EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUIT AND VEGETABLES. SCREENING METHODS 04 (EUPT-FV-SM-04) 2012

Pesticide Residues in Pear Homogenate

Final Report

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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[1]: 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 national monitoring and surveillance programmes.

Regulation (EC) No 882/2004[2] lays down the general tasks, duties and requirements of European Union Reference Laboratories (EURLs) for Food, Feed and Animal Health. Among these tasks is the provision for independently-organised comparative tests. This is the fourth time that the EURL for pesticides in fruit and vegetables at the University of Almería, Spain[3] has organised a proficiency test on qualitative screening methods for pesticides in fruit and vegetable commodities.

The aim of these tests is to evaluate laboratory capability when using wide-scope qualitative and/or semi-quantitative screening methods during routine analysis, for detecting and identifying unexpected pesticides at levels at, or above 0.01 mg/kg – included in and/or in addition to the laboratories' quantitative methods used for frequently-detected pesticides. A second aim is to encourage official laboratories to extend the scope of their methods in a cost-effective way, by using the different MS instruments/software and methods available (whether they be old or new).

Participation in this PT remains on a voluntary basis. Besides this, official laboratories have a significant number of mandatory PTs annually given that the EURL-FV already organises the PT for quantitative multi-residue pesticide analysis (EUPT-FV14), 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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[1] Regulation (EC) No 396/2005, published in the OJ of the EU L70 of 16.03.2005, as last amended by Regulation 839/2008 published in the OJ of the EU L234 of 30.08.2008.

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

[3] 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

In this fourth year, consolidation of inter-laboratory tests for pesticide screening methods among laboratories seems to be assured; and are even requested by those laboratories not performing routine analysis on fruit and vegetables. Responding to this demand, the EURL-FV has decided to continue its 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 observed that many laboratories not only use a full-scan approach to perform screening but that some also employ 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 Document N° SANCO/12495/2011 (which supersedes 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. Amongst 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-SM04 is aimed at all National Reference Laboratories (NRLs) and all Official Laboratories (OfLs) for fruit 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 the pesticide-residue analysis of pears. The pears were grown in Aragón, Spain.

The pesticide treatments were carried out post-harvest using either commercial formulation in micro-spray solutions or using standard solutions. The test material was frozen (using liquid nitrogen), chopped, homogenised and sub-sampled into polyethylene bottles that had previously been coded.

Ten of these bottles containing the test material were chosen randomly and analysed to check for homogeneity.

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

Two bottles, again chosen randomly, were analysed over a period of time to confirm the stability of the pesticides in the test material (firstly, when the test materials were shipped, and then a few days after the deadline for receipt of participants´ results). There was a further analysis during this period reproducing the sample shipment i.e. maintaining the sample at room temperature for a few days to see if there was any degradation of any of the pesticides present in the test material. These results were not included in the statistical analysis of the proficiency test. The aim was solely to check pesticide stability during the shipping process and for the duration of the proficiency test.

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

The pesticides used to spike the pear 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 for treating the test material for this EUPT-FV-SM04 were mainly chosen taking into account the following considerations:

- that they were not included in the EU-Coordinated Multiannual Control Programme for 2011 (Regulation (EC) 915/2011).
- that they had particularly acute toxicity and/or had low ARfD values.

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

Table 2.1. The spiked pesticides used.

Spiked Pesticides		
Bromacil	Fenpropidin	Napropamide
Bromoxinyl	Flufenacet	Prometryn
Cadusafos	Flutolanil	Propaquizafop
Diflubenzuron	Imazapyr	Pyrifenox
Diniconazole	Ioxynil	Terbacil
Ethoxiquin	Isoproturon	Terbumeton
Etrimfos	Metazachlor	Vamidothion

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: The sample is extracted with ethyl acetate along with sodium chloride and magnesium sulphate anhydrous. The mixture is shaken and centrifuged. The final extract is injected into GC-MS/MS.

- LC method: The sample is extracted with ethyl acetate and sodium hydroxide. The mixture is shaken and centrifuged. The extract is evaporated and redissolved in methanol and directly injected into LC-MS/MS.

2.3 Prior analysis of the spiked pear test material

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

To confirm the homogeneity of the test material sent, ten spiked test samples 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 was determined from a table of randomly-generated numbers. The relative standard deviation (RSD) of these 20 analyses needed to be at 15%, or below, to consider the material homogeneous.

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

Table 2.3.1 Homogeneity tests

Test material No.	026 a	026 b	040 a	040 b	046 a	046 b	054 a	054 b	074 a	074 b	078 a	078 b	081 a	081 b	114 a	114 b	132 a	132 b	141 a	141 b	A. Cc (µg/kg)	RSD (%)
Bromacil	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	437.7	8.4
Bromoxinyl	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	291.2	14.1
Cadusafos	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	209.4	10.6
Diflubenzuron	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	379.5	14.6
Diniconazole	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	209.2	8.2
Ethoxiquin	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	101.1	10.6
Etrimfos	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	56.5	1.1
Fenpropidin	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	59.5	12.5
Flufenacet	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	77.8	2.4
Flutolanil	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	79.8	2.3
Imazapyr	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	196.0	9.9
Ioxynil	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	148.1	10.6
Isoproturon	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	160.8	4.8
Metazachlor	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	80.2	2.7
Napropamide	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	51.6	1.5
Prometryn	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	62.7	1.6
Propaquizafop	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	16.5	4.5
Pyrifenox	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	37.5	4.2
Terbacil	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	204.6	8.3
Terbumeton	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	382.3	7.0
Vamidothion	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	95.4	10.9

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 sample 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 10th February 2012.
- Day 2: on 20th February 2012 after reproducing the sample shipment conditions.
- Day 3: soon after the deadline for reporting results on 23rd February 2012.

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

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

Table 2.3.2 Stability tests performed.

Stability tests						
Test material Number	**057 Day 1**	**057 Day 1**	**062 Day 2**	**062 Day 2**	**137 Day 3**	**137 Day 3**
Bromacil	D	D	D	D	D	D
Bromoxinyl	D	D	D	D	D	D
Cadusafos	D	D	D	D	D	D
Diflubenzuron	D	D	D	D	D	D
Diniconazole	D	D	D	D	D	D
Ethoxiquin	D	D	D	D	D	D
Etrimfos	D	D	D	D	D	D
Fenpropidin	D	D	D	D	D	D
Flufenacet	D	D	D	D	D	D
Flutolanil	D	D	D	D	D	D
Imazapyr	D	D	D	D	D	D
Ioxynil	D	D	D	D	D	D
Isoproturon	D	D	D	D	D	D
Metazachlor	D	D	D	D	D	D
Napropamide	D	D	D	D	D	D
Prometryn	D	D	D	D	D	D
Propaquizafop	D	D	D	D	D	D
Pyrifenox	D	D	D	D	D	D
Terbacil	D	D	D	D	D	D
Terbumeton	D	D	D	D	D	D
Vamidothion	D	D	D	D	D	D

D: Detected

2.4 Distribution of test materials and protocol to participants

Approximately 300 g of treated pear homogenate together with another 300 g of 'blank' pear homogenate were shipped to participants on 20th February 2012. 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 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 General and Specific Protocols, were uploaded onto the EUPT-FV-SM04 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 that 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 were 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 was made on a case-by-case basis.

Organiser's 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 detection and full identification.

3.1.2 Not-Reported Pesticides

These were 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-three laboratories agreed to participate in this fourth proficiency test on screening methods. Fifty-one 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 (available only as a pdf document uploaded to the EUPT-SM04 webpage, not in the printed version). The laboratories that agreed to participate are listed in Annex 1.

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*
Bromacil	44	86	7	14
Bromoxinyl	37	73	14	27
Cadusafos	46	90	5	10
Diflubenzuron	42	82	9	18
Diniconazole	47	92	4	8
Ethoxyquin	37	73	14	27
Etrimfos	48	94	3	6
Fenpropidin	44	86	7	14
Flufenacet	39	76	12	24
Flutolanil	43	84	8	16
Imazapyr	18	35	33	65
Ioxynil	28	55	23	45
Isoproturon	46	90	5	10
Metazachlor	45	88	6	12
Napropamide	43	84	8	16
Prometryn	45	88	6	12
Propaquizafop	24	47	27	53
Pyrifenox	40	78	11	22
Terbacil	38	75	13	25
Terbumeton	38	75	13	25
Vamidothion	36	71	15	29
Spinosad**	43	84	8	16

* The % of laboratories is calculated based on the total number of laboratories submitting results (51).
** Reported but not spiked

4.1.1 Other Reported Pesticides

Many laboratories reported additional pesticides to those used to spike the test material. These reported pesticides are presented in Table 4.1.1.

Table 4.1.1. 'Other reported pesticides' in the test material given by laboratories.

LABORATORY CODE	OTHER REPORTED PESTICIDES
Lab003*	Secbumeton
Lab008	Atrazine-2-hydroxy, Atrazine-desethyl-2-hydroxy
Lab009*	Ametryn, Carbaryl, Endosulfan alpha, Imibenconazol, Propiconazole, Terbutylazin
Lab010*	3-Chloroaniline, Prometon, Terbutryn, Secbumeton
Lab012*	3-Chloroaniline
Lab016	Formothion, Omethoate, Secbumeton
Lab017*	Phenothrin, Photodieldrin, Tebufenocide, Thiodemeton
Lab019*	3-Chloroaniline
Lab020	3-Chloroaniline
Lab022	Terbutryn
Lab025	Cyproconazole, Diclobutrazol, Propiconazole
Lab028*	Atrazine-desethyl-2-hydroxy
Lab029	Cymoxanil, Terbutryn
Lab030*	Ametryn, Cyazofomid, Diclobutrazol, Molinate, Terbutryn
Lab040*	3-Chloroaniline, Atraton, Demeton O, Isobornyl Thiocyanoacetate
Lab044	Difenoconazole, Folpet, Procloraz
Lab045	Chlorbufam, o-Phenylphenol, Trichlorfon
Lab048	Atraton
Lab050	Bifenox

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

Those pesticides reported by more than two laboratories were analysed by the Organiser, but none of these pesticides was detected after repeated analyses.

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-one pesticides were used to spike the pear test material at different levels, in the range between 50 and 1000 µg/kg. Spinosad was present in the sample at around 5µg/kg. This EUPT was focused only on detection capabilities; therefore no quantitative data were requested.

4.3 Assessment of laboratory performance.

No z-score values were carried out for laboratories to assess their performance as no numerical results were reported by the participants. However, classification was considered important, based on the number of detected results each laboratory reported and also on which methods they used.

Table 4.3.1 classifies the laboratories according to the number of spiked pesticides reported.

Table 4.3.1 Classification of laboratories according to the number of spiked pesticides reported.

Laboratory Code	Reported	Other Reported Pesticides	
		Not Confirmed by the Organiser	Confirmed by the Organiser
Lab024	22	0	
Lab034*	22	0	
Lab028*	22	1	
Lab048	22	1	
Lab008	22	2	0
Lab040*	22	4	0
Lab001	21	0	
Lab014	21	0	
Lab023	21	0	
Lab049	21	0	0
Lab010*	21	4	0
Lab004*	20	0	
Lab015	20	0	
Lab038	20	0	
Lab047*	20	0	
Lab003*	20	1	
Lab025	20	3	0
Lab006	19	0	
Lab018	19	0	
Lab021	19	0	
Lab041	19	0	
Lab046	19	0	
Lab030*	19	5	
Lab002*	18	0	
Lab011	18	0	
Lab031	18	0	0
Lab039	18	0	0
Lab043*	18	0	0
Lab020	18	1	0
Lab036	17	0	
Lab037	17	0	
Lab042	17	0	
Lab019*	17	1	0
Lab016	17	3	0
Lab045	17	3	0
Lab026	16	0	
Lab050	16	1	

Laboratory Code	Reported	Other Reported Pesticides	
		Not Confirmed by the Organiser	Confirmed by the Organiser
Lab029	16	2	0
Lab017*	16	4	0
Lab007*	15	0	
Lab053	15	0	
Lab009*	15	6	0
Lab027*	14	0	
Lab012*	14	1	0
Lab033	13	0	0
Lab032	11	0	
Lab051	11	0	0
Lab022	10	1	
Lab035*	6	0	
Lab013	1	0	
Lab044	1	3	0
Lab005	No Results Reported		
Lab052	No Results Reported		

* 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 (available only as a pdf document uploaded to the EUPT-SM04 webpage, not in the printed version). In Table 4.3.2, there is a summary of the chromatographic techniques used for each pesticide, and a graphical representation is shown in Appendix 2.

Table 4.3.2 Chromatographic techniques used to determine each pesticide in the test material

Pesticide	Total no. of Reports	GC	Full-scan GC	LC	Full-scan LC
Bromacil	46	18	14	28	10
Bromoxinyl	39	11	11	28	6
Cadusafos	49	26	15	23	5
Diflubenzuron	44	3	2	41	10
Diniconazole	50	20	15	30	6
Ethoxyquin	39	24	17	15	4
Etrimfos	50	34	21	16	5
Fenpropidin	47	18	13	29	5
Flufenacet	41	14	12	27	7
Flutolanil	46	25	16	21	6
Imazapyr	18	0	0	18	4
Ioxynil	29	4	4	25	5
Isoproturon	46	2	2	44	10
Metazachlor	47	27	18	20	6

Pesticide	Total no. of Reports	GC	Full-scan GC	LC	Full-scan LC
Napropamide	45	18	12	27	8
Prometryn	48	26	18	22	6
Propaquizafop	25	0	0	25	5
Pyrifenox	44	20	14	24	6
Spinosad*	53	0	0	53	13
Terbacil	39	30	20	9	6
Terbumeton	41	23	18	18	7
Vamidothion	44	5	5	39	7

*Spinosad was present in the sample but not spiked.
Note: The number of reports for each of the pesticides could be different to the reports shown in Table 4.1 because a particular laboratory might analize each of the pesticides with more than one technique.

In Appendix 2, graphical representations of the techniques used can be seen.

Table 4.3.3 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.

Table 4.3.3. Number and Percentage of Present Pesticides Reported by Laboratory

Laboratory Code	Number of Present Pesticides Reported	% of Present Pesticides Reported
Lab001	21	95
Lab002*	18	82
Lab003*	20	91
Lab004*	20	91
Lab006	19	86
Lab007*	15	68
Lab008	22	100
Lab009*	15	68
Lab010*	21	95
Lab011	18	82
Lab012*	14	64
Lab013	1	5
Lab014	21	95
Lab015	20	91
Lab016	17	77
Lab017*	16	73
Lab018	19	86
Lab019*	17	77
Lab020	18	82
Lab021	19	86
Lab022	10	45
Lab023	21	95

Laboratory Code	Number of Present Pesticides Reported	% of Present Pesticides Reported
Lab024	22	100
Lab025	20	91
Lab026	16	73
Lab027*	14	64
Lab028*	22	100
Lab029	16	73
Lab030*	19	86
Lab031	18	82
Lab032	11	50
Lab033	13	59
Lab034*	22	100
Lab035*	6	27
Lab036	17	77
Lab037	17	77
Lab038	20	91
Lab039	18	82
Lab040*	22	100
Lab041	19	86
Lab042	17	77
Lab043*	18	82
Lab044	1	5
Lab045	17	77
Lab046	19	86
Lab047*	20	91
Lab048	22	100
Lab049	21	95
Lab050	16	73
Lab051	11	50
Lab053	15	68

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

5. CONCLUSIONS

Fifty-three laboratories applied to participate in this test and fifty-one laboratories submitted results. Seventeen of the laboratories which applied were National Reference Laboratories for Fruit 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 three non-EU/EFTA countries (Egypt, Serbia 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. Of 930 detections, 395 were made using full-scan, meaning 43% of detections; 248 by LC techniques and 147 by GC techniques.

Four of the 53 laboratories were able to detect all 22 pesticides in the spiked pear test material (21 spiked plus spinosad, which was present in the blank). Only 4 laboratories failed to detect less than 50% of the pesticides present.
Seventy-six percent of the laboratories (39 laboratories) that reported results were able to find more than 70% of the pesticides used to spike the sample whereas last year (EUPT-FV-SM03) only 71% managed to do so.

Nineteen participants reported thirty-two different pesticides which were not used for spiking the pears. 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 fourth 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 the 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 fourth 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, potato matrix test material will be used. If laboratories have a particular interest in specific matrices, they should inform the EURL-FV and their suggestions will be evaluated. The date of the test material delivery will be January 2013; and 72 hours will be allowed for results submission (given that this should be enough time to carry out screening methods). There will be no target list, as was the case in this test.

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8. ACKNOWLEDGEMENTS

The Organiser is grateful to the European Commission for funding this 4th European Proficiency Test for Screening Methods in Fruit and Vegetables.

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

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

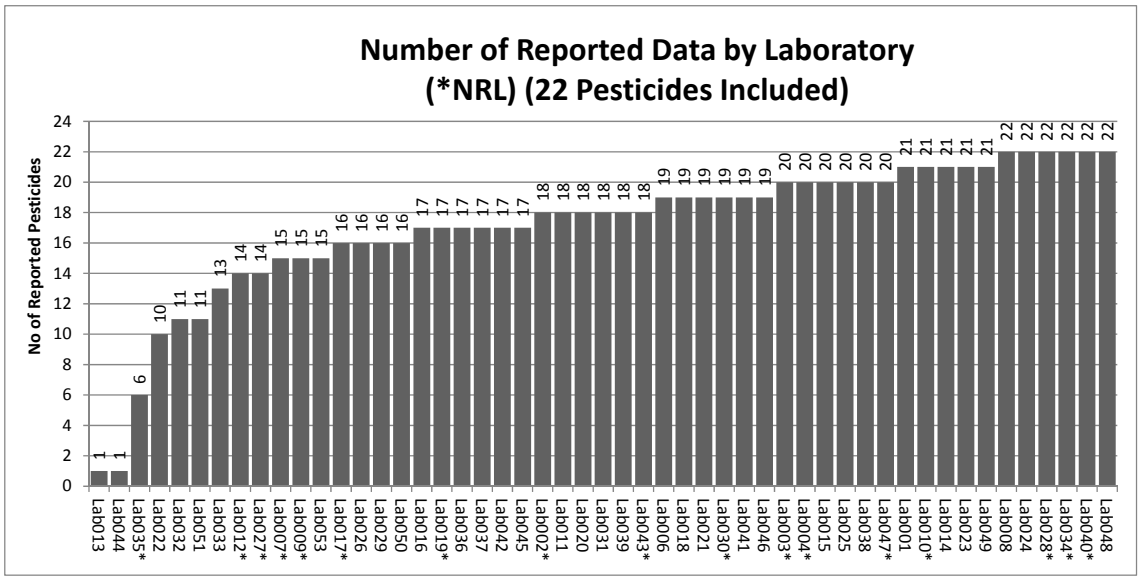
APPENDIX 1. Results

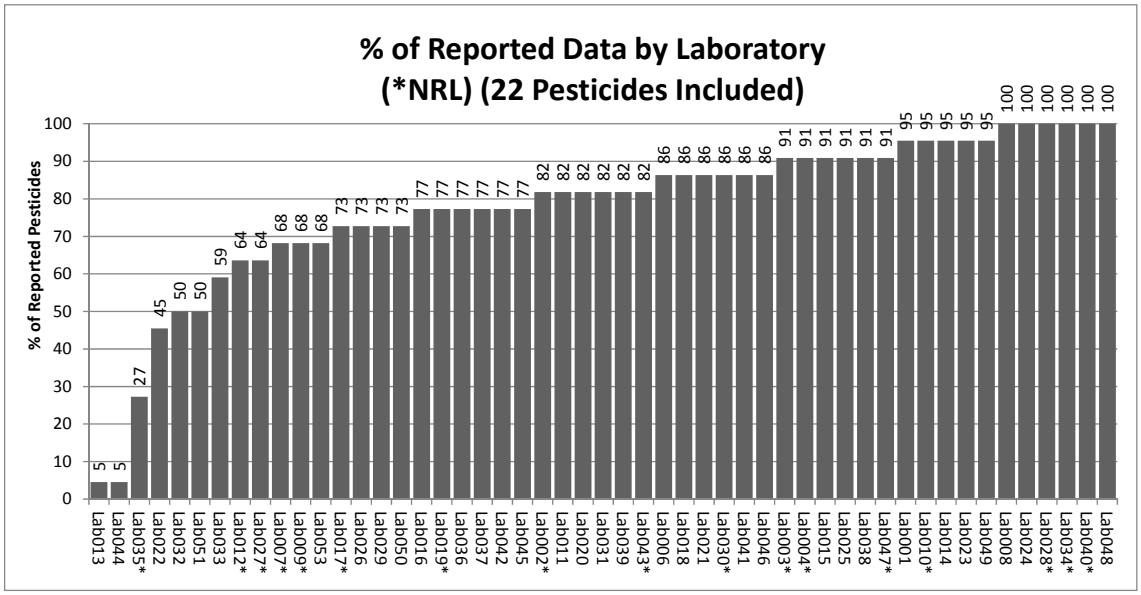
Laboratory Code Total No of Reporting Laboratories = 51	Evaluated Pesticides (21 added + 1 present in the blank)																					Reported Pesticides by Laboratory	% of Reported Pesticides by Laboratory
	Bromacil	Bromoxinyl	Cadusafos	Diflubenzuron	Diniconazole	Ethoxyquin	Etrimfos	Fenpropidin	Flufenacet	Flutolanil	Imazapyr	Ioxynil	Isoproturon	Metazachlor	Napropamide	Prometryn	Propaquizafop	Pyrifenox	Terbacil	Terbumeton	Vamidothion	Spinosad		
001	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	21	95
002*	R	R	R		R	R	R	R	R	R			R	R	R	R		R	R	R	R	R	18	82
003*	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R		R	R	20	91
004*	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	R		R	R	R	20	91
005	No Results Reported																						0	0
006	R	R	R	R	R	R	R	R	R	R			R	R	R	R		R	R	R	R	R	19	86
007*	R		R	R			R	R		R	R		R	R	R	R	R		R	R		R	15	68
008	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	22	100
009*		R	R	R	R	R	R	R		R		R	R	R	R	R		R	R				15	68
010*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	21	95
011	R	R	R	R	R	R	R	R	R	R			R	R	R	R		R	R	R		R	18	82
012*			R	R	R	R	R	R	R	R		R	R	R	R						R	R	14	64
013																					R		1	5
014	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	21	95
015	R	R	R	R	R	R	R	R	R	R			R	R	R	R	R	R	R	R	R	R	20	91
016	R		R	R	R	R	R	R	R	R			R	R	R	R		R		R	R	R	17	77
017*	R	R	R	R	R	R	R	R				R	R	R		R			R	R	R	R	16	73
018	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R		R	R	R		R	19	86
019*	R	R	R	R	R		R	R	R	R		R	R	R	R	R		R			R	R	17	77
020	R		R	R	R	R	R	R	R	R			R	R	R	R		R	R	R	R	R	18	82
021	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R				R	19	86
022	R		R	R	R		R				R		R	R						R	R		10	45
023	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	21	95
024	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	22	100
025	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R		R	R	R	R	R	20	91
026	R	R	R	R	R	R	R	R		R			R	R	R			R	R	R		R	16	73
027*			R	R	R		R	R	R				R	R	R	R		R	R	R		R	14	64
028*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	22	100
029	R		R		R		R	R	R	R	R		R	R	R	R		R	R	R		R	16	73
030*	R	R		R	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R		19	86
031	R	R	R	R	R	R	R	R		R		R	R	R	R	R	R	R			R	R	18	82
032		R	R		R		R	R	R	R			R				R				R	R	11	50
033	R		R		R		R	R	R	R				R	R	R		R	R	R			13	59
034*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	22	100
035*	R					R								R		R			R	R			6	27
036	R		R		R		R	R	R	R			R	R	R	R	R	R	R	R	R	R	17	77
037	R	R	R	R	R		R	R	R	R		R	R	R	R	R	R				R	R	17	77
038	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R		R	R	R	20	91
039		R	R	R	R	R	R	R		R			R	R	R	R	R	R	R	R	R	R	18	82
040*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	22	100
041	R	R	R	R	R	R	R			R	R	R	R	R	R	R		R	R	R	R	R	19	86
042	R	R	R	R	R		R	R	R	R		R	R	R	R	R		R			R	R	17	77
043*	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R		R	R			R	18	82
044													R										1	5
045	R	R	R		R	R	R	R	R	R		R	R	R	R	R		R	R	R			17	77
046	R		R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	R		R	R	19	86
047*	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R		R	R	R	R	R	20	91
048	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	22	100
049	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	21	95
050	R	R	R	R	R		R	R	R			R	R		R	R			R	R	R	R	16	73
051	R	R		R	R	R	R			R						R			R	R		R	11	50
052	No Results Reported																						0	0
053	R		R	R	R	R	R		R	R				R		R	R	R	R	R		R	15	68
Reported Pesticides	44	37	46	42	47	37	48	44	39	43	18	28	46	45	43	45	24	40	38	38	36	43		
% of Reported Pesticides	86	73	90	82	92	73	94	86	76	84	35	55	90	88	84	88	47	78	75	75	71	84		

R: Reported pesticide

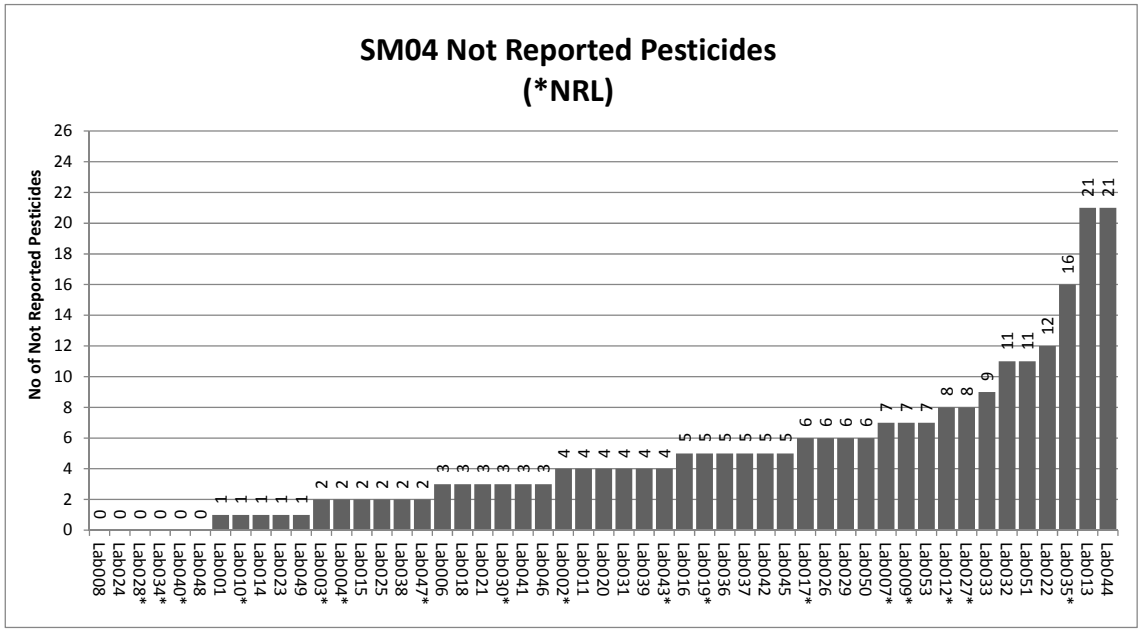
* NRLs from EU

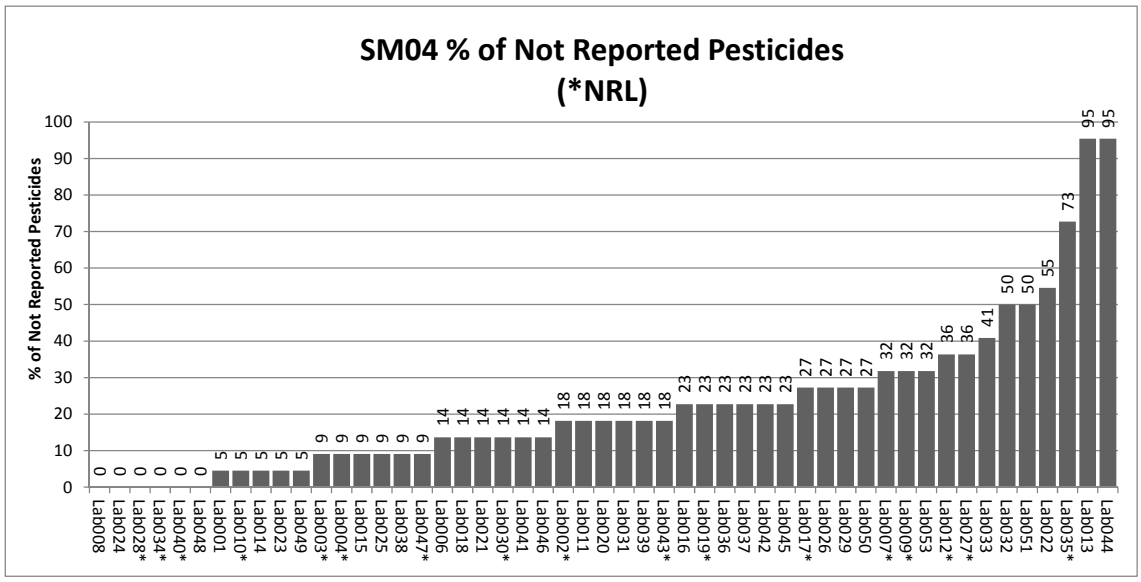
APPENDIX 2. Graphical Representations



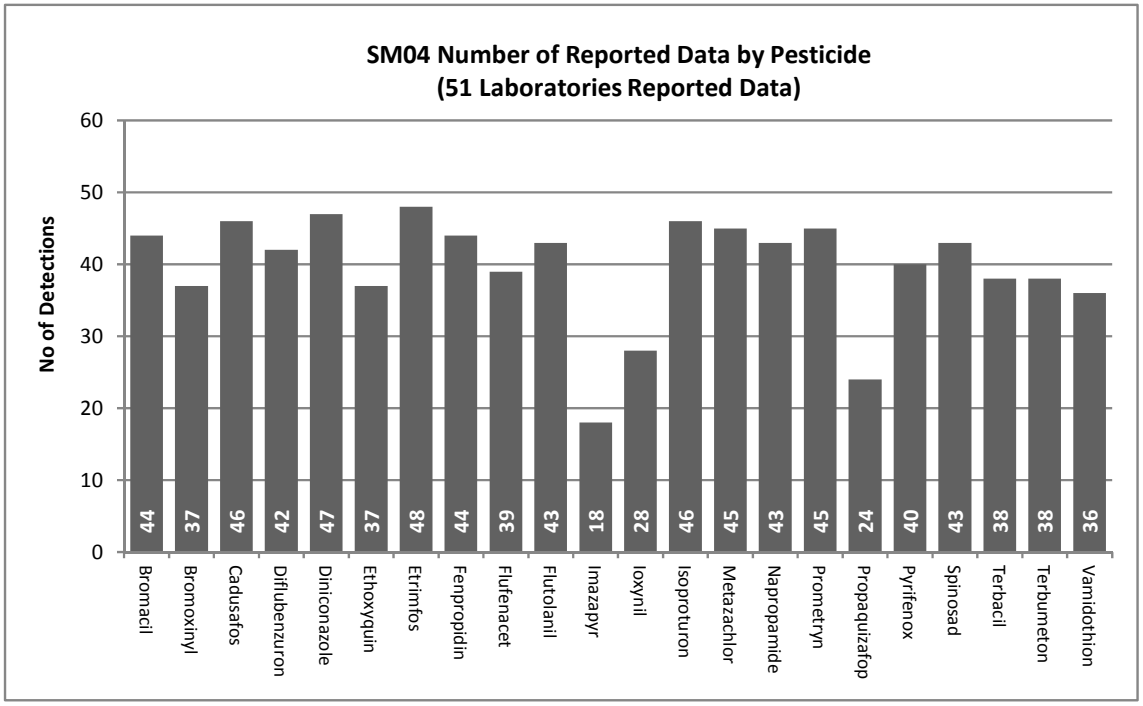


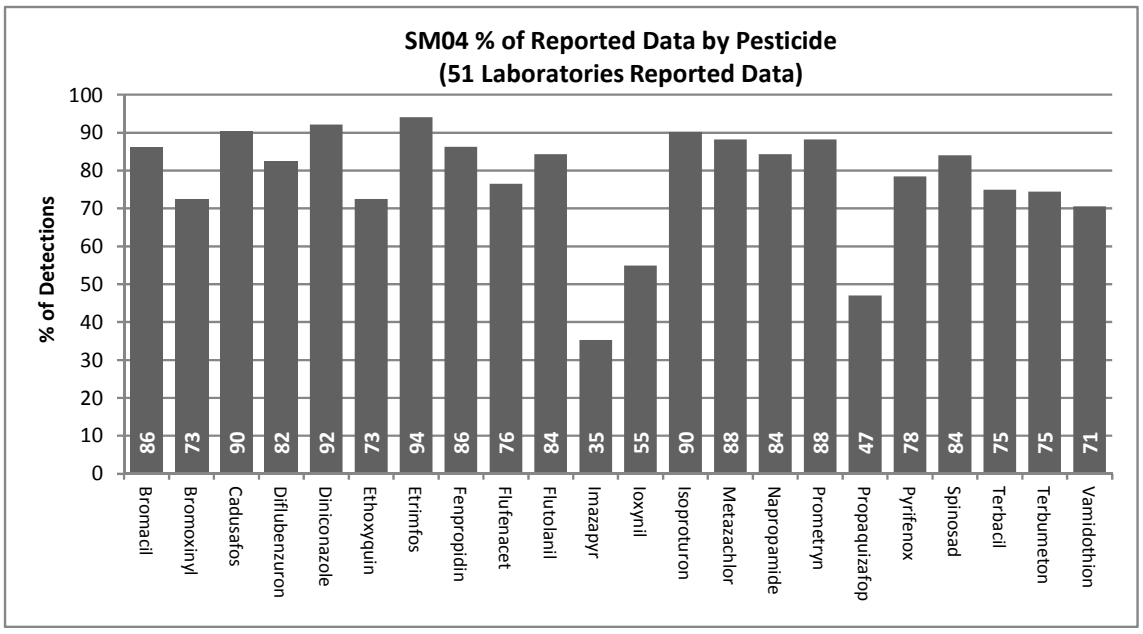
APPENDIX 2. Graphical Representations





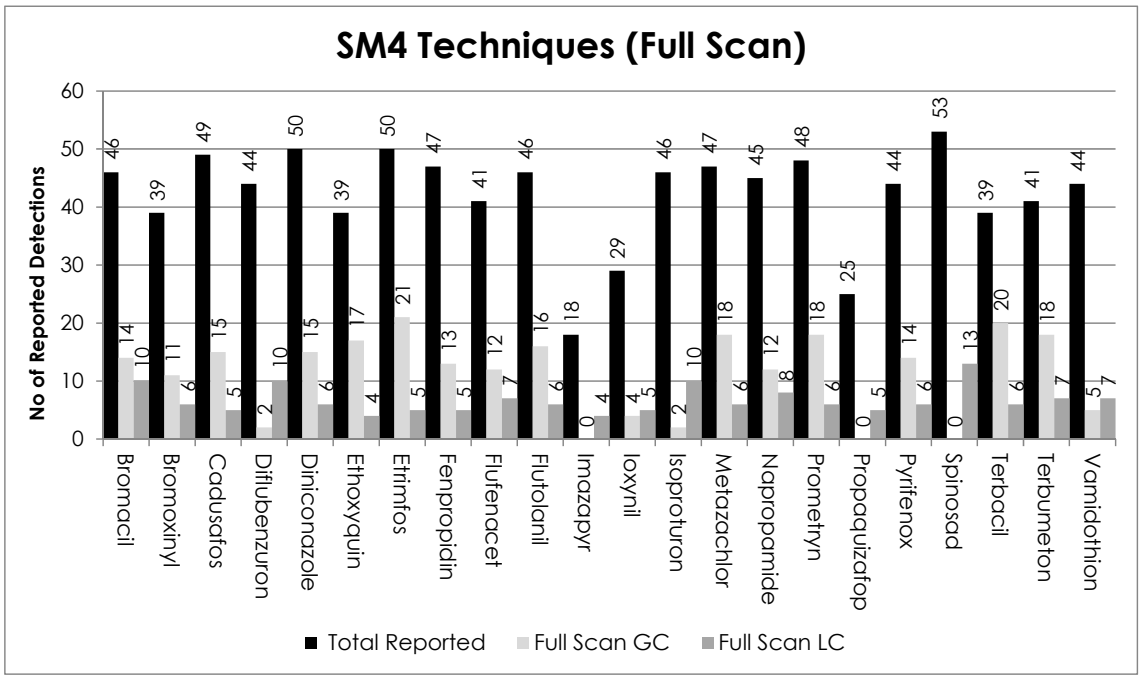
APPENDIX 2. Graphical Representations





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.

BROMACIL																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6490	5	ACN		RP-C18	10	Both		Daily
002	Confirmed with second method	11.2			GC	MSD	Q	EI	+	YES	YES	not validated	YES	AT 5975	10	ACN	SPE	HP5-MS	10	Auto	aprox 750	Always
003	Confirmed with second method	0.05	5	2 transitions	LC		Q-TOF	ESI	+	NO	NO		YES	Bruker Maxis	10	ACN	PSA	RSLC C18	1	Both	aprox 400	
004	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA		LC	MSD	QQQ	ESI	+	YES	NO	NA	NO	Agilent 6460, 1290HPLC	10	EtOAc	None	Waters, HSS T3, 150 x 2.1mm	2	Both	500	Each Batch
004	Confirmed with second method	-1.8	0.6		LC	MSD	TOF	ESI	+	NO	NO	NA	YES	Bruker Maxis	10	EtOAc	None	Acclaim RSLC 120 C18, 2,1*100 mm	1	Both	1000	Each Batch
006	Fully identified (SANCO/12495/2011 p.74-80)	<2	0	2 transitions	LC	MSD	QQQ	ESI	-	YES	YES	>0.010 mg/kg	NO	Waters quattro premier	15	ACN	None	BEH C18	3	Both		
007	Fully identified (SANCO/12495/2011 p.74-80)	1	None		GC	MSD	Q	EI	+	YES	YES	0.01	NO	Agilent 6890/5973	10	ACN	DSPE	HP-5MS	2	Manual	183	Each Batch
008	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions +accurate mass	LC	MSD	Q-TRAP	ESI	+	YES	NO	0.01	NO	QTrap 5500	10	ACN	None	reversed phase	2.5	Auto	300	Each Batch
010	Fully identified (SANCO/12495/2011 p.74-80)	6.3		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01	YES	HP5975C	10	ACN	DSPE	HP5MS	10	Both	aprox 1000	Always
011	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	NA	GC	MSD	Q	EI	+	NO	NO	NA	YES	5790 msd	10	ACN	PSA	HP-5MS	20	Auto	600 aprox	0-1
014	Fully identified (SANCO/12495/2011 p.74-80)	±2.5%		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010 mg/kg	YES	Waters-TQD	15	ACN	PSA	UPLC-BEH C18	10	Auto	331	Each Batch
015	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	2 transitions	GC		QQQ	EI	+	YES	YES	0.01	NO	7000B	10	ACN	PSA	DB-5MS UI	2	Both	440	
016	Confirmed with second method					MSD	QQQ	CI	+	YES	YES	0.010	NO	LC-MS/MS (Waters)	15	ACN	QuEChERS	C18	10	Both	370	Always
017	Tentative detected				GC	MSD	TOF	EI		NO	NO		YES	LECO PEGASUS	15	Acetone/PE/DCM	NO	DB-5	1	Both		

APPENDIX 3. Methods used by participants for detecting pesticides.

BROMACIL																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
018	Tentative detected	5	5	accurate mass	LC	Orbitrap	Orbitrap	ESI	+	YES	YES	0.01 - 0.05 mg/kg	YES	Thermo Exactive	10	ACN	PSA	C18	5	Both	120	Always
019	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	10	NO	Agilent 6490	15	ACN	PSA	C18	5	Auto		
020	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	AbSciex	10	ACN	DSPE	C18	5	Auto	200	Always
021	Fully identified (SANCO/12495/2011 p.74-80)	15		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	HPLC Agilent 1100, MS API 3000	10	ACN	DSPE (PSA, MgSO4)	C18 3µ 50x2mm	10	Both	550	Always
022	Tentative detected	0.5	0.05		LC	MSD	TOF	ESI	+	YES	YES		YES	LCT PREMIER XE	15	MINI LUKE	NO	C-18	7	Auto	343	Every Day
023	Confirmed with second method				LC		IT	ESI	-	YES	YES	0.01	NO		10	ACN				Both		Each Batch
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		3 masses	GC	MSD	IT	EI	+	YES	YES	0.01	YES	Varian Saturn 2000	10	ACN	PSA	VactorFour 5 MS	5	Auto	500	Daily
025	Fully identified (SANCO/12495/2011 p.74-80)	-0.12	1.2	isotope, 2 ions < 5ppm	LC	MSD	Orbitrap	ESI	+	NO	NO	0.01-0.1	YES	Exactive	10	ACN	None	C18 Atlantis T3	5	Both	570	Each Batch
	Fully identified (SANCO/12495/2011 p.74-80)	< 20	860	spectrum	GC	MSD	TOF	EI	+	YES	YES	0.01-0.1	YES	Leco Pegasus IV	10	ACN	DSPE (PSA)	RTX-CL pest	10	Auto	557	Each Batch
026	Fully identified (SANCO/12495/2011 p.74-80)	< 2.5 %	2.5	transition 1 and 2	LC	MSD	Q-TRAP	ESI	+	YES	YES	0.01	YES	3200 Qtrap	10	ACN	DSPE	C18	50	Auto	521	Every Week
028	Confirmed with second method			261.0>205.0, 261.0>187.0	LC	MSD	QQQ	ESI	+	NO	NO	10-100 ppb	NO	LC: Agilent 1200, MS: API 4000 QTRAP	10	ACN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Auto	591	
029	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	NO	NO				10	ACN	NO	Xterra C18	20	Both		
030	Tentative detected				LC		QQQ	ESI	+	YES	YES	0.02	NO	Varian 320	15	ACN		C18	10	Auto	150	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

BROMACIL																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
031	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.025-0.4 µg/ml	NO	UPLC-MSMS (Waters Aquity UPLC system, API 5000 Trippel quadropol, Sciex	10	EtOAc	Filter	HSS T3	2	Both	141	Each Batch
033	Fully identified (SANCO/12495/2011 p.74-80)	-5.5	20%	min 3 ions	GC	MSD	Q	EI	+	YES	YES	0.01	YES	Agilent GC-MSD 7890A/5975c	10	ACN	PSA, C18	HP-5MS	1	Auto	927	
034	Confirmed with second method			full scan	GC	MSD	Q	EI	+	NO	NO		YES	Agilent 5973 MSD	10	ACN	DSPE (PSA/MgSO4)	HP-5MS	10	Both	950	
035	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	Q	EI		NO	NO		YES	Agilent 6890N	10	ACN	DSPE	DB-5	2	Auto		
036	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES					10	ACN			5	Auto	350	
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.010	NO	4000 QTRAP	10	ACN	DSPE	C18 hydro Phenomenex	40	Manual		Always
038	Fully identified (SANCO/12495/2011 p.74-80)	None	None	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES		NO	API 4000	10	ACN	DSPE	Waters-C18 2,1x50mmx1,7,µm	5	Auto	600	Every Week
040	Confirmed with second method	3	1		LC		TOF	ESI	+	NO	NO		YES	Agilent 6230	10	ACN	None	Eclipse C18	3	Auto	aprox 500	
041	Fully identified (SANCO/12495/2011 p.74-80)	0.5%	20%	2 transitions	GC	MSD	QQQ	EI		YES	YES	0.005 mg/kg	NO	VARIAN CP-3800 MS-320	10	ACN	PSA	VF-5MS	1	Auto	210	
042	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES		NO	API 4000	10	ACN	QuEChERS	C18 Aqua	10	Both		
043	Tentative detected	0	0	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010	YES	Api 3200QT	10	ACN	DSPE	Phenomenex fusion	10	Auto	200	Daily
045	Tentative detected	1	1 unit		GC	MSD	Q	EI		YES	NO	0.01 ppm	YES	Agilent 5973	10	ACN	PSA	HP 5 MS UI	2	Auto	923	Each Batch
046	Fully identified (SANCO/12495/2011 p.74-80)	0			LC	MSD	QQQ	ESI	+	NO			NO	Waters Xevo	10	Acetone/PE/DCM	Na2SO4	C18	2	Both	286	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

BROMACIL																				
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
047	Fully identified (SANCO/12495/2011 p.74-80)	2.8		Spectrum match (reversed)	GC	MSD	IT	EI	+	YES	YES	20-10.000 µg/kg	YES	Varian Satum 2000	15	Acetone/PE/DCM	NO	VF-5-ms	5	Both	550	Quarterly
048	Confirmed with second method	8			GC	MSD	TOF	EI		YES	YES	0.005	YES	Pegasus IV	10	ACN	DSPE(PSA)	HP-5MS	5	Both	650	Weekly
049	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC		QQQ	EI	+	YES	YES		NO	Waters Quattro micro GC	10	ACN		DB-5MS	3	Both	150	Daily
050	Fully identified (SANCO/12495/2011 p.74-80)	-0.018		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01-0.05	YES	Agilent 1200 LC+6460QQQ	10	ACN	DSPE	YMC Triart C18 2*100 mm 3µm	5	Both	354	Daily
051	Tentative detected		0.5	qualifiers	GC	MSD	IT	EI		NO	NO		YES	varian GCMS 4000	10	ACN	PSA	varian factor four	5	Both		
053	Tentative detected	8		3 SIM Ions	GC	MSD	Q	EI	+	NO	NO	>0.05 mg/kg	YES	Thermo DSQ	10	DCM	Diatomaceous earth		1	Both	400	Every 6 Months

APPENDIX 3. Methods used by participants for detecting pesticides.

BROMOXINYL																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	-	YES	YES	0.01	NO	Agilent 6490	5	ACN		RP-C18	10	Both		Daily
002	Confirmed with second method	9.8			GC	MSD	Q	EI	+	YES	YES	not validated	YES	AT 5975	10	ACN	SPE	HP5-MS	10	Auto	aprox 750	Always
003	Confirmed with second method	0.05	5	2 transitions	LC		Q-TOF	ESI	+	NO	NO		YES	Bruker Maxis	10	ACN	PSA	RSLC C18	1	Both	aprox 400	
004	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA		LC	MSD	QQQ	ESI	-	YES	NO	NA	NO	Agilent 6460, 1290HPLC	10	EtOAc	None	Waters, HSS T3, 150 x 2.1mm	2	Both	500	Each Batch
006	Fully identified (SANCO/12495/2011 p.74-80)	<2	0	2 transitions	LC	MSD	QQQ	ESI	-	YES	YES	>0.010 mg/kg	NO	Waters quattro premier	15	ACN	None	BEH C18	10	Both		
008	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions +accurate mass	LC	MSD	Q-TRAP	ESI	-	YES	NO	0.01	NO	QTrap 5500	10	ACN	None	reversed phase	3	Auto	300	Each Batch
009	Tentative detected			2 transitions	LC		QQQ	ESI	-	NO	NO		NO	API4000	10	ACN	DSPE	Synergie Fusion RP80A	8	Manual	40	Scheduled MRM Method also used for quantification
010	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	NO	YES	0.01	NO	ABI4000	10	ACN	NO	C18	55	Both	aprox 580	Always
	Fully identified (SANCO/12495/2011 p.74-80)	0.2		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01	YES	HP5975C	10	ACN	DSPE	HP5MS	10	Both	aprox 1000	Always
011	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	NA	GC	MSD	Q	EI	+	NO	NO	NA	YES	5790 msd	10	ACN	PSA	HP-5MS	20	Auto	600 aprox	0-1
014	Fully identified (SANCO/12495/2011 p.74-80)	±2.5%		2 transitions	LC	MSD	QQQ	ESI	-	YES	YES	0.010 mg/kg	YES	Waters-TQD	15	ACN	PSA	UPLC-BEH C18	10	Auto	331	Each Batch
015	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	2 transitions	LC		QQQ	ESI	+	YES	YES	0.01	NO	6460	10	ACN	PSA	C18-XB	2	Both	440	

APPENDIX 3. Methods used by participants for detecting pesticides.

BROMOXINYL																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
017	Tentative detected				GC	MSD	TOF	EI		NO	NO		YES	LECO PEGASUS	15	Acetone/PE/DCM	NO	DB-5	1	Both		
018	Tentative detected	RT locking		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01-0.05 mg/kg	YES	Agilent MSD 5975B	10	ACN	PSA	HP5	10	Both	> 600	Always
019	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	LC	MSD	QQQ	ESI	-	YES	YES	10	NO	Agilent 6490	15	ACN	PSA	C18	5	Auto		
021	Fully identified (SANCO/12495/2011 p.74-80)	15		2 transitions	LC	MSD	QQQ	ESI	-	YES	YES	0.01	NO	HPLC Agilent 1100, MS API 3000	10	ACN	DSPE (PSA, MgSO4)	C18 3µ 50x2mm	10	Both	550	Always
023	Confirmed with second method				LC		IT	ESI	-	YES	YES	0.01	NO		10	ACN				Both		Each Batch
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		2 x MRM	LC	MSD	QQQ	ESI	-	YES	YES	0.01	NO	AB Sciex API 4000	10	ACN	PSA	Phenomenex C18	10	Both	500	Daily
025	Confirmed with second method	0.04	1.0	isotope	LC	MSD	Orbitrap	ESI	-	NO	NO	0.01-0.1	YES	Exactive	10	ACN	None	C18 Atlantis T3	5	Both	570	Each Batch
	Confirmed with second method	NA	776	spectrum	GC	MSD	TOF	EI	+	YES	YES	0.01-0.1	YES	Leco Pegasus IV	10	ACN	DSPE (PSA)	RTX-CL pest	10	Auto	557	Each Batch
026	Fully identified (SANCO/12495/2011 p.74-80)	<0.5%	2.5		GC	MSD	ion trap	EI		YES	YES	0.01	YES	Trap 4000 - Varian	10	ACN	DSPE	5%	1 µL	Auto	450	Every Week
028	Fully identified (SANCO/12495/2011 p.74-80)			273.7>78.9, 275.4>81.0	LC	MSD	QQQ	ESI	-	YES	YES	LOQ:2 ppb	NO	LC: Agilent 1200, MS: API 4000 QTRAP	10	ACN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Auto		
030	Tentative detected				LC		QQQ	ESI	+	YES	YES	0.02	NO	Varian 320	15	ACN		C18	10	Auto	150	Each Batch
031	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	-	YES	YES	0.005-0.04 µg/ml	NO	UPLC-MSMS (Waters Aquity UPLC system, API 5000 Tripelquadropol, Sciex	5	EtOAc	Filter	HSS T3	2	Both	250	Each Batch
032	Tentative detected	0		2 transitions	LC	MSD	QQQ	ESI	-	YES	YES	0.01	NO	Waters Acquity TQD	10	Methanol	Filter	C18	6	Auto	150	Always
034	Confirmed with second method			full scan	GC	MSD	Q	EI	+	NO	NO		YES	Agilent 5973 MSD	10	ACN	DSPE (PSA/MgSO4)	HP-5MS	10	Both	950	

APPENDIX 3. Methods used by participants for detecting pesticides.

BROMOXINYL																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	-	YES	YES	0.010	NO	4000 QTRAP	10	ACN	DSPE	C18 hydro Phenomenex	40	Manual		Always
038	Tentative detected	None	None	2 transitions	LC	MSD	QQQ	ESI	-	NO	NO		NO	API 4000	10	ACN	DSPE	Waters-C18 2,1x50mmx1,7,µm	5	Auto	600	Every Week
039	Tentative detected				GC	MSD	Q	EI	+	NO	NO		YES	HP5973 MSD	10	ACN	DSPE	HP-5MSI	20	Both	624	No standard used
040	Confirmed with second method	10	3		LC		TOF	ESI	-	NO	NO		YES	Agilent 6230	10	ACN	None	Eclipse C18	3	Auto	aprox 500	
041	Fully identified (SANCO/12495/2011 p.74-80)	2.5%		2 transitions	LC	MSD	QQQ	ESI	-	YES	YES	0.005 mg/kg	NO	AGILENT G-6410A	10	ACN	PSA	ZORBAX-ECLIPSE XDB-C8	80	Auto	143	
042	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	-	YES	YES		NO	API 4000	10	ACN	QuEChERS	C18 Aqua	10	Both		
043	Tentative detected	0	0	2 transitions	LC	MSD	QQQ	ESI	-	YES	YES	0.010	YES	Api 3200QT	10	ACN	DSPE	Phenomenex fusion	10	Auto	200	Daily
045	Tentative detected	1	1 unit		GC	MSD	Q	EI		YES	NO	0.01 ppm	YES	Agilent 5973	10	ACN	PSA	HP 5 MS UI	2	Auto	923	Each Batch
047	Fully identified (SANCO/12495/2011 p.74-80)	2.4		2 transitions + ion ratio	LC	MSD	QQQ	ESI	-	NO	NO	5-1000 µg/kg	NO	Waters Quattro Premier XE	15	Acetone/PE/DCM	NO	Alltima C18 (150x3 mm i.d.)	5	Both	27	Each Batch
048	Confirmed with second method	0.9			LC	MSD	QQQ	ESI	-	YES	YES	0.005	NO	Agilent 6410	10	ACN	DSPE(PSA)	Luna C18	5	Both	350	Daily
049	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC		QQQ	ESI	+	YES	YES		NO	Waters XEVO TQ MS	10	ACN		BEH C18 1,7µm	3	Both	60	Daily
050	Fully identified (SANCO/12495/2011 p.74-80)	-0.016		2 transitions	LC	MSD	QQQ	ESI	-	YES	YES	0.01-0.05	YES	Agilent 1200 LC+6460QQQ	10	ACN	DSPE	YMC Triart C18 2*100 mm 3µm	5	Both	7	Daily
051	Tentative detected		0.5	qualifiers	GC	MSD	IT	EI		NO	NO		YES	varian GCMS 4000	10	ACN	PSA	varian factor four	5	Both		

APPENDIX 3. Methods used by participants for detecting pesticides.

CADUSAFOS																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6490	5	ACN		RP-C18	10	Both		Daily
002	Confirmed with second method	6.8			GC	MSD	Q	EI	+	YES	YES	not validated	YES	AT 5975	10	ACN	SPE	HP5-MS	10	Auto	aprox 750	Always
003	Confirmed with second method	0.05	5	2 transitions	LC		Q-TOF	ESI	+	NO	NO		YES	Bruker Maxis	10	ACN	PSA	RSLC C18	1	Both	aprox 400	
004	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA		GC	MSD	QQQ	EI	+	YES	YES	0.01	NO	Thermo TSQ Quantum XLS	10	EtOAc	None	Thermo TG-5SILMS, 30m*0.25mm* 0.25um	2	Auto	500	Each Batch
	Confirmed with second method	1.2	0.4		LC	MSD	TOF	ESI	+	NO	NO	NA	YES	Bruker Maxis	10	EtOAc	None	Acclaim RSLC 120 C18, 2,1*100 mm	1	Both	1000	Each Batch
006	Fully identified (SANCO/12495/2011 p.74-80)	<2	0	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	>0.010 mg/kg	NO	Waters quattro premier	15	ACN	None	BEH C18	3	Both		
007	Fully identified (SANCO/12495/2011 p.74-80)	1	None		GC	MSD	Q	EI	+	YES	YES	0.01	NO	Agilent 6890/5973	10	ACN	DSPE	HP-5MS	2	Manual	183	Each Batch
008	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions +accurate mass	LC	MSD	Q-TRAP	ESI	+	YES	NO	0.01	NO	QTrap 5500	10	ACN	None	reversed phase	2.5	Auto	300	Each Batch
009	Tentative detected	36		2 transitions	LC		QQQ	ESI	+	NO	NO		NO	API4000	10	ACN	DSPE	Synergie Fusion RP80A	8	Manual	250	Scheduled MRM Method also used for quantification
010	Fully identified (SANCO/12495/2011 p.74-80)	5.0		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01	YES	HP5975C	10	ACN	DSPE	HP5MS	10	Both	aprox 1000	Always
011	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	NA	GC	MSD	Q	EI	+	NO	NO	NA	YES	5790 msd	10	ACN	PSA	HP-5MS	20	Auto	600 aprox	0-1
012	Fully identified (SANCO/12495/2011 p.74-80)	0.6		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010	NO	Waters Quattro Premier	10	Methanol	NO	C18	5	Both	211	Always

APPENDIX 3. Methods used by participants for detecting pesticides.

CADUSAFOS																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
014	Fully identified (SANCO/12495/2011 p.74-80)	±2.5%		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010 mg/kg	YES	Waters-TQD	15	ACN	PSA	UPLC-BEH C18	10	Auto	331	Each Batch
015	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	2 transitions	GC		QQQ	EI	+	YES	YES	0.01	NO	7000B	10	ACN	PSA	DB-5MS UI	2	Both	440	
016	Confirmed with second method					MSD	QQQ	EI	+	NO	YES	0.010	NO	GC-MS/MS (Agilent)	15	ACN	QuEChERS	DB 5MS	1	Both	130	Always
017	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	TOF	EI		YES	NO		YES	LECO PEGASUS	15	Acetone/PE/DCM	NO	DB-5	1	Both		
018	Tentative detected	RT locking		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01 - 0.05 mg/kg	YES	Agilent MSD 5975B	10	ACN	PSA	HP5	10	Both	> 600	Always
019	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	GC	MSD	QQQ	EI		YES	YES	10	NO	Agilent 6890	15	Acetone/PE/DCM	None	95%methyl 5% phenyl siloxane	2	Auto		
020	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	QQQ	EI		YES	YES	0.01	NO	agilent	10	ACN	DSPE	HP5MS	1		200	Always
021	Fully identified (SANCO/12495/2011 p.74-80)	15		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	HPLC Agilent 1100, MS API 3000	10	ACN	DSPE (PSA, MgSO4)	C18 3µ 50x2mm	10	Both	550	Always
022	Tentative detected	0.5	0.05		LC	MSD	TOF	ESI	+	YES	YES		YES	LCT PREMIER XE	15	MINI LUKE	NO	C-18	7	Auto	343	Every Day
023	Confirmed with second method				LC		QQQ	ESI	+	YES	YES	0.01	NO	API 5500	10	ACN		Pursuit XRs Ultra	3	Both	250	Each Batch
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		3 masses	GC	MSD	IT	EI	+	YES	YES	0.01	YES	Varian Saturn 2000	10	ACN	PSA	VactorFour 5 MS	5	Auto	500	Daily
025	Fully identified (SANCO/12495/2011 p.74-80)	-0.22	-0.05	isotope, 2 ions < 5ppm	LC	MSD	Orbitrap	ESI	+	NO	NO	0.01-0.1	YES	Exactive	10	ACN	None	C18 Atlantis T3	5	Both	570	Each Batch
	Fully identified (SANCO/12495/2011 p.74-80)	< 20	925	spectrum	GC	MSD	TOF	EI	+	YES	YES	0.01-0.1	YES	Leco Pegasus IV	10	ACN	DSPE (PSA)	RTX-CL pest	10	Auto	557	Each Batch
026	Fully identified (SANCO/12495/2011 p.74-80)	< 0.5 %	2.5		GC	MSD	ion trap	EI		YES	YES	0.01	YES	Trap 4000 - Varian	10	ACN	DSPE	5%	1 µL	Auto	450	Every Week

APPENDIX 3. Methods used by participants for detecting pesticides.

CADUSAFOS																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
027	Fully identified (SANCO/12495/2011 p.74-80)	4	0.7	3 transitions	GC	MSD	QQQ	EI	+	YES	YES	0.01	NO	Varian 3800 GC + 320-MS	10	ACN	DSPE with PSA	VF-1ms	4	Both	302	Each Batch
028	Fully identified (SANCO/12495/2011 p.74-80)			271.1>215.0, 271.1>159.0	LC	MSD	QQQ	ESI	+	YES	YES	LOQ: 2 ppb	NO	LC: Agilent 1200, MS: API 4000 QTRAP	10	ACN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Auto		
029	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO		10	ACN	NO	Xterra C18	20	Both		
031	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MS/MS	QQQ	EI	+	YES	YES	0.005-0.64 µg/ml	NO	Waters Quattro Micro GC	10	EtOAc	Filter	Rxi-5sil MS/integra-guard Restek	10	Both	373	Each Batch
032	Tentative detected	0		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Waters Acquity TQD	10	Methanol	Filter	C18	6	Auto	150	Always
033	Fully identified (SANCO/12495/2011 p.74-80)	3.5		min 3 ions	GC	MSD	Q	EI	+	YES	NO	0.01	YES	Agilent GC-MSD 7890A/5975c	10	ACN	PSA, C18	HP-5MS	1	Auto	927	
034	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC		QQQ	ESI	+	YES	YES		NO	Waters Quattro Premier	10	ACN	DSPE (PSA/MgSO4)	RP-18 endcapped	5	Both	186	Each Batch
036	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MSD	QQQ	EI	+	YES					10	ACN			5	Auto	240	
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.010	NO	4000 QTRAP	10	ACN	DSPE	C18 hydro Phenomenex	40	Manual		Always
038	Fully identified (SANCO/12495/2011 p.74-80)	None	None	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES		NO	API 4000	10	ACN	DSPE	Waters-C18 2,1x50mmx1,7,µm	5	Auto	600	Every Week
039	Fully identified (SANCO/12495/2011 p.74-80)			271.1->159.0, 271.1->130.9	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6410B	10	ACN	DSPE	C18	2	Both	187	Each Batch
040	Confirmed with second method				GC	MSD	Q	EI		NO	NO		YES	Agilent 5973	30	EtOAc	HPGPC	DB-5MS	2	Auto	aprox 500	
041	Fully identified (SANCO/12495/2011 p.74-80)	0.5%		2 transitions	GC	MSD	QQQ	EI		YES	YES	0.005 mg/kg	NO	VARIAN CP-3800 MS-320	10	ACN	PSA	VF-5MS	1	Auto	210	

APPENDIX 3. Methods used by participants for detecting pesticides.

CADUSAFOS																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
042	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES		NO	API 4000	10	ACN	QuEChERS	C18 Aqua	10	Both		
043	Tentative detected	0	0	scan	GC	MSD	IT	EI		YES	YES	0.010	YES	Varian Saturn 4000	10	ACN	DSPE	Varian VF	1	Auto	260	Daily
045	Tentative detected	1	1 unit		GC	MSD	Q	EI		YES	NO	0.01 ppm	YES	Agilent 5973	10	ACN	PSA	HP 5 MS UI	2	Auto	923	Each Batch
046	Fully identified (SANCO/12495/2011 p.74-80)	0			GC	MSD	QQQ	EI	+	YES	YES		NO	Thermo TSQ Quantum GC	10	Acetone/PE/DCM	Na2SO4	RXi-5SIL MS	1	Both	199	Each Batch
047	Fully identified (SANCO/12495/2011 p.74-80)	0		Spectrum match (reversed)	GC	MSD	IT	EI	+	YES	YES	20-10.000 µg/kg	YES	Varian Saturn 2000	15	Acetone/PE/DCM	NO	VF-5-ms	5	Both	550	Quarterly
048	Confirmed with second method	5			GC	MSD	TOF	EI		YES	YES	0.005	YES	Pegasus IV	10	ACN	DSPE(PSA)	HP-5MS	5	Both	650	Weekly
049	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC		QQQ	EI	+	YES	YES		NO	Waters Quattro micro GC	10	ACN		DB-5MS	3	Both	150	Daily
050	Fully identified (SANCO/12495/2011 p.74-80)	0.02		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01-0.05	YES	Agilent 1200 LC+6460QQQ	10	ACN	DSPE	YMC Triart C18 2*100 mm 3µm	5	Both	354	Daily
053	Fully identified (SANCO/12495/2011 p.74-80)	2		3 SIM Ions	GC	MSD	Q	EI	+	NO	NO	>0.05 mg/kg	YES	Thermo DSQ	10	DCM	Diatomaceous earth		1	Both	400	Every 6 Months
	Fully identified (SANCO/12495/2011 p.74-80)	2		2 MRM transitions	LC	MSD	QQQ	ESI	+	YES	YES	>0.005 mg/kg	NO	API4000	10	ACN	PSA		10	Both	180	Every Month

APPENDIX 3. Methods used by participants for detecting pesticides.

DIFLUBENZURON																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6490	5	ACN		RP-C18	10	Both		Daily
003	Confirmed with second method	0.05	5	2 transitions	LC		Q-TOF	ESI	+	NO	NO		YES	Bruker Maxis	10	ACN	PSA	RSLC C18	1	Both	aprox 400	
004	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA		LC	MSD	QQQ	ESI	-	YES	YES	0.01	NO	Agilent 6460, 1290HPLC	10	EtOAc	None	Waters, HSS T3, 150 x 2.1mm	2	Auto	500	Each Batch
	Confirmed with second method	-2.4	0		LC	MSD	TOF	ESI	+	NO	NO	NA	YES	Bruker Maxis	10	EtOAc	None	Acclaim RSLC 120 C18, 2,1*100 mm	1	Both	1000	Each Batch
006	Fully identified (SANCO/12495/2011 p.74-80)	<2	0	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	>0.010 mg/kg	NO	Waters quattro premier	15	ACN	None	BEH C18	3	Both		
007	Fully identified (SANCO/12495/2011 p.74-80)	1	None		LC	MSD	QQQ	ESI	-	YES	YES	0.01	NO	Agilent 6410	10	ACN	DSPE	Eclipse XDB-C18	4	Manual	139	Each Batch
008	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions +accurate mass	LC	MSD	Q-TRAP	ESI	+	YES	NO	0.01	NO	QTrap 5500	10	ACN	None	reversed phase	2.5	Auto	300	Each Batch
009	Tentative detected	35		2 transitions	LC		QQQ	ESI	+	YES	YES	0.01 (=RL)	NO	API4000	10	ACN	DSPE	Synergie Fusion RP80A	8	Manual	250	Scheduled MRM Method also used for quantification
010	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	ABI4000	10	ACN	NO	C18	55	Both	aprox 580	Always
011	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	NA	LC	MSD	Q-TRAP	ESI	+	YES	YES	0.01	NO	3200qtrap	10	ACN	PSA	T-3	5	Manual	170	1
012	Fully identified (SANCO/12495/2011 p.74-80)	0.6		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010	NO	Waters Quattro Premier	10	Methanol	NO	C18	5	Both	211	Always
014	Fully identified (SANCO/12495/2011 p.74-80)	±2.5%		2 transitions	LC	MSD	QQQ	ESI	-	YES	YES	0.010 mg/kg	YES	Waters-TQD	15	ACN	PSA	UPLC-BEH C18	10	Auto	331	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

DIFLUBENZURON																				
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
015	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	2 transitions	LC		QQQ	ESI	+	YES	YES	0.01	NO	6460	10	ACN	PSA	C18-XB	2	Both	440	
016	Confirmed with second method					MSD	QQQ	CI	+	YES	YES	0.005	NO	LC-MS/MS (Waters)	15	ACN	QuEChERS	C18	10	Both	370	Always
017	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	TOF	EI		YES	YES		YES	LECO PEGASUS	15	Acetone/PE/DCM	NO	DB-5	1	Both		
018	Tentative detected	5	5	accurate mass	LC	Orbitrap	Orbitrap	ESI	+	YES	YES	0.01 - 0.05 mg/kg	YES	Thermo Exactive	10	ACN	PSA	C18	5	Both	120	Always
019	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	LC	MSD	QQQ	ESI	-	YES	YES	10	NO	Agilent 6490	15	ACN	PSA	C18	5	Auto		
020	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	AbSciex	10	ACN	DSPE	C18	5	Auto	200	Always
021	Fully identified (SANCO/12495/2011 p.74-80)	15		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	HPLC Agilent 1100, MS API 3000	10	ACN	DSPE (PSA, MgSO4)	C18 3µ 50x2mm	10	Both	550	Always
022	Tentative detected	0.5	0.05		LC	MSD	TOF	ESI	+	YES	YES		YES	LCT PREMIER XE	15	MINI LUKE	NO	C-18	7	Auto	343	Every Day
023	Confirmed with second method				LC		IT	ESI	-	YES	YES	0.01	NO		10	ACN				Both		Each Batch
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		2 x MRM	LC	MSD	QQQ	ESI	-	YES	YES	0.01	NO	AB Sciex API 4000	10	ACN	PSA	Phenomenex C18	10	Both	500	Daily
025	Fully identified (SANCO/12495/2011 p.74-80)	-0.19	-1.0	isotope, 2 ions < 5ppm	LC	MSD	Orbitrap	ESI	+	NO	NO	0.01-0.1	YES	Exactive	10	ACN	None	C18 Atlantis T3	5	Both	570	Each Batch
026	Fully identified (SANCO/12495/2011 p.74-80)	< 2.5 %	2.5	transition 1 and 2	LC	MSD	Q-TRAP	ESI	+	YES	YES	0.01	YES	3200 Qtrap	10	ACN	DSPE	C18	50	Auto	521	Every Week
027	Fully identified (SANCO/12495/2011 p.74-80)	4	0.7	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Varian Pro Star + 320-MS	10	ACN	DSPE with PSA	Restek Ultra C18	10	Both	302	Each Batch
028	Fully identified (SANCO/12495/2011 p.74-80)			311.1>158.2, 311.1>141.2	LC	MSD	QQQ	ESI	-	YES	YES	LOQ: 2 ppb	NO	LC: Agilent 1200, MS: API 4000 QTRAP	10	ACN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Auto		

APPENDIX 3. Methods used by participants for detecting pesticides.

DIFLUBENZURON																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
030	Tentative detected				LC		QQQ	ESI	-	YES	YES	0.02	NO	Varian 320	15	ACN		C18	10	Auto	150	Each Batch
031	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	-	YES	YES	0.005-0.4 µg/ml	NO	Waters Aquity UPLC system, API 5000 Tripelquadropol, Sciex	10	EtOAc	Filter	HSS T3	2	Both	13	Each Batch
034	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC		QQQ	ESI	+	YES	YES		NO	Waters Quattro Premier	10	ACN	DSPE (PSA/MgSO4)	RP-18 endcapped	5	Both	186	Each Batch
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	-	YES	YES	0.010	NO	4000 QTRAP	10	ACN	DSPE	C18 hydro Phenomenex	40	Manual		Always
038	Fully identified (SANCO/12495/2011 p.74-80)	None	None	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES		NO	API 4000	10	ACN	DSPE	Waters-C18 2,1x50mmx1,7,µm	5	Auto	600	Every Week
039	Fully identified (SANCO/12495/2011 p.74-80)			311.0->158.0, 311.0->141.0	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6410B	10	ACN	DSPE	C18	2	Both	187	Each Batch
040	Confirmed with second method	4	4		LC		TOF	ESI	+	NO	NO		YES	Agilent 6230	10	ACN	None	Eclipse C18	3	Auto	aprox 500	
041	Fully identified (SANCO/12495/2011 p.74-80)	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.005 mg/kg	NO	AGILENT G-6410A	10	ACN	PSA	ZORBAX-ECLIPSE XDB-C8	80	Auto	143	
042	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	-	YES	YES		NO	API 4000	10	ACN	QuEChERS	C18 Aqua	10	Both		
043	Tentative detected	0	0	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010	YES	Api 3200QT	10	ACN	DSPE	Phenomenex fusion	10	Auto	200	Daily
046	Fully identified (SANCO/12495/2011 p.74-80)	0			LC	MSD	QQQ	ESI	+	NO			NO	Waters Xevo	10	Acetone/PE/DCM	Na2SO4	C18	2	Both	286	Each Batch
047	Fully identified (SANCO/12495/2011 p.74-80)	2.4		2 transitions + ion ratio	LC	MSD	QQQ	ESI	-	NO	NO	5-1000 µg/kg	NO	Waters Quattro Premier XE	15	Acetone/PE/DCM	NO	Alltima C18 (150x3 mm i.d.)	5	Both	27	Each Batch
048	Fully identified (SANCO/12495/2011 p.74-80)	0			LC	MSD	QQQ	ESI	+	YES	YES	0.005	NO	Agilent 6410	10	ACN	DSPE(PSA)	Luna C18	5	Both	350	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

DIFLUBENZURON																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
049	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC		QQQ	EI	+	YES	YES		NO	Waters Quattro micro GC	10	ACN		DB-5MS	3	Both	150	Daily
050	Fully identified (SANCO/12495/2011 p.74-80)	0.019		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01-0.05	YES	Agilent 1200 LC+6460QQQ	10	ACN	DSPE	YMC Triart C18 2*100 mm 3µm	5	Both	354	Daily
051	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.05		ABSciex API 2000	10	ACN	PSA	waters Atlantis	5	Auto	117	Each Batch
053	Fully identified (SANCO/12495/2011 p.74-80)	0		3 SIM Ions	GC	MSD	Q	EI	+	NO	NO	>0.05 mg/kg	YES	Thermo DSQ	10	DCM	Diatomaceous earth		1	Both	400	Every 6 Months
	Fully identified (SANCO/12495/2011 p.74-80)	0		2 MRM transitions	LC	MSD	QQQ	ESI	+	YES	YES	>0.005 mg/kg	NO	API4000	10	ACN	PSA		10	Both	180	Every Month

APPENDIX 3. Methods used by participants for detecting pesticides.

DINICONAZOLE																				
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6490	5	ACN		RP-C18	10	Both		Daily
002	Confirmed with second method	5.3			GC	MSD	Q	EI	+	YES	YES	not validated	YES	AT 5975	10	ACN	SPE	HP5-MS	10	Auto	aprox 750	Always
003	Confirmed with second method	0.05	5	2 transitions	LC		Q-TOF	ESI	+	NO	NO		YES	Bruker Maxis	10	ACN	PSA	RSLC C18	1	Both	aprox 400	
004	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA		LC	MSD	QQQ	ESI	+	YES	NO	NA	NO	Agilent 6460, 1290HPLC	10	EtOAc	None	Waters, HSS T3, 150 x 2.1mm	2	Both	500	Each Batch
	Confirmed with second method	-0.6	0.3		LC	MSD	TOF	ESI	+	NO	NO	NA	YES	Bruker Maxis	10	EtOAc	None	Acclaim RSLC 120 C18, 2,1*100 mm	1	Both	1000	Each Batch
006	Fully identified (SANCO/12495/2011 p.74-80)	<2	0	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	>0.010 mg/kg	NO	Waters quattro premier	15	ACN	None	BEH C18	3	Both		
008	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions+accurate mass	LC	MSD	Q-TRAP	ESI	+	YES	NO	0.01	NO	QTrap 5500	10	ACN	None	reversed phase	2.5	Auto	300	Each Batch
009	Confirmed with second method	21		2 transitions	LC		QQQ	ESI	+	YES	YES	0.01 (=RL)	NO	API5500	10	ACN	DSPE	Synergie Fusion RP80A	8	Manual	35	Scheduled MRM Method also used for quantification
010	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	ABI4000	10	ACN	NO	C18	55	Both	aprox 580	Always
011	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	NA	GC	MSD	Q	EI	+	NO	NO	NA	YES	5790 msd	10	ACN	PSA	HP-5MS	20	Auto	600 aprox	0-1
012	Fully identified (SANCO/12495/2011 p.74-80)	0.6		2 transitions	GC	MSD	QQQ	EI	+	YES	YES	0.010	NO	Varian 320	50	Acetone	L/L	DB5 type	8	Both	146	Always
014	Fully identified (SANCO/12495/2011 p.74-80)	±2.5%		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010 mg/kg	YES	Waters-TQD	15	ACN	PSA	UPLC-BEH C18	10	Auto	331	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

DINICONAZOLE																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
015	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	2 transitions	GC		QQQ	EI	+	YES	YES	0.01	NO	7000B	10	ACN	PSA	DB-5MS UI	2	Both	440	
016	Confirmed with second method					MSD	QQQ	CI	+	YES	YES	0.005	NO	LC-MS/MS (Waters)	15	ACN	QuEChERS	C18	10	Both	370	Always
017	Tentative detected				GC	MSD	TOF	EI		NO	NO		YES	LECO PEGASUS	15	Acetone/PE/DCM	NO	DB-5	1	Both		
018	Tentative detected	RT locking		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01 - 0.05 mg/kg	YES	Agilent MSD 5975B	10	ACN	PSA	HP5	10	Both	> 600	Always
019	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	10	NO	Agilent 6490	15	ACN	PSA	C18	5	Auto		
020	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	AbSciex	10	ACN	DSPE	C18	5	Auto	200	Always
021	Fully identified (SANCO/12495/2011 p.74-80)	15		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	HPLC Agilent 1100, MS API 3000	10	ACN	DSPE (PSA, MgSO4)	C18 3µ 50x2mm	10	Both	550	Always
022	Tentative detected	0.5	0.05		LC	MSD	TOF	ESI	+	YES	YES		YES	LCT PREMIER XE	15	MINI LUKE	NO	C-18	7	Auto	343	Every Day
023	Confirmed with second method				GC	MSD	TOF			YES	YES	0.01	YES	Leco Pegasus IV	10	ACN	DSPE	Phenomenenx ZB-5MS	3	Both	750	Each Batch
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		2 x MRM	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	AB Sciex API 4000	10	ACN	PSA	Phenomenex C18	10	Both	500	Daily
025	Fully identified (SANCO/12495/2011 p.74-80)	-0.2	0.03	isotope, 2 ions < 5ppm	LC	MSD	Orbitrap	ESI	+	NO	NO	0.01-0.1	YES	Exactive	10	ACN	None	C18 Atlantis T3	5	Both	570	Each Batch
	Fully identified (SANCO/12495/2011 p.74-80)	< 20	923	spectrum	GC	MSD	TOF	EI	+	YES	YES	0.01-0.1	YES	Leco Pegasus IV	10	ACN	DSPE (PSA)	RTX-CL pest	10	Auto	557	Each Batch
026	Fully identified (SANCO/12495/2011 p.74-80)	< 0.5 %	2.5		GC	MSD	ion trap	EI		YES	YES	0.01	YES	Trap 4000 Varian	10	ACN	DSPE	5%	1 µL	Auto	450	Every Week
027	Fully identified (SANCO/12495/2011 p.74-80)	1	0.7	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Varian Pro Star +320-MS	10	ACN	DSPE with PSA	Restek Ultra C18	10	Both	302	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

DINICONAZOLE																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
028	Fully identified (SANCO/12495/2011 p.74-80)			326.1>70.1, 328.1>70.1	LC	MSD	QQQ	ESI	+	YES	YES	LOQ: 8 ppb	NO	LC: Agilent 1200, MS: API 4000 QTRAP	10	ACN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Auto		
029	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	NO	NO				10	ACN	NO	Xterra C18	20	Both		
030	Tentative detected				LC		QQQ	ESI	+	YES	YES	0.02	NO	Varian 320	15	ACN		C18	10	Auto	150	Each Batch
031	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.025-0.25 µg/ml	NO	UPLC-MSMS (Waters Aquity UPLC system, API 5000 Tripelquadropol, Sciex	10	EtOAc	Filter	HSS T3	2	Both	141	Each Batch
032	Tentative detected	0		2 transitions	GC	MSD	QQQ	EI	+	YES	YES	0.01	NO	Thermo TSQ Quantum XLS Ultra	10	ACN	PSA	Capillary	1	Both	150	Always
033	Fully identified (SANCO/12495/2011 p.74-80)	-0.9		min 3 ions	GC	MSD	Q	EI	+	YES	NO	0.01	YES	Agilent GC-MSD 7890A/5975c	10	ACN	PSA, C18	HP-5MS	1	Auto	927	
034	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC		QQQ	ESI	+	YES	YES		NO	Waters Quattro Premier	10	ACN	DSPE (PSA/MgSO4)	RP-18 endcapped	5	Both	186	Each Batch
036	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES					10	ACN			5	Auto	350	
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.010	NO	4000 QTRAP	10	ACN	DSPE	C18 hydro Phenomenex	40	Manual		Always
038	Tentative detected	None	None	2 transitions	LC	MSD	QQQ	ESI	+	NO	NO		NO	API 4000	10	ACN	DSPE	Waters-C18 2,1x50mmx1,7,µm	5	Auto	600	Every Week
039	Tentative detected				GC	MSD	Q	EI	+	NO	NO		YES	HP5973 MSD	10	ACN	DSPE	HP-5MSI	20	Both	624	No standard used
040	Tentative detected	5	4		LC		TOF	ESI	+	NO	NO		YES	Agilent 6230	10	ACN	None	Eclipse C18	3	Auto	aprox 500	
041	Fully identified (SANCO/12495/2011 p.74-80)	0.5%		2 transitions	GC	MSD	QQQ	EI		YES	YES	0.005 mg/kg	NO	VARIAN CP-3800 MS-320	10	ACN	PSA	VF-5MS	1	Auto	210	

APPENDIX 3. Methods used by participants for detecting pesticides.

DINICONAZOLE																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
042	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES		NO	API 4000	10	ACN	QuEChERS	C18 Aqua	10	Both		
043	Tentative detected	0	0	scan	GC	MSD	IT	EI		YES	YES	0.010	YES	Varian Saturn 4000	10	ACN	DSPE	Varian VF	1	Auto	260	Daily
045	Tentative detected	1	1 unit		GC	MSD	Q	EI		YES	NO	0.01 ppm	YES	Agilent 5973	10	ACN	PSA	HP 5 MS UI	2	Auto	923	Each Batch
046	Fully identified (SANCO/12495/2011 p.74-80)	0			LC	MSD	QQQ	ESI	+	NO			NO	Waters Xevo	10	Acetone/PE/DCM	Na2SO4	C18	2	Both	286	Each Batch
047	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions + ion ratio	LC	MSD	QQQ	ESI	+	YES	YES	5-1000 µg/kg	NO	Waters Quattro Premier XE	15	Acetone/PE/DCM	NO	Acquity BEH (100x2.1 mm i.d.)	5	Both	230	Each Batch
048	Confirmed with second method	6			GC	MSD	TOF	EI		YES	YES	0.005	YES	Pegasus IV	10	ACN	DSPE(PSA)	HP-5MS	5	Both	650	Weekly
049	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC		QQQ	EI	+	YES	YES		NO	Waters Quattro micro GC	10	ACN		DB-5MS	3	Both	150	Daily
050	Fully identified (SANCO/12495/2011 p.74-80)	-0.026		Qion ratio	GC	MSD	Q	EI	+	YES	YES	0.01-0.05	YES	Agilent 6890GC+5975MS	10	ACN	DSPE	RESTEK-5MS	50	Both	168	Daily
051	Fully identified (SANCO/12495/2011 p.74-80)	15	0.5	qualifiers	GC	MSD	IT	EI		YES	YES	0.01	YES	Varian GCMS 4000	10	ACN	PSA	varian factor four	5	Both	130	Each Batch
	Fully identified (SANCO/12495/2011 p.74-80)	120		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.05		ABSciex API 2000	10	ACN	PSA	waters Atlantis	5	Auto	117	Each Batch
053	Tentative detected	8		3 SIM Ions	GC	MSD	Q	EI	+	NO	NO	>0.05 mg/kg	YES	Thermo DSQ	10	DCM	Diatomaceous earth		1	Both	400	Every 6 Months

APPENDIX 3. Methods used by participants for detecting pesticides.

ETHOXIQUIN																				
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6490	5	ACN		RP-C18	10	Both		Daily
002	Confirmed with second method	3.0			GC	MSD	Q	EI	+	YES	YES	not validated	YES	AT 5975	10	ACN	SPE	HP5-MS	10	Auto	aprox 750	Always
003	Confirmed with second method	0.05	5	2 transitions	LC		Q-TOF	ESI	+	NO	NO		YES	Bruker Maxis	10	ACN	PSA	RSLC C18	1	Both	aprox 400	
004	Confirmed with second method	-1.2	1.9		LC	MSD	TOF	ESI	+	NO	NO	NA	YES	Bruker Maxis	10	EtOAc	None	Acclaim RSLC 120 C18, 2,1*100 mm	1	Both	1000	Each Batch
	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA		LC	MSD	QQQ	ESI	+	YES	NO	NA	NO	Agilent 6460, 1290HPLC	10	EtOAc	None	Waters, HSS T3, 150 x 2.1mm	2	Both	500	Each Batch
006	Fully identified (SANCO/12495/2011 p.74-80)	<2	0	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	>0.010 mg/kg	NO	Waters quattro premier	15	ACN	None	BEH C18	10	Both		
008	Tentative detected			2 transitions	LC	MSD	Q-TRAP	ESI	+	NO	NO		NO	QTrap 5500	10	ACN	None	reversed phase	2.5	Auto	300	We do not have a standard
009	Tentative detected			2 transitions	GC	MSD	QQQ	EI	+	NO	NO		NO	Thermo TSQ Quantum	10	ACN	DSPE	TG-5MS 30m	2	Manual	80	Each Batch
010	Fully identified (SANCO/12495/2011 p.74-80)	0.3		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01	YES	HP5975C	10	ACN	DSPE	HP5MS	10	Both	aprox 1000	Always
011	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	NA	GC	MSD	Q	EI	+	NO	NO	NA	YES	5790 msd	10	ACN	PSA	HP-5MS	20	Auto	600 aprox	0-1
012	Fully identified (SANCO/12495/2011 p.74-80)	0.6		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010	NO	Waters Quattro Premier	10	Methanol	NO	C18	5	Both	211	Always
014	Fully identified (SANCO/12495/2011 p.74-80)	±0.5%			GC	MSD	Q	EI	+	YES	YES	0.010 mg/kg	YES	Agilent 7890-5975C	15	ACN	PSA	HP-5MS	10	Auto	211	Each Batch
015	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	2 transitions	GC		QQQ	EI	+	YES	YES	0.01	NO	7000B	10	ACN	PSA	DB-5MS UI	2	Both	440	
016	Confirmed with second method					MSD	QQQ	CI	+	YES	YES	0.005	NO	LC-MS/MS (Waters)	15	ACN	QuEChERS	C18	10	Both	370	Always

APPENDIX 3. Methods used by participants for detecting pesticides.

ETHOXIQUIN																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
017	Tentative detected				GC	MSD	TOF	EI		NO	NO		YES	LECO PEGASUS	15	Acetone/PE/DCM	NO	DB-5	1	Both		
018	Tentative detected	RT locking		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01 - 0.05 mg/kg	YES	Agilent MSD 5975B	10	ACN	PSA	HP5	10	Both	> 600	Always
020	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	QQQ	EI		YES	YES	0.01	NO	agilent	10	ACN	DSPE	HP5MS	1		200	Always
021	Fully identified (SANCO/12495/2011 p.74-80)	15		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	HPLC Agilent 1100, MS API 3000	10	ACN	DSPE (PSA, MgSO4)	C18 3µ 50x2mm	10	Both	550	Always
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		2 x MRM	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	AB Sciex API 4000	10	ACN	PSA	Phenomenex C18	10	Both	500	Daily
025	Confirmed with second method	NA	< 5	isotope, 2 ions < 5ppm	LC	MSD	Orbitrap	ESI	+	NO	NO	0.01-0.1	YES	Exactive	10	ACN	None	C18 Atlantis T3	5	Both	570	Each Batch
	Confirmed with second method	< 20	854	spectrum	GC	MSD	TOF	EI	+	YES	YES	0.01-0.1	YES	Leco Pegasus IV	10	ACN	DSPE (PSA)	RTX-CL pest	10	Auto	557	Each Batch
026	Fully identified (SANCO/12495/2011 p.74-80)	< 0.5 %	2.5		GC	MSD	ion trap	EI		YES	YES	0.01	YES	Trap 4000 - Varian	10	ACN	DSPE	5%	1 µL	Auto	450	Every Week
028	Fully identified (SANCO/12495/2011 p.74-80)			Match with analytical standard (Match 819, range 0-1000)	GC	MSD	TOF	EI		YES	YES	LOQ: 10 ppb	YES	GCxGC-TOF MS Pegasus 4D	10	ACN	QuEChERS without PSA	DB5-MS (30 m x 0,25 mm x 0,25 µm) in the 1st Dimension, BPX-50 (2 m x 0,1 mm x 0,1 µm) in the 2nd Dimension	10	Auto		
030	Tentative detected				LC		QQQ	ESI	+	YES	YES	0.02	NO	Varian 320	15	ACN		C18	10	Auto	150	Each Batch
031	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MS/MS	QQQ	EI	+	YES	YES	0.025-0.25 µg/ml	NO	Waters Quattro Micro GC	10	EtOAc	Filter	Rxi-5sil MS/integra-guard Restek	10	Both	141	Each Batch
034	Tentative detected			full scan	GC	MSD	Q	EI	+	NO	NO		YES	Agilent 5973 MSD	10	ACN	DSPE (PSA/MgSO4)	HP-5MS	10	Both	950	
035	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	Q	EI		NO	NO		YES	Agilent 6890N	10	ACN	DSPE	DB-5	2	Auto		
038	Tentative detected	None	None	2 transitions	LC	MSD	QQQ	ESI	+	NO	NO		NO	API 4000	10	ACN	DSPE	Waters-C18 2,1x50mmx1,7,µm	5	Auto	600	Every Week

APPENDIX 3. Methods used by participants for detecting pesticides.

ETHOXIQUIN																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
039	Tentative detected				GC	MSD	Q	EI	+	NO	NO		YES	HP5973 MSD	10	ACN	DSPE	HP-5MSI	20	Both	624	No standard used
040	Confirmed with second method	1	4		LC		TOF	ESI	+	NO	NO		YES	Agilent 6230	10	ACN	None	Eclipse C18	3	Auto	aprox 500	
041	Fully identified (SANCO/12495/2011 p.74-80)	0.5%		2 transitions	GC	MSD	QQQ	EI		YES	YES	0.005 mg/kg	NO	VARIAN CP-3800 MS-320	10	ACN	PSA	VF-5MS	1	Auto	210	
043	Tentative detected	0	0	scan	GC	MSD	IT	EI		YES	YES	0.010	YES	Varian Saturn 4000	10	ACN	DSPE	Varian VF	1	Auto	260	Daily
045	Tentative detected	1	1 unit		GC	MSD	Q	EI		YES	NO	0.01 ppm	YES	Agilent 5973	10	ACN	PSA	HP 5 MS UI	2	Auto	923	Each Batch
046	Fully identified (SANCO/12495/2011 p.74-80)	0			GC	MSD	QQQ	EI	+	YES	YES		NO	Thermo TSQ Quantum GC	10	Acetone/PE/DCM	Na2SO4	RXi-5SIL MS	1	Both	199	Each Batch
047	Fully identified (SANCO/12495/2011 p.74-80)	0.4		Spectrum match (reversed)	GC	MSD	IT	EI	+	YES	YES	20-10.000 µg/kg	YES	Varian Saturn 2000	15	Acetone/PE/DCM	NO	VF-5-ms	5	Both	550	Quarterly
048	Confirmed with second method	4			GC	MSD	TOF	EI		YES	YES	0.01	YES	Pegasus IV	10	ACN	DSPE(PSA)	HP-5MS	5	Both	650	Weekly
049	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC		QQQ	EI	+	YES	YES		NO	Waters Quattro micro GC	10	ACN		DB-5MS	3	Both	150	Daily
051	Tentative detected		0.5	qualifiers	GC	MSD	IT	EI		NO	NO		YES	varian GCMS 4000	10	ACN	PSA	varian factor four	5	Both		
053	Fully identified (SANCO/12495/2011 p.74-80)	2		2 MRM transitions	LC	MSD	QQQ	ESI	+	YES	YES	>0.005 mg/kg	NO	API4000	10	ACN	PSA		10	Both	180	Every Month

APPENDIX 3. Methods used by participants for detecting pesticides.

ETRIMFOS																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6490	5	ACN		RP-C18	10	Both		Daily
002	Confirmed with second method	1.6			GC	MSD	Q	EI	+	YES	YES	not validated	YES	AT 5975	10	ACN	SPE	HP5-MS	10	Auto	aprox 750	Always
003	Confirmed with second method	0.05	5	2 transitions	LC		Q-TOF	ESI	+	NO	NO		YES	Bruker Maxis	10	ACN	PSA	RSLC C18	1	Both	aprox 400	
004	Confirmed with second method	0.6	0.8		LC	MSD	TOF	ESI	+	NO	NO	NA	YES	Bruker Maxis	10	EtOAc	None	Acclaim RSLC 120 C18, 2,1*100 mm	1	Both	1000	Each Batch
	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA		LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6460, 1290HPLC	10	EtOAc	None	Waters, HSS T3, 150 x 2.1mm	2	Both	500	Each Batch
006	Fully identified (SANCO/12495/2011 p.74-80)	<2	<1	3 diagnostic ions	GC	MSD	Q	EI	+	YES	YES	>0.010 mg/kg	YES	Trace DSQ	15	ACN	PSA/C18	DB5MS	0.8	Both		
007	Fully identified (SANCO/12495/2011 p.74-80)	1	None		GC	MSD	Q	EI	+	YES	YES	0.01	NO	Agilent 6890/5973	10	ACN	DSPE	HP-5MS	2	Manual	183	Each Batch
008	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions +accurate mass	LC	MSD	Q-TRAP	ESI	+	YES	NO	0.01	NO	QTrap 5500	10	ACN	None	reversed phase	2.5	Auto	300	Each Batch
009	Tentative detected			2 transitions	GC	MSD	QQQ	EI	+	NO	NO		NO	Thermo TSQ Quantum	10	ACN	DSPE	TG-5MS 30m	2	Manual	180	Each Batch
010	Fully identified (SANCO/12495/2011 p.74-80)	1.3		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01	YES	HP5975C	10	ACN	DSPE	HP5MS	10	Both	aprox 1000	Always
011	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	NA	GC	MSD	Q	EI	+	NO	NO	NA	YES	5790 msd	10	ACN	PSA	HP-5MS	20	Auto	600 aprox	0-1
012	Fully identified (SANCO/12495/2011 p.74-80)	0.6		2 transitions	GC	MSD	QQQ	EI	+	YES	YES	0.010	NO	Varian 320	50	Acetone	L/L	DB5 type	8	Both	146	Always
014	Fully identified (SANCO/12495/2011 p.74-80)	±0.5%			GC	MSD	Q	EI	+	YES	YES	0.010 mg/kg	YES	Agilent 7890-5975C	15	ACN	PSA	HP-5MS	10	Auto	211	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

ETRIMFOS																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
015	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	2 transitions	GC		QQQ	EI	+	YES	YES	0.01	NO	7000B	10	ACN	PSA	DB-5MS UI	2	Both	440	
016	Confirmed with second method					MSD	QQQ	EI	+	NO	YES	0.010	NO	GC-MS/MS (Agilent)	15	ACN	QuEChERS	DB 5MS	1	Both	130	Always
017	Tentative detected				GC	MSD	TOF	EI		NO	NO		YES	LECO PEGASUS	15	Acetone/PE/DCM	NO	DB-5	1	Both		
018	Tentative detected	RT locking		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01 - 0.05 mg/kg	YES	Agilent MSD 5975B	10	ACN	PSA	HP5	10	Both	> 600	Always
019	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	GC	MSD	QQQ	EI		YES	YES	10	NO	Agilent 6890	15	Acetone/PE/DCM	None	95%methyl 5% phenyl siloxane	2	Auto		
020	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	QQQ	EI		YES	YES	0.01	NO	agilent	10	ACN	DSPE	HP5MS	1		200	Always
021	Fully identified (SANCO/12495/2011 p.74-80)	15		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	HPLC Agilent 1100, MS API 3000	10	ACN	DSPE (PSA, MgSO4)	C18 3µ 50x2mm	10	Both	550	Always
022	Tentative detected	0.5	0.05		LC	MSD	TOF	ESI	+	YES	YES		YES	LCT PREMIER XE	15	MINI LUKE	NO	C-18	7	Auto	343	Every Day
023	Confirmed with second method				LC		QQQ	ESI	+	YES	YES	0.01	NO	API 5500	10	ACN		Pursuit XRs Ultra	3	Both	250	Each Batch
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		3 masses	GC	MSD	IT	EI	+	YES	YES	0.01	YES	Varian Saturn 2000	10	ACN	PSA	VactorFour 5 MS	5	Auto	500	Daily
025	Confirmed with second method	-0.23	-0.04	isotope	LC	MSD	Orbitrap	ESI	+	NO	NO	0.01-0.1	YES	Exactive	10	ACN	None	C18 Atlantis T3	5	Both	570	Each Batch
	Fully identified (SANCO/12495/2011 p.74-80)	< 20	943	spectrum	GC	MSD	TOF	EI	+	YES	YES	0.01-0.1	YES	Leco Pegasus IV	10	ACN	DSPE (PSA)	RTX-CL pest	10	Auto	557	Each Batch
026	Fully identified (SANCO/12495/2011 p.74-80)	< 0.5 %	2.5		GC	MSD	ion trap	EI		YES	YES	0.01	YES	Trap 4000 - Varian	10	ACN	DSPE	5%	1 µL	Auto	450	Every Week
027	Fully identified (SANCO/12495/2011 p.74-80)	2			GC	FPD				YES	YES	0.01	NO	Varian 3800 GC	10	ACN	DSPE with PSA	Rtx-OPP	1	Both	302	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

ETRIMFOS																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
028	Fully identified (SANCO/12495/2011 p.74-80)			Match with analytical standard (Match 927, range 0-1000)	GC	MSD	TOF	EI		YES	YES	LOQ: 2 ppb	YES	GCxGC-TOF MS Pegasus 4D	10	ACN	QuEChERS without PSA	DB5-MS (30 m x 0,25 mm x 0,25 µm) in the 1st Dimension, BPX-50 (2 m x 0,1 mm x 0,1 µm) in the 2nd Dimension	10	Auto		
029	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	IT	EI	+	YES	YES	0.01	YES		10	ACN	NO	DB-5MS	5	Both		
030	Tentative detected				LC		QQQ	ESI	+	YES	YES	0.02	NO	Varian 320	15	ACN		C18	10	Auto	150	Each Batch
031	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.005-0.4 µg/ml	NO	UPLC-MSMS (Waters Aquity UPLC system, API 5000 Tripelquadropol, Sciex	10	EtOAc	Filter	HSS T3	2	Both	373	Each Batch
032	Tentative detected	0		2 transitions	GC	MSD	QQQ	EI	+	YES	YES	0.01	NO	Thermo TSQ Quantum XLS Ultra	10	ACN	PSA	Capillary	1	Both	150	Always
033	Fully identified (SANCO/12495/2011 p.74-80)	-0.5		min 3 ions	GC	MSD	Q	EI	+	YES	YES	0.01	YES	Agilent GC-MSD 7890A/5975c	10	ACN	PSA, C18	HP-5MS	1	Auto	927	
034	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC		QQQ	EI	+	YES	YES		NO	Waters Quattro Micro	10	ACN	DSPE (PSA/MgSO4)	HP-5MS	5	Both	110	Each Batch
036	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES					10	ACN			5	Auto	350	
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.010	NO	4000 QTRAP	10	ACN	DSPE	C18 hydro Phenomenex	40	Manual		Always
038	Tentative detected	None	None	2 transitions	LC	MSD	QQQ	ESI	+	NO	NO		NO	API 4000	10	ACN	DSPE	Waters-C18 2,1x50mmx1,7,µm	5	Auto	600	Every Week
039	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	Q	EI	+	YES	YES	0.01	YES	HP5973 MSD	10	ACN	DSPE	HP-5MSI	20	Both	113	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

ETRIMFOS																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
040	Confirmed with second method				GC	MSD	Q	EI		NO	NO		YES	Agilent 5973	30	EtOAc	HPGPC	DB-5MS	2	Auto	aprox 500	
041	Fully identified (SANCO/12495/2011 p.74-80)	0.5%		2 transitions	GC	MSD	QQQ	EI		YES	YES	0.005 mg/kg	NO	VARIAN CP-3800 MS-320	10	ACN	PSA	VF-5MS	1	Auto	210	
042	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MSD	QQQ	EI		YES	YES		NO	TSQ Quantum	10	ACN	QuEChERS	VF 5 MS	3	Both		
043	Tentative detected	0	0	scan	GC	MSD	IT	EI		YES	YES	0.010	YES	Varian Saturn 4000	10	ACN	DSPE	Varian VF	1	Auto	260	Daily
045	Tentative detected	1	1 unit		GC	MSD	Q	EI		YES	NO	0.01 ppm	YES	Agilent 5973	10	ACN	PSA	HP 5 MS UI	2	Auto	923	Each Batch
046	Fully identified (SANCO/12495/2011 p.74-80)	0			GC	MSD	QQQ	EI	+	YES	YES		NO	Thermo TSQ Quantum GC	10	Acetone/PE/DCM	Na2SO4	RXi-5SIL MS	1	Both	199	Each Batch
047	Fully identified (SANCO/12495/2011 p.74-80)	0.4		Spectrum match (reversed)	GC	MSD	IT	EI	+	YES	YES	20-10.000 µg/kg	YES	Varian Saturn 2000	15	Acetone/PE/DCM	NO	VF-5-ms	5	Both	550	Quarterly
048	Confirmed with second method	3			GC	MSD	TOF	EI		YES	YES	0.005	YES	Pegasus IV	10	ACN	DSPE(PSA)	HP-5MS	5	Both	650	Weekly
049	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC		QQQ	EI	+	YES	YES		NO	Waters Quattro micro GC	10	ACN		DB-5MS	3	Both	150	Daily
050	Fully identified (SANCO/12495/2011 p.74-80)	-0.039		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01-0.05	YES	Agilent 1200 LC+6460QQQ	10	ACN	DSPE	YMC Triart C18 2*100 mm 3µm	5	Both	354	Daily
051	Tentative detected		0.5	qualifiers	GC	MSD	IT	EI		NO	NO		YES	varian GCMS 4000	10	ACN	PSA	varian factor four	5	Both		
053	Tentative detected	5		3 SIM Ions	GC	MSD	Q	EI	+	NO	NO	>0.05 mg/kg	YES	Thermo DSQ	10	DCM	Diatomaceous earth		1	Both	400	Every 6 Months

APPENDIX 3. Methods used by participants for detecting pesticides.

FENPROPIDIN																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	Q	EI	+	YES	NO	0.01	YES	Agilent 5973 inert	5	ACN		HP-5 MSUI	5	Both		Daily
002	Confirmed with second method	0.2			GC	MSD	Q	EI	+	YES	YES	not validated	YES	AT 5975	10	ACN	SPE	HP5-MS	10	Auto	aprox 750	Always
003	Confirmed with second method	0.05	5	2 transitions	LC		Q-TOF	ESI	+	NO	NO		YES	Bruker Maxis	10	ACN	PSA	RSLC C18	1	Both	aprox 400	
004	Confirmed with second method	1.2	1		LC	MSD	TOF	ESI	+	NO	NO	NA	YES	Bruker Maxis	10	EtOAc	None	Acclaim RSLC 120 C18, 2,1*100 mm	1	Both	1000	Each Batch
	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA		LC	MSD	QQQ	ESI	+	YES	NO	NA	NO	Agilent 6460, 1290HPLC	10	EtOAc	None	Waters, HSS T3, 150 x 2.1mm	2	Both	500	Each Batch
006	Fully identified (SANCO/12495/2011 p.74-80)	<2	0	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	>0.010 mg/kg	NO	Waters quattro premier	15	ACN	None	BEH C18	3	Both		
007	Fully identified (SANCO/12495/2011 p.74-80)	1	None		GC	MSD	Q	EI	+	YES	YES	0.01	NO	Agilent 6890/5973	10	ACN	DSPE	HP-5MS	2	Manual	183	Each Batch
008	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions +accurate mass	LC	MSD	Q-TRAP	ESI	+	YES	NO	0.01	NO	QTrap 5500	10	ACN	None	reversed phase	2.5	Auto	300	Each Batch
009	Tentative detected			2 transitions	GC	MSD	QQQ	EI	+	NO	NO		NO	Thermo TSQ Quantum	10	ACN	DSPE	TG-5MS 30m	2	Manual	80	Each Batch
	Confirmed with second method	13.8		2 transitions	LC		QQQ	ESI	+	YES	YES	0.01 (=RL)	NO	API5500	10	ACN	DSPE	Synergie Fusion RP80A	8	Manual	35	Scheduled MRM Method also used for quantification
010	Fully identified (SANCO/12495/2011 p.74-80)	0.8		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01	YES	HP5975C	10	ACN	DSPE	HP5MS	10	Both	aprox 1000	Always
011	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	NA	GC	MSD	Q	EI	+	NO	NO	NA	YES	5790 msd	10	ACN	PSA	HP-5MS	20	Auto	600 aprox	0-1

APPENDIX 3. Methods used by participants for detecting pesticides.

FENPROPIDIN																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
012	Fully identified (SANCO/12495/2011 p.74-80)	0.6		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010	NO	Waters Quattro Premier	10	Methanol	NO	C18	5	Both	211	Always
014	Fully identified (SANCO/12495/2011 p.74-80)	±0.5%			GC	MSD	Q	EI	+	YES	YES	0.010 mg/kg	YES	Agilent 7890-5975C	15	ACN	PSA	HP-5MS	10	Auto	211	Each Batch
015	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	2 transitions	GC		QQQ	EI	+	YES	YES	0.01	NO	7000B	10	ACN	PSA	DB-5MS UI	2	Both	440	
016	Confirmed with second method					MSD	QQQ	CI	+	YES	YES	0.005	NO	LC-MS/MS (Waters)	15	ACN	QuEChERS	C18	10	Both	370	Always
017	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	TOF	EI		YES	NO		YES	LECO PEGASUS	15	Acetone/PE/DCM	NO	DB-5	1	Both		
018	Tentative detected	RT locking		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01 - 0.05 mg/kg	YES	Agilent MSD 5975B	10	ACN	PSA	HP5	10	Both	> 600	Always
019	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	10	NO	Agilent 6490	15	ACN	PSA	C18	5	Auto		
020	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	QQQ	EI		YES	YES	0.01	NO	agilent	10	ACN	DSPE	HP5MS	1		200	Always
021	Fully identified (SANCO/12495/2011 p.74-80)	15		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	HPLC Agilent 1100, MS API 3000	10	ACN	DSPE (PSA, MgSO4)	C18 3µ 50x2mm	10	Both	550	Always
023	Confirmed with second method				LC		QQQ	ESI	+	YES	YES	0.01	NO	API 5500	10	ACN		Pursuit XRs Ultra	3	Both	250	Each Batch
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		2 x MRM	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	AB Sciex API 4000	10	ACN	PSA	Phenomenex C18	10	Both	500	Daily
025	Fully identified (SANCO/12495/2011 p.74-80)	0.15	0.12	isotope, 2 ions < 5ppm	LC	MSD	Orbitrap	ESI	+	NO	NO	0.01-0.1	YES	Exactive	10	ACN	None	C18 Atlantis T3	5	Both	570	Each Batch
	Fully identified (SANCO/12495/2011 p.74-80)	< 20	918	spectrum	GC	MSD	TOF	EI	+	YES	YES	0.01-0.1	YES	Leco Pegasus IV	10	ACN	DSPE (PSA)	RTX-CL pest	10	Auto	557	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

FENPROPIDIN																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
026	Fully identified (SANCO/12495/2011 p.74-80)	< 0.5 %	2.5		GC	MSD	ion trap	EI		YES	YES	0.01	YES	Trap 4000 - Varian	10	ACN	DSPE	5%	1 µL	Auto	450	Every Week
027	Fully identified (SANCO/12495/2011 p.74-80)	1	0.7	3 transitions	GC	MSD	QQQ	EI	+	YES	YES	0.01	NO	Varian 3800 GC + 320-MS	10	ACN	DSPE with PSA	VF-1ms	4	Both	302	Each Batch
028	Fully identified (SANCO/12495/2011 p.74-80)			274.2>147.1, 274.2>117.0	LC	MSD	QQQ	ESI	+	YES	YES	LOQ: 2 ppb	NO	LC: Agilent 1200, MS: API 4000 QTRAP	10	ACN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Auto		
029	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	NO	NO				10	ACN	NO	Xterra C18	20	Both		
030	Tentative detected				LC		QQQ	ESI	+	YES	YES	0.02	NO	Varian 320	15	ACN		C18	10	Auto	150	Each Batch
031	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.025-0.25 µg/ml	NO	UPLC-MSMS (Waters Aquity UPLC system, API 5000 Tripelquadropol, Sciex	10	EtOAc	Filter	HSS T3	2	Both	141	Each Batch
032	Tentative detected	0		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Waters Acquity TQD	10	Methanol	Filter	C18	6	Auto	150	Always
033	Fully identified (SANCO/12495/2011 p.74-80)	-2.0		min 3 ions	GC	MSD	Q	EI	+	YES	NO	0.05	YES	Agilent GC-MSD 7890A/5975c	10	ACN	PSA, C18	HP-5MS	1	Auto	927	
034	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC		QQQ	ESI	+	YES	YES		NO	Waters Quattro Premier	10	ACN	DSPE (PSA/MgSO4)	RP-18 endcapped	5	Both	186	Each Batch
036	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES					10	ACN			5	Auto	350	
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.010	NO	4000 QTRAP	10	ACN	DSPE	C18 hydro Phenomenex	40	Manual		Always
038	Tentative detected	None	None	2 transitions	LC	MSD	QQQ	ESI	+	NO	NO		NO	API 4000	10	ACN	DSPE	Waters-C18 2,1x50mmx1,7,µm	5	Auto	600	Every Week
039	Fully identified (SANCO/12495/2011 p.74-80)			274.3->147.1, 274.3->86.1	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6410B	10	ACN	DSPE	C18	2	Both	187	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

FENPROPIDIN																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
040	Tentative detected	16	3		LC		TOF	ESI	+	NO	NO		YES	Agilent 6230	10	ACN	None	Eclipse C18	3	Auto	aprox 500	
042	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES		NO	API 4000	10	ACN	QuEChERS	C18 Aqua	10	Both		
043	Tentative detected	0	0	scan	GC	MSD	IT	EI		YES	YES	0.010	YES	Varian Saturn 4000	10	ACN	DSPE	Varian VF	1	Auto	260	Daily
045	Tentative detected	1	1 unit		GC	MSD	Q	EI		YES	NO	0.01 ppm	YES	Agilent 5973	10	ACN	PSA	HP 5 MS UI	2	Auto	923	Each Batch
046	Fully identified (SANCO/12495/2011 p.74-80)	0			LC	MSD	QQQ	ESI	+	NO			NO	Waters Xevo	10	Acetone/PE/DCM	Na2SO4	C18	2	Both	286	Each Batch
047	Fully identified (SANCO/12495/2011 p.74-80)	1.2		2 transitions + ion ratio	LC	MSD	QQQ	ESI	+	YES	YES	5-1000 µg/kg	NO	Waters Quattro Premier XE	15	Acetone/PE/DCM	NO	Acquity BEH (100x2.1 mm i.d.)	5	Both	230	Each Batch
048	Confirmed with second method	3			GC	MSD	TOF	EI		YES	YES	0.005	YES	Pegasus IV	10	ACN	DSPE(PSA)	HP-5MS	5	Both	650	Weekly
049	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC		QQQ	ESI	+	YES	YES		NO	Waters XEVO TQ MS	10	ACN		BEH C18 1,7µm	3	Both	60	Daily
050	Fully identified (SANCO/12495/2011 p.74-80)	-0.038		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01-0.05	YES	Agilent 1200 LC+6460QQQ	10	ACN	DSPE	YMC Triart C18 2*100 mm 3µm	5	Both	354	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

FLUFENACET																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	Q	EI	+	YES	YES	0.01	YES	Agilent 5973 inert	5	ACN		HP-5 MSUI	5	Both		Daily
002	Confirmed with second method	3.1			GC	MSD	Q	EI	+	YES	YES	not validated	YES	AT 5975	10	ACN	SPE	HP5-MS	10	Auto	aprox 750	Always
003	Confirmed with second method	0.05	5	2 transitions	LC		Q-TOF	ESI	+	NO	NO		YES	Bruker Maxis	10	ACN	PSA	RSLC C18	1	Both	aprox 400	
004	Confirmed with second method	-1.2	2.2		LC	MSD	TOF	ESI	+	NO	NO	NA	YES	Bruker Maxis	10	EtOAc	None	Acclaim RSLC 120 C18, 2,1*100 mm	1	Both	1000	Each Batch
	Tentative detected	NA	NA		LC	MSD	QQQ	ESI	+	YES	NO	NA	NO	Agilent 6460, 1290HPLC	10	EtOAc	None	Waters, HSS T3, 150 x 2.1mm	2	Both	500	Each Batch
006	Tentative detected		<1	3 diagnostic ions	GC	MSD	Q	EI	+	YES	YES	>0.010 mg/kg	YES	Trace DSQ	15	ACN	PSA/C18	DB5MS	0.8	Both		
008	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions +accurate mass	LC	MSD	Q-TRAP	ESI	+	YES	NO	0.01	NO	QTrap 5500	10	ACN	None	reversed phase	2.5	Auto	300	Each Batch
010	Fully identified (SANCO/12495/2011 p.74-80)	3.2		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01	YES	HP5975C	10	ACN	DSPE	HP5MS	10	Both	aprox 1000	Always
011	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	NA	GC	MSD	Q	EI	+	NO	NO	NA	YES	5790 msd	10	ACN	PSA	HP-5MS	20	Auto	600 aprox	0-1
012	Fully identified (SANCO/12495/2011 p.74-80)	0.6		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010	NO	Waters Quattro Premier	10	Methanol	NO	C18	5	Both	211	Always
014	Fully identified (SANCO/12495/2011 p.74-80)	±2.5%		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010 mg/kg	YES	Waters-TQD	15	ACN	PSA	UPLC-BEH C18	10	Auto	331	Each Batch
015	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	2 transitions	LC		QQQ	ESI	+	YES	YES	0.01	NO	6460	10	ACN	PSA	C18-XB	2	Both	440	
016	Confirmed with second method					MSD	QQQ	CI	+	YES	YES	0.005	NO	LC-MS/MS (Waters)	15	ACN	QuEChERS	C18	10	Both	370	Always
018	Tentative detected	RT locking		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01 - 0.05 mg/kg	YES	Agilent MSD 5975B	10	ACN	PSA	HP5	10	Both	> 600	Always

APPENDIX 3. Methods used by participants for detecting pesticides.

FLUFENACET																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
019	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	10	NO	Agilent 6490	15	ACN	PSA	C18	5	Auto		
020	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	QQQ	EI		YES	YES	0.01	NO	agilent	10	ACN	DSPE	HP5MS	1		200	Always
021	Fully identified (SANCO/12495/2011 p.74-80)	15		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	HPLC Agilent 1100, MS API 3000	10	ACN	DSPE (PSA, MgSO4)	C18 3µ 50x2mm	10	Both	550	Always
023	Confirmed with second method				LC		QQQ	ESI	+	YES	YES	0.01	NO	API 5500	10	ACN		Pursuit XRs Ultra	3	Both	250	Each Batch
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		2 x MRM	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	AB Sciex API 4000	10	ACN	PSA	Phenomenex C18	10	Both	500	Daily
025	Fully identified (SANCO/12495/2011 p.74-80)	-0.21	-0.35	isotope, 2 ions < 5ppm	LC	MSD	Orbitrap	ESI	+	NO	NO	0.01-0.1	YES	Exactive	10	ACN	None	C18 Atlantis T3	5	Both	570	Each Batch
	Fully identified (SANCO/12495/2011 p.74-80)	< 20	905	spectrum	GC	MSD	TOF	EI	+	YES	YES	0.01-0.1	YES	Leco Pegasus IV	10	ACN	DSPE (PSA)	RTX-CL pest	10	Auto	557	Each Batch
027	Fully identified (SANCO/12495/2011 p.74-80)	1	0.7	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Varian Pro Star +320-MS	10	ACN	DSPE with PSA	Restek Ultra C18	10	Both	302	Each Batch
028	Tentative detected			364.1>152.2, 364.1>194.2	LC	MSD	QQQ	ESI	+	NO	NO	10-100 ppb	NO	LC: Agilent 1200, MS: API 4000 QTRAP	10	ACN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Auto	591	
029	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	NO	NO				10	ACN	NO	Xterra C18	20	Both		
030	Tentative detected				LC		QQQ	ESI	+	YES	YES	0.02	NO	Varian 320	15	ACN		C18	10	Auto	150	Each Batch
032	Tentative detected	0		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Waters Acquity TQD	10	Methanol	Filter	C18	6	Auto	150	Always
033	Fully identified (SANCO/12495/2011 p.74-80)	-1.3		min 3 ions	GC	MSD	Q	EI	+	YES	NO	0.05	YES	Agilent GC-MSD 7890A/5975c	10	ACN	PSA, C18	HP-5MS	1	Auto	927	
034	Confirmed with second method			full scan	GC	MSD	Q	EI	+	NO	NO		YES	Agilent 5973 MSD	10	ACN	DSPE (PSA/MgSO4)	HP-5MS	10	Both	950	

APPENDIX 3. Methods used by participants for detecting pesticides.

FLUFENACET																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
036	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES					10	ACN			5	Auto	350	
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.010	NO	4000 QTRAP	10	ACN	DSPE	C18 hydro Phenomenex	40	Manual		Always
038	Tentative detected	None	None	2 transitions	LC	MSD	QQQ	ESI	+	NO	NO		NO	API 4000	10	ACN	DSPE	Waters-C18 2,1x50mmx1,7,µm	5	Auto	600	Every Week
040	Confirmed with second method	0	4		LC		TOF	ESI	+	NO	NO		YES	Agilent 6230	10	ACN	None	Eclipse C18	3	Auto	aprox 500	
042	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES		NO	API 4000	10	ACN	QuEChERS	C18 Aqua	10	Both		
043	Tentative detected	0	0	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010	YES	Api 3200QT	10	ACN	DSPE	Phenomenex fusion	10	Auto	200	Daily
045	Tentative detected	1	1 unit		GC	MSD	Q	EI		YES	NO	0.01 ppm	YES	Agilent 5973	10	ACN	PSA	HP 5 MS UI	2	Auto	923	Each Batch
046	Fully identified (SANCO/12495/2011 p.74-80)	0			LC	MSD	QQQ	ESI	+	NO			NO	Waters Xevo	10	Acetone/PE/DCM	Na2SO4	C18	2	Both	286	Each Batch
047	Fully identified (SANCO/12495/2011 p.74-80)	0.6		2 transitions + ion ratio	LC	MSD	QQQ	ESI	+	YES	YES	5-1000 µg/kg	NO	Waters Quattro Premier XE	15	Acetone/PE/DCM	NO	Acquity BEH (100x2.1 mm i.d.)	5	Both	230	Each Batch
048	Confirmed with second method	4			GC	MSD	TOF	EI		YES	YES	0.01	YES	Pegasus IV	10	ACN	DSPE(PSA)	HP-5MS	5	Both	650	Weekly
049	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC		QQQ	EI	+	YES	YES		NO	Waters Quattro micro GC	10	ACN		DB-5MS	3	Both	150	Daily
050	Fully identified (SANCO/12495/2011 p.74-80)	-0.058		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01-0.05	YES	Agilent 1200 LC+6460QQQ	10	ACN	DSPE	YMC Triart C18 2*100 mm 3µm	5	Both	354	Daily
053	Tentative detected	12		3 SIM Ions	GC	MSD	Q	EI	+	NO	NO	>0.05 mg/kg	YES	Thermo DSQ	10	DCM	Diatomaceous earth		1	Both	400	Every 6 Months

APPENDIX 3. Methods used by participants for detecting pesticides.

FLUTOLANIL																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6490	5	ACN		RP-C18	10	Both		Daily
002	Confirmed with second method	2.8			GC	MSD	Q	EI	+	YES	YES	not validated	YES	AT 5975	10	ACN	SPE	HP5-MS	10	Auto	aprox 750	Always
003	Confirmed with second method	0.05	5	2 transitions	LC		Q-TOF	ESI	+	NO	NO		YES	Bruker Maxis	10	ACN	PSA	RSLC C18	1	Both	aprox 400	
004	Confirmed with second method	-1.2	1.1		LC	MSD	TOF	ESI	+	NO	NO	NA	YES	Bruker Maxis	10	EtOAc	None	Acclaim RSLC 120 C18, 2,1*100 mm	1	Both	1000	Each Batch
	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA		GC	MSD	QQQ	EI	+	YES	NO	NA	NO	Thermo TSQ Quantum XLS	10	EtOAc	None	Thermo TG-5SILMS, 30m*0.25mm* 0.25um	2	Auto	500	Each Batch
006	Tentative detected		<1	3 diagnostic ions	GC	MSD	Q	EI	+	YES	YES	>0.010 mg/kg	YES	Trace DSQ	15	ACN	PSA/C18	DB5MS	0.8	Both		
007	Fully identified (SANCO/12495/2011 p.74-80)	1	None		GC	MSD	Q	EI	+	YES	YES	0.01	NO	Agilent 6890/5973	10	ACN	DSPE	HP-5MS	2	Manual	183	Each Batch
008	Tentative detected		1.3		LC	MSD	Q-TOF	ESI	+	NO	NO		YES	5600 triple TOF	10	ACN	None	reversed phase	2	Auto	600	We do not have a standard
009	Tentative detected	35		2 transitions	LC		QQQ	ESI	+	YES	YES	0.01 (=RL)	NO	API4000	10	ACN	DSPE	Synergie Fusion RP80A	8	Manual	250	Scheduled MRM Method also used for quantification
010	Fully identified (SANCO/12495/2011 p.74-80)	2.5		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01	YES	HP5975C	10	ACN	DSPE	HP5MS	10	Both	aprox 1000	Always
011	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	NA	GC	MSD	Q	EI	+	NO	NO	NA	YES	5790 msd	10	ACN	PSA	HP-5MS	20	Auto	600 aprox	0-1

APPENDIX 3. Methods used by participants for detecting pesticides.

FLUTOLANIL																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
012	Fully identified (SANCO/12495/2011 p.74-80)	0.6		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010	NO	Waters Quattro Premier	10	Methanol	NO	C18	5	Both	211	Always
014	Fully identified (SANCO/12495/2011 p.74-80)	±0.5%			GC	MSD	Q	EI	+	YES	YES	0.010 mg/kg	YES	Agilent 7890-5975C	15	ACN	PSA	HP-5MS	10	Auto	211	Each Batch
015	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	2 transitions	LC		QQQ	ESI	+	YES	YES	0.01	NO	6460	10	ACN	PSA	C18-XB	2	Both	440	
016	Confirmed with second method					MSD	QQQ	CI	+	YES	YES	0.005	NO	LC-MS/MS (Waters)	15	ACN	QuEChERS	C18	10	Both	370	Always
018	Tentative detected	5	5	accurate mass	LC	Orbitrap	Orbitrap	ESI	+	YES	YES	0.01 - 0.05 mg/kg	YES	Thermo Exactive	10	ACN	PSA	C18	5	Both	120	Always
019	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	GC	MSD	QQQ	EI		YES	YES	10	NO	Agilent 6890	15	Acetone/PE/DCM	None	95%methyl 5% phenyl siloxane	2	Auto		
020	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	AbSciex	10	ACN	DSPE	C18	5	Auto	200	Always
021	Fully identified (SANCO/12495/2011 p.74-80)	15		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	HPLC Agilent 1100, MS API 3000	10	ACN	DSPE (PSA, MgSO4)	C18 3µ 50x2mm	10	Both	550	Always
023	Confirmed with second method				GC	MSD	TOF			YES	YES	0.01	YES	Leco Pegasus IV	10	ACN	DSPE	Phenomenenx ZB-5MS	3	Both	750	Each Batch
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		2 x MRM	LC	MSD	QQQ	ESI	-	YES	YES	0.01	NO	AB Sciex API 4000	10	ACN	PSA	Phenomenex C18	10	Both	500	Daily
025	Fully identified (SANCO/12495/2011 p.74-80)	-0.17	-0.71	isotope, 2 ions < 5ppm	LC	MSD	Orbitrap	ESI	+	NO	NO	0.01-0.1	YES	Exactive	10	ACN	None	C18 Atlantis T3	5	Both	570	Each Batch
	Fully identified (SANCO/12495/2011 p.74-80)	< 20	933	spectrum	GC	MSD	TOF	EI	+	YES	YES	0.01-0.1	YES	Leco Pegasus IV	10	ACN	DSPE (PSA)	RTX-CL pest	10	Auto	557	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

FLUTOLANIL																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
026	Fully identified (SANCO/12495/2011 p.74-80)	< 0.5 %	2.5		GC	MSD	ion trap	EI		YES	YES	0.01	YES	Trap 4000 - Varian	10	ACN	DSPE	5%	1 µL	Auto	450	Every Week
028	Tentative detected			Match with NIST Library (Match 828, range 0-1000)	GC	MSD	TOF	EI		NO	NO	10-100 ppb	YES	GCxGC-TOF MS Pegasus 4D	10	ACN	QuEChERS without PSA	DB5-MS (30 m x 0,25 mm x 0,25 µm) in the 1st Dimension, BPX-50 (2 m x 0,1 mm x 0,1 µm) in the 2nd Dimension	10	Auto	500	
029	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	NO	NO				10	ACN	NO	Xterra C18	20	Both		
031	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MS/MS	QQQ	EI	+	YES	YES	0.025-0.25 µg/ml	NO	Waters Quattro Micro GC	10	EtOAc	Filter	Rxi-5sil MS/integra-guard Restek	10	Both	141	Each Batch
032	Tentative detected	0		2 transitions	GC	MSD	QQQ	EI	+	YES	YES	0.01	NO	Thermo TSQ Quantum XLS Ultra	10	ACN	PSA	Capillary	1	Both	150	Always
033	Fully identified (SANCO/12495/2011 p.74-80)	-1.9		min 3 ions	GC	MSD	Q	EI	+	YES	NO	0.01	YES	Agilent GC-MSD 7890A/5975c	10	ACN	PSA, C18	HP-5MS	1	Auto	927	
034	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC		QQQ	ESI	+	YES	YES		NO	Waters Quattro Premier	10	ACN	DSPE (PSA/MgSO4)	RP-18 endcapped	5	Both	186	Each Batch
036	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES					10	ACN			5	Auto	350	
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.010	NO	4000 QTRAP	10	ACN	DSPE	C18 hydro Phenomenex	40	Manual		Always
038	Tentative detected	None	None	2 transitions	LC	MSD	QQQ	ESI	+	NO	NO		NO	API 4000	10	ACN	DSPE	Waters-C18 2,1x50mmx1,7,µm	5	Auto	600	Every Week
039	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	Q	EI	+	YES	YES	0.01	YES	HP5973 MSD	10	ACN	DSPE	HP-5MSI	20	Both	113	Each Batch
040	Confirmed with second method				GC	MSD	Q	EI		NO	NO		YES	Agilent 5973	30	EtOAc	HPGPC	DB-5MS	2	Auto	aprox 500	

APPENDIX 3. Methods used by participants for detecting pesticides.

FLUTOLANIL																				
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
041	Fully identified (SANCO/12495/2011 p.74-80)	0.5%		2 transitions	GC	MSD	QQQ	EI		YES	YES	0.005 mg/kg	NO	VARIAN CP-3800 MS-320	10	ACN	PSA	VF-5MS	1	Auto	210	
042	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MSD	QQQ	EI		YES	YES		NO	TSQ Quantum	10	ACN	QuEChERS	VF 5 MS	3	Both		
043	Tentative detected	0	0	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010	YES	Api 3200QT	10	ACN	DSPE	Phenomenex fusion	10	Auto	200	Daily
045	Tentative detected	1	1 unit		GC	MSD	Q	EI		YES	NO	0.01 ppm	YES	Agilent 5973	10	ACN	PSA	HP 5 MS UI	2	Auto	923	Each Batch
046	Fully identified (SANCO/12495/2011 p.74-80)	0			LC	MSD	QQQ	ESI	+	NO			NO	Waters Xevo	10	Acetone/PE/DCM	Na2SO4	C18	2	Both	286	Each Batch
047	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions + ion ratio	LC	MSD	QQQ	ESI	+	YES	YES	5-1000 µg/kg	NO	Waters Quattro Premier XE	15	Acetone/PE/DCM	NO	Acquity BEH (100x2.1 mm i.d.)	5	Both	230	Each Batch
048	Confirmed with second method	6			GC	MSD	TOF	EI		YES	YES	0.005	YES	Pegasus IV	10	ACN	DSPE(PSA)	HP-5MS	5	Both	650	Weekly
049	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC		QQQ	EI	+	YES	YES		NO	Waters Quattro micro GC	10	ACN		DB-5MS	3	Both	150	Daily
051	Tentative detected		0.5	qualifiers	GC	MSD	IT	EI		NO	NO		YES	varian GCMS 4000	10	ACN	PSA	varian factor four	5	Both		
053	Tentative detected	9		3 SIM Ions	GC	MSD	Q	EI	+	NO	NO	>0.05 mg/kg	YES	Thermo DSQ	10	DCM	Diatomaceous earth		1	Both	400	Every 6 Months

APPENDIX 3. Methods used by participants for detecting pesticides.

IMAZAPYR																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
003	Confirmed with second method	0.05	5	2 transitions	LC		Q-TOF	ESI	-	NO	NO		YES	Bruker Maxis	10	ACN	PSA	RSLC C18	1	Both	aprox 400	
007	Fully identified (SANCO/12495/2011 p.74-80)	1	None		LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6410	10	ACN	DSPE	Eclipse XDB-C18	4	Manual	139	Each Batch
008	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions +accurate mass	LC	MSD	Q-TRAP	ESI	+	YES	NO	0.01	NO	QTrap 5500	10	ACN	None	reversed phase	2.5	Auto	300	Each Batch
010	Tentative detected			2 transitions	LC	MSD	QQQ	ESI	+	NO	NO		NO	ABI4000	10	ACN	NO	C18	55	Both	aprox 580	Always
021	Fully identified (SANCO/12495/2011 p.74-80)	15		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	HPLC Agilent 1100, MS API 3000	10	ACN	DSPE (PSA, MgSO4)	C18 3µ 50x2mm	10	Both	550	Always
022	Tentative detected	0.5	0.05		LC	MSD	TOF	ESI	+	YES	YES		YES	LCT PREMIER XE	15	MINI LUKE	NO	C-18	7	Auto	343	Every Day
023	Confirmed with second method				LC		IT	ESI	-	YES	YES	0.01	NO		10	ACN				Both		Each Batch
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		2 x MRM	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	AB Sciex API 4000	10	ACN	PSA	Phenomenex C18	10	Both	500	Daily
028	Fully identified (SANCO/12495/2011 p.74-80)			262.2>217.0, 262.2>149.0	LC	MSD	QQQ	ESI	+	YES	YES	LOQ: 2 ppb	NO	LC: Agilent 1200, MS: API 4000 QTRAP	10	ACN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Auto		
029	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	NO	NO				10	ACN	NO	Xterra C18	20	Both		
030	Tentative detected				LC		QQQ	ESI	+	YES	YES	0.02	NO	Varian 320	15	ACN		C18	10	Auto	150	Each Batch
034	Tentative detected			5 transitions	LC		QQQ	ESI	+	NO	NO		NO	Waters Quattro Premier	10	ACN	DSPE (PSA/MgSO4)	RP-18 endcapped	5	Both	500	
038	Tentative detected	None	None	2 transitions	LC	MSD	QQQ	ESI	+	NO	NO		NO	API 4000	10	ACN	DSPE	Waters-C18 2,1x50mmx1,7,µm	5	Auto	600	Every Week
040	Tentative detected	2	0		LC		TOF	ESI	+	NO	NO		YES	Agilent 6230	10	ACN	None	Eclipse C18	3	Auto	aprox 500	

APPENDIX 3. Methods used by participants for detecting pesticides.

IMAZAPYR																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
041	Fully identified (SANCO/12495/2011 p.74-80)	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.005 mg/kg	NO	AGILENT G-6410A	10	ACN	PSA	ZORBAX-ECLIPSE XDB-C8	20	Auto	143	
043	Tentative detected	0	0	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010	YES	Api 3200QT	10	ACN	DSPE	Phenomenex fusion	10	Auto	200	Daily
046	Fully identified (SANCO/12495/2011 p.74-80)	0			LC	MSD	QQQ	ESI	+	NO			NO	Waters Xevo	10	Acetone/PE/DCM	Na2SO4	C18	2	Both	286	Each Batch
048	Fully identified (SANCO/12495/2011 p.74-80)	1.1			LC	MSD	QQQ	ESI	+	YES	YES	0.005	NO	Agilent 6410	10	ACN	DSPE(PSA)	Luna C18	5	Both	350	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

IOXYNIL																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	-	YES	YES	0.01	NO	Agilent 6490	5	ACN		RP-C18	10	Both		Daily
003	Confirmed with second method	0.05	5	2 transitions	LC		Q-TOF	ESI	+	NO	NO		YES	Bruker Maxis	10	ACN	PSA	RSLC C18	1	Both	aprox 400	
004	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA		LC	MSD	QQQ	ESI	-	YES	NO	NA	NO	Agilent 6460, 1290HPLC	10	EtOAc	None	Waters, HSS T3, 150 x 2.1mm	2	Both	500	Each Batch
008	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions +accurate mass	LC	MSD	Q-TRAP	ESI	-	YES	NO	0.01	NO	QTrap 5500	10	ACN	None	reversed phase	3	Auto	300	Each Batch
009	Tentative detected			2 transitions	LC		QQQ	ESI	-	NO	NO		NO	API4000	10	ACN	DSPE	Synergie Fusion RP80A	8	Manual	40	Scheduled MRM Method also used for quantification
010	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	-	YES	YES	0.02	NO	ABI4000	10	ACN	NO	C18	55	Both	aprox 580	Always
012	Fully identified (SANCO/12495/2011 p.74-80)	0.6		2 transitions	LC	MSD	QQQ	ESI	-	YES	YES	0.010	NO	Waters Quattro Premier	10	Methanol	NO	C18	5	Both	211	Always
014	Fully identified (SANCO/12495/2011 p.74-80)	±2.5%		2 transitions	LC	MSD	QQQ	ESI	-	YES	YES	0.010 mg/kg	YES	Waters-TQD	15	ACN	PSA	UPLC-BEH C18	10	Auto	331	Each Batch
017	Tentative detected				GC	MSD	TOF	EI		NO	NO		YES	LECO PEGASUS	15	Acetone/PE/DCM	NO	DB-5	1	Both		
018	Tentative detected	RT locking		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01 - 0.05 mg/kg	YES	Agilent MSD 5975B	10	ACN	PSA	HP5	10	Both	> 600	Always
019	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	LC	MSD	QQQ	ESI	-	YES	YES	10	NO	Agilent 6490	15	ACN	PSA	C18	5	Auto		
021	Fully identified (SANCO/12495/2011 p.74-80)	15		2 transitions	LC	MSD	QQQ	ESI	-	YES	YES	0.01	NO	HPLC Agilent 1100, MS API 3000	10	ACN	DSPE (PSA, MgSO4)	C18 3µ 50x2mm	10	Both	550	Always
023	Confirmed with second method				LC		IT	ESI	-	YES	YES	0.01	NO		10	ACN				Both		Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

IOXYNIL																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		2 x MRM	LC	MSD	QQQ	ESI	-	YES	YES	0.01	NO	AB Sciex API 4000	10	ACN	PSA	Phenomenex C18	10	Both	500	Daily
025	Confirmed with second method	-0.02	0.9	isotope	LC	MSD	Orbitrap	ESI	-	NO	NO	0.01-0.1	YES	Exactive	10	ACN	None	C18 Atlantis T3	5	Both	570	Each Batch
	Fully identified (SANCO/12495/2011 p.74-80)	< 20	910	spectrum	GC	MSD	TOF	EI	+	YES	YES	0.01-0.1	YES	Leco Pegasus IV	10	ACN	DSPE (PSA)	RTX-CL pest	10	Auto	557	Each Batch
028	Fully identified (SANCO/12495/2011 p.74-80)			369.6>126.9, 369.6>214.8	LC	MSD	QQQ	ESI	-	YES	YES	LOQ: 2 ppb	NO	LC: Agilent 1200, MS: API 4000 QTRAP	10	ACN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Auto		
030	Tentative detected				LC		QQQ	ESI	+	YES	YES	0.02	NO	Varian 320	15	ACN		C18	10	Auto	150	Each Batch
031	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	-	YES	YES	0.005-0.04 µg/ml	NO	UPLC-MSMS (Waters Aquity UPLC system, API 5000 Tripelquadropol, Sciex	5	EtOAc	Filter	HSS T3	2	Both	250	Each Batch
034	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC		QQQ	ESI	-	YES	YES		NO	Waters Quattro Premier	10	ACN	DSPE (PSA/MgSO4)	RP-18 endcapped	5	Both	186	Each Batch
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	-	YES	YES	0.010	NO	4000 QTRAP	10	ACN	DSPE	C18 hydro Phenomenex	40	Manual		Always
040	Confirmed with second method	9	3		LC		TOF	ESI	-	NO	NO		YES	Agilent 6230	10	ACN	None	Eclipse C18	3	Auto	aprox 500	
041	Fully identified (SANCO/12495/2011 p.74-80)	2.5%		2 transitions	LC	MSD	QQQ	ESI	-	YES	YES	0.005 mg/kg	NO	AGILENT G-6410A	10	ACN	PSA	ZORBAX-ECLIPSE XDB-C8	80	Auto	143	
042	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	-	YES	YES		NO	API 4000	10	ACN	QuEChERS	C18 Aqua	10	Both		
045	Tentative detected	1	1 unit		GC	MSD	Q	EI		YES	NO	0.01 ppm	YES	Agilent 5973	10	ACN	PSA	HP 5 MS UI	2	Auto	923	Each Batch
047	Fully identified (SANCO/12495/2011 p.74-80)	3.6		2 transitions + ion ratio	LC	MSD	QQQ	ESI	-	NO	NO	5-1000 µg/kg	NO	Waters Quattro Premier XE	15	Acetone/PE/DCM	NO	Alltima C18 (150x3 mm i.d.)	5	Both	27	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

IOXYNIL																				
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
048	Fully identified (SANCO/12495/2011 p.74-80)	0.8			LC	MSD	QQQ	ESI	-	YES	YES	0.005	NO	Agilent 6410	10	ACN	DSPE(PSA)	Luna C18	5	Both	350	Daily
049	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC		QQQ	ESI	+	YES	YES		NO	Waters XEVO TQ MS	10	ACN		BEH C18 1,7µm	3	Both	60	Daily
050	Fully identified (SANCO/12495/2011 p.74-80)	0.017		2 transitions	LC	MSD	QQQ	ESI	-	YES	YES	0.01-0.05	YES	Agilent 1200 LC+6460QQQ	10	ACN	DSPE	YMC Triart C18 2*100 mm 3µm	5	Both	7	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

ISOPROTURON																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6490	5	ACN		RP-C18	10	Both		Daily
002	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC		QQQ	ESI	+	YES	YES		NO	API4000	10	ACN	None	C18	5	Manual		
003	Confirmed with second method	0.05	5	2 transitions	LC		Q-TOF	ESI	+	NO	NO		YES	Bruker Maxis	10	ACN	PSA	RSLC C18	1	Both	aprox 400	
004	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA		LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6460, 1290HPLC	10	EtOAc	None	Waters, HSS T3, 150 x 2.1mm	2	Both	500	Each Batch
	Confirmed with second method	-1.2	2.2		LC	MSD	TOF	ESI	+	NO	NO	NA	YES	Bruker Maxis	10	EtOAc	None	Acclaim RSLC 120 C18, 2,1*100 mm	1	Both	1000	Each Batch
006	Fully identified (SANCO/12495/2011 p.74-80)	<2	0	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	>0.010 mg/kg	NO	Waters quattro premier	15	ACN	None	BEH C18	3	Both		
007	Fully identified (SANCO/12495/2011 p.74-80)	1	None		LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6410	10	ACN	DSPE	Eclipse XDB-C18	4	Manual	139	Each Batch
008	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions +accurate mass	LC	MSD	Q-TRAP	ESI	+	YES	NO	0.01	NO	QTrap 5500	10	ACN	None	reversed phase	2.5	Auto	300	Each Batch
009	Tentative detected	28.8		2 transitions	LC		QQQ	ESI	+	YES	YES	0.01 (=RL)	NO	API4000	10	ACN	DSPE	Synergie Fusion RP80A	8	Manual	250	Scheduled MRM Method also used for quantification
010	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	ABI4000	10	ACN	NO	C18	55	Both	aprox 580	Always
011	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	NA	LC	MSD	Q-TRAP	ESI	+	YES	YES	0.01	NO	3200qtrap	10	ACN	PSA	T-3	5	Manual	170	1
012	Fully identified (SANCO/12495/2011 p.74-80)	0.6		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010	NO	Waters Quattro Premier	10	Methanol	NO	C18	5	Both	211	Always

APPENDIX 3. Methods used by participants for detecting pesticides.

ISOPROTURON																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
014	Fully identified (SANCO/12495/2011 p.74-80)	±2.5%		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010 mg/kg	YES	Waters-TQD	15	ACN	PSA	UPLC-BEH C18	10	Auto	331	Each Batch
015	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	2 transitions	LC		QQQ	ESI	+	YES	YES	0.01	NO	6460	10	ACN	PSA	C18-XB	2	Both	440	
016	Confirmed with second method					MSD	QQQ	CI	+	YES	YES	0.005	NO	LC-MS/MS (Waters)	15	ACN	QuEChERS	C18	10	Both	370	Always
017	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	TOF	EI		YES	NO		YES	LECO PEGASUS	15	Acetone/PE/DCM	NO	DB-5	1	Both		
018	Tentative detected	5	5	accurate mass	LC	Orbitrap	Orbitrap	ESI	+	YES	YES	0.01 - 0.05 mg/kg	YES	Thermo Exactive	10	ACN	PSA	C18	5	Both	120	Always
019	Fully identified (SANCO/12495/2011 p.74-80)	3			LC	MSD	QQQ	ESI	+	YES	YES		NO	Agilent 6490	15	ACN	PSA		5	Auto		
020	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	AbSciex	10	ACN	DSPE	C18	5	Auto	200	Always
021	Fully identified (SANCO/12495/2011 p.74-80)	15		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	HPLC Agilent 1100, MS API 3000	10	ACN	DSPE (PSA, MgSO4)	C18 3µ 50x2mm	10	Both	550	Always
022	Tentative detected	0.5	0.05		LC	MSD	TOF	ESI	+	YES	YES		YES	LCT PREMIER XE	15	MINI LUKE	NO	C-18	7	Auto	343	Every Day
023	Confirmed with second method				LC		QQQ	ESI	+	YES	YES	0.01	NO	API 5500	10	ACN		Pursuit XRs Ultra	3	Both	250	Each Batch
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		2 x MRM	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	AB Sciex API 4000	10	ACN	PSA	Phenomenex C18	10	Both	500	Daily
025	Fully identified (SANCO/12495/2011 p.74-80)	-0.2	0.64	isotope, 2 ions < 5ppm	LC	MSD	Orbitrap	ESI	+	NO	NO	0.01-0.1	YES	Exactive	10	ACN	None	C18 Atlantis T3	5	Both	570	Each Batch
026	Fully identified (SANCO/12495/2011 p.74-80)	< 2.5 %	2.5	transition 1 and 2	LC	MSD	Q-TRAP	ESI	+	YES	YES	0.01	YES	3200 Qtrap	10	ACN	DSPE	C18	50	Auto	521	Every Week

APPENDIX 3. Methods used by participants for detecting pesticides.

ISOPROTURON																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
027	Fully identified (SANCO/12495/2011 p.74-80)	1	0.7	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Varian Pro Star +320-MS	10	ACN	DSPE with PSA	Restek Ultra C18	10	Both	302	Each Batch
028	Fully identified (SANCO/12495/2011 p.74-80)			207.2>72.1, 207.2>134.2	LC	MSD	QQQ	ESI	+	YES	YES	LOQ: 2 ppb	NO	LC: Agilent 1200, MS: API 4000 QTRAP	10	ACN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Auto		
029	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO		10	ACN	NO	Xterra C18	20	Both		
030	Tentative detected				LC		QQQ	ESI	+	YES	YES	0.02	NO	Varian 320	15	ACN		C18	10	Auto	150	Each Batch
031	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.005-0.125 µg/ml	NO	UPLC-MSMS (Waters Aquity UPLC system, API 5000 Tripelquadropol, Sciex	10	EtOAc	Filter	HSS T3	2	Both	373	Each Batch
032	Tentative detected	0		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Waters Acquity TQD	10	Methanol	Filter	C18	6	Auto	150	Always
034	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC		QQQ	ESI	+	YES	YES		NO	Waters Quattro Premier	10	ACN	DSPE (PSA/MgSO4)	RP-18 endcapped	5	Both	186	Each Batch
036	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES					10	ACN			5	Auto	350	
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.010	NO	4000 QTRAP	10	ACN	DSPE	C18 hydro Phenomenex	40	Manual		Always
038	Fully identified (SANCO/12495/2011 p.74-80)	None	None	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES		NO	API 4000	10	ACN	DSPE	Waters-C18 2,1x50mmx1,7,µm	5	Auto	600	Every Week
039	Fully identified (SANCO/12495/2011 p.74-80)			207.1->72.0, 207.1->165.1	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6410B	10	ACN	DSPE	C18	2	Both	187	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

ISOPROTURON																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
040	Confirmed with second method	3	2		LC		TOF	ESI	+	NO	NO		YES	Agilent 6230	10	ACN	None	Eclipse C18	3	Auto	aprox 500	
041	Fully identified (SANCO/12495/2011 p.74-80)	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.005 mg/kg	NO	AGILENT G-6410A	10	ACN	PSA	ZORBAX-ECLIPSE XDB-C8	20	Auto	143	
042	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES		NO	API 4000	10	ACN	QuEChERS	C18 Aqua	10	Both		
043	Tentative detected	0	0	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010	YES	Api 3200QT	10	ACN	DSPE	Phenomenex fusion	10	Auto	200	Daily
044	Tentative detected				LC		QQQ	EI	+	YES				Agilent 6410	10	ACN		Zorbax SB C-18	5	Auto		
046	Fully identified (SANCO/12495/2011 p.74-80)	0			LC	MSD	QQQ	ESI	+	NO			NO	Waters Xevo	10	Acetone/PE/DCM	Na2SO4	C18	2	Both	286	Each Batch
047	Tentative detected	2		Spectrum match (reversed)	GC	MSD	IT	EI	+	YES	YES	20-10.000 µg/kg	YES	Varian Satum 2000	15	Acetone/PE/DCM	NO	VF-5-ms	5	Both	550	Quarterly
048	Fully identified (SANCO/12495/2011 p.74-80)	0.9			LC	MSD	QQQ	ESI	+	YES	YES	0.005	NO	Agilent 6410	10	ACN	DSPE(PSA)	Luna C18	5	Both	350	Daily
049	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC		QQQ	ESI	+	YES	YES		NO	Waters XEVO TQ MS	10	ACN		BEH C18 1,7µm	3	Both	60	Daily
050	Fully identified (SANCO/12495/2011 p.74-80)	-0.055		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01-0.05	YES	Agilent 1200 LC+6460QQQ	10	ACN	DSPE	YMC Triart C18 2*100 mm 3µm	5	Both	354	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

METAZACHLOR																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	Q	EI	+	YES	YES	0.01	YES	Agilent 5973 inert	5	ACN		HP-5 MSUI	5	Both		Daily
002	Confirmed with second method	3.1			GC	MSD	Q	EI	+	YES	YES	not validated	YES	AT 5975	10	ACN	SPE	HP5-MS	10	Auto	aprox 750	Always
004	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA		GC	MSD	QQQ	EI	+	YES	YES	0.01	NO	Thermo TSQ Quantum XLS	10	EtOAc	None	Thermo TG-5SILMS, 30m*0.25mm* 0.25um	2	Auto	500	Each Batch
	Confirmed with second method	-0.6	2.4		LC	MSD	TOF	ESI	+	NO	NO	NA	YES	Bruker Maxis	10	EtOAc	None	Acclaim RSLC 120 C18, 2,1*100 mm	1	Both	1000	Each Batch
006	Fully identified (SANCO/12495/2011 p.74-80)	<2	<1	3 diagnostic ions	GC	MSD	Q	EI	+	YES	YES	>0.010 mg/kg	YES	Trace DSQ	15	ACN	PSA/C18	DB5MS	0.8	Both		
007	Fully identified (SANCO/12495/2011 p.74-80)	1	None		GC	MSD	Q	EI	+	YES	YES	0.01	NO	Agilent 6890/5973	10	ACN	DSPE	HP-5MS	2	Manual	183	Each Batch
008	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions +accurate mass	LC	MSD	Q-TRAP	ESI	+	YES	NO	0.01	NO	QTrap 5500	10	ACN	None	reversed phase	2.5	Auto	300	Each Batch
009	Tentative detected	36.6		2 transitions	LC		QQQ	ESI	+	YES	YES	0.01 (=RL)	NO	API4000	10	ACN	DSPE	Synergie Fusion RP80A	8	Manual	250	Scheduled MRM Method also used for quantification
010	Fully identified (SANCO/12495/2011 p.74-80)	2.3		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01	YES	HP5975C	10	ACN	DSPE	HP5MS	10	Both	aprox 1000	Always
011	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	NA	GC	MSD	Q	EI	+	NO	NO	NA	YES	5790 msd	10	ACN	PSA	HP-5MS	20	Auto	600 aprox	0-1
012	Fully identified (SANCO/12495/2011 p.74-80)	0.6		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010	NO	Waters Quattro Premier	10	Methanol	NO	C18	5	Both	211	Always

APPENDIX 3. Methods used by participants for detecting pesticides.

METAZACHLOR																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
014	Fully identified (SANCO/12495/2011 p.74-80)	±0.5%			GC	MSD	Q	EI	+	YES	YES	0.010 mg/kg	YES	Agilent 7890-5975C	15	ACN	PSA	HP-5MS	10	Auto	211	Each Batch
015	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	2 transitions	GC		QQQ	EI	+	YES	YES	0.01	NO	7000B	10	ACN	PSA	DB-5MS UI	2	Both	440	
016	Confirmed with second method					MSD	QQQ	CI	+	YES	YES	0.005	NO	LC-MS/MS (Waters)	15	ACN	QuEChERS	C18	10	Both	370	Always
017	Tentative detected				GC	MSD	TOF	EI		NO	NO		YES	LECO PEGASUS	15	Acetone/PE/DCM	NO	DB-5	1	Both		
018	Tentative detected	RT locking		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01 - 0.05 mg/kg	YES	Agilent MSD 5975B	10	ACN	PSA	HP5	10	Both	> 600	Always
019	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	GC	MSD	QQQ	EI		YES	YES	10	NO	Agilent 6890	15	Acetone/PE/DCM	None	95%methyl 5% phenyl siloxane	2	Auto		
020	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	QQQ	EI		YES	YES	0.01	NO	agilent	10	ACN	DSPE	HP5MS	1		200	Always
021	Fully identified (SANCO/12495/2011 p.74-80)	15		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	HPLC Agilent 1100, MS API 3000	10	ACN	DSPE (PSA, MgSO4)	C18 3µ 50x2mm	10	Both	550	Always
022	Tentative detected	0.5	0.05		LC	MSD	TOF	ESI	+	YES	YES		YES	LCT PREMIER XE	15	MINI LUKE	NO	C-18	7	Auto	343	Every Day
023	Confirmed with second method				LC		QQQ	ESI	+	YES	YES	0.01	NO	API 5500	10	ACN		Pursuit XRs Ultra	3	Both	250	Each Batch
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		3x SIM masses	GC	MSD	Q	EI	+	YES	YES	0.01	YES	Agilent 5975	10	ACN	PSA	GsBP-5MS	5	Both	500	Daily
025	Confirmed with second method	-0.19	-0.43	isotope	LC	MSD	Orbitrap	ESI	+	NO	NO	0.01-0.1	YES	Exactive	10	ACN	None	C18 Atlantis T3	5	Both	570	Each Batch
	Fully identified (SANCO/12495/2011 p.74-80)	< 20	840	spectrum	GC	MSD	TOF	EI	+	YES	YES	0.01-0.1	YES	Leco Pegasus IV	10	ACN	DSPE (PSA)	RTX-CL pest	10	Auto	557	Each Batch
026	Fully identified (SANCO/12495/2011 p.74-80)	< 0.5 %	2.5		GC	MSD	ion trap	EI		YES	YES	0.01	YES	Trap 4000 - Varian	10	ACN	DSPE	5%	1 µL	Auto	450	Every Week

APPENDIX 3. Methods used by participants for detecting pesticides.

METAZACHLOR																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
027	Fully identified (SANCO/12495/2011 p.74-80)	1	0.7	4 transitions	GC	MSD	QQQ	EI	+	YES	YES	0.01	NO	Varian 3800 GC + 320-MS	10	ACN	DSPE with PSA	VF-1ms	4	Both	302	Each Batch
028	Confirmed with second method			278.2>143.2, 278.2>210.2	LC	MSD	QQQ	ESI	+	NO	NO	10-100 ppb	NO	LC: Agilent 1200, MS: API 4000 QTRAP	10	ACN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Auto	591	
029	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	NO	NO				10	ACN	NO	Xterra C18	20	Both		
030	Tentative detected				LC		QQQ	ESI	+	YES	YES	0.02	YES	Varian 320	15	ACN		C18	10	Auto	150	Each Batch
031	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MS/MS	QQQ	EI	+	YES	YES	0.005-0.4 µg/ml	NO	Waters Quattro Micro GC	10	EtOAc	Filter	Rxi-5sil MS/integra-guard Restek	10	Both	373	Each Batch
033	Fully identified (SANCO/12495/2011 p.74-80)	-2.1		min 3 ions	GC	MSD	Q	EI	+	YES	NO	0.05	YES	Agilent GC-MSD 7890A/5975c	10	ACN	PSA, C18	HP-5MS	1	Auto	927	
034	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC		QQQ	ESI	+	YES	YES		NO	Waters Quattro Premier	10	ACN	DSPE (PSA/MgSO4)	RP-18 endcapped	5	Both	186	Each Batch
035	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	Q	EI		NO	NO		YES	Agilent 6890N	10	ACN	DSPE	DB-5	2	Auto		
036	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES					10	ACN			5	Auto	350	
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.010	NO	4000 QTRAP	10	ACN	DSPE	C18 hydro Phenomenex	40	Manual		Always
038	Fully identified (SANCO/12495/2011 p.74-80)	None	None	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES		NO	API 4000	10	ACN	DSPE	Waters-C18 2,1x50mmx1,7,µm	5	Auto	600	Every Week
039	Tentative detected				GC	MSD	Q	EI	+	NO	NO		YES	HP5973 MSD	10	ACN	DSPE	HP-5MSI	20	Both	624	No standard used
040	Confirmed with second method	7	3		LC		TOF	ESI	+	NO	NO		YES	Agilent 6230	10	ACN	None	Eclipse C18	3	Auto	aprox 500	

APPENDIX 3. Methods used by participants for detecting pesticides.

METAZACHLOR																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
041	Fully identified (SANCO/12495/2011 p.74-80)	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.005 mg/kg	NO	AGILENT G-6410A	10	ACN	PSA	ZORBAX-ECLIPSE XDB-C8	20	Auto	143	
042	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MSD	QQQ	EI		YES	YES		NO	TSQ Quantum	10	ACN	QuEChERS	VF 5 MS	3	Both		
043	Tentative detected	0	0	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010	YES	Api 3200QT	10	ACN	DSPE	Phenomenex fusion	10	Auto	200	Daily
045	Tentative detected	1	1 unit		GC	MSD	Q	EI		YES	NO	0.01 ppm	YES	Agilent 5973	10	ACN	PSA	HP 5 MS UI	2	Auto	923	Each Batch
046	Fully identified (SANCO/12495/2011 p.74-80)	0			LC	MSD	QQQ	ESI	+	NO			NO	Waters Xevo	10	Acetone/PE/DCM	Na2SO4	C18	2	Both	286	Each Batch
047	Fully identified (SANCO/12495/2011 p.74-80)	0.2		Spectrum match (reversed)	GC	MSD	IT	EI	+	YES	YES	20-10.000 µg/kg	YES	Varian Saturn 2000	15	Acetone/PE/DCM	NO	VF-5-ms	5	Both	550	Quarterly
048	Confirmed with second method	4			GC	MSD	TOF	EI		YES	YES	0.005	YES	Pegasus IV	10	ACN	DSPE(PSA)	HP-5MS	5	Both	650	Weekly
049	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC		QQQ	EI	+	YES	YES		NO	Waters Quattro micro GC	10	ACN		DB-5MS	3	Both	150	Daily
053	Tentative detected	9		3 SIM Ions	GC	MSD	Q	EI	+	NO	NO	>0.05 mg/kg	YES	Thermo DSQ	10	DCM	Diatomaceous earth		1	Both	400	Every 6 Months

APPENDIX 3. Methods used by participants for detecting pesticides.

NAPROPAMIDE																				
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	Q	EI	+	YES	YES	0.01	YES	Agilent 5973 inert	5	ACN		HP-5 MSUI	5	Both		Daily
002	Confirmed with second method	4.2			GC	MSD	Q	EI	+	YES	YES	not validated	YES	AT 5975	10	ACN	SPE	HP5-MS	10	Auto	aprox 750	Always
003	Confirmed with second method	0.05	5	2 transitions	LC		Q-TOF	ESI	+	NO	NO		YES	Bruker Maxis	10	ACN	PSA	RSLC C18	1	Both	aprox 400	
004	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA		LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6460, 1290HPLC	10	EtOAc	None	Waters, HSS T3, 150 x 2.1mm	2	Both	500	Each Batch
	Confirmed with second method	-0.6	0.9		LC	MSD	TOF	ESI	+	NO	NO	NA	YES	Bruker Maxis	10	EtOAc	None	Acclaim RSLC 120 C18, 2,1*100 mm	1	Both	1000	Each Batch
006	Tentative detected		<1	3 diagnostic ions	GC	MSD	Q	EI	+	YES	YES	>0.010 mg/kg	YES	Trace DSQ	15	ACN	PSA/C18	DB5MS	0.8	Both		
007	Fully identified (SANCO/12495/2011 p.74-80)	1	None		GC	MSD	Q	EI	+	YES	YES	0.01	NO	Agilent 6890/5973	10	ACN	DSPE	HP-5MS	2	Manual	183	Each Batch
008	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions +accurate mass	LC	MSD	Q-TRAP	ESI	+	YES	NO	0.01	NO	QTrap 5500	10	ACN	None	reversed phase	2.5	Auto	300	Each Batch
009	Tentative detected			2 transitions	GC	MSD	QQQ	EI	+	NO	NO		NO	Thermo TSQ Quantum	10	ACN	DSPE	TG-5MS 30m	2	Manual	180	Each Batch
010	Fully identified (SANCO/12495/2011 p.74-80)	5.4		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01	YES	HP5975C	10	ACN	DSPE	HP5MS	10	Both	aprox 1000	Always
011	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	NA	GC	MSD	Q	EI	+	NO	NO	NA	YES	5790 msd	10	ACN	PSA	HP-5MS	20	Auto	600 aprox	0-1
012	Fully identified (SANCO/12495/2011 p.74-80)	0.6		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010	NO	Waters Quattro Premier	10	Methanol	NO	C18	5	Both	211	Always
014	Fully identified (SANCO/12495/2011 p.74-80)	±2.5%		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010 mg/kg	YES	Waters-TQD	15	ACN	PSA	UPLC-BEH C18	10	Auto	331	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

NAPROPAMIDE																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
015	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	2 transitions	LC		QQQ	ESI	+	YES	YES	0.01	NO	6460	10	ACN	PSA	C18-XB	2	Both	440	
016	Confirmed with second method					MSD	QQQ	CI	+	YES	YES	0.005	NO	LC-MS/MS (Waters)	15	ACN	QuEChERS	C18	10	Both	370	Always
018	Tentative detected	5	5	accurate mass	LC	Orbitrap	Orbitrap	ESI	+	YES	YES	0.01 - 0.05 mg/kg	YES	Thermo Exactive	10	ACN	PSA	C18	5	Both	120	Always
019	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	GC	MSD	QQQ	EI		YES	YES	10	NO	Agilent 6890	15	Acetone/PE/DCM	None	95%methyl 5% phenyl siloxane	2	Auto		
020	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	AbSciex	10	ACN	DSPE	C18	5	Auto	200	Always
021	Fully identified (SANCO/12495/2011 p.74-80)	15		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	HPLC Agilent 1100, MS API 3000	10	ACN	DSPE (PSA, MgSO4)	C18 3µ 50x2mm	10	Both	550	Always
023	Confirmed with second method				LC		QQQ	ESI	+	YES	YES	0.01	NO	API 5500	10	ACN		Pursuit XRs Ultra	3	Both	250	Each Batch
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		2 x MRM	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	AB Sciex API 4000	10	ACN	PSA	Phenomenex C18	10	Both	500	Daily
025	Fully identified (SANCO/12495/2011 p.74-80)	< 20	964	spectrum	GC	MSD	TOF	EI	+	YES	YES	0.01-0.1	YES	Leco Pegasus IV	10	ACN	DSPE (PSA)	RTX-CL pest	10	Auto	557	Each Batch
	Fully identified (SANCO/12495/2011 p.74-80)	-0.28	1.85	isotope, 2 ions < 5ppm	LC	MSD	Orbitrap	ESI	+	NO	NO	0.01-0.1	YES	Exactive	10	ACN	None	C18 Atlantis T3	5	Both	570	Each Batch
026	Fully identified (SANCO/12495/2011 p.74-80)	< 2.5 %	2.5	transition 1 and 2	LC	MSD	Q-TRAP	ESI	+	YES	YES	0.01	YES	3200 Qtrap	10	ACN	DSPE	C18	50	Auto	521	Every Week
027	Fully identified (SANCO/12495/2011 p.74-80)	1	0.7	3 transitions	GC	MSD	QQQ	EI	+	YES	YES	0.01	NO	Varian 3800 GC + 320-MS	10	ACN	DSPE with PSA	VF-1ms	4	Both	302	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

NAPROPAMIDE																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
028	Confirmed with second method			272.1>129.3, 272.1>171.1	LC	MSD	QQQ	ESI	+	NO	NO	10-100 ppb	NO	LC: Agilent 1200, MS: API 4000 QTRAP	10	ACN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Auto	591	
029	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	NO	NO				10	ACN	NO	Xterra C18	20	Both		
030	Tentative detected				GC		QQQ	EI	+	YES	YES	0.02	NO	Varian 450	15	Acetone		C18	10	Auto	150	Each Batch
031	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.005-0.125 µg/ml	NO	UPLC-MSMS (Waters Aquity UPLC system, API 5000 Tripelquadropol, Sciex	10	EtOAc	Filter	HSS T3	2	Both	373	Each Batch
033	Fully identified (SANCO/12495/2011 p.74-80)	0.1		min 3 ions	GC	MSD	Q	EI	+	YES	NO	0.01	YES	Agilent GC-MSD 7890A/5975c	10	ACN	PSA, C18	HP-5MS	1	Auto	927	
034	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC		QQQ	ESI	+	YES	YES		NO	Waters Quattro Premier	10	ACN	DSPE (PSA/MgSO4)	RP-18 endcapped	5	Both	186	Each Batch
036	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES					10	ACN			5	Auto	350	
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.010	NO	4000 QTRAP	10	ACN	DSPE	C18 hydro Phenomenex	40	Manual		Always
038	Tentative detected	None	None	2 transitions	LC	MSD	QQQ	ESI	+	NO	NO		NO	API 4000	10	ACN	DSPE	Waters-C18 2,1x50mmx1,7,µm	5	Auto	600	Every Week
039	Tentative detected				GC	MSD	Q	EI	+	NO	NO		YES	HP5973 MSD	10	ACN	DSPE	HP-5MSI	20	Both	624	No standard used
040	Confirmed with second method	11	3		LC		TOF	ESI	+	NO	NO		YES	Agilent 6230	10	ACN	None	Eclipse C18	3	Auto	aprox 500	
041	Fully identified (SANCO/12495/2011 p.74-80)	0.5%		2 transitions	GC	MSD	QQQ	EI		YES	YES	0.005 mg/kg	NO	VARIAN CP-3800 MS-320	10	ACN	PSA	VF-5MS	1	Auto	210	

APPENDIX 3. Methods used by participants for detecting pesticides.

NAPROPAMIDE																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
042	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES		NO	API 4000	10	ACN	QuEChERS	C18 Aqua	10	Both		
043	Tentative detected	0	0	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010	YES	Api 3200QT	10	ACN	DSPE	Phenomenex fusion	10	Auto	200	Daily
045	Tentative detected	1	1 unit		GC	MSD	Q	EI		YES	NO	0.01 ppm	YES	Agilent 5973	10	ACN	PSA	HP 5 MS UI	2	Auto	923	Each Batch
046	Fully identified (SANCO/12495/2011 p.74-80)	0			LC	MSD	QQQ	ESI	+	NO			NO	Waters Xevo	10	Acetone/PE/DCM	Na2SO4	C18	2	Both	286	Each Batch
047	Fully identified (SANCO/12495/2011 p.74-80)	0.5		Spectrum match (reversed)	GC	MSD	IT	EI	+	YES	YES	20-10.000 µg/kg	YES	Varian Satum 2000	15	Acetone/PE/DCM	NO	VF-5-ms	5	Both	550	Quarterly
048	Confirmed with second method	3			GC	MSD	TOF	EI		YES	YES	0.01	YES	Pegasus IV	10	ACN	DSPE(PSA)	HP-5MS	5	Both	650	Weekly
049	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC		QQQ	ESI	+	YES	YES		NO	Waters XEVO TQ MS	10	ACN		BEH C18 1,7µm	3	Both	60	Daily
050	Fully identified (SANCO/12495/2011 p.74-80)	0.017		Qion ratio	GC	MSD	Q	EI	+	YES	YES	0.01-0.05	YES	Agilent 6890GC+5975MS	10	ACN	DSPE	RESTEK-5MS	50	Both	168	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

PROMETRYN																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	Q	EI	+	YES	YES	0.01	YES	Agilent 5973 inert	5	ACN		HP-5 MSUI	5	Both		Daily
002	Confirmed with second method	5.3			GC	MSD	Q	EI	+	YES	YES	not validated	YES	AT 5975	10	ACN	SPE	HP5-MS	10	Auto	aprox 750	Always
003	Confirmed with second method	0.05	5	2 transitions	LC		Q-TOF	ESI	+	NO	NO		YES	Bruker Maxis	10	ACN	PSA	RSLC C18	1	Both	aprox 400	
004	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA		LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6460, 1290HPLC	10	EtOAc	None	Waters, HSS T3, 150 x 2.1mm	2	Both	500	Each Batch
	Confirmed with second method	3	0.9		LC	MSD	TOF	ESI	+	NO	NO	NA	YES	Bruker Maxis	10	EtOAc	None	Acclaim RSLC 120 C18, 2,1*100 mm	1	Both	1000	Each Batch
006	Fully identified (SANCO/12495/2011 p.74-80)	<2	<1	3 diagnostic ions	GC	MSD	Q	EI	+	YES	YES	>0.010 mg/kg	YES	Trace DSQ	15	ACN	PSA/C18	DB5MS	0.8	Both		
007	Fully identified (SANCO/12495/2011 p.74-80)	1	None		GC	MSD	Q	EI	+	YES	YES	0.01	NO	Agilent 6890/5973	10	ACN	DSPE	HP-5MS	2	Manual	183	Each Batch
008	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions +accurate mass	LC	MSD	Q-TRAP	ESI	+	YES	NO	0.01	NO	QTrap 5500	10	ACN	None	reversed phase	2.5	Auto	300	Each Batch
009	Tentative detected			2 transitions	GC	MSD	QQQ	EI	+	NO	NO		NO	Thermo TSQ Quantum	10	ACN	DSPE	TG-5MS 30m	2	Manual	180	Each Batch
010	Tentative detected			2 transitions	LC	MSD	QQQ	ESI	+	NO	NO		NO	ABI4000	10	ACN	NO	C18	55	Both	aprox 580	Always
	Fully identified (SANCO/12495/2011 p.74-80)	4.7		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01	YES	HP5975C	10	ACN	DSPE	HP5MS	10	Both	aprox 1000	Always
011	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	NA	GC	MSD	Q	EI	+	NO	NO	NA	YES	5790 msd	10	ACN	PSA	HP-5MS	20	Auto	600 aprox	0-1
014	Fully identified (SANCO/12495/2011 p.74-80)	±2.5%		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010 mg/kg	YES	Waters-TQD	15	ACN	PSA	UPLC-BEH C18	10	Auto	331	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

PROMETRYN																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
015	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	2 transitions	GC		QQQ	EI	+	YES	YES	0.01	NO	7000B	10	ACN	PSA	DB-5MS UI	2	Both	440	
016	Confirmed with second method					MSD	QQQ	CI	+	YES	YES	0.005	NO	LC-MS/MS (Waters)	15	ACN	QuEChERS	C18	10	Both	370	Always
017	Tentative detected				GC	MSD	TOF	EI		NO	NO		YES	LECO PEGASUS	15	Acetone/PE/DCM	NO	DB-5	1	Both		
018	Tentative detected	RT locking		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01 - 0.05 mg/kg	YES	Agilent MSD 5975B	10	ACN	PSA	HP5	10	Both	> 600	Always
019	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	10	NO	Agilent 6490	15	ACN	PSA	C18	5	Auto		
020	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	QQQ	EI		YES	YES	0.01	NO	agilent	10	ACN	DSPE	HP5MS	1		200	Always
021	Fully identified (SANCO/12495/2011 p.74-80)	15		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	HPLC Agilent 1100, MS API 3000	10	ACN	DSPE (PSA, MgSO4)	C18 3µ 50x2mm	10	Both	550	Always
023	Confirmed with second method				LC		QQQ	ESI	+	YES	YES	0.01	NO	API 5500	10	ACN		Pursuit XRs Ultra	3	Both	250	Each Batch
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		3 masses	GC	MSD	IT	EI	+	YES	YES	0.01	YES	Varian Saturn 2000	10	ACN	PSA	VactorFour 5 MS	5	Auto	500	Daily
025	Fully identified (SANCO/12495/2011 p.74-80)	< 20	917	spectrum	GC	MSD	TOF	EI	+	YES	YES	0.01-0.1	YES	Leco Pegasus IV	10	ACN	DSPE (PSA)	RTX-CL pest	10	Auto	557	Each Batch
	Confirmed with second method	0.28	1.2	isotope, 2 ions < 5ppm	LC	MSD	Orbitrap	ESI	+	NO	NO	0.01-0.1	YES	Exactive	10	ACN	None	C18 Atlantis T3	5	Both	570	Each Batch
027	Fully identified (SANCO/12495/2011 p.74-80)	3	0.7	3 transitions	GC	MSD	QQQ	EI	+	YES	YES	0.01	NO	Varian 3800 GC + 320-MS	10	ACN	DSPE with PSA	VF-1ms	4	Both	302	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

PROMETRYN																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
028	Confirmed with second method			242.2>200.1, 242.2>158.1	LC	MSD	QQQ	ESI	+	NO	NO	10-100 ppb	NO	LC: Agilent 1200, MS: API 4000 QTRAP	10	ACN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Auto	591	
029	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO		10	ACN	NO	Xterra C18	20	Both		
030	Tentative detected				LC		QQQ	ESI	+	YES	YES	0.02	NO	Varian 320	15	ACN		C18	10	Auto	150	Each Batch
031	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.005-0.125 µg/ml	NO	UPLC-MSMS (Waters Aquity UPLC system, API 5000 Tripelquadropol, Sciex	10	EtOAc	Filter	HSS T3	2	Both	373	Each Batch
033	Fully identified (SANCO/12495/2011 p.74-80)	0.5		min 3 ions	GC	MSD	Q	EI	+	YES	YES	0.01	YES	Agilent GC-MSD 7890A/5975c	10	ACN	PSA, C18	HP-5MS	1	Auto	927	
034	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC		QQQ	EI	+	YES	YES		NO	Waters Quattro Micro	10	ACN	DSPE (PSA/MgSO4)	HP-5MS	5	Both	110	Each Batch
035	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	Q	EI		NO	NO		YES	Agilent 6890N	10	ACN	DSPE	DB-5	2	Auto		
036	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES					10	ACN			5	Auto	350	
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.010	NO	4000 QTRAP	10	ACN	DSPE	C18 hydro Phenomenex	40	Manual		Always
038	Fully identified (SANCO/12495/2011 p.74-80)	None	None	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES		NO	API 4000	10	ACN	DSPE	Waters-C18 2,1x50mmx1,7,µm	5	Auto	600	Every Week
039	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	Q	EI	+	YES	YES	0.01	YES	HP5973 MSD	10	ACN	DSPE	HP-5MSI	20	Both	113	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

PROMETRYN																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
040	Confirmed with second method	30	3		LC		TOF	ESI	+	NO	NO		YES	Agilent 6230	10	ACN	None	Eclipse C18	3	Auto	aprox 500	
041	Fully identified (SANCO/12495/2011 p.74-80)	0.5%		2 transitions	GC	MSD	QQQ	EI		YES	YES	0.005 mg/kg	NO	VARIAN CP-3800 MS-320	10	ACN	PSA	VF-5MS	1	Auto	210	
042	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES		NO	API 4000	10	ACN	QuEChERS	C18 Aqua	10	Both		
043	Tentative detected	0	0	scan	GC	MSD	IT	EI		YES	YES	0.010	YES	Varian Saturn 4000	10	ACN	DSPE	Varian VF	1	Auto	260	Daily
045	Tentative detected	1	1 unit		GC	MSD	Q	EI		YES	NO	0.01 ppm	YES	Agilent 5973	10	ACN	PSA	HP 5 MS UI	2	Auto	923	Each Batch
046	Fully identified (SANCO/12495/2011 p.74-80)	0			GC	MSD	QQQ	EI	+	YES	YES		NO	Thermo TSQ Quantum GC	10	Acetone/PE/DCM	Na2SO4	RXi-5SIL MS	1	Both	199	Each Batch
047	Fully identified (SANCO/12495/2011 p.74-80)	0.1		Spectrum match (reversed)	GC	MSD	IT	EI	+	YES	YES	20-10.000 µg/kg	YES	Varian Saturn 2000	15	Acetone/PE/DCM	NO	VF-5-ms	5	Both	550	Each Batch
048	Confirmed with second method	4			GC	MSD	TOF	EI		YES	YES	0.005	YES	Pegasus IV	10	ACN	DSPE(PSA)	HP-5MS	5	Both	650	Weekly
049	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC		QQQ	ESI	+	YES	YES		NO	Waters XEVO TQ MS	10	ACN		BEH C18 1,7µm	3	Both	60	Daily
050	Fully identified (SANCO/12495/2011 p.74-80)	0.054		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01-0.05	YES	Agilent 1200 LC+6460QQQ	10	ACN	DSPE	YMC Triart C18 2*100 mm 3µm	5	Both	354	Daily
051	Tentative detected		0.5	qualifiers	GC	MSD	IT	EI		NO	NO		YES	varian GCMS 4000	10	ACN	PSA	varian factor four	5	Both		
053	Tentative detected	2		3 SIM Ions	GC	MSD	Q	EI	+	NO	NO	>0.05 mg/kg	YES	Thermo DSQ	10	DCM	Diatomaceous earth		1	Both	400	Every 6 Months

APPENDIX 3. Methods used by participants for detecting pesticides.

PROPAQUIZAFOP																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6490	5	ACN		RP-C18	10	Both		Daily
003	Confirmed with second method	0.05	5	2 transitions	LC		Q-TOF	ESI	+	NO	NO		YES	Bruker Maxis	10	ACN	PSA	RSLC C18	1	Both	aprox 400	
004	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA		LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6460, 1290HPLC	10	EtOAc	None	Waters, HSS T3, 150 x 2.1mm	2	Both	500	Each Batch
	Confirmed with second method	1.2	3.7		LC	MSD	TOF	ESI	+	NO	NO	NA	YES	Bruker Maxis	10	EtOAc	None	Acclaim RSLC 120 C18, 2,1*100 mm	1	Both	1000	Each Batch
007	Fully identified (SANCO/12495/2011 p.74-80)	1	None		LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6410	10	ACN	DSPE	Eclipse XDB-C18	4	Manual	139	Each Batch
008	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions +accurate mass	LC	MSD	Q-TRAP	ESI	+	YES	NO	0.01	NO	QTrap 5500	10	ACN	None	reversed phase	2.5	Auto	300	Each Batch
014	Fully identified (SANCO/12495/2011 p.74-80)	±2.5%		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010 mg/kg	YES	Waters-TQD	15	ACN	PSA	UPLC-BEH C18	10	Auto	331	Each Batch
015	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	2 transitions	LC		QQQ	ESI	+	YES	YES	0.01	NO	6460	10	ACN	PSA	C18-XB	2	Both	440	
021	Fully identified (SANCO/12495/2011 p.74-80)	15		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	HPLC Agilent 1100, MS API 3000	10	ACN	DSPE (PSA, MgSO4)	C18 3µ 50x2mm	10	Both	550	Always
023	Confirmed with second method				LC		Q-TOF	ESI	+	NO	NO	0.01		Bruker Maxis	10	ACN				Both	700	Each Batch
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		2 x MRM	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	AB Sciex API 4000	10	ACN	PSA	Phenomenex C18	10	Both	500	Daily
028	Tentative detected			444.1>299.1, 444.1>100.0	LC	MSD	QQQ	ESI	+	NO	NO	10-100 ppb	NO	LC: Agilent 1200, MS: API 4000 QTRAP	10	ACN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Auto	591	
030	Tentative detected				LC		QQQ	ESI	+	YES	YES	0.02	YES	Varian 320	15	ACN		C18	10	Auto	150	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

PROPAQUIZAFOP																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
031	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.005-0.4 µg/ml	NO	UPLC-MSMS (Waters Aquity UPLC system, API 5000 Tripelquadropol, Sciex	10	EtOAc	Filter	HSS T3	2	Both	373	Each Batch
032	Tentative detected	0		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Waters Acquity TQD	10	Methanol	Filter	C18	6	Auto	150	Always
034	Tentative detected			3 transitions	LC		QQQ	ESI	+	NO	NO		NO	Waters Quattro Premier	10	ACN	DSPE (PSA/MgSO4)	RP-18 endcapped	5	Both	500	
036	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES					10	ACN			5	Auto	350	
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.010	NO	4000 QTRAP	10	ACN	DSPE	C18 hydro Phenomenex	40	Manual		Always
038	Tentative detected	None	None	2 transitions	LC	MSD	QQQ	ESI	+	NO	NO		NO	API 4000	10	ACN	DSPE	Waters-C18 2,1x50mmx1,7,µm	5	Auto	600	Every Week
039	Fully identified (SANCO/12495/2011 p.74-80)			444.1->100.1, 444.1->299.1	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6410B	10	ACN	DSPE	C18	2	Both	187	Each Batch
040	Confirmed with second method	4	5		LC		TOF	ESI	+	NO	NO		YES	Agilent 6230	10	ACN	None	Eclipse C18	3	Auto	aprox 500	
046	Fully identified (SANCO/12495/2011 p.74-80)	0			LC	MSD	QQQ	ESI	+	NO			NO	Waters Xevo	10	Acetone/PE/DCM	Na2SO4	C18	2	Both	286	Each Batch
048	Fully identified (SANCO/12495/2011 p.74-80)	2.3			LC	MSD	QQQ	ESI	+	YES	YES	0.005	NO	Agilent 6410	10	ACN	DSPE(PSA)	Luna C18	5	Both	350	Daily
049	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC		QQQ	ESI	+	YES	YES		NO	Waters XEVO TQ MS	10	ACN		BEH C18 1,7µm	3	Both	60	Daily
053	Fully identified (SANCO/12495/2011 p.74-80)	1		2 MRM transitions	LC	MSD	QQQ	ESI	+	YES	YES	>0.005 mg/kg	NO	API4000	10	ACN	PSA		10	Both	180	Every Month

APPENDIX 3. Methods used by participants for detecting pesticides.

PYRIFENOX																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6490	5	ACN		RP-C18	10	Both		Daily
002	Confirmed with second method	3.0 and 3.9			GC	MSD	Q	EI	+	YES	YES	not validated	YES	AT 5975	10	ACN	SPE	HP5-MS	10	Auto	aprox 750	Always
003	Confirmed with second method	0.05	5	2 transitions	LC		Q-TOF	ESI	+	NO	NO		YES	Bruker Maxis	10	ACN	PSA	RSLC C18	1	Both	aprox 400	
004	Confirmed with second method	3	0.6		LC	MSD	TOF	ESI	+	NO	NO	NA	YES	Bruker Maxis	10	EtOAc	None	Acclaim RSLC 120 C18, 2,1*100 mm	1	Both	1000	Each Batch
	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA		LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6460, 1290HPLC	10	EtOAc	None	Waters, HSS T3, 150 x 2.1mm	2	Both	500	Each Batch
006	Fully identified (SANCO/12495/2011 p.74-80)	<2	0	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	>0.010 mg/kg	NO	Waters quattro premier	15	ACN	None	BEH C18	3	Both		
008	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions +accurate mass	LC	MSD	Q-TRAP	ESI	+	YES	NO	0.01	NO	QTrap 5500	10	ACN	None	reversed phase	2.5	Auto	300	Each Batch
009	Tentative detected			2 transitions	GC	MSD	QQQ	EI	+	NO	NO		NO	Thermo TSQ Quantum	10	ACN	DSPE	TG-5MS 30m	2	Manual	180	Each Batch
010	Fully identified (SANCO/12495/2011 p.74-80)	0.5		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01	YES	HP5975C	10	ACN	DSPE	HP5MS	10	Both	aprox 1000	Always
011	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	NA	GC	MSD	Q	EI	+	NO	NO	NA	YES	5790 msd	10	ACN	PSA	HP-5MS	20	Auto	600 aprox	0-1
014	Fully identified (SANCO/12495/2011 p.74-80)	±2.5%		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010 mg/kg	YES	Waters-TQD	15	ACN	PSA	UPLC-BEH C18	10	Auto	331	Each Batch
015	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	2 transitions	GC		QQQ	EI	+	YES	YES	0.01	NO	7000B	10	ACN	PSA	DB-5MS UI	2	Both	440	
016	Confirmed with second method					MSD	QQQ	CI	+	YES	YES	0.005	NO	LC-MS/MS (Waters)	15	ACN	QuEChERS	C18	10	Both	370	Always

APPENDIX 3. Methods used by participants for detecting pesticides.

PYRIFENOX																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
018	Tentative detected	RT locking		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01 - 0.05 mg/kg	YES	Agilent MSD 5975B	10	ACN	PSA	HP5	10	Both	> 600	Always
019	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	GC	MSD	QQQ	EI		YES	YES	10	NO	Agilent 6890	15	Acetone/PE/DCM	None	95%methyl 5% phenyl siloxane	2	Auto		
020	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	QQQ	EI		YES	YES	0.01	NO	agilent	10	ACN	DSPE	HP5MS	1		200	Always
021	Fully identified (SANCO/12495/2011 p.74-80)	15		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	HPLC Agilent 1100, MS API 3000	10	ACN	DSPE (PSA, MgSO4)	C18 3µ 50x2mm	10	Both	550	Always
023	Confirmed with second method				GC	MSD	TOF			YES	YES	0.01	YES	Leco Pegasus IV	10	ACN	DSPE	Phenomenenx ZB-5MS	3	Both	750	Each Batch
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		3 masses	GC	MSD	IT	EI	+	YES	YES	0.01	YES	Varian Saturn 2000	10	ACN	PSA	VactorFour 5 MS	5	Auto	500	Daily
025	Confirmed with second method	-0.16	0.22	isotope, two isomer peaks	LC	MSD	Orbitrap	ESI	+	NO	NO	0.01-0.1	YES	Exactive	10	ACN	None	C18 Atlantis T3	5	Both	570	Each Batch
	Fully identified (SANCO/12495/2011 p.74-80)	< 20	916	spectrum, two isomer peaks	GC	MSD	TOF	EI	+	YES	YES	0.01-0.1	YES	Leco Pegasus IV	10	ACN	DSPE (PSA)	RTX-CL pest	10	Auto	557	Each Batch
026	Fully identified (SANCO/12495/2011 p.74-80)	< 2.5 %	2.5	transition 1 and 2	LC	MSD	Q-TRAP	ESI	+	YES	YES	0.01	YES	3200 Qtrap	10	ACN	DSPE	C18	50	Auto	521	Every Week
027	Fully identified (SANCO/12495/2011 p.74-80)	1	0.7	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Varian Pro Star +320-MS	10	ACN	DSPE with PSA	Restek Ultra C18	10	Both	302	Each Batch
028	Fully identified (SANCO/12495/2011 p.74-80)			297.1>93.1, 295.1>92.9	LC	MSD	QQQ	ESI	+	YES	YES	LOQ: 8 ppb	NO	LC: Agilent 1200, MS: API 4000 QTRAP	10	ACN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Auto		
029	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	NO	NO				10	ACN	NO	Xterra C18	20	Both		
030	Tentative detected				LC		QQQ	ESI	+	YES	YES	0.02	NO	Varian 320	15	ACN		C18	10	Auto	150	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

PYRIFENOX																				
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
031	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.005-0.4 µg/ml	NO	UPLC-MSMS (Waters Aquity UPLC system, API 5000 Tripelquadropol, Sciex	10	EtOAc	Filter	HSS T3	2	Both	373	Each Batch
033	Fully identified (SANCO/12495/2011 p.74-80)	-0.8		min 3 ions	GC	MSD	Q	EI	+	YES	NO	0.01	YES	Agilent GC-MSD 7890A/5975c	10	ACN	PSA, C18	HP-5MS	1	Auto	927	
034	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC		QQQ	ESI	+	YES	YES		NO	Waters Quattro Premier	10	ACN	DSPE (PSA/MgSO4)	RP-18 endcapped	5	Both	186	Each Batch
036	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES					10	ACN			5	Auto	350	
038	Tentative detected	None	None	2 transitions	LC	MSD	QQQ	ESI	+	NO	NO		NO	API 4000	10	ACN	DSPE	Waters-C18 2,1x50mmx1,7,µm	5	Auto	600	Every Week
039	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	Q	EI	+	YES	YES	0.02	YES	HP5973 MSD	10	ACN	DSPE	HP-5MSI	20	Both	113	Each Batch
040	Confirmed with second method	2	4		LC		TOF	ESI	+	NO	NO		YES	Agilent 6230	10	ACN	None	Eclipse C18	3	Auto	aprox 500	
041	Fully identified (SANCO/12495/2011 p.74-80)	0.5%		2 transitions	GC	MSD	QQQ	EI		YES	YES	0.005 mg/kg	NO	VARIAN CP-3800 MS-320	10	ACN	PSA	VF-5MS	1	Auto	210	
042	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES		NO	API 4000	10	ACN	QuEChERS	C18 Aqua	10	Both		
043	Tentative detected	0	0	scan	GC	MSD	IT	EI		YES	YES	0.010	YES	Varian Saturn 4000	10	ACN	DSPE	Varian VF	1	Auto	260	Daily
045	Tentative detected	1	1 unit		GC	MSD	Q	EI		YES	NO	0.01 ppm	YES	Agilent 5973	10	ACN	PSA	HP 5 MS UI	2	Auto	923	Each Batch
046	Fully identified (SANCO/12495/2011 p.74-80)	0			LC	MSD	QQQ	ESI	+	NO			NO	Waters Xevo	10	Acetone/PE/DCM	Na2SO4	C18	2	Both	286	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

PYRIFENOX																				
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
047	Fully identified (SANCO/12495/2011 p.74-80)	12		2 transitions + ion ratio	LC	MSD	QQQ	ESI	+	YES	YES	5-1000 µg/kg	NO	Waters Quattro Premier XE	15	Acetone/PE/DCM	NO	Acquity BEH (100x2.1 mm i.d.)	5	Both	230	Each Batch
048	Confirmed with second method	4			GC	MSD	TOF	EI		YES	YES	0.005	YES	Pegasus IV	10	ACN	DSPE(PSA)	HP-5MS	5	Both	650	Weekly
049	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC		QQQ	EI	+	YES	YES		NO	Waters Quattro micro GC	10	ACN		DB-5MS	3	Both	150	Daily
053	Fully identified (SANCO/12495/2011 p.74-80)	0		2 MRM transitions	LC	MSD	QQQ	ESI	+	YES	YES	>0.005 mg/kg	NO	API4000	10	ACN	PSA		10	Both	180	Every Month
	Confirmed with second method	0		3 SIM Ions	GC	MSD	Q	EI	+	NO	NO	>0.05 mg/kg	YES	Thermo DSQ	10	DCM	Diatomaceous earth		1	Both	400	Every 6 Months

APPENDIX 3. Methods used by participants for detecting pesticides.

SPINOSAD																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6490	5	ACN		RP-C18	10	Both		Daily
002	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC		QQQ	ESI	+	YES	YES		NO	API4000	10	ACN	None	C18	5	Manual		
004	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA		LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6460, 1290HPLC	10	EtOAc	None	Waters, HSS T3, 150 x 2.1mm	2	Both	500	Each Batch
006	Fully identified (SANCO/12495/2011 p.74-80)	<2	0	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	>0.010 mg/kg	NO	Waters quattro premier	15	ACN	None	BEH C18	10	Both		
007	Fully identified (SANCO/12495/2011 p.74-80)	1	None		LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6410	10	ACN	DSPE	Eclipse XDB-C18	4	Manual	139	Each Batch
008	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions +accurate mass	LC	MSD	Q-TRAP	ESI	+	YES	NO	0.01	NO	QTrap 5500	10	ACN	None	reversed phase	2.5	Auto	300	Each Batch
010	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	ABI4000	10	ACN	NO	C18	55	Both	aprox 580	Always
011	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	NA	LC	MSD	Q-TRAP	ESI	+	YES	YES	0.01	NO	3200qtrap	10	ACN	PSA	T-3	5	Manual	170	1
012	Fully identified (SANCO/12495/2011 p.74-80)	0.6		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010	NO	Waters Quattro Premier	10	Methanol	NO	C18	5	Both	211	Always
014	Fully identified (SANCO/12495/2011 p.74-80)	±2.5%		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010 mg/kg	YES	Waters-TQD	15	ACN	PSA	UPLC-BEH C18	10	Auto	331	Each Batch
015	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	2 transitions	LC		QQQ	ESI	+	YES	YES	0.01	NO	6460	10	ACN	PSA	C18-XB	2	Both	440	
016	Confirmed with second method					MSD	QQQ	CI	+	YES	YES	0.005	NO	LC-MS/MS (Waters)	15	ACN	QuEChERS	C18	10	Both	370	Always

APPENDIX 3. Methods used by participants for detecting pesticides.

SPINOSAD																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
017	Tentative detected				LC	MSD	QQQ	ESI		NO	NO			Agilent 6410	15	Acetone/PE/DCM	NO	C-18	3	Both		
018	Tentative detected	5	5	accurate mass	LC	Orbitrap	Orbitrap	ESI	+	YES	YES	0.01 - 0.05 mg/kg	YES	Thermo Exactive	10	ACN	PSA	C18	5	Both	120	Always
019	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	10	NO	Agilent 6490	15	ACN	PSA	C18	5	Auto		
020	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	AbSciex	10	ACN	DSPE	C18	5	Auto	200	Always
021	Fully identified (SANCO/12495/2011 p.74-80)	15		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	HPLC Agilent 1100, MS API 3000	10	ACN	DSPE (PSA, MgSO4)	C18 3µ 50x2mm	10	Both	550	Always
023	Confirmed with second method				LC		QQQ	ESI	+	YES	YES	0.01	NO	API 5500	10	ACN		Pursuit XRs Ultra	3	Both	250	Each Batch
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		2 x MRM	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	AB Sciex API 4000	10	ACN	PSA	Phenomenex C18	10	Both	500	Daily
025	Fully identified (SANCO/12495/2011 p.74-80)	-0.27	1.3	isotope, 2 ions < 5ppm	LC	MSD	Orbitrap	ESI	+	NO	NO	0.01-0.1	YES	Exactive	10	ACN	None	C18 Atlantis T3	5	Both	570	Each Batch
	Tentative detected	0.22	1.4	other spinosyns	LC	MSD	Orbitrap	ESI	+	NO	NO	0.01-0.1	YES	Exactive	10	ACN	None	C18 Atlantis T3	5	Both	570	Each Batch
026	Fully identified (SANCO/12495/2011 p.74-80)	< 2.5 %	2.5	transition 1 and 2	LC	MSD	Q-TRAP	ESI	+	YES	YES	0.01	YES	3200 Qtrap	10	ACN	DSPE	C18	50	Auto	521	Every Week
027	Fully identified (SANCO/12495/2011 p.74-80)	3	0.7	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Varian Pro Star +320-MS	10	ACN	DSPE with PSA	Restek Ultra C18	10	Both	302	Each Batch
028	Fully identified (SANCO/12495/2011 p.74-80)			732.6>142.2, 732.6>98.2	LC	MSD	QQQ	ESI	+	YES	YES	LOQ: 8 ppb	NO	LC: Agilent 1200, MS: API 4000 QTRAP	10	ACN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Auto		
029	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO		10	ACN	NO	Xterra C18	20	Both		

APPENDIX 3. Methods used by participants for detecting pesticides.

SPINOSAD																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
031	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.005-0.4 µg/ml	NO	UPLC-MSMS (Waters Aquity UPLC system, API 5000 Tripelquadropol, Sciex	10	EtOAc	Filter	HSS T3	2	Both	373	Each Batch
032	Tentative detected	0		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Waters Acquity TQD	10	Methanol	Filter	C18	6	Auto	150	Always
034	Fully identified (SANCO/12495/2011 p.74-80)			1 transitions	LC		QQQ	ESI	+	YES	YES		NO	Waters Quattro Premier	10	ACN	DSPE (PSA/MgSO4)	RP-18 endcapped	5	Both	186	Each Batch
036	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES					10	ACN			5	Auto	350	
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.010	NO	4000 QTRAP	10	ACN	DSPE	C18 hydro Phenomenex	40	Manual		Always
038	Fully identified (SANCO/12495/2011 p.74-80)	None	None	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES		NO	API 4000	10	ACN	DSPE	Waters-C18 2,1x50mmx1,7,µm	5	Auto	600	Every Week
039	Fully identified (SANCO/12495/2011 p.74-80)			732.4->142.0, 732.4->98.1, 746.5->142.1, 746.5->98.1	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6410B	10	ACN	DSPE	C18	2	Both	187	Each Batch
040	Confirmed with second method	1	7		LC		TOF	ESI	+	NO	NO		YES	Agilent 6230	10	ACN	None	Eclipse C18	3	Auto	aprox 500	
041	Fully identified (SANCO/12495/2011 p.74-80)	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.005 mg/kg	NO	AGILENT G-6410A	10	ACN	PSA	ZORBAX-ECLIPSE XDB-C8	20	Auto	143	
042	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES		NO	API 4000	10	ACN	QuEChERS	C18 Aqua	10	Both		
043	Tentative detected	0	0	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010	YES	Api 3200QT	10	ACN	DSPE	Phenomenex fusion	10	Auto	200	Daily
046	Fully identified (SANCO/12495/2011 p.74-80)	0			LC	MSD	QQQ	ESI	+	NO			NO	Waters Xevo	10	Acetone/PE/DCM	Na2SO4	C18	2	Both	286	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

SPINOSAD																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
047	Fully identified (SANCO/12495/2011 p.74-80)	0.6		2 transitions + ion ratio	LC	MSD	QQQ	ESI	+	YES	YES	5-1000 µg/kg	NO	Waters Quattro Premier XE	15	Acetone/PE/DCM	NO	Acquity BEH (100x2.1 mm i.d.)	5	Both	230	Each Batch
048	Fully identified (SANCO/12495/2011 p.74-80)	1.2			LC	MSD	QQQ	ESI	+	YES	YES	0.005	NO	Agilent 6410	10	ACN	DSPE(PSA)	Luna C18	5	Both	350	Daily
049	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC		QQQ	ESI	+	YES	YES		NO	Waters XEVO TQ MS	10	ACN		BEH C18 1,7µm	3	Both	60	Daily
050	Fully identified (SANCO/12495/2011 p.74-80)	0.078		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01-0.05	YES	Agilent 1200 LC+6460QQQ	10	ACN	DSPE	YMC Triart C18 2*100 mm 3µm	5	Both	354	Daily
051	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.05		ABSciex API 2000	10	ACN	PSA	waters Atlantis	5	Auto	117	Each Batch
053	Fully identified (SANCO/12495/2011 p.74-80)	3		2 MRM transitions	LC	MSD	QQQ	ESI	+	YES	YES	>0.005 mg/kg	NO	API4000	10	ACN	PSA		10	Both	180	Every Month

APPENDIX 3. Methods used by participants for detecting pesticides.

TERBACIL																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	Q	EI	+	YES	YES	0.01	YES	Agilent 5973 inert	5	ACN		HP-5 MSUI	5	Both		Daily
002	Confirmed with second method	8.0			GC	MSD	Q	EI	+	YES	YES	not validated	YES	AT 5975	10	ACN	SPE	HP5-MS	10	Auto	aprox 750	Always
003	Confirmed with second method	0.05	5	2 transitions	LC		Q-TOF	ESI	-	NO	NO		YES	Bruker Maxis	10	ACN	PSA	RSLC C18	1	Both	aprox 400	
006	Tentative detected		<1	3 diagnostic ions	GC	MSD	Q	EI	+	YES	YES	>0.010 mg/kg	YES	Trace DSQ	15	ACN	PSA/C18	DB5MS	0.8	Both		
007	Fully identified (SANCO/12495/2011 p.74-80)	1	None		GC	MSD	Q	EI	+	YES	YES	0.01	NO	Agilent 6890/5973	10	ACN	DSPE	HP-5MS	2	Manual	183	Each Batch
008	Tentative detected		5		GC	MSD	TOF	EI		NO	NO		YES	GCT Micromass	12.5	EtOAc	GPC	capillary	1	Auto	NIST library	We do not have a standard
009	Tentative detected			2 transitions	GC	MSD	QQQ	EI	+	NO	NO		NO	Thermo TSQ Quantum	10	ACN	DSPE	TG-5MS 30m	2	Manual	180	Each Batch
010	Fully identified (SANCO/12495/2011 p.74-80)	0.7		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01	YES	HP5975C	10	ACN	DSPE	HP5MS	10	Both	aprox 1000	Always
011	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	NA	GC	MSD	Q	EI	+	NO	NO	NA	YES	5790 msd	10	ACN	PSA	HP-5MS	20	Auto	600 aprox	0-1
014	Fully identified (SANCO/12495/2011 p.74-80)	±2.5%		2 transitions	LC	MSD	QQQ	ESI	-	YES	YES	0.010 mg/kg	YES	Waters-TQD	15	ACN	PSA	UPLC-BEH C18	10	Auto	331	Each Batch
015	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	2 transitions	GC		QQQ	EI	+	YES	YES	0.01	NO	7000B	10	ACN	PSA	DB-5MS UI	2	Both	440	
017	Tentative detected				GC	MSD	TOF	EI		NO	NO		YES	LECO PEGASUS	15	Acetone/PE/DCM	NO	DB-5	1	Both		

APPENDIX 3. Methods used by participants for detecting pesticides.

TERBACIL																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
018	Tentative detected	RT locking		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01 - 0.05 mg/kg	YES	Agilent MSD 5975B	10	ACN	PSA	HP5	10	Both	> 600	Always
020	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	QQQ	EI		YES	YES	0.01	NO	agilent	10	ACN	DSPE	HP5MS	1		200	Always
023	Confirmed with second method				LC		IT	ESI	-	YES	YES	0.01	NO		10	ACN				Both		Each Batch
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		2 x MRM	LC	MSD	QQQ	ESI	-	YES	YES	0.01	NO	AB Sciex API 4000	10	ACN	PSA	Phenomenex C18	10	Both	500	Daily
025	Fully identified (SANCO/12495/2011 p.74-80)	-0.3	1.7	isotope, 2 ions < 5ppm	LC	MSD	Orbitrap	ESI	+	NO	NO	0.01-0.1	YES	Exactive	10	ACN	None	C18 Atlantis T3	5	Both	570	Each Batch
	Fully identified (SANCO/12495/2011 p.74-80)	< 20	939	spectrum	GC	MSD	TOF	EI	+	YES	YES	0.01-0.1	YES	Leco Pegasus IV	10	ACN	DSPE (PSA)	RTX-CL pest	10	Auto	557	Each Batch
026	Fully identified (SANCO/12495/2011 p.74-80)	< 0.5 %	2.5		GC	MSD	ion trap	EI		YES	YES	0.01	YES	Trap 4000 - Varian	10	ACN	DSPE	5%	1 µL	Auto	450	Every Week
027	Fully identified (SANCO/12495/2011 p.74-80)	4	0.7	3 transitions	GC	MSD	QQQ	EI	+	YES	YES	0.01	NO	Varian 3800 GC + 320-MS	10	ACN	DSPE with PSA	VF-1ms	4	Both	302	Each Batch
028	Tentative detected			Match with NIST Library (Match 925, range 0-1000)	GC	MSD	TOF	EI		NO	NO	10-100 ppb	YES	GCxGC-TOF MS Pegasus 4D	10	ACN	QuEChERS without PSA	DB5-MS (30 m x 0,25 mm x 0,25 µm) in the 1st Dimension, BPX-50 (2 m x 0,1 mm x 0,1 µm) in the 2nd Dimension	10	Auto	500	
029	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	NO	NO				10	ACN	NO	Xterra C18	20	Both		
030	Tentative detected				GC		QQQ	EI	+	NO	NO	0.02	NO	Varian 450	15	Acetone		V5MS	1	Auto	150	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

TERBACIL																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
033	Fully identified (SANCO/12495/2011 p.74-80)	-4.1		min 3 ions	GC	MSD	Q	EI	+	YES	NO	0.01	YES	Agilent GC-MSD 7890A/5975c	10	ACN	PSA, C18	HP-5MS	1	Auto	927	
034	Confirmed with second method			full scan	GC	MSD	Q	EI	+	NO	NO		YES	Agilent 5973 MSD	10	ACN	DSPE (PSA/MgSO4)	HP-5MS	10	Both	950	
035	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	Q	EI		NO	NO		YES	Agilent 6890N	10	ACN	DSPE	DB-5	2	Auto		
036	Tentative detected				GC	MSD	Q	EI							10	ACN			5	Auto		
039	Tentative detected				GC	MSD	Q	EI	+	NO	NO		YES	HP5973 MSD	10	ACN	DSPE	HP-5MSI	20	Both	624	No standard used
040	Confirmed with second method	17	8		LC		TOF	ESI	-	NO	NO		YES	Agilent 6230	10	ACN	None	Eclipse C18	3	Auto	aprox 500	
041	Fully identified (SANCO/12495/2011 p.74-80)	0.5%		2 transitions	GC	MSD	QQQ	EI		YES	YES	0.005 mg/kg	NO	VARIAN CP-3800 MS-320	10	ACN	PSA	VF-5MS	1	Auto	210	
043	Tentative detected	0	0	2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010	YES	Api 3200QT	10	ACN	DSPE	Phenomenex fusion	10	Auto	200	Daily
045	Tentative detected	1	1 unit		GC	MSD	Q	EI		YES	NO	0.01 ppm	YES	Agilent 5973	10	ACN	PSA	HP 5 MS UI	2	Auto	923	Each Batch
046	Fully identified (SANCO/12495/2011 p.74-80)	0			GC	MSD	QQQ	EI	+	YES	YES		NO	Thermo TSQ Quantum GC	10	Acetone/PE/DCM	Na2SO4	RXi-5SIL MS	1	Both	199	Each Batch
047	Fully identified (SANCO/12495/2011 p.74-80)	0.7		Spectrum match (reversed)	GC	MSD	IT	EI	+	YES	YES	20-10.000 µg/kg	YES	Varian Satum 2000	15	Acetone/PE/DCM	NO	VF-5-ms	5	Both	550	Quarterly
048	Confirmed with second method	6			GC	MSD	TOF	EI		YES	YES	0.005	YES	Pegasus IV	10	ACN	DSPE(PSA)	HP-5MS	5	Both	650	Weekly

APPENDIX 3. Methods used by participants for detecting pesticides.

TERBACIL																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
049	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC		QQQ	EI	+	YES	YES		NO	Waters Quattro micro GC	10	ACN		DB-5MS	3	Both	150	Daily
050	Fully identified (SANCO/12495/2011 p.74-80)	0.036		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01-0.05	YES	Agilent 1200 LC+6460QQQ	10	ACN	DSPE	YMC Triart C18 2*100 mm 3µm	5	Both	354	Daily
051	Tentative detected		0.5	qualifiers	GC	MSD	IT	EI		NO	NO		YES	varian GCMS 4000	10	ACN	PSA	varian factor four	5	Both		
053	Tentative detected	12		3 SIM Ions	GC	MSD	Q	EI	+	NO	NO	>0.05 mg/kg	YES	Thermo DSQ	10	DCM	Diatomaceous earth		1	Both	400	Every 6 Months

APPENDIX 3. Methods used by participants for detecting pesticides.

TERBUMETON																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	Ionization/Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6490	5	ACN		RP-C18	10	Both		Daily
002	Confirmed with second method	5.0			GC	MSD	Q	EI	+	YES	YES	not validated	YES	AT 5975	10	ACN	SPE	HP5-MS	10	Auto	aprox 750	Always
004	Tentative detected	NA	NA		LC	MSD	QQQ	ESI	+	YES	NO	NA	NO	Agilent 6460, 1290HPLC	10	EtOAc	None	Waters, HSS T3, 150 x 2.1mm	2	Both	500	Each Batch
	Confirmed with second method	-1.2	0.6		LC	MSD	TOF	ESI	+	NO	NO	NA	YES	Bruker Maxis	10	EtOAc	None	Acclaim RSLC 120 C18, 2,1*100 mm	1	Both	1000	Each Batch
006	Tentative detected		<1	3 diagnostic ions	GC	MSD	Q	EI	+	YES	YES	>0.010 mg/kg	YES	Trace DSQ	15	ACN	PSA/C18	DB5MS	0.8	Both		
007	Fully identified (SANCO/12495/2011 p.74-80)	1	None		GC	MSD	Q	EI	+	YES	YES	0.01	NO	Agilent 6890/5973	10	ACN	DSPE	HP-5MS	2	Manual	183	Each Batch
008	Tentative detected		0.1		LC	MSD	Q-TOF	ESI	+	NO	NO		YES	5600 triple TOF	10	ACN	None	reversed phase	2	Auto	600	We do not have a standard
010	Tentative detected	3.8		2 transitions	GC	MSD	Q	EI	+	YES	NO	0.01	YES	HP5975C	10	ACN	DSPE	HP5MS	10	Both	aprox 1000	Always
011	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	NA	GC	MSD	Q	EI	+	NO	NO	NA	YES	5790 msd	10	ACN	PSA	HP-5MS	20	Auto	600 aprox	0-1
014	Fully identified (SANCO/12495/2011 p.74-80)	±2.5%		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010 mg/kg	YES	Waters-TQD	15	ACN	PSA	UPLC-BEH C18	10	Auto	331	Each Batch
015	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	2 transitions	GC		QQQ	EI	+	YES	YES	0.01	NO	7000B	10	ACN	PSA	DB-5MS UI	2	Both	440	
016	Confirmed with second method					MSD	QQQ	CI	+	YES	YES	0.005	NO	LC-MS/MS (Waters)	15	ACN	QuEChERS	C18	10	Both	370	Always
017	Tentative detected				GC	MSD	TOF	EI		NO	NO		YES	LECO PEGASUS	15	Acetone/PE/DCM	NO	DB-5	1	Both		
018	Tentative detected	RT locking		2 transitions	GC	MSD	Q	EI	+	YES	YES	0.01 - 0.05 mg/kg	YES	Agilent MSD 5975B	10	ACN	PSA	HP5	10	Both	> 600	Always

APPENDIX 3. Methods used by participants for detecting pesticides.

TERBUMETON																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
020	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	AbSciex	10	ACN	DSPE	C18	5	Auto	200	Always
022	Tentative detected	0.5	0.05		LC	MSD	TOF	ESI	+	YES	YES		YES	LCT PREMIER XE	15	MINI LUKE	NO	C-18	7	Auto	343	Every Day
023	Confirmed with second method				LC		QQQ	ESI	+	YES	YES	0.01	NO	API 5500	10	ACN		Pursuit XRs Ultra	3	Both	250	Each Batch
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		3 masses	GC	MSD	IT	EI	+	YES	YES	0.05	YES	Varian Saturn 2000	10	ACN	PSA	VactorFour 5 MS	5	Auto	500	Daily
025	Fully identified (SANCO/12495/2011 p.74-80)	-0.16	-1.5	isotope, 2 ions < 5ppm	LC	MSD	Orbitrap	ESI	+	NO	NO	0.01-0.1	YES	Exactive	10	ACN	None	C18 Atlantis T3	5	Both	570	Each Batch
025	Fully identified (SANCO/12495/2011 p.74-80)	< 20	755	spectrum	GC	MSD	TOF	EI	+	YES	YES	0.01-0.1	YES	Leco Pegasus IV	10	ACN	DSPE (PSA)	RTX-CL pest	10	Auto	557	Each Batch
	Fully identified (SANCO/12495/2011 p.74-80)	< 0.5 %	2.5		GC	MSD	ion trap	EI		YES	YES	0.01	YES	Trap 4000 - Varian	10	ACN	DSPE	5%	1 µL	Auto	450	Every Week
027	Fully identified (SANCO/12495/2011 p.74-80)	3	0.7	3 transitions	GC	MSD	QQQ	EI	+	YES	YES	0.01	NO	Varian 3800 GC + 320-MS	10	ACN	DSPE with PSA	VF-1ms	4	Both	302	Each Batch
028	Tentative detected			226.0>170.0, 226.0>114.0	LC	MSD	QQQ	ESI	+	NO	NO	10-100 ppb	NO	LC: Agilent 1200, MS: API 4000 QTRAP	10	ACN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Auto	591	
029	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	NO	NO				10	ACN	NO	Xterra C18	20	Both		
030	Tentative detected				LC		QQQ	ESI	+	YES	YES	0.02	NO	Varian 320	15	ACN		C18	10	Auto	150	Each Batch
033	Fully identified (SANCO/12495/2011 p.74-80)	0.3		min 3 ions	GC	MSD	Q	EI	+	YES	NO	0.01	YES	Agilent GC-MSD 7890A/5975c	10	ACN	PSA, C18	HP-5MS	1	Auto	927	
034	Confirmed with second method			full scan	GC	MSD	Q	EI	+	NO	NO		YES	Agilent 5973 MSD	10	ACN	DSPE (PSA/MgSO4)	HP-5MS	10	Both	950	

APPENDIX 3. Methods used by participants for detecting pesticides.

TERBUMETON																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
035	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	Q	EI		NO	NO		YES	Agilent 6890N	10	ACN	DSPE	DB-5	2	Auto		
036	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES					10	ACN			5	Auto	350	
038	Tentative detected	None	None	2 transitions	LC	MSD	QQQ	ESI	+	NO	NO		NO	API 4000	10	ACN	DSPE	Waters-C18 2,1x50mmx1,7,µm	5	Auto	600	Every Week
039	Tentative detected				GC	MSD	Q	EI	+	NO	NO		YES	HP5973 MSD	10	ACN	DSPE	HP-5MSI	20	Both	624	No standard used
040	Confirmed with second method	17	3		LC		TOF	ESI	+	NO	NO		YES	Agilent 6230	10	ACN	None	Eclipse C18	3	Auto	aprox 500	
	Tentative detected		3		LC		TOF	ESI	+	NO	NO		YES	Agilent 6230	10	ACN	None	Eclipse C18	3	Auto	aprox 500	
041	Fully identified (SANCO/12495/2011 p.74-80)	0.5%		2 transitions	GC	MSD	QQQ	EI		YES	YES	0.005 mg/kg	NO	VARIAN CP-3800 MS-320	10	ACN	PSA	VF-5MS	1	Auto	210	
045	Tentative detected	1	1 unit		GC	MSD	Q	EI		YES	NO	0.01 ppm	YES	Agilent 5973	10	ACN	PSA	HP 5 MS UI	2	Auto	923	Each Batch
047	Fully identified (SANCO/12495/2011 p.74-80)	0.2		Spectrum match (reversed)	GC	MSD	IT	EI	+	YES	YES	20-10.000 µg/kg	YES	Varian Saturn 2000	15	Acetone/PE/DCM	NO	VF-5-ms	5	Both	550	Quarterly
048	Confirmed with second method	4			GC	MSD	TOF	EI		YES	YES	0.005	YES	Pegasus IV	10	ACN	DSPE(PSA)	HP-5MS	5	Both	650	Weekly
049	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC		QQQ	ESI	+	YES	YES		NO	Waters XEVO TQ MS	10	ACN		BEH C18 1,7µm	3	Both	60	Daily
050	Fully identified (SANCO/12495/2011 p.74-80)	0.032		Qion ratio	GC	MSD	Q	EI	+	YES	YES	0.01-0.05	YES	Agilent 6890GC+5975MS	10	ACN	DSPE	RESTEK-5MS	50	Both	168	Daily
051	Tentative detected		0.5	qualifiers	GC	MSD	IT	EI		NO	NO		YES	varian GCMS 4000	10	ACN	PSA	varian factor four	5	Both		
053	Tentative detected	10		3 SIM Ions	GC	MSD	Q	EI	+	NO	NO	>0.05 mg/kg	YES	Thermo DSQ	10	DCM	Diatomaceous earth		1	Both	400	Every 6 Months

APPENDIX 3. Methods used by participants for detecting pesticides.

VAMIDOTHION																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6490	5	ACN		RP-C18	10	Both		Daily
002	Confirmed with second method	8.4			GC	MSD	Q	EI	+	YES	YES	not validated	YES	AT 5975	10	ACN	SPE	HP5-MS	10	Auto	aprox 750	Always
002	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC		QQQ	ESI	+	YES	YES		NO	API4000	10	ACN	None	C18	5	Manual		
003	Confirmed with second method	0.05	5	2 transitions	LC		Q-TOF	ESI	+	NO	NO		YES	Bruker Maxis	10	ACN	PSA	RSLC C18	1	Both	aprox 400	
004	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA		LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6460, 1290HPLC	10	EtOAc	None	Waters, HSS T3, 150 x 2.1mm	2	Both	500	Each Batch
	Confirmed with second method	-0.6	2.4		LC	MSD	TOF	ESI	+	NO	NO	NA	YES	Bruker Maxis	10	EtOAc	None	Acclaim RSLC 120 C18, 2,1*100 mm	1	Both	1000	Each Batch
006	Tentative detected		<1	3 diagnostic ions	GC	MSD	Q	EI	+	YES	YES	>0.010 mg/kg	YES	Trace DSQ	15	ACN	PSA/C18	DB5MS	0.8	Both		
008	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions +accurate mass	LC	MSD	Q-TRAP	ESI	+	YES	NO	0.01	NO	QTrap 5500	10	ACN	None	reversed phase	2.5	Auto	300	Each Batch
010	Tentative detected	6.5		2 transitions	GC	MSD	Q	EI	+	YES	NO		YES	HP5975C	10	ACN	DSPE	HP5MS	10	Both	aprox 1000	Always
012	Fully identified (SANCO/12495/2011 p.74-80)	0.6		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010	NO	Waters Quattro Premier	10	Methanol	NO	C18	5	Both	211	Always
013	Fully identified (SANCO/12495/2011 p.74-80)	4	NA	ion ratio - quantifier 288/146 qualifier 288/118	LC	MSD	QQQ	ESI	+	YES	YES	0.010 mg/Kg	NO	API 3200 QTRAP	10.0	ACN	d-SPE	C18	20	Manual	120	Each Batch
014	Fully identified (SANCO/12495/2011 p.74-80)	±2.5%		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.010 mg/kg	YES	Waters-TQD	15	ACN	PSA	UPLC-BEH C18	10	Auto	331	Each Batch
015	Fully identified (SANCO/12495/2011 p.74-80)	NA	NA	2 transitions	LC		QQQ	ESI	+	YES	YES	0.01	NO	6460	10	ACN	PSA	C18-XB	2	Both	440	

APPENDIX 3. Methods used by participants for detecting pesticides.

VAMIDOTHION																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
016	Confirmed with second method					MSD	QQQ	CI	+	YES	YES	0.005	NO	LC-MS/MS (Waters)	15	ACN	QuEChERS	C18	10	Both	370	Always
017	Tentative detected				GC	MSD	TOF	EI		NO	NO		YES	LECO PEGASUS	15	Acetone/PE/DCM	NO	DB-5	1	Both		
019	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	10	NO	Agilent 6490	15	ACN	PSA	C18	5	Auto		
020	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	AbSciex	10	ACN	DSPE	C18	5	Auto	200	Always
022	Tentative detected	0.5	0.05		LC	MSD	TOF	ESI	+	YES	YES		YES	LCT PREMIER XE	15	MINI LUKE	NO	C-18	7	Auto	343	Every Day
023	Confirmed with second method				LC		Q-TOF	ESI	+	NO	NO	0.01		Bruker Maxis	10	ACN				Both	700	Each Batch
024	Fully identified (SANCO/12495/2011 p.74-80)	0.1		2 x MRM	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	AB Sciex API 4000	10	ACN	PSA	Phenomenex C18	10	Both	500	Daily
025	Fully identified (SANCO/12495/2011 p.74-80)	-0.12	0.76	isotope, 2 ions < 5ppm	LC	MSD	Orbitrap	ESI	+	NO	NO	0.01-0.1	YES	Exactive	10	ACN	None	C18 Atlantis T3	5	Both	570	Each Batch
	Fully identified (SANCO/12495/2011 p.74-80)	< 20	901	spectrum	GC	MSD	TOF	EI	+	YES	YES	0.01-0.1	YES	Leco Pegasus IV	10	ACN	DSPE (PSA)	RTX-CL pest	10	Auto	557	Each Batch
028	Fully identified (SANCO/12495/2011 p.74-80)			288.1>118.0, 288.1>146.0	LC	MSD	QQQ	ESI	+	YES	YES	LOQ: 2 ppb	NO	LC: Agilent 1200, MS: API 4000 QTRAP	10	ACN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Auto		
030	Tentative detected				LC		QQQ	ESI	+	YES	YES	0.02	NO	Varian 320	15	ACN		C18	10	Auto	150	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

VAMIDOTHION																					
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
031	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.005-0.4 µg/ml	NO	UPLC-MSMS (Waters Aquity UPLC system, API 5000 Tripelquadropol, Sciex	10	EtOAc	Filter	HSS T3	2	Both	373	Each Batch
032	Tentative detected	0		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Waters Acquity TQD	10	Methanol	Filter	C18	6	Auto	150	Always
034	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC		QQQ	ESI	+	YES	YES		NO	Waters Quattro Premier	10	ACN	DSPE (PSA/MgSO4)	RP-18 endcapped	5	Both	186	Each Batch
036	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES					10	ACN			5	Auto	350	
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	YES	YES	0.010	NO	4000 QTRAP	10	ACN	DSPE	C18 hydro Phenomenex	40	Manual		Always
038	Tentative detected	None	None	2 transitions	LC	MSD	QQQ	ESI	+	NO	NO		NO	API 4000	10	ACN	DSPE	Waters-C18 2,1x50mmx1,7,µm	5	Auto	600	Every Week
039	Fully identified (SANCO/12495/2011 p.74-80)			288.1->146.1, 288.1->58.1	LC	MSD	QQQ	ESI	+	YES	YES	0.01	NO	Agilent 6410B	10	ACN	DSPE	C18	2	Both	187	Each Batch
040	Confirmed with second method	2	2		LC		TOF	ESI	+	NO	NO		YES	Agilent 6230	10	ACN	None	Eclipse C18	3	Auto	aprox 500	
041	Fully identified (SANCO/12495/2011 p.74-80)	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.005 mg/kg	NO	AGILENT G-6410A	10	ACN	PSA	ZORBAX-ECLIPSE XDB-C8	20	Auto	143	
042	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	YES	YES		NO	API 4000	10	ACN	QuEChERS	C18 Aqua	10	Both		
046	Fully identified (SANCO/12495/2011 p.74-80)	0			LC	MSD	QQQ	ESI	+	NO			NO	Waters Xevo	10	Acetone/PE/DCM	Na2SO4	C18	2	Both	286	Each Batch
047	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions + ion ratio	LC	MSD	QQQ	ESI	+	YES	YES	5-1000 µg/kg	NO	Waters Quattro Premier XE	15	Acetone/PE/DCM	NO	Acquity BEH (100x2.1 mm i.d.)	5	Both	230	Each Batch

APPENDIX 3. Methods used by participants for detecting pesticides.

VAMIDOTHION																				
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Techique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range?	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
048	Confirmed with second method	2.7			LC	MSD	QQQ	ESI	+	YES	YES	0.005	NO	Agilent 6410	10	ACN	DSPE(PSA)	Luna C18	5	Both	350	Daily
049	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC		QQQ	ESI	+	YES	YES		NO	Waters XEVO TQ MS	10	ACN		BEH C18 1,7µm	3	Both	60	Daily
050	Fully identified (SANCO/12495/2011 p.74-80)	0.039		2 transitions	LC	MSD	QQQ	ESI	+	YES	YES	0.01-0.05	YES	Agilent 1200 LC+6460QQQ	10	ACN	DSPE	YMC Triart C18 2*100 mm 3µm	5	Both	354	Daily

ANNEX 1. List of Laboratories that participate in EUPT-FV-SM04.

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
AUSTRIA	AUSTRIAN AGENCY FOR HEALTH AND FOOD SAFETY, COMPETENCE CENTER FOR RESIDUES OF PLANT PROTECTION PRODUCTS	INNSBRUCK	YES
BELGIUM	FYTOLAB	ZWIJNAARDE	YES
BELGIUM	LOVAP	GEEL	YES
BELGIUM	SCIENTIFIC INSTITUTE OF PUBLIC HEALTH	BRUXELLES	YES
CZECH REPUBLIC	CZECH AGRICULTURE AND FOOD INSPECTION AUTHORITY	PRAHA	YES
CZECH REPUBLIC	INSTITUTE OF CHEMICAL TECHNOLOGY PRAGUE, DEPT. OF FOOD CHEMISTRY AND ANALYSIS	PRAGUE	YES
DENMARK	DANISH VETERINARY AND FOOD ADMINISTRATION REGION 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	DOKKI, GIZA	YES
ESTONIA	LABORATORY FOR RESIDUES AND CONTAMINANTS, AGRICULTURAL RESEARCH CENTRE	SAKU	YES
FINLAND	FINNISH CUSTOMS LABORATORY	ESPOO	YES
FRANCE	CERECO SUD	GARONS	YES
FRANCE	GIRPA	BEAUCOUZE	YES
FRANCE	LABORATOIRE DU SCL DE MONTPELLIER	MONTPELLIER	YES
FRANCE	LABORATORY SCL-RENNES	RENNES	YES
FRANCE	SERVICE COMMUN DES LABORATOIRES - LABORATOIRE SCL D'ILE DE FRANCE	MASSY CEDEX	YES
GERMANY	BAYERISCHES LANDESAMT FÜR GESUNDHEIT UND LEBENSMITTELSICHERHEIT	ERLANGEN	YES
GERMANY	CHEMICAL AND VETERINARY ANALYTICAL INSTITUTE MUENSTERLAND-EMSCHER LIPPE	MUENSTER	YES
GERMANY	EUROFINS DR. SPECHT LABORATORIEN GMBH	HAMBURG	YES
GERMANY	FEDERAL OFFICE OF CONSUMER PROTECTION AND FOOD SAFETY (BVL)	BERLIN	YES
GERMANY	GALAB LABORATORIES GMBH	GEESTHACHT	YES
GERMANY	INSTITUTE FOR HYGIENE AND ENVIRONMENT	HAMBURG	YES
GERMANY	LABOR FRIEDLE GMBH	REGENSBURG	YES
GERMANY	LUA SACHSEN, DEUTSCHLAND	DRESDEN	YES
GERMANY	NIEDERSAECHSISCHES LANDESAMT FUER VERBRAUCHERSCHUTZ UND LEBENSMITTELSICHERHEIT, LVI OLDENBURG	OLDENBURG	YES
GREECE	PESTICIDE RESIDUES LABORATORY, D CHEMICAL DIVISION, GENERAL CHEMICAL STATE LABORATORY	ATHENS	YES
HUNGARY	AGRICULTURAL OFFICE, DPPSCA PESTICIDE RESIDUE ANALYTICAL LABORATORY, MISKOLC	MISKOLC	YES
HUNGARY	WESSLING HUNGARY LTD. FOOD TESTING LABORATORY	BUDAPEST	YES
IRELAND	THE PESTICIDE CONTROL LABORATORY	CELBRIDGE	YES

ANNEX 1. List of Laboratories that participate in EUPT-FV-SM04.

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
ITALY	AGENZIA REGIONALE PROTEZIONE AMBIENTALE-LABORATORIO DI PORDENONE	PORDENONE	YES
ITALY	ARPA PUGLIA DIPARTIMENTO DI BRINDISI	BRINDISI	NO
ITALY	ARPA VENETO, S.L. VERONA	VERONA	YES
ITALY	LABORATORIO CONTAMINANTI AMBIENTALI - ISTITUTO ZOOPROFILATTICO SPERIMENTALE UMBRIA MARCHE	PERUGIA	YES
ITALY	LANDESAGENTUR FÜR UMWELT - LABOR FÜR CHROMATOGRAPHIE	BOZEN	YES
ITALY	SERVIZIO LABORATORIO CHIMICO - TOSSICOLOGICO ARPACAL	COSENZA	YES
LATVIA	INSTITUTE OF FOOD SAFETY, ANIMAL HEALTH AND ENVIRONMENT "BIOR"	RIGA	YES
NORWAY	BIOFORSK - NORWEGIAN INSTITUTE FOR AGRICULTURAL AND ENVIRONMENTAL RESEARCH, PLANT HEALTH AND PLANT PROTECTION DIVISION, PESTICIDE CHEMISTRY SECTION	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	INSTITUTE OF PUBLIC HEALTH MARIBOR, ENVIRONMENTAL PROTECTION INSTITUTE	MARIBOR	YES
SPAIN	LABORATORIO AGROALIMENTARIO DE GRANADA (ATARFE)	ATARFE (GRANADA)	YES
SPAIN	LABORATORIO AGROALIMENTARIO DE LA GENERALITAT VALENCIANA	BURJASSOT	YES
SPAIN	LABORATORIO AGROALIMENTARIO Y DE SANIDAD ANIMAL	EL PALMAR (MURCIA)	YES
SPAIN	LABORATORIO DE SANIDAD VEGETAL	OVIEDO	NO
SWEDEN	EUROFINS FOOD & AGRO SWEDEN AB	LIDKÖPING	YES
SWEDEN	NATIONAL FOOD AGENCY (NFA), CHEMICAL UNIT 1	UPPSALA	YES
SWITZERLAND	SERVICE DE LA CONSOMMATION ET DES AFFAIRES VETERINAIRES (SCAV)	GENEVE	YES
THE NETHERLANDS	NVWA - NETHERLANDS FOOD AND CONSUMER PRODUCT SAFETY AUTHORITY	WAGENINGEN	YES
THE NETHERLANDS	RIKILT	WAGENINGEN	YES
TURKEY	MSM FOOD CONTROL LABORATORY	MERSIN	YES
UNITED KINGDOM	EUROFINS LABORATORIES LTD	WOLVERHAMPTON	YES
UNITED KINGDOM	THE FOOD AND ENVIRONMENT RESEARCH AGENCY (FERA)	YORK	YES

Protocol

EUPT-FV-SM04 SPECIFIC PROTOCOL

European Union Proficiency Test for

Pesticide Residues in Fruits and Vegetables

Screening Multiresidue Methods

(2012)

Introduction

This protocol is complementary to the General Protocol for EU Proficiency Tests (EUPT) dealing with Pesticide Residues in Food and Feed. This Proficiency Test is organised by the EURL for Pesticide Residues in Fruits and Vegetables and covers the screening of pesticides using multiresidue methods of analysis.

The aim of this test is to evaluate laboratory capability when using large-scope quantitative and/or screening methods during routine analysis, for detecting and identifying unexpected pesticides at levels at, or above 0.01 mg/kg – included in and/or in addition to the laboratories' quantitative methods used for frequently-detected pesticides. A second aim is to encourage official laboratories to extend the scope of their methods in a cost-effective way, by using the different MS instruments/software and methods available (whether old or new).

Test material

This proficiency test is based on the pesticide-residue analysis of pears. The pears were grown in Aragón, Spain.

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

Ten of these bottles containing the test material will be chosen randomly and analysed to check for homogeneity.

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

Two bottles, again chosen randomly, will be analysed over a period of time to confirm the stability of the pesticides in the test material (firstly, when the test materials are shipped, and then a few days after the deadline for receipt of participants´ results). There will be a further analysis during this period reproducing the sample shipment i.e. maintaining the sample at room temperature for a few days to see if there is any degradation of any of the pesticides present in the test material.

These results will not be included in the statistical analysis of the proficiency test. The aim is solely to check pesticide stability during the shipping process and for the duration of the proficiency test.

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 online, 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 unique Laboratory Code. With this code, laboratories will be able to access the restricted area containing the reply forms using their login information - 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. The sample delivery will be free of charge to those laboratories already participating in EUPT-FV14. For those who are not EUPT-FV14 participants, please see Cost for shipment of the test material for further details. The payment procedure must have started before 20th February. An e-mail showing the bank transfer confirmation, or similar, must have been sent beforehand, or may be requested by the Organiser at any time. Payments without a Laboratory Code or Invoice Number to identify them will not be considered paid.
4. When the participant laboratories receive the test material (and not before), they must enter the restricted area and submit Form 0 - Test Material Receipt to inform the Organiser that they have accepted the test material. If no test material has been received by 23rd February, please contact the Organiser by e-mail (cferrer@ual.es and omalato@ual.es).
5. The participating laboratories must respect the deadline for submitting the results. Results must be reported using Form 1 - Results within 72 hours after the arrival of the test item.
6. The Organiser will evaluate the results at the end of the proficiency test, once the deadline for the receipt of results has passed. The Organiser will prepare a Preliminary Report that will be sent to the participants and uploaded to the website to show the pesticides reported,

after the revision of all the data by the Scientific Panel a Final Report will be done and the organiser will upload an electronic version on the EURL-FV website and, afterwards, send a hard copy to each participant laboratory. This report will include information regarding the design of the test, the homogeneity and stability test results, an evaluation of the participant's results as well as graphical displays of the results and any conclusions. Any other relevant information considered of value may also be included.

Amount of Test Material

Participants will receive:

- Approximately 300 g of pear test material treated with pesticides.
- Approximately 300 g of 'blank' pear test material.

Shipment of Test Materials

All test materials will be frozen and packed in polystyrene boxes surrounded by dry ice and packed into cardboard boxes.

The shipment of the test materials will start on 20th February 2012. An information message will be sent out by e-mail before shipment. Laboratories must make their own arrangements for the receipt of the package. They must inform the Organiser of any public holidays in their country/city during the delivery period given in the calendar as well as making the necessary arrangements to receive the shipment, even if the laboratory is closed.

Advice on Test Material Handling

Once received, the test material should be stored deeply frozen (-18ºC or less) prior to analysis to avoid any possible deterioration/spoilage. 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 measurement and their own reference standards for identification.

Form 0 - Test Material Receipt

Once the laboratory has received the test material, the Organiser must be notified using Form 0 in the restricted area; filling in the date of receipt, the condition of the test material, and acknowledging its acceptance. If the laboratory does not inform the Organiser by 23rd February 2012 (at the latest) via email (to cferrer@ual.es and omalato@ual.es), stating that no sample has been received, the Organiser will assume that the test material has been received and accepted.

Form 1 - Results

Laboratories must enter their results in Form 1 by accessing the restricted area on the EURL-FV web site: http://www.eurl-pesticides.eu

On this form, the laboratory should report the name of each of the pesticides detected.

Each pesticide may be reported more than once if it has been detected by more than one method or identification criteria, as long as details of each method used are also provided.

Information on the parameters and/or criteria used for detecting and reporting the pesticides found will be requested, such as deviation from expected retention time, and MS identification details.

The idea is to ascertain if the methods are used in routinely or just specifically for this test and if the identification is undertaken manually or automatically. Moreover, the range over which your method operates will be required. This is the concentration range - the minimum and maximum level of your screening method that is used to detect pesticides.

Information in this form will be saved and may be changed or updated up to 72 hours after sample arrival in the laboratory. After the deadline, results submission and/or changes to the results form will no longer be possible.

Calendar

ACTIVITY	DATE
Publishing the Calendar and Matrix on the EURL-FV Web page.	30th November 2011
Submission of Application Form by invited laboratories.	15th Dec. 2011-18th Jan 2012
Sample distribution.	20th February 2012
Submission of sample receipt and acceptance - Form 0.	As soon as sample is received
Deadline for receiving results - Form1	72 hours after receiving the sample
Preliminary Report.	March 2012
Final Report distributed to the Laboratories.	December 2012

Cost for shipment of the test material

Only those laboratories not participating in EUPT-FV 14 will have to pay the following fee for sample shipment: EU and EFTA laboratories will be charged 175€ and other laboratories will be charged 200 €. For the payment procedures, each laboratory can specify their details and requests for invoices when applying for the test. Payment details are as follows:

BANK NAME: CAJAMAR - Caja Rural Sociedad Corporativa de Crédito
BANK ACCOUNT OWNER: Universidad de Almeria
BANK ADDRESS: Office Number 990. Universidad de Almeria. Spain
ACCOUNT NUMBER: 30580130172731005000
IBAN: ES0730580130172731005000
SWIFT: CCRIES2A
CONCEPT: Invoice No. or Lab Code

Contact information

The official organising group details are as follows:

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Dr. Miguel Gamón, Senior Chemist, Laboratorio Agroalimentario, Valencia, Spain.
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Mr. Ralf Lippold, Senior Chemist, CVUA, Freiburg, Germany.
Dr. Michelangelo Anastassiades, Senior Chemist, CVUA, Stuttgart, Germany.