

**EUROPEAN UNION PROFICIENCY TEST FOR  
PESTICIDES IN FRUITS AND VEGETABLES  
SCREENING METHODS 03  
(EUP-T-FV-SM-03)  
2011**

**Pesticide Residues in Mandarin Homogenate**

**Final Report**

**Organiser:**

**Dr. Amadeo R. Fernández-Alba**

Co-Head of EURL-FV

University of Almería, Edificio Químicas CITE I, Ctra. Sacramento s/n  
04120 Almería, SPAIN

Phone: +34 950015034; Fax: +34 950015645

E-mail: amadeo@ual.es

[www.eurl-pesticides.eu](http://www.eurl-pesticides.eu)

**Organising team at the University of Almería:**

Dr. Milagros Mezcua, Senior Chemist.

University of Almería

Dr. Paula Medina, Chemist.

University of Almería

Mr. Octavio Malato, Chemist.

University of Almería

Ms. Noelia Belmonte, Chemist.

University of Almería

Ms. María Ángeles Martínez Uroz, Chemist.

University of Almería

Ms. María del Mar Gómez Ramos, Chemist.

University of Almería

Ms. Ana Lozano, Chemist.

University of Almería

**Scientific Committee:**

Dr. Antonio Valverde, Senior Chemist (QCG).  
Mr. Stewart Reynolds, Senior Chemist (QCG).

University of Almería, Spain.  
The Food and Environment Research Agency, York, United Kingdom.

Dr. Carmelo Rodríguez, Senior Chemist (SG).  
Dr. Sonja Masselter (AG)

University of Almería, Spain.  
AGES GmbH, Competence Center for Residues of Plant Protection Products, Innsbruck, Austria.

Dr. André de Kok, Senior Chemist (AG).

Food and Consumer Product Safety Authority (VWA), Amsterdam, The Netherlands.

Dr. Tuija Pihlström, Senior Chemist (AG).

National Food Administration, Uppsala, Sweden.

Dr. Hans Mol, Senior Chemist (AG)  
Dr. Magnus Jezussek, Senior Chemist (AG).

RIKILT, Wageningen, The Netherlands.  
Bavarian Health and Food Safety Authority, Erlangen, Germany.

Dr. Darinka Stajnbaher, Senior Chemist (AG).

Institute of Public Health, Maribor, Slovenia.

Dr. Miguel Gamón, Senior Chemist (AG).

Co-Head of EURL-FV. Generalitat Valenciana, Spain.

Dr. Michlangelo Anastassiades, Senior Chemist (AG).  
Dr. Mette Erecius Poulsen, Senior Chemist (AG).

CVUA Stuttgart, Fellbach, Germany.  
National Food Institute, Soeborg, Denmark.

Mr. Ralf Lippold, Senior Chemist (AG)

CVUA, Freiburg, Germany

QCG: Quality Control Group

SG: Statistical Group

AG: Advisory Group



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# **EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUITS AND VEGETABLES**

## **SCREENING METHODS 03**

**2011**

### **BACKGROUND**

According to Article 28 of Regulation 396/2005/EC of the European Parliament and European Council regarding maximum residue levels for pesticides in, or on, food and feed of plant and animal origin<sup>1</sup>: all laboratories analysing samples for the official control of 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 coordinated 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 for independently-organised comparative tests. This year, for the third time, the EURL for pesticides in Fruit and Vegetables at the University of Almería, Spain<sup>3</sup> organised a proficiency test on qualitative screening methods for pesticides in vegetable/fruit commodities. This test was organised because many laboratories are now using screening methods on routine samples allowing a significant increase in their analytical scope.

Participation in this PT remains on a voluntary basis. Besides this, official laboratories have a significant number of mandatory PTs annually as the EURL-FV already organises the PT for quantitative multi-residue pesticide analysis (EUPT-FV13), organised over 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 EUPT data including the individual lab-codes/lab-name keys.

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<sup>1</sup> Regulation (EC) No 396/2005, published in OJ of the EU L70 of 16.03.2005, as last amended by Regulation 839/2008 published in 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 in 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 of Regulation (EC) No 882/2004 of the European Parliament and of the Council as regards Community Reference Laboratories.

## 1. INTRODUCTION

Now in this third year, consolidation of inter-laboratory tests for pesticide screening methods among laboratories seems to be assured; even being requested by those laboratories not performing routine analysis on fruit and vegetables. Responding to this demand, the EURL-FV has decided to continue with their operation. The support given by DG SANCO for these methods has greatly assisted laboratory acceptance by allowing laboratories to conduct screening methods on the EU-Coordinated Multiannual Programme samples.

Over recent years, it has been found that many laboratories not only used a full-scan approach to perform screening but that some also employed modern tandem-mass spectrometers, even if their sensitivity has to be reduced.

Mass spectrometry plays an essential role in the everyday work carried out by laboratories. Technological improvements in modern MS systems offer new possibilities for greatly increasing the scope of MRM analysis. Whereas full-scan measurements are theoretically the best approach for MS screening, developments in targeted measurements also offer the potential for a substantially increased scope of analysis. Another reason for conducting this proficiency test on screening methods is to gather information from laboratories as to the type of software they use for processing data: whether laboratories are using commercial software and databases or whether they are internally constructed and search manually. This type of test provides an overview of such information as well as valuable insight into the possible need for further software development in the near future.

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 Document No. SANCO/10684/2009 - Method validation and quality control procedures for pesticide residues analysis in food and feed.

Only qualitative information was requested for those pesticides detected. It was decided, as in previous PTs, not to provide the laboratories with a Target Pesticide List so that their capability in detecting whatever pesticides were present was also evaluated.

Regulation (EC) No 882/2004 lays down the general tasks, duties and requirements for EURLs in Food, Feed and Animal Health. Among these tasks is the provision for independently-organised comparative tests. The EURL for pesticides in Fruit and Vegetables at the University of Almería, Spain, has organised the proficiency test on qualitative screening methods for pesticides in fruit/vegetable commodities. This EUPT-FV-SM03 is aimed at all National Reference Laboratories (**NRLs**) and all Official Laboratories (**Ofls**) for fruits and vegetables in EU Member States. Laboratories outside this EURL/NRL/Ofl-Network were also able to participate on a case-by-case basis, following consultation with DG SANCO.

## **2. TEST MATERIALS**

### **2.1 Test material**

This proficiency test is based on pesticide residue detection in mandarins. The mandarins used in this study were grown in Valencia, Spain.

The pesticides used to treat the mandarins were spiked with a mixture of standard solutions on a post-harvest treatment. The mandarins were then frozen (using liquid nitrogen), chopped, homogenised and sub-sampled into polyethylene bottles that had previously been coded. The bottles containing the test material were stored frozen (at approximately -20°C) prior to shipment to participants.

Ten of these bottles were randomly chosen and analysed to check the presence of the spiked pesticides.

Two bottles, again randomly chosen, were analysed over a period of time to confirm the stability of the pesticides in the test material (a week prior to the sample delivery and a few days after the deadline for receipt of the participants' results). There was an extra analysis during this period, in which the test material underwent the same conditions as the sample shipment for 48h in order to check if there had been any degradation of any of the pesticides during sample delivery.

The pesticides used to spike the mandarin test material were decided upon by the Quality Control Group. 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-SM03 were chosen taking into account the following considerations:

- Pesticides that were not included in the EU-Coordinated Multiannual Control Programme for 2010 (Regulation (EC) 915/2010).
- Pesticides that had particularly acute toxicity and/or had low ARfD values.

Table 2.1 shows the 26 pesticides used to spike the mandarin sample.

Table 2.1. The spiked pesticides used.

| Spiked Pesticides |                 |                 |               |
|-------------------|-----------------|-----------------|---------------|
| Alachlor          | Fenamidone      | Ofurace         | Quinalphos    |
| Atrazine          | Flucythrinate   | O-Phenylphenol  | Sulfotep      |
| Benalaxydil       | Fonofos         | Phorate         | Terbufos      |
| Carbophenothion   | Forchlorfenuron | Prometryn       | Terbutylazine |
| Chinomethionat    | Mecarbam        | Propoxur        | Tolfenpyrad   |
| Chlozolinate      | Mevinphos       | Prothiophos     |               |
| Etrimfos          | Nuarimol        | Pyridaphenthion |               |

Blank material without spiked pesticides was also prepared and shipped for confirmation purposes.

## **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 and negative ion mode.

## **2.3 Prior analysis of the spiked mandarin test material**

The Organiser's homogeneity and stability tests associated with 'quantitative' PTs were conducted with other acceptance criterion than classical EUPT-FVs. Hence the PT test material was analysed in order to detect the presence of the spiked pesticides consistently confirmed to be above the Organiser's LOD.

For a test aiming to confirm the homogeneity of the test material sent, ten spiked test materials were randomly chosen from those stored in the freezer and analysed in duplicate so as to check for 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. Relative standard deviation (RSD) of these 20 analyses needed to be below or at a 15% to consider the material as homogeneous.

Table 2.3.1 shows the summary of these tests, together with the average concentration values for each of the pesticides used to treat the sample and the RSDs.

Table 2.3.1 Homogeneity tests

| Test material No. | 006<br>a | 006<br>b | 012<br>a | 012<br>b | 035<br>a | 035<br>b | 041<br>a | 041<br>b | 061<br>a | 061<br>b | 085<br>a | 085<br>b | 089<br>a | 089<br>b | 093<br>a | 093<br>b | 104<br>a | 104<br>b | 110<br>a | 110<br>b | A. Cc<br>(µg/kg) | RSD<br>(%) |
|-------------------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|------------------|------------|
| Alachlor          | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 109.3            | 5.4        |
| Atrazine          | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 292.2            | 4.0        |
| Benalaxyll        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 135.0            | 9.7        |
| Carbophenothon    | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 367.8            | 5.5        |
| Chinomethionat    | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 120.8            | 6.5        |
| Chlozolinate      | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 222.0            | 10.9       |
| Etrifos           | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 146.1            | 6.9        |
| Fenamidone        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 320.9            | 12.2       |
| Flucythrinate     | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 752.8            | 6.2        |
| Fonofos           | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 302.3            | 14.4       |
| Forchlorfuron     | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 264.2            | 4.5        |
| Mecarbam          | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 412.5            | 9.7        |
| Mevinphos         | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 283.4            | 10.0       |
| Nuarimol          | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 379.3            | 3.7        |
| Ofurace           | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 95.1             | 5.2        |
| o-Phenylphenol    | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 218.9            | 14.8       |
| Phorate           | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 265.2            | 7.8        |
| Prometryn         | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 695.5            | 2.3        |
| Propoxur          | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 194.8            | 3.6        |
| Prothiophos       | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 191.0            | 8.2        |
| Pyridaphenthion   | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 207.3            | 8.8        |
| Quinalphos        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 205.6            | 8.4        |
| Sulfotep          | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 242.6            | 12.6       |
| Terbufos          | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 328.5            | 8.3        |
| Terbutylazine     | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 396.4            | 4.0        |
| Tolfenpyrad       | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | D        | 38.9             | 15.0       |

D: Detected

A. Cc: Average Concentration

RSD: Relative Standard Deviation

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

The three occasions were:

- Day 1: one week before the sample shipment on 17<sup>th</sup> January 2011.
- Day 2: on 26<sup>th</sup> January 2011 after reproducing the sample shipment conditions.
- Day 3: soon after the deadline for reporting results on 28<sup>th</sup> January 2011.

For all the analyses, the two analytical methods described briefly above (in section 2.2) were used.

Table 2.3.2 Stability tests performed.

| Stability tests      |           |           |           |           |           |           |
|----------------------|-----------|-----------|-----------|-----------|-----------|-----------|
| Test material Number | 015 Day 1 | 015 Day 1 | 042 Day 2 | 042 Day 2 | 071 Day 3 | 071 Day 3 |
| Alachlor             | D         | D         | D         | D         | D         | D         |
| Atrazine             | D         | D         | D         | D         | D         | D         |
| Benalaxyll           | D         | D         | D         | D         | D         | D         |
| Carbophenothion      | D         | D         | D         | D         | D         | D         |
| Chinomethionat       | D         | D         | D         | D         | D         | D         |
| Chlozolinate         | D         | D         | D         | D         | D         | D         |
| Etrimfos             | D         | D         | D         | D         | D         | D         |
| Fenamidone           | D         | D         | D         | D         | D         | D         |
| Flucythrinate        | D         | D         | D         | D         | D         | D         |
| Fonofos              | D         | D         | D         | D         | D         | D         |
| Forchlorfenuron      | D         | D         | D         | D         | D         | D         |
| Mecarbam             | D         | D         | D         | D         | D         | D         |
| Mevinphos            | D         | D         | D         | D         | D         | D         |
| Nuarimol             | D         | D         | D         | D         | D         | D         |
| Ofurace              | D         | D         | D         | D         | D         | D         |
| o-Phenylphenol       | D         | D         | D         | D         | D         | D         |
| Phorate              | D         | D         | D         | D         | D         | D         |
| Prometryn            | D         | D         | D         | D         | D         | D         |
| Propoxur             | D         | D         | D         | D         | D         | D         |
| Prothiophos          | D         | D         | D         | D         | D         | D         |
| Pyridaphenthion      | D         | D         | D         | D         | D         | D         |
| Quinalphos           | D         | D         | D         | D         | D         | D         |
| Sulfotep             | D         | D         | D         | D         | D         | D         |
| Terbufos             | D         | D         | D         | D         | D         | D         |
| Terbutylazine        | D         | D         | D         | D         | D         | D         |
| Tolfenpyrad          | D         | D         | D         | D         | D         | D         |

D: Detected

All the pesticides used to spike the samples demonstrated sufficient stability even after reproducing the 48h delivery conditions. All were detected on each occasion.

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

Approximately 300 g of treated mandarin homogenate together with another 300 g of 'blank' mandarin homogenate were shipped to participants on 24<sup>th</sup> January 2011. The deadline for results submission to the Organiser was 72 hours after receipt of the test material. Participants 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 would 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 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 the Protocol, were uploaded onto the EUPT-FV-SM03 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 Form 0 (Sample Receipt) and Form 1 (Results). These allowed the evaluation of the mass-spectrometric screening methods that each of the participants used.

### **3. STATISTICAL METHODS**

#### **3.1 Type of results reported**

The results evaluation is concerned with the results themselves matching the pesticides the Organiser used to treat the sample: or otherwise stating a 'not-reported pesticide' or 'other reported pesticide' from those used to treat the sample. After receiving the results, the Organiser may consider further evaluation highlighted by important information received.

##### 3.1.1 Other Reported Pesticides

These will be considered as those results showing the apparent presence of pesticides which were: (i) not used in the test material treatment, or (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 an 'Other Reported Pesticide' result will be made on a case-by-case basis.

##### Organiser Note:

- Not all screening methods immediately provide sufficient information to allow full identification. In such cases, when they detect a pesticide in real life, laboratories normally do a follow-up confirmatory analysis; using, for example, LC-MS/MS and based on two transitions. In future PTs of this nature, there will be a need to distinguish between suspect or tentative detections and full identification.

##### 3.1.2 Not-Reported Pesticides

These will be considered as any pesticides present in the sample but not reported by the lab even though the Organiser had used it to spike the test material and it was detected by the majority of participants.

## 4. RESULTS

### 4.1 Summary of reported results

Fifty-two laboratories agreed to participate in this third proficiency test on screening methods. Forty-six 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 | % of Reported* | No. of Not Reported | % of Not Reported* |
|-----------------|-----------------|----------------|---------------------|--------------------|
| Alachlor        | 38              | 83%            | 8                   | 17%                |
| Atrazine        | 41              | 89%            | 5                   | 11%                |
| Benalaxyl       | 43              | 93%            | 3                   | 7%                 |
| Carbophenothion | 34              | 74%            | 12                  | 26%                |
| Chinomethionat  | 29              | 63%            | 17                  | 37%                |
| Chlozolinate    | 35              | 76%            | 11                  | 24%                |
| Etrimfos        | 40              | 87%            | 6                   | 13%                |
| Fenamidone      | 32              | 70%            | 14                  | 30%                |
| Flucythrinate   | 39              | 85%            | 7                   | 15%                |
| Fonofos         | 39              | 85%            | 7                   | 15%                |
| Forchlорfenuron | 11              | 24%            | 35                  | 76%                |
| Mecarbam        | 43              | 93%            | 3                   | 7%                 |
| Mevinphos       | 42              | 91%            | 4                   | 9%                 |
| Nuarimol        | 38              | 83%            | 8                   | 17%                |
| Ofurace         | 23              | 50%            | 23                  | 50%                |
| o-Phenylphenol  | 40              | 87%            | 6                   | 13%                |
| Phorate         | 37              | 80%            | 9                   | 20%                |
| Prometryn       | 41              | 89%            | 5                   | 11%                |
| Propoxur        | 38              | 83%            | 8                   | 17%                |
| Prothiophos     | 40              | 87%            | 6                   | 13%                |
| Pyridaphenthion | 32              | 70%            | 14                  | 30%                |
| Quinalphos      | 43              | 93%            | 3                   | 7%                 |
| Sulfotep        | 41              | 89%            | 5                   | 11%                |
| Terbufos        | 38              | 83%            | 8                   | 17%                |
| Terbutylazine   | 43              | 93%            | 3                   | 7%                 |
| Tolfenpyrad     | 13              | 28%            | 33                  | 72%                |

\* The % of laboratories is calculated based on the total number of laboratories submitting results (46).

#### 4.1.1 Other Reported Pesticides

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

Table 4.2. 'Other reported pesticides' in the test material given by laboratories.

| LABORATORY CODE | REPORTED PESTICIDE    | LABORATORY CODE | REPORTED PESTICIDE |
|-----------------|-----------------------|-----------------|--------------------|
| Lab003          | Phenthioate           | Lab035          | Aldimorph          |
|                 | Trietazina            |                 | Butralin           |
| Lab008          | Ametryn               |                 | Fenazaquin         |
|                 | Tralkoxydim           |                 | Fluacrypyrim       |
| Lab011          | Clomazone             |                 | Fluazifop butyl    |
|                 | Ditalimfos            | Lab040          | Tribenuron methyl  |
|                 | Eoprocarb             | Lab045          | Imazalil           |
|                 | Fensulfothion sulfone | Lab046          | Dimethomorph       |
|                 | Merphos               | Lab047          | Terbutryn          |
|                 | Metolachlor           | Lab051          | Bromophos ethyl    |
|                 | Tralkoxydim           | Lab053          | Alpha Cypermetrin  |
|                 | Triazamate            |                 | Bitertanol         |
| Lab013          | Phoxim                |                 | Clomazone          |
| Lab020          | Pyridaben             |                 | Dichlofenthion     |
|                 | Thiabendazole         |                 | Methfuroxan        |
| Lab022          | Ametryn               |                 | Metoxychlor        |
| Lab023          | Alpha Cypermetrin     |                 | Propachlor         |
| Lab030          | Fluquinconazole       |                 | Pymetrazine        |
|                 | Flutriafol            |                 | Pyridaben          |
|                 | Imazalil              |                 | Sebutylazine       |
|                 | Triadimefon           |                 | Siduron            |
| Lab033          | Naphthalene           |                 |                    |

There were three pesticides reported by some laboratories and detected in repeated analyses by the Organiser. These were chlorpyrifos, propazine and simazine. The laboratories that reported them are shown in Table 4.3.

Table 4.3 Pesticides detected by laboratories and the Organiser, other than those spiked

| LABORATORY CODE | REPORTED PESTICIDE | LABORATORY CODE | REPORTED PESTICIDE |
|-----------------|--------------------|-----------------|--------------------|
| Lab011          | Chlorpyrifos ethyl | Lab032          | Chlorpyrifos ethyl |
| Lab012          | Chlorpyrifos ethyl |                 | Propazine          |
| Lab013          | Propazine          | Lab039          | Simazine           |
| Lab016          | Propazine          |                 | Chlorpyrifos ethyl |
|                 | Simazine           |                 | Propazine          |
| Lab022          | Propazine          | Lab040          | Chlorpyrifos ethyl |
|                 | Simazine           | Lab046          | Propazine          |

| LABORATORY CODE | REPORTED PESTICIDE |  | LABORATORY CODE | REPORTED PESTICIDE |
|-----------------|--------------------|--|-----------------|--------------------|
| Lab023          | Chlorpyrifos ethyl |  |                 | Simazine           |
|                 | Propazine          |  | Lab047          | Chlorpyrifos ethyl |
| Lab025          | Chlorpyrifos ethyl |  | Lab051          | Chlorpyrifos ethyl |

#### 4.1.2 Not-Reported Pesticides

In Table 4.1, the number and percentage of laboratories not reporting each of the pesticides used to spike the sample can be seen. The individual results for each laboratory are given in Appendix 1. Graphical representations can be seen in Appendix 2.

#### **4.2 Concentration levels.**

Twenty-six pesticides were used to spike the mandarin test material at different levels, in the range between 50 and 1000 ppb. This EUPT was focused only on detection capabilities; therefore no quantitative data were requested.

#### **4.3 Assessment of laboratory performance.**

No z-score values (nor any other statistical calculations) have been carried out for laboratories to assess their performance as no numerical results were reported by the participants. However, classification has been considered important, based on the number of detected results each laboratory reported and also which methods they used.

Table 4.4 classifies the laboratories according to the number of spiked pesticides reported.

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

| Laboratory Code | Reported | Other Detected Pesticides  |                                |
|-----------------|----------|----------------------------|--------------------------------|
|                 |          | Confirmed by the Organiser | Not confirmed by the Organiser |
| 4               | 26       | -                          |                                |
| 5*              | 26       | -                          |                                |
| 11*             | 26       | 1                          | 8                              |
| 22              | 26       | 2                          | 1                              |
| 13              | 25       | 1                          | 1                              |
| 18              | 25       | -                          |                                |
| 25              | 25       | 1                          | 0                              |
| 28*             | 25       | -                          |                                |
| 32              | 25       | 3                          | 0                              |
| 33              | 25       |                            | 1                              |
| 46              | 25       | 2                          | 1                              |
| 1               | 24       | -                          |                                |
| 8               | 24       |                            | 2                              |
| 17              | 24       | -                          |                                |
| 34*             | 24       | -                          |                                |

| Laboratory Code | Reported | Other Detected Pesticides  |                                |
|-----------------|----------|----------------------------|--------------------------------|
|                 |          | Confirmed by the Organiser | Not confirmed by the Organiser |
| 50              | 24       | -                          |                                |
| 51*             | 24       | 1                          | 1                              |
| 10              | 23       | -                          |                                |
| 16              | 23       | 2                          | 0                              |
| 21              | 23       | -                          |                                |
| 12*             | 22       | 1                          | 0                              |
| 14              | 22       | -                          |                                |
| 26              | 22       | -                          |                                |
| 39              | 22       | 2                          | 0                              |
| 41*             | 22       | -                          |                                |
| 52              | 22       | -                          |                                |
| 40              | 21       | 1                          | 1                              |
| 53*             | 21       | -                          | 11                             |
| 45              | 20       | -                          | 1                              |
| 6*              | 19       | -                          |                                |
| 15              | 19       | -                          |                                |
| 19              | 19       | -                          |                                |
| 23              | 19       | 2                          | 1                              |
| 35*             | 18       | -                          | 5                              |
| 42*             | 18       | -                          |                                |
| 44*             | 18       | -                          |                                |
| 9               | 17       | -                          |                                |
| 30*             | 16       | -                          | 4                              |
| 37*             | 16       | -                          |                                |
| 20              | 15       | -                          | 2                              |
| 3               | 14       | -                          | 2                              |
| 47*             | 13       | 1                          | 1                              |
| 24*             | 12       | -                          |                                |
| 29              | 9        | -                          |                                |
| 49              | 3        | -                          |                                |
| 36              | 2        | -                          |                                |

\* National Reference Laboratories for Fruit and Vegetables from the EU participating in this test.

The methods used by the laboratories, the chromatographic techniques, detectors, instrumentation, etc... are detailed in Appendix 3. In Table 4.4, there is a summary of the chromatographic techniques used for each pesticide, and a graphical representation is shown in Appendix 2.

Table 4.4 Chromatographic techniques used to determine each pesticide in the test material

| Pesticide       | Total no. of Reported Pesticides | Total % of Reported Pesticides | GC | IC |
|-----------------|----------------------------------|--------------------------------|----|----|
| Benalaxyd       | 43                               | 93%                            | 25 | 18 |
| Mecarbam        | 43                               | 93%                            | 20 | 23 |
| Quinalphos      | 43                               | 93%                            | 32 | 11 |
| Terbutylazine   | 43                               | 93%                            | 22 | 21 |
| Mevinphos       | 42                               | 91%                            | 28 | 14 |
| Atrazine        | 41                               | 89%                            | 19 | 22 |
| Prometryn       | 41                               | 89%                            | 26 | 15 |
| Sulfotep        | 41                               | 89%                            | 30 | 11 |
| Etrimfos        | 40                               | 87%                            | 29 | 11 |
| o-Phenylphenol  | 40                               | 87%                            | 39 | 1  |
| Prothiophos     | 40                               | 87%                            | 36 | 4  |
| Flucythrinate   | 39                               | 85%                            | 34 | 5  |
| Fonofos         | 39                               | 85%                            | 28 | 11 |
| Alachlor        | 38                               | 83%                            | 24 | 14 |
| Nuarimol        | 38                               | 83%                            | 25 | 13 |
| Propoxur        | 38                               | 83%                            | 14 | 24 |
| Terbufos        | 38                               | 83%                            | 23 | 15 |
| Phorate         | 37                               | 80%                            | 27 | 10 |
| Chlozolinate    | 35                               | 76%                            | 35 | 0  |
| Carbophenothion | 34                               | 74%                            | 29 | 5  |
| Fenamidone      | 32                               | 70%                            | 9  | 23 |
| Pyridaphenthion | 32                               | 70%                            | 18 | 14 |
| Chinomethionat  | 29                               | 63%                            | 27 | 2  |
| Ofurace         | 23                               | 50%                            | 8  | 15 |
| Tolfenpyrad     | 13                               | 28%                            | 6  | 7  |
| Forchlorfenuron | 11                               | 24%                            | 0  | 11 |

In Table 4.4, it can be observed that 72% of the overall results (857 out of 1,196) for the pesticides present in the sample are reported at or above 70%; and that 61% of the overall results are at or above 80%. In Appendix 2, graphical representations of the coloured-bar techniques used can be seen.

Table 4.5 shows the number and percentage of the pesticides used by the Organiser to spike the sample which were reported by each laboratory. National Reference Laboratories are marked with an asterisk sign.

Table 4.5. Number and Percentage of Pesticides Present Reported by Laboratory

| Laboratory Code | Number of Pesticides Present Reported | % of Pesticides Present Reported |
|-----------------|---------------------------------------|----------------------------------|
| 1               | 24                                    | 92                               |
| 2*              | No Results Reported                   |                                  |
| 3               | 14                                    | 54                               |
| 4               | 26                                    | 100                              |
| 5*              | 26                                    | 100                              |
| 6*              | 19                                    | 73                               |
| 7               | No Results Reported                   |                                  |
| 8               | 24                                    | 92                               |
| 9               | 17                                    | 65                               |
| 10              | 23                                    | 88                               |
| 11*             | 26                                    | 100                              |
| 12*             | 22                                    | 85                               |
| 13              | 25                                    | 96                               |
| 14              | 22                                    | 85                               |
| 15              | 19                                    | 73                               |
| 16              | 23                                    | 88                               |
| 17              | 24                                    | 92                               |
| 18              | 25                                    | 96                               |
| 19              | 19                                    | 73                               |
| 20              | 15                                    | 58                               |
| 21              | 23                                    | 88                               |
| 22              | 26                                    | 100                              |
| 23              | 19                                    | 73                               |
| 24*             | 12                                    | 46                               |
| 25              | 25                                    | 96                               |
| 26              | 22                                    | 85                               |
| 27              | No Results Reported                   |                                  |
| 28*             | 25                                    | 96                               |
| 29              | 9                                     | 35                               |
| 30*             | 16                                    | 62                               |
| 31              | No Results Reported                   |                                  |
| 32              | 25                                    | 96                               |
| 33              | 25                                    | 96                               |
| 34*             | 24                                    | 92                               |
| 35*             | 18                                    | 69                               |
| 36              | 2                                     | 8                                |
| 37*             | 16                                    | 62                               |
| 38              | No Results Reported                   |                                  |
| 39              | 22                                    | 85                               |
| 40              | 21                                    | 81                               |
| 41*             | 22                                    | 85                               |
| 42*             | 18                                    | 69                               |

| Laboratory Code | Number of Pesticides Present Reported | % of Pesticides Present Reported |
|-----------------|---------------------------------------|----------------------------------|
| 43              | No Results Reported                   |                                  |
| 44*             | 18                                    | 69                               |
| 45              | 20                                    | 77                               |
| 46              | 25                                    | 96                               |
| 47*             | 13                                    | 50                               |
| 49              | 3                                     | 12                               |
| 50              | 24                                    | 92                               |
| 51*             | 24                                    | 92                               |
| 52              | 22                                    | 85                               |
| 53*             | 21                                    | 81                               |

\* National Reference Laboratories for Fruit and Vegetables from the EU participating in this test.

## **5. CONCLUSIONS**

Fifty-two laboratories applied to participate in this test and forty-six laboratories submitted results. Seventeen of the laboratories which applied were National Reference Laboratories for Fruits and Vegetables (marked with an asterisk on the graphs and tables) representing nineteen Member States. In addition to these, 2 EFTA countries (Norway and Switzerland) and two non-EU/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 the case of LC-MS analysis, targeted acquisition methods using triple quadrupole instruments were the most widely used.

Four of the 46 laboratories were able to detect all 26 pesticides in the spiked mandarin test material. Only 4 laboratories failed to detect below 50% of the pesticides present.

Seventy-one percent of the laboratories that reported results were able to find more than 70% of the pesticides used to spike the sample whereas last year (EUPT-FV-SM02) only 50% managed to do so. This gives an idea of the scale of improvement possible for laboratories using these types of methods.

Sixteen participants reported forty different pesticides which were not used to spike the mandarins. Whether this should be judged as poor performance, or not, depends on how each participant would act on these positive findings in routine analysis. If the detected pesticide were reported as positive with no further identifying confirmation, then the result would be a false positive and hence erroneous monitoring data would be reported. If the detected pesticide is regarded simply as 'suspect' or 'indicatively present', leading to additional analysis to confirm identity before reporting the result, then those pesticides indicated as 'other reported pesticides' in this report are not really an issue.

This third 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 feed, or not monitored by the laboratories because they are not part of the EU-Coordinated Programme. 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 screening methods performance (i.e. validation) are necessary to improve reliability of such methods.

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

The Organiser and the Scientific Committee consider that screening methods have provided additional value to the current quantitative multiresidue methods routinely used for monitoring purposes. The results of this third test are most 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 screening method validation has been recognised and guidelines for such validation have been prepared and included in the updated SANCO document: "Method validation and quality control procedures for pesticide residue analysis in food and feed" (SANCO/12495/2011).

Next year, pear matrix test material will be used. If laboratories have a particular interest for specific matrices, they should inform the EURL-FV and their suggestions will be evaluated. The timing for the test material delivery will be February 2012; and 72 hours will be allowed for results submission (given that this should be time enough to undertake screening methods). There will not be a target list, as was the case in this test.

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## **8. ACKNOWLEDGEMENTS**

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## APPENDIX 1. Results

| Laboratory Code  | Alachlor | Atrazine | Benazolin | Carbofenthion | Chinomethionat | Chlozolinate | Etrimesfos | Fenamidone | Flucythrinate | Fonofos | Forchlorfenuron | Mecarbam | Mevinphos | Nuarimol | Oflurace | <i>o</i> -Phenyphenol | Phorate | Prometryn | Propoxur | Prothiophos | Pyridaphenthion | Quinalphos | Sulfotep | Terbufos | Terbutylazine | Tolfenpyrad | Total | %  |    |
|------------------|----------|----------|-----------|---------------|----------------|--------------|------------|------------|---------------|---------|-----------------|----------|-----------|----------|----------|-----------------------|---------|-----------|----------|-------------|-----------------|------------|----------|----------|---------------|-------------|-------|----|----|
| 1                | D        | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 24          | 92    |    |    |
| 2*               |          |          |           |               |                |              |            |            |               |         |                 |          |           |          |          | No Results Reported   |         |           |          |             |                 |            |          |          |               |             |       |    |    |
| 3                |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 14          | 54    |    |    |
| 4                |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 26          | 100   |    |    |
| 5*               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 26          | 100   |    |    |
| 6*               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 19          | 73    |    |    |
| 7                |          |          |           |               |                |              |            |            |               |         |                 |          |           |          |          | No Results Reported   |         |           |          |             |                 |            |          |          |               |             |       |    |    |
| 8                |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 24          | 92    |    |    |
| 9                |          |          |           |               |                |              |            |            |               |         |                 |          |           |          |          |                       |         |           |          |             |                 |            |          |          |               |             |       | 17 | 65 |
| 10               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 23          | 88    |    |    |
| 11*              |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 26          | 100   |    |    |
| 12*              |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 22          | 85    |    |    |
| 13               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 25          | 96    |    |    |
| 14               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 22          | 85    |    |    |
| 15               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 19          | 73    |    |    |
| 16               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 23          | 88    |    |    |
| 17               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 24          | 92    |    |    |
| 18               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 25          | 96    |    |    |
| 19               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 19          | 73    |    |    |
| 20               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 15          | 58    |    |    |
| 21               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 23          | 88    |    |    |
| 22               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 26          | 100   |    |    |
| 23               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 19          | 73    |    |    |
| 24               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 12          | 46    |    |    |
| 25               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 25          | 96    |    |    |
| 26               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 22          | 85    |    |    |
| 27               |          |          |           |               |                |              |            |            |               |         |                 |          |           |          |          | No Results Reported   |         |           |          |             |                 |            |          |          |               |             |       |    |    |
| 28*              |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 25          | 96    |    |    |
| 29               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 9           | 35    |    |    |
| 30*              |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 16          | 62    |    |    |
| 31               |          |          |           |               |                |              |            |            |               |         |                 |          |           |          |          | No Results Reported   |         |           |          |             |                 |            |          |          |               |             |       |    |    |
| 32               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 25          | 96    |    |    |
| 33               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 25          | 96    |    |    |
| 34*              |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 24          | 92    |    |    |
| 35*              |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 18          | 69    |    |    |
| 36               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 2           | 8     |    |    |
| 37*              |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 16          | 62    |    |    |
| 38               |          |          |           |               |                |              |            |            |               |         |                 |          |           |          |          | No Results Reported   |         |           |          |             |                 |            |          |          |               |             |       |    |    |
| 39               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 22          | 85    |    |    |
| 40               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 21          | 81    |    |    |
| 41*              |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 22          | 85    |    |    |
| 42*              |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 18          | 69    |    |    |
| 43               |          |          |           |               |                |              |            |            |               |         |                 |          |           |          |          | No Results Reported   |         |           |          |             |                 |            |          |          |               |             |       |    |    |
| 44*              |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 18          | 69    |    |    |
| 45               |          |          |           |               |                |              |            |            |               |         |                 |          |           |          |          |                       |         |           |          |             |                 |            |          |          |               |             |       | 20 | 77 |
| 46               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 25          | 96    |    |    |
| 47*              |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 13          | 50    |    |    |
| 49               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 3           | 12    |    |    |
| 50               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 24          | 92    |    |    |
| 51*              |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 24          | 92    |    |    |
| 52               |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 22          | 85    |    |    |
| 53*              |          | D        | D         | D             | D              | D            | D          | D          | D             | D       | D               | D        | D         | D        | D        | D                     | D       | D         | D        | D           | D               | D          | D        | D        | D             | 21          | 81    |    |    |
| Total Detections | 38       | 41       | 43        | 34            | 29             | 35           | 40         | 32         | 39            | 39      | 11              | 43       | 42        | 38       | 23       | 40                    | 37      | 41        | 38       | 40          | 32              | 43         | 41       | 38       | 43            | 13          |       |    |    |
| % of Detections  | 84       | 91       | 96        | 76            | 64             | 78           | 89         | 71         | 87            | 87      | 24              | 96       | 93        | 84       | 51       | 89                    | 82      | 91        | 84       | 89          | 71              | 96         | 91       | 84       | 96            | 29          |       |    |    |

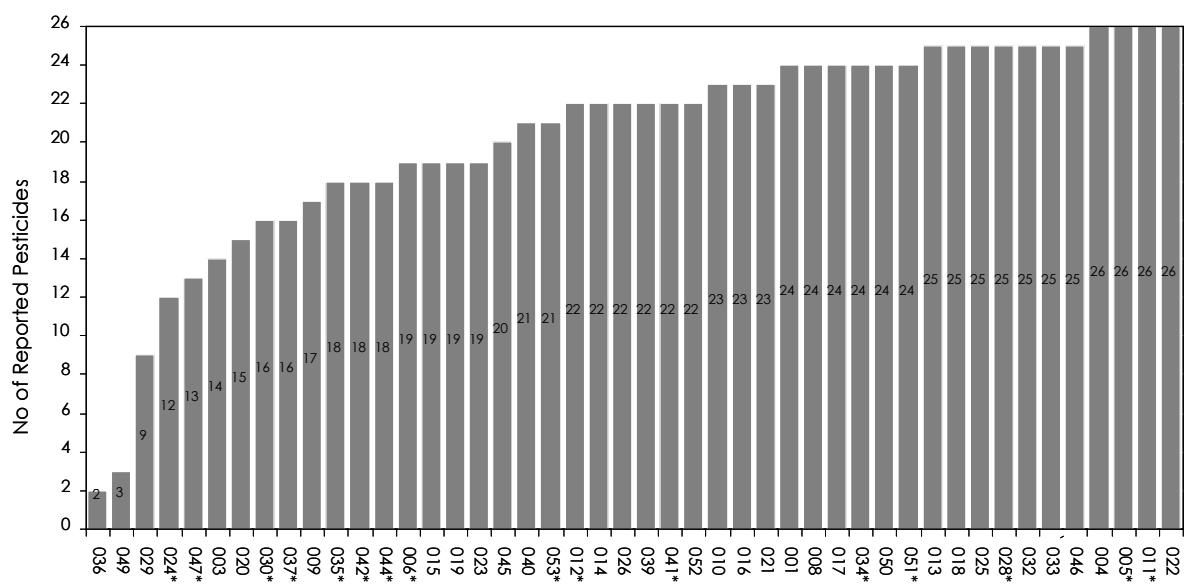
D: Detected pesticide reported

\* NRLs from EU

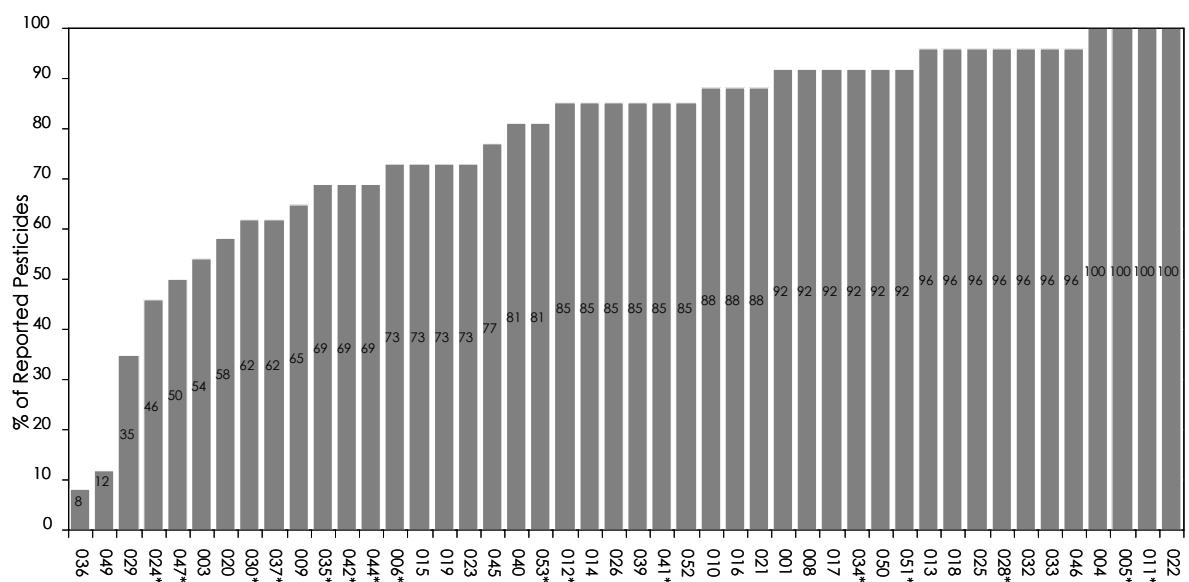


## APPENDIX 2. Graphical Representations

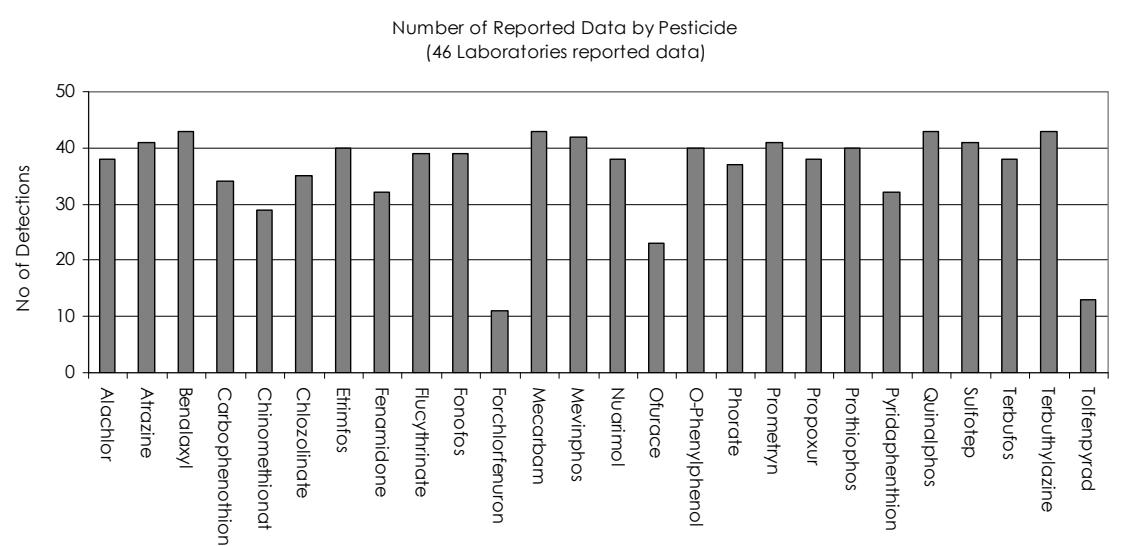
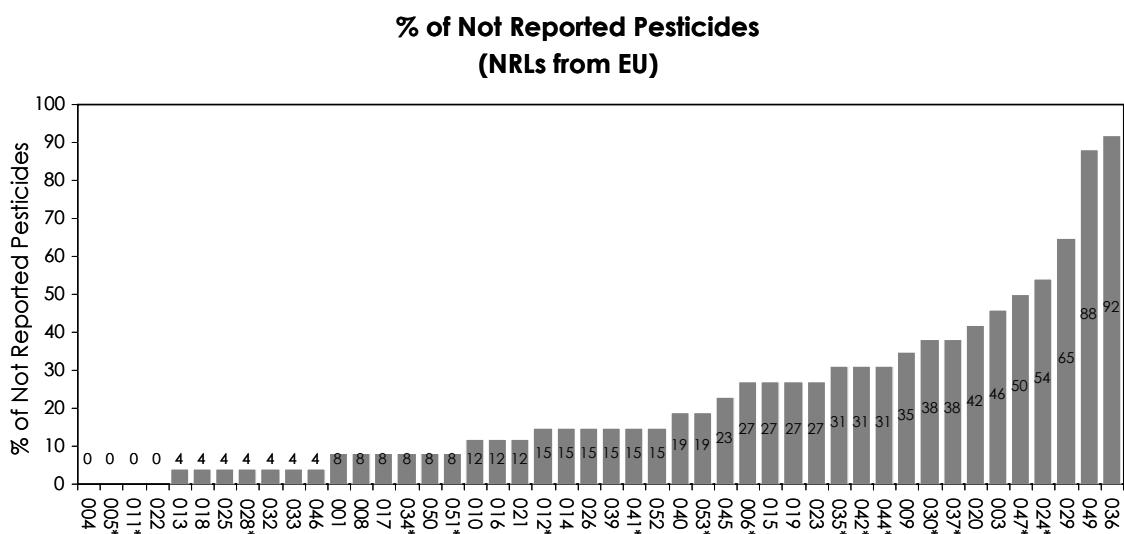
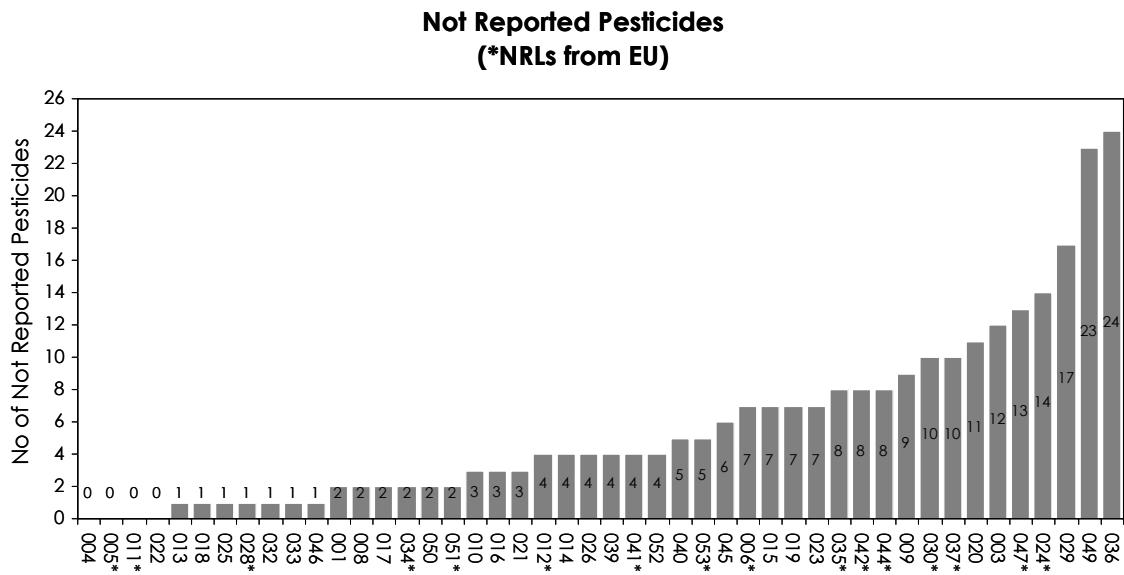
**Number of Reported Data by Laboratory**  
(\*NRLs from EU) (26 Pesticides Included)



**Number of Reported Data by Laboratory**  
(\*NRLs from EU) (26 Pesticides Included)

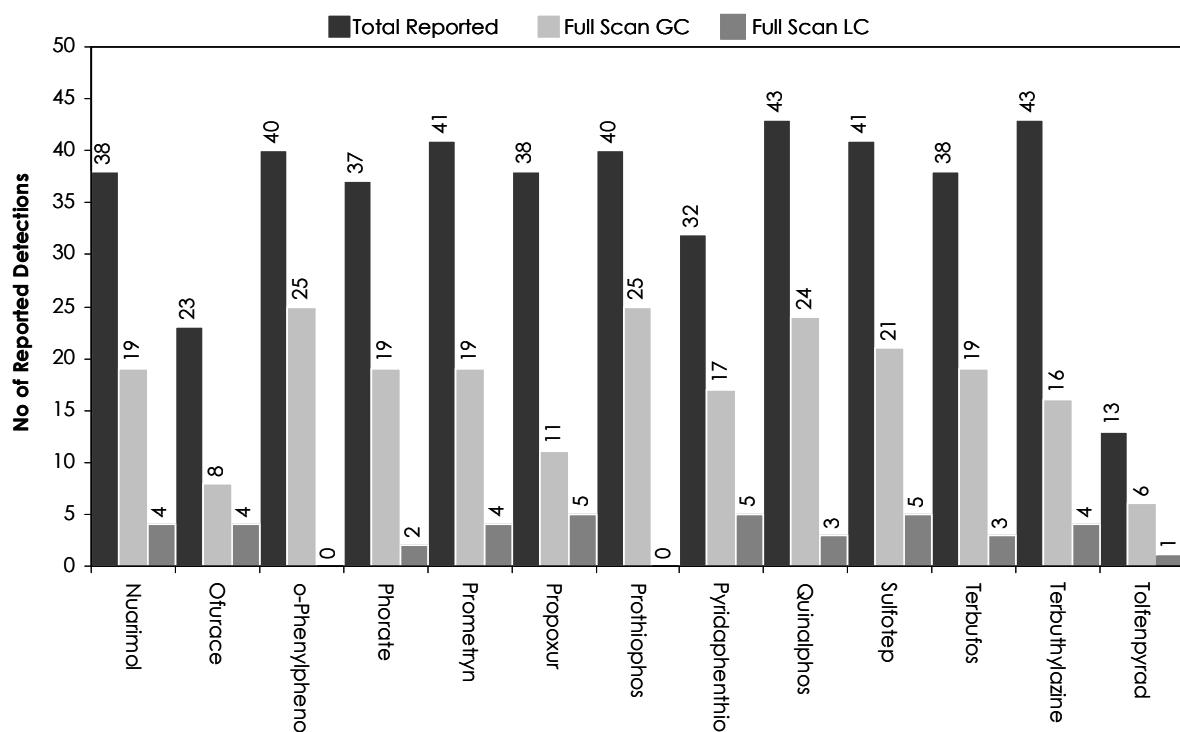
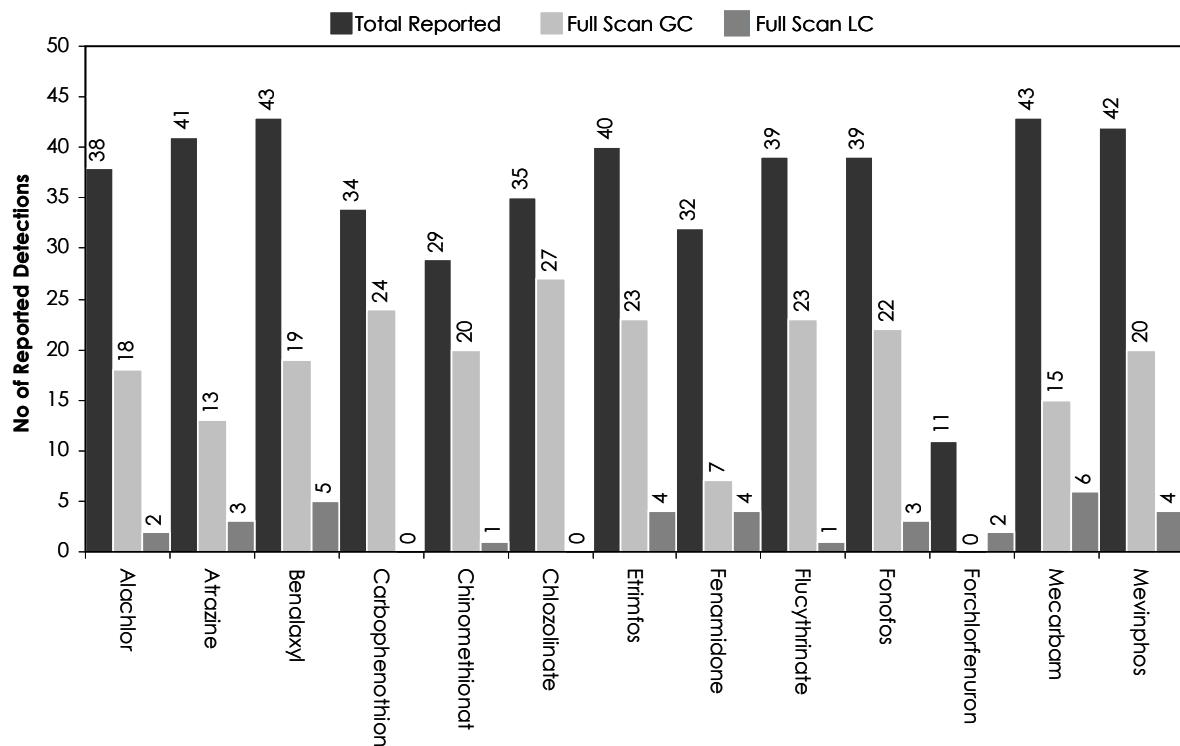


## APPENDIX 2. Graphical Representations



## APPENDIX 2. Graphical Representations

Chromatographic Techniques used in Full Scan to determine each pesticide in the test material





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

| Laboratory Code | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Detector | Instrument Model | Sample Weight (g)      | Clean-up                       | Column Type | Injection Volume (µl) | No. of Compounds  | Standard Solution         | ALACHLOR     |            |  |
|-----------------|------------------|--------------|--------------------------|----------|------------------|------------------------|--------------------------------|-------------|-----------------------|-------------------|---------------------------|--------------|------------|--|
|                 |                  |              |                          |          |                  |                        |                                |             |                       |                   |                           | Frequency of | Each Batch |  |
| 001             | 60               |              | LC                       | No       | MS               | QQQ                    | API 4000                       | 10          | ACN                   | DSPE              | Waters C18 2.1x50mmx1.7µm | 5            | Both       | 600  |
| 004             |                  |              | LC                       | No       | MS               | QQQ                    | Applied Biosystems API5000     | 10          | EtOAc                 | Filter            | HSS T3                    | 2            | Both       | 104  |
| 005             |                  |              | LC                       | No       | MS               | QQQ                    | Agilent 6460                   | 10          | EtOAc                 |                   | C18                       | 2            | Automatic  | 500  |
| 008             | 359              | 100          | LC                       | No       | MS               | Q-Trap                 | API 4000 Qtrap                 | 10          | ACN                   | Pursuit XRs Ultra | 6                         | Both         | 250        |  |
| 010             |                  |              | GC                       | Yes      | MS               | Q                      | HP5973 MSD                     | 10          | ACN                   | DSPE              | HP-5MSI                   | 20           | Both       | 624  |
| 011             | 0.1              | 0.0          | GC                       | Yes      | MS               | Q                      | HP5975C                        | 10          | ACN                   | PSA               | HP5MS                     | 5            | Both       | approx.1.000                                       |
| 012             | 30               | 20           | GC                       | Yes      | MS               | IT                     | Saturn 4000 Varian             | 10          | ACN                   | DSPE              | DB5MS                     | 1            | Both       | 350  |
| 013             | -3.6             | 95           | GC                       | Yes      | MS               | Q                      | Agilent 5975                   | 10          | ACN                   | MgSO4/PSA         | HP5MS                     | 20           | Automatic  | 550  |
| 014             |                  |              | GC                       | Yes      | MS               | Q                      | Trace DSQ                      | 15          | ACN                   | QuEChERS          | DB5-MS                    | 0.8          | Both       |  |
| 015             | > 70%            | GC           | Yes                      | MS       | IT               | ThermoFinniganPolarisQ | 10                             | ACN         |                       | DB-5MS            |                           | 8            | Both       | 500  |
| 016             |                  |              | LC                       | No       | MS               | QQQ                    | Waters Premier Quattro XE      | 10          | ACN                   | PSA               | Kinetex XB-C18            | 10           | Both       | Targeted Analysis Only                             |
| 017             | 1.2              | -17.20       | LC                       | No       | MS               | QQQ                    | HPLC Agilent 1100, MS API 3000 | 10          | ACN                   | DSPE              | C 18 3µm 50x2mm           | 10           | Both       | 525 method<br>590 library<br>Never                 |
| 018             | 0.0              | 0.0          | GC                       | No       | MS               | QQQ                    | Thermo TSQ Quantum GC          | 10          | Acetone/PE/DCM        | Na2CO4            | RXi-5SIL MS               | 1            | Automatic  | 164  |
| 019             | 9                | 30           | GC                       | Yes      | MS               | Q                      | Agilent GC7890-M5975C          | 10          | ACN                   | DSPE              | HP5-MS                    | 1            | Both       | 150 method<br>500 library<br>(and very rare cases) |
| 021             |                  |              | GC                       | Yes      | MS               | IT                     | Varian 4000                    | 10          | ACN                   | DSPE              | Capillary                 | 3            | Automatic  | 450  |
| 022             | <1.5%            | LC           | No                       | MS       | QQQ              | Waters Aquity TQD      | 15                             | ACN         | DSPE                  | UPLC-BEH C18      | 10                        | Both         | 340        |  |
| 023             | 0.0              |              | LC                       | No       | MS/MS            | Q                      | API 4000                       | 10          | ACN                   | DSPE              | C18 hydro Phenomenex      | 40           | Manual     | Once a Day   |
| 025             | 5                | 13           | GC                       | Yes      | MS               | TOF                    | LECO Pegasus IV                | 10          | ACN                   | DSPE(PSA)         | HP-5MS                    | 5            | Both       | 650  |
| 026             | 0.0              | 0.0          | LC                       | No       | MS               | QQQ                    | Xevo TQ                        | 15          | ACN                   | LLE               | C18                       | 10           | Both       | 360  |
| 029             |                  |              | GC                       | Yes      | MS               | IT                     | Varian GCmasse 4000            | 10          | ACN                   | DSPE (MgSO4)      | Capillary db 5            | 5            | Both       | Each Batch   |
| 030             |                  |              | GC                       | Yes      | MS               | TOF                    | Leco Pegasus                   | 15          | Acetone, DCM, PE      |                   | DB-5                      | 1            | Automatic  |  |
| 032             | RT locking       |              | GC                       | Yes      | MS               | GCMSD                  | Agilent                        | 10          | ACN                   | PSA               | HP5                       | 10           | Both       | > 600  |
|                 |                  |              |                          |          |                  |                        |                                |             |                       |                   |                           |              | Always     |  |

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

| Laboratory Code | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Detector | Analyser | Instrument Model | Sample Weight (g)          | ALACHLOR |             |                         | No. of Compounds  | Software  | Frequency of Standard Solution |                 |                           |
|-----------------|------------------|--------------|--------------------------|----------|----------|------------------|----------------------------|----------|-------------|-------------------------|-------------------|-----------|--------------------------------|-----------------|---------------------------|
|                 |                  |              |                          |          |          |                  |                            | Clean-up | Column Type | Injection Volume (μl)   |                   |           |                                |                 |                           |
| 033             | 9                | 20%          | GC                       | Yes      | MS       | Q                | Agilent GC/MSD 7890A/5975C | 10       | ACN         | PSA, C18                | HP-5MS            | 1         | Automatic                      | 927             | Each Batch                |
| 034             | 60               | 10 ppm       | LC                       | Yes      | MS       | IT               | Agilent 6230               | 10       | ACN         | Zorbax Eclipse plus C18 | 3                 | Automatic | 517                            | Not for routine |                           |
| 036             |                  |              | GC                       | No       | MS       | Q                | Agilent Tech. 5973 inert   | 15       | Acetone     |                         | HP-5MS            | 2         | Both                           |                 | Always                    |
| 037             |                  |              | GC                       | Yes      | MS       | Q                | Agilent 6890N              | 10       | ACN         | PSA                     | DB-5              | 1         | Both                           | 80              |                           |
| 039             |                  |              | GC                       | No       | MS       | QQQ              | Agilent 7000B              | 10       | ACN         | DSPE                    | HP-5MS            | 1         | Automatic                      | 400             | Daily                     |
| 040             | 5                | 20           | LC                       | No       | MS       | QQQ              | Quattro Premier XE         | 10       | ACN         |                         | Reversed Phase    | 5         | Manual                         | 320             | Each Batch                |
| 041             | 0.0              |              | GC                       | No       | MS       | QQQ              | Variian 3800 GC+ 320-MS    | 10       | ACN         | DSPE(PSA)               | VF-1 ms           | 3         | Both                           | 150             | Each Batch                |
| 042             | 5                | Unit         | GC                       | No       | MS       | QQQ              | Variian 320MS              | 50       | Acetone     | L/L                     | DB5               | 8         | Both                           | 128             | Each Time                 |
| 044             | <3%              | >20%         | GC                       | No       | MS       | QQQ              | Agilent7000a               | 15       | Acetone     | ne                      | HP5MSI            | 2         | Automatic                      | 200             | Each batch                |
| 046             | -1.8             | 0.5 ppm      | LC                       | Yes      | MS       | Orbitrap         | Exactive                   | 10       | ACN         |                         | Atlantis T3       | 5         | Both                           | 461             | 100 compounds every batch |
| 047             | 0.1              |              | LC                       | No       | MS       | QQQ              | Agilent 6410               | 10       | ACN         | DSPE                    | C-18              | 10        | Automatic                      |                 | Each Batch                |
| 049             | 60               | 20%          | LC                       | No       | MS       | QQQ              | Variian 310-MS             | 10       | ACN         | DSPE                    | XDB-C18           | 20        | Automatic                      | 91              | Every 15 days             |
| 050             | -1.55            | 6            | GC                       | Yes      | MS       | Q                | 5975C inert XL EI/CI/MSD   | 10       | ACN         | PSA                     | HP5-MS            | 2         | Automatic                      | 200             | Always                    |
| 051             | 2.9              | 1.2          | GC                       | Yes      | MS       | IT               | Variian Saturn 2000        | 15       | Acetone     | Partitioning            | FactorFour VF-5ms | 5         | Both                           | 500             | Quarterly                 |
| 052             | 84%              | GC           | Yes                      | MS       | Q        | QQQ              | Thermo trace DSQ           | 10       | DCM         | Supelco SLB-5MS         | 1                 | Both      | 400                            | Never           |                           |
| 053             |                  |              | GC                       | Yes      | MS       | QQQ              | Varian                     | 7.5      | Acetone     | Yes                     | DB5 MS            | 1         | Automatic                      | 100             |                           |

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

| ATRAZINE        |                  |              |                          |           |          |                  |                                   |                    |                |             |                              |                  |                                |                         |                                    |
|-----------------|------------------|--------------|--------------------------|-----------|----------|------------------|-----------------------------------|--------------------|----------------|-------------|------------------------------|------------------|--------------------------------|-------------------------|------------------------------------|
| Laboratory Code | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Full Scan | Detector | Instrument Model | Sample Weight (g)                 | Extraction Solvent | Clean-Up       | Column Type | Injection Volume (μl)        | No. of Compounds | Frequency of Standard Solution |                         |                                    |
|                 |                  |              |                          |           |          |                  |                                   |                    |                |             |                              |                  |                                |                         |                                    |
| 001             | 60               | none         | LC                       | No        | MS       | QQQ              | API 4000                          | 10                 | ACN            | DSPE        | Waters-C18<br>2.1x50mmx1.7μm | 5                | Both                           | 600                     | every week 200 pesticides          |
| 003             | 0.30             | 0.05 Da      | LC                       | Yes       | MS       | TOF              | ACQUITY/LCT PREMIER XE            | 15                 | MINI LUKE      | No          | C18                          | 5                | Both                           | 345                     | Once a Day                         |
| 004             |                  |              | LC                       | No        | MS       | QQQ              | Applied Biosystems API5000        | 10                 | EtOAc          | Filter      | HSS T3                       | 2                | Both                           | 227                     | Each Batch                         |
| 005             |                  |              | LC                       | No        | MS       | QQQ              | Agilent 6460                      | 10                 | EtOAc          |             | C18                          | 2                | Automatic                      | 500                     | Each Batch                         |
| 006             | 0.03             |              | GC                       | No        | MS       | Q                | Agilent 6890/5973                 | 10                 | ACN            | DSPE        | HP-5MS                       | 1                | Manual                         | 177                     | Once a Month                       |
| 008             | 238              | 100          | LC                       | No        | MS       | Q-TRAP           | API 4000 Qtrap                    | 10                 | ACN            |             | Pursuit XRs Ultra            | 6                | Both                           | 250                     | Each Batch                         |
| 009             | 0.00             |              | LC                       | No        | MS       | QQQ              | Waters Aquity TQD                 | 10                 | Methanol       | Filter      | C18 UPLC                     | 6                | Automatic                      | 120                     |                                    |
| 010             |                  |              | LC                       | No        | MS       | QQQ              | Agilent 6410B                     | 10                 | ACN            | DSPE        | C18                          | 2                | Both                           | 182                     | Each Batch                         |
| 011             |                  |              | LC                       | No        | MS       | Q-TRAP           | ABl 4000                          | 10                 | ACN            |             | C18                          | 55               | Both                           | approx. 580             | Always                             |
| 012             | 30               | 20           | GC                       | Yes       | MS       | IT               | Saturn 4000 varian                | 10                 | ACN            | DSPE        | DB5MS                        | 1                | Both                           | 350                     | Daily                              |
| 013             | 14.8             | 96           | GC                       | Yes       | MS       | Q                | Agilent 5975                      | 10                 | ACN            | MgSO4/PSA   | HP5MS                        | 20               | Automatic                      | 550                     |                                    |
| 014             |                  |              | GC                       | Yes       | MS       | Q                | Trace DSQ                         | 15                 | ACN            | QuEChERS    | DB5-MS                       | 0.8              | Both                           |                         |                                    |
| 015             |                  |              | LC                       | No        | MS       | QQQ              | ThermoFinnigan TSQ Quantum        | 10                 | ACN            |             | XterraC18MS                  | 20               | Automatic                      | 130                     | On a Daily Basis                   |
| 016             |                  |              | LC                       | No        | MS       | QQQ              | Waters Premier Quattro XE         | 10                 | ACN            | PSA         | Kinetex XB-C18               | 10               | Both                           |                         | Targeted Analysis Only             |
| 017             | 0.0              | 4.06         | LC                       | No        | MS       | QQQ              | HPIC Agilent 1100,<br>MS API 3000 | 10                 | ACN            | DSPE        | C18 3μm 50x2mm               | 10               | Both                           | \$25 method 590 library | Always                             |
| 018             | 0.0              | 0.0          | LC                       | No        | MS       | QQQ              | Waters Xevo                       | 10                 | Acetone/PE/DCM | Na23O4      | C18                          | 2                | Automatic                      | 250                     | Each Batch                         |
| 019             | 9                | 30           | GC                       | Yes       | MS       | Q                | Agilent GC7890-M5975C             | 10                 | ACN            | DSPE        | HP5-MS                       | 1                | Both                           | 150 method 500 library  | Method Setup (and very rare cases) |
| 020             | <0.5             |              | GC                       | Yes       | MS       | IT               | Varian 4000                       | 10                 | ACN            | PSA         | 5 % diphenyl                 | 1                | Automatic                      | 148 method NIST library | Each Batch                         |

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

| ATRAZINE        |              |                          |           |          |                   |                          |                           |                      |                         |                       |                  |                                |                  |                           |            |
|-----------------|--------------|--------------------------|-----------|----------|-------------------|--------------------------|---------------------------|----------------------|-------------------------|-----------------------|------------------|--------------------------------|------------------|---------------------------|------------|
| Laboratory Code | MS Tolerance | Cromatographic Technique | Full Scan | Detector | Instrument Model  | Sample Weight (g)        | Extraction Solvent        | Clean-Up             | Column Type             | Injection Volume (μl) | No. of Compounds | Frequency of Standard Solution |                  |                           |            |
|                 |              |                          |           |          |                   |                          |                           |                      |                         |                       |                  |                                |                  |                           |            |
| 021             | GC           | Yes                      | MS        | IT       | Varian 4000       | 10                       | ACN                       | DSPE                 | Capillary               | 3                     | Automatic        | 450                            | Daily/Weekly     |                           |            |
| 022             | <0.1%        | LC                       | No        | MS       | Waters Aquity TQD | 5                        | ACN                       | DSPE                 | UPC-8EH C18             | 10                    | Both             | 340                            | Once a Day       |                           |            |
| 023             | 0.0          | LC                       | No        | MS/MS    | API 4000          | 10                       | ACN                       | DSPE                 | C18 hydro Phenomenex    | 40                    | Manual           |                                | Each Time        |                           |            |
| 024             | 6 s          | LC                       | No        | MS       | QQQQ              | 10                       | ACN                       | Freeze out/DSPE(PSA) | Synergie Fusion RP80A   | 8                     | Both             | 400                            | At least monthly |                           |            |
| 025             | 6            | GC                       | Yes       | MS       | TOF               | 10                       | ACN                       | DSPE(PSA)            | HP-5MS                  | 5                     | Both             | 650                            | Weekly           |                           |            |
| 028             |              | LC                       | No        | MS       | QQQQ              | 10                       | ACN                       | DSPE                 | C18                     | 5                     | Both             | 180                            |                  |                           |            |
| 030             |              | GC                       | Yes       | MS       | TOF               | 15                       | Acetone. DCM,PE           |                      | DB-5                    | 1                     | Automatic        |                                |                  |                           |            |
| 032             | RT locking   | GC                       | Yes       | MS       | GCMSD             | Agilent                  | 10                        | ACN                  | PSA                     | HP5                   | 10               | Both                           | > 600            | Always                    |            |
| 033             | 9            | 20%                      | GC        | Yes      | MS                | Q                        | Agilent GCMSD 7890A/5975C | 10                   | ACN                     | PSA. C18              | HP-5MS           | 1                              | Automatic        | 927                       | Each Batch |
| 034             | 60 ppm       | LC                       | Yes       | MS       | IT                | Agilent 6230             | 10                        | ACN                  | Zorbax eclipse plus C18 | 3                     | Automatic        | 517                            | Not for routine  |                           |            |
| 035             | 0.01         | GC                       | No        | MS       | QQQQ              | Waters Micromass         | 10                        | ACN                  | PSA                     | DB5                   | 5                | Automatic                      |                  | Daily                     |            |
| 037             |              | GC                       | Yes       | MS       | Q                 | Agilent 6890N            | 10                        | ACN                  | PSA                     | DB-5                  | 1                | Both                           | 80               |                           |            |
| 039             |              | GC                       | No        | MS       | QQQQ              | Agilent 7000B            | 10                        | ACN                  | DSPE                    | HP-5MS                | 1                | Automatic                      | 400              | Daily                     |            |
| 040             | 5            | 20 LC                    | No        | MS       | QQQQ              | Quattro Premier XE       | 10                        | ACN                  |                         | Reversed Phase        | 5                | Manual                         | 320              | Each Batch                |            |
| 041             | 0.0          | GC                       | No        | MS       | QQQQ              | Varian 3800 GC + 320-MS  | 10                        | ACN                  | DSPE(PSA)               | VF-1 ms               | 3                | Both                           | 150              | Each Batch                |            |
| 044             | <3%          | <20% GC                  | No        | MS       | QQQQ              | Agilent7000a             | 15                        | Acetone              | NO                      | HP5MSI                | 2                | Automatic                      | 200              | Each batch                |            |
| 046             | 1.2 ppm      | LC                       | Yes       | MS       | Orbitrap          | Excitive                 | 10                        | ACN                  |                         | Atlantis T3           | 5                | Both                           | 461              | 100 compounds every batch |            |
| 047             | 0.0          | LC                       | No        | MS       | QQQQ              | Agilent 6410             | 10                        | ACN                  | DSPE                    | C-18                  | 10               | Automatic                      |                  | Each Batch                |            |
| 049             | 60 20%       | LC                       | No        | MS       | QQQQ              | Varian 310-MS            | 10                        | ACN                  | DSPE                    | XDB-C18               | 20               | Automatic                      | 91               | Every 15 days             |            |
| 050             | 5.25 15.5    | GC                       | Yes       | MS       | Q                 | 5975C inert XL EI/CI MSD | 10                        | ACN                  | PSA                     | HP5-MS                | 2                | Automatic                      | 200              | Always                    |            |
| 051             | 0.2 0.6      | GC                       | Yes       | MS       | IT                | Varian Saturn 2000       | 15                        | Acetone              | Partitioning            | FactorFour VF-5ms     | 5                | Both                           | 500              | Quarterly                 |            |

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

| Instrument Configuration for Atrazine Analysis |                                |                   |                    |          |                                   |                       |                                   |
|--|--------------------------------|-------------------|--------------------|----------|-----------------------------------|-----------------------|-----------------------------------|
| Parameter                                      | Atrazine                       |                   |                    | Ammonium |                                   |                       |                                   |
|  | Method Name                    | Sample Weight (g) | Extraction Solvent | Clean-Up | Column Type                       | Injection Volume (μL) | Software                          |
| 052  | Atrazine                       | 0.1               | LC                 | No       | MS                                | 10                    | ACN                               |
| 053  | Ammonium                       | 0.2               | GC                 | No       | MS                                | 7.5                   | Acetone                           |
|  | RT Tolerance (s)               | 8                 | IC                 | Yes      | Varian                            | 10                    | QuEChERS                          |
|  | MS Tolerance                   | 0.5               | Full Scan          | Yes      | Supelco Ascentis Express RP-Amide | 180                   | Supelco Ascentis Express RP-Amide |
|  | Detector                       | 0.1               | Full Scan          | Yes      | DB5 MS                            | 1                     | Supelco Ascentis Express RP-Amide |
|  | Analyser                       | 0.05              | Full Scan          | Yes      | DB5 MS                            | 1                     | Supelco Ascentis Express RP-Amide |
|  | Instrument Model               | Varian            | Full Scan          | Yes      | DB5 MS                            | 1                     | Supelco Ascentis Express RP-Amide |
|  | Clean-Up                       | 0.05              | Full Scan          | Yes      | DB5 MS                            | 1                     | Supelco Ascentis Express RP-Amide |
|  | Extraction Solvent             | 0.05              | Full Scan          | Yes      | DB5 MS                            | 1                     | Supelco Ascentis Express RP-Amide |
|  | Column Type                    | 0.05              | Full Scan          | Yes      | DB5 MS                            | 1                     | Supelco Ascentis Express RP-Amide |
|  | Injection Volume (μL)          | 0.05              | Full Scan          | Yes      | DB5 MS                            | 1                     | Supelco Ascentis Express RP-Amide |
|  | Software                       | 0.05              | Full Scan          | Yes      | DB5 MS                            | 1                     | Supelco Ascentis Express RP-Amide |
|  | No. of Compounds               | 0.05              | Full Scan          | Yes      | DB5 MS                            | 1                     | Supelco Ascentis Express RP-Amide |
|  | Frequency of Standard Solution | 0.05              | Full Scan          | Yes      | DB5 MS                            | 1                     | Supelco Ascentis Express RP-Amide |
|  | Each Batch                     | 0.05              | Full Scan          | Yes      | DB5 MS                            | 1                     | Supelco Ascentis Express RP-Amide |

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

| Laboratory Code  | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Detector | Instrument Model | Extraction Solvent | Column Type                     | Injection Volume (lL) | No. of Compounds | Standard Solution Frequency (s) |
|------------------|------------------|--------------|--------------------------|----------|------------------|--------------------|---------------------------------|-----------------------|------------------|---------------------------------|
|                  |                  |              |                          |          |                  |                    |                                 |                       |                  |                                 |
| <b>BENALAXYL</b> |                  |              |                          |          |                  |                    |                                 |                       |                  |                                 |
| 001              | 60sec            | none         | LC                       | No       | MS               | QQQ                | API 4000                        | 10                    | ACN              | DSPE                            |
| 003              | 0.30             | 0.05 Da      | LC                       | Yes      | MS               | TOF                | ACQUITY/LCT PREMIER XE          | 15                    | MINI LUKE        | No                              |
| 004              |                  |              | LC                       | No       | MS               | QQQ                | Applied Biosystems API5000      | 10                    | EtOAc            | Filter                          |
| 005              |                  |              | LC                       | No       | MS               | QQQ                | Agilent 6460                    | 10                    | EtOAc            | C18                             |
| 006              | 0.06             |              | LC                       | No       | MS               | QQQ                | Agilent 6410                    | 10                    | ACN              | Eclipse XDB-C18                 |
| 008              | 1.110            | 946          | GC                       | Yes      | MS               | TOF                | LECO Pegasus IV                 | 10                    | ACN              | PSA                             |
| 009              | 0.00             |              | GC                       | Yes      | MS               | II                 | Thermo Polaris Q                | 10                    | ACN              | PSA                             |
| 010              |                  |              | GC                       | Yes      | MS               | Q                  | HP 5973 MSD                     | 10                    | ACN              | DSPE                            |
| 011              | 3.8              | 0.0          | GC                       | Yes      | MS               | Q                  | HP5975C                         | 10                    | ACN              | PSA                             |
| 012              | 30               | 20           | GC                       | Yes      | MS               | II                 | Saturn 4000 varian              | 10                    | ACN              | DSPE                            |
| 013              | 1.5              | 91           | GC                       | Yes      | MS               | Q                  | Agilent 5975                    | 10                    | ACN              | MgSO4/PSA                       |
| 014              |                  |              | LC                       | No       | MS               | QQQ                | Accuity UPLC Quattro Premier XE | 15                    | ACN              | QuEChERS                        |
| 015              | >70% match       |              | GC                       | Yes      | MS               | II                 | ThermoFinniganPolarisQ          | 10                    | ACN              | DB-5MS                          |
| 016              |                  |              | LC                       | No       | MS               | QQQ                | Waters Premier Quattro XE       | 10                    | ACN              | PSA                             |
| 017              | 0.0              | -5.97        | LC                       | No       | MS               | QQQ                | HPLC Agilent 1100. MS API 3000  | 10                    | ACN              | DSPE                            |
| 018              | 0.0              | 0.0          | GC                       | No       | MS               | QQQ                | Thermo TSQ Quantum GC           | 10                    | Acetone/PE/DCM   | Na2SO4                          |
| 019              | 9                | 30           | GC                       | Yes      | MS               | Q                  | Agilent GC 7890-MS5975C         | 10                    | ACN              | DSPE                            |
| 020              | <0.5             |              | GC                       | Yes      | MS               | II                 | Varian 4000                     | 10                    | ACN              | PSA                             |

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

| BENALAXYL        |                    |          |             |                       |                  |                                |                           |                  |                       |                         |                  |
|------------------|--------------------|----------|-------------|-----------------------|------------------|--------------------------------|---------------------------|------------------|-----------------------|-------------------------|------------------|
| Instrument Model | Extraction Solvent | Clean-Up | Column Type | Injection Volume (μl) | No. of Compounds | Frequency of Standard Solution | Software                  | Column Type      | Injection Volume (μl) | Sample Weight (g)       | RT Tolerance (s) |
| 021              | GC                 | Yes      | MS          | IT                    | Varian 4000      | 10                             | ACN                       | DSPE             | Capillary             | 3                       | Automatic        |
| 022              | <20%               | GC       | Yes         | MS                    | Q                | Agilent 7890A/5975C            | 15                        | ACN              | DSPE                  | HP5MS                   | 10               |
| 023              | 0.0                | LC       | No          | MS/MS                 | Q                | API 4000                       | 10                        | ACN              | DSPE                  | C18 hydro Phenomenex    | 40               |
| 024              | 2.4                | LC       | No          | MS                    | QQQ              | API4000                        | 10                        | ACN              | Freeze out/ DSPE(PSA) | Synergie Fusion RP80A   | 8                |
| 025              | 2                  | 12       | GC          | Yes                   | MS               | TOF                            | LECO Pegasus IV           | 10               | ACN                   | DSPE(PSA)               | HP-5MS           |
| 026              | 0.0                | 0.0      | LC          | No                    | MS               | QQQ                            | Xevo TQ                   | 15               | ACN                   | LL.E                    | C18              |
| 028              |                    | LC       | No          | MS                    | QQQ              | Quattro Premier                | 10                        | ACN              | DSPE                  | C18                     | 5                |
| 029              |                    | GC       | Yes         | MS                    | IT               | Varian GCmass 4000             | 10                        | ACN              | DSPE(MgSO4)           | Capillary db 5          | 5                |
| 030              |                    | GC       | Yes         | MS                    | TOF              | Leco Pegasus                   | 15                        | Acetone, DCM, PE | DB-5                  | 1                       | Automatic        |
| 032              | 0.5                | 5 ppm    | LC          | Yes                   | MS               | Orbitrap                       | Exactive                  | 10               | ACN                   | PSA                     | C18              |
| 033              | 9                  | 20%      | GC          | Yes                   | MS               | Q                              | Agilent GCMSD 7890A/5975C | 10               | ACN                   | PSA, C18                | 5                |
| 034              | 60                 | 10 ppm   | LC          | Yes                   | MS               | IT                             | Agilent 6230              | 10               | ACN                   | Zorbax eclipse plus C18 | 3                |
| 035              | 0.005 Da           | 0.005 Da | LC          | Yes                   | MS               | Q-TOF                          | Bruker Maxis              | 10               | ACN                   | PSA                     | C18 poroshell    |
| 037              |                    | GC       | Yes         | MS                    | Q                | Agilent 8890N                  | 10                        | ACN              | PSA                   | DB-5                    | 1                |
| 039              |                    | GC       | No          | MS                    | QQQ              | Agilent 7000B                  | 10                        | ACN              | DSPE                  | HP-5MS                  | 1                |
| 040              | 5                  | 20       | LC          | No                    | MS               | QQQ                            | Quattro Premier XE        | 10               | ACN                   | Reversed Phase          | 5                |
| 041              | 0.5                |          | GC          | No                    | MS               | QQQ                            | Varian 3800 GC+ 320-MS    | 10               | ACN                   | DSPE(PSA)               | VF-1 ms          |
| 042              | 5                  | unit     | GC          | No                    | MS               | QQQ                            | Varian 320MS              | 50               | Acetone               | DB5                     | 8                |
| 044              | <3%                | <20%     | GC          | No                    | MS               | QQQ                            | Agilent7000a              | 15               | Acetone               | ne                      | HP5MSI           |
| 045              | 0.0                | 0.0      | GC          | Yes                   | MS               | Q                              | Agilent                   | 50               | Acetone               | DCM                     | 1                |
| 046              | 0.0                | 1.2 ppm  | LC          | Yes                   | MS               | Orbitrap                       | Exactive                  | 10               | ACN                   | Atlantis T3             | 5                |
| 047              | 0.02               |          | GC          | No                    | QQQ              | Varian 1200L                   | 10                        | ACN              | DSPE                  | DB-5                    | 5                |

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

| BENALAXYL       |                  |              |                          |           |              |                          |                                   |                   |                   |
|-----------------|------------------|--------------|--------------------------|-----------|--------------|--------------------------|-----------------------------------|-------------------|-------------------|
| Laboratory Code | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Full Scan | Detector     | Analyser                 | Instrument Model                  | Sample Weight (g) | Column Type       |
| 050             | 0.2              | LC           | No                       | QQQ       | Agilent 6410 | 10                       | ACN                               | DSPE              | C-18              |
| 051             | 0.85             | 1            | GC                       | MS        | Q            | 5975C Inert XL EI/CI MSD | 10                                | ACN               | PSA               |
| 052             | 6.1              | 0.8          | GC                       | Yes       | MS           | IT                       | Varian Saturn 2000                | 15                | Acetone           |
|                 | 6                | 92%          | LC                       | No        | MS           | QQQ                      | API 4000                          | 10                | ACN               |
|                 |                  |              |                          |           |              |                          | QuEChERS                          |                   | FactorFour VF-5ms |
|                 |                  |              |                          |           |              |                          | Supelco Ascentis express RP-Amide |                   | Partitioning      |
|                 |                  |              |                          |           |              |                          |                                   | 10                | Automatic         |
|                 |                  |              |                          |           |              |                          |                                   | 180               | Both              |
|                 |                  |              |                          |           |              |                          |                                   |                   | 500               |
|                 |                  |              |                          |           |              |                          |                                   |                   | 200               |
|                 |                  |              |                          |           |              |                          |                                   |                   | Always            |
|                 |                  |              |                          |           |              |                          |                                   |                   | Each Batch        |
|                 |                  |              |                          |           |              |                          |                                   |                   | Each Batch        |
|                 |                  |              |                          |           |              |                          |                                   |                   | Quarterly         |
|                 |                  |              |                          |           |              |                          |                                   |                   |                   |

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

| CARBOPHENOTHION |                  |              |                  |                    |          |             |   |                  |                |                                |                              |         |
|-----------------|------------------|--------------|------------------|--------------------|----------|-------------|---|------------------|----------------|--------------------------------|------------------------------|---------|
| Laboratory Code | RT Tolerance (s) | MS Tolerance | Instrument Model | Extraction Solvent | Clean-Up | Column Type | Injection Volume (μl)                           | No. of Compounds | Software       | Frequency of Standard Solution | Frequency of 200 pesticides  |         |
|                 |                  |              |                  |                    |          |             |   |                  |                |                                |                              |         |
| 001             | 12               | none         | LC               | No                 | MS       | QQQ         | API 4000  | 10               | ACN            | DSPE                           | Waters-C18<br>2.1x50mmx1.7μm |         |
| 004             |                  |              | LC               | No                 | MS       | QQQ         | Applied Biosystems API5000                      | 10               | EtOAc          | Filter                         | HSS T3                       |         |
| 005             |                  |              | LC               | No                 | MS       | QQQ         | Agilent 6460                                    | 10               | EtOAc          |                                | C18                          |         |
| 006             | 0.01             |              | GC               | No                 | MS       | Q           | Agilent 6890/5973                               | 10               | ACN            | DSPE                           | HP-5MS                       |         |
| 009             | 0.00             |              | GC               | Yes                | MS       | IT          | Thermo Polaris Q                                | 10               | ACN            | PSA                            | Capillary GC                 |         |
| 010             |                  |              | GC               | Yes                | MS       | Q           | HP5973 MSD                                      | 10               | ACN            | DSPE                           | HP-5MSI                      |         |
| 011             | 4.4              | 0.0          | GC               | Yes                | MS       | Q           | HP5975C   | 10               | ACN            | PSA                            | HP5MS                        |         |
| 012             | 30               | 20           | GC               | Yes                | MS       | IT          | Saturn 4000 varian                              | 10               | ACN            | DSPE                           | DB5MS                        |         |
| 013             | 5.6              | 95           | GC               | Yes                | MS       | Q           | Agilent 5975                                    | 10               | ACN            | MgSO4/PSA                      | HP5MS                        |         |
| 014             |                  |              | GC               | Yes                | MS       | Q           | Trace DSQ                                       | 15               | ACN            | QuEChERS                       | DB5-MS                       |         |
| 015             | >70% match       | GC           | Yes              | MS                 | IT       |             | ThermoFinniganPolarisQ                          | 10               | ACN            |                                | DB-5MS                       |         |
| 016             |                  |              | GC               | Yes                | MS       | Q           |   | 5973             | 10             | ACN                            | PSA                          | DB-35ms |
| 017             | 0.0              | 5.00         | GC               | No                 | MS       | Q           | GC Shimadzu GC-2010, MS<br>Shimadzu GCMS-QP2010 | 10               | ACN            | DSPE                           | HP-5MS                       | 3       |
| 018             | 0.0              | 0.0          | GC               | No                 | MS       | QQQ         | Thermo TSQ Quantum GC                           | 10               | Acetone/PE/DCM | Na2SO4                         | Rxi-5SiL MS                  | 1       |
| 019             | 9                | 30           | GC               | Yes                | MS       | Q           | Agilent GC7890-N5975C                           | 10               | ACN            | DSPE                           | HP5-MS                       | 1       |
| 021             |                  |              | GC               | Yes                | MS       | IT          | Varian 4000                                     | 10               | ACN            | DSPE                           | Capillary                    | 3       |
| 022             | <0.2             | <20%         | GC               | Yes                | MS       | Q           | Agilent 7890A/5975C                             | 15               | ACN            | DSPE                           | HP5MS                        | 10      |
|                 |                  |              |                  |                    |          |             |   |                  | Both           |                                | Once a Day                   |         |

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

| CARBOPHENOTHION |                  |              |                           |          |                  |                           |          |             |                 |                      |                                |           |
|-----------------|------------------|--------------|---------------------------|----------|------------------|---------------------------|----------|-------------|-----------------|----------------------|--------------------------------|-----------|
| Laboratory Code | RT Tolerance (s) | MS Tolerance | Chromatographic Technique | Detector | Instrument Model | Extraction Solvent        | Clean-Up | Column Type | Software        | No. of Compounds     | Frequency of Standard Solution |           |
|                 |                  |              |                           |          |                  |                           |          |             |                 |                      |                                |           |
| 025             | 1                | 10.6         | GC                        | Yes      | MS TOF           | LECO Pegasus IV           | 10       | ACN         | DSPE(PSA)       | HP-5MS               | 5                              | Both      |
| 026             | 0.0              | 0.0          | GC                        | Yes      | MS IT            | Polidis Q                 | 25       | Hexane      | LL              | DB5 MS               | 1                              | Both      |
| 028             |                  |              | LC                        | No       | MS QQQ           | Quattro Premier           | 10       | ACN         | DSPE            | C18                  | 5                              | Both      |
| 029             |                  |              | GC                        | Yes      | MS IT            | Varian GCmasse 4000       | 10       | ACN         | DSPE (MgSO4)    | Capillary db 5       | 5                              | Both      |
| 032             | RT locking       | GC           | Yes                       | MS GCMSD | Agilent          | 10                        | ACN      | PSA         | HP5             | 10                   | Both                           |           |
| 033             | 9                | 20%          | GC                        | Yes      | MS Q             | Agilent GCMSD 7890A/5975C | 10       | ACN         | PSA, C18        | HP-5MS               | 1                              | Automatic |
| 034             | 12               |              | GC                        | Yes      | MS IT            | Saturn 2000               | 30       | EtOAc       | HPGPC           | DB-5MS               | 2                              | Automatic |
| 035             | 0.01             |              | GC                        | No       | MS QQQ           | Waters Micromass          | 10       | ACN         | PSA             | DB5                  | 5                              | Automatic |
| 037             |                  |              | GC                        | Yes      | MS Q             | Agilent 6890N             | 10       | ACN         | PSA             | DB-5                 | 1                              | Both      |
| 040             | 5                | 20           | LC                        | No       | MS QQQ           | Quattro Premier XE        | 10       | ACN         |                 | Reversed Phase       | 5                              | Manual    |
| 041             | 0.0              |              | GC                        | Yes      | MS QQQ           | Varian 3800 GC + 320-MS   | 10       | ACN         | DSPE(PSA)       | VF-1 ms              | 3                              | Both      |
| 042             | 5                | unit         | GC                        | No       | MS QQQ           | Varian 320MS              | 50       | Acetone     | L/L             | DB5                  | 8                              | Both      |
| 045             | 0.0              | 0.0          | GC                        | Yes      | MS Q             | Agilent                   | 50       | Acetone     | DCM             |                      | 1                              | Both      |
| 046             | <20              | 919          | GC                        | Yes      | MS TOF           | Leco                      | 10       | ACN         | DSPE(PSA)       | Restek CI Pesticides | 10                             | Both      |
| 050             | 1.65             | 1            | GC                        | Yes      | MS Q             | 5975C inert XL EI/CI MSD  | 10       | ACN         | PSA             | HP5-MS               | 2                              | Automatic |
| 051             | 4.8              | 3.5          | GC                        | Yes      | MS IT            | Varian Saturn 2000        | 15       | Acetone     | Partitioning    | FactorFour VF-5ms    | 5                              | Both      |
| 052             | 92%              | GC           | Yes                       | MS Q     | Thermo trace DSQ | 10                        | DCM      |             | Supelco SLB-5MS | 1                    | Both                           |           |

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

| CHINOMETHIONAT  |                  |                          |           |                        |  |                    |                   |                                  |                       |
|-----------------|------------------|--------------------------|-----------|------------------------|--|--------------------|-------------------|----------------------------------|-----------------------|
| Laboratory Code | RT Tolerance (s) | Cromatographic Technique | Detector  | Instrument Model       | Sample Weight (g)                            | Extraction Solvent | Clean-Up          | Column Type                      | No. of Compounds      |
|                 |                  |                          |           |                        |  |                    |                   |                                  | Injection Volume (µl) |
|                 |                  |                          |           |                        |  |                    |                   | Software                         | Solution Standard     |
| 001             | none             | none                     | LC        | No MS QQQQ             | API 4000                                     | 10 ACN             | DSPE              | Waters-C18 2.1x50mmx1.7µm        | 5 Both                |
| 004             |                  |                          | GC        | No MS QQQQ             | Waters                                       | 10 EtOAc           | Filter            | Rxi-5Sil MS/integra-guard Restek | 10 Both               |
| 005             |                  |                          | GC        | No MS QQQQ             | Varian 1200L GC3800                          | 10 EtOAc           | FactorFour VF-5ms | 2 Automatic                      | 500 Each Batch        |
| 008             | 916              | 924                      | GC        | Yes MS TOF             | LECO Pegasus IV                              | 10 ACN             | PSA               | Phenomenex ZB-5MS                | 3 Both                |
| 010             |                  |                          | GC        | Yes MS Q               | HP 5973 MSD                                  | 10 ACN             | DSPE              | HP 5MSI                          | 20 Both               |
| 011             | 4.6              | 0.0                      | GC        | Yes MS Q               | HP5975C                                      | 10 ACN             | PSA               | HP5MS                            | 5 Both                |
| 012             | 30               | 20                       | GC        | Yes MS IT              | Saturn 4000 varian                           | 10 ACN             | DSPE              | DB5MS                            | 1 Both                |
| 013             | 15.2             | 93                       | GC        | Yes MS Q               | Agilent 5975                                 | 10 ACN             | MgSO4/PSA         | HP5MS                            | 20 Automatic          |
| 014             |                  |                          | GC        | Yes MS Q               | Trace DSQ                                    | 15 ACN             | QuICkERS          | DB5-MS                           | 0.8 Both              |
| 015             | >70% match       | GC                       | Yes MS IT | ThermoFinniganPolarisQ |  | 10 ACN             |                   | DB-5MS                           | 8 Both                |
| 017             | -0.6             | 1.00                     | GC        | No MS Q                | GC Shimadzu GC-2010. MS Shimadzu GCMS-QP2010 | 10 ACN             | DSPE              | HP-5MS                           | 3 Both                |
| 018             | 0.0              | 0.0                      | GC        | No MS QQQQ             | Thermo TSQ Quantum GC                        | 10 Acetone/FE/DCM  | Na2SO4            | RXi-5SiL MS                      | 1 Automatic           |
| 020             | <0.5             |                          | GC        | Yes MS IT              | Variian 4000                                 | 10 ACN             | PSA               | 5 % diphenyl                     | 1 Automatic           |
| 021             |                  |                          | GC        | Yes MS IT              | Variian 4000                                 | 10 ACN             | DSPE              | Capillary                        | 3 Automatic           |
| 022             | <0.2             | <20%                     | GC        | Yes MS Q               | Agilent 7890A/5975C                          | 15 ACN             | DSPE              | HP5MS                            | 10 Both               |
| 025             | 3                | 18.1                     | GC        | Yes MS TOF             | LECO Pegasus IV                              | 10 ACN             | DSPE(PSA)         | HP-5MS                           | 5 Both                |
| 026             | 0.0              | 0.0                      | GC        | Yes MS IT              | Polaris Q                                    | 25 Hexane          | LLE               | DB5 MS                           | 1 Both                |
|                 |                  |                          |           |                        |  |                    |                   |                                  | 150 Each Time         |

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

| CHINOMETHIONAT  |                  |                           |               |          |                          |                   |                    |                      |              |                       |                                |
|-----------------|------------------|---------------------------|---------------|----------|--------------------------|-------------------|--------------------|----------------------|--------------|-----------------------|--------------------------------|
| Laboratory Code | MS Tolerance (s) | Chromatographic Technique | Full Scan     | Detector | Analyser                 | Instrument Model  | Extraction Solvent | Clean-Up             | Column Type  | Injection Volume (µl) | No. of Compounds               |
|                 |                  |                           |               |          |                          |                   |                    |                      |              |                       |                                |
| 028             |                  | GC                        | No MS QQQ     |          | Agilent                  | Quattro micro     | 10 ACN             | DSPE                 | HP5_MS       | 1 Both                | 110                            |
| 032             | RI locking       | GC                        | Yes MS/GC/MSD |          |                          |                   | 10 ACN             | PSA                  | HP5          | 10 Both               | > 600 Always                   |
| 033             | 9                | 20%                       | GC Yes MS Q   |          | Agilent                  | GCMSD 7890A/5975C | 10 ACN             | PSA, C18             | HP-5MS       | 1 Automatic           | 927 Each Batch                 |
| 035             | 0.01             |                           | GC No MS QQQ  |          |                          | Waters Micromass  | 10 ACN             | PSA                  | DB5          | 5 Automatic           | Daily                          |
| 040             | 5                | 10                        | GC Yes MS TOF |          | GCT Premier              | EI/OAC            | 12.5 GPC           | Capillary            | 1 Both       | NIST                  |                                |
| 041             | 0.5              |                           | GC No MS QQQ  |          | Varian 3800 GC+ 320-MS   | 10 ACN            | DSPE(PSA)          | VF-1 ms              | 3 Both       | 150                   | Each Batch                     |
| 045             | 0.0              | 0.0                       | GC Yes MS Q   |          | Agilent                  | 50 Acetone        | DCM                |                      | 1 Both       | 220                   | Daily                          |
| 046             | <20              | 960                       | GC Yes MS TOF |          | Leco                     | 10 ACN            | DSPE(PSA)          | Restek CI Pesticides | 10 Both      | 700                   | 235 compounds with every batch |
| 050             | 0.15             | 2                         | GC Yes MS Q   |          | 5975C Inert XL EI/CI MSD | 10 ACN            | PSA                | HP5-MS               | 2 Automatic  | 200                   | Always                         |
| 051             | 8.8              | 13.1                      | GC Yes MS IT  |          | Varian Saturn 2000       | 15 Acetone        | Partitioning       | FactorFour VF-5ms    | 5 Both       | 500                   | Quarterly                      |
| 052             | 90%              | GC Yes MS Q               |               |          | Thermo trace DSQ         | 10 DCM            |                    | Supelco SLB-5MS      | 1 Both       | 400                   | Never                          |
| 053             |                  | LC Yes MS QQQ             |               |          | Varian                   | 12 ACN            | Yes                | C18                  | 10 Automatic | 70                    |                                |

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

| CHLOZOLINATE    |                  |              |                          |                    |                  |                   |   |          |                                |
|-----------------|------------------|--------------|--------------------------|--------------------|------------------|-------------------|---|----------|--------------------------------|
| Laboratory Code | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Detector Full Scan | Instrument Model | Sample Weight (g) | Extraction Solvent                          | Clean-up | Column Type                    |
|                 |                  |              |                          |                    |                  |                   |   |          | Injection Volume (μl)          |
|                 |                  |              |                          |                    |                  |                   |   |          | No. of Compounds               |
|                 |                  |              |                          |                    |                  |                   |   |          | Solution Frequency of Standard |
| 004             |                  | GC           | No                       | MS                 | QQQ              |                   | Waters                                      | 10       | EtOAc                          |
| 005             |                  | GC           | No                       | MS                 | QQQ              |                   | Varien 1200L GC3800                         | 10       | EtOAc                          |
| 006             | 0.01             | GC           | No                       | MS                 | Q                |                   | Agilent 6890/5973                           | 10       | ACN                            |
| 008             | 853              | GC           | Yes                      | MS                 | TQF              |                   | LECO Pegasus IV                             | 10       | ACN                            |
| 010             |                  | GC           | Yes                      | MS                 | Q                |                   | HP 5973 MSD                                 | 10       | ACN                            |
| 011             | 3.6              | 0.0          | GC                       | Yes                | MS               | Q                 | HP 5975C                                    | 10       | ACN                            |
| 012             | 30               | 20           | GC                       | Yes                | MS               | II                | Saturn 4000 varian                          | 10       | ACN                            |
| 013             | 8.5              | 92           | GC                       | Yes                | MS               | Q                 | Agilent 5975                                | 10       | ACN                            |
| 014             |                  | GC           | Yes                      | MS                 | Q                |                   | Trace DSQ                                   | 15       | ACN                            |
| 015             | >70% match       | GC           | Yes                      | MS                 | II               |                   | ThermoFinniganPolarisQ                      | 10       | ACN                            |
| 016             |                  | GC           | Yes                      | MS                 | Q                |                   | 5973  | 10       | ACN                            |
| 017             | 0.0              | 3.00         | GC                       | No                 | MS               | Q                 | GC Shimadzu GC-2010 MS Shimadzu GCMS-QP2010 | 10       | ACN                            |
| 018             | 0.0              | 0.0          | GC                       | No                 | MS               | QQQ               | Thermo TSQ Quantum GC                       | 10       | Acetone/PE/DCM                 |
| 019             | 9                | 30           | GC                       | Yes                | MS               | Q                 | Agilent GC7890-NSS5975C                     | 10       | ACN                            |
| 020             | <0.5             |              | GC                       | Yes                | MS               | II                | Varian 4000                                 | 10       | ACN                            |
| 021             |                  | GC           | Yes                      | MS                 | II               |                   | Varian 4000                                 | 10       | ACN                            |
| 022             | <20%             | GC           | Yes                      | MS                 | Q                |                   | Agilent 7890A/5975C                         | 15       | ACN                            |
| 025             | 5                | 96           | GC                       | Yes                | MS               | TOF               | LECO Pegasus IV                             | 10       | ACN                            |
| 026             | 0.0              | 0.0          | GC                       | Yes                | MS               | II                | Polidis Q                                   | 25       | Hexane                         |

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

| CHLOZOLINATE    |                  |                          |           |          |                  |                   |                           |          |                  |                 |
|-----------------|------------------|--------------------------|-----------|----------|------------------|-------------------|---------------------------|----------|------------------|-----------------|
| Laboratory Code | MS Tolerance (s) | Cromatographic Technique | Full Scan | Detector | Instrument Model | Sample Weight (g) | Extraction Solvent        | Clean-Up | Column Type      | Software        |
|                 |                  |                          |           |          |                  |                   |                           |          |                  |                 |
|                 |                  |                          |           |          |                  |                   |                           |          |                  |                 |
| 028             |                  | GC                       | No        | MS       | QQQ              |                   | Quattro micro             | 10       | ACN              | DSPF            |
| 030             |                  | GC                       | Yes       | MS       | TOF              |                   | Leco Pegasus              | 15       | Acetone, DCM, PE | DB-5            |
| 032             | RT locking       | GC                       | Yes       | MS       | GCMSD            |                   | Agilent                   | 10       | ACN              | PSA             |
| 033             | 9                | 20%                      | GC        | Yes      | MS               | Q                 | Agilent GCMSD 7890A/5975C | 10       | ACN              | PSA, C18        |
| 034             | 12               | GC                       | Yes       | MS       | IT               |                   | Saturn 2000               | 30       | EtOAc            | HGPC            |
| 037             |                  | GC                       | Yes       | MS       | Q                |                   | Agilent 6890N             | 10       | ACN              | PSA             |
| 039             |                  | GC                       | No        | MS       | QQQ              |                   | Agilent 7000B             | 10       | ACN              | DSPF            |
| 040             | 5                | 10                       | GC        | Yes      | MS               | TOF               | GCT Premier               | 12.5     | EtOAc            | GPC             |
| 041             | 0.6              |                          | GC        | Yes      | MS               | QQQ               | Variian 3800 GC + 320-MS  | 10       | ACN              | DSPF(PSA)       |
| 044             | <3%              | <20%                     | GC        | No       | MS               | QQQ               | Agilent7000a              | 15       | Acetone          | re              |
| 045             | 0.0              | 0.0                      | GC        | Yes      | MS               | Q                 | Agilent                   | 50       | Acetone          | DCM             |
| 046             | <20              | 964                      | GC        | Yes      | MS               | TOF               | Leco                      | 10       | ACN              | DSPF(PSA)       |
| 050             | 0.95             | 1                        | GC        | Yes      | MS               | Q                 | 5975Cinert XL EI/CI MSD   | 10       | ACN              | PSA             |
| 051             | 5.3              | 3.1                      | GC        | Yes      | MS               | IT                | Varian Saturn 2000        | 15       | Acetone          | Partitioning    |
| 052             | 85%              | GC                       | Yes       | MS       | Q                |                   | Thermo trace DSQ          | 10       | DCM              | FctFour VF-5ms  |
| 053             |                  | GC                       | Yes       | MS       | QQQ              |                   | Varian                    | 7.5      | Acetone          | Supelco SLB-5MS |
|                 |                  |                          |           |          |                  |                   |                           |          |                  | 1 Automatic     |
|                 |                  |                          |           |          |                  |                   |                           |          |                  | 100             |

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

| Laboratory Code | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Full Scan | Detector | Analyser  | Instrument Model               | Excitation Solvent | Clean-Up       | ETRIMFOS              |                              |           | No. of Compounds | Frequency of Standard Solution |                           |
|-----------------|------------------|--------------|--------------------------|-----------|----------|-----------|--------------------------------|--------------------|----------------|-----------------------|------------------------------|-----------|------------------|--------------------------------|---------------------------|
|                 |                  |              |                          |           |          |           |                                |                    |                | Injection Volume (μl) | Column Type                  | Software  |                  |                                |                           |
| 001             | 12               | none         | LC                       | No        | MS       | QQQ       | API 4000                       | 10                 | ACN            | DSPE                  | Waters-C18 2.1x50mmx1.7 Å μm | 5         | Both             | 600                            | Every week 200 pesticides |
| 003             | 0.30             | 0.05 Da      | LC                       | Yes       | MS       | TOF       | ACQUITY/LCT PREMIER XE         | 15                 | MINI LUKE      | No                    | C18                          | 5         | Both             | 345                            | Once a Day                |
| 004             |                  |              | LC                       | No        | MS       | QQQ       | Applied Biosystems API5000     | 10                 | EtOAC          | Filter                | HSS T3                       | 2         | Both             | 227                            | Each Batch                |
| 005             |                  |              | GC                       | No        | MS       | QQQ       | Varian 1200L GC-3800           | 10                 | EtOAC          | FactorFour, VF-5ms    | 2                            | Automatic | 500              | Each Batch                     |                           |
| 006             | 0.01             |              | LC                       | No        | MS       | QQQ       | Agilent 6460                   | 10                 | EtOAC          |                       | C18                          | 2         | Automatic        | 500                            | Each Batch                |
| 008             | 673              | 957          | GC                       | Yes       | MS       | TOF       | Agilent 6890/5973              | 10                 | ACN            | DSPE                  | HP-5MS                       | 1         | Manual           | 177                            | Once a Month              |
| 009             | 0.00             |              | GC                       | Yes       | MS       | IT        | LECO Pegasus IV                | 10                 | ACN            | PSA                   | Phenomenex ZB-5MS            | 3         | Both             | 750                            | Each Batch                |
| 010             |                  |              | GC                       | Yes       | MS       | Q         | Thermo Polaris Q               | 10                 | ACN            | PSA                   | Capillary GC                 | 1         | Automatic        | 130                            |                           |
| 011             | 0.5              | 0.0          | GC                       | Yes       | MS       | Q         | HP 5973 MSD                    | 10                 | ACN            | DSPE                  | HP-5MSI                      | 20        | Both             | 111                            | Each Batch                |
| 012             | 30               | 20           | GC                       | Yes       | MS       | IT        | LECO Pegasus IV                | 10                 | ACN            | PSA                   | HP5MS                        | 5         | Both             | approx. 1.000                  | Always                    |
| 013             | -5.4             | 97           | GC                       | Yes       | MS       | Q         | Saturn 4000 varian             | 10                 | ACN            | DSPE                  | DB5MS                        | 1         | Both             | 350                            | Daily                     |
| 014             |                  |              | GC                       | Yes       | MS       | Q         | Agilent 5975                   | 10                 | ACN            | MgSO4/PSA             | HP5MS                        | 20        | Automatic        | 550                            |                           |
| 015             | >70% match       | GC           | Yes                      | MS        | IT       | Trace DSQ | 15                             | ACN                | QUEChERS       | DB5-MS                | 0.8                          | Both      |                  |                                |                           |
| 016             |                  |              | GC                       | Yes       | MS       | Q         | ThermoFinniganPolarisQ         | 10                 | ACN            | PSA                   | DB-5MS                       | 8         | Both             | 500                            | Often                     |
| 017             | 0.0              | 9.27         | LC                       | No        | MS       | QQQ       | HPLC Agilent 1100, MS API 3000 | 10                 | ACN            | DSPE                  | C18 3μm 50x2mm               | 10        | Both             | 325 method library             | Always                    |
| 018             | 0.0              | 0.0          | GC                       | No        | MS       | QQQ       | Thermo TSQ Quantum GC          | 10                 | Acetone/PE/DCM | Na2SO4                | RX-5SiL MS                   | 1         | Automatic        | 164                            | Each Batch                |
| 021             |                  |              | GC                       | Yes       | MS       | IT        | Varian 4000                    | 10                 | ACN            | DSPE                  | Capillary                    | 3         | Automatic        | 450                            | Daily/Weekly              |
| 022             | <0.2             | <20%         | GC                       | Yes       | MS       | Q         | Agilent 7890A/5975C            | 15                 | ACN            | DSPE                  | HP5MS                        | 10        | Both             | 214                            | Once a Day                |
| 023             | 0.0              |              | LC                       | No        | MS/MS    | Q         | API 4000                       | 10                 | ACN            | DSPE                  | C18 hydro Phenomenex         | 40        | Manual           |                                | Each Time                 |
| 025             | 3                | 11.8         | GC                       | Yes       | MS       | TOF       | LECO Pegasus IV                | 10                 | ACN            | DSPE(PSA)             | HP-5MS                       | 5         | Both             | 650                            | Weekly                    |

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

| Laboratory Code | MS Tolerance (s) | MS Tolerance | Chromatographic Technique | Full Scan | Detector | Instrument Model | Sample Weight (g)         | Extraction Solvent | Clean-Up         | Column Type     | Injection Volume (μl) |      | No. of Compounds | Software | Frequency of Standard Solution |
|-----------------|------------------|--------------|---------------------------|-----------|----------|------------------|---------------------------|--------------------|------------------|-----------------|-----------------------|------|------------------|----------|--------------------------------|
|                 |                  |              |                           |           |          |                  |                           |                    |                  |                 | Inj                   | Time |                  |          |                                |
| <b>ETRIAFOS</b> |                  |              |                           |           |          |                  |                           |                    |                  |                 |                       |      |                  |          |                                |
| 026             | 0.0              | 0.0          | LC                        | No        | MS       | QQQ              | Xevo TQ                   | 15                 | ACN              | LLE             | C18                   | 10   | Both             | 360      | Each Time                      |
| 028             |                  |              | GC                        | No        | MS       | QQQ              | Quattro micro             | 10                 | ACN              | DSPE            | HP5MSI                | 1    | Both             | 110      |                                |
| 030             |                  |              | GC                        | Yes       | MS       | TOF              | Leco Pegasus              | 15                 | Acetone, DCM, PE |                 | DB-5                  | 1    | Automatic        |          |                                |
| 032             | 0.5              | 5 ppm        | LC                        | Yes       | MS       | Orbitrap         | Exactive                  | 10                 | ACN              | PSA             | C18                   | 5    | Both             | > 200    | Always                         |
| 033             | 9                | 20%          | GC                        | Yes       | MS       | Q                | Agilent GCMSD 7890A/5975C | 10                 | ACN              | PSA, C18        | HP-5MS                | 1    | Automatic        | 927      | Each Batch                     |
| 034             | 12               |              | GC                        | Yes       | MS       | IT               | Saturn 2000               | 30                 | EiOAC            | HGPC            | DB-5MS                | 2    | Automatic        | 380      | Not for routine                |
| 035             | 0.005 Da         |              | LC                        | Yes       | MS       | Q-TOF            | Bruker Maxis              | 10                 | ACN              | PSA             | C18 poroshell         | 1    | Automatic        | 2000     |                                |
| 037             |                  |              | GC                        | Yes       | MS       | Q                | Agilent 6890N             | 10                 | ACN              | PSA             | DB-5                  | 1    | Both             | 80       |                                |
| 039             |                  |              | LC                        | No        | MS       | QQQ              | API 4000Q                 | 10                 | ACN              | DSPE            | C18                   | 5    | Automatic        | 400      | Daily                          |
| 040             | 5                | 20           | LC                        | No        | MS       | QQQ              | Quattro Premier XE        | 10                 | ACN              |                 | Reversed Phase        | 5    | Manual           | 320      | Each Batch                     |
| 041             | 0.0              |              | GC                        | Yes       | MS       | QQQ              | Varian 3800 GC + 320-MS   | 10                 | ACN              | DSPE(PSA)       | VF-1 ms               | 3    | Both             | 500      | Each Batch                     |
| 042             | 5                | unit         | GC                        | No        | MS       | QQQ              | Varian 320MS              | 50                 | Acetone          | I/I             | DB5                   | 8    | Both             | 128      | Each Time                      |
| 044             | <3%              | <20%         | GC                        | No        | MS       | QQQ              | Agilent 7000a             | 15                 | Acetone          | ne              | HP5MSI                | 2    | Automatic        | 200      | Each batch                     |
| 045             | 0.0              | 0.0          | GC                        | Yes       | MS       | Q                | Agilent                   | 50                 | Acetone          | DCM             |                       | 1    | Both             | 220      | Daily                          |
| 046             | <20              | 91.5         | GC                        | Yes       | MS       | TOF              | Leco                      | 10                 | ACN              | DSPE(PSA)       | Restek CI Pesticides  | 10   | Both             | 700      | 235 compounds with every batch |
| 047             | 0.1              | 3.7 ppm      | LC                        | Yes       | TOF      |                  | Agilent 6220              | 10                 | ACN              | DSPE            | C-18                  | 10   | Automatic        | 800      | Each Batch                     |
| 050             | -1.35            | 3            | GC                        | Yes       | MS       | Q                | 5975C inert XL EI/CI MSD  | 10                 | ACN              | PSA             | HP5-MS                | 2    | Automatic        | 200      | Always                         |
| 051             | 3.8              | 0.8          | GC                        | Yes       | MS       | IT               | Varian Saturn 2000        | 15                 | Acetone          | Partitioning    | FactorFour VF-5ms     | 5    | Both             | 500      | Quarterly                      |
| 052             | 96%              |              | GC                        | Yes       | MS       | Q                | Thermo Trace DSQ          | 10                 | DCM              | Supelco SLB-5MS |                       | 1    | Both             | 400      | Never                          |
| 053             |                  |              | GC                        | Yes       | MS       | QQQ              | Varian                    | 7.5                | Acetone          | Yes             | DB5 MS                | 1    | Automatic        | 100      |                                |

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

| Laboratory Code | RT Tolerance (s) | Cromatographic Technique | Full Scan | Detector | Instrument Model | Extraction Solvent | Clean-Up                       | Column Type | Software       | No. of Compounds      | Frequency of Standard Solution | FENAMIDONE            |                  |                        |
|-----------------|------------------|--------------------------|-----------|----------|------------------|--------------------|--------------------------------|-------------|----------------|-----------------------|--------------------------------|-----------------------|------------------|------------------------|
|                 |                  |                          |           |          |                  |                    |                                |             |                |                       |                                | Injection Volume (µl) | No. of Compounds |                        |
| 001             | 60               | none                     | LC        | No       | MS               | QQQ                | API 4000                       | 10          | ACN            | DSPE                  | Waters-C18 2.1x50mmx1.7µm      | 5                     | Both             | 600                    |
| 004             |                  |                          | LC        | No       | MS               | QQQ                | Applied Biosystems API5000     | 10          | EtOAC          | Filter                | HSS T3                         | 2                     | Both             | 104                    |
| 005             |                  |                          | LC        | No       | MS               | QQQ                | Agilent 6460                   | 10          | EtOAC          |                       | C18                            | 2                     | Automatic        | 500                    |
| 006             | 0.02             | GC                       | No        | MS       | Q                | Agilent 6890/5973  | 10                             | ACN         | DSPE           | HP-5MS                | 1                              | Manual                | 177              |                        |
| 008             | 1238             | 935                      | GC        | Yes      | MS               | TOF                | LECO Pegasus IV                | 10          | ACN            | PSA                   | Phenomenex ZB-5MS              | 3                     | Both             | 750                    |
| 010             |                  |                          | LC        | No       | MS               | QQQ                | Agilent 6410B                  | 10          | ACN            | DSPE                  | C18                            | 2                     | Both             | 182                    |
| 011             |                  |                          | LC        | No       | MS               | Q-TRAP             | ABI 4000                       | 10          | ACN            |                       | C18                            | 55                    | Both             | approx. 580            |
| 012             | 30               | 20                       | GC        | Yes      | MS               | IT                 | Saturn 4000 varian             | 10          | ACN            | DSPE                  | DB5MS                          | 1                     | Both             | 350                    |
| 013             | 10.3             | 9.6                      | GC        | Yes      | MS               | Q                  | Agilent 5975                   | 10          | ACN            | MgSO4/PSA             | HP5MS                          | 20                    | Automatic        | 550                    |
| 016             |                  |                          | LC        | No       | MS               | QQQ                | Waters Premier Quattro XE      | 10          | ACN            | PSA                   | Kinetex XB-C18                 | 10                    | Both             | Targeted Analysis Only |
| 017             | 0.0              | -15.43                   | LC        | No       | MS               | QQQ                | HPLC Agilent 1100. MS API 3000 | 10          | ACN            | DSPE                  | C18 3µm 50x2mm                 | 10                    | Both             | 525 method 590 library |
| 018             | 0.0              | 0.0                      | LC        | No       | MS               | QQQ                | Waters Xevo                    | 10          | Acetone/PE/DCM | Na2SO4                | C18                            | 2                     | Automatic        | 250                    |
| 020             | <2.5             |                          | LC        | No       | MS               | Q-TRAP             | 3200 Qtrap                     | 10          | ACN            | PSA                   | C18                            | 50                    | Automatic        | library : 500          |
| 022             | <0.1             | <15%                     | LC        | No       | MS               | QQQ                | Waters Aquity TQD              | 15          | ACN            | DSPE                  | UPLC-BEH C18                   | 10                    | Both             | 340                    |
| 023             | 0.0              |                          | LC        | No       | MS/MS            | Q                  | API 4000                       | 10          | ACN            | DSPE                  | C18 hydro Phenomenex           | 40                    | Manual           | Once a Day             |
| 024             | 3                |                          | LC        | No       | MS               | QQQ                | API4000                        | 10          | ACN            | Freeze out/ DSPE(PSA) | Synergie Fusion RP80A          | 8                     | Both             | 400                    |
| 025             | 2                | 8.1                      | GC        | Yes      | MS               | TOF                | LECO Pegasus IV                | 10          | ACN            | DSPE(PSA)             | HP-5MS                         | 5                     | Both             | 650                    |
| 026             | 0.0              | 0.0                      | LC        | No       | MS               | QQQ                | Xevo TQ                        | 15          | ACN            | LLE                   | C18                            | 10                    | Both             | 360                    |
| 028             |                  |                          | LC        | No       | MS               | QQQ                | Quattro Premier                | 10          | ACN            | DSPE                  | C18                            | 5                     | Both             | 180                    |
| 032             | RT locking       | GC                       | Yes       | MS       | GCMSD            | Agilent            | 10                             | ACN         | PSA            | HP5                   | 10                             | Both                  | >600             | Always                 |

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

| Laboratory Code   | RT Tolerance (s) | Chromatographic Technique | Full Scan | Instrument Model | Extraction Solvent | Clean-Up     | Column Type                | Software | No. of Compounds | Frequency of Standard Solution    |
|-------------------|------------------|---------------------------|-----------|------------------|--------------------|--------------|----------------------------|----------|------------------|-----------------------------------|
|                   |                  |                           |           |                  |                    |              |                            |          |                  |                                   |
| <b>FENAMIDONE</b> |                  |                           |           |                  |                    |              |                            |          |                  |                                   |
| 033               | 9                | 20%                       | GC        | Yes              | MS                 | Q            | Agilent GC/MSD 7890A/5975C | 10       | ACN              | PSA, C18                          |
| 034               | 60               | 10 ppm                    | LC        | Yes              | MS                 | IT           | Agilent 6230               | 10       | ACN              | Zorbax eclipse plus C18           |
| 035               |                  | 0.005 Da                  | LC        | Yes              | MS                 | Q-TOF        | Bruker Maxis               | 10       | ACN              | C18 poroshell                     |
| 039               |                  |                           | LC        | No               | MS                 | QQQ          | API 4000Q/T                | 10       | ACN              | PSA                               |
| 042               | 6                | unit                      | LC        | No               | MS                 | QQQ          | Waters Premier             | 10       | Methanol         | DSPE                              |
| 044               | <3%              | <20%                      | GC        | No               | MS                 | QQQ          | Agilent 7000a              | 15       | Acetone          | ne                                |
| 045               | 0.0              | 0.0                       | GC        | Yes              | MS                 | Q            | Agilent                    | 50       | Acetone          | DCM                               |
| 046               | 1.2              | 1.0 ppm                   | LC        | Yes              | MS                 | Orbitrap     | Exactive                   | 10       | ACN              | Atlantis T3                       |
| 047               | 0.0              |                           | LC        | No               | QQQ                | Agilent 6410 | 10                         | ACN      | DSPE             | C-18                              |
| 051               | 1.2              | 2.3                       | LC        | No               | MS                 | QQQ          | Waters Premier XE          | 15       | Acetone          | Partitioning                      |
| 052               | 4                |                           | LC        | No               | MS                 | QQQ          | API 4000                   | 10       | ACN              | QuEChERS                          |
| 053               |                  |                           | LC        | Yes              | MS                 | QQQ          | Varian                     | 12       | ACN              | Supelco Ascentis express RP-Amide |
|                   |                  |                           |           |                  |                    |              | Yes                        |          | C18              | 10                                |
|                   |                  |                           |           |                  |                    |              |                            |          | Automatic        | 70                                |

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

| FLUCYTHIRNATE   |                  |                          |          |                  |                   |                        |   |          |                              |                          |
|-----------------|------------------|--------------------------|----------|------------------|-------------------|------------------------|---|----------|------------------------------|--------------------------|
| Laboratory Code | RT Tolerance (s) | Cromatographic Technique | Detector | Instrument Model | Sample Weight (g) | Clean-Up               | Column Type                                 | Software | No. of Compounds             |                          |
| 001             | 60               | none                     | LC       | No               | MS                | QQQ                    | ACN   | DSPE     | Waters-C18<br>2.1x50mmx.7Åµm |                          |
| 004             |                  |                          | LC       | No               | MS                | QQQ                    | Applied Biosystems API5000                  | 10       | EtOAC                        |                          |
| 005             |                  |                          | LC       | No               | MS                | QQQ                    | Agilent 6460                                | 10       | EtOAC                        |                          |
| 006             | 0.02             | GC                       | No       | MS               | Q                 | Agilent 6890/5973      | 10  | ACN      | DSPE                         |                          |
| 008             | 1528             | 964                      | GC       | Yes              | MS                | TOF                    | LECO Pegasus IV                             | 10       | ACN                          |                          |
| 009             | 0.00             | GC                       | No       | MS               | QQQ               | Thermo XLS             | 10  | ACN      | PSA                          |                          |
| 010             |                  | GC                       | Yes      | MS               | Q                 | HP 5973 MSD            | 10  | ACN      | DSPE                         |                          |
| 011             | 7.5              | 0.0                      | GC       | Yes              | MS                | Q                      | HP5975C                                     | 10       | ACN                          | PSA                      |
| 012             | 30               | 20                       | GC       | Yes              | MS                | IT                     | Saturn 4000 varian                          | 10       | ACN                          | DSPE                     |
| 013             | -14.8            | 86                       | GC       | Yes              | MS                | Q                      | Agilent 5975                                | 10       | ACN                          | MgSO4/PSA                |
| 014             |                  | GC                       | Yes      | MS               | Q                 | Trace DSQ              | 15  | ACN      | QUEChERS                     |                          |
| 015             | >70%             | GC                       | Yes      | MS               | IT                | ThermoFinniganPolarisQ | 10  | ACN      | DB5-MS                       |                          |
| 016             |                  | GC                       | Yes      | MS               | Q                 | Agilent 5973 MSD       | 10  | ACN      | PSA                          |                          |
| 017             | 0.6              | 3.00                     | GC       | No               | MS                | Q                      | GC Shimadzu GC-2010-MS Shimadzu GCMS-QP2010 | 10       | ACN                          | DSPE                     |
| 018             | 0.0              | 0.0                      | GC       | No               | MS                | QQQ                    | Thermo TSQ Quantum GC                       | 10       | Acetone/PE/DCM               | Na23O4                   |
| 019             | 9                | 30                       | GC       | Yes              | MS                | Q                      | Agilent GC7890A-MS5975C                     | 10       | ACN                          | DSPE                     |
| 021             |                  | GC                       | Yes      | MS               | IT                |                        | Varian 4000                                 | 10       | ACN                          | DSPE                     |
| 022             | <0.2             | <20%                     | GC       | Yes              | MS                | Q                      | Agilent 7890A/5975C                         | 15       | ACN                          | DSPE                     |
| 023             | 0.0              |                          | LC       | No               | MS/MS             | Q                      | API 4000                                    | 10       | ACN                          | DSPE                     |
| 024             |                  |                          | GC       | No               | MS                | QQQ                    | TSQ Thermo                                  | 10       | ACN                          | Freeze out/<br>DSPE/PSA) |
| 025             | 4                | 17.6                     | GC       | Yes              | MS                | TOF                    | LECO Pegasus IV                             | 10       | ACN                          | DSPE/PSA)                |
|                 |                  |                          |          |                  |                   |                        | HP-5MS                                      | 2        | Both                         | 450                      |
|                 |                  |                          |          |                  |                   |                        | HP-5MS                                      | 5        | Both                         | 650                      |
|                 |                  |                          |          |                  |                   |                        |   |          |                              | Weekly                   |

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

| Laboratory Code | RT Tolerance (s) | Chromatographic Technique | MS Tolerance | Full Scan | Detector | Analyser                | FLUCYTHIRINATE            |          | Column Type | Software                | No. of Compounds     | Frequency of standard Solution |           |
|-----------------|------------------|---------------------------|--------------|-----------|----------|-------------------------|---------------------------|----------|-------------|-------------------------|----------------------|--------------------------------|-----------|
|                 |                  |                           |              |           |          |                         | Sample Weight (g)         | Clean-Up |             |                         |                      |                                |           |
| 026             | 0.0              | GC                        | Yes          | MS        | IT       | Polaris Q               | 25                        | Hexane   | LE          | DB5 MS                  | 1                    | Both                           |           |
| 028             |                  | GC                        | Yes          | MS        | Q        | HP-5973N                | 10                        | ACN      | DSPE        | HP5MSI                  | 1                    | Both                           |           |
| 032             | RT locking       | GC                        | Yes          | MS        | GCMSD    | Agilent                 | 10                        | ACN      | PSA         | HP5                     | 10                   | Both                           |           |
| 033             | 9                | 20%                       | GC           | Yes       | MS       | Q                       | Agilent GCMSD 7890A/5975C | 10       | ACN         | PSA, C18                | HP-5MS               | 1                              | Automatic |
| 034             | 60               | 10                        | LC           | Yes       | MS       | TOF                     | Agilent 6230              | 10       | ACN         | Zorbax eclipse plus C18 | 3                    | Automatic                      |           |
| 035             | 0.01             | GC                        | No           | MS        | QQQ      | Waters Micromass        | 10                        | ACN      | PSA         | DB5                     | 5                    | Automatic                      |           |
| 037             |                  | GC                        | Yes          | MS        | Q        | Agilent 6890N           | 10                        | ACN      | PSA         | DB-5                    | 1                    | Both                           |           |
| 039             |                  | GC                        | No           | MS        | QQQ      | Agilent 7000B           | 10                        | ACN      | DSPE        | HP-5MS                  | 1                    | Automatic                      |           |
| 040             | 5                | 10                        | GC           | Yes       | MS       | TOF                     | GCT Premier               | 12.5     | EtOAc       | GPC                     | Capillary            | 1                              | Both      |
| 041             | 0.5              | GC                        | Yes          | MS        | QQQ      | Varian 3800 GC + 320-MS | 10                        | ACN      | DSPE(PSA)   | VF-I ms                 | 3                    | Both                           |           |
| 042             | 5                | unit                      | GC           | No        | MS       | QQQ                     | Varian 320MS              | 50       | Acetone     | I/L                     | DB5                  | 8                              | Both      |
| 044             | < 3%             | < 20%                     | GC           | No        | MS       | QQQ                     | Agilent 7000a             | 15       | Acetone     | ne                      | HP5MSI               | 2                              | Automatic |
| 045             | 0.0              | 0.0                       | GC           | Yes       | MS       | Q                       | Agilent                   | 50       | Acetone     | DCM                     |                      | 1                              | Both      |
| 046             | <30              | 954                       | GC           | Yes       | MS       | TOF                     | Leco                      | 10       | ACN         | DSPE(PSA)               | Restek CI Pesticides | 10                             | Both      |
| 047             | 0.04             |                           | GC           | No        | QQQ      | Varian 1200L            | 10                        | ACN      | DSPE        | DB-5                    | 5                    | Automatic                      |           |
| 047             | 0.1              |                           | LC           | No        | QQQ      | Agilent 6410            | 10                        | ACN      | DSPE        | C-18                    | 10                   | Automatic                      |           |
| 050             | 1.72             | 13                        | GC           | Yes       | MS       | Q                       | 5975C Inert XL EI/CI MSD  | 10       | ACN         | PSA                     | HP5MS                | 2                              | Automatic |
| 051             | 0.8              | 2.9                       | GC           | Yes       | MS       | IT                      | Varian Saturn 2000        | 15       | Acetone     | Partitioning            | FactorFour VF-5ms    | 5                              | Both      |
| 053             |                  |                           | GC           | No        | MS       | QQQ                     | Varian                    | 7.5      | Acetone     | Yes                     | DB5 MS               | 1                              | Automatic |
|                 |                  |                           |              |           |          |                         |                           |          |             |                         |                      | 100                            |           |

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

| Laboratory Code | MS Tolerance (s) | Cromatographic Technique | Detector | Analyser | FONOFOS          |                   |  | No. of Compounds | Frequency of Standard Solution |
|-----------------|------------------|--------------------------|----------|----------|------------------|-------------------|--|------------------|--------------------------------|
|                 |                  |                          |          |          | Instrument Model | Sample Weight (g) | Clean-Up                                     | Column Type      |                                |
| 001             | 12               | none                     | LC       | No       | MS               | QQQ               | API 4000                                     | 10               | ACN                            |
| 003             | 0.30             | 0.05 Da                  | LC       | Yes      | MS               | TOF               | ACQUITY/LCT PREMIER XE                       | 15               | MINI LUKE                      |
| 004             |                  |                          | LC       | No       | MS               | QQQ               | AB API5000                                   | 10               | EIOAC                          |
| 005             |                  |                          | LC       | No       | MS               | QQQ               | Agilent 6460                                 | 10               | EIOAC                          |
| 006             | 0.02             |                          | GC       | No       | MS               | Q                 | Agilent 6890/5973                            | 10               | ACN                            |
| 008             | 660              | 947                      | GC       | Yes      | MS               | TOF               | LECO Pegasus IV                              | 10               | ACN                            |
| 009             | 0.00             |                          | GC       | Yes      | MS               | IT                | Thermo Polaris Q                             | 10               | ACN                            |
| 010             |                  |                          | GC       | Yes      | MS               | Q                 | HP 5973 MSD                                  | 10               | ACN                            |
| 011             | 0.2              | 0.0                      | GC       | Yes      | MS               | Q                 | HP 5975C                                     | 10               | ACN                            |
| 012             | 30               | 20                       | GC       | Yes      | MS               | IT                | Saturn 4000 varian                           | 10               | ACN                            |
| 013             | -3.0             | 96                       | GC       | Yes      | MS               | Q                 | Agilent 5975                                 | 10               | ACN                            |
| 014             |                  |                          | GC       | Yes      | MS               | Q                 | Trace DSQ                                    | 15               | ACN                            |
| 016             |                  |                          | GC       | Yes      | MS               | Q                 | 5973   | 10               | ACN                            |
| 017             | 0                | 1.00                     | GC       | No       | MS               | Q                 | GC Shimadzu GC-2010, MS Shimadzu GCMS-QP2010 | 10               | ACN                            |
| 018             | 0                | 0%                       | LC       | No       | MS               | QQQ               | Waters Xevo                                  | 10               | Acetone/PE/DCM                 |
| 019             | 9                | 30                       | GC       | Yes      | MS               | Q                 | Agilent GC7890-M5975C                        | 10               | ACN                            |
| 020             | < 0.5            | %                        | GC       | Yes      | MS               | IT                | Varian 4000                                  | 10               | ACN                            |
| 021             |                  |                          | GC       | Yes      | MS               | IT                | Varian 4000                                  | 10               | ACN                            |
| 022             | <0.1             | <15%                     | LC       | No       | MS               | QQQ               | Waters Aquity TQD                            | 15               | ACN                            |
| 023             | 0                |                          | LC       | No       | MS/MS            | Q                 | API 4000                                     | 10               | ACN                            |

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

| FONOFOS            |                  |                          |           |          |          |                  |                           |          |             |                       |                   |
|--------------------|------------------|--------------------------|-----------|----------|----------|------------------|---------------------------|----------|-------------|-----------------------|-------------------|
| Labradoratory Code | MS Tolerance (s) | Cromatographic Technique | Full Scan | Detector | Analyser | Instrument Model | Extraction Solvent        | Clean-Up | Column Type | Injection Volume (µl) | No. of Compounds  |
|                    |                  |                          |           |          |          |                  |                           |          |             |                       |                   |
| 025                | 3                | 9.7                      | GC        | Yes      | MS       | TOF              | Pegasus IV                | 10       | ACN         | DSPE(PSA)             | HP-5MS            |
| 026                | 0                | 0%                       | LC        | No       | MS       | QQQ              | Xevo TQ                   | 15       | ACN         | LLE                   | C18               |
| 028                |                  | GC                       | Yes       | MS       | Q        |                  | HP-5973N                  | 10       | ACN         | DSPE                  | HP5MSI            |
| 029                |                  | GC                       | Yes       | MS       | IT       |                  | Varian GCmasse 4000       | 10       | ACN         | DSPE (MgSO4)          | Capillary db 5    |
| 032                | RT locking       | GC                       | Yes       | MS       | GCMSD    | Agilent          | 10                        | ACN      | PSA         | HP5                   | 10                |
| 033                | 9                | 20%                      | GC        | Yes      | MS       | Q                | Agilent GCMSD 7890A/5975C | 10       | ACN         | PSA, C18              | HP-5MS            |
| 034                | 12               |                          | GC        | Yes      | MS       | TOF              | Saturn 2000               | 30       | EIOAC       | HGPC                  | DB-5 MS           |
| 037                |                  |                          | GC        | Yes      | MS       | Q                | Agilent 3890N             | 10       | ACN         | PSA                   | DB-5              |
| 039                |                  |                          | GC        | No       | MS       | QQQ              | Agilent 7000B             | 10       | ACN         | DSPE                  | HP-5MS            |
| 040                | 5                | 20                       | LC        | No       | MS       | QQQ              | Quattro Premier XE        | 10       | ACN         |                       | Reversed phase    |
| 041                | 0.5              |                          | GC        | No       | MS       | QQQ              | Varian 3800 GC+ 320-MS    | 10       | ACN         | DSPE (PSA)            | VF-1 ms           |
| 042                | 5                | unit                     | GC        | No       | MS       | QQQ              | VARIAN 320MS              | 50       | Acetone     | L/L                   | DB5               |
| 044                | <3%              | <20%                     | GC        | No       | MS       | QQQ              | Agilent 7000a             | 15       | Acetone     | HP5MSI                | 2                 |
| 045                | 0                | 0                        | GC        | Yes      | MS       | Q                | Agilent                   | 50       | Acetone     | DCM                   | 0                 |
| 046                | -0.6             | 0.7 ppm                  | LC        | Yes      | MS       | Orbitrap         | Exactive                  | 10       | ACN         |                       | Atlantis T3       |
| 050                | -0.95            | 5                        | GC        | Yes      | MS       | Q                | 5975C Inert XL EI/CI MSD  | 10       | ACN         | PSA                   | HP5-MS            |
| 051                | 2.6              | 3.1                      | GC        | Yes      | MS       | IT               | Varian Saturn 2000        | 15       | Acetone     | Partitioning          | FactorFour VF-5ms |
| 052                | 9.6%             | GC                       | Yes       | MS       | Q        |                  | Thermo trace DSQ          | 10       | DCM         | No                    | Supelco SLB-5MS   |
| 053                |                  | LC                       | Yes       | MS       | QQQ      |                  | Varian                    | 12       | ACN         | Yes                   | C18               |

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

| FORCHLORFENURON |                  |              |                           |           |          |          |                            |                   |                |                         |                             |                                |
|-----------------|------------------|--------------|---------------------------|-----------|----------|----------|----------------------------|-------------------|----------------|-------------------------|-----------------------------|--------------------------------|
| Laboratory Code | RT Tolerance (s) | MS Tolerance | Chromatographic Technique | Full Scan | Detector | Analyser | Instrument Model           | Sample Weight (g) | Column Type    | Injection Volume (µl)   | No. of Compounds            | Frequency of Standard Solution |
|                 |                  |              |                           |           |          |          |                            |                   |                |                         |                             |                                |
| 001             | 60               | none         | LC                        | No        | MS       | QQQ      | API 4000                   | 10                | ACN            | DSPE                    | Waters C18 2.1x50mmx1.7Å µm | 5                              |
| 004             |                  |              | LC                        | No        | MS       | QQQ      | Applied Biosystems API5000 | 10                | EtOAc          | Filter                  | HSS T3                      | 2                              |
| 005             |                  |              | LC                        | No        | MS       | QQQ      | Agilent 6460               | 10                | EtOAc          | C18                     | C18                         | 2                              |
| 011             |                  |              | LC                        | No        | MS       | Q-TRAP   | AB 4000                    | 10                | ACN            |                         |                             | Automatic                      |
| 013             |                  |              | LC                        | No        | MS       | QQQ      | Applied 3200 Qtrap         | 10                | ACN            | C18                     | 55                          |                                |
| 018             | 0.0              | 0.0          | LC                        | No        | MS       | QQQ      | Waters Xevo                | 10                | Acetone/PE/DCM | Na2SO4                  | Atlantis T3                 | 10                             |
| 022             | <0.1             | <15%         | LC                        | No        | MS       | QQQ      | Waters Aquity TQD          | 15                | ACN            | DSPE                    | UPLC-BEH C18                | 10                             |
| 028             |                  |              | LC                        | No        | MS       | QQQ      | Quattro Premier            | 10                | ACN            | DSPE                    | C18                         | 5                              |
| 034             | 60               | 10 ppm       | LC                        | Yes       | MS       | TOF      | Agilent 6230               | 10                | ACN            | Zorbax eclipse plus C18 | 3                           |                                |
| 046             | 8.4              | 3.5 ppm      | LC                        | Yes       | MS       | Orbitrap | Exactive                   | 10                | ACN            |                         | Atlantis T3                 | 5                              |
| 050             | -0.006           | -3.84        | LC                        | No        | MS       | QQQ      | G6410A                     | 10                | ACN            | PSA                     | Zorbax-Eclipse XDB-C8       | 20                             |
|                 |                  |              |                           |           |          |          |                            |                   |                | Automatic               | 106                         | Always                         |

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

| MECARBAM        |                  |              |                          |           |          |                        |                                |                    |                |                        |                            |
|-----------------|------------------|--------------|--------------------------|-----------|----------|------------------------|--------------------------------|--------------------|----------------|------------------------|----------------------------|
| Laboratory Code | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Full Scan | Detector | Instrument Model       | Sample Weight (g)              | Extraction Solvent | Clean-up       | Column Type            | No. of Compounds           |
|                 |                  |              |                          |           |          |                        |                                |                    |                |                        | Injection Volume (μl)      |
| 001             | 60               | none         | LC                       | No        | MS       | QQQ                    | API 4000                       | 10                 | ACN            | DSPE                   | Waters-C18 2.1x50mmx1.7Åum |
| 003             | 0.30             | 0.05 Da      | LC                       | Yes       | MS       | TOF                    | ACQUITY/LCT PREMIER XE         | 15                 | MINI LUKE      | No                     | C18                        |
| 004             |                  |              | LC                       | No        | MS       | QQQ                    | Applied Biosystems API5000     | 10                 | EIOAc          | Filter                 | HSS 13                     |
| 005             |                  |              | LC                       | No        | MS       | QQQ                    | Agilent 6460                   | 10                 | EIOAc          |                        | C18                        |
| 006             | 0.01             | GC           | No                       | MS        | Q        | Agilent 6890/5973      | 10                             | ACN                | DSPE           | HP-5MS                 | 1                          |
| 008             | 864              | 978          | GC                       | Yes       | MS       | TOF                    | LECO Pegasus IV                | 10                 | ACN            | PSA                    | Phenomenex ZB-5MS          |
| 009             | 0.00             |              | LC                       | No        | MS       | QQQ                    | Waters Aquity TQD              | 10                 | Methanol       | Filter                 | C18 UPLC                   |
| 010             |                  |              | LC                       | No        | MS       | QQQ                    | Agilent 6410B                  | 10                 | ACN            | DSPE                   | C18                        |
| 011             | 2.4              | 0.0          | GC                       | Yes       | MS       | Q                      | HP5975C                        | 10                 | ACN            | PSA                    | HP5MS                      |
| 012             | 30               | 20           | GC                       | Yes       | MS       | IT                     | Saturn 4000 varian             | 10                 | ACN            | DSPE                   | DB5MS                      |
| 013             | -0.4             | 93           | GC                       | Yes       | MS       | Q                      | Agilent 5975                   | 10                 | ACN            | MgSO <sub>4</sub> /PSA | HP5MS                      |
| 014             |                  |              | LC                       | No        | MS       | QQQ                    | Acuity UPC Quattro Premier XE  | 15                 | ACN            | QuECHERS               | BEHC 18 1.7 μm             |
| 015             | > 70%            | GC           | Yes                      | MS        | IT       | ThermoFinniganPolarisQ | 10                             | ACN                |                | DB-5MS                 | 8                          |
| 016             |                  |              | GC                       | Yes       | MS       | Q                      |                                | 5973               | 10             | ACN                    | PSA                        |
| 017             | 0.0              | -6.10        | LC                       | No        | MS       | QQQ                    | HPLC Agilent 1100, MS API 3000 | 10                 | ACN            | DSPE                   | C18 3μm 50x2mm             |
| 018             | 0.0              | 0.0          | GC                       | No        | MS       | QQQ                    | Thermo TSQ Quantum GC          | 10                 | Acetone/PE/DCM | Na25O4                 | RXi-5SIL MS                |
| 019             | 9                | 30           | GC                       | Yes       | MS       | Q                      | Agilent GC7890-M5975C          | 10                 | ACN            | DSPE                   | HP-5MS                     |
| 021             |                  |              | GC                       | Yes       | MS       | IT                     |                                | Varian 4000        | 10             | ACN                    | DSPE                       |
| 022             | <0.1             | <15%         | LC                       | No        | MS       | QQQ                    | Waters Aquity TQD              | 15                 | ACN            | DSPE                   | UPLC-BEH C18               |
| 023             | 0.0              |              | LC                       | No        | MS/MS    | Q                      |                                | API 4000           | 10             | ACN                    | DSPE                       |
| 024             | 1.2              |              | LC                       | No        | MS       | QQQ                    |                                | API4000            | 10             | ACN                    | Freeze out/DSPE(PSA)       |
| 025             | 3                | 6.9          | GC                       | Yes       | MS       | TOF                    | LECO Pegasus IV                | 10                 | ACN            | DSPE(PSA)              | HP-5MS                     |
| 026             | 0.0              | 0.0          | LC                       | No        | MS       | QQQ                    | Xevo TQ                        | 15                 | ACN            | LLE                    | C18                        |
| 028             |                  |              | GC                       | No        | MS       | QQQ                    | Quattro micro                  | 10                 | ACN            | DSPE                   | HFS_MSi                    |

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

| MECARBAM        |                  |              |           |                  |                   |                     |                           |                  |                          |                         |
|-----------------|------------------|--------------|-----------|------------------|-------------------|---------------------|---------------------------|------------------|--------------------------|-------------------------|
| Laboratory Code | RT Tolerance (s) | MS Tolerance | Technique | Instrument Model | Sample Weight (g) | Extraction Solvent  | Clean-Up                  | Column Type      | Injection Volume (μl)    | No. of Compounds        |
|                 |                  |              |           |                  |                   |                     |                           |                  |                          |                         |
|                 |                  |              |           |                  |                   |                     |                           |                  |                          |                         |
| 029             |                  | GC           | Yes       | MS               | IT                | Variian GCmass 4000 | 10                        | ACN              | DSPE(MgSO <sub>4</sub> ) | Capillary db 5          |
| 030             |                  | GC           | Yes       | MS               | TOF               | Leco Pegasus        | 15                        | Acetone, DCM, PE | DB-5                     | 5 Both                  |
| 032             | 0.5              | 5 ppm        | LC        | Yes              | MS                | Orbitrap            | Exactive                  | 10               | ACN                      | PSA                     |
| 033             | 9                | 20%          | GC        | Yes              | MS                | Q                   | Agilent GCMSD 7890A/5975C | 10               | ACN                      | FSA, C18                |
| 034             | 60               | 10 ppm       | LC        | Yes              | MS                | TOF                 | Agilent 6230              | 10               | ACN                      | Zorbax eclipse plus C18 |
| 035             | 0.005 Da         | LC           | Yes       | MS               | Q-TOF             | Bruker Maxis        | 10                        | ACN              | PSA                      | C18 poroshell           |
| 037             |                  | GC           | Yes       | MS               | Q                 | Agilent 6890N       | 10                        | ACN              | PSA                      | DB-5                    |
| 039             |                  | LC           | No        | MS               | QQQ               | API 4000QT          | 10                        | ACN              | DSPE                     | C18                     |
| 040             | 5                | 20           | LC        | No               | MS                | QQQ                 | Quattro Premier XE        | 10               | ACN                      | Reversed Phase          |
| 041             | 0.5              |              | GC        | Yes              | MS                | QQQ                 | Variian 3800 GC + 320-MS  | 10               | ACN                      | DSPE(PSA)               |
| 042             | 5                | unit         | GC        | No               | MS                | QQQ                 | Variian 320MS             | 50               | Acetone                  | L/L                     |
| 044             | <3%              | <20%         | GC        | No               | MS                | QQQ                 | Agilent 7000a             | 15               | Acetone                  | ne                      |
| 045             | 0.0              | 0.0          | GC        | Yes              | MS                | Q                   | Agilent                   | 50               | Acetone                  | DCM                     |
| 046             | 1.2              | 0.3 ppm      | LC        | Yes              | MS                | Orbitrap            | Exactive                  | 10               | ACN                      | Atlantis T3             |
| 047             | 0.1              |              | LC        | No               | MS                | QQQ                 | Agilent 6410              | 10               | ACN                      | DSPE                    |
| 050             | -0.004           | -5.31        | LC        | No               | MS                | QQQ                 | G6410A                    | 10               | ACN                      | PSA                     |
| 051             | 0.6              | 0.1          | LC        | No               | MS                | QQQ                 | Waters Premier XE         | 15               | Acetone                  | Zorbax-Eclipse XDB-C8   |
| 052             | 6                | 93%          | LC        | No               | MS                | QQQ                 | API 4000                  | 10               | ACN                      | UPLC Acuity BEH         |
| 053             |                  |              | LC        | Yes              | MS                | QQQ                 | Varian                    | 12               | ACN                      | QuECHERS                |
|                 |                  |              |           |                  |                   |                     |                           |                  | Yes                      | C18                     |

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

| MEVINPHOS       |                  |              |                          |                        |                                |                   |                    |           |                                    |
|-----------------|------------------|--------------|--------------------------|------------------------|--------------------------------|-------------------|--------------------|-----------|------------------------------------|
| Laboratory Code | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Full Scan              | Instrument Model               | Sample Weight (g) | Extraction Solvent | Clean-Up  | Column Type                        |
| Software        |                  |              |                          |                        |                                |                   |                    |           |                                    |
| 001             | 60               | none         | LC No                    | QQQ                    | API 4000                       | 10                | ACN                | DSPE      | Waters-C18 2.1x50mmx1.7 $\mu$ m    |
| 003             | 0.30             | 0.05 Da      | LC Yes                   | TOF                    | ACQUITY/LCT PREMIER XE         | 15                | MINI LUKE          | No        | C18                                |
| 004             |                  |              | GC No                    | QQQ                    | Waters                         | 10                | EtOAc              | Filter    | Rxi-5sil MS/integra-guard Restek   |
| 005             |                  |              | GC No                    | QQQ                    | Varian 1200L GC3800            | 10                | EtOAc              |           | FactorFour, VF-5ms                 |
| 006             | 0.0              |              | GC No                    | Q                      | Agilent 6890/5973              | 10                | ACN                | DSPE      | HP-5MS                             |
| 008             | 170              | 100          | LC No                    | Q-TRAP                 | API 4000 Qtrap                 | 10                | ACN                |           | Pursuit XRs Ultra                  |
| 009             | 0.00             |              | LC No                    | QQQ                    | Waters Aquity TQD              | 10                | Methanol           | Filter    | C18 UPICL                          |
| 010             |                  |              | LC No                    | QQQ                    | Agilent 6410B                  | 10                | ACN                | DSPE      | C18                                |
| 011             | 3.0              | 0.0          | GC Yes                   | Q                      | HP5973C                        | 10                | ACN                | PSA       | HP5MS                              |
| 012             | 30               | 20           | GC Yes                   | IT                     | Saturn 4000 varian             | 10                | ACN                | DSPE      | DB5MS                              |
| 013             | -0.3             | 95           | GC Yes                   | Q                      | Agilent 5975                   | 10                | ACN                | MgSO4/PSA | HP5MS                              |
| 014             |                  |              | GC Yes                   | Q                      | Trace DSQ                      | 15                | ACN                | QuECHERS  | DB5MS                              |
| 015             | > 70% match      | GC Yes       | IT                       | ThermoFinniganPolarisQ | 10                             | ACN               |                    | DB-5MS    | 0.8                                |
| 016             |                  |              | GC Yes                   | Q                      | 5973                           | 10                | ACN                | PSA       | DB-35ms                            |
| 017             | 0.0              | 1.44         | LC No                    | QQQ                    | HPLC Agilent 1100. MS API 3000 | 10                | ACN                | DSPE      | C18 3 $\mu$ m 50x2mm               |
| 018             | 0.0              | 0.0          | GC No                    | QQQ                    | Thermo TSQ Quantum GC          | 10                | Acetone/PE/DCM     | Na2SO4    | Rxi-5SiL MS                        |
| 019             | 9                | 30           | GC Yes                   | Q                      | Agilent GC7890-MSS973C         | 10                | ACN                | DSPE      | HP-5MS                             |
| 020             | <0.5             |              | GC Yes                   | IT                     | Varian 4000                    | 10                | ACN                | PSA       | 5 % diphenyl                       |
|                 |                  |              |                          |                        |                                |                   |                    |           | 1 Automatic                        |
|                 |                  |              |                          |                        |                                |                   |                    |           | 148 method NIST library            |
|                 |                  |              |                          |                        |                                |                   |                    |           | Each Batch                         |
|                 |                  |              |                          |                        |                                |                   |                    |           | Targeted Analysis Only             |
|                 |                  |              |                          |                        |                                |                   |                    |           | Always                             |
|                 |                  |              |                          |                        |                                |                   |                    |           | 525 method 590 library             |
|                 |                  |              |                          |                        |                                |                   |                    |           | Each Batch                         |
|                 |                  |              |                          |                        |                                |                   |                    |           | Method Setup (and very rare cases) |
|                 |                  |              |                          |                        |                                |                   |                    |           | Each Batch                         |

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

| Laboratory Code | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Full Scan       | Analyzer                  | Instrument Model | Sample Weight (g) | Extraction Solvent    | Clean-Up                | MEVINPHOS             |           | No. of Compounds | Frequency of Standard Solution |
|-----------------|------------------|--------------|--------------------------|-----------------|---------------------------|------------------|-------------------|-----------------------|-------------------------|-----------------------|-----------|------------------|--------------------------------|
|                 |                  |              |                          |                 |                           |                  |                   |                       |                         | Injection Volume (μl) | Software  |                  |                                |
| 021             |                  |              |                          | GC Yes IT       | Variian 4000              | 10               | ACN               | DSPE                  | Capillary               | 3                     | Automatic | 450              | Daily/Weekly                   |
| 022             | <0.2             | <20%         |                          | GC Yes Q        | Agilent 7890A/5975C       | 15               | ACN               | DSPE                  | HP5MS                   | 10                    | Both      | 214              | Once a Day                     |
| 023             | 0.0              |              |                          | LC No Q         | API 4000                  | 10               | ACN               | DSPE                  | C18 hydro Phenomenex    | 40                    | Manual    |                  | Each Time                      |
| 024             | 4.2              |              |                          | LC No QQQ       | API 4000                  | 10               | ACN               | Freeze out/ DSPE(PSA) | Synergie Fusion RP80A   | 8                     | Both      | 400              | At least monthly               |
| 025             | 3                | 19.5         |                          | GC Yes TOF      | LECO Pegasus IV           | 10               | ACN               | DSPE(PSA)             | HP-5MS                  | 5                     | Both      | 650              | Weekly                         |
| 026             | 0.0              | 0.0          |                          | LC No QQQ       | Xevo TQ                   | 15               | ACN               | LLE                   | C18                     | 10                    | Both      | 360              | Each Time                      |
| 028             |                  |              |                          | GC No QQQ       | Quattro micro             | 10               | ACN               | DSPE                  | HP5 MSI                 | 1                     | Both      | 110              |                                |
| 029             |                  |              |                          | GC Yes IT       | Variian GCmasse 4000      | 10               | ACN               | DSPE (MgSO4)          | Capillary db 5          | 5                     | Both      |                  | Each Batch                     |
| 030             |                  |              |                          | GC Yes TOF      | Leco Pegasus              | 15               | Acetone, DCM, PE  |                       | DB-5                    | 1                     | Automatic |                  |                                |
| 032             | 0.5              | 5 ppm        |                          | LC Yes Orbitrap | Exactive                  | 10               | ACN               | PSA                   | C18                     | 5                     | Both      | > 200            | Always                         |
| 033             | 9                | 20%          |                          | GC Yes Q        | Agilent GCMSD 7890A/5975C | 10               | ACN               | PSA, C18              | HP-5MS                  | 1                     | Automatic | 927              | Each Batch                     |
| 034             | 60               | 10 ppm       |                          | LC Yes TOF      | Agilent 6230              | 10               | ACN               |                       | Zorbax eclipse plus C18 | 3                     | Automatic | 517              | Not for routine                |
| 035             | 0.005 Da         |              |                          | LC Yes Q-TOF    | Bruker Maxis              | 10               | ACN               | PSA                   | C18 poroshell           | 1                     | Automatic | 2,000            |                                |
| 039             |                  |              |                          | GC No QQQ       | Agilent 7000B             | 10               | ACN               | DSPE                  | HP-5MS                  | 1                     | Automatic | 400              | Daily                          |
| 040             | 5                | 20           |                          | LC No QQQ       | Quattro Premier XE        | 10               | ACN               |                       | Reversed Phase          | 5                     | Manual    | 320              | Each Batch                     |
| 041             | 0.6              |              |                          | GC Yes QQQ      | Variian 3800 GC + 320-MS  | 10               | ACN               | DSPE(PSA)             | VF-1 ms                 | 3                     | Both      | 500              | Each Batch                     |
| 042             | 6                | unit         |                          | LC No QQQ       | Waters Premier            | 10               | Methanol          |                       | C18                     | 5                     | Both      | 201              | Each Time                      |
| 044             | <3%              | <20%         |                          | GC No QQQ       | Agilent 7000a             | 15               | Acetone           | re                    | HP5MSI                  | 2                     | Automatic | 200              | Each batch                     |
| 045             | 0.0              | 0.0          |                          | GC Yes Q        | Agilent                   | 50               | Acetone           | DCM                   |                         | 1                     | Both      | 220              | Daily                          |
| 046             | <40              | 893          |                          | GC Yes TOF      | Leco                      | 10               | ACN               | DSPE(PSA)             | Restek CI Pesticides    | 10                    | Both      | 700              | 235 compounds with every batch |
| 050             | -0.7             | 5.5          |                          | GC Yes Q        | 5975C inert XI E/CI MSD   | 10               | ACN               | PSA                   | HP5-MS                  | 2                     | Automatic | 200              | Always                         |
| 051             | 1.0              | 12.4         |                          | GC Yes IT       | Variian Saturn 2000       | 15               | Acetone           | Partitioning          | FactorFour VF-5ms       | 5                     | Both      | 500              | Quarterly                      |

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

| MEVINPHOS       |                  |              |                          |           |                  |                    |                   |                 |                  |                                |
|-----------------|------------------|--------------|--------------------------|-----------|------------------|--------------------|-------------------|-----------------|------------------|--------------------------------|
| Laboratory Code | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Full Scan | Analyser         | Instrument Model   | Sample Weight (g) | Column Type     | No. of Compounds | Frequency of Standard Solution |
|                 |                  |              |                          |           |                  | Extraction Solvent | Clean-Up          |                 |                  |                                |
| 052             | 99%              | GC           | Yes                      | Q         | Thermo trace DSQ | 10                 | DCM               | Supelco SLB-5MS | 1                | Both                           |
| 053             | GC               | No           | QQQ                      |           | Varian           | 7.5                | Acetone           | DB5 MS          | —                | Automatic                      |
|                 |                  |              |                          |           |                  |                    |                   |                 | 400              | Never                          |
|                 |                  |              |                          |           |                  |                    |                   |                 | 100              |                                |

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

| NUARIMOL        |                  |                          |          |           |                    |          |                                 |                       |                  |                                |
|-----------------|------------------|--------------------------|----------|-----------|--------------------|----------|---------------------------------|-----------------------|------------------|--------------------------------|
| Laboratory Code | RT Tolerance (s) | Cromatographic Technique | Defector | Full Scan | Extraction Solvent | Clean-Up | Column Type                     | Injection Volume (lL) | No. of Compounds | Frequency of Standard Solution |
| 001             | 60               | none                     | LC       | No        | MS                 | QQQ      | API 4000                        | No                    | 10               | ACN                            |
| 003             | 0.30             | 0.05 Da                  | LC       | Yes       | MS                 | TOF      | ACQUITY/LC/ PREMIER XE          | Yes                   | 15               | MINI LUKE                      |
| 004             |                  |                          | GC       | No        | MS                 | QQQ      | Waters                          | No                    | 10               | EtOAC                          |
| 005             |                  |                          | GC       | No        | MS                 | QQQ      | Variam 1200L, GC 3800           | No                    | 10               | EtOAC                          |
| 008             | 1155             | 975                      | GC       | Yes       | MS                 | TOF      | LECO Pegasus IV                 | Yes                   | 10               | ACN                            |
| 009             | 0.01             |                          | LC       | No        | MS                 | QQQ      | Waters Aquity TQD               | No                    | 10               | Methanol                       |
| 010             |                  |                          | GC       | Yes       | MS                 | Q        | HP5973 MSD                      | Yes                   | 10               | ACN                            |
| 011             |                  |                          | LC       | No        | MS                 | Q-TRAP   | ABI 4000                        | No                    | 10               | ACN                            |
| 012             | 30               | 20                       | LC       | Yes       | MS                 | QQQ      | API 3200 QT                     | Yes                   | 10               | ACN                            |
| 013             | 11.5             | 87                       | GC       | Yes       | MS                 | Q        | Agilent 5975                    | Yes                   | 10               | ACN                            |
| 014             |                  |                          | LC       | No        | MS                 | QQQ      | Acquity UPLC Quattro Premier XE | No                    | 15               | ACN                            |
| 015             | > 70% match      |                          | GC       | Yes       | MS                 | IT       | ThermoFinniganPolarisQ          | Yes                   | 10               | ACN                            |
| 016             |                  |                          | GC       | Yes       | MS                 | Q        |                                 | 5973                  | 10               | ACN                            |
| 017             | 0.0              | -0.50                    | LC       | No        | MS                 | QQQ      | HPLC Agilent 1100. MS API 3000  | No                    | 10               | ACN                            |
| 018             | 0.0              | 0.0                      | LC       | No        | MS                 | QQQ      | Waters Xevo                     | No                    | 10               | Acetone/PE/DCM                 |
| 019             | 9                | 30                       | GC       | Yes       | MS                 | Q        | Agilent GC7890-NS5975C          | Yes                   | 10               | ACN                            |
| 021             |                  |                          | GC       | Yes       | MS                 | IT       | Varian 4000                     | Yes                   | 10               | ACN                            |
| 022             | <0.1             | <1.5%                    | LC       | No        | MS                 | QQQ      | Waters Aquity TQD               | No                    | 15               | ACN                            |
| 023             | 0.0              |                          | LC       | No        | MS/MS              | Q        | API 4000                        | No                    | 10               | ACN                            |
| 025             | 1                | 9.3                      | GC       | Yes       | MS                 | TOF      | LECO Pegasus IV                 | Yes                   | 10               | ACN                            |
| 026             | 0.0              | 0.0                      | LC       | No        | MS                 | QQQ      | Xevo TQ                         | No                    | 15               | ACN                            |
| 028             |                  |                          | GC       | Yes       | MS                 | Q        | HP-5973N                        | Yes                   | 10               | ACN                            |
| 030             |                  |                          | GC       | Yes       | MS                 | TOF      | Leco Pegasus                    | Yes                   | 15               | Acetone, DCM, PE               |

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

| NUARIMOL        |                  |                          |          |          |                  |                   |                          |          |             |                       |                  |                                |      |           |        |                                |
|-----------------|------------------|--------------------------|----------|----------|------------------|-------------------|--------------------------|----------|-------------|-----------------------|------------------|--------------------------------|------|-----------|--------|--------------------------------|
| Laboratory Code | RT Tolerance (s) | Cromatographic Technique | Detector | Analyzer | Instrument Model | Sample Weight (g) | Extraction Solvent       | Clean-Up | Column Type | Injection Volume (μl) | No. of Compounds | Frequency of Standard Solution |      |           |        |                                |
| 032             | RT locking       | GC                       | Yes      | MS       | GCMSSD           | Agilent           | Yes                      | 10       | ACN         | PSA                   | HP5              | 10                             | Both | >600      | Always | Each Batch                     |
| 033             | 9                | 20%                      | GC       | Yes      | MS               | Q                 | Agilent GCMSD            | Yes      | 10          | ACN                   | PSA, C18         | HP-5MS                         | 1    | Automatic | 927    | Each Batch                     |
| 034             | 60               | 10 ppm                   | LC       | Yes      | MS               | TOF               | Agilent 6230             | Yes      | 10          | ACN                   |                  | Zorbax eclipse plus C18        | 3    | Automatic | 517    | Not for routine                |
| 035             |                  |                          | GC       | Yes      | MS               | Q                 | Waters Micromass         | Yes      | 10          | ACN                   | PSA              | DB5                            | 5    | Manual    | 500    |                                |
| 039             |                  |                          | GC       | No       | MS               | QQQ               | Agilent 7000B            | No       | 10          | ACN                   | DSPE             | HP-5MS                         | 1    | Automatic | 400    | Daily                          |
| 040             | 5                | 10                       | GC       | Yes      | MS               | TOF               | GCT Premier              | Yes      | 12.5        | EtOAc                 | GPC              | Capillary                      | 1    | Both      | NIST   |                                |
| 041             | 0.0              |                          | GC       | No       | MS               | QQQ               | Varian 3800 GC+ 320-MS   | No       | 10          | ACN                   | DSPE(PSA)        | VF-1 ms                        | 3    | Both      | 150    | Each Batch                     |
| 042             | 5                | unit                     | GC       | No       | MS               | QQQ               | Varian 320MS             | No       | 50          | Acetone               | L/L              | DB5                            | 8    | Both      | 128    | Each Time                      |
| 044             | <3%              | <20%                     | GC       | No       | MS               | QQQ               | Agilent 7000a            | No       | 15          | Acetone               | ne               | HP5MSI                         | 2    | Automatic | 200    | Each batch                     |
| 045             | 0.0              | 0.0                      | GC       | Yes      | MS               | Q                 | Agilent                  | Yes      | 50          | Acetone               | DCM              |                                | 1    | Both      | 220    | Daily                          |
| 046             | -0.6             | 0.2 ppm                  | LC       | Yes      | MS               | Orbitrap          | Exactive                 | Yes      | 10          | ACN                   |                  | Atlantis T3                    | 5    | Both      | 461    | 100 compounds with every batch |
| 050             | 2.05             | 5.5                      | GC       | Yes      | MS               | Q                 | 5975C inert XL EI/CI MSD | Yes      | 10          | ACN                   | PSA              | HP5-MS                         | 2    | Automatic | 200    | Always                         |
| 051             | 3.9              | 3.2                      | GC       | Yes      | MS               | IT                | Varian Saturn 2000       | Yes      | 15          | Acetone               | Partitioning     | FactorFour VF-5ms              | 5    | Both      | 500    | Quarterly                      |
| 052             | 92%              |                          | GC       | Yes      | MS               | Q                 | Thermo trace DSQ         | Yes      | 10          | DCM                   |                  | Supelco SLB-5MS                | 1    | Both      | 400    | Never                          |
| 053             |                  |                          | GC       | Yes      | MS               | QQQ               | Varian                   | Yes      | 7.5         | Acetone               | Yes              | DB5 MS                         | 1    | Automatic | 100    |                                |

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

| Laboratory Code | RT Tolerance (s) | MS Tolerance    | Chromatographic Technique | Full Scan                      | Detector | Analyser       | Instrument Model | Sample Weight (g)       | Extraction Solvent | Clean-Up | OFURACE               |             | Software         | No. of Compounds | Frequency of Standard Solution |
|-----------------|------------------|-----------------|---------------------------|--------------------------------|----------|----------------|------------------|-------------------------|--------------------|----------|-----------------------|-------------|------------------|------------------|--------------------------------|
|                 |                  |                 |                           |                                |          |                |                  |                         |                    |          | Injection Volume (μl) | Column Type |                  |                  |                                |
| 003             | 0.30             | 0.05 Da         | LC Yes MS TOF             | ACQUITY/LCT PREMIER XE         | 15       | MINI LUKE      | No               | C18                     |                    |          | 5                     | Both        | 345              |                  | Once a Day                     |
| 004             |                  |                 | LC No MS QQQ              | Applied Biosystems API5000     | 10       | EtOAc          | Filter           | HSS T3                  |                    |          | 2                     | Both        | 227              |                  | Each Batch                     |
| 005             |                  |                 | LC No MS QQQ              | Agilent 6460                   | 10       | EtOAc          |                  | C18                     |                    |          | 2                     | Automatic   | 500              |                  | Each Batch                     |
| 008             | 1098             | 889             | GC Yes MS TOF             | LECO Pegasus IV                | 10       | ACN            | PSA              | Phenomenex ZB-5MS       |                    |          | 3                     | Both        | 750              |                  | Each Batch                     |
| 009             | 0.00             |                 | LC No MS QQQ              | Waters Aquity TQD              | 10       | Methanol       | Filter           | C18 UPIC                |                    |          | 6                     | Automatic   | 120              |                  |                                |
| 011             | 6.3              | 0.0             | GC Yes MS Q               | HP5975C                        | 10       | ACN            | PSA              | HP5MS                   |                    |          | 5                     | Both        | approx. 1,000    |                  | Always                         |
| 016             |                  |                 | GC Yes MS Q               | 5973                           | 10       | ACN            | PSA              | DB-35ms                 |                    |          | 2                     | Both        |                  |                  | Targeted Analysis Only         |
| 017             | 0.0              | 10.36           | LC No MS QQQ              | HPLC Agilent 1100, MS API 3000 | 10       | ACN            | DSPE             | C18 3um 50x2mm          |                    |          | 10                    | Both        | 525 meth 590 lib |                  | Always                         |
| 018             | 0.0              | 0.0             | LC No MS QQQ              | Waters Xevo                    | 10       | Acetone/PE/DCM | Na2SO4           | C18                     |                    |          | 2                     | Automatic   | 250              |                  | Each Batch                     |
| 021             |                  |                 | GC Yes MS IT              | Varian 4000                    | 10       | ACN            | DSPE             | Capillary               |                    |          | 3                     | Automatic   | 450              |                  | Daily/Weekly                   |
| 022             | <0.1             | <15%            | LC No MS QQQ              | Waters Aquity TQD              | 15       | ACN            | DSPE             | UPLC-BEH C18            |                    |          | 10                    | Both        | 340              |                  | Once a Day                     |
| 025             | 4                | 14.7            | GC Yes MS TOF             | LECO Pegasus IV                | 10       | ACN            | DSPE(PSA)        | HP-5MS                  |                    |          | 5                     | Both        | 650              |                  | Weekly                         |
| 026             | 0.0              | 0.0             | LC No MS QQQ              | Xevo TQ                        | 15       | ACN            | LLE              | C18                     |                    |          | 10                    | Both        | 360              |                  | Each Time                      |
| 028             |                  |                 | LC No MS QQQ              | Quattro Premier                | 10       | ACN            | DSPE             | C18                     |                    |          | 5                     | Both        |                  |                  |                                |
| 032             | RT locking       | GC Yes MS GCMSD | Agilent                   | 10                             | ACN      | PSA            | HP5              |                         |                    |          | 10                    | Both        | >600             |                  | Always                         |
| 033             | 9                | 20%             | GC Yes MS Q               | Agilent GCMSD 7890A/5975C      | 10       | ACN            | PSA, C18         | HP-5MS                  |                    |          | 1                     | Automatic   | 927              |                  | Each Batch                     |
| 034             | 60               | 10 ppm          | LC Yes MS TOF             | Agilent 6230                   | 10       | ACN            |                  | Zorbax eclipse plus C18 |                    |          | 3                     | Automatic   | 517              |                  | Not for routine                |
| 039             |                  |                 | LC No MS QQQ              | API 4000QIT                    | 10       | ACN            | DSPE             | C18                     |                    |          | 5                     | Automatic   | 400              |                  | Daily                          |
| 046             | 0.0              | 0.4 ppm         | LC Yes MS Orbitrap        | Exactive                       | 10       | ACN            |                  | Atlantis T3             |                    |          | 5                     | Both        | 461              |                  | 100 compounds every batch      |
| 050             | -0.008           | 3.79            | LC No MS QQQ              | G6410A                         | 10       | ACN            | PSA              | Zorbax-Eclipse XDB-C8   |                    |          | 20                    | Automatic   | 106              |                  | Always                         |
| 051             | 0.0              | 4.6             | LC No MS QQQ              | Waters Premier XE              | 15       | Acetone        | Partitioning     | UPLC Acuity BEH         |                    |          | 5                     | Both        | 200              |                  | Daily                          |
| 052             |                  | 80%             | GC Yes MS Q               | Thermo trace DSQ               | 10       | DCM            |                  | Supelco SLB-5MS         |                    |          | 1                     | Both        | 400              |                  | Never                          |
| 053             |                  |                 | LC Yes MS QQQ             | Varian                         | 12       | ACN            | Yes              | C18                     |                    |          | 10                    | Automatic   | 70               |                  |                                |

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

| o-PHENYLPHENOL   |                           |           |                          |                              |                  |                         |  |  |  |  |
|--|---------------------------|-----------|--------------------------|------------------------------|------------------|-------------------------|--|--|--|--|
| Instrument Model   | Extraction Solvent        | Clean-up  | Column Type              | Injection Volume (μl)        | No. of Compounds | Solution                | Frequency of Standard                  |  |  |  |
| 001 12 none LC No MS QQQ   | API 4000                  | 10 ACN    | DSPE                     | Waters C18<br>2.1x50mmx1.7μm | 5 Both           | 600                     | every week<br>200 pesticides           |  |  |  |
| 004      GC No MS QQQ  | Waters                    | 10 EtOAc  | Filter                   | Integra-guard Restek         | 10 Both          | 135                     | Each Batch                             |  |  |  |
| 005      GC No MS QQQ  | Varien 1200L GC3800       | 10 EtOAc  | FactorFour, VF-5ms       | 2 Automatic                  | 500              | 500                     | Each Batch                             |  |  |  |
| 006 0.0 GC No MS Q Agilent 6890/5973                                 | 10 ACN                    | DSPE      | HP-5MS                   | 1 Manual                     | 177              | Once a Month            | Once a Month                           |  |  |  |
| 008 551 979 GC Yes MS TOF LECO Pegasus IV                            | 10 ACN                    | PSA       | Phenomenex ZB-5MS        | 3 Both                       | 750              | 750                     | Each Batch                             |  |  |  |
| 009 0.00 GC Yes MS IT Thermo Polaris Q                               | 10 ACN                    | PSA       | Capillary GC             | 1 Automatic                  | 130              | 130                     | Each Batch                             |  |  |  |
| 010 3.5 0.0 GC Yes MS Q HP 5973 MSD                                  | 10 ACN                    | DSPE      | HP-5MSI                  | 20 Both                      | 111              | 111                     | Each Batch                             |  |  |  |
| 011 30 20 GC Yes MS IT HP5975C                                       | 10 ACN                    | PSA       | HP5MS                    | 5 Both                       | approx. 1.000    | Always                  | Always                                 |  |  |  |
| 012 7.1 97 GC Yes MS Q Saturn 4000 varian                            | 10 ACN                    | DSPE      | DB5MS                    | 1 Both                       | 350              | 350                     | Daily                                  |  |  |  |
| 013 7.1 97 GC Yes MS Q Agilent 5975                                  | 10 ACN                    | MgSO4/PSA | HP5MS                    | 20 Automatic                 | 550              | 550                     | 2 months                               |  |  |  |
| 014      GC Yes MS Q Trace DSQ                                       | 15 ACN                    | QuEChERS  | DB5-MS                   | 0.8 Both                     | Both             | Both                    | Targeted Analysis Only                 |  |  |  |
| 016      GC Yes MS Q 5973  | 10 ACN                    | PSA       | DB-35ms                  | 2 Both                       | 525 method       | 590 library             | Each Batch                             |  |  |  |
| 017 0.0 2.00 GC No MS Q GC Shimadzu GC-2010, MS Shimadzu GCMS-QP2010 | 10 Acetone/PE/DCM         | Na2SO4    | RXi-5SiL MS              | 1 Automatic                  | 164              | 164                     | Always                                 |  |  |  |
| 018 0.0 0.0 GC No MS QQQ Thermo TSQ Quantum GC                       | 10 Agilent GC7890-MS5975C | DSPE      | HP-5MS                   | 1 Both                       | 150 method       | 500 library             | Method Set up<br>(and very rare cases) |  |  |  |
| 019 9 30 GC Yes MS Q Agilent GC7890-MS5975C                          | 10 ACN                    | PSA       | 5% diphenyl              | 1 Automatic                  | 148 method       | 148 method NIST library | Each Batch                             |  |  |  |
| 020 <0.5 GC Yes MS IT Varian 4000                                    | 10 ACN                    | DSPE      | Capillary                | 3 Automatic                  | 450              | 450                     | Daily/Weekly                           |  |  |  |
| 021      GC Yes MS IT Varian 4000                                    | 10 ACN                    | DSPE      | HP5MS                    | 10 Both                      | 214              | 214                     | Once a Day                             |  |  |  |
| 022 <0.2 <20% GC Yes MS Q Agilent 7890A/5975C                        | 15 ACN                    | DSPE      | CP Sil 8 CB/MS (Varian)  | 2 Manual                     | Manual           | Manual                  | Each Time                              |  |  |  |
| 023 0.0 GC No MS IT Varian Saturn 2000                               | 10 ACN                    | DSPE      | Freeze out/<br>DSPE(PSA) | 2 Both                       | 450              | 450                     | At least monthly                       |  |  |  |
| 024      GC No MS QQQ TSQ Thermo                                     | 10 ACN                    | DSPE(PSA) | HP-5MS                   | 5 Both                       | 650              | 650                     | Weekly                                 |  |  |  |

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

| o-PHENYLPHENOL  |                  |              |                          |                  |                   |                           |          |             |                       |                      |                                |
|-----------------|------------------|--------------|--------------------------|------------------|-------------------|---------------------------|----------|-------------|-----------------------|----------------------|--------------------------------|
| Laboratory Code | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Instrument Model | Sample Weight (g) | Extraction Solvent        | Clean-up | Column Type | Injection Volume (μl) | No. of Compounds     | Frequency of Standard Solution |
|                 |                  |              |                          |                  |                   |                           |          |             |                       |                      |                                |
| 026             | 0.0              | 0.0          | GC                       | Yes              | MS IT             | Polaris Q                 | 25       | Hexane      | LLE                   | DB5 MS               | 1 Both                         |
| 028             |                  |              | GC                       | No               | MS QQQ            | Quattro micro             | 10       | ACN         | DSPE                  | HP5_MS               | 1 Both                         |
| 029             |                  |              | GC                       | Yes              | MS IT             | Varian GCmass 4000        | 10       | ACN         | DSPE(MS04)            | Capillary db 5       | 5 Both                         |
| 032             | RT locking       |              | GC                       | Yes              | MS GCMSD          | Agilent                   | 10       | ACN         | PSA                   | HP5                  | 10 Both                        |
| 033             | 9                | 20%          | GC                       | Yes              | MS Q              | Agilent GCMSD 7890A/5975C | 10       | ACN         | PSA, C18              | HP-5MS               | 1 Automatic                    |
| 034             | 12               |              | GC                       | Yes              | MS IT             | Saturn 2000               | 30       | EtOAc       | HPGPC                 | DB-5 MS              | 2 Automatic                    |
| 035             | 0.01             |              | GC                       | No               | MS QQQ            | Waters Micromass          | 10       | ACN         | PSA                   | DB5                  | 5 Automatic                    |
| 037             |                  |              | GC                       | Yes              | MS Q              | Agilent 6890N             | 10       | ACN         | PSA                   | DB-5                 | 1 Both                         |
| 039             |                  |              | GC                       | No               | MS QQQ            | Agilent 7000B             | 10       | ACN         | DSPE                  | HP-5MS               | 1 Automatic                    |
| 040             | 5                | 10           | GC                       | Yes              | MS TOF            | GCT Premier               | 12.5     | EtOAc       | GPC                   | Capillary            | 1 Manual                       |
| 041             | 1.2              |              | GC                       | No               | MS QQQ            | Varian 3800 GC+ 320-Ms    | 10       | ACN         | DSPE(PSA)             | VF-1 ms              | 3 Both                         |
| 042             | 5                | unit         | GC                       | No               | MS QQQ            | Varian 320MS              | 50       | Acetone     | L/L                   | DB5                  | 8 Both                         |
| 044             | <3%              | <20%         | GC                       | No               | MS QQQ            | Agilent7000a              | 15       | Acetone     | ne                    | HP5MSI               | 2 Automatic                    |
| 045             | 0.0              | 0.0          | GC                       | Yes              | MS Q              | Agilent                   | 50       | Acetone     | DCM                   |                      | 1 Both                         |
| 046             | <20              | 961          | GC                       | Yes              | MS TOF            | Leco                      | 10       | ACN         | DSPE(PSA)             | Restek CI Pesticides | 10 Both                        |
| 047             | 0.02             | 0.5          | GC                       | No               | QQQ               | Varian 1200L              | 10       | ACN         | DSPE                  | DB-5                 | 5 Automatic                    |
| 050             | -0.05            | 0.5          | GC                       | Yes              | MS Q              | 5975C inert XL EI/CI MSD  | 10       | ACN         | PSA                   | HP5-MS               | 2 Automatic                    |
| 051             | 0.5              | 0.5          | GC                       | Yes              | MS IT             | Varian Saturn 2000        | 15       | Acetone     | Partitioning          | FactorFour VF-5ms    | 5 Both                         |
| 052             | 80%              | GC           | Yes                      | MS Q             | Thermo trace DSQ  | 10                        | DCM      |             | Supelco SLB-5MS       | 1 Both               | 400 Daily                      |
|                 |                  |              |                          |                  |                   |                           |          |             |                       |                      | Never                          |

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

| Laboratory Code | RT Tolerance (s) | Chromatographic Technique | MS Tolerance | Analisyser | Instrument Model | Sample Weight (g) | Extraction Solvent                | Clean-Up | Column Type    | Injection Volume (μl) | No. of Compounds           | Frequency of Standard Solution |           |                                     |                              |
|-----------------|------------------|---------------------------|--------------|------------|------------------|-------------------|-----------------------------------|----------|----------------|-----------------------|----------------------------|--------------------------------|-----------|-------------------------------------|------------------------------|
|                 |                  |                           |              |            |                  |                   |                                   |          |                |                       |                            |                                |           |                                     |                              |
| 001             | 12               | none                      | LC           | No         | MS               | QQQ               | API 4000                          | 10       | ACN            | DSPE                  | Waters-C18 2.1x50mmx1.7Åμm | 5                              | Both      | 600                                 | every week<br>200 pesticides |
| 004             |                  |                           | LC           | No         | MS               | QQQ               | Applied Biosystems API5000        | 10       | EtOAC          | Filter                | HSS T3                     | 2                              | Both      | 227                                 | Each Batch                   |
| 005             |                  |                           | LC           | No         | MS               | QQQ               | Agilent 6460                      | 10       | EtOAC          |                       | C18                        | 2                              | Automatic | 500                                 | Each Batch                   |
| 006             | 0.03             |                           | GC           | No         | MS               | Q                 | Agilent 6890/5973                 | 10       | ACN            | DSPE                  | HP-5MS                     | 1                              | Manual    | 177                                 | Once a Month                 |
| 008             | 616              | 929                       | GC           | Yes        | MS               | TOF               | LECO Pegasus IV                   | 10       | ACN            | PSA                   | Phenomenex ZB-5MS          | 3                              | Both      | 750                                 | Each Batch                   |
| 009             | 0.00             |                           | GC           | Yes        | MS               | IT                | Thermo Polaris Q                  | 10       | ACN            | PSA                   | Capillary GC               | 1                              | Automatic | 130                                 |                              |
| 010             |                  |                           | LC           | No         | MS               | QQQ               | Agilent 6410B                     | 10       | ACN            | DSPE                  | C18                        | 2                              | Both      | 182                                 | Each Batch                   |
| 011             | -0.4             | 0.0                       | GC           | Yes        | MS               | Q                 | HP5975C                           | 10       | ACN            | PSA                   | HP5MS                      | 5                              | Both      | approx.1,000                        | Always                       |
| 012             | 30               | 20                        | GC           | Yes        | MS               | IT                | Saturn 4000 varian                | 10       | ACN            | DSPE                  | DB5MS                      | 1                              | Both      | 350                                 | Daily                        |
| 013             | 30               | 20                        | LC           | Yes        | MS               | QQQ               | API 3200 QT                       | 10       | ACN            | DSPE                  | Fusion C18                 | 20                             | Automatic | 150                                 | Daily                        |
| 014             | -2.5             | 56                        | GC           | Yes        | MS               | Q                 | Agilent 5975                      | 10       | ACN            | MgSO4/PSA             | HP5MS                      | 20                             | Automatic | 550                                 | 2 months                     |
| 016             |                  |                           | GC           | Yes        | MS               | Q                 | 5973                              | 10       | ACN            | PSA                   | DB-35ms                    | 2                              | Both      |                                     | Targeted Analysis Only       |
| 017             | -1.2             | -19.44                    | LC           | No         | MS               | QQQ               | Waters Premier Quattro XE         | 10       | ACN            | PSA                   | Kinetex XB-C18             | 10                             | Both      |                                     | Targeted Analysis Only       |
| 018             | 0.0              | 0.0                       | GC           | No         | MS               | QQQ               | HPLC Agilent 1100.<br>MS API 3000 | 10       | Acetone/PE/DCM | DSPE                  | C18 3μm 50x2mm             | 10                             | Both      | 525 method<br>590 library           |                              |
| 019             | 9                | 30                        | GC           | Yes        | MS               | Q                 | Agilent GC7890A/MS5975C           | 10       | ACN            | Na2SO4                | RXi-5SiL MS                | 1                              | Automatic | 164                                 | Each Batch                   |
| 020             | <0.5             |                           | GC           | Yes        | MS               | IT                | Varian 4000                       | 10       | ACN            | DSPE                  | HP5-MS                     | 1                              | Both      | 150 method<br>(and very rare cases) |                              |
| 021             |                  |                           | GC           | Yes        | MS               | IT                | Varian 4000                       | 10       | ACN            | PSA                   | 5 % diphenyl               | 1                              | Automatic | 148 method<br>NST library           | Each Batch                   |
| 022             | <0.2             | <20%                      | GC           | Yes        | MS               | Q                 | Agilent 7890A/5975C               | 15       | ACN            | DSPE                  | Capillary                  | 3                              | Automatic | 450                                 | Daily/Weekly                 |
| 023             | 0.0              |                           | LC           | No         | MS/MS            | Q                 | API 4000                          | 10       | ACN            | DSPE                  | HP5MS                      | 10                             | Both      | 214                                 | Once a Day                   |
| 024             |                  |                           | GC           | No         | MS               | QQQ               | TSQ Thermo                        | 10       | ACN            | Freeze out/DSPE/PSA   | TR5MS                      | 2                              | Both      | 450                                 | Each Time                    |
|                 |                  |                           |              |            |                  |                   |                                   |          |                |                       |                            |                                |           | At least monthly                    |                              |

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

| Laboratory Code | RT Tolerance (s) | Mass Tolerance | Cromatographic Technique | Full Scan | Detector | Analyser | Instrument Model          | Sample Weight (g) | PHORATE          |              | No. of Compounds    | Software | Frequency of Standard Solution |                                    |
|-----------------|------------------|----------------|--------------------------|-----------|----------|----------|---------------------------|-------------------|------------------|--------------|---------------------|----------|--------------------------------|------------------------------------|
|                 |                  |                |                          |           |          |          |                           |                   | Clean-up         | Column Type  |                     |          |                                |                                    |
| 025             | 1                | 11.0           | GC                       | Yes       | MS       | TOF      | LECO Pegasus IV           | 10                | ACN              | DSPE(PSA)    | HP-5MS              | 5        | Both                           | 650 Weekly                         |
| 026             | 0.0              | 0.0            | GC                       | Yes       | MS       | IT       | Polaris Q                 | 25                | Hexane           | LLE          | DB5 MS              | 1        | Both                           | 150 Each Time                      |
| 028             |                  |                | LC                       | No        | MS       | QQQ      | Quattro Premier           | 10                | ACN              | DSPE         | C18                 | 5        | Both                           | 180                                |
| 030             |                  |                | GC                       | Yes       | MS       | TOF      | Leco Pegasus              | 15                | Acetone, DCM, PE |              | DB-5                | 1        | Automatic                      |                                    |
| 032             | RT Locking       | GC             | Yes                      | MS        | GCMSD    |          | Agilent                   | 10                | ACN              | PSA          | HP5                 | 10       | Both                           | Always                             |
| 033             | 9                | 20%            | GC                       | Yes       | MS       | Q        | Agilent GCMSD 7890A/5975C | 10                | ACN              | PSA, C18     | HP-5MS              | 1        | Automatic                      | 927 Each Batch                     |
| 034             | 12               |                | GC                       | Yes       | MS       | IT       | Saturn 2000               | 30                | EtoAC            | HPGPC        | DB-5 MS             | 2        | Automatic                      | 380 Not for routine                |
| 035             | 2.50%            |                | LC                       | No        | MS/MS    | MS/MS    | Waters Premier XE         | 10                | ACN              |              | Acquity UPLC HSS T3 | 20       | Both                           | 200 Routine Target Suite           |
| 036             | 0.01             |                | GC                       | No        | MS       | QQQ      | Waters Micromass          | 10                | ACN              | PSA          | DB5                 | 5        | Automatic                      | Daily                              |
| 039             |                  |                | LC                       | No        | MS       | QQQ      | API 4000QT                | 10                | ACN              | DSPE         | C18                 | 5        | Automatic                      | 400 Daily                          |
| 040             | 5                | 20             | LC                       | No        | MS       | QQQ      | Quattro Premier XE        | 10                | ACN              |              | Reversed Phase      | 5        | Manual                         | 320 Each Batch                     |
| 041             | 0.5              |                | GC                       | No        | MS       | QQQ      | Varian 3800 GC+ 320-MS    | 10                | ACN              | DSPE(PSA)    | VF-1 ms             | 3        | Both                           | 150 Each Batch                     |
| 042             | 5                | Unit           | GC                       | No        | MS       | QQQ      | Varian 320MS              | 50                | Acetone          | L/L          | DB5                 | 8        | Both                           | 128 Each Time                      |
| 045             | 0.0              | 0.0            | GC                       | Yes       | MS       | Q        | Agilent                   | 50                | Acetone          | DCM          |                     | 1        | Both                           | 220 Daily                          |
| 046             | -1.8             | 0.3 ppm        | LC                       | Yes       | MS       | Orbitrap | Exactive                  | 10                | ACN              |              | Atlantis 3          | 5        | Both                           | 461 100 compounds with every batch |
|                 | 0.0              | 2.5 ppm        | LC                       | Yes       | MS       | Orbitrap | Exactive                  | 10                | ACN              |              | Atlantis 3          | 5        | Both                           | 461 100 compounds with every batch |
|                 | -0.6             | 0.4 ppm        | LC                       | Yes       | MS       | Orbitrap | Exactive                  | 10                | ACN              |              | Atlantis 3          | 5        | Both                           | 461 100 compounds with every batch |
| 047             | 0.02             |                | GC                       | No        | MS       | QQQ      | Varian 1200L              | 10                | ACN              | DSPE         | DB-5                | 5        | Automatic                      | Each Batch                         |
| 050             | -1.2             | 9.5            | GC                       | Yes       | MS       | Q        | 5975C Inert XL EI/CI MSD  | 10                | ACN              | PSA          | HP5-MS              | 2        | Automatic                      | 200 Always                         |
| 051             | 2.6              | 5.6            | GC                       | Yes       | MS       | IT       | Varian Saturn 2000        | 15                | Acetone          | Partitioning | FactorFour VF-5ms   | 5        | Both                           | 500 Quarterly                      |
|                 | 1.2              | 1.7            | LC                       | No        | MS       | QQQ      | Waters Premier XE         | 15                | Acetone          | Partitioning | UPLC Acuity BEH     | 5        | Both                           | 200 Weekly                         |
| 053             |                  |                | GC                       | No        | MS       | QQQ      | Varian                    | 7.5               | Acetone          | Yes          | DB5 MS              | 1        | Automatic                      | 100                                |

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

| PROMETRYN       |                  |              |                          |           |          |                  |                                |          |                |           |                                    |                                |           |
|-----------------|------------------|--------------|--------------------------|-----------|----------|------------------|--------------------------------|----------|----------------|-----------|------------------------------------|--------------------------------|-----------|
| Laboratory Code | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Full Scan | Detector | Instrument Model | Sample Weight (g)              | Clean-Up | Column Type    | Software  | No. of Compounds                   | Frequency of Standard Solution |           |
| 001             | 60               | none         | LC                       | No        | MS       | QQQ              | API 4000                       | 10       | ACN            | DSPE      | Waters-C18<br>2.1x50mmx1.7 $\mu$ m | 5                              | Both      |
| 003             | 0.30             | 0.05 Da      | LC                       | Yes       | MS       | TOF              | ACQUITY/LCT PREMIER XE         | 15       | MINI LUKE      | No        | C18                                | 5                              | Both      |
| 004             |                  |              | LC                       | No        | MS       | QQQ              | Applied Biosystems API5000     | 10       | EtOAc          | Filter    | HSS T3                             | 2                              | Both      |
| 005             |                  |              | LC                       | No        | MS       | QQQ              | Agilent 6460                   | 10       | EtOAc          |           | C18                                | 2                              | Automatic |
| 006             | 0.0              |              | GC                       | No        | MS       | Q                | Agilent 6890/5973              | 10       | ACN            | DSPE      | HP-5MS                             | 1                              | Manual    |
| 008             | 310              | 100          | LC                       | No        | MS       | Q-TRAP           | API 4000 Qtrap                 | 10       | ACN            |           | Pursuit XRs Ultra                  | 6                              | Both      |
| 010             |                  |              | GC                       | Yes       | MS       | Q                | HP 5973 MSD                    | 10       | ACN            | DSPE      | HP-5MSI                            | 20                             | Both      |
| 011             | 3.5              | 0.0          | GC                       | Yes       | MS       | Q                | HP5975C                        | 10       | ACN            | PSA       | HP5MS                              | 5                              | Both      |
| 012             | 30               | 20           | GC                       | Yes       | MS       | T                | Saturn 4000 varian             | 10       | ACN            | DSPE      | DB5MS                              | 1                              | Both      |
| 013             | 11.8             | 95           | GC                       | Yes       | MS       | Q                | Agilent 5975                   | 10       | ACN            | MGSO4/PSA | HP5MS                              | 20                             | Automatic |
| 014             |                  |              | GC                       | Yes       | MS       | Q                | Trace DSQ                      | 15       | ACN            | QuEChERS  | DB5-MS                             | 0.8                            | Both      |
| 015             |                  |              | LC                       | No        | MS       | QQQ              | ThermoFinnigan TSQQuantum      | 10       | ACN            |           | XterraC18MS                        | 20                             | Automatic |
| 016             |                  |              | LC                       | No        | MS       | QQQ              | Waters Premier Quattro XE      | 10       | ACN            | PSA       | Kinetex XB-C18                     | 10                             | Both      |
| 017             | -0.6             | -20.39       | LC                       | No        | MS       | QQQ              | HPLC Agilent 1100, MS API 3000 | 10       | ACN            | DSPE      | C 18 3 $\mu$ m 50x2mm              | 10                             | Both      |
| 018             | 0.0              | 0.0          | GC                       | No        | MS       | QQQ              | Thermo TSQ Quantum GC          | 10       | Acetone/PE/DCM | No2SO4    | RXI-SSL MS                         | 1                              | Automatic |
| 019             | 9                | 30           | GC                       | Yes       | MS       | Q                | Agilent GC/890-MS5975C         | 10       | ACN            | DSPE      | HP5-MS                             | 1                              | Both      |
| 020             | <0.5             |              | GC                       | Yes       | MS       | T                | Varian 4000                    | 10       | ACN            | PSA       | 5 % diphenyl                       | 1                              | Automatic |
| 021             |                  |              | GC                       | Yes       | MS       | T                | Variion 4000                   | 10       | ACN            | DSPE      | Capillary                          | 3                              | Automatic |
| 022             | <0.1             | <15%         | LC                       | No        | MS       | QQQ              | Waters Aquity TQD              | 15       | ACN            | DSPE      | UPLC-BEH C18                       | 10                             | Both      |
| 023             | 0.0              |              | LC                       | No        | MS/MS    | Q                | API 4000                       | 10       | ACN            | DSPE      | C18 hydro Phenomenex               | 40                             | Manual    |

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

| Labordatory Code | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Full Scan | Detector | Analyser | Instrument Model          | Sample Weight (g) | PROMETRYN             |                           |                         | No. of Compounds | Frequency of Standard Solution |            |
|------------------|------------------|--------------|--------------------------|-----------|----------|----------|---------------------------|-------------------|-----------------------|---------------------------|-------------------------|------------------|--------------------------------|------------|
|                  |                  |              |                          |           |          |          |                           |                   | Clean-Up              | Column Type               | Injection Volume (µl)   |                  |                                |            |
| 024              |                  |              |                          |           |          |          |                           |                   | Freeze out/ DSPE(PSA) | TR5MS                     | 2                       | Both             | 450                            |            |
| 025              | 3                | 10.2         | GC                       | Yes       | MS       | TOF      | LECO Pegasus IV           | 10                | ACN                   | DSPE(PSA)                 | 5                       | Both             | 650                            |            |
| 026              | 0.0              | 0.0          | GC                       | Yes       | MS       | IT       | Polaris Q                 | 25                | Hexane                | LLE                       | 1                       | Both             | 150                            |            |
| 028              |                  |              | GC                       | No        | MS       | QQQ      | Quattro micro             | 10                | ACN                   | DSPE                      | HP5_MSi                 | 1                | Both                           | 110        |
| 029              |                  |              | GC                       | Yes       | MS       | IT       | Varian GCmass 4000        | 10                | ACN                   | DSPE (MgSO <sub>4</sub> ) | Capillary db 5          | 5                | Both                           | Each Batch |
| 030              |                  |              | GC                       | Yes       | MS       | TOF      | Leco Pegasus              | 15                | Acetone, DCM, PE      |                           | DB-5                    | 1                | Automatic                      |            |
| 032              | 0.5              | 5 ppm        | LC                       | Yes       | MS       | Orbitrap | Exactive                  | 10                | ACN                   | PSA                       | C18                     | 5                | Both                           | > 200      |
| 033              | 9                | 20%          | GC                       | Yes       | MS       | Q        | Agilent GCMSD 7890A/5975C | 10                | ACN                   | PSA, C18                  | HP-5MS                  | 1                | Automatic                      | 927        |
| 034              | 60               | 10 ppm       | LC                       | Yes       | MS       | TOF      | Agilent 6230              | 10                | ACN                   |                           | Zorbax eclipse plus C18 | 3                | Automatic                      | 517        |
| 035              |                  |              | GC                       | Yes       | MS       | Q        | Waters Micromass          | 10                | ACN                   | PSA                       | DB5                     | 5                | Manual                         | 500        |
| 037              |                  |              | GC                       | Yes       | MS       | Q        | Agilent 6890N             | 10                | ACN                   | PSA                       | DB-5                    | 1                | Both                           | 80         |
| 039              |                  |              | GC                       | No        | MS       | QQQ      | Agilent 7000B             | 10                | ACN                   | DSPE                      | HP-5MS                  | 1                | Automatic                      | 400        |
| 040              | 5                | 20           | LC                       | No        | MS       | QQQ      | Quattro Premier XE        | 10                | ACN                   |                           | Reversed Phase          | 5                | Manual                         | 320        |
| 041              | 0.5              | GC           | No                       | MS        | QQQ      |          | Varian 3800 GC+ 320-MS    | 10                | ACN                   | DSPE(PSA)                 | VF-1 ms                 | 3                | Both                           | 150        |
| 044              | <2.5% < 20%      | LC           | No                       | MS        | QQQ      |          | API 4000                  | 15                | ACN                   | PSA                       | SpeedRod                | 20               | Manual                         | 153        |
| 046              | <20              | 926          | GC                       | Yes       | MS       | TOF      | Leco                      | 10                | ACN                   | DSPE(PSA)                 | Restek CI Pesticides    | 10               | Both                           | 700        |
| 047              | 0.01             | GC           | No                       | QQQ       |          |          | Varian 1200L              | 10                | ACN                   | DSPE                      | DB-5                    | 5                | Automatic                      | Each Batch |
| 047              | 0.1              | LC           | No                       | QQQ       |          |          | Agilent 6410              | 10                | ACN                   | DSPE                      | C-18                    | 10               | Automatic                      | Each Batch |
| 050              | 2.2              | 4            | GC                       | Yes       | MS       | Q        | 5975C inert XL EI/CI MSD  | 10                | ACN                   | PSA                       | HP5_MS                  | 2                | Automatic                      | 200        |
| 051              | 1.2              | 0.7          | GC                       | Yes       | MS       | IT       | Varian Saturn 2000        | 15                | Acetone               | Partitioning              | FactorFour VF-5ms       | 5                | Both                           | 500        |
| 052              | 93%              | GC           | Yes                      | MS        | Q        |          | Thermo Trace DSQ          | 10                | DCM                   |                           | Supelco SLB-5MS         | 1                | Both                           | 400        |
| 053              |                  | LC           | Yes                      | MS        | QQQ      |          | Varian                    | 12                | ACN                   | Yes                       | C18                     | 10               | Automatic                      | 70         |

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

| PROPOXUR        |                  |              |                          |          |                  |                   |                                    |             |                       |                        |                                |
|-----------------|------------------|--------------|--------------------------|----------|------------------|-------------------|------------------------------------|-------------|-----------------------|------------------------|--------------------------------|
| Laboratory Code | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Detector | Instrument Model | Sample Weight (g) | Extraction Solvent                 | Column Type | Injection Volume (μl) | No. of Compounds       | Frequency of Standard Solution |
|                 |                  |              |                          |          |                  |                   |                                    |             |                       |                        |                                |
| 001             | 60               | none         | LC                       | No       | MS               | QQQ               | API 4000                           | 10          | ACN                   | DSPE                   | Waters-C18<br>2.1x50mmx1.7μm   |
| 003             | 0.30             | 0.05 Da      | LC                       | Yes      | MS               | TOF               | ACQUITY/LCT PREMIER XE             | 15          | MINI LUKE             | No                     | C18                            |
| 004             |                  |              | LC                       | No       | MS               | QQQ               | Applied Biosystems API5000         | 10          | EtOAC                 | Filter                 | HSS T3                         |
| 005             |                  |              | LC                       | No       | MS               | QQQ               | Agilent 6460                       | 10          | EtOAC                 |                        | C18                            |
| 006             | 0.03             |              | LC                       | No       | MS               | QQQ               | Agilent 6410                       | 10          | ACN                   |                        | Eclipse XDB-C18                |
| 008             | 204              | 100          | LC                       | No       | MS               | Q-TRAP            | API 4000 Qtrap                     | 10          | ACN                   |                        | Pursuit XRs Ultra              |
| 009             | 0.00             |              | LC                       | No       | MS               | QQQ               | Waters Aquity TQD                  | 10          | Methanol              | Filter                 | C18 UPLC                       |
| 010             |                  |              | LC                       | No       | MS               | QQQ               | Agilent 6410B                      | 10          | ACN                   | DSPE                   | C18                            |
| 011             |                  |              | LC                       | No       | MS               | Q-TRAP            | ABi 4000                           | 10          | ACN                   |                        | C18                            |
| 013             | 0.7              | 94           | GC                       | Yes      | MS               | Q                 | Agilent 5975                       | 10          | ACN                   | MgSO <sub>4</sub> /PSA | HP5MS                          |
| 014             |                  |              | LC                       | No       | MS               | QQQ               | Acquity UPLC<br>Quattro Premier XE | 15          | ACN                   | QuECHERS               | BEH C18 1.7 μm                 |
| 015             |                  |              | LC                       | No       | MS               | QQQ               | ThermoFinnigan TSQ Quantum         | 10          | ACN                   |                        | XterraC18MS                    |
| 016             |                  | GC           | Yes                      | MS       | Q                | 5973              | 10                                 | ACN         | PSA                   | DB-35ms                | 2                              |
| 017             | 0.0              | -0.52        | LC                       | No       | MS               | QQQ               | HPLC Agilent 1100.<br>MS API 3000  | 10          | ACN                   | DSPE                   | C18 3μm 50x2mm                 |
| 018             | 0.0              | 0.0          | LC                       | No       | MS               | QQQ               | Waters Xevo                        | 10          | Acetone/PE/DCM        | Nas2O4                 | C18                            |
| 019             | 9                | 30           | GC                       | Yes      | MS               | Q                 | Agilent GC7890-MSS75C              | 10          | ACN                   | DSPE                   | HP5-MS                         |
| 020             | <2.5             |              | LC                       | No       | MS               | Q-TRAP            | 3200 Qtrap                         | 10          | ACN                   | PSA                    | C18                            |
| 021             | GC               | Yes          | MS                       | IT       |                  |                   | Variion 4000                       | 10          | ACN                   | DSPE                   | Capillary                      |
| 022             | <0.1 s           | <15%         | LC                       | No       | MS               | QQQ               | Waters Aquity TQD                  | 15          | ACN                   | DSPE                   | UPLC-BEH C18                   |
| 023             | 0.0              |              | LC                       | No       | MS/MS            | Q                 | API 4000                           | 10          | ACN                   | DSPE                   | C18 hydro Phenomenex           |

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

| Laboratory Code | RT Tolerance (s) | Cromatographic Technique | Full Scan | Analyzer | Instrument Model | Sample Weight (g) | Clean-Up                     | Column Type | Injection Volume (μl) | Software         | No. of Compounds        | Frequency of Standard Solution | PROPOXUR    |                       |                                |
|-----------------|------------------|--------------------------|-----------|----------|------------------|-------------------|------------------------------|-------------|-----------------------|------------------|-------------------------|--------------------------------|-------------|-----------------------|--------------------------------|
|                 |                  |                          |           |          |                  |                   |                              |             |                       |                  |                         |                                | Column Type | Injection Volume (μl) |                                |
| 025             | 3                | 10.8                     | GC        | Yes      | MS               | TOF               | LECO Pegasus IV              | 10          | ACN                   | DSPE(PSA)        | HP-5MS                  | 5                              | Both        | 650                   | Weekly                         |
| 026             | 0.0              | 0.0                      | LC        | No       | MS               | QQQ               | Xevo TQ                      | 15          | ACN                   | LLE              | C18                     | 10                             | Both        | 360                   | Each Time                      |
| 027             |                  |                          | LC        | No       | MS               | QQQ               | Agilent 6410                 | 10          | ACN                   | PSA+MgSO4        | Fusion C18              | 10                             | Both        | 108                   | Always                         |
| 028             |                  |                          | GC        | No       | MS               | QQQ               | Quattro micro                | 10          | ACN                   | DSPE             | HP5_MSI                 | 1                              | Both        | 110                   |                                |
| 030             |                  |                          | GC        | Yes      | MS               | TOF               | Leco Pegasus                 | 15          | Acetone DCM.<br>PE    |                  | DB-5                    | 1                              | Automatic   |                       |                                |
| 032             | 0.5              | 5 ppm                    | LC        | Yes      | MS               | Orbitrap          | Exactive                     | 10          | ACN                   | PSA              | C18                     | 5                              | Both        | > 200                 | Always                         |
| 033             | 9                | 20%                      | GC        | Yes      | MS               | Q                 | Agilent GCMSD<br>7890A/5975C | 10          | ACN                   | PSA. C18         | HP-5MS                  | 1                              | Automatic   | 927                   | Each Batch                     |
| 034             | 60               | 10 ppm                   | LC        | Yes      | MS               | TOF               | Agilent 6230                 | 10          | ACN                   |                  | Zorbax eclipse plus C18 | 3                              | Automatic   | 517                   | Not for routine                |
| 039             |                  |                          | LC        | No       | MS               | QQQ               | API 4000QTRAP                | 10          | ACN                   | DSPE             | C18                     | 5                              | Automatic   | 400                   | Daily                          |
| 040             | 5                | 20                       | LC        | No       | MS               | QQQ               | Quattro Premier XE           | 10          | ACN                   |                  | Reversed Phase          | 5                              | Manual      | 320                   | Each Batch                     |
| 041             | 0.0              |                          | GC        | Yes      | MS               | QQQ               | Varian 3800 GC + 320-MS      | 10          | ACN                   | DSPE<br>with PSA | VF-1 ms                 | 3                              | Both        | 500                   | Each Batch                     |
| 042             | 5                | unit                     | GC        | No       | MS               | QQQ               | Varian 320MS                 | 50          | Acetone               | L/L              | DB5                     | 8                              | Both        | 128                   | Each Time                      |
| 044             | <3%              | <20%                     | GC        | No       | MS               | QQQ               | Agilent 7000a                | 15          | Acetone               | ne               | HP5MSI                  | 2                              | Automatic   | 200                   | Each batch                     |
| 045             | 0.0              | 0.0                      | GC        | Yes      | MS               | Q                 | Agilent                      | 50          | Acetone               | DCM              |                         | 1                              | Both        | 220                   | Daily                          |
| 046             | -1.2             | 1.7                      | LC        | Yes      | MS               | Orbitrap          | Exactive                     | 10          | ACN                   |                  | Atlantis T3             | 5                              | Both        | 461                   | 100 compounds with every batch |
| 050             | -0.01            | 9.01                     | LC        | No       | MS               | QQQ               | G6410A                       | 10          | ACN                   | PSA              | Zorbax-Eclipse XDB-C8   | 20                             | Automatic   | 106                   | Always                         |
| 051             | 0.7              | 7.4                      | GC        | Yes      | MS               | T                 | Varian Saturn 2000           | 15          | Acetone               | Partitioning     | FactoFour VF-5ms        | 5                              | Both        | 500                   | Quarterly                      |
| 052             | 95%              | GC                       | Yes       | MS       | Q                | Thermo Trace DSQ  | 10                           | DCM         |                       | Supelco SLB-5MS  | 1                       | Both                           | 400         | Never                 |                                |
| 053             |                  |                          | LC        | Yes      | MS               | QQQ               | Varian                       | 12          | ACN                   | Yes              | C18                     | 10                             | Automatic   | 70                    |                                |

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

| PROTHIOPHOS                                  |                            |          |                |                       |                            |           |                                |                        |                                    |                            |
|--|----------------------------|----------|----------------|-----------------------|----------------------------|-----------|--------------------------------|------------------------|------------------------------------|----------------------------|
| Instrument Model                             | Extraction Solvent         | Clean-Up | Column Type    | Injection Volume (μL) | No. of Compounds           | Software  | Frequency of Standard Solution |                        |                                    |                            |
|  |                            |          |                |                       |                            |           |                                | Sample Weight (g)      | Acquisition Mode                   | Retention Time Range (min) |
| Varian 1200L, GC3800                         | Applied Biosystems API5000 | 10       | ACN            | DSPE                  | Waters C18 2.1x50mmx1.74μm | 5         | Both                           | 600                    | every week                         | 200 pesticides             |
| Agilent 6890/5973                            | MS Q                       | 10       | EIOAC          | Filter                | HSS T3                     | 2         | Both                           | 227                    | Each Batch                         |                            |
| LECO Pegasus IV                              | TOF                        | 10       | EIOAC          | FactorFour, VF-5ms    | 2                          | Automatic | 500                            | 500                    | Each Batch                         |                            |
| Thermo Polaris Q                             | MS IT                      | 10       | ACN            | DSPE                  | HP-5MS                     | 1         | Manual                         | 177                    | Once a Month                       |                            |
| HP 5973 MSD                                  | MS Q                       | 10       | ACN            | PSA                   | Phenomenex ZB-5MS          | 3         | Both                           | 750                    | Each Batch                         |                            |
| HP 5973 MSD                                  | MS Q                       | 10       | ACN            | PSA                   | Capillary GC               | 1         | Automatic                      | 130                    |                                    |                            |
| HP5975C                                      | MS Q                       | 10       | ACN            | DSPE                  | HP-5MSI                    | 20        | Both                           | 111                    | Each Batch                         |                            |
| Saturn 4000 Varian                           | MS IT                      | 10       | ACN            | PSA                   | HP5MS                      | 5         | Both                           | approx. 1,000          | Always                             |                            |
| Agilent 5975                                 | MS Q                       | 10       | ACN            | DSPE                  | DB5MS                      | 1         | Both                           | 350                    | Daily                              |                            |
| Trace DSQ                                    | MS Q                       | 15       | ACN            | MgSO4/PSA             | HP5MS                      | 20        | Automatic                      | 550                    | Yearly                             |                            |
| ThermoFinniganPolarisQ                       | MS IT                      | 10       | ACN            | QUECHERS              | DB5-MS                     | 0.8       | Both                           |                        |                                    |                            |
| Thermo TSQ Quantum GC                        | MS Q                       | 10       | ACN            | DSPE                  | DB-5MS                     | 8         | Both                           | 500                    | Often                              |                            |
| GC Shimadzu GC-2010, MS Shimadzu GCMS-QP2010 | MS Q                       | 10       | ACN            | PSA                   | DB-35ms                    | 2         | Both                           | 525 method 590 library | Targeted Analysis Only             |                            |
| Na2SO4                                       | No QQQ                     | 10       | Acetone/PE/DCM | HP-5MS                | 3                          | Both      | 525 method 590 library         | 164                    | Each Batch                         |                            |
| Agilent GC7890-MS5975C                       | MS Q                       | 10       | ACN            | DSPE                  | HP5-MS                     | 1         | Both                           | 150 method 500 library | Method Setup (and very rare cases) |                            |
| VariAN 4000                                  | MS IT                      | 10       | ACN            | DSPE                  | Capillary                  | 3         | Automatic                      | 450                    | Daily/Weekly                       |                            |
| Agilent 7890A/5975C                          | MS Q                       | 15       | ACN            | DSPE                  | HP5MS                      | 10        | Both                           | 214                    | Once a Day                         |                            |

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

| PROTHIOPHOS     |                  |              |                          |          |                  |                   |                           |                  |           |                                |
|-----------------|------------------|--------------|--------------------------|----------|------------------|-------------------|---------------------------|------------------|-----------|--------------------------------|
| Laboratory Code | RT Tolerance (%) | MS Tolerance | Cromatographic Technique | Detector | Instrument Model | Sample Weight (g) | Column Type               | No. of Compounds | Software  | Frequency of Standard Solution |
| 023             | 0.0              | LC           | No                       | MS/MS    | Q                | API 4000          | 10                        | ACN              | DSPE      | C18 hydro Phenomenex           |
| 025             | 1                | 8.8          | GC                       | Yes      | MS               | TOF               | 10                        | ACN              | DSPE(PSA) | HF-5MS                         |
| 026             | 0.0              | 0.0          | GC                       | Yes      | MS               | IT                | 25                        | Hexane           | LLE       | DB5 MS                         |
| 028             |                  |              | GC                       | No       | MS               | Quattro micro     | 10                        | ACN              | DSPE      | HP5_MS                         |
| 030             |                  |              | GC                       | Yes      | MS               | TOF               | 15                        | Acetone, DCM, PE | DB-5      | 1                              |
| 032             | RT locking       |              | GC                       | Yes      | MS               | GCMSD             | 10                        | ACN              | PSA       | HP5                            |
| 033             | 9                | 20%          | GC                       | Yes      | MS               | Q                 | Agilent GCMSD 7890A/5975C | 10               | PSA, C18  | HF-5MS                         |
| 034             | 12               |              | GC                       | Yes      | MS               | IT                | Saturn 2000               | 30               | EtOAc     | HPGPC                          |
| 035             | 0.01             |              | GC                       | No       | MS               | QQQ               | Waters Micromass          | 10               | ACN       | DB5                            |
| 037             |                  |              | GC                       | Yes      | MS               | Q                 | Agilent 6890N             | 10               | ACN       | PSA                            |
| 039             |                  |              | GC                       | No       | MS               | QQQ               | Agilent 7000B             | 10               | ACN       | DB-5                           |
| 040             | 5                | 20           | LC                       | No       | MS               | QQQ               | Quattro Premier XE        | 10               | ACN       | Reversed Phase                 |
| 041             | 0.6              |              | GC                       | Yes      | MS               | QQQ               | Variion 3800 GC + 320-MS  | 10               | ACN       | DSPE(PSA)                      |
| 042             | 5                | unit         | GC                       | No       | MS               | QQQ               | Variion 320MS             | 50               | Acetone   | L/L                            |
| 044             | <3%              | <20%         | GC                       | No       | MS               | QQQ               | Agilent 7000a             | 15               | Acetone   | ne                             |
| 045             | 0.0              | 0.0          | GC                       | Yes      | MS               | Q                 | Agilent                   | 50               | Acetone   | DCM                            |
| 046             | <20              | 946          | GC                       | Yes      | MS               | TOF               | Leco                      | 10               | ACN       | DSPE(PSA)                      |
| 047             | 0.01             |              | GC                       | No       |                  | QQQ               | Variion 1200L             | 10               | ACN       | DSPE                           |
| 050             | 0.95             | 3.5          | GC                       | Yes      | MS               | Q                 | 5975C inert XL EI/CI/MSD  | 10               | ACN       | PSA                            |
| 051             | 5.5              | 1.2          | GC                       | Yes      | MS               | IT                | Variion Saturn 2000       | 15               | Acetone   | Partitioning                   |
| 052             | 95%              |              | GC                       | Yes      | MS               | Q                 | Thermo trace DSQ          | 10               | DCM       | FactorFour VF-5ms              |
| 053             |                  |              | GC                       | No       | MS               | QQQ               | Variion                   | 7.5              | Acetone   | Supelco SLB-5MS                |
|                 |                  |              |                          |          |                  |                   | Yes                       |                  | DB5 MS    | 1                              |

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

| PYRIDAPHENTHION |                  |              |                          |                                 |          |                  |                        |                           |                  |                            |                                |            |
|-----------------|------------------|--------------|--------------------------|---------------------------------|----------|------------------|------------------------|---------------------------|------------------|----------------------------|--------------------------------|------------|
| Laboratory Code | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Full Scan                       | Detector | Instrument Model | Sample Weight (g)      | Injection Volume (μl)     | No. of Compounds | Software                   | Frequency of Standard Solution |            |
|                 |                  |              |                          |                                 |          |                  |                        |                           |                  |                            |                                |            |
| 001             | 60               | none         | LC No MS QQQ             | API 4000                        | 10       | ACN              | DSPE                   | Waters-C18 2.1x50mmx1.7Åμ | 5                | Both                       | 600                            |            |
| 004             |                  |              | LC No MS QQQ             | Applied Biosystems API5000      | 10       | EtOAc            | Filter                 | HSS T3                    | 2                | Both                       | 227                            |            |
| 005             |                  |              | LC No MS QQQ             | Agilent 6460                    | 10       | EtOAc            |                        | C18                       | 2                | Automatic                  | 500                            |            |
| 008             | 1204             | 974          | GC Yes MS TOF            | LECO Pegasus IV                 | 10       | ACN              | PSA                    | Phenomenex ZB-5MS         | 3                | Both                       | 750                            |            |
| 009             | 0.00             |              | LC No MS QQQ             | Waters Aquity TQD               | 10       | Methanol         | Filter                 | C18 UPLC                  | 6                | Automatic                  | 120                            |            |
| 010             |                  |              | GC Yes MS Q              | HP5973 MSD                      | 10       | ACN              | DSPE                   | HP-5MSI                   | 20               | Both                       | 624                            |            |
| 011             | 5.8              | 0.0          | GC Yes MS Q              | HP5975C                         | 10       | ACN              | PSA                    | HP5MS                     | 5                | Both                       | approx. 1.000                  |            |
| 012             | 30               | 20           | GC Yes MS IT             | Saturn 4000 varian              | 10       | ACN              | DSPE                   | DB5MS                     | 1                | Both                       | 350                            |            |
| 013             | 5.4              | 91           | GC Yes MS Q              | Agilent 5975                    | 10       | ACN              | MgSO <sub>4</sub> /PSA | HP5MS                     | 20               | Automatic                  | 550                            |            |
| 014             |                  |              | LC No MS QQQ             | Acquity UPLC Quattro Premier XE | 15       | ACN              | QuEChERS               | BEH C18 1.7 μm            | 3                | Both                       |                                |            |
| 015             | >70% match       | GC Yes MS IT | ThermoFinniganPolarisQ   | 10                              | ACN      |                  |                        | DB-5MS                    | 8                | Both                       | 500                            |            |
| 016             |                  |              | GC Yes MS Q              | 5973                            | 10       | ACN              | PSA                    | DB-35ms                   | 2                | Both                       | Targeted Analysis Only         |            |
| 017             | 0.0              | -10.51       | LC No MS QQQ             | HPLC Agilent 1100, MS API 3000  | 10       | ACN              | DSPE                   | C18 3μm 50x2mm            | 10               | Both                       | 525 method<br>590 library      |            |
| 018             | 0.0              | 0.0          | LC No MS QQQ             | Waters Xevo                     | 10       | Acetone/PE/DCM   | Na23O4                 | C18                       | 2                | Automatic                  | 250                            |            |
| 020             | <0.5             | GC Yes MS IT | Varian 4000              | 10                              | ACN      | PSA              | 5% diphenyl            | 1                         | Automatic        | 148 method<br>NIST library | Always                         |            |
| 021             |                  | GC Yes MS IT | Varian 4000              | 10                              | ACN      | DSPE             | Capillary              | 3                         | Automatic        | 450                        | Daily/Weekly                   |            |
| 022             | <0.1             | <15%         | LC No MS QQQ             | Waters Aquity TQD               | 15       | ACN              | DSPE                   | UPLC-BEH C18              | 10               | Both                       | 340                            | Once a Day |
| 025             | 2                | 14.1         | GC Yes MS TOF            | LECO Pegasus IV                 | 10       | ACN              | DSPE(PSA)              | HP-5MS                    | 5                | Both                       | 650                            | Weekly     |
| 028             |                  |              | GC Yes MS Q              | HP-5973N                        | 10       | ACN              | DSPE                   | HP5MSI                    | 1                | Both                       |                                |            |
| 032             | 0.5              | 5 ppm        | LC Yes MS Orbitrap       | Exactive                        | 10       | ACN              | PSA                    | C18                       | 5                | Both                       | >200                           | Always     |

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

| PYRIDAPHENTHION |                  |              |                          |           |         |          |                           |                   |                       |                         |                   |                                |
|-----------------|------------------|--------------|--------------------------|-----------|---------|----------|---------------------------|-------------------|-----------------------|-------------------------|-------------------|--------------------------------|
| Laboratory Code | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Full Scan | Detecto | Analyser | Instrument Model          | Sample Weight (g) | Injection Volume (lL) | No. of Compounds        | Software          | Frequency of Standard Solution |
| 033             | 9                | 20%          | GC                       | Yes       | MS      | Q        | Agilent GCMSD 7890A/5975C | 10                | ACN                   | PSA, C18                | HP-5MS            | 1 Automatic                    |
| 034             | 60               | 10 ppm       | LC                       | Yes       | MS      | TOF      | Agilent 6230              | 10                | ACN                   | Zorbax eclipse plus C18 | C18               | 3 Automatic                    |
| 035             |                  | 0.005 Da     | LC                       | Yes       | MS      | Q-TOF    | Bruker Maxis              | 10                | ACN                   | PSA                     | C18 poroshell     | 1 Automatic                    |
| 039             |                  |              | GC                       | No        | MS      | QQQ      | Agilent 7000B             | 10                | ACN                   | DSPE                    | HP-5MS            | 1 Automatic                    |
| 041             | 0.6              |              | GC                       | Yes       | MS      | QQQ      | Varian 3800 GC + 320-MS   | 10                | ACN                   | DSPE(PSA)               | VF-1 ms           | 3 Both                         |
| 044             | <2.5%            | < 20%        | LC                       | No        | MS      | QQQ      | API4000                   | 15                | ACN                   | PSA                     | SpeedRod          | 20 Manual                      |
| 045             | 0.0              | 0.0          | GC                       | Yes       | MS      | Q        | Agilent                   | 50                | Acetone               | DCM                     |                   | 1 Both                         |
| 046             | 1.8              | 2.0 ppm      | LC                       | Yes       | MS      | Orbitrap | Exactive                  | 10                | ACN                   |                         | Atlantis T3       | 5 Both                         |
| 050             | 2.45             | 5.5          | GC                       | Yes       | MS      | Q        | 5975C inert XL EI/CI MSD  | 10                | ACN                   | PSA                     | HP5-MS            | 2 Automatic                    |
| 051             | 4.4              | 6.9          | GC                       | Yes       | MS      | T        | Varian Saturn 2000        | 15                | Acetone               | Partitioning            | FactorFour VF-5ms | 5 Both                         |
| 052             |                  | 88%          | GC                       | Yes       | MS      | Q        | Thermo trace DSQ          | 10                | DCM                   | Supelco SLB-5MS         | Supelco SLB-5MS   | 1 Both                         |
| 053             |                  |              | LC                       | Yes       | MS      | QQQ      | Varian                    | 12                | ACN                   | Yes                     | C18               | 10 Automatic                   |
|                 |                  |              |                          |           |         |          |                           |                   |                       |                         | 70                |                                |

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

| Laboratory Code | RT Tolerance (s) | Cromatographic Technique | Full Scan | Detector | Analyser | Instrument Model       | Sample Weight (g)             | Extraction Solvent | Clean-Up       | Column Type                      | Software                  | Injection Volume (μl) | No. of Compounds        | Frequency of Standard Solution | QUINALPHOS                         |                  |
|-----------------|------------------|--------------------------|-----------|----------|----------|------------------------|-------------------------------|--------------------|----------------|----------------------------------|---------------------------|-----------------------|-------------------------|--------------------------------|------------------------------------|------------------|
|                 |                  |                          |           |          |          |                        |                               |                    |                |                                  |                           |                       |                         |                                | Column Type                        | No. of Compounds |
| 001             | 60               | none                     | LC        | No       | MS       | QQQ                    | API 4000                      | 10                 | ACN            | DSP-E                            | Waters-C18 2.1x50mmx1.7μm | 5                     | Both                    | 600                            | every week 200 pesticides          |                  |
| 003             | 0.30             | 0.05 Da                  | LC        | Yes      | MS       | TOF                    | ACQUITY/LCT PREMIER XE        | 15                 | MINI LUKE      | No                               | C18                       | 5                     | Both                    | 345                            | Once a Day                         |                  |
| 004             |                  | GC                       | No        | MS       | QQQ      | Wafers                 | 10                            | EtOAC              | Filter         | Rxi-5Sil MS/Integra-guard Restek | 10                        | Both                  | 135                     | Each Batch                     |                                    |                  |
| 005             |                  | GC                       | No        | MS       | QQQ      | Varian 1200L GC3800    | 10                            | EtOAC              |                | FactorFour: VF-5ms               | 2                         | Automatic             | 500                     | Each Batch                     |                                    |                  |
| 006             | 0.03             | GC                       | No        | MS       | Q        | Agilent 8890/5973      | 10                            | ACN                | DSP-E          | HP-5MS                           | 1                         | Manual                | 177                     | Once a Month                   |                                    |                  |
| 008             | 875              | 964                      | GC        | Yes      | MS       | TOF                    | LECO Pegasus IV               | 10                 | ACN            | PSA                              | Phenomenex ZB-5MS         | 3                     | Both                    | 750                            | Each Batch                         |                  |
| 009             | 0.00             | GC                       | Yes       | MS       | IT       | Thermo Polaris Q       | 10                            | ACN                | PSA            | Capillary GC                     | 1                         | Automatic             | 130                     |                                |                                    |                  |
| 010             |                  | GC                       | Yes       | MS       | Q        | HP 5973 MSD            | 10                            | ACN                | DSP-E          | HP-5MSI                          | 20                        | Both                  | 111                     | Each Batch                     |                                    |                  |
| 011             | 2.9              | 0.0                      | GC        | Yes      | MS       | Q                      | HP5975C                       | 10                 | ACN            | PSA                              | HP5MS                     | 5                     | Both                    | approx. 1.000                  | Always                             |                  |
| 012             | 30               | 20                       | GC        | Yes      | MS       | IT                     | Scurve 4000 varian            | 10                 | ACN            | DSP-E                            | DB5MS                     | 1                     | Both                    | 350                            | Daily                              |                  |
| 013             | 4.0              | 89                       | GC        | Yes      | MS       | Q                      | Agilent 5975                  | 10                 | ACN            | MGSOr/PSA                        | HP5MS                     | 20                    | Automatic               | 550                            | Yearly                             |                  |
| 014             |                  | GC                       | Yes       | MS       | Q        | Trace DSQ              | 15                            | ACN                | QuECHERS       | DB5-MS                           | 0.8                       | Both                  |                         |                                |                                    |                  |
| 015             | > 70%            | GC                       | Yes       | MS       | IT       | ThermoFinniganPolarisQ | 10                            | ACN                |                | DB-5MS                           | 8                         | Both                  | 500                     | Often                          |                                    |                  |
| 016             |                  | GC                       | Yes       | MS       | Q        | 5973                   | 10                            | ACN                | PSA            | DB-35ms                          | 2                         | Both                  |                         | Targeted Analysis Only         |                                    |                  |
| 017             | -1.2             | -12.21                   | LC        | No       | MS       | QQQ                    | HPLC Agilent 1100 MS API 3000 | 10                 | ACN            | DSP-E                            | C18 3μm 50x2mm            | 10                    | Both                    | 525 method<br>590 library      | Always                             |                  |
| 018             | 0.0              | 0.0                      | GC        | No       | MS       | QQQ                    | Thermo TSQ Quantum GC         | 10                 | Acetone/PE/DCM | Na2SO4                           | RXi-5SiL MS               | 1                     | Automatic               | 164                            | Each Batch                         |                  |
| 019             | 9                | 30                       | GC        | Yes      | MS       | Q                      | Agilent GC7890A-MS5975C       | 10                 | ACN            | DSP-E                            | HP5-MS                    | 1                     | Both                    | 150 method<br>500 library      | Method Setup (and very rare cases) |                  |
| 020             | <0.5             | GC                       | Yes       | MS       | IT       | Varian 4000            | 10                            | ACN                | PSA            | 5 % diphenyl                     | 1                         | Automatic             | 148 method NIST library | Each Batch                     |                                    |                  |

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

| QUINALPHOS      |                 |                          |           |          |                  |                         |                    |                 |                           |                         |                  |
|-----------------|-----------------|--------------------------|-----------|----------|------------------|-------------------------|--------------------|-----------------|---------------------------|-------------------------|------------------|
| Laboratory Code | RT Tolerance(s) | Cromatographic Technique | Full Scan | Detector | Instrument Model | Sample Weight (g)       | Extraction Solvent | Clean-Up        | Column Type               | Injection Volume (µl)   | No. of Compounds |
|                 |                 |                          |           |          |                  |                         |                    |                 |                           |                         |                  |
|                 |                 |                          |           |          |                  |                         |                    |                 |                           |                         |                  |
| 021             |                 | GC                       | Yes       | MS       | IT               | Varian 4000             | 10                 | ACN             | DSPE                      | Capillary               | 3 Automatic      |
| 022             | <0.2            | GC                       | Yes       | MS       | Q                | Agilent 7890A/5975C     | 15                 | ACN             | DSPE                      | HP5MS                   | 10 Both          |
| 023             | 0.0             | LC                       | No        | MS/MS    | Q                | API 4000                | 10                 | ACN             | DSPE                      | C18 hydro Phenomenex    | 40 Manual        |
| 024             |                 | GC                       | No        | MS       | QQQ              | TSQ Thermo              | 10                 | ACN             | Freeze out/ DSPE(PSA)     | TR5MS                   | 2 Both           |
| 025             | 2               | GC                       | Yes       | MS       | TOF              | LECO Pegasus IV         | 10                 | ACN             | DSPE(PSA)                 | HP-5MS                  | 5 Both           |
| 026             | 0.0             | LC                       | No        | MS       | QQQ              | Xevo TQ                 | 15                 | ACN             | LLE                       | C18                     | 10 Both          |
| 028             |                 | GC                       | No        | MS       | QQQ              | Quattro micro           | 10                 | ACN             | DSPE                      | HP5_MS <i>i</i>         | 1 Both           |
| 029             |                 | GC                       | Yes       | MS       | IT               | Varian GCmass 4000      | 10                 | ACN             | DSPE (MgSO <sub>4</sub> ) | Capillary db 5          | 5 Both           |
| 030             |                 | GC                       | Yes       | MS       | TOF              | Leco Pegasus            | 15                 | Acetone, DCM,PE |                           | DB-5                    | 1 Automatic      |
| 032             | RT locking      | GC                       | Yes       | MS       | GCMSD            | Agilent                 | 10                 | ACN             | PSA                       | HP5                     | 10 Both          |
| 033             | 9               | 20%                      | GC        | Yes      | MS               | Q                       | Agilent GCMSD      | 10              | ACN                       | PSA, C18                | HP-5MS           |
|                 |                 |                          |           |          |                  | 7890A/5975C             |                    |                 |                           |                         | 1 Automatic      |
| 034             | 60              | 10 ppm                   | LC        | Yes      | MS               | TOF                     | Agilent 6230       | 10              | ACN                       | Zorbax eclipse plus C18 | 3 Automatic      |
| 035             | 0.005 Da        | LC                       | Yes       | MS       | Q-TOF            | Bruker Maxis            | 10                 | ACN             | PSA                       | C18 poroshell           | 1 Automatic      |
| 037             |                 | GC                       | Yes       | MS       | Q                | Agilent 6890N           | 10                 | ACN             | PSA                       | DB-5                    | 1 Both           |
| 039             |                 | GC                       | No        | MS       | QQQ              | Agilent 7000B           | 10                 | ACN             | DSPE                      | HP-5MS                  | 1 Automatic      |
| 040             | 5               | 20                       | LC        | No       | MS               | QQQ                     | Quattro Premier XE | 10              | ACN                       | Reversed Phase          | 5 Manual         |
| 041             | 0.6             | GC                       | Yes       | MS       | QQQ              | Varian 3800 GC + 320-MS | 10                 | ACN             | DSPE(PSA)                 | VF-1 ms                 | 3 Both           |
| 042             | 6               | unit                     | LC        | No       | MS               | QQQ                     | Waters Premier     | 10              | Methanol                  | C18                     | 5 Both           |
| 044             | <20%            | GC                       | No        | MS       | QQQ              | Agilent7000 <i>a</i>    | 15                 | Aceton          | re                        | HP5MS <i>i</i>          | 2 Automatic      |
| 045             | 0.0             | 0.0                      | GC        | Yes      | MS               | Q                       | Agilent            | DCM             | DCM                       | 1 Both                  | 200              |
|                 |                 |                          |           |          |                  |                         |                    |                 |                           |                         | Daily            |

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

| QUINALPHOS      |                  |              |                          |           |          |          |                          |                   |                       |              |                                   |    |           |     |                                |
|-----------------|------------------|--------------|--------------------------|-----------|----------|----------|--------------------------|-------------------|-----------------------|--------------|-----------------------------------|----|-----------|-----|--------------------------------|
| Laboratory Code | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Full Scan | Detector | Analyzer | Instrument Model         | Sample Weight (g) | Injection Volume (µl) |              |                                   |    |           |     |                                |
|                 |                  |              |                          |           |          |          |                          |                   | No. of Compounds      |              |                                   |    |           |     |                                |
| 046             | <20              | 929          | GC                       | Yes       | MS       | TOF      | Leco                     | 10                | ACN                   | DSPE(PSA)    | Restek CI Pesticides              | 10 | Both      | 700 | 235 compounds with every batch |
| 050             | -0.05            | 6.5          | GC                       | Yes       | MS       | Q        | 5975C inert XL EI/CI MSD | 10                | ACN                   | PSA          | HP5-MS                            | 2  | Automatic | 200 | Always                         |
| 051             | 3.8              | 3.8          | GC                       | Yes       | MS       | IT       | Varian Saturn 2000       | 15                | Acetone               | Partitioning | FactorFour VF-5ms                 | 5  | Both      | 500 | Quarterly                      |
| 052             | 6                | 93%          | LC                       | No        | MS       | QQQ      | API 4000                 | 10                | ACN                   | QuECHERS     | Supelco Ascentis express RP-Amide | 10 | Automatic | 180 | Each Batch                     |
| 053             |                  |              | LC                       | No        | MS       | QQQ      | Varian                   | 12                | ACN                   | Yes          | C18                               | 10 | Automatic | 70  |                                |

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

| Laboratory Code | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Full Scan | Detector | Instrument Model | Extraction Solvent             | Clean-Up | Column Type    | Injection Volume (µl) | No. of Compounds                 | Frequency of Standard Solution | SULFOTOP          |                           |                                       |
|-----------------|------------------|--------------|--------------------------|-----------|----------|------------------|--------------------------------|----------|----------------|-----------------------|----------------------------------|--------------------------------|-------------------|---------------------------|---------------------------------------|
|                 |                  |              |                          |           |          |                  |                                |          |                |                       |                                  |                                | Sample Weight (g) | Software                  |                                       |
| 001             | 60               | none         | LC                       | No        | MS       | QQQ              | API 4000                       | 10       | ACN            | DSPE                  | Waters-C18<br>2.1x50mmx1.7µm     | 5                              | Both              | 600                       | every week 200 pesticides             |
| 003             | 0.30             | 0.05 Da      | LC                       | Yes       | MS       | TOF              | ACQUITY/LCT PREMIER XE         | 15       | MINI LUKE      | No                    | C18                              | 5                              | Both              | 345                       | Once a Day                            |
| 004             |                  |              | GC                       | No        | MS       | QQQ              | Waters                         | 10       | EtOAC          | Filter                | Rxi-5sil MS/integra-guard Restek | 10                             | Both              | 135                       | Each Batch                            |
| 005             |                  |              | GC                       | No        | MS       | QQQ              | Varian 1200L GC3800            | 10       | EtOAC          | FactorFour, VF-5ms    | 2                                | Automatic                      | 500               | Each Batch                |                                       |
| 006             | 0.03             |              | GC                       | No        | MS       | Q                | Agilent 6890/5973              | 10       | ACN            | DSPE                  | HP-5MS                           | 1                              | Manual            | 177                       | Once a Month                          |
| 008             | 604              | 938          | GC                       | Yes       | MS       | TOF              | LECO Pegasus IV                | 10       | ACN            | PSA                   | Phenomenex ZB-5MS                | 3                              | Both              | 750                       | Each Batch                            |
| 009             | 0.00             |              | GC                       | Yes       | MS       | IT               | Thermo Polaris Q               | 10       | ACN            | PSA                   | Capillary GC                     | 1                              | Automatic         | 130                       |                                       |
| 010             |                  |              | GC                       | Yes       | MS       | Q                | HP 5973 MSD                    | 10       | ACN            | DSPE                  | HP-5MSI                          | 20                             | Both              | 111                       | Each Batch                            |
| 011             | -0.7             | 0.0          | GC                       | Yes       | MS       | Q                | HP5975C                        | 10       | ACN            | PSA                   | HP2MS                            | 5                              | Both              | approx. 1.000             | Always                                |
| 012             | 30               | 20           | GC                       | Yes       | MS       | IT               | Saturn 4000 varian             | 10       | ACN            | DSPE                  | DB5MS                            | 1                              | Both              | 350                       | Daily                                 |
| 013             | -8.1             | 98           | GC                       | Yes       | MS       | Q                | Agilent 5975                   | 10       | ACN            | MgSO4/PSA             | HP5MS                            | 20                             | Automatic         | 550                       |                                       |
| 014             |                  |              | GC                       | Yes       | MS       | Q                | Trace DSQ                      | 15       | ACN            | QuICkFRS              | DB5-MS                           | 0.8                            | Both              |                           |                                       |
| 015             |                  | >70%         | GC                       | Yes       | MS       | IT               | ThermoFinnigan/PolarisQ        | 10       | ACN            | PSA                   | DB-5MS                           | 8                              | Both              | 500                       | Often                                 |
| 016             |                  |              | LC                       | No        | MS       | QQQ              | Waters Premier Quattro XE      | 10       | ACN            | DSPE                  | Kinetex XB-C18                   | 10                             | Both              |                           | Targeted Analysis Only                |
| 017             | 0.0              | 4.70         | LC                       | No        | MS       | QQQ              | HPLC Agilent 1100, MS API 3000 | 10       | ACN            | DSPE                  | C18 3µm 50x2mm                   | 10                             | Both              | 525 method<br>590 library | Always                                |
| 018             | 0.0              | 0.0          | GC                       | No        | MS       | QQQ              | Thermo TSQ Quantum GC          | 10       | Acetone/PE/DCM | Na2SO4                | Rxi-5SIL MS                      | 1                              | Automatic         | 164                       | Each Batch                            |
| 019             | 9                | 30           | GC                       | Yes       | MS       | Q                | Agilent GC7890-MS5975C         | 10       | ACN            | DSPE                  | HP5-MS                           | 1                              | Both              | 150 method<br>500 library | Method Setup<br>(and very rare cases) |
| 020             | <0.5             |              | GC                       | Yes       | MS       | IT               | Varian 4000                    | 10       | ACN            | PSA                   | 5% diphenyl                      | 1                              | Automatic         | 148 method NST library    | Each Batch                            |

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

| SULFOTEP                       |                  |              |                          |                        |                           |                  |                       |                      |                         |                       |                                    |                     |
|--------------------------------|------------------|--------------|--------------------------|------------------------|---------------------------|------------------|-----------------------|----------------------|-------------------------|-----------------------|------------------------------------|---------------------|
| Laboratory Code                | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Full Scan              | Detector                  | Instrument Model | Extraction Solvent    | Clean-Up             | Column Type             | Injection Volume (μL) | No. of Compounds                   |                     |
|                                |                  |              |                          |                        |                           |                  |                       |                      |                         |                       |                                    |                     |
| Frequency of Standard Solution |                  |              |                          |                        |                           |                  |                       |                      |                         |                       |                                    |                     |
| 021                            |                  | GC Yes       | MS IT                    | Varian 4000            | 10                        | ACN              | DSPE                  | Capillary            | 3                       | Automatic             | 450                                |                     |
| 022                            | <0.2             | <20%         | GC Yes                   | MS Q                   | Agilent 7890A/5975C       | 15               | ACN                   | DSPE                 | HP5MS                   | 10 Both               | 214                                |                     |
| 023                            | 0.0              | LC No        | MS/MS Q                  | API 4000               | 10                        | ACN              | DSPE                  | C18 hydro Phenomenex | 40                      | Manual                | Each Time                          |                     |
| 024                            |                  | GC No        | MS QQQ                   | TSQ Thermo             | 10                        | ACN              | Freeze out/ DSPE(PSA) | TR5MS                | 2                       | Both                  | 450 At least monthly               |                     |
| 025                            | 1                | 12.1         | GC Yes                   | MS TOF                 | LECO Pegasus IV           | 10               | ACN                   | DSPE(PSA)            | HP-5MS                  | 5 Both                | 650 Weekly                         |                     |
| 026                            | 0.0              | 0.0          | LC No                    | MS QQQ                 | Xevo TQ                   | 15               | ACN                   | LLE                  | C18                     | 10 Both               | 360 Each Time                      |                     |
| 028                            |                  | GC No        | MS QQQ                   | Quattro micro          | 10                        | ACN              | DSPE                  | HP5_MS               | 1                       | Both                  | 110                                |                     |
| 030                            |                  | GC Yes       | MS TOF                   | Leco Pegasus           | 15                        | Acetone, DCM, PE |                       | DB-5                 | 1                       | Automatic             | Always                             |                     |
| 032                            | RT locking       | GC Yes       | MS GCMSD                 | Agilent                | 10                        | ACN              | PSA                   | HP5                  | 10 Both                 | > 600                 | Each Batch                         |                     |
| 033                            | 9                | 20%          | GC Yes                   | MS Q                   | Agilent GCMSD 7890A/5975C | 10               | ACN                   | PSA, C18             | HP-5MS                  | 1                     | Automatic                          | 927 Not for routine |
| 034                            | 60               | 10 ppm       | LC Yes                   | MS TOF                 | Agilent 6230              | 10               | ACN                   |                      | Zorbax eclipse plus C18 | 3                     | Automatic                          | 517                 |
| 035                            | 0.005 Da         | LC Yes       | MS QTOF                  | Bruker Microvis        | 10                        | ACN              | PSA                   | C18 poroshell        | 1                       | Automatic             | 2,000                              |                     |
| 037                            |                  | GC Yes       | MS Q                     | Agilent 8890N          | 10                        | ACN              | PSA                   | DB-5                 | 1                       | Both                  | 80 Daily                           |                     |
| 039                            |                  | GC No        | MS QQQ                   | Agilent 7000B          | 10                        | ACN              | DSPE                  | HP-5MS               | 1                       | Automatic             | 400                                |                     |
| 040                            | 5                | 20           | LC No                    | MS QQQ                 | Quattro Premier XE        | 10               | ACN                   |                      | Reversed Phase          | 5                     | Manual                             | 320 Each Batch      |
| 041                            | 0.5              | GC No        | MS QQQ                   | Varian 3800 GC+ 320-MS | 10                        | ACN              | DSPE(PSA)             | VF-1 ms              | 3 Both                  | 150                   | Each Batch                         |                     |
| 042                            | 5                | unit         | GC No                    | MS QQQ                 | Varian 320MS              | 50               | Acetone               | L/L                  | DB5                     | 8 Both                | 128                                |                     |
| 045                            | 0.0              | 0.0          | GC Yes                   | MS Q                   | Agilent                   | 50               | Acetone               | DCM                  |                         | 1 Both                | 220                                |                     |
| 046                            | 2.4              | 1.5 ppm      | LC Yes                   | MS Orbitrap            | Exactive                  | 10               | ACN                   |                      | Atlantis T3             | 5 Both                | 461 100 compounds with every batch |                     |
| 050                            | -1.15            | 1            | GC Yes                   | MS Q                   | 5975C inert XL EI/CI MSD  | 10               | ACN                   | PSA                  | HP5-MS                  | 2 Automatic           | 200 Always                         |                     |
| 051                            | 2.9              | 0.6          | GC Yes                   | MS IT                  | Varian Saturn 2000        | 15               | Acetone               | Partitioning         | FactorFour VF-5ms       | 5 Both                | 500 Quarterly                      |                     |
| 052                            | 95%              | GC Yes       | MS Q                     | Thermo trace DSQ       | 10                        | DCM              |                       | Supelco SLB-5MS      | 1 Both                  | 400 Never             |                                    |                     |
| 053                            |                  | LC Yes       | MS QQQ                   | Varian                 | 12                        | ACN              | Yes                   | C18                  | 10 Automatic            | 70                    |                                    |                     |

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

| TERBUFOs        |              |              |                           |                                     |                   |                    |                |                            |                        |
|-----------------|--------------|--------------|---------------------------|-------------------------------------|-------------------|--------------------|----------------|----------------------------|------------------------|
| Laboratory Code | RT Tolerance | MS Tolerance | Detector                  | Instrument Model                    | Sample Weight (g) | Extraction Solvent | Clean-Up       | Column Type                | No. of Compounds       |
|                 |              |              |                           |                                     |                   |                    |                |                            | Injection Volume (μl)  |
|                 |              |              |                           |                                     |                   |                    |                |                            | Solution Standard      |
|                 |              |              |                           |                                     |                   |                    |                |                            | Frequency of Standard  |
| 001             | none         | none         | LC No MS                  | QQQ API 4000                        | 10                | ACN                | DSPE           | Waters-C18 2.1x50mmx1.7Åμm | 5 Both                 |
| 004             |              |              | LC No MS                  | QQQ Applied Biosystems API5000      | 10                | EtOAc              | Filter         | HSS T3                     | 2 Both                 |
| 005             |              |              | LC No MS                  | QQQ Agilent 6460                    | 10                | EtOAc              |                | C18                        | 2 Automatic            |
| 006             | 0.03         |              | GC No MS                  | Q Agilent 6890/5973                 | 10                | ACN                | DSPE           | HP-5MS                     | 1 Manual               |
| 008             | 653          | 959          | GC Yes MS                 | TOF LECO Pegasus IV                 | 10                | ACN                | PSA            | Phenomenex ZB-5MS          | 3 Both                 |
| 008             | 308          | 100          | LC No MS                  | Q-TRAP API 4000 Qtrap               | 10                | ACN                |                | Pursuit XRs Ultra          | 6 Both                 |
| 010             |              |              | LC No MS                  | QQQ Agilent 6410B                   | 10                | ACN                | DSPE           | C18                        | 2 Both                 |
| 011             | -0.5         | 0.0          | GC Yes MS                 | Q HF5975C                           | 10                | ACN                | PSA            | HP5MS                      | 5 Both                 |
| 012             | 30           | 20           | GC Yes MS                 | IT Saturn 4000 varian               | 10                | ACN                | DSPE           | DB5MS                      | 1 Both                 |
| 013             | -5           | 98           | GC Yes MS                 | Q Agilent 5975                      | 10                | ACN                | MgSO4/PSA      | HP5MS                      | 20 Automatic           |
| 014             |              |              | LC No MS                  | QQQ Accuity UPLC Quattro Premier XE | 15                | ACN                | QuEChERS       | BEH C 18 1.7 μm            | 3 Both                 |
| 015             | >70%         | GC Yes MS    | IT ThermofinniganPolarisQ | 10                                  | ACN               |                    |                | DB-5MS                     | 8 Both                 |
| 016             |              |              | GC Yes MS                 | Q 5973                              | 10                | ACN                | PSA            | DB-35ms                    | 2 Both                 |
| 017             | -1.8         | -4.66        | LC No MS                  | QQQ HPLC Agilent 1100. MS API 3000  | 10                | ACN                | DSPE           | Kinetex XB-C 18            | 10 Both                |
| 018             | 0.0          | 0.0          | LC No MS                  | QQQ Waters Xevo                     | 10/Acetone/PE/DCM | Nα2SO4             | C18 3μm 50x2mm | 10 Both                    | 525 method 590 library |
| 019             | 9            | 30           | GC Yes MS                 | Q Agilent GC7890-MSS975C            | 10                | ACN                | DSPE           | HP5-MS                     | 2 Automatic            |
| 020             | <0.5         |              | GC Yes MS                 | IT Varian 4000                      | 10                | ACN                | PSA            | 5 % diphenyl               | 1 Automatic            |
| 021             |              |              | GC Yes MS                 | IT Varian 4000                      | 10                | ACN                | DSPE           | Capillary                  | 3 Automatic            |
|                 |              |              |                           |                                     |                   |                    |                |                            | Daily/Weekly           |

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

| TERBUFOOS       |              |              |                           |           |          |                          |                            |                    |           |                      |                         |                  |           |                                |                           |
|-----------------|--------------|--------------|---------------------------|-----------|----------|--------------------------|----------------------------|--------------------|-----------|----------------------|-------------------------|------------------|-----------|--------------------------------|---------------------------|
| Laboratory Code | RT Tolerance | MS Tolerance | Chromatographic Technique | Full Scan | Defector | Instrument Model         | Sample Weight (g)          | Extraction Solvent | Clean-Up  | Column Type          | Injection Volume (μl)   | No. of Compounds | Software  | Frequency of Standard Solution |                           |
|                 |              |              |                           |           |          |                          |                            |                    |           |                      |                         | 340              |           |                                |                           |
| 022             | <0.1         | <15%         | LC                        | No        | MS       | QQQ                      | Waters Aquity TQD          | 15                 | ACN       | DSPE                 | UPLC-BEH C18            | 10               | Both      | Once a Day                     |                           |
| 023             | 0.0          | LC           | No                        | MS/MS     | Q        | API 4000                 | 10                         | ACN                | DSPE      | C18 hydro Phenomenex | 40                      | Manual           | Each Time |                                |                           |
| 025             | 2            | 12.5         | GC                        | Yes       | MS       | TOF                      | LECO Pegasus IV            | 10                 | ACN       | DSPE(PSA)            | HP-5MS                  | 5                | Both      | Weekly                         |                           |
| 026             | 0.0          | 0.0          | LC                        | No        | MS       | QQQ                      | Xevo TQ                    | 15                 | ACN       | LLE                  | C18                     | 10               | Both      | Each Time                      |                           |
| 028             |              | GC           | Yes                       | MS        | Q        | HP-5973N                 | 10                         | ACN                | DSPE      | HP5MSi               | 1                       | Both             |           |                                |                           |
| 030             |              | GC           | No                        | MS        | QQQ      | Quattro Premier          | 10                         | ACN                | DSPE      | C18                  | 5                       | Both             |           |                                |                           |
| 032             |              | GC           | Yes                       | MS        | GCMSD    | Leco Pegasus             | 15                         | Acetone. DCM. PE   |           | DB-5                 | 1                       | Automatic        |           |                                |                           |
| 033             | 9            | 20%          | GC                        | Yes       | MS       | Q                        | Agilent GC/MSD 7890A/5975C | 10                 | ACN       | PSA. C18             | HP5                     | 10               | Both      | >600                           |                           |
| 034             | 60           | 10 ppm       | LC                        | Yes       | MS       | TOF                      | Agilent 6230               | 10                 | ACN       |                      | Zorbax eclipse plus C18 | 3                | Automatic | Always                         |                           |
| 034             | 2.50%        | LC           | No                        | MS/MS     | MS/MS    | Waters Premier XE        | 10                         | ACN                |           | Acquity UPLC HSS T3  | 20                      | Both             | 200       | Routine Target Suite           |                           |
| 034             | 2.50%        | IC           | No                        | MS/MS     | MS/MS    | Waters Premier XF        | 10                         | ACN                |           | Acquity UPLC HSS T3  | 20                      | Both             | 200       | Routine Target Suite           |                           |
| 037             |              | GC           | Yes                       | MS        | Q        | Agilent 6890N            | 10                         | ACN                | PSA       | DB-5                 | 1                       | Both             | 80        |                                |                           |
| 039             |              | GC           | No                        | MS        | QQQ      | Agilent 7000B            | 10                         | ACN                | DSPE      | HP-5MS               | 1                       | Automatic        | 400       | Daily                          |                           |
| 040             | 5            | 20           | LC                        | No        | MS       | QQQ                      | Quattro Premier XE         | 10                 | ACN       |                      | Reversed Phase          | 5                | Manual    | 320                            | Routine Target Suite      |
| 041             | 0.6          | GC           | No                        | MS        | QQQ      | Varian 3800 GC+ 320-N/MS | 10                         | ACN                | DSPE(PSA) | VF-1 ms              | 3                       | Both             | 150       | Each Batch                     |                           |
| 042             | 5            | unit         | GC                        | No        | MS       | QQQ                      | Varian 320MS               | 50                 | Acetone   | L/L                  | DB5                     | 8                | Both      | 128                            | Each Time                 |
| 045             | 0.0          | 0.0          | GC                        | Yes       | MS       | Q                        | Agilent                    | 50                 | Acetone   | DCM                  |                         | 1                | Both      | 220                            | Daily                     |
|                 | -6.6         | 1.1 ppm      | LC                        | Yes       | MS       | Orbitrap                 | Exactive                   | 10                 | ACN       |                      | Atlantis T3             | 5                | Both      | 461                            | 100 compounds every batch |
| 046             | 0.0          | -0.8 ppm     | LC                        | Yes       | MS       | Orbitrap                 | Exactive                   | 10                 | ACN       |                      | Atlantis T3             | 5                | Both      | 461                            | 100 compounds every batch |
|                 | -1.2         | -2.9 ppm     | LC                        | Yes       | MS       | Orbitrap                 | Exactive                   | 10                 | ACN       |                      | Atlantis T3             | 5                | Both      | 461                            | 100 compounds every batch |
| 047             | 0.0          |              | LC                        | No        |          | QQQ                      | Agilent 6410               | 10                 | ACN       | DSPE                 | C-18                    | 10               | Automatic | Each Batch                     |                           |
| 047             | 0.0          |              | LC                        | No        |          | QQQ                      | Agilent 6410               | 10                 | ACN       | DSPE                 | C-18                    | 10               | Automatic | Each Batch                     |                           |
| 050             | -0.7         | 3            | GC                        | Yes       | MS       | Q                        | 5975C inert XL EI/CI MSD   | 10                 | ACN       | PSA                  | HP5-MS                  | 2                | Automatic | 200                            | Always                    |
| 051             | 3.9          | 0.5          | GC                        | Yes       | MS       | IT                       | Varian Saturn 2000         | 15                 | Acetone   | Partitioning         | FactorFour VF-5ms       | 5                | Both      | 500                            | Quarterly                 |

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

| TERBUFO\$                |               | Software           |          | No. of Compounds | Frequency of Standard Solution |           |
|--------------------------|---------------|--------------------|----------|------------------|--------------------------------|-----------|
| Instrument Model         |               | Extraction Solvent | Clean-Up |                  |                                |           |
| Analyser                 |               |                    |          |                  |                                |           |
| Detector                 |               |                    |          |                  |                                |           |
| Cromatographic Technique | Full Scan     |                    |          |                  |                                |           |
| MS Tolerance             | GC Yes MS Q   | Thermo trace DSQ   | DCM      | Supelco SB-5MS   | 1                              | Both      |
| RT Tolerance             | LC Yes MS QQQ | Varian             | ACN      | C18              | 10                             | Automatic |
| Laboratory Code          |               |                    |          |                  | 400                            | Never     |
| 052                      | 97%           |                    |          |                  | 70                             |           |
| 053                      |               |                    |          |                  |                                |           |

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

| TERBUTHYLAZINE  |                  |                          |                    |                  |                   |                                |             |                       |                              |                                |                           |                        |                                    |
|-----------------|------------------|--------------------------|--------------------|------------------|-------------------|--------------------------------|-------------|-----------------------|------------------------------|--------------------------------|---------------------------|------------------------|------------------------------------|
| Laboratory Code | RT Tolerance (s) | Cromatographic Technique | Detector Full Scan | Instrument Model | Sample Weight (g) | Clean-Up                       | Column Type | Injection Volume (μl) | No. of Compounds             | Frequency of Standard Solution |                           |                        |                                    |
| 001             | none             | LC                       | No                 | MS QQQ           | API 4000          | 10                             | ACN         | DSP-E                 | Waters C18<br>2.1x50mmx1.7μm | 5 Both                         | every week 200 pesticides |                        |                                    |
| 003             | 0.30             | 0.05 Da                  | LC                 | Yes              | MS TOF            | ACQUITY/ICT PREMIER XE         | 15          | MINI LUKE             | No                           | C18                            | 5 Both                    | Once a Day             |                                    |
| 004             |                  |                          | LC                 | No               | MS QQQ            | Applied Biosystems API5000     | 10          | EFOAC                 | Filter                       | HSS T3                         | 2 Both                    | Each Batch             |                                    |
| 005             |                  |                          | LC                 | No               | MS QQQ            | Agilent 6460                   | 10          | EFOAC                 |                              | C18                            | 2 Automatic               | 500                    |                                    |
| 006             | 0.04             |                          | LC                 | No               | MS QQQ            | Agilent 6410                   | 10          | ACN                   | Eclipse XDB-C18              | 4 Manual                       | 123                       | Always                 |                                    |
| 008             | 653              | 956                      | GC                 | Yes              | MS TOF            | LECO Pegasus IV                | 10          | ACN                   | PSA                          | Phenomenex ZB-5MS              | 3 Both                    | 750                    | Each Batch                         |
| 010             |                  |                          | GC                 | Yes              | MS Q              | HP 5973 MSD                    | 10          | ACN                   | DSP-E                        | HP-5MSI                        | 20 Both                   | 111                    | Each Batch                         |
| 011             |                  |                          | LC                 | No               | MS Q-TRAP         | ABI 4000                       | 10          | ACN                   |                              | C18                            | 55 Both                   | approx. 580            | Always                             |
| 012             | 30               | 20                       | LC                 | Yes              | MS QQQ            | API 3200 QT                    | 10          | ACN                   | DSP-E                        | Fusion C18                     | 20 Automatic              | 150                    | Daily                              |
| 013             | 11.4             | 98                       | GC                 | Yes              | MS Q              | Agilent 5975                   | 10          | ACN                   | MGSO4/PSA                    | HP5MS                          | 20 Automatic              | 550                    |                                    |
| 014             |                  |                          | LC                 | No               | MS QQQ            | Acquity UPC Quattro Premier XE | 15          | ACN                   | QuEChERS                     | BEH C18 1.7 μm                 | 3 Both                    |                        |                                    |
| 015             |                  |                          | LC                 | No               | MS QQQ            | ThermoFinnigan TSQQuantum      | 10          | ACN                   |                              | XterraC18MS                    | 20 Automatic              | 130                    | On a Daily Basis                   |
| 016             |                  |                          | LC                 | No               | MS QQQ            | Waters Premier Quattro XE      | 10          | ACN                   | PSA                          | Kinetex XB-C18                 | 10 Both                   | 525 method 590 library | Targeted Analysis Only             |
| 017             | 0.0              | 10.55                    | LC                 | No               | MS QQQ            | HPLC Agilent 1100, MS API 3000 | 10          | ACN                   | DSP-E                        | C18 3μm 50x2mm                 | 10 Both                   |                        | Always                             |
| 018             | 0.0              | 0.0                      | GC                 | No               | MS QQQ            | Thermo TSQ Quantum GC          | 10          | Acetone/PEDCM         | Nd2SO4                       | RXi-5SiL MS                    | 1 Automatic               | 164                    | Each Batch                         |
| 019             | 9                | 30                       | GC                 | Yes              | MS Q              | Agilent GC7890-MSS975C         | 10          | ACN                   | DSP-E                        | HP5-MS                         | 1 Both                    | 150 method 500 library | Method Setup (and very rare cases) |
| 021             |                  |                          | GC                 | Yes              | MS IT             | Varian 4000                    | 10          | ACN                   | DSP-E                        | Capillary                      | 3 Automatic               | 450                    | Daily/Weekly                       |
| 022             | <0.2             | <20%                     | GC                 | Yes              | MS Q              | Agilent 7890A/5975C            | 15          | ACN                   | DSP-E                        | HP5MS                          | 10 Both                   | 214                    | Once a Day                         |
| 023             | 0.0              |                          | LC                 | No               | MS/MS Q           | API 4000                       | 10          | ACN                   | DSP-E                        | C18 hydro Phenomenex           | 40 Manual                 |                        | Each Time                          |

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

| TERBUTHYLAZINE   |                  |              |                          |          |                  |                          |                           |             |                                |                |                      |                  |            |                                |
|------------------|------------------|--------------|--------------------------|----------|------------------|--------------------------|---------------------------|-------------|--------------------------------|----------------|----------------------|------------------|------------|--------------------------------|
| Laboratory Code  | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Detector | Instrument Model | Sample Weight (g)        | Clean-Up                  | Column Type | Injection Volume (μL)          |                |                      |                  |            |                                |
| No. of Compounds | Software         |              |                          |          |                  |                          |                           |             |                                |                |                      |                  |            |                                |
|                  |                  |              |                          |          |                  |                          |                           |             | Frequency of Standard Solution |                |                      |                  |            |                                |
| 024              | 12.6             | LC           | No                       | MS       | QQQ              | API 4000                 | 10                        | ACN         | Freeze out/ DSPE(PSA)          | 8 Both         | 400                  | At least monthly |            |                                |
| 025              | 2                | 14.6         | GC                       | Yes      | MS               | TOF                      | LECO Pegasus IV           | 10          | ACN                            | DSPE(PSA)      | 5 Both               | 650              | Weekly     |                                |
| 026              | 0.0              | 0.0          | LC                       | No       | MS               | QQQ                      | Xevo TQ                   | 15          | ACN                            | LE             | 10 Both              | 360              | Each Time  |                                |
| 028              |                  |              | GC                       | No       | MS               | QQQ                      | Quattro micro             | 10          | ACN                            | DSPE           | HP5_MSI              | 1 Both           | 110        |                                |
| 030              |                  |              | GC                       | Yes      | MS               | TOF                      | Leco Pegasus              | 15          | Acetone, DCM, PE               |                | DB-5                 | 1 Automatic      |            |                                |
| 032              | RT locking       | GC           | Yes                      | MS       | GCMSD            | Agilent                  | 10                        | ACN         | PSA                            | HP5            | 10 Both              | > 600            | Always     |                                |
| 033              | 9                | 20%          | GC                       | Yes      | MS               | Q                        | Agilent GCMSD 7890A/5975C | 10          | ACN                            | PSA, C18       | HP-5MS               | 1 Automatic      | 927        | Each Batch                     |
| 034              | 12               |              | GC                       | Yes      | MS               | IT                       | Saturn 2000               | 30          | EtOAc                          | HPGPC          | DB-5 MS              | 2 Automatic      | 380        | Not for routine                |
| 035              | 0.005 Da         | LC           | Yes                      | MS       | Q-TOF            | Bruker Maxis             | 10                        | ACN         | PSA                            | C18 poroshell  | 1 Automatic          | 2000             |            |                                |
| 036              |                  | GC           | No                       | MS       | Q                | Agilent Tech. 5973 inert | 15                        | Acetone     |                                | HP-5MS         | 2 Both               |                  | Always     |                                |
| 037              |                  | GC           | Yes                      | MS       | Q                | Agilent 6890N            | 10                        | ACN         | PSA                            | DB-5           | 1 Both               | 80               |            |                                |
| 039              |                  | GC           | No                       | MS       | QQQ              | Agilent 7000B            | 10                        | ACN         | DSPE                           | HP-5MS         | 1 Automatic          | 400              | Daily      |                                |
| 040              | 5                | 20           | LC                       | No       | MS               | QQQ                      | Quattro Premier XE        | 10          | ACN                            | Reversed Phase | 5 Manual             | 320              | Each Batch |                                |
| 041              | 0.6              |              | GC                       | No       | MS               | QQQ                      | Varian 3800 GC+ 320-MS    | 10          | ACN                            | DSPE(PSA)      | VF-1 ms              | 3 Both           | 150        | Each Batch                     |
| 042              | 6                | unit         | LC                       | No       | MS               | QQQ                      | Waters Premier            | 10          | Methanol                       |                | C18                  | 5 Both           | 201        | Each Time                      |
| 044              | <3%              | <20%         | GC                       | No       | MS               | QQQ                      | Agilent 7000a             | 15          | Acetone                        | ne             | HP5MSI               | 2 Automatic      | 200        | Each batch                     |
| 045              | 0.0              | 0.0          | GC                       | Yes      | MS               | Q                        | Agilent                   | 50          | Acetone                        | DCM            |                      | 1 Both           | 220        | Daily                          |
| 046              | <20              | 932          | GC                       | Yes      | MS               | TOF                      | Leco                      | 10          | ACN                            | DSPE(PSA)      | Restek CI Pesticides | 10 Both          | 700        | 235 compounds with every batch |
| 047              | 0.1              |              | LC                       | No       | MS               | QQQ                      | Agilent 6410              | 10          | ACN                            | DSPE           | C-18                 | 10 Automatic     |            | Each Batch                     |
| 049              | 60               | 20 %         | LC                       | No       | MS               | QQQ                      | Varian 310-MS             | 10          | ACN                            | DSPE           | XDB-C18              | 20 Automatic     | 91         | Every 15 days                  |
| 050              | 1.4              | 8            | GC                       | Yes      | MS               | Q                        | 5975C inert XL EI/CI MSD  | 10          | ACN                            | PSA            | HP5-MS               | 2 Automatic      | 200        | Always                         |

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

| TERBUTHYLAZINE  |                  |              |                          |           |          |          |                    |                   |                                |
|-----------------|------------------|--------------|--------------------------|-----------|----------|----------|--------------------|-------------------|--------------------------------|
| Laboratory Code | RT Tolerance (s) | MS Tolerance | Cromatographic Technique | Full Scan | Detector | Analyser | Instrument Model   | Sample Weight (g) | Injection Volume (μl)          |
|                 |                  |              |                          |           |          |          |                    | No. of Compounds  | Frequency of Standard Solution |
| 051             | 2.8              | 1.0          | GC                       | Yes       | MS       | IT       | Varian Saturn 2000 | 15                | Acetone                        |
| 052             | 6                | 96%          | LC                       | No        | MS       | QQQ      | API 4000           | 10                | ACN                            |
| 053             |                  |              | LC                       | Yes       | MS       | QQQ      | Varian             | 12                | ACN                            |
|                 |                  |              |                          |           |          |          |                    | Yes               | C18                            |
|                 |                  |              |                          |           |          |          |                    |                   | 10 Automatic                   |
|                 |                  |              |                          |           |          |          |                    |                   | 70                             |
|                 |                  |              |                          |           |          |          |                    |                   | Each Batch                     |
|                 |                  |              |                          |           |          |          |                    |                   | Daily                          |

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

| TOLFENPYRAD      |                   |                    |                            |              |                                |                   |              |                           |            |  |
|------------------|-------------------|--------------------|----------------------------|--------------|--------------------------------|-------------------|--------------|---------------------------|------------|--|
| Instrument Model | Sample Weight (g) | Extraction Solvent | Clean-Up                   | Column Type  | Injection Volume (μl)          | No. of Compounds  | Solution     | Frequency of Standard     | Software   |  |
| 001 none         | 0.05 Da           | LC No MS QQQQ      | API 4000                   | 10 ACN       | DSPE Waters-C18 2.1x50mmx1.7Åμ | 5 Both            | 600          | Every week 200 pesticides |            |  |
| 003 0.30         | 0.05 Da           | LC Yes MS TOF      | ACQUITY/LCT PREMIER XE     | 15 MINI LUKE | No                             | C18               | 5 Both       | 345                       | Once a Day |  |
| 004              |                   | LC No MS QQQQ      | Applied Biosystems API5000 | 10 EtOAc     | Filter                         | HSS T3            | 2 Both       | 104                       | Each Batch |  |
| 005              |                   | LC No MS QQQQ      | Agilent 6460               | 10 EtOAc     |                                | C18               | 2 Automatic  | 500                       | Each Batch |  |
| 008 474          | 100               | LC No MS Q-Trap    | API 4000 Qtrap             | 10 ACN       |                                | Pursuit XRs Ultra | 6 Both       | 250                       | Each Batch |  |
| 011 15.9         | 0.0               | GC Yes MS Q        | HP5975C                    | 10 ACN       | PSA                            | HP5MS             | 5 Both       | approx. 1.000             | Always     |  |
| 013 15.5         | 88                | GC Yes MS Q        | Agilent 5975               | 10 ACN       | MgSO4/PSA                      | HP5MS             | 20 Automatic | 550                       | Yearly     |  |
| 014              |                   | GC Yes MS Q        | Trace DSQ                  | 15 ACN       | QuEChERS                       | DB5-MS            | 0.8 Both     |                           |            |  |
| 022 <0.1         | <1%               | LC No MS QQQQ      | Waters Aquity TQD          | 15 ACN       | DSPE                           | UPLC-BEH C18      | 10 Both      | 340                       | Once a Day |  |
| 025 5            | 37.2              | GC Yes MS TOF      | LECO Pegasus IV            | 10 ACN       | DSPE(PSA)                      | HP-5MS            | 5 Both       | 650                       | Weekly     |  |
| 028              |                   | LC No MS QQQQ      | Quattro Premier            | 10 ACN       | DSPE                           | C18               | 5 Both       |                           |            |  |
| 032 RT locking   | GC Yes MS GCMSD   | Agilent            | 10 ACN                     | PSA          | HP5                            | 10 Both           | > 600        | Always                    |            |  |
| 033 9            | 20%               | GC Yes MS Q        | Agilent GCMSD 7890A/5975C  | 10 ACN       | PSA, C18                       | HP-5MS            | 1 Automatic  | 927                       | Each Batch |  |



# Protocol

## EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUITS AND VEGETABLES SCREENING METHODS 03 - 2011

### **Introduction:**

Over recent years inter-laboratory tests for pesticide screening method were well accepted by laboratories. Therefore, the EUR-LFV has decided to follow on this year with a new test. The main reason for this broad acceptance comes from the fact that many laboratories have invested in new higher mass accuracy MS systems that allow them to greatly increase the scope of their multiresidue methods.

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

Another reason for conducting these screening method proficiency tests 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 the near future, there is the need to develop new software.

Furthermore, this year, the Coordinated Multiannual Control Programme of the Union (Commission Regulation (EU) No 915/2010) allows member state laboratories to use multi-residue methods to conduct qualitative screening methods on up to 15 % of the samples taken.

The EUR-LFV 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")

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 sample for this EUPT-FV-SM03 will take into account the following considerations:

- Pesticides that are **not included** in the Coordinated Multiannual Community Control Programme for 2011 (Regulation (EC) No. 915/2010).
- Pesticides that are particularly acutely toxic and/or have low ARfD values.

Regulation (EC) No 882/2004 lays down the general tasks, duties and requirements for EUR-LFs for Food, Feed and Animal Health. Among these tasks is the provision to independently organise comparative tests. As we have done for the last two years, the EUR-LF 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-03 is directed at all National Reference Laboratories (**NRLs**) and all Official Laboratories (**Ofls** for Fruits and Vegetables) in the EU Member States. Laboratories outside this EUR-LF/NRL/Ofls-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 residue detection in mandarin. The mandarins to be used in this study were grown in Malaga, Spain.

The pesticide treatments on the mandarins will be carried out post-harvest by spiking with a mixture of standard solutions. The mandarins will then be frozen (using liquid nitrogen), chopped, homogenized and subsampled 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.

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 sample 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 EURL-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. Sample delivery will cost **150 Euros**, except for those Laboratories already participating in EUPT-FV13, which will not be charged. If participation is solely in EUPT-FV-SM-03, then the payment is expected well in advance of the scheduled shipment date. 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 sample has been accepted. If no test material has been received by 29<sup>th</sup> January 2011, please contact the Organiser by e-mail ([pmedina@ual.es](mailto:pmedina@ual.es) or [omalato@ual.es](mailto:omalato@ual.es))
5. The participant laboratories must respect the deadline for submitting the results – 72 hours after the sample 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 EURL-FV web site. This report will include information regarding the design of the test, the evaluation of the participants' results as well as graphical displays of the results and any conclusions. Other relevant information considered of value may also be included.

#### **Sample Amount**

Participants will receive:

- Approximately 300 g of mandarin test material with spiked pesticides, labelled as **EUPT-FV-SM-03 Sample**
- Approximately 300 g of 'blank' mandarin test material. **Note: the 'blank' sample will be the same if the laboratory also participated in EUPT-FV13.**

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

### **Shipment of Samples**

All samples 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 (24<sup>th</sup> January 2011). 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 Sample 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.eupt-sm.com> by filling in 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 sample has been received and accepted.  
If any laboratory has not received the test material by 31<sup>st</sup> January 2011, 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 EURL –FV web site: <http://www.eupt-sm.com>  
As there is no Target Pesticide List for this PT, the laboratory will have to enter the name of the pesticides that they detected and related information concerning the procedure for detecting it.

### **Calendar:**

| <b>Activity</b>  | <b>Date</b>                                     |
|--|---|
| Publish the Calendar and Matrix on the Web page  | October 2010                                    |
| Receipt of Application Form from invited laboratories                                  | 1 <sup>st</sup> -30 <sup>th</sup> November 2010 |
| Specific Protocol published on the Web site  | January 2011                                    |
| Sample distribution  | 24th January 2011                               |
| Deadline for informing Organiser of sample 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.  | February 2011                                   |
| Final Report distributed to the Laboratories.  | October 2011                                    |

### **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.

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

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

### **Evaluation of the Results:**

The procedures used for the evaluation of results will be based mainly around "not reported pesticides" and "other reported pesticides". After receiving the results, the Organiser may consider further evaluation that could highlight important information received. Therefore:

#### 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, or (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 an "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 by, for example, 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 detections and full identifications.
- The Scientific Committee considered that the term "Other Reported Pesticide" is more suitable than "False Positive" for the EUPT-FV-SM.

#### Not Reported Pesticides

These will be considered as the absence of a result for any pesticide present but 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.

#### Organiser Note:

- The term "False Negative" has been changed for 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.

### **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. EURL-FV        | amadeo@ual.es  | +34 950015034  |               |
| Paula Medina Pastor.                     | EURL-FV        | pmedina@ual.es | +34 950014102 |
| Octavio Malato Rodríguez. EURL-FV        | omalato@ual.es | +34 950015531  |               |
| Ms. Noelia Belmonte, Chemist.            |                | EURL-FV        |               |
| Ms. María Ángeles Martínez Uroz, Chemist |                | EURL-FV        |               |
| Dr. Milagros Mezcua, Senior Chemist.     |                | EURL-FV        |               |
| Ms. Ana Lozano, Chemist.                 |                | EURL-FV        |               |

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

### **Quality Control Group**

Dr. Antonio Valverde, University of Almería, Spain.

Mr. Stewart Reynolds, Senior Chemist from FERA, York, United Kingdom

### **Statistical Group**

Dr. Carmelo Rodriguez, Senior Mathematics. University of Almeria, Spain

### **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 Anastassiades, Senior Chemist, CVUA, Stuttgart, Germany.

Dr. Metter Erecius Poulsen, Senior Chemist, NFI, Copenhagen, Denmark.

Dr. Ralf Lippold, Senior Chemist, CVUA, Freiburg, Germany.

Dr. Hans Mol, Senior Chemist, RIKILT, Wageningen, The Netherlands.

Dr. Magnus Jezussek, Senior Chemist, Erlangen, Germany.

Dr. Darinka Stajnbaher, Senior Chemist, Maribor, Slovenia.

**Application Form****General Data**

|                  |   |   |                |
|------------------|---|---|----------------|
| <b>NRL in FV</b> | <b>No of Analysis by MRM done last year</b> | <b>No EU Official Samples Received last year from Coordinated Programme</b> | <b>Country</b> |
|------------------|---|---|----------------|

Yes  No



**Laboratory Data****Laboratory name**

**Laboratory Address**

|                     |                      |
|---------------------|----------------------|
| <b>Contact Name</b> | <input type="text"/> |
|---------------------|----------------------|

**Street**

|               |                      |                    |                      |             |                      |
|---------------|----------------------|--------------------|----------------------|-------------|----------------------|
| <b>Number</b> | <input type="text"/> | <b>Postal code</b> | <input type="text"/> | <b>City</b> | <input type="text"/> |
|---------------|----------------------|--------------------|----------------------|-------------|----------------------|

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

|               |                      |
|---------------|----------------------|
| <b>Street</b> | <input type="text"/> |
|---------------|----------------------|

|               |                      |                    |                      |             |                      |                |                      |
|---------------|----------------------|--------------------|----------------------|-------------|----------------------|----------------|----------------------|
| <b>Number</b> | <input type="text"/> | <b>Postal code</b> | <input type="text"/> | <b>City</b> | <input type="text"/> | <b>Country</b> | <input type="text"/> |
|---------------|----------------------|--------------------|----------------------|-------------|----------------------|----------------|----------------------|

|   |                                   |                                |              |
|---|-----------------------------------|--------------------------------|--------------|
| <b>Telephone including country code</b> | <b>Fax Including country code</b> | <b>Mobile (Optional field)</b> | <b>Email</b> |
|---|-----------------------------------|--------------------------------|--------------|




**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**

**Payment agreement**

**Invoice type**

12/22/2010

Yes  No

Yes  No

Invoice  Receipt  Nothing

**Invoice Laboratory Address**

|                   |                      |                        |                      |
|-------------------|----------------------|------------------------|----------------------|
| <b>Vat number</b> | <input type="text"/> | <b>Laboratory name</b> | <input type="text"/> |
|-------------------|----------------------|------------------------|----------------------|

**Street**

|               |                      |                    |                      |             |                      |                |                      |
|---------------|----------------------|--------------------|----------------------|-------------|----------------------|----------------|----------------------|
| <b>Number</b> | <input type="text"/> | <b>Postal code</b> | <input type="text"/> | <b>City</b> | <input type="text"/> | <b>Country</b> | <input type="text"/> |
|---------------|----------------------|--------------------|----------------------|-------------|----------------------|----------------|----------------------|

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

|                        |                      |
|------------------------|----------------------|
| <b>Laboratory name</b> | <input type="text"/> |
|------------------------|----------------------|

**Street**

|               |                      |                    |                      |             |                      |                |                      |
|---------------|----------------------|--------------------|----------------------|-------------|----------------------|----------------|----------------------|
| <b>Number</b> | <input type="text"/> | <b>Postal code</b> | <input type="text"/> | <b>City</b> | <input type="text"/> | <b>Country</b> | <input type="text"/> |
|---------------|----------------------|--------------------|----------------------|-------------|----------------------|----------------|----------------------|

**[Clear] [Send]**

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

 **EURL-FV-SM03**  
European Union Reference Laboratory for Residues of Pesticides in Fruits and Vegetables

### Sample Receipt EUPT-FV-SM 03

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: LabXXX

Contact name: XXXXXXXXXXXX -

Sample number: XX

Frozen: Yes ▶

No ▶

Blank number: XX

Date of receipt (DD-MM-YYYY): 03/03/2011

Frozen: Yes ▶

No ▶

Losses: I accept the test material and need no replacement

### Form 0 (Sample Receipt)

Contact Persons:

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

**THIS FORM WILL BE AVAILABLE TO BE FILLED IN ONLY ONE TIME, PLEASE CHECK YOUR DATA BEFORE SAVING IT.**  
If you have entered your data and you want to change them, please contact us.

**Form 1 (Results)**

**EUPT-FV-SM03**

European Union Reference Laboratory for Residues of Pesticides in Fruits and Vegetables

**EUURL**

**1. Results Page EUPT-FV-SM 03**

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.

Lab code: LabXXX  
 Contact Name: xxxxxxxxxxxxxxxx  
[Back to Main page](#) [Save this page](#)

| Pesticide No:   | Pesticide name:   | Methods as pesticide No.: | Desviation (s):     | Desviation RT (specify unit or %): | MS            | Chromatographic Detector: | Analyzer:                              |
|---|-------------------|---------------------------|---------------------|------------------------------------|---------------|---------------------------|--|
| 1   | quinalphos        |                           |                     | 60sec                              | 60sec         | 10 ppm                    | LC <input checked="" type="checkbox"/> |
| <input type="checkbox"/> MS <input checked="" type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>              |                   |                           |                     |                                    |               |                           |  |
| No of compounds in method or library that are pesticides ONLY: <input type="checkbox"/> 517 <input checked="" type="checkbox"/> we do not use screening |                   |                           |                     |                                    |               |                           |  |
| Full Scan:  | Instrument model: | Sample Weight (g):        | Extraction Solvent: | Clean up Step (specify):           | Column Type:  | Injection Volume (µl):    | Software:                              |
| <input checked="" type="checkbox"/> yes   | Agilent 6230      | 10                        | acetonitrile        | none                               | Zorbax eclips | 3                         |  |



**ANNEX 2. List of laboratories that participate in EUPT-FV-SM-03.**

| COUNTRY        | LABORATORY NAME  | CITY               | REPORTED RESULTS |
|----------------|--|--------------------|------------------|
| Austria        | AGES - Competence Center for Residues of Plant Protection Products   | Innsbruck          | Yes              |
| Belgium        | Scientific Institute of Public Health  | Bruxelles          | Yes              |
| Belgium        | LOVAP  | Geel               | Yes              |
| Belgium        | Fytolab  | Zwijnaarde         | Yes              |
| Brazil         | Institute of Technology of Pernambuco - Pesticide Residues Laboratory  | Recife - PE        | No               |
| Czech Republic | Institute of Chemical Technology Prague, Dept. of Food Chemistry and Analysis  | Prague             | Yes              |
| Denmark        | Danish Veterinary and Food Administration, Regin East  | Ringsted           | Yes              |
| Denmark        | National Food Institute, Technical University of Denmark   | Soeborg            | Yes              |
| Egypt          | Central Lab of Residue Analysis of Pesticides and Heavy Metals in Foods  | Giza               | Yes              |
| Estonia        | Laboratory for Residues and Contaminants of Agricultural Research Centre   | Saku               | Yes              |
| Finland        | Finnish Customs Laboratory   | Espoo              | Yes              |
| France         | Laboratoire du SCL   | Montpellier        | Yes              |
| France         | Laboratoire du SCL d'Ile de France Massy   | Massy Cedex France | Yes              |
| France         | GIRPA  | Beaucouze          | Yes              |
| France         | Laboratoire du SCL 35  | Rennes             | Yes              |
| France         | Laboratoire departemental de la sarthe   | Le Mans            | No               |
| France         | Laboratoire du SCL Strasbourg  | Illkirch           | Yes              |
| Germany        | Landesamt für Soziales, Gesundheit und Verbraucherschutz   | Saarbrücken        | No               |
| Germany        | Niedersaechsisches Landesamt fuer Verbraucherschutz und Lebensmittelsicherheit   | Oldenburg          | Yes              |
| Germany        | Bayerisches Landesamt fuer Gesundheit und Lebensmittelsicherheit   | Erlangen           | Yes              |
| Germany        | Federal Office of Consumer Protection and Food Safety (BVL)  | Berlin             | Yes              |
| Germany        | LUA Sachsen, Germany   | Dresden            | Yes              |
| Germany        | Chemisches und Veterinäruntersuchungsamt Münsterland-Emscher-Lippe (CVUA-MEL)  | Muenster           | Yes              |
| Greece         | General Chemical State Laboratory, Pesticide Residues Laboratory   | Athens             | Yes              |
| Greece         | Benaki Phytopathological Institute   | Kifissia           | Yes              |
| Hungary        | Agricultural Office of Somogy County, Plant Prot. & Soil Cons. Directorate, Pesticide Analytical Laboratory                  | Kaposvar           | Yes              |
| Hungary        | Agricultural Office of BAZ County Plant Protection and Soil Conservation Directorate Pesticide Residue Analytical Laboratory | Miskolc            | Yes              |

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| COUNTRY         | LABORATORY NAME  | CITY              | REPORTED RESULTS |
|-----------------|--|-------------------|------------------|
| Ireland         | Pesticide Control Laboratory   | Celbridge         | Yes              |
| Italy           | Agenzia Regionale per la Protezione Ambientale della Toscana - Dipartimento di Arezzo (A.R.P.A.T. - Arezzo)              | Arezzo            | Yes              |
| Italy           | Landesagentur für Umwelt - Labor für Chromatographie   | Bozen             | Yes              |
| Italy           | Istituto Zooprofilattico Sperimentale Lombardia Emilia Romagna - Reparto chimica alimenti - Lab. Contaminanti Ambientali | Brescia           | No               |
| Italy           | ARPACAL - Dipartimento Di Reggio Calabria  | Reggio Calabria   | Yes              |
| Italy           | A.R.P.A.V. - Servizio Laboratori Verona  | Verona            | Yes              |
| Latvia          | Institute of Food Safety, Animal Health and Environment - BIOR   | Riga              | Yes              |
| Norway          | Bioforsk, Plant Health and Plant Protection, Pesticide Chemistry   | Aas               | Yes              |
| Romania         | Laboratory for Pesticides Residues Control in Plants and Vegetable Products  | Bucharest         | Yes              |
| Romania         | Sanitary Veterinary and Food Safety Directorate  | Bucharest         | Yes              |
| Serbia          | Center for Food Analysis   | Belgrade          | Yes              |
| Slovenia        | Zavod za zdravstveno varstvo Maribor - Institute of Public Health Maribor  | Maribor           | No               |
| Spain           | Laboratorio Agroalimentario de Granada   | Santa Fe, Granada | Yes              |
| Spain           | Laboratorio Agroalimentario de la Generalitat Valenciana   | Burjassot         | Yes              |
| Spain           | Laboratorio de Sanidad Vegetal   | Oviedo            | Yes              |
| Spain           | Laboratorio Agroalimentario y de Sanidad Animal  | El Palmar, Murcia | Yes              |
| Sweden          | Eurofins Food & Agro Sweden AB   | Lidköping         | Yes              |
| Sweden          | National Food Administration (Livsmedelsverket)  | Uppsala           | Yes              |
| Switzerland     | Service de la Consommation et des Affaires Vétérinaires (SCAV)   | Genève            | Yes              |
| The Netherlands | VWA - Food and Consumer Product Safety Authority   | Amsterdam         | Yes              |
| The Netherlands | RIKILT   | Wageningen        | Yes              |
| Turkey          | MSM Food Control Laboratory  | Mersin            | Yes              |
| United Kingdom  | The Food and Environment Research Agency   | York              | Yes              |
| United Kingdom  | SASA   | Edinburgh         | No               |
| United Kingdom  | Eurofins Laboratories Ltd.   | Wolverhampton     | Yes              |