

**EUROPEAN UNION PROFICIENCY TEST FOR  
PESTICIDES IN FRUITS AND VEGETABLES FOR  
SCREENING METHODS 02  
(EUPT-FV-SM-02)  
2010**

**Pesticide Residues in Leek Homogenate**

**Final Report**

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QCG: Quality Control Group

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# EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUITS AND VEGETABLES

## SCREENING METHODS 02

2010

### BACKGROUND

According to Article 28 of Regulation 396/2005/EC of the European Parliament and European Council regarding maximum residue levels of pesticides in, or on, food and feed of plant and animal origin<sup>1</sup>: all laboratories analysing samples for the official control on pesticide residues shall participate in the European Union Proficiency Tests (EUPTs) for pesticide residues, facilitated by the Commission. These proficiency tests are carried out on an annual basis in order to ensure the quality, accuracy and comparability of the residue data reported by EU Member States to the European Commission, as well as by other Member States within the framework of co-ordinated and national monitoring and surveillance programmes.

Regulation (EC) No 882/2004<sup>2</sup> lays down the general tasks, duties and requirements for European Union Reference Laboratories (EURLs) for Food, Feed and Animal Health. Among these tasks is the provision of independently-organised comparative tests. This year, for a second time, the EURL for pesticides in Fruit and Vegetables at the University of Almería, Spain<sup>3</sup> organized a proficiency test on qualitative screening methods for pesticides in vegetable/fruit commodities. This test was organised because many laboratories have invested in modern higher mass accuracy MS systems that allows them to greatly increase their analytical scope by using screening methods.

Because the use of such screening methods is not yet common practise amongst all EU laboratories involved in official monitoring, participation in this PT was on a purely voluntary basis. Another reason for not making this PT mandatory was that a PT for quantitative pesticide multi-residue analysis (EUPT-FV12) had been organised in the same time period. Nevertheless, all FV-NRLs and FV-Official laboratories involved in the determination of pesticide residues in fruit and vegetables for the EU-coordinated monitoring programme or for their own national programmes were invited to take part.

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

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<sup>1</sup> Regulation (EC) No 396/2005, published at OJ of the EU L70 of 16.03.2005, as last amended by Regulation 839/2008 published at OJ of the EU L234 of 30.08.2008.

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

<sup>3</sup> Commission Regulation (EC) No 776/2006 of 23 May 2006 - amending Annex VII to Regulation (EC) No 882/2004 of the European Parliament and of the Council as regards Community Reference Laboratories.

## 1. INTRODUCTION

Last year the first pesticide screening method inter-laboratory test was well accepted by laboratories. Therefore the EURL-FV has decided to continue this year with a second test. The main reason for this broad acceptance is that the support for this type of approach, especially from DG SANCO.

From last year it was found that many laboratories not only used a full scan approach to perform screening but some also employed modern tandem-mass spectrometers, even if the sensitivity had to be reduced.

Mass spectrometry plays an essential role in everyday work carried out by laboratories. It is used typically for targeted analysis and the scope of many official laboratories is around 150 pesticides. Technological improvements in modern MS systems offer new possibilities for greatly increasing the scope of MRM analysis. Whereas full-scan measurement is theoretically the best approach for MS screening, developments in targeted measurement also offers the potential for a substantially increased scope of analysis. Another reason for conducting this screening method proficiency test is to gain information from the laboratories on the type of software that they use to process MS data. Whether laboratories are using commercial software and databases, or whether they are internally constructed and searched manually. This information provides an overall view of the purpose of this type of test, and if in a near future, there is the need to further develop the software.

The aim of the EURL-FV is for laboratories to be able to use mass spectrometry based screening methods routinely, following validation. This is in line with the new Document SANCO/10684/2009 ("Method validation and quality control procedures for pesticide residues analysis in food and feed"). This document is available in our webpage if required.

Only qualitative information will be requested for those pesticides that are detected. It has also been decided by the Quality Control Group, and based on the received questionnaires, that a target pesticide list **will not be provided**.

Regulation (EC) No 882/2004 lays down the general tasks, duties and requirements for EU-RLs for Food, Feed and Animal Health. Among these tasks is the provision of independently organised comparative tests. As last year the EURL for pesticides in Fruit and Vegetables at the University of Almería, Spain, has organised a proficiency test on qualitative screening methods for pesticides in vegetable/fruit commodities. This EUPT-FV-SM is directed at all National Reference Laboratories (**NRLs**) and all Official Laboratories (**OfLs**) in the EU Member States for Fruits and Vegetables. Laboratories outside this EURL/NRL/OfL-Network may also be allowed to participate on a case-by-case basis, after consultation with DG SANCO.

## 2. TEST MATERIALS

### 2.1 Test material

This proficiency test is based on pesticide residues detection in leeks. The leeks to be used in this study were grown in Catalunya, Spain.

The pesticide treatments on the leeks were carried out post-harvest by spiking with a mixture of standard solutions. The leeks were then frozen (using liquid nitrogen), chopped, homogenized and sub-sampled into polyethylene bottles that had previously been coded.

Ten of these bottles containing the prepared test material, were chosen randomly, and analysed to check the presence of all the spiked pesticides.

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

Two bottles, again chosen randomly, were analysed over a period of time to confirm the stability of the pesticides in the test material (firstly when the test material was shipped, and then a few days after the deadline for receipt of participants' results). There was an extra analysis during this period, where the test material was maintained at room temperature for a few days, again to check if there had been any degradation of any of the pesticides.

The pesticides used to spike the leek test material were decided by the Quality Control Group. Based on the received questionnaires from previous test it was decided that a target pesticide list would not be provided to participants. The pesticides selected to treat the test material for this EUPT-FV-SM02 were chosen taking into account the following considerations:

- Pesticides that were **not included** in the Coordinated Multiannual Community Control Programme for 2010 (Regulation (EC) 901/2009).
- Pesticides with toxicological interest.

**Table 2.1. Spiked Pesticides**

Spiked Pesticides		
Cyromazine	Isofenphos-methyl	Picolinafen
N,N-Diethyl-meta-toluamide(Deet)	Isoprocarb	Prometryn
Diuron	Mecarbam	Propazine
Ethoxyquin	Metolachlor	Propoxur
Fenpropidin	Metribuzin	Pyraclostrobin
Fenpyroximate	Mevinphos	Quinoclamine
Furathiocarb	Phorate	Simazine

## 2.2 Analytical methods

The two analytical methods described briefly below were used by the Organiser for the homogeneity and stability tests performed by the EURL-FV. These were:

- GC method [1]: gas chromatography/mass spectrometry (GC-q-MS) using electron impact (EI) ionisation and full-scan acquisition.
- LC method [2]: LC-TOF-MS using electrospray ionisation and operating in the positive ion mode

### 2.2 Check analyses for the presence of the pesticides in the spiked leek test material

The organiser homogeneity and stability tests associated with 'quantitative' PTs were not necessary. Hence the PT test material was only analysed in order to detect the presence of all the spiked pesticides.

Ten test materials spiked were randomly chosen from those stored in the freezer and analysed by duplicate in order to check the presence of the pesticides. The injection sequence of the 10 analyses by GC and LC were determined from a table of randomly generated numbers. Table 2.2.1 shows the summary of those tests.

Table 2.2.1 Homogeneity tests

Test material Number	097 a	097 b	009 a	009 b	063 a	063 b	057 a	057 b	049 a	049 b	006 a	006 b	012 a	012 b	079 a	079 b	087 a	087 b	070 a	070 b
Cyromazine	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
N,N-Diethyl-meta-toluamide(Deet)	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
Diuron	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
Ethoxyquin	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
Fenpropidin	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
Fenpyroximate	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
Furathiocarb	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
Isofenphos methyl	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
Isoprocab	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
Mecarbam	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
Metolachlor	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
Metribuzin	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
Mevinphos	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
Phorate	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
Picolinafen	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
Prometryn	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
Propazine	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
Propoxur	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
Pyraclostrobin	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
Quinoclamine	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
Simazine	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D
Carbofuran, Carbofuran-OH	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D

D: Detected

We have marked carbofuran with a different colour, as it was not used to spike the test material but was found to be present probably as a consequence of furathiocarb degradation, because it was not present in the blank material. This pesticide is not included in the EUPT-FV-SM02 evaluation because it is included in the Coordinated Multiannual Community Control Programme for 2010 (Regulation (EC) 901/2009 and so, it was not an objective of this PT. The carbofuran data presented has only informative value.

Stability Test:

Further analyses following different elapses of time to test for stability were performed. On each occasion, a test material stored in the freezer at -20°C was randomly chosen and analysed.

The three occasions were:

- Day 1: coinciding with the test material shipment, which took place on 12<sup>th</sup> April 2010.
- Day 2: soon after the deadline for reporting results, on 16<sup>th</sup> April 2010.
- Day 3: some days after the test material had been maintained at room temperature, on 21<sup>st</sup> April 2010.

For all the analyses, the two analytical methods described briefly above (in section 2.1) were used.

Table 2.2.2 Stability tests

Test material Number	075 Day 1	075 Day 2	075 Day 3	098 Day 1	098 Day 2	098 Day 3
Cyromazine	D	D	D	D	D	D
Deet	D	D	D	D	D	D
Diuron	D	D	D	D	D	D
Ethoxyquin	D	D	D	D	D	D
Fenpropidin	D	D	D	D	D	D
Fenpyroximate	D	D	D	D	D	D
Furathiocarb	D	D	D	D	D	D
Isofenphos-methyl	D	D	D	D	D	D
Isoprocarb	D	D	D	D	D	D
Mecarbam	D	D	D	D	D	D
Metolachlor	D	D	D	D	D	D
Metribuzin	D	D	D	D	D	D
Mevinphos	D	D	D	D	D	D
Phorate	D	D	D	D	D	D
Picolinafen	D	D	D	D	D	D
Prometryn	D	D	D	D	D	D
Propazine	D	D	D	D	D	D
Propoxur	D	D	D	D	D	D
Pyraclostrobin	D	D	D	D	D	D
Quinoclamine	D	D	D	D	D	D
Simazine	D	D	D	D	D	D
Carbofuran, Carbofuran-OH	D	D	D	D	D	D

D: Detected



The aim of these tests was to demonstrate the detectability of all the pesticides added to the treated test materials at shipment. All the pesticides that had been spiked into the test material were detected on all occasions.

#### **2.4 Distribution of test materials and protocol to participants**

The test material, approximately 300 g of leek homogenate containing residues of pesticides, together with another 300 g of 'blank' leek homogenate, were shipped to participants on the 12<sup>th</sup> April 2010. The deadline for the submission of results to the Organiser was 72 hours after receipt of the test material. Participants were not provided with a target list of pesticides that may have been used to spike the test material and they were asked to report all the pesticides that they detected.

Laboratories were asked to screen the test materials using the wide-scope screening methods they normally apply, or anticipate applying, for official monitoring purposes. This typically involves full-scan techniques like GC-MS (full-scan quadrupole, ion trap, ToF) and/or LC-TOF-MS and Orbitrap. However, extended targeted methods using LC tandem MS (triple quadrupole, Q-trap, Q-TOF) or GC-MS/MS could also be used.

Before shipment, the laboratories had received full instructions (Annex 1) for the receipt and analysis of the spiked test material although they were encouraged to use their own screening methods. These instructions, laid out as Protocol, were uploaded onto the EUPT-FV-SM02 web page, designed especially for this Proficiency Test. This information was also sent by e-mail to all participant laboratories. The Application Form was uploaded onto this same web site; together with Forms 0 (Sample Receipt) and Form 1 (Results), these allowed the evaluation of the mass-spectrometric screening methods that each one of the participants used.

### 3. STATISTICAL METHODS

#### 3.1 False positives (other Reported Pesticides) and negatives (Not Reported Pesticides)

##### 3.1.1 False positives (Other Reported Pesticides)

These will be considered as those results that show the apparent presence of pesticides which were: (i) not used in the test material treatment, (ii) not detected by the Organiser, even after repeated analyses. However, if a number of participants detect the same additional pesticide(s), then a decision as to whether, or not, this should be considered to be a false positive ("Other Reported Pesticide") result will be made on a case-by-case basis.

##### Organiser Notes:

- Not all screening methods immediately provide sufficient information to allow full identification. In such cases, in real-life, laboratories normally do a follow-up confirmatory analysis when they detect a pesticide with e.g. using LC-MS/MS and based on only one transition. In future PTs of this nature, there will be a need to distinguish between suspect or tentative detects and full identifications.
- The Scientific Committee considered that the term "Other Reported Pesticide" is more suitable than "False Positive" for the EUPT-FV-SM.

##### 3.1.2 False negatives (Not Reported Pesticides)

These will be considered as the absence of result for any present pesticide that the Organiser has used to spike the test material, and was detected by the majority of participants.

Organiser Note: This year the term "False Negative" has been changed by the term "Not Reported Pesticide" (NR). The Scientific Committee considers NR more consistent in those cases where no fixed scope is established in the Protocol.

## 4. RESULTS

### 4.1 Summary of reported results

Fifty laboratories agreed to participate in this first proficiency test on screening methods. Forty-four laboratories submitted results. All results reported by the participants are given in Appendix 1. Graphical representations of the results reported are shown in Appendix 2. Details of the screening methods used are provided in Appendix 3. The laboratories that agreed to participate are listed in Annex 2.

A summary of the results reported by pesticide and by laboratory can be seen in Table 4.1.

Table 4.1 Summary of Results Reported.

Pesticide	No of Reported	No of Not Reported	% of Reported*	% of Not Reported*
Cyromazine	20	24	45	55
Deet	20	24	45	55
Diuron	30	14	68	32
Ethoxyquin	30	14	68	32
Fenpropidin	28	16	64	36
Fenpyroximate	28	16	64	36
Furathiocarb	31	13	70	30
Isofenphos-methyl	37	7	84	16
Isoprocarb	28	16	64	36
Mecarbam	42	2	95	5
Metolachlor	37	7	84	16
Metribuzin	40	4	91	9
Mevinphos	40	4	91	9
Phorate	40	4	91	9
Picolinafen	23	21	52	48
Prometryn	39	5	89	11
Propazine	32	12	73	27
Propoxur	40	4	91	9
Pyraclostrobin	37	7	84	16
Quinoclamine	15	29	34	66
Simazine	40	4	91	9
Carbofuran, Carbofuran-OH	39	5	89	11

\* The % of Laboratories is calculated relative to the total number of laboratories submitting results (44).

#### 4.1.1 Other Reported Pesticides

Many laboratories reported additional pesticides to those spiked into the test material. These pesticides reported are presented in Table 4.2.

Table 4.2. Laboratories that reported other pesticides in the test material.

LABORATORY CODE	REPORTED PESTICIDE
Lab007	Chlorpyrifos
	Ethion
Lab014	Ametryn
	Fenpropimorph
Lab018	Ametryn
	Cymoxanil
	Methiocarb Sulfone
Lab019	Dimethoate
Lab021	Ametryn
	Chlorpyrifos
	Ethion
	Prometon
Lab022	Chlorpyrifos
	Dimethachlor
	Dimethomorph
	Fenpropimorph
	Metamitron
	Methidathion
	Phosdrin
Thiabendazole	
Lab026	Amitraz
	Difenoconazole
	Pymetrozin
	Pyridaben
	Pyridate
Lab030	Terbutylazine
Lab031	Chlorpyrifos
Lab037	Fonicamid
Lab045	Terbutylazine
Lab046	Dinobuton
	Fenuron
	Propanil
Lab047	Iprobenfos
	Mepronil
	Methazole
Lab048	Dimethametryn
	Lutiram
Lab049	Binapacryl

*Atrazine, 3,4-Dichloroaniline* Case: these pesticides were not used to spike the test material although ten laboratories and the organiser also detected them at very low concentrations. In the case of 3,4-Dichloroaniline, its presence is justified because it is a Diuron degradation product, and Diuron was spiked in the test materials. Therefore Atrazine and 3,4 Dichloroaniline

were not assigned as “Other Reported Pesticides”. Table 4.3 shows the laboratories that reported the presence of these pesticides.

Table 4.3 Laboratories that reported Atrazine and 3,4-Dichloroaniline.

LABORATORY CODE	REPORTED PESTICIDE
Lab006	3,4-Dichloroaniline
Lab016	
Lab029	
Lab007	Atrazine
Lab013	
Lab014	
Lab018	
Lab021	
Lab022	
Lab031	
Lab039	
Lab040	
Lab046	

#### 4.1.2 False negatives (Not Reported Pesticides)

Table 4.1 summarizes how many laboratories did not report each pesticide. All results are given in Appendix 1. A graphical representation can be seen in Appendix 2.

#### 4.2 Concentration levels.

Twenty-one pesticides were used to spike the leek test material at different levels, in the range between 50 and 1000 ppb. The carbofuran was also present in the sample but not spiked (>10ppb), as explained on page 5. The aim of this EUPT was focused only on detection capabilities, therefore no quantitative data were requested.

#### 4.3 Assessment of laboratory performance.

No z-score values, or any other statistical calculations, have been performed as no numerical results were reported by the participants. However, a classification has been considered of importance, based on the number of detected results each laboratory reported and also according to the methods that they used.

Table 4.4 Classification of laboratories according to the number of pesticides correctly reported.

Laboratory Code	Reported	Carbofuran	Other Detected Pesticides
Lab 017	21	1	-
Lab 023	21	1	-
Lab 036*	21	1	-
Lab 038	21	1	-
Lab 039*	21	1	-
Lab 030	21	1	1
Lab 031	21	1	1
Lab 007	21	1	2
Lab 046*	21	1	3
Lab 001	20	1	-
Lab 029	20	1	-
Lab 006*	18	1	-
Lab 014	18	1	2
Lab 009*	17	1	-
Lab 016*	17	1	-
Lab 024	17	1	-
Lab 013*	16	1	-
Lab 015*	16	1	-
Lab 041*	16	1	-
Lab 042*	16	1	-
Lab 037*	16	1	1
Lab 040	16	1	1
Lab 008*	15	-	-
Lab 002	14	1	-
Lab 003	14	1	-
Lab 004*	14	1	-
Lab 011	14	1	-
Lab 020	14	1	-
Lab 025	14	1	-
Lab 035	14	1	-
Lab 050*	14	1	-
Lab 045*	14	-	1
Lab 021	14	1	4
Lab 026*	14	-	5
Lab 012	13	1	-
Lab 048	13	1	2
Lab 047*	13	-	3
Lab 005*	12	1	-
Lab 049	11	1	1
Lab 044	9	1	-
Lab 018*	9	-	3
Lab 022*	9	1	8
Lab 019	4	1	1
Lab 033	3	1	-

\* National Reference Laboratories in Fruit and Vegetables participating in this test.

The methods used by the laboratories are detailed in Appendix 3, where the chromatographic techniques, detectors, instrumentation, etc used to analyze the test materials are given. In the table 4.5 there is a summary of the chromatographic techniques used by pesticide, and a graphical representation is shown in Appendix 2.

Table 4.5 Chromatographic techniques used to determine each pesticide in the test material

Pesticide	Total Reported	GC	LC
Cyromazine	20	1	19
Deet	20	15	5
Diuron	30	2	28
Ethoxyquin	30	19	11
Fenpropidin	28	11	17
Fenpyroximate	28	0	28
Furathiocarb	31	8	23
Isofenphos-methyl	37	25	12
Isoprocarb	28	14	14
Mecarbam	42	22	20
Metolachlor	37	21	16
Metribuzin	40	19	21
Mevinphos	40	29	11
Phorate*	40	29	11
Picolinafen	23	10	13
Prometryn	39	22	17
Propazine	32	20	12
Propoxur	40	14	26
Pyraclostrobin	37	8	29
Quinoclamine	15	11	4
Simazine	40	21	19
Carbofuran*	39	9	30

\* with metabolite

In the table 4.6 we can observe that around a 50% of the laboratories that reported results, reported more than the 70% of the present pesticides (including carbofuran) and around 80% of the laboratories more than the 60% of the pesticides, in the Appendix 2 the graphical representation can be seen.

Table 4.6. Number and % of Reported Present Pesticides by Laboratory

Laboratory Code	Number of Reported Present Pesticides	% of Reported Present Pesticides
Lab 007	22	100
Lab 017	22	100
Lab 023	22	100
Lab 030	22	100
Lab 031	22	100
Lab 036*	22	100
Lab 038	22	100
Lab 039*	22	100
Lab 046*	22	100
Lab 001	21	95
Lab 029	21	95
Lab 006*	19	86
Lab 014	19	86
Lab 009*	18	82
Lab 016*	18	82
Lab 024	18	82
Lab 013*	17	77
Lab 015*	17	77
Lab 037*	17	77
Lab 040	17	77
Lab 041*	17	77
Lab 042*	17	77
Lab 008*	15	68
Lab 002	15	68
Lab 003	15	68
Lab 004*	15	68
Lab 011	15	68
Lab 020	15	68
Lab 021	15	68
Lab 025	15	68
Lab 026*	14	64
Lab 035	15	68
Lab 045*	14	64
Lab 050*	15	68
Lab 012	14	64
Lab 047*	13	59
Lab 048	14	64
Lab 005*	13	59
Lab 049	12	55
Lab 022*	10	45
Lab 044	10	45
Lab 018*	9	41
Lab 019	5	23
Lab 033	4	18

\* National Reference Laboratories in Fruit and Vegetables participating in this test.



## 5. CONCLUSIONS

Fifty laboratories applied to participate in this test and forty-four laboratories submitted results. Twenty-two participating laboratories were National Reference Laboratories for Fruits and Vegetables (marked with an asterisk on the graphs and tables). Nineteen Member States, 2 EFTA countries (Norway and Switzerland) and two non EU or EFTA countries (Egypt, and Turkey) participated in this European Union Proficiency Test.

Most laboratories analysed the test material using methods based on both gas and liquid chromatography, combined with mass spectrometric detection. In the case of GC-MS analysis, full scan acquisition, with associated target-library software (covering a large number of pesticides), was used by the majority of the laboratories. In contrast where LC-MS analysis was used, targeted acquisition methods using triple quadrupole instruments was favoured. Most of the laboratories indicated that they could cover a scope of greater than 200 pesticides.

Nine of the laboratories were able to detect all 21 pesticides spiked into the leek test material and the carbofuran (furathiocarb degradation product). Only 5 laboratories failed to detect 50% of the present pesticides. Around 50% of the laboratories that reported results were able to find more than the 70% of the pesticides including carbofuran (more than 16 pesticides), and approximately an 80% of them found more than the 60% of the present pesticides (more than 14 pesticides).

This year the evaluation of "False Negatives" has been changed to "Not Reported Pesticides" due to the absence of a Pesticide Target List, but laboratories should check their 'Not Reported Pesticides' if they are included in their scope.

Sixteen participants reported pesticides that were not spiked into the leek test material. Whether or not this should be judged as poor performance depends on how the participant would act on these results in routine analysis. If the detected pesticide would be reported as positive without any further confirmation of identity, then the result would be a false positive and hence erroneous monitoring data would be reported. If the detected pesticide is regarded as only 'suspect' or 'indicatively present' and followed up by additional analysis to confirm identity before reporting the result, then the pesticide indicated as "other reported pesticide" in this report is not really an issue.

This second interlaboratory test on wide-scope screening methods showed that such an approach can substantially expand the scope of pesticide residue analysis. This is especially useful for pesticides not frequently found in food and, therefore, not normally included in current quantitative methods. The use of screening methods can greatly increase the chance of detecting less commonly found pesticides. However, the test also revealed that improvements in scope (both in number and the choice of pesticides included) and verification of the performance of screening methods (i.e. validation) are necessary to improve reliability of such methods.

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

The Organiser and the Scientific Committee considers that screening methods have added value in addition to the quantitative multiresidue methods currently routinely used for monitoring purposes. The results of this second test are very encouraging, but also indicate the need for continued evaluation of screening methods. Therefore, further proficiency tests will be organised to provide support to those laboratories using screening methods in order to extend their use and improve their reliability. These methods will be used more and more as screens/filters, to make routine laboratory work easier and faster. The need for validation of screening methods has been recognised and guidelines for such validation have been prepared and included in the update of the SANCO document for "Method validation and quality control procedures for pesticide residue analysis in food and feed" (SANCO/10684/2009).

For next year, a mandarin matrix test material will be used. If there is an interest from laboratories for specific matrices, they should inform the EURL-FV and their suggestions will be evaluated. The timing of delivery of the test material will be January, and 72 hours will be allowed for submission of results (given that this should be time enough to undertake screening methods). There will be no target list, the same as for this test.

Furthermore, next year the coordinated multiannual control programme of the Union (Commission Regulation (EU) No 915/2010) will allow Member State laboratories using multi-residue methods to conduct qualitative screening methods on up to 15 % of the samples to be taken.

## 7. REFERENCES

1. Mezcua M., Martinez-Uroz M. A., Wylie P. L. and Fernandez-Alba A.R. Simultaneous screening and target analytical approach by GC-q-MS for pesticide residues in fruits and vegetables. *Journal of AOAC Int.*, 2009, 92 (6).
2. Mezcua M., Malato O., Garcia-Reyes J. F., Molina-Diaz A., and Fernandez-Alba A. R. Accurate-Mass Databases for Comprehensive Screening of Pesticide Residues in Food by Fast Liquid Chromatography Time-of-Flight Mass Spectrometry. *Anal. Chem.* 2009, 81, 913-929
3. Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed, European Commission, Document No. SANCO/10684/2009.
4. ISO/IEC 17043:2010 Conformity assessment — General requirements for proficiency testing.

## **8. ACKNOWLEDGEMENTS**

The Organiser is grateful to the European Commission for funding this 2<sup>nd</sup> European Proficiency Test in Fruit and Vegetables for Screening Methods.

The Organiser wishes to thank the members of the Scientific Committee for their invaluable and knowledgeable advice.

The Organiser wishes to give a special thank-you to Almeria University for the use of their facilities.

APPENDIX 1. Results

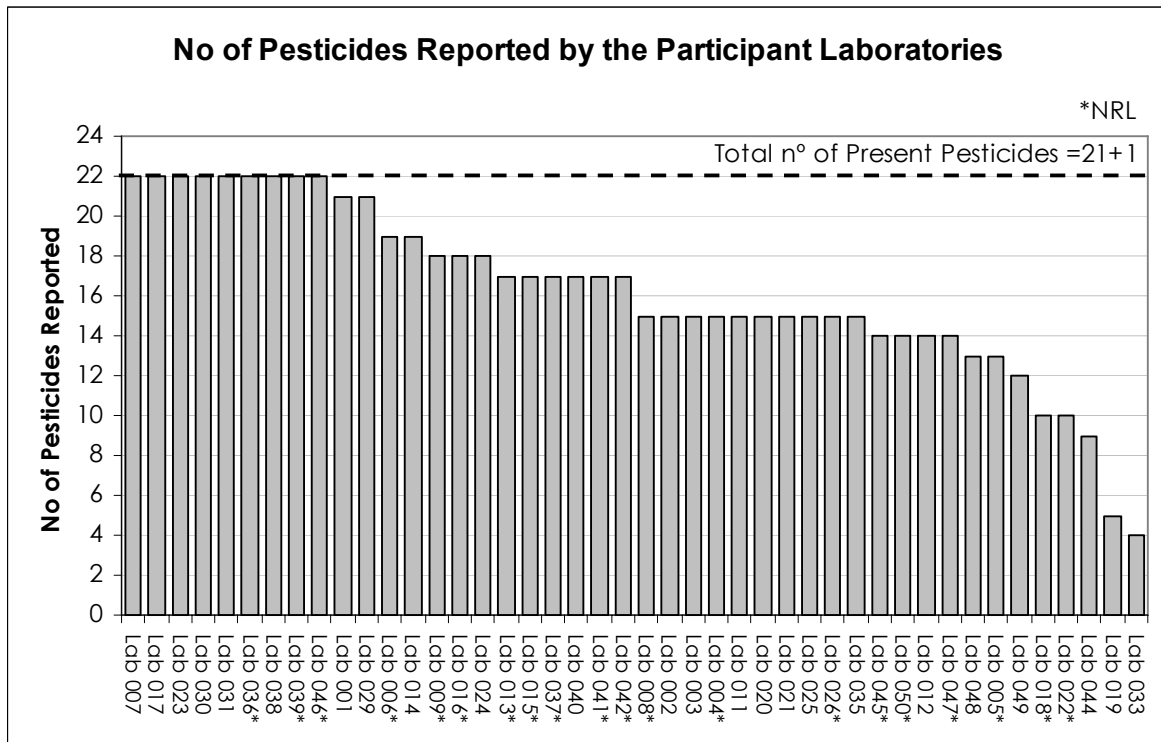
Laboratory Code Total Number of Reporting Laboratories = 44	Added Pesticides Total Number of Added Pesticides = 21																				Total Reported	Total of Not Reported	% Reported	% of Not Reported	Carbofuran-Carbofuran-OH Present at >10µg/Kg but Not Added	
	Cyromazine	Deet	Diuron	Ethoxyquin	Fenpropridin	Fenpyroximate	Furthiocarb	Isofenphos-methyl	Isoprocarb	Mecarbam	Metolachlor	Metribuzin	Mevinphos	Phorate,	Picolinafen	Promethyn	Propazine	Propoxur	Pyraclostrobin	Quinoclamine						Simazine
001		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	20	1	95	5	R
002			R	R			R	R		R	R	R	R	R		R	R	R	R	R	R	14	7	67	33	R
003	R		R		R	R	R		R		R			R	R	R	R	R	R	R	R	14	7	67	33	R
004*			R		R	R	R	R		R	R	R	R	R		R		R	R	R	R	14	7	67	33	R
005*					R	R		R	R	R		R	R	R		R		R	R	R	R	12	9	57	43	R
006*			R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	3	86	14	R
007	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	21	0	100	0	R
008*		R	R			R	R	R	R	R	R	R	R	R		R		R	R	R	R	15	6	71	29	
009*	R	R	R		R	R	R	R	R	R	R	R	R	R		R		R	R	R	R	17	4	81	19	R
011		R		R			R		R	R	R	R	R	R		R	R	R		R	R	14	7	67	33	R
012	R		R	R			R	R		R		R	R	R		R		R	R	R	R	13	8	62	38	R
013*	R		R	R			R	R	R	R	R	R	R	R		R	R	R	R	R	R	16	5	76	24	R
014	R		R	R	R	R	R	R	R	R	R	R		R	R	R		R	R	R	R	18	3	86	14	R
015*		R	R	R		R		R	R	R	R	R	R	R		R	R	R	R	R	R	16	5	76	24	R
016*	R	R	R	R		R			R	R	R	R	R	R		R	R	R	R	R	R	17	4	81	19	R
017	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	21	0	100	0	R
018*	R					R	R	R					R	R		R			R		R	9	12	43	57	
019								R		R			R					R				4	17	19	81	R
020		R	R	R				R	R	R	R	R	R	R		R	R	R			R	14	7	67	33	R
021			R	R	R	R		R		R	R	R	R	R		R		R	R	R	R	14	7	67	33	R
022*			R				R	R		R	R	R	R					R			R	9	12	43	57	R
023	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	21	0	100	0	R
024			R	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	17	4	81	19	R
025		R		R	R				R	R	R	R	R	R		R	R	R		R	R	14	7	67	33	R
026*	R			R	R					R	R	R	R	R	R	R	R	R	R	R	R	14	7	67	33	
029		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	20	1	95	5	R
030	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	21	0	100	0	R
031	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	21	0	100	0	R
033								R		R			R									3	18	14	86	R
035		R		R	R			R		R	R	R	R	R		R	R	R	R	R	R	14	7	67	33	R
036*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	21	0	100	0	R
037*					R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	5	76	24	R
038	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	21	0	100	0	R
039*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	21	0	100	0	R
040			R	R	R	R	R		R	R	R	R		R	R	R	R	R	R	R	R	16	5	76	24	R
041*	R		R	R	R	R	R	R		R	R	R		R		R	R	R	R	R	R	16	5	76	24	R
042*			R	R			R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	5	76	24	R
044				R			R	R		R	R	R	R	R				R				9	12	43	57	R
045*		R	R			R		R	R	R	R	R	R		R	R	R	R			R	14	7	67	33	
046*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	21	0	100	0	R
047*					R		R		R	R		R	R	R	R	R	R	R	R	R	R	13	8	62	38	
048	R			R		R		R		R	R	R	R	R		R	R	R	R	R	R	13	8	62	38	R
049	R				R			R		R		R	R	R				R	R		R	11	10	52	48	R
050*					R		R	R		R	R	R	R	R	R	R	R	R	R	R	R	14	7	67	33	R
Total Reported	20	20	30	30	28	28	31	37	28	42	37	40	40	40	23	39	32	40	37	15	40					39
Total Not Reported	24	24	14	14	16	16	13	7	16	2	7	4	4	4	21	5	12	4	7	29	4					5
% Reported	45	45	68	68	64	64	70	84	64	95	84	91	91	91	52	89	73	91	84	34	91					89
% Not Reported	55	55	32	32	36	36	30	16	36	5	16	9	9	9	48	11	27	9	16	66	9					11

R: Reported Pesticide

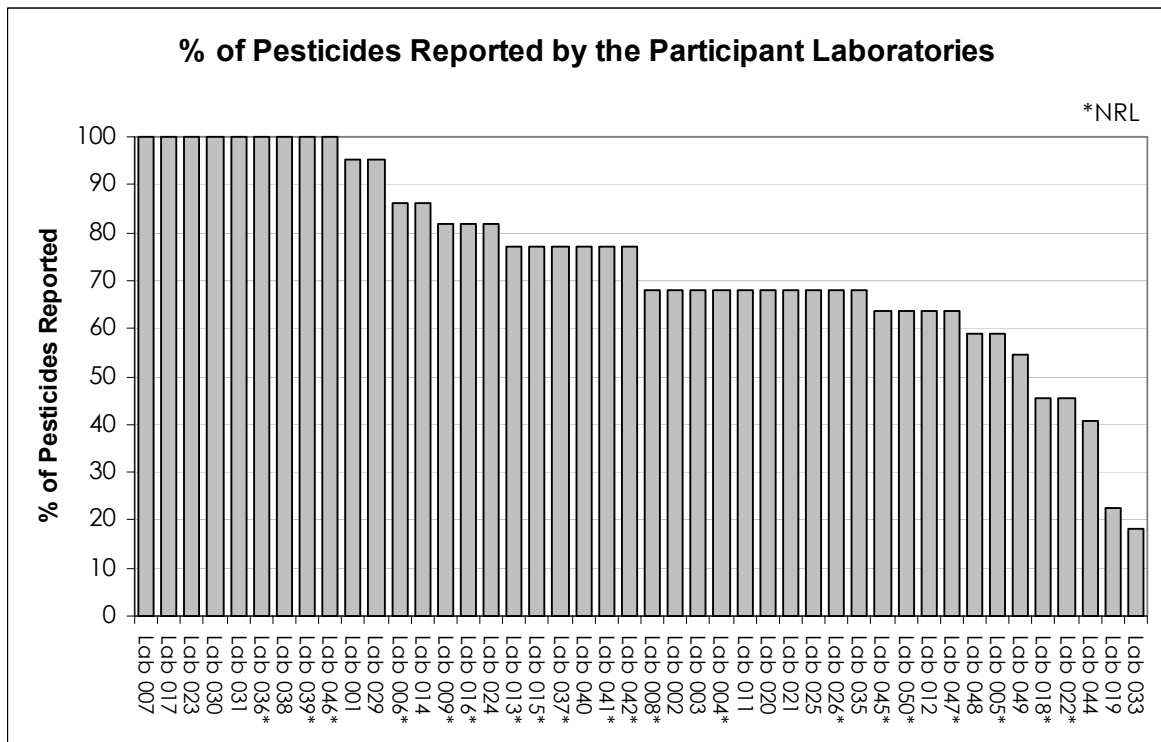
\* NRL



APPENDIX 2. Graphical Representations

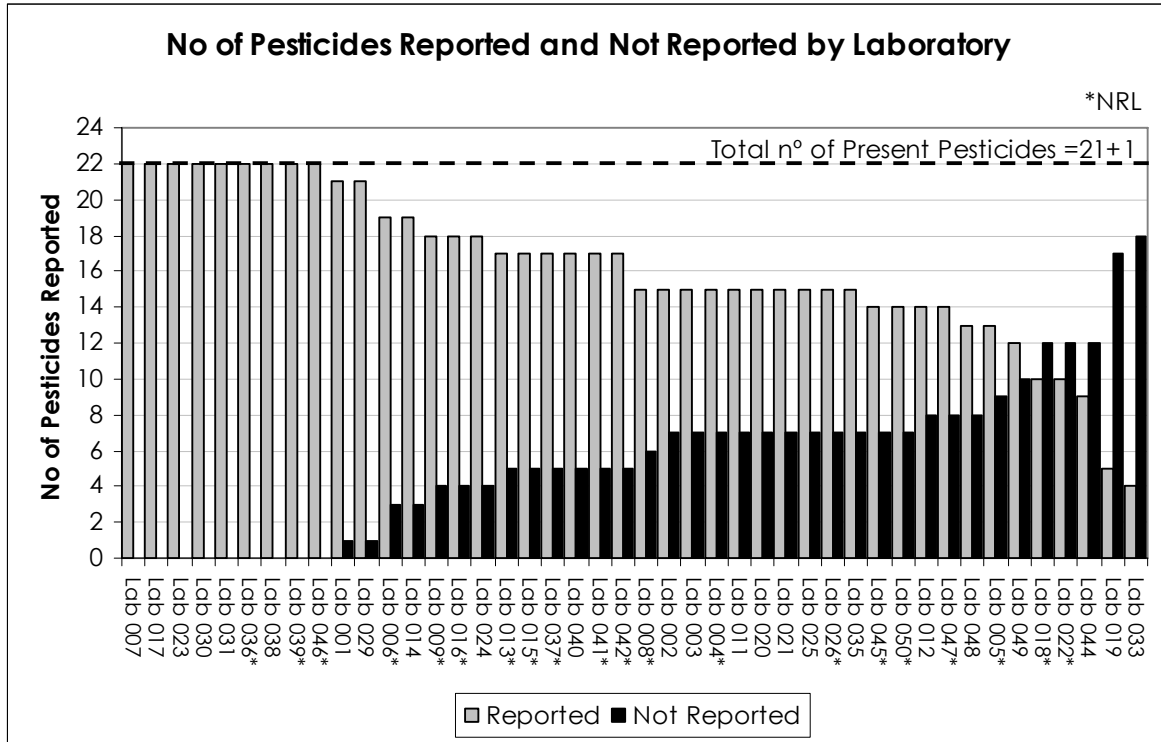


21+1(Carbofuran) pesticides

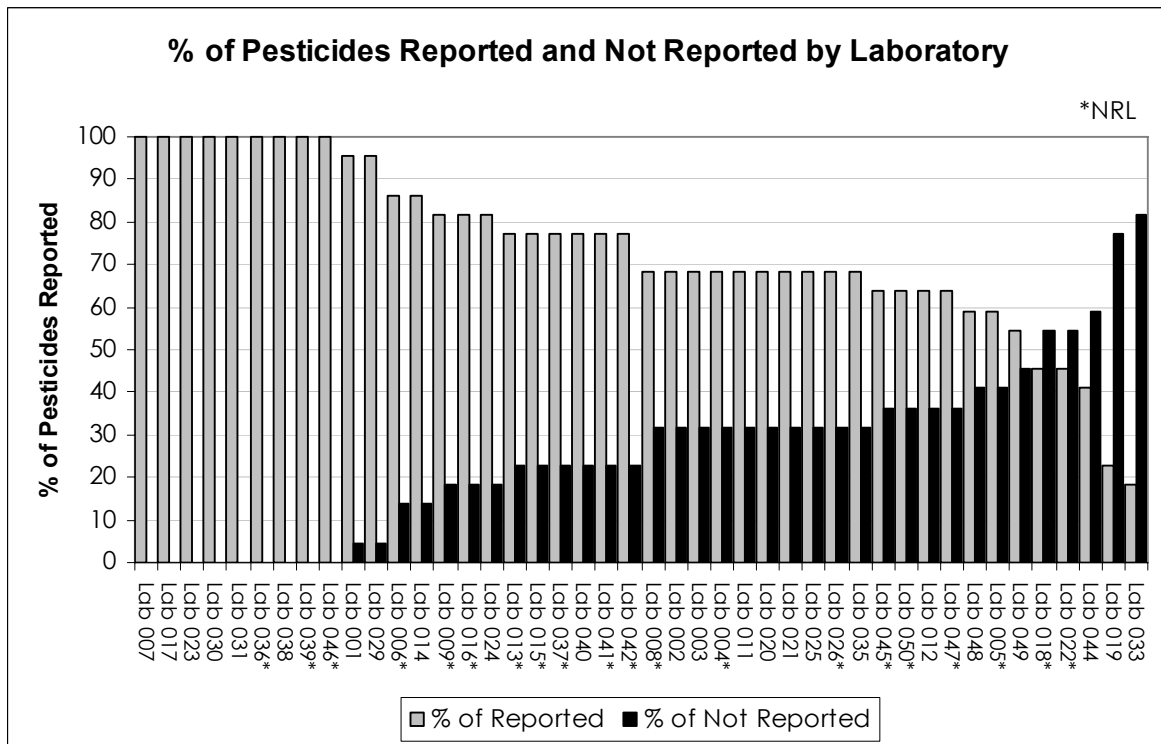


21+1(Carbofuran) pesticides

APPENDIX 2. Graphical Representations



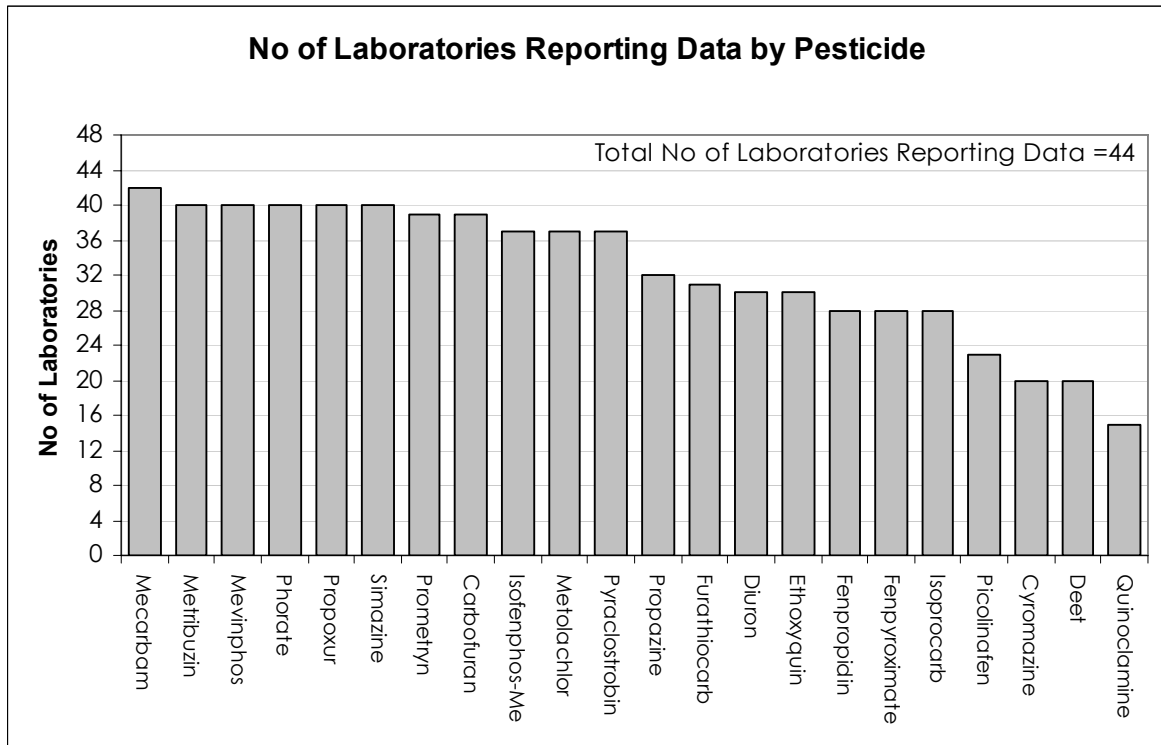
21+1(Carbofuran) pesticides



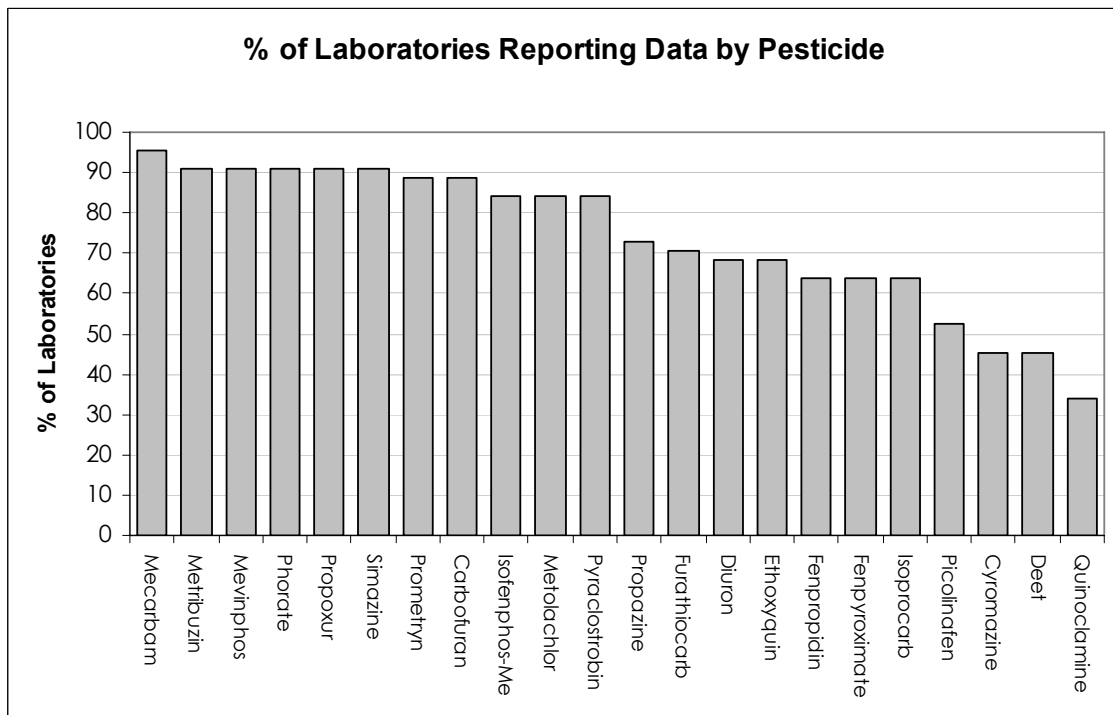
21+1(Carbofuran) pesticides



APPENDIX 2. Graphical Representations

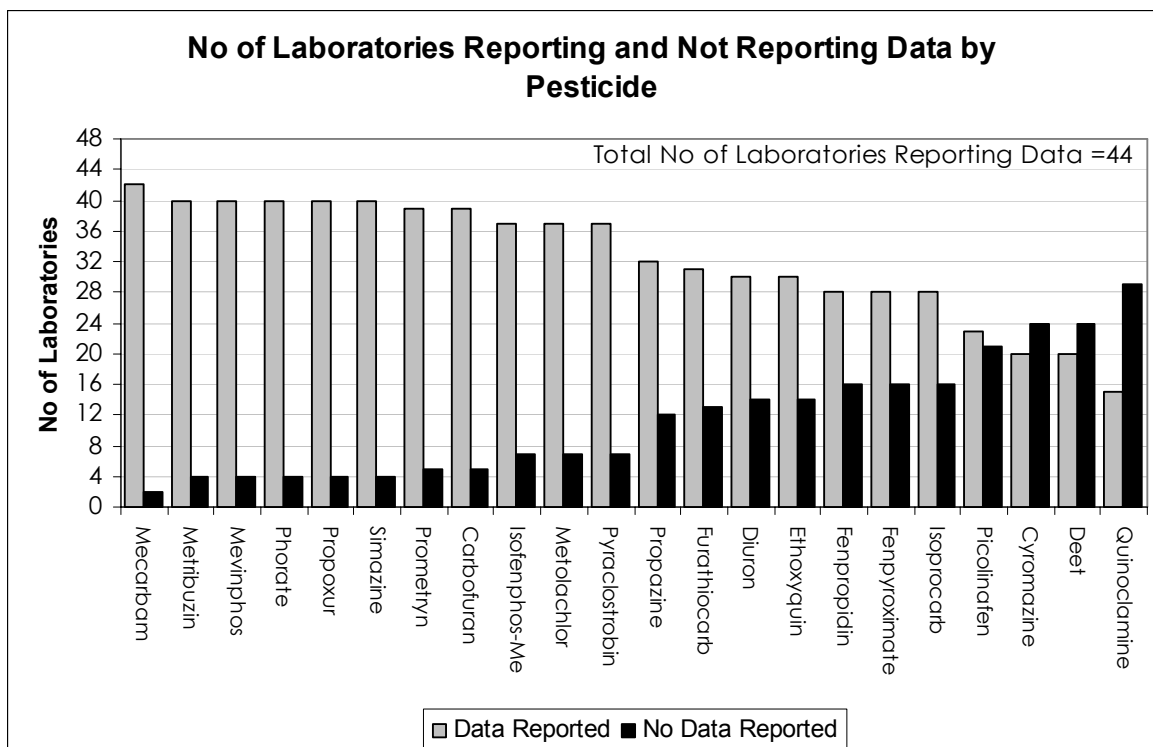


21+1(Carbofuran) pesticides

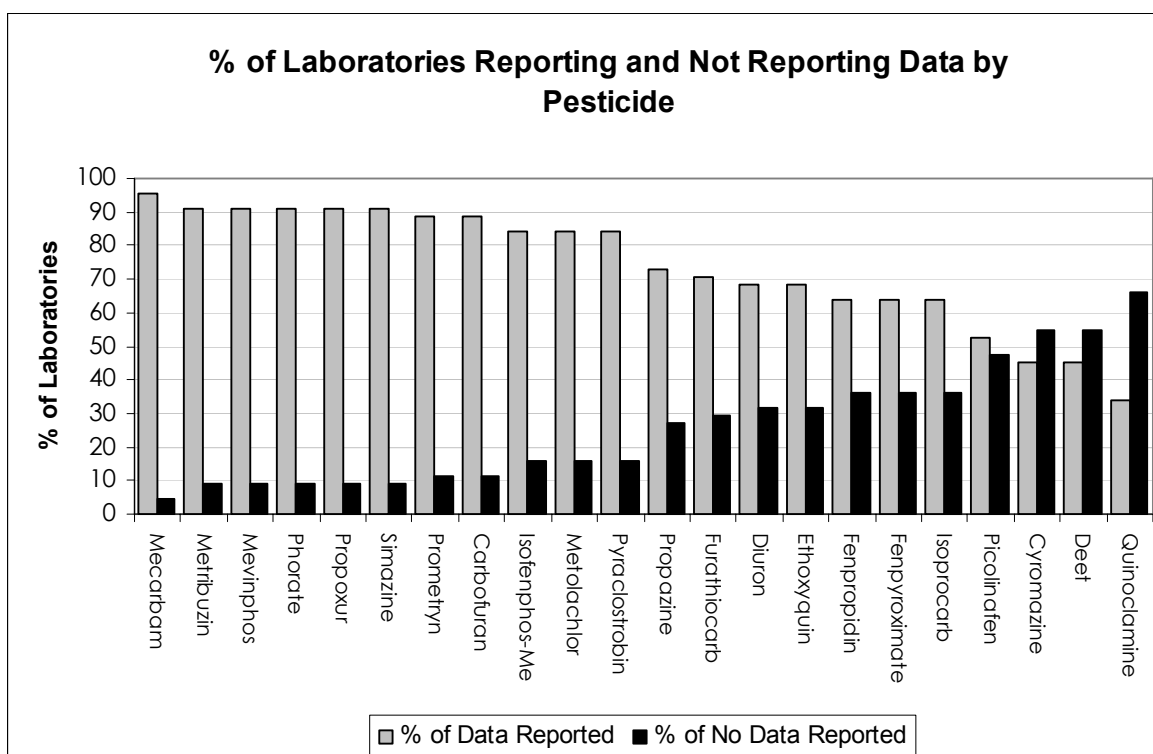


21+1(Carbofuran) pesticides

APPENDIX 2. Graphical Representations



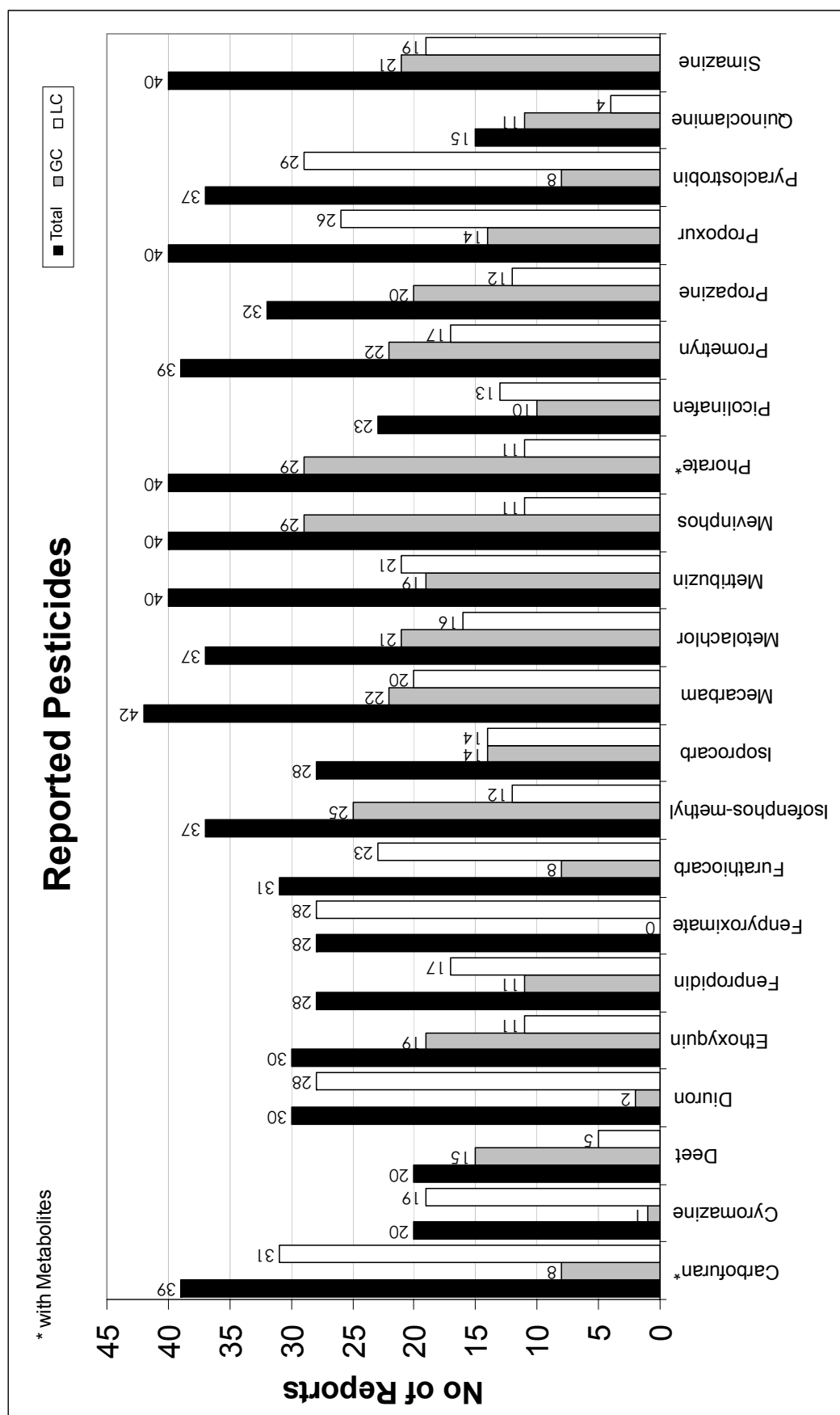
21+1(Carbofuran) pesticides



21+1(Carbofuran) pesticides

APPENDIX 2. Graphical Representations

Chromatographic Techniques used to determine each pesticide in the test material



21+1(Carbofuran) pesticides



**APPENDIX 3. Methods used by participants for detecting pesticides.**

Laboratory Methods												
Laboratory Code	Chromatographic technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software	Number of compounds in the screening method	Standard Solution Frequency
001	GC	MS		AT 5975	10	ACN	SPE	HP-5MSi	10	Automatic	750	Always
	LC	MS	QqQ	API 4000 Trap	10	ACN	No	C18 Synergi fusion	10	Manual	98	Always
002	GC	MS	IT	Varian 4000	10	ACN	PSA	5%	10	Automatic	200	Each Batch
	LC	MS	QqQ	3200 Qtrap	10	ACN	PSA	C18 2,5 µm - 5 cm	50	Automatic	85	Each Batch
003	LC	MS	Q-TOF	Waters Xevo QToF	10	Ethyl Acetate	No	Acquity BEH C18	3	Both	500	Always
004	GC	MS	QqQ	Agilent 7000A	15	Acetone/PE/DCM	No	HP5MS	2	Both	200	Always
	LC	MS	QqQ	API 4000	15	ACN	PSA	SpeedRod	20	Manual	153	Always
005	GC	MS	Q	Agilent 5973N	10	ACN	DSPE	HP5-MS(I)	20	Both	118	Each Batch
	LC	MS	QqQ	Agilent 6410B	10	ACN	DSPE	C18, 1.8µm	2	Automatic	180	Each Batch
006	GC	MS	IT	Varian 2000	15	Acetone/DCM/PE	No	VF-5ms	5	Automatic	500	Each Batch/ Quarterly
	LC	MS	QqQ	Waters Premier XE	15	Acetone/DCM/PE	No	Acquity BEH	5	Automatic	188	Each Batch/ Quarterly
007	GC	MS	TOF	Pegasus IV	10	ACN	Dispersive SPE with PSA	HP-5MS	5	Both	650	Weekly
	LC	MS	QqQ	Agilent 6410	10	ACN	Dispersive SPE with PSA	Luna C18	5	Both	200	Daily
008	GC	MS	QqQ	Varian 1200L, GC3800	10	Ethyl Acetate	No	FactorFour, VF-5ms	2	Automatic	160	Each Batch
	LC	MS	QqQ	API 4000 Qtrap	10	Ethyl Acetate	No	C18	5	Automatic	270	Each Batch
009	GC	MS	QqQ	Waters Quattro Micro GC	10	Ethyl Acetate	Filter	Fused silica capillary column	10	Both	136	Always
	LC	MS	QqQ	Waters Acquity UPLC system/ API 5000 Applied Biosystems	10	Ethyl Acetate	Filter	C18	2	Both	211	Always
011	GC	MS	Q	Agilent GC 7890 MS 5975	10	ACN	Dispersive SPE	HP-5MS	1	Both	150 method 500 library	At the method's setup

**APPENDIX 3. Methods used by participants for detecting pesticides.**

Laboratory Methods												
Laboratory Code	Chromatographic technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software	Number of compounds in the screening method	Standard Solution Frequency
012	GC	MS	Q	AGILENT GC-MSD INERT 5975C	10	ACN	PSA	HP5MS	2	Manual	215	Always
	LC	MS	QqQ	AGILENT LC-MSMS 6410	10	ACN	PSA	C18	20	Manual	106	Always
	GC	MS	Q		15	Luke	GPC	HP 5	2	Both		
013	LC	MS	QqQ		10	QuEChERS		C8	20	Both		
	LC	MS	QqQ	API4000	10	ACN	DSPE	Waters-C18 2.1x50mmx1.7µm	5	Both	600	Every Week
015	GC	MS	Q	Varian 3800 GC+320-MS	10	ACN	Dispersive SPE with PSA + GCB	VF 5ms	3	Both	150	Each Batch
	GC	FPD	no	Varian 3800 GC	10	ACN	Dispersive SPE with PSA + GCB	Rtx OPP	1	Both	45	Each Batch
	GC	MS	QqQ	Varian 3800 GC+320-MS	10	ACN	Dispersive SPE with PSA + GCB	VF 5ms	3	Both	150	Each Batch
	LC	MS	QqQ	Varian Pro Star +320-MS	10	ACN	Dispersive SPE with PSA + GCB	ChromSep C-18	20	Both	140	Each Batch
016	GC	MS	Q	Thermo DSQ	10	ACN	DSPE/SPA/C18	Varian VF-5ms	1	Both	150	Twice a Year
	LC	MS	QqQ	API 4000	10	ACN	DSPE/SPA/C18	Supelco Ascentis Express RP-AMIDE	10	Both	190	Twice a Month
017	GC	MS	Q	Agilent 6890N/5973N	10	ACN	QuEChERS	HP-5	1	Automatic	927	Each Batch
	LC	MS	QqQ	Agilent 6410 Triple Quad	10	ACN	QuEChERS	kinetex C-18	2	Manual		Each Batch
018	GC	NPD	TOF	Agilent	15	Acetone +DCM +PE	No	DB-5 & DB-17	1	Manual	250	Always
	LC	MS	QqQ	Agilent	10	ACN	DSPE	C-18	10	Automatic	250	Always
019	GC	MS	IT	Varian 2000 Ion Trap	15	Ethylacetate		DB.5 MS	10	Automatic	67	
020	GC	MS	IT	Varian 4000	10	ACN	DSPE	RTX-200	2	Automatic	400	Daily Check
021	LC	MS	QqQ	Quattro Premier XE	10	ACN	No	C18	5	Both	311	Always
022	LC	MS	QqQ	API4000	10	ACN	PSA	C18ec	8	Manual	250	

**APPENDIX 3. Methods used by participants for detecting pesticides.**

Laboratory Methods												
Laboratory Code	Chromatographic technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software	Number of compounds in the screening method	Standard Solution Frequency
023	GC	MS	Q	GC Agilent 6890N, MS Agilent 5976	5	ACN	DSPE	HP-5MS	3	Both	method 441 library 590	Never
	LC	MS	QqQ	HPLC Agilent 1100, MS API 3000	5	ACN	DSPE	C18 3µm 50x2mm	10	Both	method 441 library 590	Always
024	GC	MS	QqQ	TSQ Quantum GC	10	Acetone /PE/DCM	Na <sub>2</sub> SO <sub>4</sub>	RXi 5SiI-MS	1	Automatic	164	Every 10 Samples
	GC	MS	Q	Varian Saturn 2000	10	Acetone /PE/DCM	Na <sub>2</sub> SO <sub>4</sub>	DB 5MS UI	25	Automatic	344	Every 10 Samples
025	LC	MS	QqQ	Waters Xevo	10	Acetone/PE/DCM	Na <sub>2</sub> SO <sub>4</sub>	C18	2	Automatic	191	Every 10 Samples
	GC	MS	Q	Agilent 5973	10	ACN	SPE	HP 5 MS	2	Automatic	923	If Necessary
026	GC	MS	Q	Waters MicroGCMSMS	15	ACN	PSA	DB5	4	Manual	Varies	Sometimes
	GC	MS	QqQ	Waters MicroGCMSMS	15	ACN	PSA	DB5	4	Manual	Varies	Sometimes
029	LC	MS	Q-TOF	Brucker	15	ACN	PSA	C18	5	Manual	Varies	Sometimes
	LC	MS	QqQ	QuatroUltima, Waters	15	ACN	PSA	C18	10	Manual	Varies	Sometimes
030	GC	MS	TOF	LECO Pegasus IV	10	ACN	PSA	DB 5	3	Both	700	Each Batch
	LC	MS	QqQ	API 4000	10	ACN	PSA	Pursuit XRS Ultra	6	Both	200	Each Batch
030	GC	MS	Q	Agilent singleQ	10	ACN	PSA	HP-MS 5	20	Both	927	Everyday
	GC	MS	QqQ	Agilent 7000	10	ACN	PSA	HP-MS 5	1	Both	100	Everyday
031	LC	MS	QqQ	API 3200 Qtrap	10	ACN	PSA	Atlantis	10	Both	180	Everyday
	GC	MS	TOF	Leco Pegasus 4D	10	ACN	PSA	RTX-CL pest	10	Both	800	Each Batch 240 Pesticides
033	LC	MS	Orbitrap	Exactive	10	ACN		Atlantis	5	Both	475	Each Batch 120 Pesticides
	LC	MS	QqQ	Applied Biosystem	10	ACN	PSA/MgSO <sub>4</sub> /GCB	Atlantis T3 100*2,1mm	5	Manual	68	Each Batch
033	GC	MS	IT	Varain GC/MS 4000	10	ACN	PSA/MgSO <sub>4</sub> /GCB	VF-5ms 30m*0,25mm	5	Manual	93	Each Batch

**APPENDIX 3. Methods used by participants for detecting pesticides.**

Laboratory Methods												
Laboratory Code	Chromatographic technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software	Number of compounds in the screening method	Standard Solution Frequency
035	GC	MS	IT	Thermo POLARISQ	10	ACN	No	DB-5MS	8	Both	500	Each Batch
	LC	MS	QqQ	Thermo TSQ Quantum	10	ACN	No	XTerra	20	Both	120	Each Batch
036	GC	MS	Q	Agilent MSD 5973	30	Ethyl Acetate	HPGPC	DB5-MS	2	Automatic	500	With Each Run
	LC	MS	TOF	Agilent	10	ACN	No	Zorbax Eclipse plus	3	Both	500	With Each Run
037	LC	MS	MS/MS	Waters Permier XE	10	ACN	No	Acquity UPLC HSS T3	20	Both	200	With Each Run
	LC	MS/MS	QqQ	API 4000QT	10	ACN	DSP	C18	25	Automatic	400	Each Batch
038	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both	> 200	Always
039	GC	MS	Q	HP5975C	10	ACN	PSA	HP5MS	5	Both	1000	Always
	LC	MS	QqQ	ABI 4000	10	ACN	No	C18	55	Both	577	Always
040	GC	MS	Q	Agilent 7890A/5975C	15	ACN	PSA	HP5MS	10	Both	117	Once a Day
	LC	MS	QqQ	Waters Aquity TQD	15	ACN	PSA	UPLC-BEH C18	10	Both	123	Once a Day
041	GC	MS	IT	UltraTrace GC-Polaris Q (Thermo)	25	Hexane	LLE	ZB-5MS	1	Automatic	419	Each Time
042	LC	MS	QqQ	UPLC-Xevo (Waters)	20	Ethyl Acetate	LLE	HSS T3	10	Automatic	327	Each Time
	GC	MS	IT	Varian Saturn 4000	10	ACN	DSPE	Varian FV5 ms	1	Both	250	4/Year
044	LC	MS	QqQ	API 3200 QT	10	ACN	DSPE	Synergi Fusion RP 10	20	Both	100	1/Week
	GC	MS	Q	Agilent 5973	50	Acetone	No	HP 5 MS	1	Automatic	215	Each Batch
045	LC	MS	QqQ	Varian MS 320	10	ACN	Yes	C 18	10	Automatic	66	Each Batch
	LC	MS	TOF	LCT PREMIER XE	15	Acetone/PE/DCM	No	C18	5	Both	345	Every Day
046	GC	MS	Q	Agilent MSD 5973	10	ACN	DSPE	HP5 MS	5	Both	500	Every Day
	GC	MS	QqQ	Waters Quattro Micro	10	ACN	DSPE	HP5 MS	3	Both	110	Every Day
047	LC	MS	QqQ	Waters Quattro Premier	10	ACN	DSPE	C18	5	Both	180	Every Day
	GC	MS	TOF	Pegasus III	50	Ethyl Acetate	GPC	VF-5ms(30mx0,25x0,25)	2	Automatic	400-450	Every Day



**APPENDIX 3. Methods used by participants for detecting pesticides.**

Laboratory Methods												
Laboratory Code	Chromatographic technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software	Number of compounds in the screening method	Standard Solution Frequency
048	GC	MS	Q	Interscience DSQ	15	ACN	QuEChERS	DB5-MS 20m 0.18mm 0.18µm	0.8	Automatic	500	Each Time
	LC	LC/MSMS	QqQ	Micromass Quattro premier	15	ACN	QuEChERS	UPLC BEH C18 1.7µ 2.1mm 100mm	3	Automatic	500	Each Time
	GC	MS	QqQ	Thermo TSQ	10	ACN	PSA	Capillary	1	Both	150	Always
049	GC	MS	IT	Thermo Polaris Q	10	ACN	PSA	Capillary	1	Both	150	Always
	LC	MS	QqQ	Waters Aquiti UPLC T	10	Methanol	Filter	C18	1	Automatic	150	Always
050	GC	MS	Q	Agilent 6890/5973	10	ACN	no	HP-5MS	1	Manual	177	Once a Month
	LC	MS	QqQ	Agilent 6400	10	ACN	no	Eclipse XDB-C18	4	Manual	123	Once a Month

**APPENDIX 3. Methods used by participants for detecting pesticides.**

CYROMAZINE												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
003	1	<5	LC	MS	Q-TOF	Waters Xevo QToF	10	Ethyl Acetate	None	Acquity BEH C18	3	Both
007	2.3	0	LC	MS	QqQ	Agilent 6410	10	ACN	Dispersive SPE with PSA	Luna C18	5	Both
009	na	na	LC	MS	QqQ	Waters Acquity UPLC system/API 5000 Applied Biosystems	10	Ethyl Acetate	Filter	C18	2	Both
012	8.5	86.42%	LC	MS	QqQ	AGILENT LC-MSMS 6410	10	ACN	PSA	C18	20	Manual
013			LC	MS	QqQ		10	QuEChERS		C8	20	Manual
014	None	None	LC	MS	QqQ	API4000	10	ACN	DSPE	Waters-C18 2.1x50mmx1.7µm	5	Both
016			LC	MS	QqQ	API 4000	10	ACN	DSPE/SFA/C18	RP-AMIDE	10	Both
017			LC	MS	QqQ	Agilent 6410 Triple Quad	10	ACN	QuEChERS	kinetex C-18	2	Manual
018	0.5		LC	MS	QqQ	Agilent	10	ACN	DSPE	C-18	10	Automatic
023	5.1	0.23%	LC	MS	QqQ	HPLC Agilent 1100. MS API 3000	5	ACN	DSPE	C18 3µ 50x2mm	10	Both
026		13.5	LC	MS	Q-TOF	Brucker	15	ACN	PSA	C18	5	Manual
030	5	1	LC	MS	QqQ	API 3200 Qtrap	10	ACN	PSA	Atlantis	10	Both
031	<20	994 (match)	GC	MS	TOF	Leco Pegasus 4D	10	ACN	PSA	RTX-CL_pest	10	Both
036	2.5% max	None	LC	MS	MS/MS	Waters Permier XE	10	ACN		Acquity UPLC HSS T3	20	Both
038	0.5	5 ppm	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both
039			LC	MS	QqQ	ABI 4000	10	ACN	No	C18	55	Both
041	0	0	LC	MS	QqQ	UPLC-Xevo (Waters)	20	Ethyl Acetate	LLE	HSS T3	10	Automatic
046			LC	MS	QqQ	Waters Quattro Premier	10	ACN	DSPE	C18	5	Both
048	10	5	LC	LC/MSMS	QqQ	Micromass Quattro premier	15	ACN	QuEChERS	UPLC BEH C18 1.7µ 2.1mm 100mm	3	Automatic
049	0.02		LC	MS	QqQ	Waters Acquity UPLC T	10	Methanol	Filter	C18	1	Automatic

**APPENDIX 3. Methods used by participants for detecting pesticides.**

DEET												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
001	0.2		GC	MS		AT5975	10	ACN	SPE	HP-5MSi	10	Automatic
007	2	6.0	GC	MS	TOF	Pegasus IV	10	ACN	Dispersive SPE with PSA	HP-5MS	5	Both
008	NA	NA	GC	MS	QqQ	Varian 1200L GC3800	10	Ethyl Acetate	None	FactorFour VF-5ms	2	Automatic
009	na	na	GC	MS	QqQ	Waters Quattro Micro GC	10	Ethyl Acetate	Filter	Fused silica capillary column	10	Both
011	+/- 9	30%	GC	MS	Q	Agilent GC 7890 MS 5975	10	ACN	Dispersive SPE	HP-5MS	1	Both
015	1.0 s	16%	GC	MS	Q	Varian 3800 GC+320-MS	10	ACN	Dispersive SPE with PSA + GCB	VF 5ms	3	Both
016	8	38%	GC	MS	Q	Thermo DSQ	10	ACN	DSPE/SPA/C18	Varian VF-5ms	1	Both
017			GC	MS	Q	Agilent 6890N/5973N	10	ACN	QuEChERS	HP-5	1	Automatic
020	n/a	n/a	GC	MS	IT	Varian 4000	10	ACN	DSPE	RTX-200	2	Automatic
023	0.3	-3.16%	LC	MS	QqQ	HPLC Agilent 1100. MS API 3000	5	ACN	DSPE	C18 3µ 50x2mm	10	Both
025	2.2	1 unit	GC	MS	Q	Agilent 5973	10	ACN	SPE	HP 5 MS	2	Automatic
029	567	99	GC	MS	TOF	LECO Pegasus IV	10	ACN	PSA	DB 5	3	Both
030	5	1	LC	MS	QqQ	API 3200 Qtrap	10	ACN	PSA	Atlantis	10	Both
031	-2.1	0.7	LC	MS	Orbitrap	Exactive	10	ACN		Atlantis	5	Both
035	0.1	0.1	GC	MS	IT	Thermo POLARISQ	10	ACN	No	DB-5MS	8	Both
036	5% max	none	GC	MS	Q	Agilent MSD 5973	30	Ethyl Acetate	HGPC	DB5-MS	2	Automatic
038	0.5	5	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both
039	0.5		GC	MS	Q	HP5975C	10	ACN	PSA	HP5MS	5	Both
045	5	0.05 Da	LC	MS	TOF	LCT PREMIER XE	15	Acetone:PE:DCM	No	C:18	5	Both
046			GC	MS	Q	Agilent MSD 5973	10	ACN	DSPE	HP5 MS	5	Both

**APPENDIX 3. Methods used by participants for detecting pesticides.**

DIURON												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
001			LC	MS	QqQ	API4000Trap	10	ACN	No	C18 Synergi fusion	10	Manual
002	< 2.5%	< 2.5 %	LC	MS	QqQ	3200 Qtrap	10	ACN	PSA	C18 2.5 µm - 5 cm	50	Automatic
003	1	<5	LC	MS	Q-TOF	Waters Xevo QToF	10	Ethyl Acetate	None	Acquity BEH C18	3	Both
004	< 2%	<5%	LC	MS	QqQ	API4000	15	ACN	PSA	SpeedRod	20	Manual
006	2.9	9.2	LC	MS	QqQ	Waters Premier	15	Acetone/DCM/PE	No	Acquity BEH	5	Automatic
007	0.4	0	LC	MS	QqQ	Agilent 6410	10	ACN	Dispersive SPE with PSA	Luna C18	5	Both
008	NA	NA	LC	MS	QqQ	API 4000 Qtrap	10	Ethyl Acetate	None	C18	5	Automatic
009	na	na	LC	MS	QqQ	Waters Acquity UPLC system/API 5000 Applied Biosystems	10	Ethyl Acetate	Filter	C18	2	Both
012	1.8	95.88	LC	MS	QqQ	AGILENT LC-MSMS 6410	10	ACN	PSA	C18	20	Manual
013			GC	MS	Q		15	Luke	GPC	HP 5	2	Both
014	None	None	LC	MS	QqQ	API4000	10	ACN	DSPE	Waters-C18 2.1x50mmx1.7µm	5	Both
015	1.1 s	0.1%	LC	MS	QqQ	Varian Pro Star +320-MS	10	ACN	Dispersive SPE with PSA + GCB	ChromSep C-18	20	Both
016			LC	MS	QqQ	API 4000	10	ACN	DSPE/SPA/C18	RP-AMIDE	10	Both
017			LC	MS	QqQ	Agilent 6410 Triple Quad	10	ACN	QuEChERS	kinetex C-18	2	Manual
020	n/a	n/a	GC	MS	IT	Varian 4000	10	ACN	DSPE	RTX-200	2	Automatic

**APPENDIX 3. Methods used by participants for detecting pesticides.**

DIURON												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
021	5	25%	LC	MS	QqQ	Quattro Premier XE	10	ACN	No	C18	5	Both
022	12		LC	MS	QqQ	API4000	10	ACN	PSA	C18ec	8	Manual
023	-0.3	-0.59%	LC	MS	QqQ	HPLC Agilent 1100, MS API 3000	5	ACN	DSPE	C18 3µ 50x2mm	10	Both
024	0		LC	MS	QqQ	Waters Xevo	10	Acetone/PE/DCM	Na <sub>2</sub> SO <sub>4</sub>	C18	2	Automatic
029	242	100	LC	MS	QqQ	API 4000	10	Acetonitril	PSA	Pursuit XRS Ultra	6	Both
030	5	1	LC	MS	QqQ	API 3200 Qtrap	10	ACN	PSA	Atlantis	10	Both
031	3	1.0	LC	MS	Orbitrap	Exactive	10	ACN		Atlantis	5	Both
036	6	5 max	LC	MS	TOF	Agilent	10	ACN	No	Zorbax Eclipse plus	3	Both
038	0.5	5	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both
039			LC	MS	QqQ	ABI 4000	10	ACN	No	C18	55	Both
040	< 0.2	< 20	LC	MS	QqQ	Waters Aquity TQD	15	ACN	PSA	UPLC-BEH C18	10	Both
041	0	0	LC	MS	QqQ	UPLC-Xevo (Waters)	20	Ethyl Acetate	LLE	HSS T3	10	Automatic
042	0.1	0.05	LC	MS	QqQ	API 3200 QT	10	ACN	DSPE	Synergi Fusion RP 10	20	Both
045	5	0.05 Da	LC	MS	TOF	LCT PREMIER XE	15	Acetone:PE:DCM	No	C:18	5	Both
046			LC	MS	QqQ	Waters Quattro Premier	10	ACN	DSPE	C18	5	Both

**APPENDIX 3. Methods used by participants for detecting pesticides.**

ETHOXYQUIN												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
001	-1.3		GC	MS		AI5975	10	ACN	SPE	HP-5MSi	10	Automatic
002	< 0.5%	< 0.5%	GC	MS	IT	Varian 4000	10	ACN	PSA	5%	10	Automatic
006	0.30	2.3	GC	MS	IT	Varian 2000	15	Acetone/DCM/PE	No	VF-5ms	5	Automatic
007	2	8.5	GC	MS	TOF	Pegasus IV	10	ACN	Dispersive SPE with PSA	HP-5MS	5	Both
011	+/- 9	30%	GC	MS	Q	Agilent GC 7890 MS 5975	10	ACN	Dispersive SPE	HP-5MS	1	Both
012	-0.25	99.5%	GC	MS	Q	AGILENT GC-MSD INERT 5975C	10	ACN	PSA	HP5MS	2	Manual
013			GC	MS	Q		15	Luke	GPC	HP 5	2	Both
014	None	None	LC	MS	QpQ	API4000	10	ACN	DSPE	Waters-C18 2.1x50mmx1.7µm	5	Both
015	1.0 s	16%	GC	MS	Q	Varian 3800 GC+320-MS	10	ACN	Dispersive SPE with PSA + GCB	VF 5ms	3	Both
016			LC	MS	QpQ	API 4000	10	ACN	DSPE/SPA/C18	RP-AMIDE	10	Both
017			GC	MS	Q	Agilent 6890N/5973N	10	ACN	QuEChERS	HP-5	1	Automatic
020	n/a	n/a	GC	MS	IT	Varian 4000	10	ACN	DSPE	RTX-200	2	Automatic
021	5	25%	LC	MS	QpQ	Quattro Premier XE	10	ACN	No	C18	5	Both
023	0	1.23%	LC	MS	QpQ	HPLC Agilent 1100. MS API 3000	5	ACN	DSPE	C18 3µ 50x2mm	10	Both
024	0		GC	MS	QpQ	TSQ Quantum GC	10	Acetone/PE/DCM	Na <sub>2</sub> SO <sub>4</sub>	RXI 5SiH-MS	1	Automatic
025	0.0	1 unit	GC	MS	Q	Agilent 5973	10	ACN	SPE	HP 5 MS	2	Automatic
026	0.05		LC	MS	QpQ	QuattroUltima. Waters	15	ACN	PSA	C18	10	Manual

**APPENDIX 3. Methods used by participants for detecting pesticides.**

ETHOXYQUIN												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
029	361	100	LC	MS	QqQ	API 4000	10	ACN	PSA	Pursuit XRS Ultra	6	Both
030	5	1	LC	MS	QqQ	API 3200 Qtrap	10	ACN	PSA	Atlantis	10	Both
031	<20	884 (match)	GC	MS	TOF	Leco Pegasus 4D	10	ACN	PSA	RTX-CL pest	10	Both
035	0.1	0.1	GC	MS	IT	Thermo POLARISQ	10	ACN	No	DB-5MS	8	Both
036	2	5 max	LC	MS	TOF	Agilent	10	ACN	No	Zorbax Eclipse plus	3	Both
038	0.5	5	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both
039	-0.3		GC	MS	Q	HP5975C	10	ACN	PSA	HP5MS	5	Both
040	< 0.2	< 20	GC	MS	Q	Agilent 7890A/5975C	15	ACN	PSA	HP5MS	10	Both
041	0	0	LC	MS	QqQ	UPLC-Xevo (Waters)	20	Ethyl Acetate	LLE	HSS T3	10	Automatic
042	0.1	0.05	GC	MS	IT	Varian Saturn 4000	10	ACN	DSPE	Varian FV5 ms	1	Both
044	0	0	GC	MS	Q	Agilent 5973	50	Acetone	No	HP 5 MS	1	Automatic
046			GC	MS	QqQ	Waters Quattro Micro	10	ACN	DSPE	HP5 MS	3	Both
048	10	5	GC and LC	MS	Q and QqQ	Interscience DSQ and Micromass Quattro premier	15	ACN	QuEChERS	DB5-MS 20m 0.18mm 0.18µm for GC and UPLC BEH C18 1.7µ 2.1mm 100mm	3 LC 0.8 GC	Automatic

**APPENDIX 3. Methods used by participants for detecting pesticides.**

FENPROPIDIN												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
001	-3.9		GC	MS		AT5975	10	ACN	SPE	HP-5MSi	10	Automatic
003	1	<5	LC	MS	Q-TOF	Waters Xevo QToF	10	Ethyl Acetate	None	Acquity BEH C18	3	Both
004	< 2%	<5%	LC	MS	QqQ	API4000	15	ACN	PSA	SpeedRod	20	Manual
005			LC	MS	QqQ	Agilent 6410B	10	ACN	DSPE	C18, 1.8µm	2	Automatic
006	1.2	1.5	LC	MS	QqQ	Waters Premier XE	15	Acetone/DCM/PE	No	Acquity BEH	5	Automatic
007	5	13.9	GC	MS	TOF	Pegasus IV	10	ACN	Dispersive SPE with PSA	HP-5MS	5	Both
009	na	na	LC	MS	QqQ	Agilent 1100/API 5000 Applied Biosystems	10	Ethyl Acetate	Filter	C18		Both
014	None	None	LC	MS	QqQ	API4000	10	ACN	DSPE	Waters-C18 2.1x50mmx1.7µm	5	Both
017			GC	MS	Q	Agilent 6890N/5973N	10	ACN	QUECHERS	HP-5	1	Automatic
021	5	25%	LC	MS	QqQ	Quattro Premier XE	10	ACN	No	C18	5	Both
023	-1.35	-6.50%	LC	MS	QqQ	HPLC Agilent 1100. MS API 3000	5	ACN	DSPE	C18 3µ 50x2mm	10	Both
024	0		LC	MS	QqQ	Waters Xevo	10	Acetone/PE/DCM	Na <sub>2</sub> SO <sub>4</sub>	C18	2	Automatic
025	2.1	1 unit	GC	MS	Q	Agilent 5973	10	ACN	SPE	HP 5 MS	2	Automatic
026			LC	MS	Q-TOF	Brucker	15	ACN	PSA	C18	5	Manual
029	747	97	GC	MS	TOF	LECO Pegasus IV	10	ACN	PSA	DB 5	3	Both
030	5	1	LC	MS	QqQ	API 3200 Qtrap	10	ACN	PSA	Atlantis	10	Both



**APPENDIX 3. Methods used by participants for detecting pesticides.**

FENPROPIDIN												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
031	<20	943 (match)	GC	MS	TOF	Leco Pegasus 4D	10	ACN	PSA	RTX-CL pest	10	Both
035	0.1	0.1	GC	MS	IT	Thermo POLARISQ	10	ACN	no	DB-5MS	8	Both
036	3	5 ppm max	LC	MS	TOF	Agilent	10	ACN	no	Zorbax Eclipse plus	3	Both
037	±12	15%	LC	MS/MS	QqQ	API 4000QT	10	ACN	DSP	C19	25	Automatic
038	0.5	5 ppm	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both
039	-4.1		GC	MS	Q	HP5975C	10	ACN	PSA	HP5MS	5	Both
040	<0.2	<20	GC	MS	Q	Agilent 7890A/5975C	15	ACN	PSA	HP5MS	10	Both
041	0	0	LC	MS	QqQ	UPLC-Xevo (Waters)	20	Ethyl Acetate	LLE	HSS T3	10	Automatic
046			LC	MS	QqQ	Waters Quattro Premier	10	ACN	DSPE	C18	5	Both
047			GC	MS	TOF	Pegasus III	50	Ethyl Acetate	GPC	VF-5ms(30mx0.25x0.25)	2	Automatic
049	0		LC	MS	QqQ	Waters Aquity UPLC T	10	Methanol	Filter	C18	1	Automatic
050	0.02		GC	MS	Q	Agilent 6890/5973	10	ACN	No	HP-5MS	1	Manual

**APPENDIX 3. Methods used by participants for detecting pesticides.**

FENPYROXIMATE												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
001			LC	MS	QqQ	API4000Trap	10	ACN	No	C18 Synergi fusion	10	Manual
003	1	<5	LC	MS	Q-TOF	Waters Xevo QToF	10	Ethyl Acetate	None	Acquity BEH C18	3	Both
004	<2%	<5%	LC	MS	QpQ	API4000	15	ACN	PSA	SpeedRod	20	Manual
005			LC	MS	QpQ	Agilent 6410B	10	ACN	DSPE	C18. 1.8µm	2	Automatic
006	2.40	12.7	LC	MS	QqQ	Waters Premier XE	15	Acetone/DCM/PE	No	Acquity BEH	5	Automatic
007	0.4	0	LC	MS	QqQ	Agilent 6410	10	ACN	Dispersive SPE with PSA	Luna C18	5	Both
008	NA	NA	LC	MS	QqQ	API 4000 QTrap	10	Ethyl Acetate	None	C18	5	Automatic
009	na	na	LC	MS	QpQ	Waters Acquity UPLC system/API 5000 Applied Biosystems	10	Ethyl Acetate	Filter	C18	2	Both
014	none	none	LC	MS	QqQ	API4000	10	ACN	DSPE	Waters-C18 2.1x50mmx1.7µm	5	Both
015	0.9 s	0.1%	LC	MS	QqQ	Varian Pro Star +320-MS	10	ACN	Dispersive SPE with PSA + GCB	ChromSep C-18	20	Both
016			LC	MS	QqQ	API 4000	10	ACN	dSPE/SPA/C18	RP-AMIDE	10	Both
017			LC	MS	QqQ	Agilent 6410 Triple Quad	10	ACN	QuEChERS	kinetex C-18	2	Manual
018	0.5	%	LC	MS	QpQ	Agilent	10	ACN	DSPE	C-18	10	Automatic
021	5	25%	LC	MS	QqQ	Quattro Premier XE	10	ACN	No	C18	5	Both
023	-0.3	1.94%	LC	MS	QpQ	HPLC Agilent 1100. MS API 3000	5	ACN	DSPE	C18 3µ 50x2mm	10	Both
024	0		LC	MS	QpQ	Waters Xevo	10	Acetone/PE/DCM	Na <sub>2</sub> SO <sub>4</sub>	C18	2	Automatic
029	485	100	LC	MS	QpQ	API 4000	10	ACN	PSA	Pursuit XRS Ultra	6	Both

**APPENDIX 3. Methods used by participants for detecting pesticides.**

FENPYROXIMATE												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
030	5	1	LC	MS	QqQ	API 3200 Qtrap	10	ACN	PSA	Atlantis	10	Both
031	0	0.8	LC	MS	Orbitrap	Exactive	10	ACN		Atlantis	5	Both
036	1	5 max	LC	MS	TOF	Agilent	10	ACN	No	Zorbax Eclipse plus	3	Both
037	±12	15%	LC	MS/MS	QqQ	API 4000QT	10	ACN	DSP	C20	25	Automatic
038	0.5	5	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both
039			LC	MS	QqQ	ABI 4000	10	ACN	No	C18	55	Both
040	<0.2	< 20	LC	MS	QqQ	Waters Acquity TQD	15	ACN	PSA	UPLC-BEH C18	10	Both
041	0	0	LC	MS	QqQ	UPLC-Xevo (Waters)	20	Ethyl Acetate	LLE	HSS T3	10	Automatic
045	5	0.05 Da	LC	MS	TOF	LCT PREMIER XE	15	ACETONE:PE:DCM	No	C:18	5	Both
046			LC	MS	QqQ	Waters Quattro Premier	10	ACN	DSPE	C18	5	Both
048	10	5	LC	LC/MSMS	QqQ	Micromass Quattro premier	15	ACN	QuEChERS	UPLC BEH C18 1.7µm 2.1mm 100mm	3 LC	Automatic

APPENDIX 3. Methods used by participants for detecting pesticides.

FURATHIOCARB												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
001	-1.3		GC	MS		AT5975	10	ACN	SPE	HP-5MSi	10	Automatic
002	< 2.5%	< 2.5%	LC	MS	QqQ	3200 Qtrap	10	ACN	PSA	C18 2.5 µm - 5 cm	50	Automatic
003	1	<5	LC	MS	Q-TOF	Waters Xevo QToF	10	Ethyl Acetate	None	Acquity BEH C18	3	Both
004	< 2%	<5%	LC	MS	QqQ	API4000	15	ACN	PSA	SpeedRod	20	Manual
006	1.62	1.2	LC	MS	QqQ	Waters Premier XE	15	Acetone/DCM/PE	No	Acquity BEH	5	Automatic
007	1	38.7	GC	MS	TOF	Pegasus IV	10	ACN	Dispersive SPE with PSA	HP-5MS	5	Both
008	NA	NA	LC	MS	QqQ	API 4000 Qtrap	10	Ethyl Acetate	None	C18	5	Automatic
009	na	na	LC	MS	QqQ	Waters Acquity UPLC system/API 5000 Applied Biosystems	10	Ethyl Acetate	Filter	C18	2	Both
011	+/- 9	30%	GC	MS	Q	Agilent GC 7890 MS 5975	10	ACN	Dispersive SPE	HP-5MS	1	Both
012	2.7	99.58%	LC	MS	QqQ	AGILENT LC-MSMS 6410	10	ACN	PSA	C18	20	Manual
013			GC	MS	Q		15	Luke	GPC	HP 5	2	Both
014	none	none	LC	MS	QqQ	API4000	10	ACN	DSPE	Waters-C18 2.1x50mmx1.7µm	5	Both
017			LC	MS	QqQ	Agilent 6410 Triple Quad	10	ACN	QuEChERS	Kinetex C-18	2	Manual
018	0.5		LC	MS	QqQ	Agilent	10	ACN	DSPE	C-18	10	Automatic
022	0		LC	MS	QqQ	API4000	10	ACN	PSA	C18ec	8	Manual
023	-0.3	-0.82%	LC	MS	QqQ	HPLC Agilent 1100. MS API 3000	5	ACN	DSPE	C18 3µ 50x2mm	10	Both
024	0		LC	MS	QqQ	Waters Xevo	10	Acetone/PE/DCM	Na <sub>2</sub> SO <sub>4</sub>	C18	2	Automatic

**APPENDIX 3. Methods used by participants for detecting pesticides.**

<b>FURATHIOCARB</b>												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
029	460	100	LC	MS	QqQ	API 4000	10	ACN	PSA	Pursuit XRS Ultra	6	Both
030	5	1	GC	MS	Q	Agilent singleQ	10	ACN	PSA	HP-MS 5	20	Both
031	-3	0.1	LC	MS	Orbitrap	Exactive	10	ACN		Atlantis	5	Both
036	3.6	5 max	LC	MS	TOF	Agilent	10	ACN	no	Zorbax Eclipse plus	3	Both
037	±12	15%	LC	MS/MS	QqQ	API 4000QT	10	ACN	DSF	C22	25	Automatic
038	0.5	5 ppm	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both
039			LC	MS	QqQ	ABI 4000	10	ACN	No	C18	55	Both
040	<0.2	<20	LC	MS	QqQ	Waters Aquity TQD	15	ACN	PSA	UPLC-BEH C18	10	Both
041	0	0	LC	MS	QqQ	UPLC-Xevo (Waters)	20	Ethyl Acetate	LLE	HSS T3	10	Automatic
042	0.1	0.05	LC	MS	QqQ	API 3200 QT	10	ACN	DSPE	Synergi Fusion RP100	20	Both
044	0	0	GC	MS	Q	Agilent 5973	50	Acetone	No	HP 5 MS	1	Automatic
046			LC	MS	QqQ	Waters Quattro Premier	10	ACN	DSPE	C18	5	Both
047			GC	MS	TOF	Pegasus III	50	Ethyl Acetate	GPC	VF-5ms (30mx0.25x0.25)	2	Automatic
050	0.02		GC	MS	Q	Agilent 6890/5973	10	ACN	No	HP-5MS	1	Manual

**APPENDIX 3. Methods used by participants for detecting pesticides.**

ISOFENPHOS METHYL												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
001	0.3		GC	MS		AT5975	10	ACN	SPE	HP-5MSI	10	Automatic
002	< 0.5%	< 0.5%	GC	MS	IT	Varian 4000	10	ACN	And A	5%	10	Automatic
004	< 2%	< 5%	GC	MS	GqQ	Agilent 7000A	15	Acetone/PE/DCM	None	HP5MS	2	Both
005			GC	MS	Q	Agilent 5973N	10	ACN	DSPE	HP5-MS(I)	20	Both
006	0.72	0.6	GC	MS	IT	Varian 2000	15	Acetone/DCM/PE	No	VF-5ms	5	Automatic
007	4	9.2	GC	MS	TOF	Pegasus IV	10	ACN	Dispersive SPE with PSA	HP-5MS	5	Both
008	NA	NA	LC	MS	GqQ	API 4000 Qtrap	10	Ethyl Acetate	None	C18	5	Automatic
009	na	na	LC	MS	GqQ	Waters Acquity UPLC system/ API 5000 Applied Biosystems	10	Ethyl Acetate	Filter	C18	2	Both
012	-1.15	91.5%	GC	MS	Q	AGILENT GC-MSD INERT 5975C	10	ACN	PSA	HP5MS	2	Manual
013			GC	MS	Q		15	Luke	GPC	HP 5	2	Both
014	None	None	LC	MS	GqQ	API4000	10	ACN	DSPE	Waters-C18 2.1x50mmx1.7µm	5	Both
015	2.3 s	0.1%	GC	MS	GqQ	Varian 3800 GC+320-MS	10	ACN	Dispersive SPE with PSA + GCB	VF 5ms	3	Both
017			GC	MS	Q	Agilent 6890N/5973N	10	ACN	QuEChERS	HP-5	1	Automatic
018	0.5		LC	MS	GqQ	Agilent	10	ACN	DSPE	C-18	10	Automatic
019	30	30%	GC	MS	IT	Varian 2000 Ion Trap	15	Ethyl Acetate		DB 5 MS	10	Automatic
020	n/a	n/a	GC	MS	IT	Varian 4000	10	ACN	DSPE	RTX-200	2	Automatic
021	5	25%	LC	MS	GqQ	Quattro Premier XE	10	ACN	No	C18	5	Both
022	6		LC	MS	GqQ	API4000	10	ACN	PSA	C18ec	8	Manual
023	0	-4.52%	LC	MS	GqQ	HPLC Agilent 1100. MS API 3000	5	ACN	DSPE	C18 3µ 50x2mm	10	Both
024	0	%	GC	MS	GqQ	TSQ Quantum GC	10	Acetone/PE/DCM	Na <sub>2</sub> SO <sub>4</sub>	RXi 5Sil-MS	1	Automatic

**APPENDIX 3. Methods used by participants for detecting pesticides.**

ISOFENPHOS METHYL												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
029	822	99	GC	MS	TOF	LECO Pegasus IV	10	ACN	PSA	DB 5	3	Both
030	5	1	GC	MS	GqQ	Agilent 7000	10	ACN	PSA	HP-MS 5	1	Both
031	<20	909 (match)	GC	MS	TOF	Leco Pegasus 4D	10	ACN	PSA	RTX-CL pest	10	Both
033			GC	MS	IT	Varian GC/MS 4000	10	ACN	PSA/MgSO <sub>4</sub> /GCB	VF-5ms 30m*0.25mm	5	Manual
035	0.1	0.1	GC	MS	IT	Thermo POLARISQ	10	ACN	No	DB-5MS	8	Both
036	8	5 max	LC	MS	TOF	Agilent	10	ACN	No	Zorbax Eclipse plus	3	Both
037	±12	15%	LC	MS/MS	GqQ	API 4000QT	10	ACN	DSP	C23	25	Automatic
038	0.5	5	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both
039			GC	MS	Q	HP5975C	10	ACN	PSA	HP5MS	5	Both
041	0	0	GC	MS	IT	UltraTrace GC-Polaris Q (Thermo)	25	Hexane	LLE	ZB-5MS	1	Automatic
042	0.1	0.05	GC	MS	IT	Varian Saturn 4000	10	ACN	DSPE	Varian FV5 ms	1	Both
044	0	0	GC	MS	Q	Agilent 5973	50	Acetone	No	HP 5 MS	1	Automatic
045	5	0.05 Da	LC	MS	TOF	LCT PREMIER XE	15	Acetone:PE:DCM	No	C:18	5	Both
046			GC	MS	GqQ	Waters Quattro Micro	10	ACN	DSPE	HP5 MS	3	Both
048	10	5	GC and LC	MS	Q and GqQ	Interscience DSQ and Micromass Quattro premier	15	ACN	QuEChERS	DB5-MS 20m 0.18mm 0.18µm for GC and UPLC BEH C18 1.7µ 2.1mm 100mm	3 LC 0.8 GC	Automatic
049	0.05		GC	MS	IT	Thermo Polaris Q	10	ACN	PSA	Capillary	1	Both
050	0.02		GC	MS	Q	Agilent 6890/5973	10	ACN	No	HP-5MS	1	Manual

**APPENDIX 3. Methods used by participants for detecting pesticides.**

ISOPROCARB												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
001	1.8		GC	MS		AT5975	10	ACN	SPE	HP-5MSi	10	Automatic
003	1	<5	LC	MS	Q-TOF	Waters Xevo QToF	10	Ethyl/ Acetate	None	Acquity BEH C18	3	Both
005			LC	MS	QqQ	Agilent 6410B	10	ACN	DSPE	C18: 1.8µm	2	Automatic
006	0.84	9.0	GC	MS	IT	Varian 2000	15	Acetone/DCM/PE	No	VF-5ms	5	Automatic
007	0	10.4	GC	MS	TOF	Pegasus IV	10	ACN	Dispersive SPE with PSA	HP-5MS	5	Both
008	NA	NA	LC	MS	QqQ	API 4000 Qtrap	10	Ethyl/ Acetate	None	C18	5	Automatic
009	na	na	LC	MS	QqQ	Waters Acquity UPLC system/API 5000 Applied Biosystems	10	Ethyl/ Acetate	Filter	C18	2	Both
011	+/- 9	30%	GC	MS	Q	Agilent GC 7890 MS 5975	10	ACN	Dispersive SPE	HP-5MS	1	Both
013			GC	MS	Q		15	Luke	GPC	HP 5	2	Both
014	None	None	LC	MS	QqQ	API4000	10	ACN	DSPE	Waters-C18 2.1x50mmx1.7µm	5	Both
015	4.4 s	0.1%	LC	MS	QqQ	Varian Pro Star +320-MS	10	ACN	Dispersive SPE with PSA + GCB	ChromSep C-18	20	Both
016	15	48%	GC	MS	Q	Thermo DSQ	10	ACN	dsPE/SFA/C18	Varian VF-5ms	1	Both
017			GC	MS	Q	Agilent 6890N/5973N	10	ACN	QuEChERS	HP-5	1	Automatic
020	n/a	n/a	GC	MS	IT	Varian 4000	10	ACN	DSPE	RTX-200	2	Automatic
023	-0.3	2.20%	GC	MS	Q	GC Agilent 6890N. MS Agilent 5976	5	ACN	DSPE	HP-5MS	3	Both



**APPENDIX 3. Methods used by participants for detecting pesticides.**

ISOPROCARB												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
025	2.6	1 unit	GC	MS	Q	Agilent 5973	10	ACN	SPE	HP 5 MS	2	Automatic
029	551	96	GC	MS	TOF	LECO Pegasus IV	10	ACN	PSA	DB 5	3	Both
030	5	1	LC	MS	GqQ	API 3200 Qtrap	10	ACN	PSA	Atlantis	10	Both
031	-3.6	1.2	LC	MS	Orbitrap	Exactive	10	ACN		Atlantis	5	Both
036	2.5% max	None	LC	MS	MS/MS	Waters Premier XE	10	ACN	No	Acquity UPLC HSS T3	20	Both
037	±12	15%	LC	MS/MS	GqQ	API 4000QT	10	ACN	DSP	C24	25	Automatic
038	0.5	5 ppm	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both
039			LC	MS	GqQ	ABI 4000	10	ACN	No	C18	55	Both
040	< 0.2	< 20	GC	MS	Q	Agilent 7890A/5975C	15	ACN	PSA	HP5MS	10	Both
042	0.1	0.05	LC	MS	GqQ	API 3200 QT	10	ACN	DSPE	Synergi Fusion RP100	20	Both
045	5	0.05 Da	LC	MS	TOF	LCT PREMIER XE	15	Acetone:PE:DCM	No	C:18	5	Both
046			GC	MS	Q	Agilent MSD 5973	10	ACN	DSPE	HP5 MS	5	Both
047			GC	MS	TOF	Pegasus III	50	Ethyl Acetate	GPC	VF-5ms(30mx0.25x0.25)	2	Automatic

**APPENDIX 3. Methods used by participants for detecting pesticides.**

MECARBAM												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
001	-3.0		GC	MS		AI5975	10	ACN	SPE	HP-5MSI	10	Automatic
002	< 0.5%	< 0.5%	GC	MS	IT	Varian 4000	10	ACN	PSA	5%	10	Automatic
004	< 2%	< 5%	GC	MS	QqQ	Agilent 7000A	15	Acetone/PE/DCM	None	HP5MS	2	Both
005			LC	MS	QqQ	Agilent 6410B	10	ACN	DSPE	C18, 1.8µm	2	Automatic
006	0.42	2.4	GC	MS	IT	Varian 2000	15	Acetone/DCM/PE	No	VF-5ms	5	Automatic
007	5	6.6	GC	MS	TOF	Pegasus IV	10	ACN	Dispersive SPE with PSA	HP-5MS	5	Both
008	NA	NA	LC	MS	QqQ	API 4000 Qtrap	10	Ethyl Acetate	None	C18	5	Automatic
009	na	na	LC	MS	QqQ	Waters Acquity UPLC system/API 5000 Applied Biosystems	10	Ethyl Acetate	Filter	C18	2	Both
011	+/- 9	30%	GC	MS	Q	Agilent GC 7890 MS 5975	10	ACN	Dispersive SPE	HP-5MS	1	Both
012	4.5	87.72%	LC	MS	QqQ	AGILENT LC-MSMS 6410	10	ACN	PSA	C18	20	Manual
013			GC	MS	Q		15	Luke	GPC	HP 5	2	Both
014	None	None	LC	MS	QqQ	API4000	10	ACN	DSPE	Waters-C18 2.1x50mmx1.7µm	5	Both
015	2.5 s	0.1%	LC	MS	QqQ	Varian Pro Star + 320-MS	10	ACN	Dispersive SPE with PSA + GCB	ChromSep C-18	20	Both
016			LC	MS	QqQ	API 4000	10	ACN	DSPE/SPA/C18	RP-AMIDE	10	Both
017			LC	MS	QqQ	Agilent 6410 Triple Quad	10	ACN	QuEChERS	kinetex C-18	2	Manual

**APPENDIX 3. Methods used by participants for detecting pesticides.**

MECARBAM												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
019	30	30%	GC	MS	IT	Varian 2000 Ion Trap	15	Ethil Acetate		DB 5 MS	10	Automatic
020	n/a	n/a	GC	MS	IT	Varian 4000	10	ACN	DSPE	RTX-200	2	Automatic
021	5	25%	LC	MS	QqQ	Quattro Premier XE	10	ACN	No	C18	5	Both
022	12		LC	MS	QqQ	API4000	10	ACN	PSA	C18ec	8	Manual
023	0	-0.99%	LC	MS	QqQ	HPLC Agilent 1100. MS API 3000	5	ACN	DSPE	C18 3µ 50x2mm	10	Both
024	0		GC	MS	QqQ	TSQ Quantum GC	10	Acetone/PE/DCM	Na <sub>2</sub> SO <sub>4</sub>	RXI 5Sil-MS	1	Automatic
025	1.5	1 unit	GC	MS	Q	Agilent 5973	10	ACN	SPE	HP 5 MS	2	Automatic
026		850 out of 1000	GC	MS	Q	Waters MicroGCMSMS	15	ACN	PSA	DB5	4	Manual
029	858	97	GC	MS	TOF	LECO Pegasus IV	10	ACN	PSA	DB 5	3	Both
030	5	1	LC	MS	QqQ	API 3200 Qtrap	10	ACN	PSA	Atlantis	10	Both
031	<20	942 (match)	GC	MS	TOF	Leco Pegasus 4D	10	ACN	PSA	RTX-CL pest	10	Both
033			GC	MS	IT	Varain GC/MS 4000	10	ACN	PSA/MgSO <sub>4</sub> /GCB	VF-5ms 30m*0.25mm	5	Manual
035	0.1	0.1	GC	MS	IT	Thermo POLARISQ	10	ACN	no	DB-5MS	8	Both
036	13	5 max	LC	MS	TOF	Agilent	10	ACN	no	Zorbax Eclipse plus	3	Both
037	±12	15%	LC	MS/MS	QqQ	API 4000QT	10	ACN	DSP	C25	25	Automatic
038	0.5	5	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both

**APPENDIX 3. Methods used by participants for detecting pesticides.**

MECARBAM												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
039	0.5		GC	MS	Q	HP5975C	10	ACN	PSA	HP5MS	5	Both
040	< 0.2	< 20	LC	MS	QqQ	Waters Aquity TQD	15	ACN	PSA	UPLC-BEH C18	10	Both
041	0	0	LC	MS	QqQ	UPLC-Xevo (Waters)	20	Ethyl Acetate	LLE	HSS T3	10	Automatic
042	0.1	0.05	GC	MS	IT	Varian Saturn 4000	10	ACN	DSPE	Varian FV5 ms	1	Both
044	0	0	GC	MS	Q	Agilent 5973	50	Acetone	No	HP 5 MS	1	Automatic
045	5	0.05 Da	LC	MS	TOF	LCT PREMIER XE	15	Acetone:PE:DCM	No	C:18	5	Both
046			GC	MS	QqQ	Waters Quattro Micro	10	ACN	DSPE	HP5 MS	3	Both
047	No	No	GC	MS	TOF	Pegasus III	50	Ethyl Acetate	GPC	VF-5ms (30mx0.25x0.25)	2	Automatic
048	10	5	GC and LC	MS	Q and QqQ	Interscience DSQ and Micromass Quattro premier	15	ACN	QuEChERS	DB5-MS 20m 0.18mm 0.18µm for GC and UPLC BEH C18 1.7µ 2.1mm 100mm	3 LC 0.8 GC	Automatic
049	0.01		LC	MS	QqQ	Waters Aquity UPLC T	10	Methanol	Filter	C18	1	Automatic
050	0.02		GC	MS	Q	Agilent 6890/5973	10	ACN	No	HP-5MS	1	Manual

**APPENDIX 3. Methods used by participants for detecting pesticides.**

METOLACHLOR												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
001	-2.4		GC	MS		AT5975	10	ACN	SPE	HP-5MSi	10	Automatic
002	< 0.5%	< 0.5%	GC	MS	IT	Varian 4000	10	ACN	PSA	5%	10	Automatic
003	1	<5	LC	MS	Q-TOF	Waters Xevo QToF	10	Ethyl Acetate	None	Acquity BEH C18	3	Both
004	< 2%	<5%	GC	MS	QqQ	Agilent 7000A	15	Acetone/PE/DCM	None	HP5MS	2	Both
006	0.00	2.4	GC	MS	IT	Varian 2000	15	Acetone/DCM/PE	No	VF-5ms	5	Automatic
007	3	10.0	GC	MS	TOF	Pegasus IV	10	ACN	Dispersive SPE with PSA	HP-5MS	5	Both
008	-0.07	0.2	LC	MS	Q-TOF	Bruker Maxis	10	Ethyl Acetate	None	C18	1	Automatic
009	na	na	LC	MS	QqQ	Waters Acquity UPLC system/ API 5000 Applied Biosystems	10	Ethyl Acetate	Filter	C18	2	Both
011	+/- 9	30%	GC	MS	Q	Agilent GC 7890 MS 5975	10	ACN	Dispersive SPE	HP-5MS	1	Both
013			LC	MS	QqQ		10	QuEChERS		C8	20	Both
014	None	None	LC	MS	QqQ	API4000	10	ACN	DSPE	Waters-C18 2.1x50mmx1.7µm	5	Both
015	2.3 s	1.2%	GC	MS	QqQ	Varian 3800 GC+320-MS	10	ACN	Dispersive SPE with PSA + GCB	VF 5ms	3	Both
016	7	2%	GC	MS	Q	Thermo DSQ	10	ACN	dSPE/SPA/C18	Varian VF-5ms	1	Both
017			GC	MS	Q	Agilent 6890N/5973N	10	ACN	QuEChERS	HP-5	1	Automatic

**APPENDIX 3. Methods used by participants for detecting pesticides.**

<b>METOLACHLOR</b>												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
020	n/a	n/a	GC	MS	IT	Varian 4000	10	ACN	DSPE	RTX-200	2	Automatic
021	5	25%	LC	MS	QqQ	Quattro Premier XE	10	ACN	No	C18	5	Both
022	0		LC	MS	QqQ	API4001	10	ACN	PSA	C18ec	8	Manual
023	0	3.07%	LC	MS	QqQ	HPLC Agilent 1100, MS API 3000	5	ACN	DSPE	C18 3µ 50x2mm	10	Both
024	0		LC	MS	QqQ	Waters Xevo	10	Acetone/PE/DCM	Na <sub>2</sub> SO <sub>4</sub>	C18	2	Automatic
025	1.0	1 unit	GC	MS	Q	Agilent 5973	10	ACN	SPE	HP 5 MS	2	Automatic
026		4.7	LC	MS	Q-TOF	Brucker	15	ACN	PSA	C18	5	Manual
029	326	100	LC	MS	QqQ	API 4000	10	ACN	PSA	Pursuit XRS Ultra	6	Both
030	5	1	GC	MS	Q	Agilent singleQ	10	ACN	PSA	HP-MS 5	20	Both
031	<20	932 (match)	GC	MS	TOF	Leco Pegasus 4D	10	ACN	PSA	RTX-CL pest	10	Both
035	0.1	0.1	GC	MS	IT	Thermo POLARISQ	10	ACN	No	DB-5MS	8	Both
036	1	5 max	LC	MS	TOF	Agilent	10	ACN	No	Zorbax Eclipse plus	3	Both
037	±12	15%	LC	MS/MS	QqQ	API 4000QT	10	ACN	DSP	C26	25	Automatic
038	0.5	5	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both
039	-1.9		GC	MS	Q	HP5975C	10	ACN	PSA	HP5MS	5	Both
040	<0.2	<20	GC	MS	Q	Agilent 7890A/5975C	15	ACN	PSA	HP5MS	10	Both

**APPENDIX 3. Methods used by participants for detecting pesticides.**

<b>METOLACHLOR</b>												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
041	0	0	LC	MS	QqQ	UPLC-Xevo (Waters)	20	Ethyl Acetate	LLE	HSS T3	10	Automatic
042	0.1	0.05	GC	MS	IT	Varian Saturn 4000	10	ACN	DSPE	Varian FV5 ms	1	Both
044	0	0	GC	MS	Q	Agilent 5973	50	Acetone	No	HP 5 MS	1	Automatic
045	5	0.05 Da	LC	MS	TOF	LCT PREMIER XE	15	Acetone:PE:DCM	No	C:18	5	Both
046			GC	MS	Q	Agilent MSD 5973	10	ACN	DSPE	HP5 MS	5	Both
048	10	5	GC	MS	Q	interscience DSQ	15	ACN	QuEChERS	DB5-MS 20m 0.18mm 0.18µm	0.8	Automatic
050	0.02		GC	MS	Q	Agilent 6890/5973	10	ACN	No	HP-5MS	1	Manual

**APPENDIX 3. Methods used by participants for detecting pesticides.**

<b>METRIBUZIN</b>												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
001	3.1		GC	MS		AT5975	10	ACN	SPE	HP-5MSi	10	Automatic
002	< 0.5%	< 0.5%	GC	MS	IT	Varian 4000	10	ACN	PSA	5%	10	Automatic
004	< 2%	<5%	GC	MS	QqQ	Agilent 7000A	15	Acetone/PE/DCM	None	HP5MS	2	Both
005			LC	MS	QqQ	Agilent 6410B	10	ACN	DSPE	C18. 1.8µm	2	Automatic
006	2.76	1.5	GC	MS	IT	Varian 2000	15	Acetone/DCM/PE	No	VF-5ms	5	Automatic
007	5	12.9	GC	MS	TOF	Pegasus IV	10	ACN	Dispersive SPE with PSA	HP-5MS	5	Both
008	NA	NA	GC	MS	QqQ	Varian 1200L. GC3800	10	Ethyl Acetate	None	FactorFour. VF-5ms	2	Automatic
009	na	na	GC	MS	QqQ	Waters Quattro Micro GC	10	Ethyl Acetate	Filter	Fused silica capillary column	10	Both
011	+/- 9	30%	GC	MS	Q	Agilent GC 7890 MS 5975	10	ACN	Dispersive SPE	HP-5MS	1	Both
012	3.9	101.92%	LC	MS	QqQ	AGILENT LC-MSMS 6410	10	ACN	PSA	C18	20	Manual
013			LC	MS	QqQ		10	QuEChERS		C8	20	Both
014	None	None	LC	MS	QqQ	API4000	10	ACN	DSPE	Waters-C18 2.1x50mmx1.7µm	5	Both
015	1.8 s	0.1%	GC	MS	QqQ	Varian 3800 GC+ 320-MS	10	ACN	Dispersive SPE with PSA + GCB	VF 5ms	3	Both
016			LC	MS	QqQ	API 4000	10	ACN	DSPE/SPA/C18	RP-AMIDE	10	Both
017			GC	MS	Q	Agilent 6890N/5973N	10	ACN	QuEChERS	HP-5	1	Automatic
020	n/a	n/a	GC	MS	IT	Varian 4000	10	ACN	DSPE	RTX-200	2	Automatic



**APPENDIX 3. Methods used by participants for detecting pesticides.**

METRIBUZIN												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
021	5	25%	LC	MS	QqQ	Quattro Premier XE	10	ACN	No	C18	5	Both
022	18		LC	MS	QqQ	API4000	10	ACN	PSA	C18ec	8	Manual
023	0.6	-3.51%	LC	MS	QqQ	HPLC Agilent 1100. MS API 3000	5	ACN	DSPE	C18 3µ 50x2mm	10	Both
024	0		GC	MS	QqQ	TSQ Quantum GC	10	Acetone/PE/DCM	Na <sub>2</sub> SO <sub>4</sub>	RXI 5SiI-MS	1	Automatic
025	1.3	1 unit	GC	MS	Q	Agilent 5973	10	ACN	SPE	HP 5 MS	2	Automatic
026	0.01		GC	MS	QqQ	Waters MicroGCM SMS	15	ACN	PSA	DB5	4	Manual
029	203	100	LC	MS	QqQ	API 4000	10	ACN	PSA	Pursuit XRS Ultra	6	Both
030	5	1	LC	MS	QqQ	API 3200 Qtrap	10	ACN	PSA	Atlantis	10	Both
031	<20	888 (match)	GC	MS	TOF	Leco Pegasus 4D	10	ACN	PSA	RTX-CL pest	10	Both
035	0.1	0.1	LC	MS	QqQ	Thermo TSQ Quantum	10	ACN	No	XTerra	20	Both
036	5	5 max	LC	MS	TOF	Agilent	10	ACN	No	Zorbax Eclipse plus	3	Both
037	±12	15%	LC	MS/MS	QqQ	API 4000QT	10	ACN	DSP	C27	25	Automatic
038	0.5	5 ppm	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both
039			LC	MS	QqQ	ABI 4000	10	ACN	No	C18	55	Both
040	< 0.2	< 20	LC	MS	QqQ	Waters Aquity TQD	1.5	ACN	PSA	UPLC-BEH C18	10	Both
041	0	0	LC	MS	QqQ	UPLC-Xevo (Waters)	20	Ethyl Acetate	LLE	HSS T3	10	Automatic
042	0.1	0.05	GC	MS	IT	Varian Saturn 4000	10	ACN	DSPE	Varian FV5 ms	1	Both

**APPENDIX 3. Methods used by participants for detecting pesticides.**

<b>METRIBUZIN</b>												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
044	0	0	GC	MS	Q	Agilent 5973	50	Acetone	No	HP 5 MS	1	Automatic
045	5	0.05 Da	LC	MS	TOF	LCT PREMIER XE	15	ACETONE:PE:DCM	No	C:18	5	Both
046			GC	MS	QqQ	Waters Quattro Micro	10	ACN	DSPE	HP5 MS	3	Both
047	No	No	GC	MS	TOF	Pegasus III	50	Ethyl Acetate	GPC	VF-5ms(30mx0.25x0.25)	2	Automatic
048	10	5	GC and LC	MS	Q and QqQ	Interscience DSQ and Micromass Quattro premier	15	ACN	QuEChERS	DB5-MS 20m 0.18mm 0.18µm for GC and UPLC BEH C18 1.7µ 2.1mm 100mm	3 LC 0.8 GC	Automatic
049	0.01		LC	MS	QqQ	Waters Aquity UPLC T	10	Methanol	Filter	C18	1	Automatic
050	0		LC	MS	QqQ	Agilent 6400	10	ACN	No	Eclipse XDB-C18	4	Manual

**APPENDIX 3. Methods used by participants for detecting pesticides.**

MEVINPHOS												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
001	0.2		GC	MS		AT5975	10	ACN	SPE	HP-5MSi	10	Automatic
002	< 0.5%	< 0.5%	GC	MS	IT	Varian 4000	10	ACN	PSA	5%	10	Automatic
004	< 2%	< 5%	GC	MS	QqQ	Agilent 7000A	15	Acetone/PE/DCM	None	HP5MS	2	Both
005			LC	MS	QqQ	Agilent 6410B	10	ACN	DSPE	C18, 1.8µm	2	Automatic
006	0.60	2.2	GC	MS	IT	Varian 2000	15	Acetone/DCM/PE	No	VF-5ms	5	Automatic
007	1	18.2	GC	MS	TOF	Pegasus IV	10	ACN	Dispersive SPE with PSA	HP-5MS	5	Both
008	NA	NA	GC	MS	QqQ	Varian 1200L, GC3800	10	Ethyl Acetate	None	FactorFour, VF-5ms	2	Automatic
009	NA	NA	GC	MS	QqQ	Waters Quattro Micro GC	10	Ethyl Acetate	Filter	Fused silica capillary column	10	Both
011	+/- 9	30%	GC	MS	Q	Agilent GC 7890 MS 5975	10	ACN	Dispersive SPE	HP-5MS	1	Both
012	-0.35	98.0%	GC	MS	Q	AGILENT GC-MSD INERT 5975C	10	ACN	PSA	HP5MS	2	Manual
013			GC	MS	Q		15	Luke	GPC	HP 5	2	Both
015	1.0 s	No	GC	FPD	No	Varian 3800 GC	10	ACN	Dispersive SPE with PSA + GCB	Rtx OPP	1	Both
016	12	8%	GC	MS	Q	Thermo DSQ	10	ACN	DSPE/SPA/C18	Varian VF-5ms	1	Both
017			GC	MS	Q	Agilent 6890N/5973N	10	ACN	QUEChERS	HP-5	1	Automatic
018	0.25		GC	NPD	TOF	Agilent	15	Acetone+DCM+PE	None	DB-5 & DB-17	1	Manual

**APPENDIX 3. Methods used by participants for detecting pesticides.**

MEVINPHOS												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
019	30	30%	GC	MS	IT	Varian 2000 Ion Trap	15	Ethil Acetate		DB 5 MS	10	Automatic
020	n/a	n/a	GC	MS	IT	Varian 4000	10	ACN	DSPE	RTX-200	2	Automatic
021	5	25%	LC	MS	QqQ	Quattro Premier XE	10	ACN	No	C18	5	Both
022	12		LC	MS	QqQ	API4000	10	ACN	PSA	C18ec	8	Manual
023	2.5	0.71%	LC	MS	QqQ	HPLC Agilent 1100, MS API 3000	5	ACN	DSPE	C18 3µ 50x2mm	10	Both
024	0		GC	MS	QqQ	TSQ Quantum GC	10	Acetone/PE/DCM	Ng <sub>2</sub> SO <sub>4</sub>	RXi 5Sil-MS	1	Automatic
025	2.1	1 unit	GC	MS	Q	Agilent 5973	10	ACN	SPE	HP 5 MS	2	Automatic
026		829 out of 1000	GC	MS	Q	Waters MicroGCMSMS	15	ACN	PSA	DB5	4	Manual
029	175	100	LC	MS	QqQ	API 4000	10	ACN	PSA	Pursuit XRS Ultra	6	Both
030	5	1	LC	MS	QqQ	API 3200 Qtrap	10	ACN	PSA	Atlantis	10	Both
031	<20	914 (match)	GC	MS	TOF	Leco Pegasus 4D	10	ACN	PSA	RTX-CL pest	10	Both
033			GC	MS	IT	Varian GC/MS 4000	10	ACN	PSA/MgSO <sub>4</sub> /GCB	VF-5ms 30m*0.25mm	5	Manual
035	0.1	0.1	GC	MS	IT	Thermo POLARISQ	10	ACN	No	DB-5MS	8	Both
036	9	5 max	LC	MS	TOF	Agilent	10	ACN	No	Zorbax Eclipse plus	3	Both
037	±12	15%	LC	MS/MS	QqQ	API 4000QT	10	ACN	DSP	C28	25	Automatic
038	0.5	5 ppm	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both

**APPENDIX 3. Methods used by participants for detecting pesticides.**

MEVINPHOS												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
039	0.4		GC	MS	Q	HP5975C	10	ACN	PSA	HP5MS	5	Both
042	0.1	0.05	GC	MS	IT	Varian Saturn 4000	10	ACN	DSPE	Varian FV5 ms	1	Both
044	0	0	GC	MS	Q	Agilent 5973	50	Acetone	No	HP 5 MS	1	Automatic
045	5	0.05 Da	LC	MS	TOF	LCT PREMIER XE	15	Acetone:PE:DCM	No	C:18	5	Both
046			GC	MS	QqQ	Waters Quattro Micro	10	ACN	DSPE	HP5 MS	3	Both
047	No	No	GC	MS	TOF	Pegasus III	50	Ethyl Acetate	GPC	VF-5ms(30mx0.25x0.25)	2	Automatic
048	10	5	GC	MS	Q	interscience DSQ	15	ACN	QuEChERS	DB5-MS 20m 0.18mm 0.18µm	0.8	Automatic
049	0		LC	MS	QqQ	Waters Acquity UPLC T	10	Methanol	Filter	C18	1	Automatic
050	0		GC	MS	Q	Agilent 6890/5973	10	ACN	No	HP-5MS	1	Manual

**APPENDIX 3. Methods used by participants for detecting pesticides.**

PHORATE												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
001	-2.5		GC	MS		AT5975	10	ACN	SPE	HP-5MSI	10	Automatic
002	< 0.5%	< 0.5%	GC	MS	IT	Varian 4000	10	ACN	PSA	5%	10	Automatic
003	1	< 5ppm	LC	MS	Q-TOF	Waters Xevo QToF	10	Ethyl Acetate	None	Acquity BEH C18	3	Both
004	< 2%	< 5%	LC	MS	QqQ	API4000	15	ACN	PSA	SpeedRod	20	Manual
005			LC	MS	QqQ	Agilent 6410B	10	ACN	DSPE	C18, 1.8µm	2	Automatic
006	0.66	2.9	GC	MS	IT	Varian 2000	15	Acetone/DCM/PE	No	VF-5ms	5	Automatic
007	0	7.2	GC	MS	TOF	Pegasus IV	10	ACN	Dispersive SPE with PSA	HP-5MS	5	Both
008	NA	NA	LC	MS	QqQ	API 4000 Qtrap	10	Ethyl Acetate	none	C18	5	Automatic
009	na	na	LC	MS	QqQ	Waters Acquity UPLC system/API 5000 Applied Biosystems	10	Ethyl Acetate	Filter	C18	2	Both
011	+/- 9	30%	GC	MS	Q	Agilent GC-7890 MS 5975	10	ACN	Dispersive SPE	HP-5MS	1	Both
012	-0.45	98.0%	GC	MS	Q	AGILENT GC-MSD INERT 5975C	10	ACN	PSA	HP5MS	2	Manual
013			GC	MS	Q		15	Luke	GPC	HP 5	2	Both
014	none	none	LC	MS	QqQ	API4000	10	ACN	DSPE	Waters-C18 2.1x50mmx1.7µm	5	Both
015	2.2 s	20%	GC	MS	QqQ	Varian 3800 GC+ 320-MS	10	ACN	Dispersive SPE with PSA + GCB	VF 5ms	3	Both
016	4	2%	GC	MS	Q	Thermo DSQ	10	ACN	DSPE/SPA/C18	Varian VF-5ms	1	Both
017			GC	MS	Q	Agilent 6890N/5973N	10	ACN	QUECHERS	HP-5	1	Automatic
018	0.25		GC	NPD	TOF	Agilent	15	Acetone+DCM+PE	None	DB-5 & DB-17	1	Manual
020	n/a	n/a	GC	MS	IT	Varian 4000	10	ACN	DSPE	RTX-200	2	Automatic
021	5	25%	LC	MS	QqQ	Quattro Premier XE	10	ACN	No	C18	5	Both
023	-0.15	-2.35%	LC	MS	QqQ	HPLC Agilent 1100. MS API 3000	5	ACN	DSPE	C18 3µ 50x2mm	10	Both
024	0		GC	MS	QqQ	TSQ Quantum GC	10	Acetone/PE/DCM	Na <sub>2</sub> SO <sub>4</sub>	RXi SSiI-MS	1	Automatic
025	0.1	1 unit	GC	MS	Q	Agilent 5973	10	ACN	SPE	HP 5 MS	2	Automatic

**APPENDIX 3. Methods used by participants for detecting pesticides.**

PHORATE												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-up	Column Type	Injection Volume (µl)	Software
026		850 out of 100	GC	MS	Q	Waters MicroGCMSMS	15	ACN	PSA	DB5	4	Manual
029	613	99	GC	MS	TOF	LECO Pegasus IV	10	ACN	PSA	DB 5	3	Both
030	5	1	GC	MS	Q	Agilent singleQ	10	ACN	PSA	HP-MS 5	20	Automatic
031	<20	888 (match)	GC	MS	TOF	Leco Pegasus 4D	10	ACN	PSA	RTX-CL pest	10	Both
035	0.1	0.1	GC	MS	IT	Thermo POLARISQ	10	ACN	No	DB-5MS	8	Both
036	5% max	none	GC	MS	Q	Agilent MSD 5973	30	Ethyl Acetate	HPGPC	DB5-MS	2	Automatic
037	±12	15%	LC	MS/MS	QqQ	API 4000QT	10	ACN	DSP	C29	25	Automatic
038	0.5	5 ppm	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both
039	-2.3		GC	MS	Q	HP5975C	10	ACN	PSA	HP5MS	5	Both
040	< 0.2	< 20	GC	MS	Q	Agilent 7890A/5975C	15	ACN	PSA	HP5MS	10	Both
041	0	0	GC	MS	IT	UltraTrace GC-Polaris Q (Thermo)	25	Hexane	LLE	ZB-5MS	1	Automatic
042	0.1	0.05	GC	MS	IT	Varian Saturn 4000	10	ACN	DSPE	Varian FV5 ms	1	Both
044	0	0	GC	MS	Q	Agilent 5973	50	Acetone	No	HP 5 MS	1	Automatic
046			LC	MS	QqQ	Waters Quattro Premier	10	ACN	DSPE	C18	5	Both
047			GC	MS	TOF	Pegasus III	50	Ethyl Acetate	GPC	VF-5ms(30mx0.25x0.25)	2	Automatic
048	10	5	GC	MS	Q	Interscience DSQ	15	ACN	QuEChERS	DB5-MS 20m 0.18mm 0.18µm	0.8	Automatic
049	0.01		GC	MS	IT	Thermo Polaris Q	10	ACN	PSA	Capillary	1	Both
050	0.01		GC	MS	Q	Agilent 6890/5973	10	ACN	No	HP-5MS	1	Manual

**APPENDIX 3. Methods used by participants for detecting pesticides.**

PICOLINAFEN												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
001	0.2		GC	MS		AT5975	10	ACN	SPE	HP-5MSi	10	Automatic
003	1	<5ppm	LC	MS	Q-TOF	Waters Xevo QToF	10	Ethyl Acetate	None	Acquity BEH C18	3	Both
006	1.14	11.2	GC	MS	IT	Varian 2000	15	Acetone/DCM/PE	No	VF-5ms	5	Automatic
007	2	19.8	GC	MS	TOF	Pegasus IV	10	ACN	Dispersive SPE with PSA	HP-5MS	5	Both
014	None	None	LC	MS	QqQ	API4000	10	ACN	DSPE	Waters-C18 2.1x50mmx1.7µm	5	Both
017			LC	MS	QqQ	Agilent 6410 Triple Quad	10	ACN	QUEChERS	kinetex C-18	2	Manual
023	0	-0.65%	LC	MS	QqQ	HPLC Agilent 1100. MS API 3000	5	ACN	DSPE	C 18 3µ 50x2mm	10	Both
024	0		LC	MS	QqQ	Waters Xevo	10	Acetone/PE/DCM	Na <sub>2</sub> SO <sub>4</sub>	C18	2	Automatic
026	0.05		LC	MS	QqQ	QuattroUltima. Waters	15	ACN	PSA	C18	10	Manual
029	1220	75	GC	MS	TOF	LECO Pegasus IV	10	ACN	PSA	DB 5	3	Both
030	5	1	GC	MS	Q	Agilent singleQ	10	ACN	PSA	HP-MS 5	20	Both
031	<20	823 (match)	GC	MS	TOF	Leco Pegasus 4D	10	ACN	PSA	RTX-CL pest	10	Both
036	2.5% max	None	LC	MS	MS/MS	Waters Permier XE	10	ACN	No	Acquity UPLC HSS T3	20	Both
037	±12	1.5%	LC	MS/MS	QqQ	API 4000QT	10	ACN	DSP	C31	25	Automatic



**APPENDIX 3. Methods used by participants for detecting pesticides.**

PICOLINAFEN												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
038	0.5	5	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both
039	4.0		GC	MS	Q	HP5975C	10	ACN	PSA	HP5MS	5	Both
040	< 0.2	< 20	GC	MS	Q	Agilent 7890A/5975C	15	ACN	PSA	HP5MS	10	Both
042	0.1	0.05	LC	MS	QqQ	API 3200 QT	10	ACN	DSPE	Synergi Fusion RP 10	20	Both
045	5	0.05 Da	LC	MS	TOF	LCT PREMIER XE	15	ACETONE:PE:DCM	No	C:18	5	Both
046			LC	MS	QqQ	Waters Quattro Premier	10	ACN	DSPE	C18	5	Both
047			GC	MS	TOF	Pegasus III	50	Ethyl Acetate	GPC	VF-5ms(30mx0.25x0.25)	2	Automatic
049	0		LC	MS	QqQ	Waters Aquity UPLC T	10	Methanol	Filter	C18	1	Automatic
050	0.02		GC	MS	Q	Agilent 6890/5973	10	ACN	No	HP-5MS	1	Manual

**APPENDIX 3. Methods used by participants for detecting pesticides.**

<b>PROMETRYN</b>												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
001	2.3		GC	MS		AT5975	10	ACN	SPE	HP-5MSi	10	Automatic
002	< 0.5%	< 0.5%	GC	MS	IT	Varian 4000	10	ACN	PSA	5%	10	Automatic
003	1	<5	LC	MS	Q-TOF	Waters Xevo QToF	10	Ethyl Acetate	None	Acquity BEH C18	3	Both
004	< 2%	<5%	LC	MS	QqQ	API4000	15	ACN	PSA	SpeedRod	20	Manual
005			GC	MS	Q	Agilent 5973N	10	ACN	DSPE	HP5-MS(I)	20	Both
006	2.28	3.1	GC	MS	IT	Varian 2000	15	Acetone/CH <sub>2</sub> Cl <sub>2</sub> /PE	No	VF-5ms	5	Automatic
007	5	9.1	GC	MS	TOF	Pegasus IV	10	ACN	Dispersive SPE with PSA	HP-5MS	5	Both
008	NA	NA	LC	MS	QqQ	API 4000 Qtrap	10	Ethyl Acetate	None	C18	5	Automatic
009	na	na	LC	MS	QqQ	Waters Acquity UPLC system/API 5000 Applied Biosystems	10	Ethyl Acetate	Filter	C18	2	Both
011	+/- 9	30%	GC	MS	Q	Agilent GC 7890 MS 5975	10	ACN	Dispersive SPE	HP-5MS	1	Both
012	1.75	94.0%	GC	MS	Q	AGILENT GC-MSD INERT 5975C	10	ACN	PSA	HP5MS	2	Manual
013			LC	MS	QqQ		10	QuEChERS		C8	20	Both
014	None	None	LC	MS	QqQ	API4000	10	ACN	DSPE	Waters-C18 2.1x50mmx1.7µm	5	Both
015	1.8 s	0.1%	GC	MS	QqQ	Varian 3800 GC+320-MS	10	ACN	Dispersive SPE with PSA + GCB	VF 5ms	3	Both
016	8	2%	GC	MS	Q	Thermo DSQ	10	ACN	DSPE/SPA/C18	Varian VF-5ms	1	Both
017	0.5		GC	MS	Q	Agilent 6890N/5973N	10	ACN	QuEChERS	HP-5	1	Automatic
018	n/a	n/a	LC	MS	QqQ	Agilent	10	ACN	DSPE	C-18	10	Automatic
020	n/a		GC	MS	IT	Varian 4000	10	ACN	DSPE	RTX-200	2	Automatic
021	5	25%	LC	MS	IT	Varian 4000	10	ACN	No	C18	5	Both
023	0	-8.51%	LC	MS	QqQ	Quattro Premier XE	5	ACN	DSPE	C18 3µ 50x2mm	10	Both
024	0		GC	MS	QqQ	HPLC Agilent 1100. MS API 3000	10	Acetone/PE/DCM	Na <sub>2</sub> SO <sub>4</sub>	RXI 5SiI-MS	1	Automatic
025	3.1	1 unit	GC	MS	Q	TSQ Quantum GC	10	ACN	SPE	HP 5 MS	2	Automatic
026		850 out of 1000	GC	MS	Q	Waters MicroGCMSMS	15	ACN	PSA	DB5	4	Manual

**APPENDIX 3. Methods used by participants for detecting pesticides.**

<b>PROMETRYN</b>												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
029	314	100	LC	MS	QqQ	API 4000	10	Acetonitril	PSA	Pursuit XRS Ultra	6	Both
030	5	1	LC	MS	QqQ	API 3200 Qtrap	10	ACN	PSA	Atlantis	10	Both
031	<20	888 (match)	GC	MS	TOF	Leco Pegasus 4D	10	ACN	PSA	RTX-CL pest	10	Both
035	0.1	0.1	LC	MS	QqQ	Thermo TSQ Quantum	10	ACN	no	XTerra	20	Both
036	5% max	None	GC	MS	Q	Agilent MSD 5973	30	Ethyl Acetate	HPGPC	DB5-MS	2	Automatic
037	±12	15%	LC	MS/MS	QqQ	API 4000QT	10	ACN	DSP	C32	25	Automatic
038	0.5	5	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both
039	5.3		GC	MS	Q	HP5975C	10	ACN	PSA	HP5MS	5	Both
040	< 0.2	< 20	LC	MS	QqQ	Waters Aquity TQD	1.5	ACN	PSA	UPLC-BEH C18	10	Both
041	0	0	LC	MS	QqQ	UPLC-Xevo (Waters)	20	Ethyl Acetate	LLE	HSS T3	10	Automatic
042	0.1	0.05	GC	MS	IT	Varian Saturn 4000	10	ACN	DSPE	Varian FV5 ms	1	Both
045	5	0.05 Da	LC	MS	TOF	LCT PREMIER XE	1.5	ACETONE:PE:DCM	No	C:18	5	Both
046			GC	MS	QqQ	Waters Quattro Micro	10	ACN	DSPE	HP5 MS	3	Both
047			GC	MS	TOF	Pegasus III	50	Ethyl Acetate	GPC	VF-5ms(30mx0.25x0.25)	2	Automatic
048	10	5	GC	MS	Q	interscience DSQ	1.5	ACN	QuEChERS	DB5-MS 20m 0.18mm 0.18µm	0.8	Automatic
050	0.02		GC	MS	Q	Agilent 6890/5973	10	ACN	No	HP-5MS	1	Manual

**APPENDIX 3. Methods used by participants for detecting pesticides.**

PROPAGAZINE												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
001	4.2		GC	MS		AT5975	10	ACN	SPE	HP-5MSi	10	Automatic
002	<0.5%	<0.5%	GC	MS	IT	Varian 4000	10	ACN	PSA	5%	10	Automatic
003	1	<5	LC	MS	Q-TOF	Waters Xevo QToF	10	Ethyl Acetate	None	Acquity BEH C18	3	Both
006	1.98	1.4	GC	MS	IT	Varian 2000	15	Acetone/DCM/PE	No	VF-5ms	5	Automatic
007	3	12.8	GC	MS	TOF	Pegasus IV	10	ACN	DSPE with PSA	HP-5MS	5	Both
011	+/- 9	30%	GC	MS	Q	Agilent GC 7890 MS 5975	10	ACN	Dispersive SPE	HP-5MS	1	Both
013			GC	MS	Q		15	Luke	GPC	HP 5	2	Both
014	none	none	LC	MS	QqQ	API4000	10	ACN	DSPE	Waters-C18 2.1x50mmx1.7µm	5	Both
015	1.9 s	0.1%	GC	MS	QqQ	Varian 3800 GC+320-MS	10	ACN	DSPE with PSA + GCB	VF 5ms	3	Both
016	10	1%	GC	MS	Q	Thermo DSQ	10	ACN	DSPE/SPA/C18	Varian VF-5ms	1	Both
017			GC	MS	Q	Agilent 6890N/5973N	10	ACN	QUECHERS	HP-5	1	Automatic
020	n/a	n/a	GC	MS	IT	Varian 4000	10	ACN	DSPE	RTX-200	2	Automatic
023	0.6	-15.21%	LC	MS	QqQ	HPLC Agilent 1100. MS API 3000	5	ACN	DSPE	C18 3µ 50x2mm	10	Both
024	0		GC	MS	Q	Varian Saturn 2000	10	Acetone/PE/DCM	Na <sub>2</sub> SO <sub>4</sub>	DB 5MS UI	25	Automatic
025	2.8	1 unit	GC	MS	Q	Agilent 5973	10	ACN	SPE	HP 5 MS	2	Automatic
026		6.2	LC	MS	Q-TOF	Brucker	15	ACN	PSA	C18	5	Manual
029	275	100	LC	MS	QqQ	API 4000	10	Acetonitrile	PSA	Pursuit XRS Ultra	6	Both
030	5	1	LC	MS	QqQ	API 3200 Qtrap	10	ACN	PSA	Atlantis	10	Both

**APPENDIX 3. Methods used by participants for detecting pesticides.**

PROPAGAZINE												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
031	<20	940 (match)	GC	MS	TOF	Leco Pegasus 4D	10	ACN	PSA	RTX-CL pest	10	Both
035	0.1	0.1	LC	MS	QqQ	Thermo TSQ Quantum	10	ACN	no	Xterra	20	Both
036	5% max	none	GC	MS	Q	Agilent MSD 5973	30	Ethyl Acetate	HPGPC	DB5-MS	2	Automatic
037	±12	15%	LC	MS/MS	QqQ	API 4000GT	10	ACN	DSP	C33	25	Automatic
038	0.5	5	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both
039	6.4		GC	MS	Q	HP5975C	10	ACN	PSA	HP5MS	5	Both
040	< 0.2	< 20	LC	MS	QqQ	Waters Aquity TQD	15	ACN	PSA	UPLC-BEH C18	10	Both
041	0	0	LC	MS	QqQ	UPLC-Xevo (Waters)	20	Ethyl Acetate	LLE	HSS T3	10	Automatic
042	0.1	0.05	GC	MS	IT	Varian Saturn 4000	10	ACN	DSPE	Varian FV5	1	Both
045	5	0.05 Da	LC	MS	TOF	LCT PREMIER XE	15	Acetone/PE/DCM	NO	C:18	5	Both
046			GC	MS	Q	Agilent MSD 5973	10	ACN	DSPE	HP5 MS	5	Both
047			GC	MS	TOF	Pegasus III	50	Ethyl Acetate	GPC	VF-5ms(30mx0.25x0.25)	2	Automatic
048	10	5	GC	MS	Q	interscience DSQ	15	ACN	QuEChERS	DB5-MS 20m 0.18mm 0.18µm	0.8	Automatic
050	0.02		GC	MS	Q	Agilent 6890/5973	10	ACN	no	HP-5MS	1	Manual

APPENDIX 3. Methods used by participants for detecting pesticides.

PROPOXUR												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
001	0.3		GC	MS		AT5975	10	ACN	SPE	HP-5MSi	10	Automatic
002	< 2.5%	< 2.5%	LC	MS	QqQ	3200 Qtrap	10	ACN	PSA	C18 2.5 µm - 5 cm	50	Automatic
003	1	<5	LC	MS	Q-TOF	Waters Xevo QToF	10	Ethyl Acetate	None	Acquity BEH C18	3	Both
004	< 2%	<5%	GC	MS	QqQ	Agilent 7000A	15	Acetone/PE/DCM	None	HP5MS	2	Both
005			LC	MS	QqQ	Agilent 6410B	10	ACN	DSPE	C18. 1.8µm	2	Automatic
006	1.2	1.0	LC	MS	QqQ	Waters Premier XE	15	Acetone/DCM/PE	No	Acquity BEH	5	Automatic
007	1	12.6	GC	MS	TOF	Pegasus IV	10	ACN	Dispersive SPE with PSA	HP-5MS	5	Both
008	NA	NA	LC	MS	QqQ	API 4000 Qtrap	10	Ethyl Acetate	None	C18	5	Automatic
009	na	na	LC	MS	QqQ	Waters Aquity UPLC system/API 5000 Applied Biosystems	10	Ethyl Acetate	Filter	C18	2	Both
011	+/- 9	30%	GC	MS	Q	Agilent GC 7890 MS 5975	10	ACN	Dispersive SPE	HP-5MS	1	Both
012	0.6	105.84%	LC	MS	QqQ	AGILENT LC-MSMS 6410	10	ACN	PSA	C18	20	Manual
013			LC	MS	QqQ		10	QuEChERS		C8	20	Both
015	4.3 s	0.1%	LC	MS	QqQ	Varian Pro Star + 320-MS	10	ACN	Dispersive SPE with PSA + GCB	ChromSep C-18	20	Both
016	4	4%	GC	MS	Q	Thermo DSQ	10	ACN	dSPE/SPA/ C18	Varian VF-5ms	1	Both
017			GC	MS	Q	Agilent 6890N/5973N	10	ACN	QuEChERS	HP-5	1	Automatic
019	30	30%	GC	MS	IT	Varian 2000 Ion Trap	15	Ethyl Acetate		DB 5 MS	10	Automatic
020	n/a	n/a	GC	MS	IT	Varian 4000	10	ACN	DSPE	RTX-200	2	Automatic
021	5	25%	LC	MS	QqQ	Quattro Premier XE	10	ACN	No	C18	5	Both
023	-0.15	7.30%	LC	MS	QqQ	HPLC Agilent 1100. MS API 3000	5	ACN	DSPE	C18 3µ 50x2mm	10	Both
024	0		GC	MS	QqQ	TSQ Quantum GC	10	Acetone/PE/DCM	Na <sub>2</sub> SO <sub>4</sub>	RXi 5SiH-MS	1	Automatic
025	1.0	1 unit	GC	MS	Q	Agilent 5973	10	ACN	SPE	HP 5 MS	2	Automatic
026	0.05		LC	MS	QqQ	QuattroUltima. Waters	15	ACN	PSA	C18	10	Manual
029	206	100	LC	MS	QqQ	API 4000	10	ACN	PSA	Pursuit XRS Ultra	6	Both
030	5	1	LC	MS	QqQ	API 3200 Qtrap	10	ACN	PSA	Altantis	10	Both
031	<20	877 (match)	GC	MS	TOF	Leco Pegasus 4D	10	ACN	PSA	RTX-CL pest	10	Both

APPENDIX 3. Methods used by participants for detecting pesticides.

PROPOXUR												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
035	0.1	0.1	LC	MS	QqQ	Thermo TSQ Quantum	10	ACN	No	XTerra	20	Both
036	3	5 max	LC	MS	TOF	Agilent	10	ACN	No	Zorbax Eclipse plus	3	Both
037	±12	15%	LC	MS/MS	QqQ	API 4000QT	10	ACN	DSP	C34	25	Automatic
038	0.5	5	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both
039			LC	MS	QqQ	ABI 4000	10	ACN	No	C18	55	Both
040	<0.2	<20	LC	MS	QqQ	Waters Aquity TQD	15	ACN	PSA	UPLC-BEH C18	10	Both
041	0	0	LC	MS	QqQ	UPLC-Xevo (Waters)	20	Ethyl Acetate	LLE	HSS T3	10	Automatic
042	0.1	0.05	LC	MS	QqQ	API 3200 QT	10	ACN	DSPE	Synergi Fusion RP 10	20	Both
044	0	0	GC	MS	Q	Agilent 5973	50	Acetone	No	HP 5 MS	1	Automatic
045	5	0.05 Da	LC	MS	TOF	LCT PREMIER XE	15	ACETONE:PE::DCM	No	C:18	5	Both
046			GC	MS	QqQ	Waters Quattro Micro	10	ACN	DSPE	HP5 MS	3	Both
047	No	No	GC	MS	TOF	Pegasus III	50	Ethyl Acetate	GPC	VF-5ms(30mx0.25x0.25)	2	Automatic
048	10	5	GC and LC	MS and QqQ	Q and QqQ	interscience DSQ and Micromass Quattro premier	15	ACN	QUECHERS	DB5-MS 20m 0.18mm 0.18µm for GC and UPLC BEH C 18 1.7µ 2.1mm 100mm	3 LC 0.8 GC	Automatic
049	0		LC	MS	QqQ	Waters Aquity UPLC T	10	Methanol	Filter	C18	1	Automatic
050	0		LC	MS	QqQ	Agilent 6400	10	ACN	No	Eclipse XDB-C18	4	Manual

**APPENDIX 3. Methods used by participants for detecting pesticides.**

PYRACLOSTROBIN												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
001	0.5		GC	MS		AT5975	10	ACN	SPE	HP-5MSi	10	Automatic
002	< 0.5%	< 0.5%	GC	MS	IT	Varian 4000	10	ACN	PSA	5%	10	Automatic
003	1	<5	LC	MS	Q-TOF	Waters Xevo QToF	10	Ethyl Acetate	None	Acquity BEH C18	3	Both
004	< 2%	<5%	LC	MS	QqQ	API4000	15	ACN	PSA	SpeedRod	20	Manual
005			LC	MS	QqQ	Agilent 6410B	10	ACN	DSPE	C18, 1.8µm	2	Automatic
006	2.25	4.2	LC	MS	QqQ	Waters Premier XE	15	Acetone/DCM/PE	No	Acquity BEH	5	Automatic
007	1	6.7	GC	MS	TOF	Pegasus IV	10	ACN	DSPE with PSA	HP-5MS	5	Both
008	NA	NA	LC	MS	QqQ	API 4000 Gtrap	10	Ethyl Acetate	None	C18	5	Automatic
009	na	na	LC	MS	QqQ	Waters Acquity UPLC system/API 5000 Applied Biosystems	10	Ethyl Acetate	Filter	C18	2	Both
012	1.8	99.15%	LC	MS	QqQ	AGILENT LC-MSMS 6410	10	ACN	PSA	C18	20	Manual
013			GC	MS	Q		15	Luke	GPC	HP 5	2	Both
014	none	none	LC	MS	QqQ	API4000	10	ACN	DSPE	Waters-C18 2.1x50mmx1.7µm	5	Both
015	3.5 s	0.1%	LC	MS	QqQ	Varian Pro Star +320-MS	10	ACN	DSPE with PSA + GCB	ChromSep C-18	20	Both
016			LC	MS	QqQ	API 4000	10	ACN	DSPE/SFA/C18	RP-AMIDE	10	Both
017			LC	MS	QqQ	Agilent 6410 Triple Quad	10	ACN	QuEChERS	kinetex C-18	2	Manual
018	0.5		LC	MS	QqQ	Agilent	10	ACN	DSPE	C-18	10	Automatic
021	5	25%	LC	MS	QqQ	Quattro Premier XE	10	ACN	No	C18	5	Both
022			LC	MS	QqQ	API4000	10	ACN	PSA	C18ec	8	Manual
023	0.3	2.88%	LC	MS	QqQ	HPLC Agilent 1100. MS API 3000	5	ACN	DSPE	C18 3µ 50x2mm	10	Both
024	0		GC	MS	QqQ	TSQ Quantum GC	10	Acetone/PE/DCM	Na <sub>2</sub> SO <sub>4</sub>	RXi 5Sil-MS	1	Automatic
026	0.05		LC	MS	QqQ	QuattroUltima. Waters	15	ACN	PSA	C18	10	Manual
029	364	100	LC	MS	QqQ	API 4000	10	ACN	PSA	Pursuit XRS Ultra	6	Both



**APPENDIX 3. Methods used by participants for detecting pesticides.**

PYRACLOSTROBIN												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
030	5	1	LC	MS	QqQ	API 3200 Qtrap	10	ACN	PSA	Atlantis	10	Both
031	<20	933 (match)	GC	MS	TOF	Leco Pegasus 4D	10	ACN	PSA	RTX-CL pest	10	Both
035	0.1	0.1	LC	MS	QqQ	Thermo TSQ Quantum	10	ACN	No	XTerra	20	Both
036	4	5 max	LC	MS	TOF	Agilent	10	ACN	No	Zorbax Eclipse plus	3	Both
037	±12	15%	LC	MS/MS	QqQ	API 4000QT	10	ACN	DSP	C35	25	Automatic
038	0.5	5	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both
039			LC	MS	QqQ	ABI 4000	10	ACN	No	C18	55	Both
040	<0.2	<20	LC	MS	QqQ	Waters Aquity TQD	15	ACN	PSA	UPLC-BEH C18	10	Both
041	0	0	LC	MS	QqQ	UPLC-Xevo (Waters)	20	Ethyl Acetate	LLE	HSS T3	10	Automatic
042	0.1	0.05	LC	MS	QqQ	API 3200 QT	10	ACN	DSPE	Synergi Fusion RP 10	20	Both
046			LC	MS	QqQ	Waters Quattro Premier	10	ACN	DSPE	C18	5	Both
047			GC	MS	TOF	Pegasus III	50	Ethyl Acetate	GPC	VF-5ms(30mx0.25x0.25)	2	Automatic
048	10	5	LC	LC/MSMS	QqQ	Micromass Quattro premier	15	ACN	QuEChERS	UPLC BEH C18 1.7µm 2.1mm 100mm	3	Automatic
049	0		LC	MS	QqQ	Waters Acquity UPLC T	10	Methanol	Filter	C18	1	Automatic
050	0.01		GC	MS	Q	Agilent 6890/5973	10	ACN	No	HP-5MS	1	Manual

**APPENDIX 3. Methods used by participants for detecting pesticides.**

<b>QUINOCLOLAMINE</b>												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
001	5.6		GC	MS		AT5975	10	ACN	SPE	HP-5MSi	10	Automatic
007	6	29.7	GC	MS	TOF	Pegasus IV	10	ACN	Dispersive SPE with PSA	HP-5MS	5	Both
011	+/- 9	30%	GC	MS	Q	Agilent GC 7890 MS 5975	10	ACN	Dispersive SPE	HP-5MS	1	Both
014	none	none	LC	MS	QqQ	API4000	10	ACN	DSPE	Waters-C18 2.1x50mmx1.7µm	5	Both
016	18	6%	GC	MS	Q	Thermo DSQ	10	ACN	DSPE/SPA/C18	Varian VF-5ms	1	Both
017			GC	MS	Q	Agilent 6890N/5973N	10	ACN	QuEChERS	HP-5	1	Automatic
023	0	2.00%	GC	MS	Q	GC Shimadzu GC-2010. MS Shimadzu GCMS-QP2010	5	ACN	DSPE	HP-5MS	3	Both
025	1.3	1 unit	GC	MS	Q	Agilent 5973	10	ACN	SPE	HP 5 MS	2	Automatic
029	776	72	GC	MS	TOF	LECO Pegasus IV	10	ACN	PSA	DB 5	3	Both
030	5	1	LC	MS	QqQ	API 3200 Qtrap	10	ACN	PSA	Atlantis	10	Both
031	na	869 (match)	GC	MS	TOF	Leco Pegasus 4D	10	ACN	PSA	RTX-CL pest	10	Both
036	5% max	none	GC	MS	Q	Agilent MSD 5973	30	Ethyl Acetate	HPGPC	DB5-MS	2	Automatic
038	0.5	5	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both
039			LC	MS	QqQ	ABI 4000	10	ACN	No	C18	55	Both
046			GC	MS	Q	Agilent MSD 5973	10	ACN	DSPE	HP5 MS	5	Both

**APPENDIX 3. Methods used by participants for detecting pesticides.**

SIMAZINE												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
001	5.7		GC	MS		AT5975	10	ACN	SPE	HP-5MSi	10	Automatic
002	< 0.5%	< 0.5%	GC	MS	IT	Varian 4000	10	ACN	psa	5%	10	Automatic
003	1	<5	LC	MS	Q-TOF	Waters Xevo QToF	10	Ethyl Acetate	none	Acquity BEH C18	3	Both
004	< 2%	<5%	GC	MS	QqQ	Agilent 7000A	1.5	Acetone/PE/DCM	None	HP5MS	2	Both
005			GC	MS	Q	Agilent 5973N	10	ACN	DSPE	HP5-MS(I)	20	Both
006	2.22	3.1	GC	MS	IT	Varian 2000	15	Acetone/CH <sub>2</sub> Cl <sub>2</sub> /PE	no	VF-5ms	5	Automatic
007	3	17.0	GC	MS	TOF	Pegasus IV	10	ACN	Dispersive SPE with PSA	HP-5MS	5	Both
008	NA	NA	LC	MS	QqQ	API 4000 Qtrap	10	Ethyl Acetate	none	C 18	5	Automatic
009	na	na	LC	MS	QqQ	Waters Acquity UPLC system/API 5000 Applied Biosystems	10	Ethyl Acetate	Filter	C 18	2	Both
011	+/- 9	30%	GC	MS	Q	Agilent GC 7890 MS 5975	10	ACN	Dispersive SPE	HP-5MS	1	Both
012	0.75	92.5%	GC	MS	Q	AGILENT GC-MSD INERT 5975C	10	ACN	PSA	HP5MS	2	Manual
013			GC	MS	Q		15	Luke	GPC	HP 5	2	Both
014	none	none	LC	MS	QqQ	API4000	10	ACN	DSPE	Waters-C18 2.1x50mmx1.7µm	5	Both
015	2.0 s	0.1%	GC	MS	QqQ	Varian 3800 GC+ 320-MS	10	ACN	Dispersive SPE with PSA + GCB	VF 5ms	3	Both
016			LC	MS	QqQ	API 4000	10	ACN	DSPE/SPA/C18	Supelco Ascentis Express RP-AMIDE	10	Both
017			GC	MS	Q	Agilent 6890N/5973N	10	ACN	QUECHERS	HP-5	1	Automatic

**APPENDIX 3. Methods used by participants for detecting pesticides.**

SIMAZINE												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
018	0.5		LC	MS	QqQ	Agilent	10	ACN	DSPE	C-18	10	Automatic
020	n/a	n/a	GC	MS	IT	Varian 4000	10	ACN	DSPE	RTX-200	2	Automatic
021	5	25%	LC	MS	QqQ	Quattro Premier XE	10	ACN	No	C18	5	Both
022	6		LC	MS	QqQ	API4000	10	ACN	PSA	C18ec	8	Manual
023	0	-0.91%	LC	MS	QqQ	HPLC Agilent 1100, MS API 3000	5	ACN	DSPE	C18 3µ 50x2mm	10	Both
024	0		GC	MS	QqQ	TSQ Quantum GC	10	Acetone/PE/DCM	Na <sub>2</sub> SO <sub>4</sub>	RX1 5SiI-MS	1	Automatic
025	2.5	1 unit	GC	MS	Q	Agilent 5973	10	ACN	SPE	HP 5 MS	2	Automatic
026		6.2	LC	MS	Q-TOF	Brucker	15	ACN	PSA	C18	5	Manual
029	633	92	GC	MS	TOF	LECO Pegasus IV	10	ACN	PSA	DB 5	3	Both
030	5	1	GC	MS	Q	Agilent singleQ	10	ACN	PSA	HP-MS 5	20	Automatic
031	<20	940 (match)	GC	MS	TOF	Leco Pegasus 4D	10	ACN	PSA	RTX-CL pest	10	Both
035	0.1	0.1	LC	MS	QqQ	Thermo TSQ Quantum	10	ACN	no	Xterra	20	Both
036	5% max	none	GC	MS	Q	Agilent MSD 5973	30	Ethyl Acetate	HPGPC	DB5-MS	2	Automatic
037	±12	15%	LC	MS/MS	QqQ	API 4000QT	10	ACN	DSP	C36	25	Automatic
038	0.5	5	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both
039			LC	MS	QqQ	ABI 4000	10	ACN	No	C18	55	Both
040	< 0.2	< 20	LC	MS	QqQ	Waters Aquity TQD	15	ACN	PSA	UPLC-BEH C18	10	Both
041	0	0	LC	MS	QqQ	UPLC-Xevo (Waters)	20	Ethyl Acetate	LLE	HSS T3	10	Automatic
042	0.1	0.05	GC	MS	IT	Varian Saturn 4000	10	ACN	DSPE	Varian FV5 ms	1	Both

**APPENDIX 3. Methods used by participants for detecting pesticides.**

<b>SIMAZINE</b>												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
045	5	0.05 Da	LC	MS	TOF	LCT PREMIER XE	15	ACETONE:PE:DCM	NO	C:18	5	Both
046			LC	MS	QqQ	Waters Quattro Premier	10	ACN	DSPE	C18	5	Both
047			GC	MS	TOF	Pegasus III	50	Ethyl Acetate	GPC	VF-5ms (30mx0.25x0.25)	2	Automatic
049	0		LC	MS	QqQ	Waters Acquity UPLC T	10	Methanol	Filter	C18	1	Automatic
050	0.01		GC	MS	Q	Agilent 6890/5973	10	ACN	no	HP-5MS	1	Manual

**APPENDIX 3. Methods used by participants for detecting pesticides.**

CARBOFURAN (Present in the sample but not added)												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
001	1.4		GC	MS		AT5975	10	ACN	SPE	HP-5MSi	10	Automatic
002	< 0.5%	< 0.5%	GC	MS	IT	Varian 4000	10	ACN	PSA	5%	10	Automatic
003	1	<5	LC	MS	Q-TOF	Waters Xevo QToF	10	Ethyl Acetate	None	Acquity BEH C18	3	Both
004	< 2%	<5%	LC	MS	GqQ	API4000	15	ACN	PSA	SpeedRod	20	Manual
005			LC	MS	GqQ	Agilent 6410B	10	ACN	DSPE	C18, 1.8µm	2	Automatic
006	2.40	12.7	LC	MS	GqQ	Waters Premier XE	15	Acetone/DCM/PE	No	Acquity BEH	5	Automatic
007	0.3	0	LC	MS	GqQ	Agilent 6410	10	ACN	Dispersive SPE with PSA	Luna C18	5	Both
009	na	na	LC	MS	GqQ	Waters Acquity UPLC system/API 5000 Applied Biosystems	10	Ethyl Acetate	Filter	C18	2	Both
011	+/- 9	30%	GC	MS	Q	Agilent GC 7890 MS 5975	10	ACN	Dispersive SPE	HP-5MS	1	Both
012	1.2	101.84%	LC	MS	GqQ	AGILENT LC-MSMS 6410	10	ACN	PSA	C18	20	Manual
013			LC	MS	GqQ		10	QuEChERS		C8	20	Manual
014	none	none	LC	MS	GqQ	API4000	10	ACN	DSPE	Waters-C18 2.1x50mmx1.7µm	5	Both
015	1.6 s	0.1%	LC	MS	GqQ	Varian Pro Star +320-MS	10	ACN	DSPE with PSA + GCB	ChromSep C-18	20	Both
016			LC	MS	GqQ	API 4000	10	ACN	DSPE/SPA/C18	RP-AMIDE	10	Both
017			GC	MS	Q	Agilent 6890N/5973N	10	ACN	QuEChERS	HP-5	1	Automatic
019	30	30%	GC	MS	IT	Varian 2000 Ion Trap	15	Ethyl Acetate		DB 5 MS	10	Automatic
020	n/a	n/a	GC	MS	IT	Varian 4000	10	ACN	DSPE	RTX-200	2	Automatic

**APPENDIX 3. Methods used by participants for detecting pesticides.**

CARBOFURAN (Present in the sample but not added)												
Laboratory Code	RT Tolerance	MS Tolerance	Chromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
021	5	25%	LC	MS	GqQ	Quattro Premier XE	10	ACN	No	C18	5	Both
022	6		LC	MS	GqQ	API4000	10	ACN	PSA	C18ec	8	Manual
023	0	2.20%	LC	MS	GqQ	HPLC Agilent 1100. MS API 3000	5	ACN	DSPE	C18 3µ 50x2mm	10	Both
024	0		LC	MS	GqQ	Waters Xevo	10	Acetone/PE/DCM	Na <sub>2</sub> SO <sub>4</sub>	C18	2	Automatic
025	1.8	1 unit	GC	MS	Q	Agilent 5973	10	ACN	SPE	HP 5 MS	2	Automatic
029	223	100	LC	MS	GqQ	API 2000	10	ACN	PSA	C. 18.Synergi Fusion	7	Both
030	5	1	LC	MS	GqQ	API 3200 Qtrap	10	ACN	PSA	Atlantis	10	Both
031	3.6	0.8	LC	MS	Orbitrap	Exactive	10	ACN		Atlantis	5	Both
033			LC	MS	GqQ	Applied Biosystem	10	ACN	PSA/MgSO <sub>4</sub> /GCB	Atlantis T3 100*2.1mm	5	Manual
035	0.1	0.1	LC	MS	GqQ	Thermo TSQ Quantum	10	ACN	No	XTerra	20	Both
036	2	5 max	LC	MS	TOF	Agilent	10	ACN	No	Zorbax Eclipse plus	3	Both
037	±12	15%	LC	MS/MS	GqQ	API 4000QT	10	ACN	DSP	C18	25	Automatic
038	0.5	5	LC	MS	Orbitrap	Exactive	10	ACN	PSA	C18	5	Both
039			LC	MS	GqQ	ABI 4000	10	ACN	No	C18	55	Both
040	<0.2	< 20	LC	MS	GqQ	Waters Aquity TQD	15	ACN	PSA	UPLC-BEH C18	10	Both
041	0	0	LC	MS	GqQ	UPLC-Xevo (Waters)	20	Ethyl Acetate	LLC	HSS T3	10	Automatic
042	0.1	0.05	LC	MS	GqQ	API 3200 QT	10	ACN	DSPE	Synergi Fusion RP 10	20	Both
044	0	0	GC	MS	Q	Agilent 5973	50	Acetone	No	HP 5 MS	1	Automatic
044	0	0	LC	MS	GqQ	Varian MS 320	10	ACN	Yes	C 18	10	Automatic
046			LC	MS	GqQ	Waters Quattro Premier	10	ACN	DSPE	C18	5	Both

**APPENDIX 3. Methods used by participants for detecting pesticides.**

<b>CARBOFURAN</b>												
(Present in the sample but not added)												
Laboratory Code	RT Tolerance	MS Tolerance	Cromatographic Technique	Detector	Analyser	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean-Up	Column Type	Injection Volume (µl)	Software
048	10	5	GC and LC	MS	Q and GdQ	interscience DSQ and Micromass Quatro premier	15	ACN	QuEChERS	DB5-MS 20m 0.18mm 0.18µm for GC and UPLC BEH C18 1.7µ 2.1mm 100mm	3 LC 0.8 GC	Automatic
049	0.01		GC	MS	GdQ	Thermo TSQ	10	ACN	PSA	Capillary	1	Both
050	0		LC	MS	GdQ	Agilent 6400	10	ACN	No	Eclipse XDB-C18	4	Manual



# Protocol

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## Introduction:

Last year the first pesticide screening method inter-laboratory test was well accepted by laboratories. Therefore the EURL-FV has decided to continue this year with a second test. The main reason for this broad acceptance comes from the fact that many laboratories have recently invested in new higher mass accuracy MS systems that allow them to greatly increase the scope of their multiresidue methods.

From last year it was found that many laboratories not only used a full scan approach to perform screening but some also employed new tandem-mass spectrometers, even if the sensitivity had to be reduced.

Another reason for conducting this screening method proficiency test is to gain information from the laboratories on the type of software that they use. Whether they are using commercial software and databases, or whether they are internally constructed and searched manually. This information will provide an overall view of the purpose of this type of test, and if, in a near future, there is the need to develop new software.

The EURL-FV aim is to be able to use mass spectrometry based screening methods routinely, following validation. This is in line with the new Document SANCO/10684/2009 ("Method validation and quality control procedures for pesticide residues analysis in food and feed")

As for last year, only qualitative information will be requested for those pesticides that are detected. It has been decided by the Quality Control Group, and based on the received questionnaires, that a target pesticide list will not be provided. The pesticides selected to treat the test material for this EUPT-FV-SM02 will take into account the following considerations:

- Pesticides that are not included in the Coordinated Multiannual Community Control Programme for 2010 (Regulation (EC) 901/2009).
- Pesticides that are particularly acutely toxic and/or have low ARfD values.

Regulation (EC) No 882/2004 (Regulation (EC) No 396/2005, published at OJ of the EU L70 of 16.03.2005, and last amended by Regulation 839/2008 published at OJ of the EU L234 of 30.08.2008) lays down the general tasks, duties and requirements for EU-RLs for Food, Feed and Animal Health. Among these tasks is the provision of independently organised comparative tests. As we did last year the EURL for pesticides in Fruit and Vegetables at the University of Almería, Spain, is going to organise a proficiency test on qualitative screening methods for pesticides in vegetable/fruit commodities. This EUPT-SM-02 is directed at all National Reference Laboratories (NRLs) and all Official Laboratories (OfLs) in the EU Member States for Fruits and Vegetables. Laboratories outside this EURL/NRL/OfL-Network may also be allowed to participate on a case-by-case basis, after consultation with DG SANCO.

## Test material

This proficiency test is based on pesticide residues detection in leeks. The leeks to be used in this study were grown in Catalunya, Spain.

The pesticide treatments on the leeks will be carried out post-harvest by spiking with a mixture of standard solutions. The leeks will then be frozen (using liquid nitrogen), chopped, homogenized and sub-sampled into polyethylene bottles that have previously been coded.

Ten of these bottles containing the prepared test material, will be chosen randomly, and analysed to check the presence of all the spiked pesticides.

## **ANNEX 1. Protocol, Instructions and Forms.**

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 participants' results). There will be an extra analysis during this period after the test material has been maintained at room temperature for a few days, again to see if there has been any degradation of any of the pesticides.

### **Steps to follow**

This Proficiency Test will be made up of the following 6 essential steps:

1. To participate, each laboratory must complete the Application Form on-line, available on the CRL-FV Web page, before the deadline stipulated on the Calendar.
2. Laboratories will then receive an e-mail confirming their participation in this exercise, and assigning them a Laboratory Code. Laboratories with this information will be able to access the restricted area using their login information to be able to fill in the response forms - consisting of their USER NAME which is the Laboratory Code expressed as Labxxx (three digits with no spaces between them) and their PASSWORD, as chosen on the application form.
3. Test material delivery will cost 150 Euros, except for those Laboratories already participating in EUPT-FV12, which will not be charged. If participation is just in this EUPT-FV-SM-02, then the payment procedure must have started before the 5<sup>th</sup> April. An e-mail showing a bank transfer confirmation, or similar, must have been sent beforehand. Payments without a Laboratory Code or an Invoice Number to identify them will not be considered as paid.
4. Immediately after participant laboratories have received the test material, they must enter the restricted area and submit the Sample Receipt Form on-line to inform the Organiser that they have accepted the test material. If the Form is sent, then the Organiser will conclude that the test material has been accepted. If no test material has been received by the 14<sup>th</sup> April 2010, please contact the Organiser by e-mail (pmedina@ual.es or omalato@ual.es)
5. The participant laboratories must respect the deadline for submitting the results - 72 hours after the test material reception - using the 'Results Form' on-line. Note that in this EUPT there will only be one Form for results submission.
6. The Organiser will evaluate the results at the end of the proficiency test, once the deadline for receipt of results has passed. The Organiser will send a hard copy of the Final Report to each participant laboratory. Before this, an electronic version will be uploaded on the CRL-FV web site. This report will include information regarding the design of the test, the evaluation of the participant's results as well as graphical displays of the results and any conclusions. Other relevant information considered of value may also be included.

### **Amount of Test material**

Participants will receive:

- Approximately 300 g of leek test material with spiked pesticides, labelled as EUPT-FV-SM-02 sample
- Approximately 300 g of 'blank' leek test material. Note: the 'blank' sample will be the same if the laboratory also participated in EUPT-FV12.

### **Shipment of Test materials**

All test materials will be frozen and packed in polyethylene boxes surrounded with dry ice and packed in boxes.

The shipment of the test materials will be carried out on a single day (12<sup>th</sup> April 2010). An information message will be sent out by e-mail before shipment. Laboratories must make their own arrangements for the reception of the package. They must inform the Organiser of any public holidays in their country/city during the delivery period given in the calendar, as well as making the necessary arrangements to receive the shipment, even if the laboratory is closed.

**Advice on Test material Handling**

The test material should be mixed thoroughly, before taking the analytical portion(s).

All participants should use their own routine standard operating procedures for extraction, clean-up and analytical determination using their own reference standards for identification. No quantification is required in this test.

**Sample Receipt – Form 0**

Immediately after the laboratory has received the test materials it must be reported to the organiser via Form 0, or the Sample Receipt Form, by accessing the restricted area found at <http://www.crl-pesticides.eu> by filling in the date of receipt, the condition of the test material, and its acceptance. If the laboratory does not respond by sending this Form, the organiser will assume that the test material has been received and accepted.

If any laboratory has not received the test material by 14<sup>th</sup> April, they must inform the Organiser immediately by e-mail ([pmedina@ual.es](mailto:pmedina@ual.es) or [omalato@ual.es](mailto:omalato@ual.es))

**Result Submission – Form 1**

Once the laboratory has analysed the test material and is ready to submit their data, they must enter their results by accessing the private area in the CRL –FV web site: <http://www.crl-pesticides.eu>

As there is not a Target Pesticide List for this PT, the laboratory will have to enter the name of the pesticides that they detected and relative information concerning the procedure for detecting it.

**Calendar:**

Activity	Date
Publish the Calendar and Matrix on the Web page	December 2009
Receipt of Application Form from invited laboratories	17 <sup>th</sup> March 2010
Specific Protocol published on the Web site	29 <sup>th</sup> March 2010, at the latest
Sample distribution	12 <sup>th</sup> April 2010
Deadline for informing organiser of test material acceptance: Fill in Form 0 "Sample receipt"	As soon as received
Deadline for receipt of results: Fill in Forms 1 – 'Results'	72 hours after receipt of the sample
Preliminary Report: only results.	July 2010
Final Report distributed to the Laboratories.	December 2010

**Confidentiality:**

The results of this test will only be made known to the participants by the Organiser. Each participating laboratory will be presented as a lab code to the Commission or at a Workshop.

**Communication:**

The official language used will be English. Communication between participating laboratories during the test on matters concerning the test is not permitted.

## ANNEX 1. Protocol, Instructions and Forms.

### Evaluation of the Results:

The procedures used for the evaluation of results will be based mainly around false negatives and false positives. After receiving the results, the Organiser may consider further evaluation that could highlight important information received. Therefore:

- *False Positives*: these will be considered as those results that show the apparent presence of pesticides which were: (i) not used in the test material treatment, (ii) not detected by the Organiser, even after repeated analyses. However, if a number of participants do detect the same additional pesticide(s), 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.
- *False Negatives*: these will be considered as the absence of result for any present pesticide not reported by the lab, even though the Organiser has used it to spike the test material, and it was detected by the majority of participants.

### Contact information

The official organising group details are as follows:

Universidad de Almería. Edificio Químicas CITE I  
Ctra. Sacramento s/n  
04120 Almería - Spain  
Fax No.: +34 950015483

### Organising team (e-mail and phone no.):

Amadeo R. Fernández-Alba.	CRL-FV	amadeo@ual.es	+34 950015034
Paula Medina Pastor.	CRL-FV	pmedina@ual.es	+34 950014102
Octavio Malato Rodríguez.	CRL-FV	omalato@ual.es	+34 950015531

### Quality Control Group:

Dr. Antonio Valverde	University of Almería, Spain.
Mr. Arne Andersson	Head of Division NFA, Uppsala, Sweden – with great sorrow and hoping for a prompt recovery
Mr. Stewart Reynolds,	Senior Chemist from FERA, York, United Kingdom, has taken his place.

### Statistical Group:

Dr. Carmelo Rodriguez, senior Mathematics.	University of Almeria, Spain
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### Advisory Group:

Dr. Miguel Gamón, senior Chemist	Laboratorio Agroalimentario, Valencia, Spain.
Dr. Tuija Pihlström, senior Chemist	NFA, Uppsala, Sweden.
Dr. André de Kok, senior Chemist	VWA, Amsterdam, The Netherlands.
Dr. Sonja Masselter, senior Chemist,	AGES, Innsbruck, Austria
Dr. Michelangelo Anastasiades, senior Chemist	CVUA, Stuttgart, Germany.
Dr. Metter Erecius Poulsen, senior Chemist	NFI, Copenhagen, Denmark.
Dr. Ralf Lippold, senior Chemist	CVUA, Freiburg, Germany.

## Application Form

The deadline for the Application Form is  
17th March 2010.  
If you need further information or have any comment or question, please send an e-mail to [pmedina@ual.es](mailto:pmedina@ual.es) or [omalato@ual.es](mailto:omalato@ual.es)

**General Data**

**NRL in FV**  Yes  No

**No of Analysis by MRM done last year**

**No EU Official Samples Received last year from Coordinated Programme**

**Country**

**Laboratory Data**

**Laboratory name**

**Laboratory Address**

**Contact Name**

**Street**

**Number**  **Postal code**  **City**

**Postal Address (Optional fields. Only fill in if the postal address is diferent from the delivery address)**

**Street**

**Number**  **Postal code**  **City**  **Country**

**Telephone including country code**

**Fax Including country code**

**Mobile (Optional field)**

**Email**

**Register Data**  
(Remember that your username will be your Lab code that will be sent to you once you have been accepted by the organization and here you have to choose your password)

**Date**

**Choose your password**

**Receive standard solvent solution**  Yes  No

**Payment agreement**  Yes  No

**Invoice type**  Invoice  Receipt  Nothing

**Invoice Laboratory Address**

**Vat number**  **Laboratory name**

**Street**

**Number**  **Postal code**  **City**  **Country**

**Invoice Postal Address (Optional fields. Only fill in if the postal address is diferent from the delivery address)**

**Laboratory name**



**Street**

**Number**  **Postal code**  **City**  **Country**

If you have any problem filling the forms please contact with Octavio Malato: [omalato@ual.es](mailto:omalato@ual.es) (+34) 950 015 531  
If you have any doubt about the required fields please contact with Paula Medina: [pmedina@ual.es](mailto:pmedina@ual.es) (+34) 950 015 645

Form 0 (Sample Receipt)

**Community Reference Laboratories for Residues of Pesticides**  
Pesticides in Fruits and Vegetables



### Sample Receipt EUPT-FV-SM 02

Please fill in the form as soon as possible after receiving the sample. If not the Organiser will understand you have accepted it.

[Back to Main page](#) | [Save this page](#)

Lab code: **Lab XXX**

Contact name: **Name XXXXXX**

Sample number:

Blank number:

Date of receipt (DD-MM-YYYY):

Frozen:

Losses:

I accept the test material and need no replacement

**Contact Persons:**

Octavio Malato  
[omalato@ual.es](mailto:omalato@ual.es)  
Paula Medina  
[pmedina@ual.es](mailto:pmedina@ual.es)  
**EURL-FV**

Form 1 (Results)



**1. Results Page EUPT-FV-SM 02**

Please indicate which pesticide you have detected. Please also type all the other fields taking into account if you are requested to enter a number or text.

Please specify the methods used for each detected pesticide. When you have described a method for one pesticide (source) and the same method is used for other pesticides (targets), you don't need to put in all the details again. In the column "Method as pesticide No.", simply write the number of the source pesticide, where details of the methods are already given. When you save the page, all fields with methods are copied from the source to the targets pesticide, start to copy all the fields as you described.

When this page is finished click on the "Save this page" button and await a status message to show up.

When all pesticides are done click on "Back to Main page" button.

<input type="button" value="Back to Main page"/> <input type="button" value="Save this page"/>																																			
<table border="0"> <tr> <td style="text-align: right;"><b>Pesticide No:</b></td> <td>1</td> <td style="text-align: right;"><b>Pesticide name:</b></td> <td>mevinphos</td> <td style="text-align: right;"><b>Methods as pesticide No.:</b></td> <td></td> <td style="text-align: right;"><b>Desviation RT (s):</b></td> <td>2.1</td> <td style="text-align: right;"><b>Desviation MS (specify unit or %):</b></td> <td>1 unit</td> <td style="text-align: right;"><b>Chromatographic Technique:</b></td> <td>GC</td> <td style="text-align: right;"><b>Detector:</b></td> <td>MS</td> <td style="text-align: right;"><b>Analyzer:</b></td> <td>Q</td> </tr> </table>	<b>Pesticide No:</b>	1	<b>Pesticide name:</b>	mevinphos	<b>Methods as pesticide No.:</b>		<b>Desviation RT (s):</b>	2.1	<b>Desviation MS (specify unit or %):</b>	1 unit	<b>Chromatographic Technique:</b>	GC	<b>Detector:</b>	MS	<b>Analyzer:</b>	Q	<table border="0"> <tr> <td style="text-align: right;"><b>Instrument model:</b></td> <td>Agilent 5973</td> <td style="text-align: right;"><b>Sample Weight (g):</b></td> <td>10</td> <td style="text-align: right;"><b>Extraction Solvent:</b></td> <td>acetonitrile</td> <td style="text-align: right;"><b>Clean up Step (specify):</b></td> <td>SPE</td> <td style="text-align: right;"><b>Column Type:</b></td> <td>HP 5 MS</td> <td style="text-align: right;"><b>Injecton Volume (µL):</b></td> <td>2 µl</td> <td style="text-align: right;"><b>Software:</b></td> <td>Automatic</td> <td style="text-align: right;"><b>No of compounds in method or library that are pesticides ONLY:</b></td> <td>923</td> <td style="text-align: right;"><b>How frequently do you use a standard solution for your screening method:</b></td> <td>if necessary</td> </tr> </table>	<b>Instrument model:</b>	Agilent 5973	<b>Sample Weight (g):</b>	10	<b>Extraction Solvent:</b>	acetonitrile	<b>Clean up Step (specify):</b>	SPE	<b>Column Type:</b>	HP 5 MS	<b>Injecton Volume (µL):</b>	2 µl	<b>Software:</b>	Automatic	<b>No of compounds in method or library that are pesticides ONLY:</b>	923	<b>How frequently do you use a standard solution for your screening method:</b>	if necessary
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<input type="button" value="Save this page"/> <input type="button" value="Add New One"/>																																			





**ANNEX 2. List of laboratories that participate in EUPT-FV-SM-02.**

<b>COUNTRY</b>	<b>CITY</b>	<b>LABORATORY NAME</b>	<b>REPORTED RESULTS</b>
AUSTRIA	INNSBRUCK	AGES GMBH, COMPETENCE CENTER FOR RESIDUES OF PLANT PROTECTION PRODUCTS	YES
BELGIUM	BELGIUM	LOVAP NV	YES
BELGIUM	ZWIJNAARDE	FYTOLAB	YES
CZECH REPUBLIC	PRAGUE	INSTITUTE OF CHEMICAL TECHNOLOGY PRAGUE, DEPT. OF FOOD CHEMISTRY AND ANALYSIS	YES
DENMARK	RINGSTED	DANISH VET AND FOOD ADM REGION EAST	YES
DENMARK	SOEBORG	NATIONAL FOOD INSTITUTE	YES
DENMARK	PORDENONE	ARPA-FVG	YES
EGYPT	GIZA	CENTRAL LAB OF RESIDUE ANALYSIS OF PESTICIDES AND HEAVY METALS IN FOODS	YES
ESTONIA	SAKU	LABORATORY FOR RESIDUES AND CONTAMINANTS, AGRICULTURAL RESEARCH CENTRE (ARC)	YES
FINLAND	ESPOO	FINNISH CUSTOMS LABORATORY	YES
FRANCE	ILLKIRCH	SCL STRASBOURG	YES
FRANCE	MASSY	ILE DE FRANCE - MASSY SCL LABORATORY	YES
FRANCE	MONTPELLIER	LABORATOIRE DU SCL DE MONTPELLIER	YES
FRANCE	RENNES	SCL-RENNES	YES
FRANCE	SAINT DENIS	SERVICE COMMUN DES LABORATOIRES SAINT DENIS REUNION	NO
GERMANY	BERLIN	FEDERAL OFFICE OF CONSUMER PROTECTION AND FOOD SAFETY (BVL)	YES
GERMANY	DRESDEN	LUA SACHSEN, DEUTSCHLAND	YES
GERMANY	ERLANGEN	BAYERISCHES LANDESAMT FÜR GESUNDHEIT UND LEBENSMITTELSICHERHEIT	YES
GERMANY	MÜNSTER	CHEMISCHES UND VETERINÄRUNTERSUCHUNGSAMT MÜNSTERLAND-EMSCHER-LIPPER	YES
GERMANY	OLDENBURG	NIEDERSAECHSISCHES LANDESAMT FUER VERBRAUCHERSCHUTZ UND LEBENSMITTELSICHERHEIT	YES
GREECE	ATHENS	GENERAL CHEMICAL STATE LABORATORY	YES
GREECE	KIFISSIA	PESTICIDE RESIDUES LAB., BENAKI PHYTOPATHOLOGICAL INSTITUTE	YES
HUNGARY	KAPOSVAR	AGRICULTURAL OFFICE OF COUNTY SOMOGY - PPSCD- PESTICIDE RESIDUE ANALYTICAL LABORATORY	YES
HUNGARY	MISKOLC	AGRICULTURAL OFFICE OF BAZ COUNTY PLANT PROTECTION AND SOIL CONSERVATION DIRECTORATE PESTICIDE RESIDUE ANALYTICAL LABORATORY	YES
IRELAND	CELBRIDGE	PESTICIDE CONTROL LABORATORY	YES
ITALY	AREZZO	AGENZIA REGIONALE PER LA PROTEZIONE AMBIENTALE DELLA TOSCANA - DIPARTIMENTO DI AREZZO (ARPAT - AREZZO)	YES
ITALY	BOZEN	LANDESAGENTUR FÜR UMWELT-LABOR FÜR CHROMATOGRAPHIE	YES
ITALY	VERONA	ARPAV VENETO - SERVIZIO LABORATORI VERONA - U.O. PESTICIDI	YES

**ANNEX 2. List of laboratories that participate in EUPT-FV-SM-02.**

COUNTRY	CITY	LABORATORY NAME	REPORTED RESULTS
NORWAY	AAS	BIOFORSK, PLANT HEALTH AND PLANT PROTECTION, PESTICIDE CHEMISTRY	YES
POLAND	OLSZTYN	WOJEWÓDZKA STACJA SANITARNO - EPIDEMIOLOGICZNA	NO
POLAND	POZNAN	PLANT PROTECTION INSTITUTE, DEPARTMENT OF PESTICIDE RESIDUE RESEARCH	NO
POLAND	TRZEBNICA	INSTITUTE OF PLANT PROTECTION - NATIONAL RESEARCH INSTITUTE	NO
PORTUGAL	OEIRAS	L-INIA - LABORATÓRIO DE RESÍDUOS DE PESTICIDAS	YES
SLOVENIA	KRANJ	INSTITUTE OF PUBLIC HEALTH KRANJ	YES
SLOVENIA	MARIBOR	INSTITUTE OF PUBLIC HEALTH MARIBOR	YES
SPAIN	OVIEDO	LABORATORIO DE SANIDAD VEGETAL	NO
SPAIN	ALMERÍA	LABORATORIO DEL SOIVRE DE ALMERÍA	YES
SPAIN	BURJASOT (VALENCIA)	LABORATORIO AGROALIMENTARIO DE LA GENERALITAT VALENCIANA	YES
SPAIN	EL PALMAR (MURCIA)	LABORATORIO AGROALIMENTARIO Y DE SANIDAD ANIMAL	YES
SPAIN	MADRID	LABORATORIO ARBITRAL AGROALIMENTARIO	YES
SPAIN	MENGÍBAR (JAÉN)	LABORATORIO DE PRODUCCION Y SANIDAD VEGETAL	NO
SPAIN	SANTA FE (GRANADA)	LABORATORIO AGROALIMENTARIO DE GRANADA	YES
SWEDEN	LIDKÖPING	EUROFINS FOOD & AGRO SWEDEN AB	YES
SWEDEN	UPPSALA	CHEMISTRY DIVISION 1, NATIONAL FOOD ADMINISTRATION	YES
SWITZERLAND	GENÈVE	SERVICE DE LA CONSOMMATION ET DES AFFAIRES VÉTÉRINAIRES (SCAV)	YES
THE NETHERLANDS	AMSTERDAM	VWA - FOOD AND CONSUMER PRODUCT SAFETY AUTHORITY, NRL PESTICIDES IN FOOD	YES
THE NETHERLANDS	WAGENINGEN	RIKILT	YES
TURKEY	MERSIN	PRIVATE MSM FOOD CONTROL LABORATORY INC	YES
UNITED KINGDOM	EDINBURGH	SASA	YES
UNITED KINGDOM	YORK	THE FOOD AND ENVIRONMENT AGENCY (FERA)	YES