Residue Analysis Vienna	Yes
BELGIUM	Brussels	Scientific Institute of Public Health (IPH)	Yes
BELGIUM	Zwijnaarde	Fytolab	Yes
CYPRUS	Nicosia	State General Laboratory	Yes
CYPRUS	Nicosia	Pesticide Residue Laboratory, Analytical Laboratories Section, Department of Agriculture	Yes
CZECH REPUBLIC	Prague 6	Institute of Chemical Technology (VŠCHT Praha)	Yes
CZECH REPUBLIC	Praha 5	Statni zemedelska a potravinarska Inspeckce (Czech Agriculture and Food Inspectorate)	Yes
DENMARK	Copenhagen V	Fodevareregion København	Yes
DENMARK	Soeborg	Danish Institute for Food and Veterinary Research	Yes
ESTONIA	Saku (Harjumaa)	Lab for Residues and Contaminants of Agricultural Research Centre	Yes
ESTONIA	Tartu	Health Protection Inspectorate Laboratory	Yes
FINLAND	Espoo	Finnish Customs Laboratory	Yes
FINLAND	Helsinki	Enviroment Centre of Helsinki	Yes
FRANCE	Illkirch	Laboratoire DGCCRF Strasbourg	Yes
FRANCE	Massy Cedex	DGCCRF-Laboratoire de Paris Massy	Yes
FRANCE	Montpellier Cedex 5	Laboratoire de la DGCCRF	Yes
FRANCE	Rennes	Laboratoire DGCCRF-Rennes	Yes
FRANCE	Talence	DGCCRF Laboratoire de Bordeaux	Yes
FRANCE	Villeneuve d'Ascq	D.G.C.C.R.F.- L59	No
GERMANY	Aachen	Chemisches und Lebensmitteluntersuchungsamt Stadt Aachen	Yes
GERMANY	Berlin	BBGes-ILAT, FB 26	Yes
GERMANY	Bielefeld	Chemisches Untersuchungsamt OWL	Yes
GERMANY	Bonn	Amt für Umwelt, Verbraucherschutz und Lokale Agenda	Yes

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COUNTRY	City	Laboratory Name	Reported Results
GERMANY	Bremen	Landesuntersuchungsamt Bremen für Chemie, Hygiene und Veterinärmedizin	Yes
GERMANY	Dortmund	Chemisches Untersuchungsamt der Stadt Dortmund	Yes
GERMANY	Dusseldorf	Amt für Verbrauchers.-Chem. Lebensmittelunters.	Yes
GERMANY	Erfurt	Thüringer Landesamt für Lebensmittelsicherheit und Verbraucherschutz (TLLV)	Yes
GERMANY	Erlangen	Bayer. Landesamt für Gesundheit und Lebensmittelsicherheit	Yes
GERMANY	Essen	CGI Essen/Oberhausen	Yes
GERMANY	Fellbach	CVUA Stuttgart	Yes
GERMANY	Hagen	Chemisches Untersuchungsamt der Stadt Hagen	Yes
GERMANY	Halle	Landesamt für Verbraucherschutz, Fachbereich 3	Yes
GERMANY	Hamburg	Institut Fur Hygiene und Umwelt	Yes
GERMANY	Kassel	Hessisches Landeslabor, Standort Kassel	Yes
GERMANY	Münster	Chemisches Landes- und Staatliches Veterinäruntersuchungsamt	Yes
GERMANY	Neumuenster	Landeslabor Schleswig-Holstein	Yes
GERMANY	Oldenburg	LAVES Lebensmittelinstitut Oldenburg	Yes
GERMANY	Potsdam	Landeslabor Brandenburg; Fachbereich L5	Yes
GERMANY	Recklinghausen	Gemeinsames Chemisches und Lebensmitteluntersuchungsamt für den Kreis Recklinghausen und die Stadt Gelsenkirchen (CEL)	Yes
GERMANY	Rostock	Landesveterinär- und Lebensmitteluntersuchungsamt M-V	Yes
GERMANY	Saarbrücken	LVGA Landesamt für Verbraucher-, Gesundheits- und Arbeitsschutz; Abt. G	Yes
GERMANY	Speyer	Landesuntersuchungsamt, Institut fur Lebensmittelchemie Speyer	Yes
GERMANY	Trier	Landesuntersuchungsamt-Institut für Lebensmittelchemie Trier	Yes
GERMANY	Wiesbaden	Hessisches Landeslabor	Yes
GERMANY	Wuppertal	Chemisches Untersuchungsinstitut Bergisches Land	Yes

**ANNEX 2. List of laboratories invited to participate in PT7.**

COUNTRY	City	Laboratory Name	Reported Results
GREECE	Athens	General Chemical State Laboratory. Division Pesticide Residue	Yes
GREECE	Athens	RCPP&QC, Pesticide Residue Laboratory	Yes
GREECE	Ioannina	Regional Centre of Plant Protection and Quality Control	Yes
GREECE	Iraklion, Crete	Regional Centre of Plant Protection and Quality Control of Iraklion	Yes
GREECE	Kavala	Ministry Rural Development and Food, Peripheral Center of Kavala	Yes
GREECE	Kifissia, Athens	Pesticide Residues Laboratory, Benaki Phytopathological Institute	Yes
GREECE	Thessaloniki	Regional Center of Plant Protection and Quality Control	Yes
HUNGARY	Fácánkert	Pesticide Residue Analytical Laboratory of Plant Protection and Soil Conservation Service of Tolna County	Yes
HUNGARY	Hódmezovásárhely	Plant Health and Soil Conservation Service of Csongrad County	Yes
HUNGARY	Kaposvár	Plant Protection and Soil Conservation Service of Somogy County	Yes
HUNGARY	Miskolc	Pesticide Residue Analytical Laboratory PPSCS-BAZ	Yes
HUNGARY	Nyiregyháza	Plant Protection and Soil Conservation Service of Szabolcs-Szatmár-Bereg county	Yes
HUNGARY	Szolnok	Plant Protection and Soil Conservation Service	Yes
HUNGARY	Tanakajd	Pesticide Residue Analytical Laboratory of County Vas	Yes
HUNGARY	Velence	Plant Protection and Soil Conservation Service of Féjer County	Yes
ICELAND	Reykjavík	Environment and Food Agency of Iceland	Yes
IRELAND	Dublin	Pesticide Residue Laboratory. Abbotstown Laboratory Complex	Yes
ITALY	Arezzo	ARPAT DIP. di Arezzo	Yes
ITALY	Bari	ARPA Puglia - Dipartimento di Bari	Yes
ITALY	Bolzano	Agenzia Ambiente Bolzano	Yes
ITALY	Cagliari	Presidio Multizionale di Prevenzione Cagliari - Area Chimica-	Yes
ITALY	Catania	ARPA Sicilia-DAP Catania	Yes

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COUNTRY	CITY	Laboratory Name	Reported Results
ITALY	Chiesuol del Fosso (Ferrara)	ARPA Sezione di Ferrara	Yes
ITALY	La Loggia (Torino)	A.R.P.A Piemonte - Polo Regionale Alimenti	Yes
ITALY	La Spezia	A.R.P.A.L. Laboratorio "Centro Pesticidi Regionale"	Yes
ITALY	Nuoro	Presidio Multizionale di Prevenzione Area Chimica	Yes
ITALY	Pordenone	ARPA - FVG Dipartimento di Podenone	Yes
ITALY	Ragusa	AUSL n7 Ragusa DAP RAGUSA ARPA SICILIA L.I.P. Sezione Chimica	Yes
ITALY	Roma	Dip. Ambiente e Connessa Prevenzione Primaria-Reparto Antiparassitari	Yes
ITALY	Saint Christophe (Aosta)	ARPA Valle Dáosta	Yes
ITALY	Sondrio	ARPA-Dip. Sondrio-UO Laboratorio	Yes
ITALY	Trento	A.P.P.A. Trento-Settore Lab. e Controlli	Yes
ITALY	Verona	APRAV-Verona	Yes
ITALY	Vicenza	A.R.P.A.V. Dipartimento di Vicenza	Yes
LATVIA	Riga	State Veterinary Medicine Diagnostic Centre	Yes
LATVIA	Riga	State Agency "Public Health Agency". Laboratory of Chemistry	Yes
LITHUANIA	Vilnius	National Veterinary Laboratory	Yes
LUXEMBOURG	Luxembourg	Côntrole des Denrées Alimentaries	Yes
NORWAY	Aas	Norwegian Crop Research Institute, Pesticide Laboratory	Yes
POLAND	Bialystok	Plant Protection Institute, Experimental Field Station in Bialystok	Yes
POLAND	Katowice	Wojewodzka Stacja Sanitarno-Epidemiologiczna	Yes
POLAND	Kielce	Wojewodzka Stacja Sanitarno-Epidemiologiczna	Yes
POLAND	Krakow	Wojewodzka Stacja Sanitarno-Epidemiologiczna	Yes
POLAND	Lodz	Wojewodzka Stacja Sanitarno-Epidemiologiczna	Yes
POLAND	Opole	Wojewodzka Stacja Sanitarno-Epidemiologiczna	Yes
POLAND	Poznan	Plant Protection Institute, Department of Pesticide Residue Research	Yes

**ANNEX 2. List of laboratories invited to participate in PT7.**

COUNTRY	CITY	Laboratory Name	Reported Results
POLAND	Poznan	Wojewodzka Stacja Sanitarno-Epidemiologiczna	Yes
POLAND	Rzeszow	Wojewodzka Stacja Sanitarno-Epidemiologiczna	Yes
POLAND	Rzeszów	Plant Protection Institute	Yes
POLAND	Skierniewice	Laboratory of Contamination & Pesticides Residue Analyses. Research institute of Pomology and Floriculture	Yes
POLAND	Sosnicowice	Plant Protection Institute	Yes
POLAND	Trzebnica	Plant Protection Institute	Yes
POLAND	Warsaw	National Institute of Hygiene. Dept. Environmental Toxicology	No
PORTUGAL	Camacha	Laboratório Agrícola Da Madeira	Yes
PORTUGAL	Oeiras	Direcção-Geral de Protecção das Culturas	Yes
ROMANIA	Bucuresti	Central Laboratory for Pesticide Residues Control	Yes
SLOVAKIA	Bratislava	State Veterinary and Food Institute	Yes
SLOVENIA	Ljubljana	Agricultural Institute of Slovenia, Central Laboratories	Yes
SLOVENIA	Ljubljana	Institute of Public Health	Yes
SLOVENIA	Maribor	Public Health Institute, Environmental Protection Institute	Yes
SPAIN	Almeria	Laboratorio de la Dirección Territorial del Comercio (SOIVRE de Almería)	Yes
SPAIN	A Coruña	Laboratorio Agrario y Fitopatológico de Galicia	Yes
SPAIN	Alicante	Laboratorio SOIVRE de Alicante	Yes
SPAIN	Aravaca (Madrid)	Laboratorio Arbitral Agroalimentario del MAPA	Yes
SPAIN	Burgos	Laboratorio Agrario Regional. Junta de Castilla y León	Yes
SPAIN	Burjassot (Valencia)	Agroalimentario Generalitat Valenciana	Yes

**ANNEX 2. List of laboratories invited to participate in PT7.**

COUNTRY	City	Laboratory Name	Reported Results
SPAIN	Cabril	Laboratori Agroalimentri Generalitat de Catalunya	Yes
SPAIN	El Palmar, Murcia	Laboratorio Agroalimentario y de Sanidad Animal	Yes
SPAIN	Jaén	Laboratorio de Sanidad Vegetal	Yes
SPAIN	La Mojonera, Almeria	Laboratorio de Producción y Sanidad Vegetal de Almería	Yes
SPAIN	Logroño	Laboratorio Regional de la CCAA La Rioja	Yes
SPAIN	Majadahonda (Madrid)	Centro Nacional de Alimentacion	Yes
SPAIN	Murcia	S.O.I.V.R.E. Murcia	Yes
SPAIN	Sevilla	Laboratorio del Catice (SOIVRE) de Sevilla	Yes
SPAIN	Villava Navarra	Gobierno de Navarra - Negociado de Análisis Instrumental	Yes
SPAIN	Zizurkil	Laboratorio Agrario. Diputación Foral de Gipuzkoa	No
SWEDEN	Lidköping	AnalyCen Nordic AB	Yes
THE NETHERLANDS	Amsterdam	VWA-Food and Consumer Product Safety Authority	Yes
UNITED KINGDOM	Edinburgh, Scotland	Scottish Agricultural Science Agency	Yes
UNITED KINGDOM	Teddington, Middlesex	Laboratory of the Government Chemist Limited	Yes
UNITED KINGDOM	Wolverhampton	Direct Laboratories	Yes
UNITED KINGDOM	York	Central Science Laboratory	Yes