

EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUIT AND VEGETABLES. SCREENING METHODS 06 (EUPPT-FV-SM-06)

Pesticide Residues in Pepper Homogenate

Final Report (29th November 2014)

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EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUIT AND VEGETABLES.

SCREENING METHODS 06

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 origin¹: 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 882/2004² 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 sixth time that the EURL for pesticides in fruit and vegetables³ at the University of Almería, Spain, 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 (OfLs) to extend the scope of their methods in a cost-effective way, by using the different MS instruments/software and methods available (whether they are old or new).

Participation in this PT remains on a voluntary basis. Besides this one, 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 (EUPPT-FV16) over the same time period. Nevertheless, all FV-National Reference Laboratories (FV-NRLs) and FV-Official laboratories (FV-OfLs) 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.

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

¹Regulation (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.

²Regulation (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

³Commission 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

The EURL-FV has decided to continue its operation in these screening proficiency tests because of the good acceptance in the EURL-FV laboratory network.

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 (multiresidue methods) 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/12571/2013 (which supersedes Document No. SANCO/12495/2011) - Method validation and quality control procedures for pesticide residues analysis in food and feed.

This EUPT-FV-SM06 is aimed at all NRLs and all 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.

Only qualitative information was requested for those pesticides reported. 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.

2. TEST ITEMS

2.1 Preparation of the treated test item.

This proficiency test is based on the pesticide-residue analysis of peppers. The peppers were organically grown in Almería by the organisers in a greenhouse with passive ventilation - typically used for horticulture in the Almeria region ('raspa y amagado' type), located at the agricultural research farm belonging to the University of Almeria, in south-eastern Spain.

The soil used for the crops has an artificial layer of sand mulch on top of the soil surface; these kinds of mulched soils are known locally as 'enarenado'. They consist of two layers: a top layer of sand around 10-15 cm thick, and a bottom organic layer 2-3 cm thick, placed directly over the indigenous soil.

Drip fertigation was used to supply both water and fertilizers to the crop.

The pesticides used to spike the pepper test item 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 item for this EUPT-FV-SM06 were mainly chosen taking into account the following considerations:

- That they were not included in the EU-Coordinated Multiannual Control Programme for 2012 (Regulation (EC) 788/2012).
- That they had particularly acute toxicity and/or had low ARfD values.

Table 2.1 shows the 21 pesticides present in the pepper sample. The pesticide treatments were carried out post-harvest using either commercial formulation in micro-spray solutions or using standard solutions. The test item was frozen (using liquid nitrogen) and chopped. The frozen minced peppers were mixed in a constantly-spinning container until a homogeneous item was obtained. Finally, 300 g portions of the well-mixed homogenate were weighed out into screw-capped polyethylene plastic bottles, sealed and stored in a freezer at about -20 °C prior to distribution to participants.

Table 2.1 Pesticides present in the sample.

Present Pesticides		
Atrazine	Ethoprophos	HCB
Cadusafos	Fenpropidin	Metosulam
Carbetamide	Fipronil	Phorate
Demeton-S-methylsulfone	Flubendiamide	Prosulfocarb
Dinocap	Fluometuron	Secbumeton
Dodemorph	Fuberidazole	Spiromesifen
Endrin	Furathiocarb	Terbufos

2.2 Preparation of "blank" test item.

The peppers used for the production of the blank item were organically grown in the same field as the test item. A homogenate was prepared in the same way as the treated test item described previously.

2.3 Homogeneity and stability tests.

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 item

was analysed in order to identify the present pesticides, which were consistently confirmed to be above the Organiser's LODs.

To confirm the homogeneity of the test item sent, ten 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 item 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 item No.	017 A	017 B	027 A	027 B	056 A	056 B	072 A	072 B	082 A	082 B	098 A	098 B	108 A	108 B	124 A	124 B	141 A	141 B	165 A	165 B	A. Cc (mg/kg)	RSD (%)
Atrazine	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.059	5.0
Cadusafos	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.038	10.4
Carbetamide	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.068	7.8
Demeton-S-methylsulfone	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.058	10.9
Dinocap	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.218	10.8
Dodemorph	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.086	10.7
Endrin	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.038	13.1
Ethoprophos	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.049	8.2
Fenpropidin	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.074	6.5
Fipronil	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.048	9.2
Flubendiamide	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.163	13.1
Fluometuron	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.060	3.6
Fuberidazole	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.041	6.3
Furathiocarb	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.046	14.5
HCB	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.039	10.0
Metosulam	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.031	8.2
Phorate	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.058	12.0
Prosulfocarb	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.057	10.5
Secbumeton	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.073	13.2
Spiromesifen	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.106	10.5
Terbufos	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.030	14.8

I: Identified

A. Cc: Average Concentration

RSD: Relative Standard Deviation

Further analyses to test for stability were performed following varying time lapses. Two bottles, chosen randomly were analysed, the first one, when the test items were shipped, and the second one a few days after the deadline for receipt the participants' results. There will be a further analysis of the first sample 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 item

All the present pesticides in the samples demonstrated sufficient stability even after reproducing the 48 h delivery conditions. All were reported on each occasion.

Table 2.3.2 Stability tests performed.

Test item Number	Stability tests					
	093 Shipment Day (24 th February)	093 Shipment Day (24 th February)	093 48 h later Shipment Day (26 th February)	093 48 h later Shipment Day (26 th February)	131 Few days after deadline 5 th March	131 Few days after deadline 5 th March
Atrazine	I	I	I	I	I	I
Cadusafos	I	I	I	I	I	I
Carbetamide	I	I	I	I	I	I
Demeton-S-methylsulfone	I	I	I	I	I	I
Dinocap	I	I	I	I	I	I
Dodemorph	I	I	I	I	I	I
Endrin	I	I	I	I	I	I
Ethoprophos	I	I	I	I	I	I
Fenpropidin	I	I	I	I	I	I
Fipronil	I	I	I	I	I	I
Flubendiamide	I	I	I	I	I	I
Fluometuron	I	I	I	I	I	I
Fuberidazole	I	I	I	I	I	I
Furathiocarb	I	I	I	I	I	I
HCB	I	I	I	I	I	I
Metosulam	I	I	I	I	I	I
Phorate	I	I	I	I	I	I
Prosulfocarb	I	I	I	I	I	I
Sebumeton	I	I	I	I	I	I
Spiromesifen	I	I	I	I	I	I
Terbufos	I	I	I	I	I	I

I: Identified

2.4 Distribution of test items and protocol to participants

Approximately 300 g of treated pepper homogenate together with another 300 g of 'blank' pepper homogenate were shipped to participants on 24th February 2014. The deadline for results submission to the Organiser was 72 hours after receipt of the test item. Participants were asked to report all the pesticides that they detected.

Laboratories were asked to screen the test items 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 test item 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-SM06 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 'non-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 item treatment, or (ii) not identified 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.

3.1.2 Non-Reported Pesticides

These were considered as any pesticide present in the sample but not reported by the lab even though the Organiser had confirmed it as present in the test item above 0.010 mg/kg and it was reported by the majority of participants.

4. RESULTS

4.1 Summary of reported results

Sixty-eight laboratories agreed to participate in this sixth proficiency test on screening methods. Sixty-seven laboratories submitted results on time and only one finally didn't participate. 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 on the EUPT-SM06 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	Reported		Non Reported	
	No. of laboratories	% of laboratories *	No. of laboratories	% of laboratories *
Atrazine	60	90	7	10
Cadusafos	64	96	3	4
Carbetamide	37	55	30	45
Demeton-S-methylsulfone	57	85	10	15
Dinocap	25	37	42	63
Dodemorph	39	58	28	42
Endrin	54	81	13	19
Ethoprophos	66	99	1	1
Fenpropidin	56	84	11	16
Fipronil	62	93	5	7
Flubendiamide	33	49	34	51
Fluometuron	35	52	32	48
Fuberidazole	39	58	28	42
Furathiocarb	55	82	12	18
HCB	52	78	15	22
Metosulam	34	51	33	49
Phorate	50	75	17	25
Prosulfocarb	56	84	11	16
Secbumeton	35	52	32	48
Spiromesifen	51	76	16	24
Terbufos	53	79	14	21

* The % of laboratories is calculated based on the total number of laboratories submitting results (67).

Other compounds were identified and quantified by the organizer at concentrations below 0.010 mg/kg:

- Atrazine-desethyl-2-hydroxy
- Carbofuran
- Difenoconazole
- Dimefuron
- Endrin ketone
- Fenamiphos
- Fenamiphos sulfoxide
- Fipronil sulfide
- Fipronil sulfone
- Phorate sulfoxide
- Terbufos sulfoxide
- Triadimenol

Two of these compounds were present in the blank at concentrations below 0.010 mg/kg, difenoconazole and triadimenol.

4.1.1 Other Reported Pesticides

Many laboratories reported additional pesticides to those present in the test item. These reported pesticides are presented in Table 4.1.1.

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

LABORATORY CODE	OTHER REPORTED PESTICIDES
Lab 002	Chlormephos, Diniconazole, Dioxation, Disulfoton, Diuron, Endosulfan Beta, Etrifos, Fenarimol, Fenitrothion, Terbucarb, Trifloxystrobin
Lab 003	Flonicamid, Terbumeton
Lab 004*	Azoxystrobin, Buprofezin, Chlorpyrifos
Lab 006*	Binapacryl, Promecarb, Simazine
Lab 007*	Dinoseb, Fenobucarb, Resmethrin, Terbucarb
Lab 009	Chlorothalonil, Lenacil, Spiroxamine, Tridemorph
Lab 010*	Diuron
Lab 013	3-Trifluormethylaniline, Carbofuran(7-phenol), Dinobuton, Prometon
Lab 014	Terbumeton
Lab 018	MCPB
Lab 020	Formetanate
Lab 033*	Diuron, Methoxychlor, Tetramethrin
Lab 039*	Chlorpyrifos-ethyl
Lab 040	Prometon
Lab 044*	Diuron, Fenpyroximate
Lab 047	Formetanate
Lab 050	Fosthiazate, Prometon, Tefluthrin, Terbumeton
Lab 051*	Vernolate
Lab 054	Spiromesifen
Lab 055	Diuron, Terbumeton
Lab 056	Ethopenprox, Exaltolide, Tebufenpyrad
Lab 057*	Propazine
Lab 060	Cyprodinil
Lab 062	Hexachlorocyclohexane
Lab 064	Binapacryl

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

Those pesticides reported were analysed by the Organiser, but none was identified after repeated analyses.

4.1.2 Non-Reported Pesticides

In Table 4.1.1, the number and percentage of laboratories not reporting each of the pesticides present in 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 pepper test item at different levels, in the range between 0.030 mg/kg and 0.200 mg/kg according to the homogeneity table 2.3.1, most of them in concentrations lower than 0.100 mg/kg.

Other compounds have been identified and quantified by the organizer at concentrations below 0.010 mg/kg:

- Atrazine-desethyl-2-hydroxy
- Carbofuran
- Difenoconazole
- Dimefuron
- Endrin ketone
- Fenamiphos
- Fenamiphos sulfoxide
- Fipronil sulfide
- Fipronil sulfone
- Phorate sulfoxide
- Terbufos sulfoxide
- Triadimenol

Two of these compounds were present in the blank at concentrations below 0.010 mg/kg, difenoconazole and triadimenol.

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 results each laboratory reported. Table 4.3.1 classifies the laboratories according to the number of present pesticides reported.

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

Laboratory Code	No of Reported Pesticides	% of Reported Pesticides	Other Reported Pesticides	
			Not Confirmed by the Organiser	Confirmed by the Organiser (below 0.010 mg/kg)
Lab037	21	100		6
Lab024*	21	100		2
Lab016*	21	100		1
Lab038	21	100		1
Lab015	21	100		
Lab057*	21	100	1	2
Lab026	20	95		6
Lab011	20	95		3
Lab029	20	95		1
Lab035	20	95		
Lab046*	20	95		
Lab053	20	95		
Lab044*	20	95	2	4
Lab007*	20	95	4	3
Lab042	19	90		2
Lab008	19	90		1
Lab027	19	90		1
Lab058	19	90		
Lab060	19	90	1	5
Lab003	19	90	2	1

Laboratory Code	No of Reported Pesticides	% of Reported Pesticides	Other Reported Pesticides	
			Not Confirmed by the Organiser	Confirmed by the Organiser (below 0.010 mg/kg)
Lab013	19	90	4	7
Lab036	18	86		1
Lab032	18	86		
Lab048	18	86		
Lab055	18	86	2	
Lab006*	18	86	3	
Lab009	18	86	4	4
Lab001	17	81		2
Lab041*	17	81		2
Lab010*	17	81	1	
Lab050	17	81	4	2
Lab031	16	76		3
Lab030	16	76		2
Lab039	16	76	1	6
Lab018	16	76	1	3
Lab047	16	76	1	1
Lab051*	16	76	1	1
Lab040	16	76	1	
Lab065	15	71		5
Lab052	15	71		4
Lab049*	15	71		2
Lab025*	15	71		1
Lab028*	15	71		1
Lab067	15	71		
Lab064	15	71	1	
Lab014	14	67	1	2
Lab066	13	62		
Lab020	13	62	1	2
Lab062	13	62	1	
Lab043	12	57		1
Lab012	12	57		
Lab004*	12	57	3	6
Lab056	12	57	3	
Lab054	11	52	1	
Lab021	10	48		
Lab045	10	48		
Lab034	9	43		
Lab059	9	43		
Lab068	9	43		
Lab033*	9	43	3	2
Lab023	6	29		
Lab002	6	29	11	1
Lab019*	5	24		1
Lab017	5	24		
Lab022	5	24		
Lab063	3	14		3
Lab061	3	14		1

* 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 on the EUPT-SM06 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 item

Pesticide	Total No. of Reports	GC	Full-scan GC	LC	Full-scan LC
Atrazine	60	29	14	35	13
Cadusafos	64	32	13	38	12
Carbetamide	37	9	7	31	11
Demeton-S-methylsulfone	57	5	1	56	16
Dinocap	25	1	1	25	6
Dodemorph	39	22	15	20	6
Endrin	54	52	20	2	0
Ethoprophos	66	36	16	33	12
Fenpropidin	56	20	11	39	10
Fipronil	62	35	13	31	10
Flubendiamide	33	1	0	32	8
Fluometuron	35	4	3	31	14
Fuberidazole	39	11	6	30	13
Furathiocarb	55	16	10	43	14
HCB	52	50	22	2	0
Metosulam	34	0	0	35	11
Phorate	50	34	16	19	5
Prosulfocarb	56	16	8	42	16
Secbumeton	35	22	17	14	10
Spiromesifen	51	29	13	25	10
Terbufos	53	35	18	20	6

Note: The number of reports for each of the pesticides could be different to the reports shown in Table 4.1.1 because a particular laboratory might have analyzed one pesticide 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 present in 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	17	81
Lab002	6	29
Lab003	19	90
Lab004*	12	57
Lab006*	18	86
Lab007*	20	95
Lab008	19	90
Lab009	18	86
Lab010*	17	81
Lab011	20	95
Lab012	12	57
Lab013	19	90
Lab014	14	67
Lab015	21	100
Lab016*	21	100
Lab017	5	24
Lab018	16	76
Lab019*	5	24
Lab020	13	62
Lab021	10	48
Lab022	5	24
Lab023	6	29
Lab024*	21	100
Lab025*	15	71
Lab026	20	95
Lab027	19	90
Lab028*	15	71
Lab029	20	95
Lab030	16	76
Lab031	16	76
Lab032	18	86
Lab033*	9	43
Lab034	9	43
Lab035	20	95
Lab036	18	86
Lab037	21	100
Lab038	21	100
Lab039*	16	76
Lab040	16	76
Lab041*	17	81
Lab042	19	90
Lab043	12	57
Lab044*	20	95
Lab045	10	48

Laboratory Code	Number of Present Pesticides Reported	% of Present Pesticides Reported
Lab046*	20	95
Lab047	16	76
Lab048	18	86
Lab049*	15	71
Lab050	17	81
Lab051*	16	76
Lab052	15	71
Lab053	20	95
Lab054	12	57
Lab055	18	86
Lab056	12	57
Lab057*	21	100
Lab058	19	90
Lab059	9	43
Lab060	19	90
Lab061	3	14
Lab062	13	62
Lab063	3	14
Lab064	15	71
Lab065	15	71
Lab066	13	62
Lab067	15	71
Lab068	9	43

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

5. CONCLUSIONS

Sixty-eight laboratories agreed to participate in this sixth proficiency test on screening methods. Sixty-seven laboratories submitted results on time and only one finally didn't participate. Eighteen of the laboratories which applied were National Reference Laboratories for Fruit and Vegetables (marked with an asterisk on the graphs and tables) representing twenty one Member States. In addition to these, 1 EFTA country (Switzerland) and four non-EU/EFTA countries (China, Egypt, Serbia and Turkey) participated in this European Union Proficiency Test.

Most laboratories analysed the test item 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 1062 detections, 427 were made using full-scan, meaning 40 % of detections (203 by full scan LC techniques and 224 by full scan GC techniques).

Six of the 67 laboratories were able to detect all 21 present pesticides in the pepper test item. 13 laboratories detected less than 50 % of the pesticides present.

Sixty-seven percent of the laboratories (45 laboratories) that reported results were able to find more than 70 % of the present pesticides in the sample.

Forty-one laboratories reported 12 different pesticides present in the sample below 0.010 mg/kg. Two of these pesticides (difenoconazole and triadimenol) were found in the blank items at concentrations below 0.010 mg/kg.

Twenty-five participants reported 46 different pesticides which were not present in the peppers. 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 reported pesticide was 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 reported 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 EUPT-SM06 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 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 SANCO Document/12571/2013 (Supersedes SANCO/12495/2011) Analytical Quality Control and Method Validation Procedures for Pesticide Residues Analysis in Food and Feed.

Next year, broccoli matrix test item will be used. If laboratories have a particular interest in specific matrices, they should inform the EURL-FV and their suggestions will be evaluated. There will be no target list, as was the case in this test.

7. REFERENCES

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- ISO/IEC 17043:2010 Conformity assessment - General requirements for proficiency testing.
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8. ACKNOWLEDGEMENTS

The Organiser is grateful to the European Commission for funding this 6th 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

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Laboratory Code Total No of Reporting Laboratories = 67	Evaluated Pesticides (21)																				Reported Pesticides by Laboratory	% of Reported Pesticides by Laboratory		
	Atrazine	Cadusafos	Carbetamide	Demeton-S-methylsulfone	Dinocap	Dodemorph	Endrin	Ethoprophos	Fenpropidin	Fipronil	Flubendiamide	Fluometuron	Fuberidazole	Furathiocarb	HCB	Metosulam	Phorate	Prosulfocarb	Secbumeton	Spiromesifen	Terbufos			
Lab001	R	R		R	R	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	17	81	
Lab002	R	R						R	R								R			R	R	6	29	
Lab003	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	19	90	
Lab004*	R	R		R			R	R	R	R				R	R	R	R	R	R	R	R	R	12	57
Lab006*	R	R	R	R		R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	18	86	
Lab007*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	20	95	
Lab008	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	19	90	
Lab009	R	R	R	R		R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	18	86	
Lab010*	R	R	R	R		R	R	R	R	R			R	R	R	R	R	R	R	R	R	R	17	81
Lab011	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	20	95	
Lab012	R	R	R			R	R	R	R	R				R			R	R	R	R	R	R	12	57
Lab013	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	19	90	
Lab014	R	R	R			R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	14	67	
Lab015	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	21	100	
Lab016*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	21	100	
Lab017	R					R	R						R						R			5	24	
Lab018	R	R	R	R	R		R	R	R	R			R	R	R	R	R	R	R	R	R	16	76	
Lab019*							R	R	R	R			R						R			5	24	
Lab020	R	R		R			R	R	R	R		R		R	R	R	R	R	R	R	R	13	62	
Lab021	R	R		R		R		R				R	R	R	R	R	R	R	R	R	R	10	48	
Lab022	R					R	R	R				R		R								5	24	
Lab023	R						R	R					R	R	R	R	R	R	R	R	R	6	29	
Lab024*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	21	100	
Lab025*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	15	71	
Lab026	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	20	95	
Lab027	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	19	90	
Lab028*	R	R		R	R	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	15	71	
Lab029	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	20	95	
Lab030	R	R	R	R			R	R	R	R	R		R	R	R	R	R	R	R	R	R	16	76	
Lab031	R	R	R	R		R	R	R	R	R			R	R	R	R	R	R	R	R	R	16	76	
Lab032	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	86	
Lab033*	R	R	R			R	R	R											R	R	R	9	43	
Lab034	R		R			R	R	R	R				R	R	R	R	R	R	R	R	R	9	43	
Lab035	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	20	95	
Lab036	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	86	
Lab037	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	21	100	
Lab038	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	21	100	
Lab039*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	76	
Lab040	R	R	R	R			R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	76	
Lab041*	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	17	81	
Lab042	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	19	90	
Lab043	R	R	R	R			R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	12	57	
Lab044*	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	20	95	
Lab045	R	R	R			R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	10	48	
Lab046*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	20	95	
Lab047	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	76	
Lab048	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	86	
Lab049*	R	R	R			R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	15	71	
Lab050	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	17	81	
Lab051*	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	76	

APPENDIX 1. Results

Laboratory Code Total No of Reporting Laboratories = 67	Evaluated Pesticides (21)																			Reported Pesticides by Laboratory	% of Reported Pesticides by Laboratory		
	Atrazine	Cadusafos	Carbetamide	Demeton-S-methylsulfone	Dinocap	Dodemorph	Endrin	Ethoprophos	Fenopropidin	Fipronil	Flubendiamide	Fluometuron	Fuberidazole	Furathioicarb	HC _B	Metosulam	Phorate	Prosulfocarb	Sectobuton	Spiromesifen	Terbufos		
Lab052	R	R	R		R	R	R	R	R					R	R	R		R	R	R	R	15	71
Lab053	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	20	95	
Lab054	R	R		R		R	R				R		R	R	R	R	R	R	R	R	12	57	
Lab055	R	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	18	86	
Lab056	R					R		R	R	R				R	R	R	R	R	R	R	R	12	57
Lab057*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	21	100	
Lab058	R	R	R	R		R	R	R	R	R	R	R	R	R		R	R	R	R	R	19	90	
Lab059	R	R	R	R		R	R		R	R		R				R						9	43
Lab060	R	R	R	R	R	R		R	R	R	R	R		R	R	R	R	R	R	R	19	90	
Lab061		R						R	R													3	14
Lab062	R	R				R	R	R	R					R		R	R	R	R	R	R	13	62
Lab063	R		R				R															3	14
Lab064	R	R	R	R		R		R	R	R			R	R		R	R	R	R	R	R	15	71
Lab065	R	R	R	R			R	R	R	R			R	R		R	R	R	R	R	R	15	71
Lab066	R	R		R		R		R	R	R	R	R	R	R		R	R					13	62
Lab067	R	R		R		R	R	R	R	R			R	R		R	R	R	R	R	R	15	71
Lab068	R	R		R			R		R			R	R	R			R					9	43
Reported Pesticides	60	64	37	57	25	39	54	66	56	62	33	35	39	55	52	34	50	56	35	52	53		
% of Reported Pesticides	90	96	55	85	37	58	81	99	84	93	49	52	58	82	78	51	75	84	52	78	79		

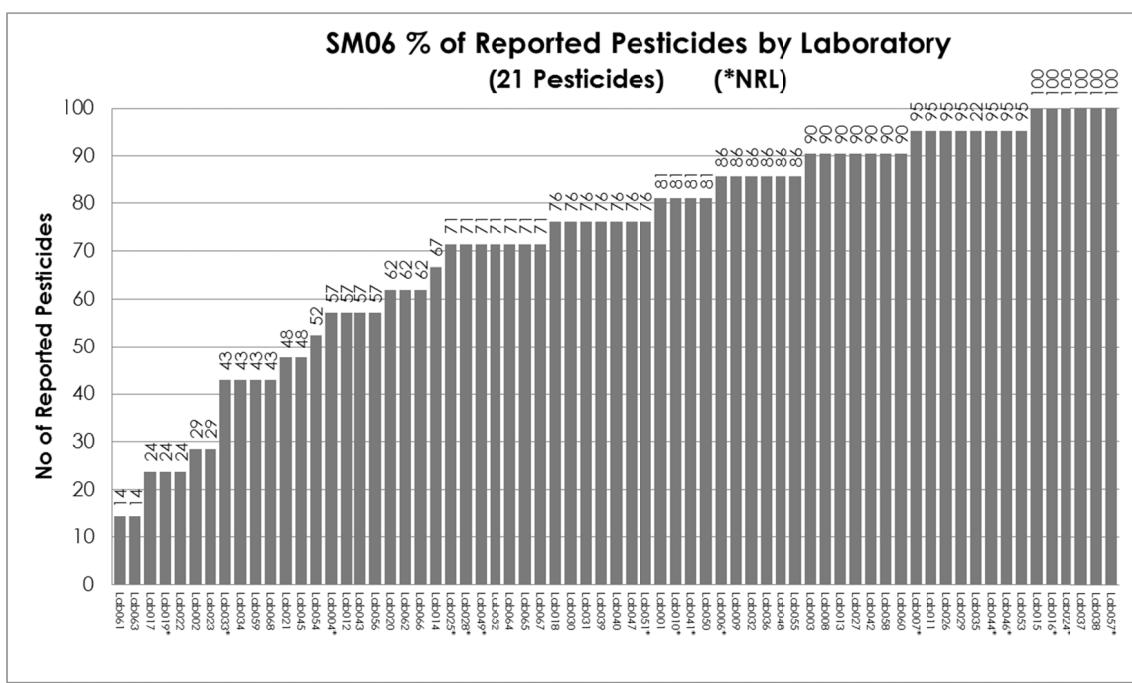
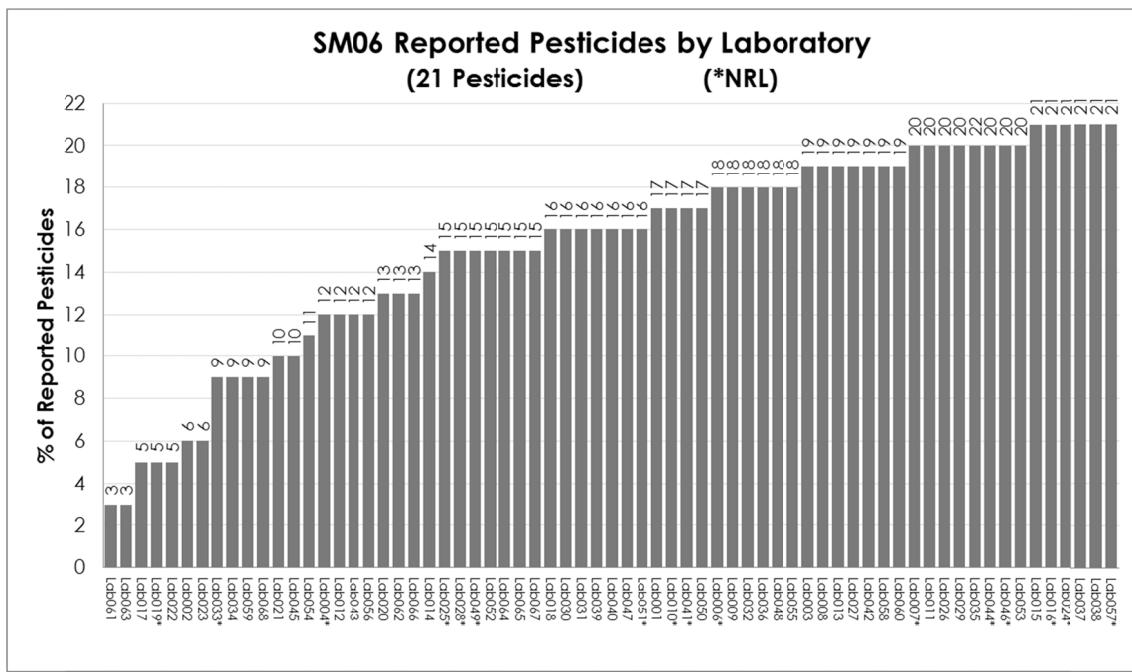
R: Reported pesticide

*NRLs from EU

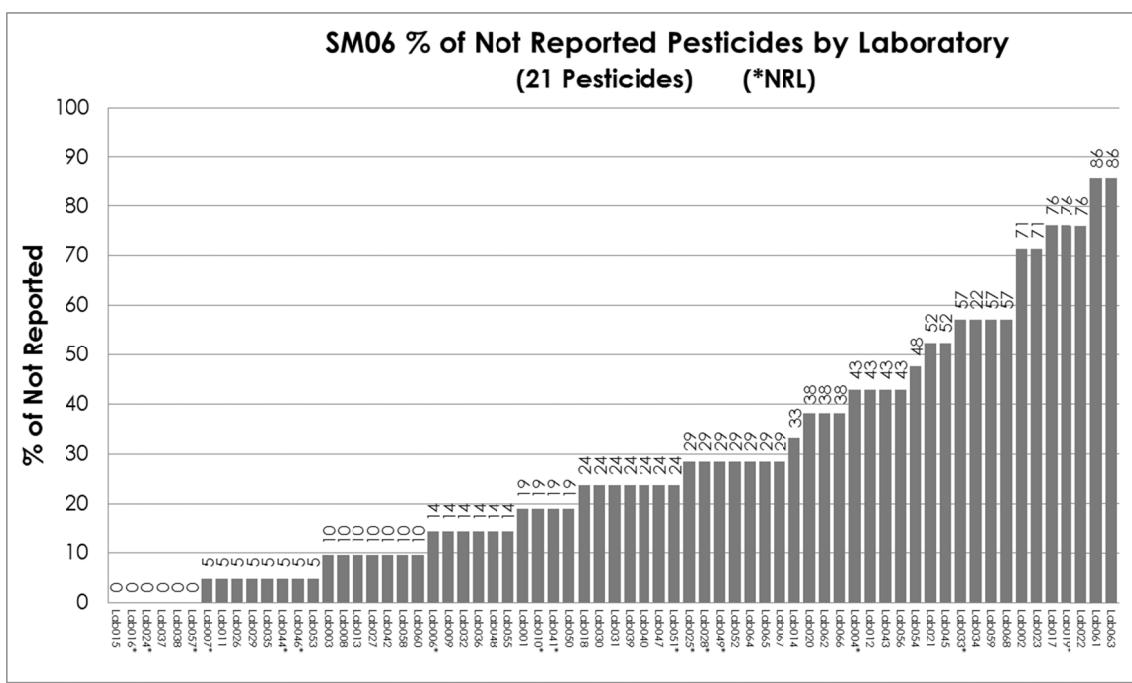
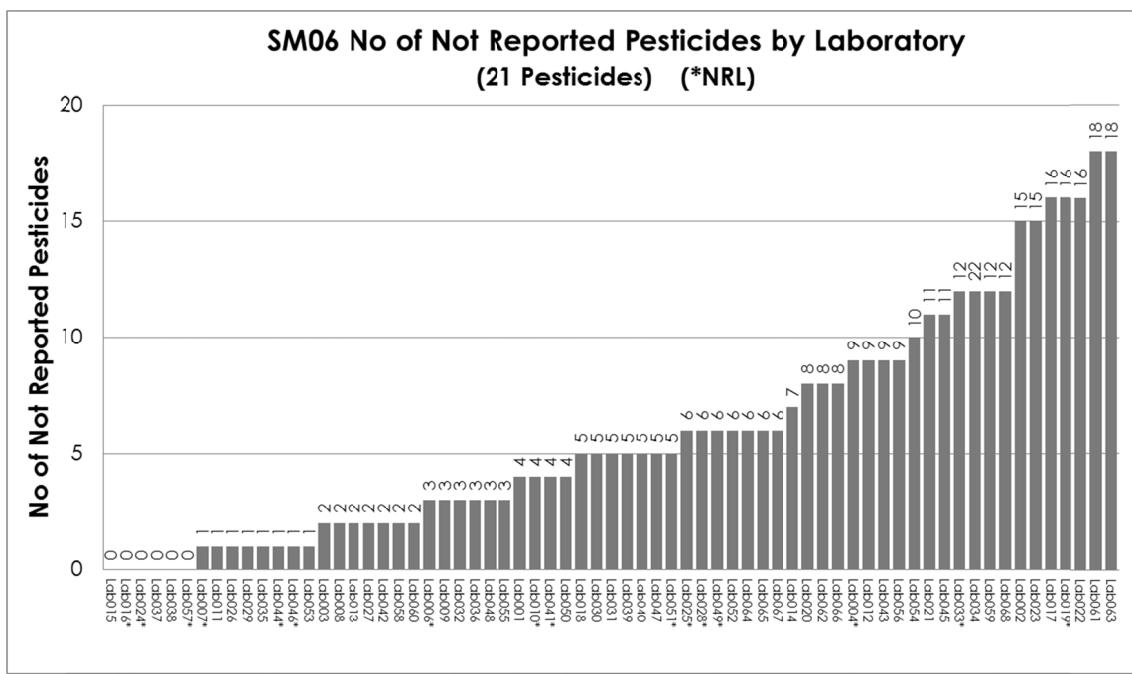
Other compounds have been reported or identified and quantified by the organizer at concentrations below 0.010 mg/kg (these compounds have not been included in the table):

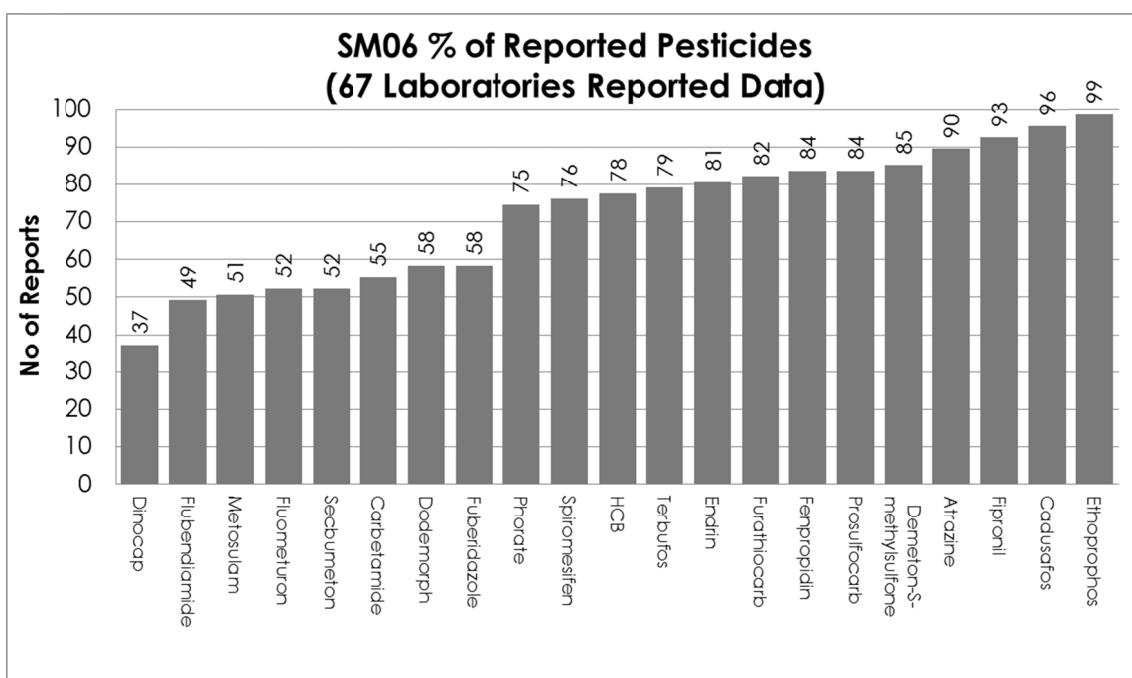
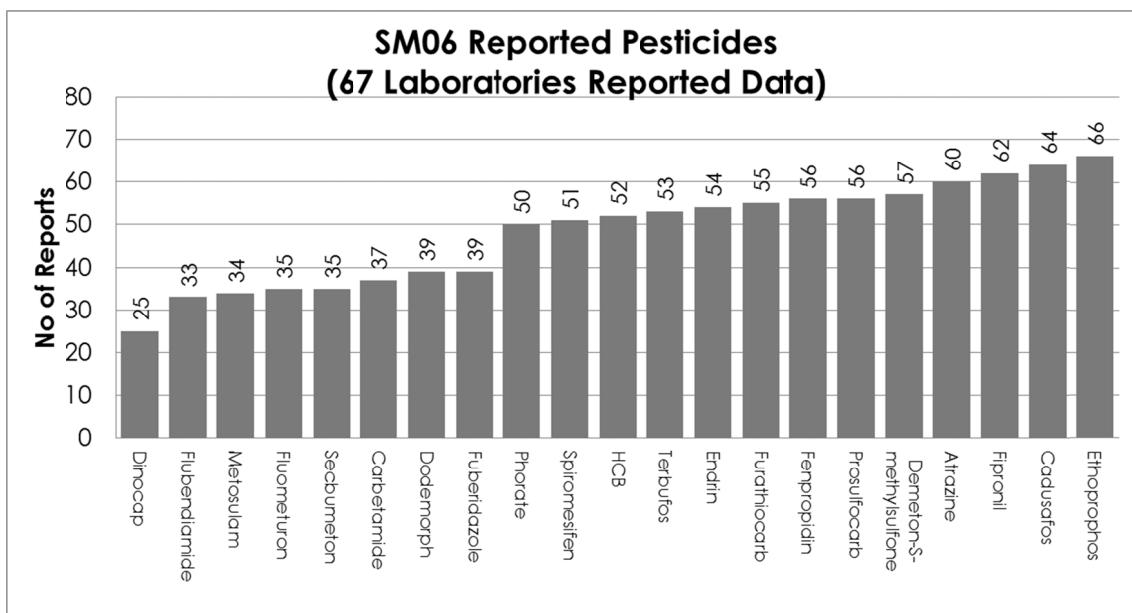
- Atrazine-desethyl-2-hydroxy
- Carbofuran
- Difenoconazole
- Dimefuron
- Endrin ketone
- Fenamiphos
- Fenamiphos sulfoxide
- Fipronil sulfide
- Fipronil sulfone
- Phorate sulfoxide
- Terbufos sulfoxide
- Triadimenol

APPENDIX 2. Graphical Representations



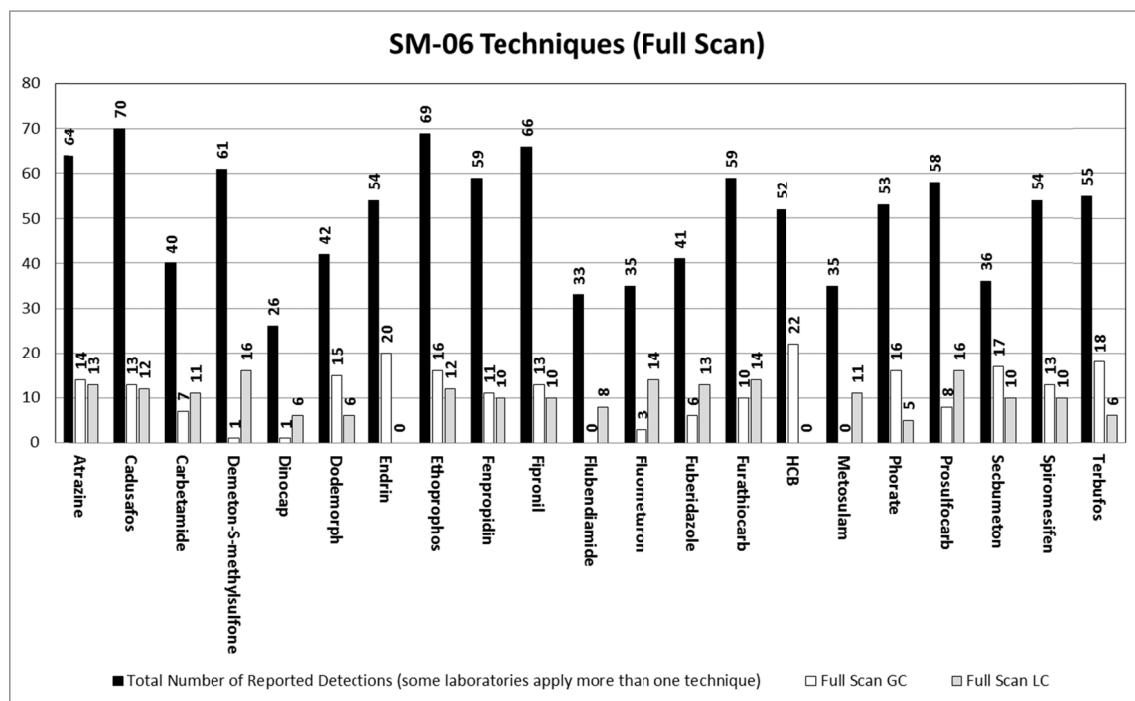
APPENDIX 2. Graphical Representations





APPENDIX 2. Graphical Representations

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



APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/identification was done?	RT Deviation (s)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Ionization Mode	Routine Method?	Routine Scope?	Screening Detection Limit (SDL)? (mg/Kg)	Instrument Scan	Extraction Solvent	Agitation Mode	Injection Volume (µL)	Software	Compounds in method (pesticides Only)	Standard Solution Frequency				
001 [SANCO/1257/2013]	Fully identified	3 Transitions	GC MS/MS	QQQ	EI +	Yes	Yes	0.01	No	Agilent 7000	10	ACN	No	1	None	HP5-MS	1	Mass Hunter	350	Daily	
002 [SANCO/1257/2013]	Fully identified		LC MS/MS	QQQ	ESI +	Yes	Yes			Agilent 6410	10	ACN/ACN		1	SPE	C18 4 micras	5	Mass Hunter	221		
003 [SANCO/1257/2013]	Fully identified		LC MS/MS		ESI +	Yes	No				10	ACN	Yes	Manual	1	DSPE					
004 Tentatively detected	90	5 fragmentations	LC MS/MS	Q-TOF	ESI +	Yes	Yes		Yes	Agilent 6520	10	ACN	Yes	Mechanical	15	DSPE	Synergie Fusion RF80A,	8	Mass Hunter	>800	3 Months
005 Fully identified [SANCO/1257/2013]	0	2 Transitions	GC MS/MS	QQQ	EI +	Yes	Yes		No	Agilent 10	10	ACN	Yes	Mechanical	15	DSPE	HP5-MS	2	Mass Hunter	>300	Every batch
006 Fully identified [SANCO/1257/2013]			GC MS/MS	QQQ	EI +	Yes	Yes		No	Agilent 15	Acetone/DCM/PE	No	Mechanical	1	None	DB5	1	Mass Hunter	176	No screening methods	
007 Fully identified [SANCO/1257/2013]	30	5 confirmed on ToF, LC-MS/MS, GC-MS	LC TOF	Q-TOF	Jet Stream	+	No	No	NA	Yes	Agilent 6530 accurate mass Q-ToF LCMS	10	ACN		0.5	None	C18	3	Mass Hunter	550	NA
008 Fully identified [SANCO/1257/2013]			GC MS/MS				Yes	Yes				13	ACN	No	Mechanical	None					
009 Fully identified [SANCO/1257/2013]	+/- 0.2 min	<5 acc. m/z + isotope + fragment	LC MS/MS	Q-TOF	ESI +	No	No	0.05-0.1	Yes	API 5600 Q-Tof	10	ACN	No	Mechanical	5	None	C18 Aqua Phenomenex	20	Both	600	2 Months
010 Fully identified [SANCO/1257/2013]			LC MS/MS	QQQ	ESI +	Yes	Yes	0.010		Waters Xevo TQ	10	ACN	No	Manual	2	DSPE	Acuity BEH C18	10	Waters	195	Daily
011 Tentatively detected	3	2 Transitions	LC MS/MS	QQQ	ESI +	Yes	Yes	0.005-0.1	No	Xevo TQS	10	ACN	No	2	None	C18	2.5	Masslynx	300	Every batch	
012 Fully identified [SANCO/1257/2013]	0.2	2 Transitions	LC MS/MS	Q-Q	ESI +	No	No	0.005-0.1	Yes	TripleTOF 10	ACN	No		2	None	C18	2	PeakView	620	Every batch	
013 Tentatively detected	10	2	GC MS/MS	QQQ	EI +	Yes	Yes	0.01		Pegasus 4D	10	ACN	No		2	DSPE	HP5-MS	2	ChromatOF	According to NIST 2.0	Every batch
014 Fully identified [SANCO/1257/2013]	5	10 Accurate mass	GC MS/MS	Q-TAP	ESI +	Yes	Yes	0.01	Yes	977	10 Cyclohexane/EtOAc/Acetone	No	3	DSPE	HP5-MS	1	Mass Hunter	~500	Daily		
015 Fully identified [SANCO/1257/2013]	0		LC MS/MS	Q-TAP	ESI +	Yes	Yes	0.01	No	ABSciex 4000 QTrap	10	ACN	Yes	1	None	Phenomenex Synergie Fusion RF 2.5 µm 50x2.0	55	MultiQuant	~250	Always	

APPENDIX 3. Methods used by participants for detecting pesticides.

Atrazine											
Laboratory Code *NRL	How was the detection/differentiation done?	RT Deviation (s)	MS Tolerance (ppm)	Other differentiation Details	Chromatographic Technique	Detector	Polarity	Routine Method?	Screening Detection Limit (SDL) (mg/kg)	Instrument Model	Extraction Solvent
018 [SANCO/1257/2013]	Fully identified			LC MS/MS QQQ	ESI + Yes Yes	0.010	No Qtrap3200	10 ACN	No Manual	DSPE C18	5 Analyst 205 Every batch
020 [SANCO/1257/2013]	Fully identified	2 Transitions		GC MS/MS QQQ	EI + Yes Yes	0.005	No Quantum XLS Ultra	10 ACN	No Mechanical	DSPE RTX5-MS	1 TraceFinder 300 Always
021 [SANCO/1257/2013]	Fully identified	2 Transitions		LC MS/MS QQQ	ESI + Yes Yes	0.005	No Waters Xevo TQ	15 ACN	No Mechanical	5 None HSS T3	10 Masslynx 400 Every batch
023 [SANCO/1257/2013]	Fully identified			GC MS IT	EI No	No	Yes Varian 240MS/15 PE	No	1 Liquid-Liquid Partitioning VF-5MS	3 Auto 127	
024 Tentatively detected	3 5	2 Transitions		LC MS Q-TOF	ESI + No	0.01-0.1	Yes Agilent 6550 QTOF	10 ACN	No Manual	1 QueChERS without PSA Atlantis 3, 2.1x100 mm, 3µm	2 Mass Hunter 1660
025 [SANCO/1257/2013]	Fully identified	2 0	2 Transitions	GC MS/MS QQQ	EI + Yes Yes	0.02	No Varian 320	50 Acetone	No Mechanical	2 Liquid-Liquid Partitioning DB5	8 Star 146
026 Tentatively detected	0.01 5	Accurate mass		LC MS/MS Q-Cirbitrap	ESI + Yes Yes	0.002	Yes Q Exactive 10	10 Acetone/DCM/PE	No Mechanical	10 DSPE BEH C18	5 700 Always
027 [SANCO/1257/2013]	Fully identified	0	2 Transitions	LC MS/MS QQQ	ESI + Yes Yes	0.010	No Xevo 10	Agilent 6890/5973N	No Mechanical	1 None C18	2 Waters 286
028 [SANCO/1257/2013]	Fully identified	0.1 0	no	GC MS Q	EI + Yes Yes	0.01	No Agilent 6890/5973N	10 ACN	No Manual	2 HP-5MS Pursuit XRs Ultra	2 Agilent ChemStation 184
029 [SANCO/1257/2013]	Fully identified		2 Transitions	LC QQQ	ESI + Yes Yes	0.01	No API 5500	10 ACN	Yes Mechanical	15 DSPE Pursuit XRs Ultra	3 Both 550 Every batch
030 [SANCO/1257/2013]	Fully identified	max 0.5%	1 5 ion ratios	GC MS Q	EI + Yes Yes	0.01	Yes Varian 4000	10 ACN	No Mechanical	1 SPE DB5MS	Rxi 5ms 5% diphenyl / 95% dimethyl polysiloxane 1 Varian Ms Workstation 196
031 [SANCO/1257/2013]	Fully identified			LC MS/MS QQQ	EI + Yes Yes	0.01	Yes Trace DSQ	15 ACN	Yes Manual	1 SPE DB5MS	0.8 Xcalibur 350 Every batch
032 [SANCO/1257/2013]	Fully identified		2 Transitions							HSS T3 2 Analyst 404	404 Every batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/denification done?	RT Deviation (s)	MS Tolerance (ppm)	Other details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Sample Weight (g)	Instrument Model	Full Scan	Extraction Solvent	pH adjusted	Agitation Mode	Agitation time (min)	Injection Volume (μL)	Compounds in method (pesticides Only)	Standard Solution Frequency	Atrazine			
																			Column Type	Software		
033	Fully identified (SANCO/1257/2013)	2 Transitions	GC	MS/MS	QQQ	EI + Yes	Yes	No	Varian 320	7.5	Acetone/DCM/PE	No	Manual	1	Liquid-Liquid Partitioning	DB5	1	MS Workstation				
035	Fully identified (SANCO/1257/2013)	2.5%	2 Transitions	LC	MS/MS	QQQ	EI + Yes	Yes	0.01	No 1100 MS API 3000	Agilent 10	ACN	Yes		15	DSPE	C18 50x2mm	10	Both	method 550	Always	
036	Fully identified (SANCO/1257/2013)		GC	MS/MS	QQQ	EI + Yes	Yes	0.01	No Agilent 7000B	5	ACN	Yes		3	None	HP-5MS UI	2	Mass Hunter	550	Daily		
037	Tentatively detected <30	5	isotope spectrum	LC	MS	Orbitrap	ESI + No	No	0.01	Yes	Excative	AcN/1% acetic acid	No	Mechanical	30	Liquid-Liquid Partitioning	C18 Atlantis	5	ToxiD	600	Every batch (100+ pesticides)	
038	Fully identified (SANCO/1257/2013)	<20	2 Transitions	LC	MS/MS	QQQ	EI + Yes	Yes	0.05	Yes Leco Pegasus IV	AcN/1% acetic acid	No	Mechanical	30	Liquid-Liquid Partitioning + DSPE	RTX-Clpesticides	10	MetAlign + ChromatOF	560	Every batch (235 pesticides)		
039	Tentatively detected 0.48		Rev. Match	GC	MS	IT	EI + Yes	Yes	0.02	Yes Varian Saturn 2000	15	Acetone/DCM/PE	No	Mechanical	5	DSPE	C18	10	Analyst		Daily	
040	Fully identified (SANCO/1257/2013)	0	+4.2 ppm	LC	MS	Q-TOF	ESI + No	Yes	0.01	Yes Wafers Xevo Q-ToF (1st gen)	10	EtOAc	Yes	Mechanical	4	None	Acquity BEH C18	3	TargetLynx + MassFragment tool	500	Always	
041	Tentatively detected -4	5	GC-QTOF	LC	MS	Q-TOF	ESI + No	Yes		Yes Ifunnel QTOF 10 LC/MS	10	ACN	No		2	DSPE	C18	5	Mass Hunter	1700	Always	
042	Fully identified (SANCO/1257/2013)		2 Transitions	LC	MS/MS	QQQ	ESI + No	No	0.01	No Agilent 6490	10	EtOAc	No	Mechanical	20	None	HSST3	2	Mass Hunter	97	Every batch	
043	Tentatively detected 0.5%	0.3	2 Transitions	GC	MS	Q	EI + Yes	Yes	0.01	No Hewlett Packard 5870 & 5973	15	Acetone/DCM/PE	No	Mechanical	1	Liquid-Liquid Partitioning	HP5-MS	1	Chemstation	201	Day of run	
044	Fully identified (SANCO/1257/2013)	2.6		GC	MS	Q	EI + Yes	Yes	0.02	Yes AT5975 GC-MS	10	AcN/EtOAc	No		2	HP5-MS	10	DRS/Chemstation	-750	Always		
045	Fully identified (SANCO/1257/2013)	0.2	2 Transitions	LC	MS/MS	QQQ	ESI + Yes	Yes	0.010	No 6460 Agilent 10	ACN	No	Mechanical	5	DSPE	C18	5	Mass Hunter	150	No screening methods		
046	Tentatively detected 0	1.5	Accurate mass	LC	MS	TOF	ESI + Yes	Yes	0.02	Yes Brukermaxis 10	EtOAc			15	None	C18	2	TargetAnalysis	750	Always		
047	Fully identified (SANCO/1257/2013)	2	0.08	2 Transitions	GC	MS	IT	EI + Yes	Yes	0.01	No Varian Saturn 4000	10	ACN	No	Mechanical	20	None	Restek Rxi-5ms	3	Variam MS Workstation	282	Every batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Atrazine												
Laboratory Code *NRL	How was the detection/differentiation was done?	RT Differentiation (s)	MS Tolerance (ppm)	Other differentiation Details	Chromatographic Technique	Detector	Analyzer	Polarity	Routine Method?	Screening Detection limit (SDL) (mg/kg)	Injection Volume (μl)	
048 Fully identified (SANCO/1257/2013)	1 2 Transitions	LC MS/MS	QQQ	ESI +	Yes	0.010	Yes	Agilent UPLC 15 TQD	ACN	Yes	UPLC BEH C18	10
049 Fully identified (SANCO/1257/2013)		LC MS/MS	QQQ	ESI +	Yes	0.01	No	Agilent 6490 LC/MS/MS	Acetone/DCM/PE	No	DSPE	1
050 Fully identified (SANCO/1257/2013)	2 Transitions	LC MS	QQQ	ESI +	Yes	0.01	No	TSQ QUANTUM	ACN	No	None	C18
051 Fully identified (SANCO/1257/2013)	3 Transitions	GC MS/MS	QQQ	EI	Yes	0.01	No	Varian 3800 GC + 320-LMS	ACN	Yes	DSPE	3
052 Fully identified (SANCO/1257/2013)	2 Transitions ratio 30%	LC MS/MS	QQQ	ESI +	Yes	0.010	Yes	AB SCIEX 4000 QTRAP	ACN	No	Mechanical	20
053 Fully identified (SANCO/1257/2013)	0 1	Accurate mass	LC MS/MS	TOF	ESI +	Yes	0.01	XEV G2-S QTOF	ACN	Yes	DSPE	15
054 Fully identified (SANCO/1257/2013)	0.5 3 Transitions	GC MS	Q	EI +	Yes	0.01	Yes	Agilent 6890M	ACN	Yes	SPE	25
055 Fully identified (SANCO/1257/2013)	none 2 Transitions	LC MS/MS	QQQ	ESI +	Yes	0.01	No	API 4000	ACN	Yes	Waters-C18 2.1x50mm x1.7μm	5
056 Tentatively detected	1 1	GC MS	Q	EI +	Yes	0.01	Yes	Agilent 5973	ACN	No	DSPE	5
057 Fully identified (SANCO/1257/2013)	2 Transitions	GC MS/MS	QQQ	EI +	Yes	0.01	No	Quattro micro	ACN	No	PSA	HP-5MS UI
058 Tentatively detected	15 1	target + qualifier	GC MS	Q	EI +	Yes	0.01 - 0.05	Agilent GC - MSD	ACN	Yes	Mechanical	10
059 Fully identified (SANCO/1257/2013)	2	GC MS/MS	QQQ	EI +	Yes	0.010	No	SCION 10	ACN	No	Manual	3
060 Fully identified (SANCO/1257/2013)	2 Transitions + GC/MS scan	LC MS/MS	Q-TRAP	ESI +	Yes	Yes		Ap 3200 Trap	ACN		DSPE	2
062 Tentatively detected	-0.2	GC MS	Q	EI +	Yes	0.05	Yes	Agilent GC-MSD 7890A/5975C	ACN	Yes	Manual	5
064 Fully identified (SANCO/1257/2013)		GC MS	TOF	EI +	Yes	Yes	Yes	Pegasus 4D	Acetone/PE/DCM	No	DSPE	1
065 Fully identified (SANCO/1257/2013)	3 diagnostic ions	GC MS	Q	EI +	Yes	0.02	Yes	Shimadzu QP2010+	ACN	No	TR5MS	1
066 Fully identified (SANCO/1257/2013)	2 Transitions	LC MS/MS	QQQ	ESI +	Yes	0.01	No	Waters QD 10	ACN	Yes	Acuity UPLC BECH C18 1.7 mm 2.1 x 100	14
											200	

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/denification done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Chromatographic Technique	Detector	Analyzer	Polarity	Routine Method?	Routine Scope?	Scanning Detection Limit (SDL) (mg/kg)	Instrument Model	Sample Weight (g)	Extraction Solvent	pH adjusted	Agitation Mode	Agitation time (min)	Column Type	Injection Volume (μL)	Software	Standard Solution Frequency		
067 (SANCO/1/257/2013)	Fully identified	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.01	No	Agilent 6460	10	ACN	No	Mechanical	1	DSPE	C18	10	Mass Hunter	193	Daily
068 (SANCO/1/257/2013)	Fully identified	2 Transitions	LC	MS/MS	QQQ	+	Yes	Yes	10	No	API4000	10	ACN	Yes		2	SPE	RP-amide	10	Analyst	170	Every batch	

APPENDIX 3. Methods used by participants for detecting pesticides.

Cadausafos									
Laboratory Code *NRL	How was the detection/differentiation done?	Other identification Details	Detector	Chromatographic Technique	Analyzer	Ionization Mode	Routine Method?	Sample Weight (g)	Standard Solution Frequency
								Volumen(µL)	Software
002 Fully identified (SANCO/1/257/2013)			LC MS/MS	QQQ	ESI + Yes	Agilent 6410	AcN/ACN	1	SPE C18 4 micras 5 Mass Hunter 221
003 Fully identified (SANCO/1/257/2013)			GC MS/MS		+ Yes No		10 AcN	Yes Manual	SPE C18 176 No screening methods
006 Fully identified (SANCO/1/257/2013)	30 5 Confirmed on TOF, GC-MS	GC MS/MS	QQQ	EI + Yes	No Agilent 15 Acetone/DCM/ PE	No Mechanical	1	DBS None	
007 Fully identified (SANCO/1/257/2013)		GC TOF Stream	TOF	Jet + No	No NA	Agilent 6530	10 AcN	0.5 None	C18 3 Mass Hunter 550 NA
008 Fully identified (SANCO/1/257/2013)		GC MS/MS		Yes Yes			13 AcN	No Mechanical	None
012 Fully identified (SANCO/1/257/2013)	2 Transitions	GC MS/MS	QQQ	EI + Yes Yes	10 No TSQ QUANTUM XLS Thermo	10 AcN	No	5 SPE C18 1	
013 Tentatively detected	10 2	GC MS/MS	QQQ	EI + Yes	0.01 Agilent7890/ Agilent7000	10 AcN	Yes	5 DSPE DB5-MS 5 Mass Hunter ~500	
Tentatively detected	0.45 2	LC MS/MS	Q-Orbitrap	ESI + Yes	0.01 Thermo Q-Exactive	10 AcN	Yes	5 DSPE C18 5 Xcalibur ~500	
017 Fully identified (SANCO/1/257/2013)	10 0.02	GC MS	Q	EI + Yes Yes	0.01 Yes Thermo DSQ II	10 AcN	Yes	2 QueChERS ZB-5ms 1 Xcalibur 300	Weekly
022 Tentatively detected		GC MS/MS	ion trap	CI	Yes Yes 0.002 saturno 2000 varian	10 AcN	No	5 PSA/ MgSO4 factor four 10	
026 Tentatively detected 0.01	5 Acccurate mass	LC MS/MS	Q-Orbitrap	ESI + Yes Yes 0.005	Yes Q Exactive 10 AcN	No Mechanical	10 DSPE BEH C18 5	700	
027 Fully identified (SANCO/1/257/2013)	0 2 Transitions	GC MS/MS	QQQ	EI + Yes Yes	0.010 No Thermo 10 Acetone/DCM/ PE	No	1 None HP5-MS 1 Thermo 201 Every batch		
Fully identified (SANCO/1/257/2013)	0 0 no	GC MS	Q	EI + Yes Yes	0.01 No Agilent 6890/5973N	10 AcN	No Manual	2 None HP5-MS 2 Agilent Chemstation 184	No
Fully identified (SANCO/1/257/2013)	max 2.5%	1 2 Transitions	LC MS/MS	QQQ	ESI + Yes Yes 0.01 No Quattro Premier Xe	15 AcN	Yes Manual	1 None C18 3 Qualitlynx 350	Every batch
033 Tentatively detected	2 Transitions	LC MS/MS	QQQ	ESI + No No	No Varian 320 7.5 AcN/DCM	No	Manual 1 Liquid-Liquid Partitioning Polaris C-18 10 MS Workstation		
037 Tentatively detected <30	5 Isotope	LC MS	Orbitrap	ESI + No Not Established	Yes Executive AcN/1% acetic acid	No Mechanical	30 Liquid-Liquid Partitioning C18 Atlantis 5 ToxID 600	Every batch (100+ pesticides)	
Fully identified (SANCO/1/257/2013)	<20 spectum	GC MS	TOF	EI + Yes Yes	Not Established Yes Leco Pegasus IV AcN/1% acetic acid	No Mechanical	30 RTX-Clipescides + DSPE 10 MetAlign + ChromatOF	Every batch (235 pesticides)	560
Fully identified (SANCO/1/257/2013)	2 Transitions	LC MS/MS	QQQ	ESI + Yes Yes 0.01 No Agilent6490	10 EtOAc	No Mechanical 20 None HSS13 2 Mass Hunter	265 Every batch		

APPENDIX 3. Methods used by participants for detecting pesticides.

Cadausafos									
Laboratory Code *NRL	How was the detection/denification done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Analyzer	Chromatographic Technique	Polarity	Routine Method?	Routine Scope?
044 Fully identified (SANCO/1/257/1/2013)	2 Transitions	LC	MS/MS	QQQ	ESI + Yes	No	AB4000	10 ACN	No
045 Fully identified (SANCO/1/257/1/2013)	2 Transitions	GC	MS/MS	QQQ	EI Yes	0.010	No	7000 Agilent 10	ACN No
049 Fully identified (SANCO/1/257/1/2013)		LC	MS/MS	QQQ	ESI + Yes	0.01	No	Agilent 6490 LC/MS/MS	15 Acetone/DCM/PE
050 Fully identified (SANCO/1/257/1/2013)	2 Transitions	LC	MS	QQQ	ESI + Yes	0.010	No	TSQ QUANTUM 10	ACN No
051 Fully identified (SANCO/1/257/1/2013)	3 Transitions	GC	MS/MS	QQQ	EI Yes	Yes	0.005	No	Varians 3800 10 GC + 320-MS
053 Fully identified (SANCO/1/257/1/2013)	0 1 3	GC	MS/MS	QQQ	ESI + Yes	Yes	0.01	Yes	7000A 10 ACN Yes
055 Fully identified (SANCO/1/257/1/2013)	2 Transitions	LC	MS/MS	QQQ	ESI + Yes	Yes	0.01	No	API 4000 10 ACN Yes
057 Fully identified (SANCO/1/257/1/2013)	2 Transitions	LC	MS/MS	QQQ	ESI + Yes	Yes	0.01	No	Quattro Premier 10
060 Fully identified (SANCO/1/257/1/2013)	2 Transitions + GC/MS scan	LC	MS/MS	Q-Trap	ESI + Yes	Yes		Ap 3200 10 Trap	10 ACN
061 Fully identified (SANCO/1/257/1/2013)	0 0 2	LC	MS/MS	QQQ	ESI + No	No	0.005	No	Thermo TSQ Vantage 10 ACN Yes
062 Tentatively detected	2.6	GC	MS	Q	EI + Yes	No	0.01	Yes	Agilent GC-7890A/5975c 10 ACN Yes
064 Tentatively detected		GC	MS	TQF	EI + No	No		Yes	Pegasus 4D 15 Acetone/PE/DCM
066 Fully identified (SANCO/1/257/1/2013)	2 Transitions	GC	MS/MS	QQQ	EI Yes	Yes	0.01	No	Agilent 7000 15 EtOAc No
068 Fully identified (SANCO/1/257/1/2013)	2 Transitions	LC	MS/MS	QQQ	+ Yes	Yes	10 No	API4000 10 ACN Yes	2 SPE ip-amide 10 Analyst 170 Every batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Carbetamide											
Laboratory Code *NRL	How was the detection/differentiation done?	RT Deviation (s)	MS Tolerance (ppm)	Other differentiation Details	Chromatographic Technique	Detector	Polarity	Routine Method?	Screening Detection limit (mg/kg)	Injection Volume (μl)	Software
					Extraction Solvent	pH adjusted	Agitation Mode	Clean up Step	Compounds in method (pesticides Only)	Standard Solution	Frequency of library screening methods
003 Fully identified (SANCO/1257/2013)	Tentatively detected	LC MS/MS	GC MS/MS	ESI + No	Agilent QQQ	TOF	Jet Stream	No	Agilent 6530 accurate mass Q-ToF LC/MS	Acetonitrile/DCM/PE 10	DSPE 1
006 Fully identified (SANCO/1257/2013)	30 5 confirmed on TOF, GC-MS	LC MS/MS	GC MS/MS	ESI + No	Agilent Q-ToF	TOF	Q-ToF	No	Agilent 6530 accurate mass Q-ToF LC/MS	Acetonitrile/DCM/PE 15	None DB5 1
007 Fully identified (SANCO/1257/2013)	+/- 0.2 min	<5 acc. m/z + isotope + fragment	LC MS/MS	ESI + No	Agilent QQQ	TOF	TOF	No	Agilent 6530 accurate mass Q-ToF LC/MS	Acetonitrile/DCM/PE 10	Mass Hunter 176
009 Fully identified (SANCO/1257/2013)	0.2 min	0.05 - 0.1	LC MS/MS	ESI + No	Waters Xevo TQ	TOF	Q-ToF	Yes	API 5600 Q-ToF	Acetonitrile/DCM/PE 10	NA
010 Fully identified (SANCO/1257/2013)	0.1 Tentatively detected	0.005-0.25 Yes	GC MS	ESI + No	Waters Xevo TQ	TOF	Q-ToF	No	Waters Xevo TQ	Acetonitrile/DCM/PE 10	Waters 550
011 Tentatively detected	0.1 2 Transitions	0.005-0.1 Yes	LC MS/MS	ESI + No	Waters Xevo TQ	TOF	Q-ToF	No	Pegasus 4D TOF	Acetonitrile/DCM/PE 10	Both 600
012 Fully identified (SANCO/1257/2013)	2 Transitions	0.01 Yes	LC MS/MS	ESI + Yes	Thermo Q-Exactive	TOF	Q-ToF	No	Thermo Q-Exactive	Acetonitrile/DCM/PE 10	PeakView 2 Months
013 Tentatively detected	0 0	0.01 Yes	LC MS/MS	ESI + Yes	Thermo Q-Exactive	TOF	Q-ToF	No	Thermo Q-Exactive	Acetonitrile/DCM/PE 10	Mass Hunter 195
014 Tentatively detected	5 5	0.01 - 0.10 Yes	LC MS	ESI + Yes	Thermo Q-Exactive	TOF	Q-ToF	No	Thermo Q-Exactive	Acetonitrile/DCM/PE 10	Masslynx 350
015 Fully identified (SANCO/1257/2013)	3 10 Accurate mass	0.01 Yes	GC MS/MS	ESI + Yes	Thermo Q-Exactive	TOF	Q-ToF	No	Thermo Q-Exactive	Acetonitrile/DCM/PE 10	Mass Hunter 850
016 Fully identified (SANCO/1257/2013)	0	0.01 Yes	LC MS/MS	ESI + Yes	Thermo Q-Exactive	TOF	Q-ToF	No	Thermo Q-Exactive	Acetonitrile/DCM/PE 10	Mass Hunter 1000
018 Fully identified (SANCO/1257/2013)	5	0.01-0.1 Yes	LC MS/MS	ESI + Yes	Thermo Q-Exactive	TOF	Q-ToF	No	Thermo Q-Exactive	Acetonitrile/DCM/PE 10	Every batch
024 Tentatively detected	Match spectra with library: 92%	0.01-0.1 Yes	GC MS	ESI + Yes	Thermo Q-Exactive	TOF	Q-ToF	No	Thermo Q-Exactive	Acetonitrile/DCM/PE 10	Mass Hunter 1000

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/differentiation done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification details	Chromatographic Technique	Detector	Analyzer	Instrument Model	Sample Weight (g)	Extraction Solvent	pH adjusted	Agitation Mode	Agitation time (min)	Injection Volume (μL)	Software	Compounds in method (pesticides Only)	Standard Solution Frequency					
025 Fully identified (SANCO/1257/2013)	0 Tentatively detected	3 5	0 Accurate mass	LC MS/MS	QQQ	ESI	+	Yes	0.01	No	Waters Quattro 1st	10	MeOH/Water	No	Mechanical	1	Acuity BEH C18	5	Masslynx	224	Always	
026 Fully identified (SANCO/1257/2013)	0 Tentatively detected	2 Transitions 5	2 Transitions Accurate mass	LC MS/MS	Q-Orbitrap	ESI	+	No	0.010	Yes	Q Exactive	10	AcN	No	Mechanical	10	DSPE	BEH C18	5	700	Every batch	
027 Fully identified (SANCO/1257/2013)	0 Tentatively detected	2 Transitions 2 Transitions	2 Transitions Accurate mass	LC MS/MS	QQQ	ESI	+	Yes	0.010	No	Xevo	10	Acetone/DCM/PE	No	Mechanical	1	None	C18	2	Waters	286	Every batch
029 Fully identified (SANCO/1257/2013)	0 Tentatively detected	2 Transitions 2 Transitions	2 Transitions Accurate mass	LC MS/MS	QQQ	ESI	+	Yes	0.01	No	API 5500	10	AcN	Yes	Mechanical	15	DSPE	Pursuit XRs Ultra	3	Both	550	Every batch
030 Fully identified (SANCO/1257/2013)	0 Tentatively detected	5 2.5%	2 Transitions 1 Transition	LC MS/MS	QQQ	ESI	+	Yes	0.02	No	Abscix 3200 QTRAP	10	AcN	No	Mechanical	1	SPE	Acuity BEH RP 18	50	Analyst	153	Every batch
031 Fully identified (SANCO/1257/2013)	1 Tentatively detected	1 2.5%	1 Transition 2.5%	LC MS/MS	QQQ	ESI	+	No	0.01	No	Quattro Premier Xe	15	AcN	Yes	Manual	1	None	C18	3	Quantlynx	350	Every batch
035 Fully identified (SANCO/1257/2013)	<30 2.5%	2 Transitions 5	isotope spectrum	LC MS	Orbitrap	ESI	+	No	0.2	Yes	Exactive	10	AcN/1% acetic acid	No	Mechanical	30	DSPE	C18 50x2mm	10	Both	method 550	Always
037 Fully identified (SANCO/1257/2013)	<30 20	5 spectrum	spectrum	GC	MS	TOF	El	+	Yes	Not Established	Yes	Leco Pegasus IV	AcN/1% acetic acid	No	Mechanical	30	Liquid-Liquid Partitioning + DSPE	C18 Atlantis	5	ToxiD	600	Every batch (100+ pesticides)
038 Fully identified (SANCO/1257/2013)	10 -2	2 Transitions 6	2 Transitions 1 Transition	LC MS/MS	QQQ	ESI	+	Yes	0.01	No	Agilent 1100, MS API 3000	10	AcN	Yes	15	Liquid-Liquid Partitioning	RTX-Capillaries	10	MetAlign + ChromatOF	560	Every batch (235 pesticides)	
041 Fully identified (SANCO/1257/2013)	-2 0.3	6 0.3	6 0.3	LC MS	Q-TOF	ESI	+	No	0.05	Yes	Agilent 6550 iFunnel QTOF LC/MS	10	AcN	No	Mechanical	5	DSPE	C18	10	Analyst	Daily	
042 Fully identified (SANCO/1257/2013)	7.2 2.0	2 Transitions Accurate mass	2 Transitions Accurate mass	LC MS/MS	QQQ	ESI	+	Yes	0.01	No	Agilent 6490	10	EIOAC	No	Mechanical	20	None	HSS3	2	Mass Hunter	265	Every batch
043 Fully identified (SANCO/1257/2013)	0.3 0.01	1 Transition 2.0	1 Transition Accurate mass	LC MS	Q	El	+	Yes	0.005	No	Agilent 6550 iFunnel QTOF LC/MS	10	EIOAC	No	Mechanical	1	Liquid-Liquid Partitioning	Alltima C18 150*2.1 mm (Alltech 88370)	25	LC quan	150	Day of run
044 Fully identified (SANCO/1257/2013)	12 12	2 Transitions 30%	2 Transitions 30%	LC MS/MS	QQQ	ESI	+	No	0.010	Yes	Agilent 6550 iFunnel QTOF LC/MS	10	ACN/EIOAC	No	2	HP5-MS	10 Chemstation	-750	Always			
050 Fully identified (SANCO/1257/2013)	20 12	Target Analysis	Target Analysis	LC MS/MS	QQQ	ESI	+	Yes	0.010	Yes	Bruker maxis 10	10	AcN	No	Mechanical	20	DSPE	C18	20	Xcalibur	25	Every batch
052 Fully identified (SANCO/1257/2013)	1 1	Target Analysis	Target Analysis	LC MS/MS	QQQ	ESI	+	Yes	0.010	Yes	AB SCIEX 4000 QTRAP	10	AcN	No	Mechanical	20	DSPE	C18	1	Analyst	1	Every batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Carbetamide									
Laboratory Code *NRL	How was the detection/differentiation done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Detector	Analyser	Polarity	Routine Method?	Screening Detection limit (SDL) (mg/kg)
053 (SANCO/1257/2013)	Fully identified	0	1	Accurate mass	LC MS/MS	TOF	ESI +	Yes	0.01
055	Tentatively detected	none	2 Transitions	LC MS/MS	QQQ	ESI +	No	No	0.01
057	Tentatively detected		6 transitions	LC MS/MS	QQQ	ESI +	No	No	No
058	Tentatively detected	1.5	1 target + qualifier	GC MS	Q	EI +	Yes	Yes	0.01 - 0.05
059 (SANCO/1257/2013)	Fully identified	2	LC MS/MS	QQQ	ESI +	Yes	Yes	0.01	No
060 (SANCO/1257/2013)	Fully identified		2 Transitions	LC MS/MS	QTRAP	ESI +	Yes	Yes	Agilent GC - MSD
064	Tentatively detected			LC MS	QQQ	ESI +	No	No	Varian-320
065 (SANCO/1257/2013)	Fully identified	60	3 diagnostic ions	GC	MS	Q	EI +	Yes	Shimadzu QP2010+
Extraction Solvent									
Sample Weight (g)									
Agriflution Mode									
Agriflution time (min)									
Column Type									
Injection Volume (μL)									
Software									
Standard Solution Frequency									

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/differentiation done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Chromatographic Technique	Detector	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Detection Limit (SDL) (mg/Kg)	Sample Weight (g)	Injection Volume(µl)	Software	Column Type	Agitation time (min)	Clean up Step	Standard Solution (pesticides or library only)	Frequency			
001 Fully identified (SANCO/1257/2013)	Fully identified	2 Transitions	LC MS/MS	QQQ	ESI +	Yes	0.01	No	Agilent 6460	10	ACN	No	1	None	Zorbax C18	5	Mass Hunter	200	Daily			
003 Fully identified (SANCO/1257/2013)	Fully identified		LC MS/MS	ESI	+ Yes	No				10	ACN	Yes	Manual	1	DSPE							
004 Tentatively detected 5.76	Tentatively detected	5.76	5 fragment ions	LC MS/MS	Q-TOF	ESI +	Yes	Yes	Agilent 6520	10	ACN	Yes	Mechanical	15	DSPE	Synergie Fusion RP80A	8	Mass Hunter	>800	3 Months		
006 Fully identified (SANCO/1257/2013)	Fully identified		LC MS/MS	QQQ	ESI +	Yes	Yes	No	Agilent 15	Acetone/DCM/PE	No	Mechanical	1	None	XDB C18	3	Mass Hunter	50	No screening methods			
007 Fully identified (SANCO/1257/2013)	Fully identified	30	5 confirmed on TOF, LC-MS/MS	LC TOF	Q-TOF	Jet Stream +	No	No	Agilent 6530 Q-TOF LCMS	10	ACN			0.5	None	C18	3	Mass Hunter	550	NA		
008 Fully identified (SANCO/1257/2013)	Fully identified		LC MS/MS			Yes	Yes			13	ACN	No	Mechanical		None							
009 Fully identified <5 min	Fully identified	0.2	occ. m/z + isotope + fragment	LC NS/MS	Q-TOF	ESI +	No	No	AP 5600 Q-TOF	10	ACN	No	Mechanical	5	None	C18 Aqua Phenomenex	20	Both	600	2 Months		
010 Fully identified (SANCO/1257/2013)	Fully identified		LC NS/MS	QQQ	ESI +	Yes	Yes	0.010	Waters Xevo TQ	10	ACN	No	Manual	2	DSPE	Acquity BEH C18	10	Waters	195	Daily		
011 Fully identified (SANCO/1257/2013)	Fully identified	3	2 Transitions	LC NS/MS	QQQ	ESI +	Yes	Yes	0.005-0.1	No	Xevo TQS 10	ACN	No	2	None	C18	2.5	Mastlynx	300	Every batch		
012 Tentatively detected 0.8	Tentatively detected	3	2 Transitions	LC NS/MS	Q-TOF	ESI +	No	No	0.005-0.1	Yes	TripleTOF 10	ACN	No	2	None	C18	2	PeakView	620	Every batch		
013 Tentatively detected 10	Tentatively detected	10	2	GC MS/MS	QQQ	EI +	Yes	0.01	Agilent 7890/ Agilent 7000	10	ACN	Yes		5	DSPE	DB5-MS	5	Mass Hunter	~500			
014 Tentatively detected 0.83	Tentatively detected	0	2	LC MS/MS	Q-Orbitrap	ESI +	Yes	0.01	Thermo Q Exactive	10	ACN	Yes		5	DSPE	C18	5	Xcalibur	~500			
015 Fully identified 5	Fully identified (SANCO/1257/2013)	5	Accurate mass	LC MS/MS	QQQ	EI +	Yes	Yes	0.01	0.01 - 0.10	LCI PREMIER XE 15	Acetone/DCM/PE	No	1.5	None	C18	10	Mastlynx	350	Daily		
016 Fully identified (SANCO/1257/2013)	Fully identified	0		LC MS/MS	Q-TRAP	ESI +	Yes	Yes	0.01	No	ABSciex 4000 QTrap	10	ACN	Yes	1	None	Synergie Fusion RP 2.5 µm 50x2.0	55	MultiQuant	~250	Always	
018 Fully identified (SANCO/1257/2013)	Fully identified		2 Transitions	GC MS/MS	QQQ	ESI +	Yes	Yes	0.010	No	Qtrap3200	10	ACN	No	3	DSPE	Poroshell	1	Mass Hunter	300	Daily	
020 Fully identified (SANCO/1257/2013)	Fully identified		2 Transitions	LC MS/MS	QQQ	EI +	Yes	Yes	0.005	No	TSQ Quantum XLS Ultra	10	ACN	No	Mechanical	30	DSPE	RTX5-MS	1	TraceFinder	300	Always
021 Fully identified (SANCO/1257/2013)	Fully identified	263.1>169.1, 263.1>109.0	LC MS/MS	QQQ	ESI +	Yes	Yes	0.005	Waters Xevo TQ	15	ACN	No	Mechanical	5	None	HSS T3	10	Mastlynx	400	Every batch		
024 Fully identified (SANCO/1257/2013)	Fully identified	263.1>169.1, 263.1>109.0	LC MS/MS	QQQ	ESI +	Yes	Yes	0.002	No	ABSciex 4000 QTRAP	10	ACN	No	Manual	1	QueCh ERS Atlantis T3, 2.1x100 mm, 3µm without PSA	2	Anlyst	250	Before and after sample		

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/differentiation done?	MS Tolerance (ppm)	RT Deviation (s)	Other identification Details	Chromatographic Technique	Detector	Analyzer	Polarity	Routine Method?	Screening Detection Limit (SDL) (mg/kg)	Instrument Model	Extraction Solvent	pH adjusted	Agitation Mode	Injection Volume (μl)	Software	Compounds in library (pesticides Only)	Standard Solution Method or library (pesticides Only)	Frequency						
Demeton-S-methylsulfone																									
025 [SANCO/1/257/2013]	Fully identified	3	0	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	0.01	No	Waters Quattro 1st	10	MeOH/Water	No	Mechanical	1	None	Agility BEH C18	5	Masslynx	224	Always	
026 Tentatively detected	0.01	5	Accurate mass	LC	MS/MS	Q-Orbitrap	ESI	+	Yes	Yes	0.005	Yes	Q Exactive	10	Acetone/DCM/PE	No	Mechanical	10	DSPE	BEH C18	5		700		
027 [SANCO/1/257/2013]	Fully identified	0	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.010	No	Xevo	10	Acetone/DCM/PE	No	Mechanical	1	None	C18	2	Waters	286	Every batch	
028 [SANCO/1/257/2013]	Fully identified	0.1	0	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	0.005	No	Agilent 6410 Triple Quad	10	ACN	No	Manual	2	None	Zorbax Eclipse XDB-C18	4	Agilent ChemStation	144	No	
029 [SANCO/1/257/2013]	Fully identified		2 Transitions	LC		QQQ	ESI	+	Yes	Yes	0.01	No	API 4000	10	ACN	Yes	Mechanical	15	DSPE	Luna 5 μm PFP	3	Both	550	Every batch	
030 [SANCO/1/257/2013]	Fully identified	0	5	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.01	No	Absciex 3200 QTRAP	10	ACN	No	Mechanical	1	SPE	Acuity BEH RP 1/8	50	Analyst	153	Every batch
031 [SANCO/1/257/2013]	Fully identified	max 2.5%	1	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	0.01	No	Quattro Xe	15	ACN	Yes	Manual	1	None	C18	3	Quaternyx	350	Every batch	
032 [SANCO/1/257/2013]	Fully identified		2 Transitions	LC	MS/MS	QQQ	EI	+	Yes	Yes	0.01	No	Premier Xe	15	EtOAc	Yes	Mechanical	18	Filtration	HSS T3	2	Analyst	404	Every batch	
033 Tentatively detected		2 Transitions	LC	MS/MS	QQQ	ESI	+	No	No	No	Varian 320	7.5	AcN/DCM	No	Manual	1	Liquid-Liquid Partitioning	Polaris C-18	10	MS Workstation					
034 [SANCO/1/257/2013]	Fully identified	<0.2 min	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.01	No	API 3200	10	ACN	Yes	Mechanical	20	DSPE	Kinetex C18	20	Analyst	139	Every batch	
035 [SANCO/1/257/2013]	Fully identified	2.5%	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.01	No	HPLC Agilent 1100, MS API 3000	10	ACN	Yes	Mechanical	15	DSPE	C18 50x2mm	10	Both	method 550	Always	
036 [SANCO/1/257/2013]	Fully identified			LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.01	No	Agilent 6490	5	ACN	Yes		3	None	RP18	5	Mass Hunter	550	Daily	
037 [SANCO/1/257/2013]	Fully identified	<30	5	Isotope	LC	MS	Orbitrap	ESI	+	No	Not established	Yes	Q Exactive		AcCN/1% acetic acid	No	Mechanical	30	Liquid-Liquid Partitioning	C18 Atlantis	5	ToxID	600	Every batch (100+ pesticides)	
038 [SANCO/1/257/2013]	Fully identified	10	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.01	No	4000	10	ACN	No	Mechanical	5	DSPE	C18	10	Analyst		Daily	
039 [SANCO/1/257/2013]	Fully identified	1.20	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.005	No	Waters Quattro Premier XE	15	Acetone/DCM/PE	No	Mechanical	0.5	None	Acuity BEH 100X 2.1 mm i.d.	5	TargetLynx	215	Every batch	

APPENDIX 3. Methods used by participants for detecting pesticides.

Demeton-S-methyl/sulfone									
Laboratory Code *NRL	How was the detection/denomination?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Chromatographic Technique	Detector	IonizationMode	Polarity	Routine Method?
040	Fully identified (SANCO/1/257/2013)	0 +0.8 ppm	accurate mass + fragment + MRM	LC	MS	Q-TOF	ESI	+	No
041	Tentatively detected	-2 5		LC	MS	Q-TOF	ESI	+	Yes
042	Fully identified (SANCO/1/257/2013)		2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes
043	Tentatively detected	2.5% 0.3	1 Transition	LC	MS/MS	QQQ	ESI	+	Yes
044	Fully identified (SANCO/1/257/2013)	2.3		GC	MS	Q	EI	+	Yes
045	Fully identified (SANCO/1/257/2013)	0.2	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes
046	Tentatively detected	0.01 1.8	Accurate mass	LC	MS	TOF	ESI	+	Yes
047	Fully identified (SANCO/1/257/2013)	1	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes
048	Fully identified (SANCO/1/257/2013)	1	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes
049	Fully identified (SANCO/1/257/2013)			GC	MS/MS	QQQ	EI	Yes	Yes
050	Fully identified (SANCO/1/257/2013)		2 Transitions	LC	MS	MS	QQQ	+	Yes
051	Fully identified (SANCO/1/257/2013)	0	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes
053	Fully identified (SANCO/1/257/2013)	0 1	Accurate mass	LC	MS/MS	TOF	ESI	+	Yes
054	Fully identified (SANCO/1/257/2013)	2.5	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes
055	Fully identified (SANCO/1/257/2013)	none	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes
057	Fully identified (SANCO/1/257/2013)		2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes
Compounds in library (pesticides Only)									
Standard Solution Frequency									
Software									
Injection Volume(µl)									
Column Type									
Clean up Step									
Agrifaction time (min)									
Target Lynx + MassFragment tool									
500									
Always									
Instrument Model									
Waters Xevo Q-TOF (1st gen)									
Agilent 6550 iFunnel QTOF LC/MS									
Agilent 6490									
TSQ Quantum discovery max thermo									
Acetone/DCM/PE									
No									
Mechanical									
20									
Liquid-Liquid Partitioning									
Altimo C18 150 *2.1 mm (Alltech 88370)									
25									
LC quan									
150									
Day of run									
DRI/Chemstation									
-750									
Always									
HP5-MS									
10									
No screening methods									
110									
150									
Mass Hunter									
265									
Every batch									
Target Analysis									
750									
Always									
Xcalibur									
282									
Every batch									
31									
Auto									
30									
Every batch									
150									
Workstation									
363									
Every batch									
C18									
900									
Often									
ZORBAX ECLIPSE									
20									
Auto									
143									
Every batch									
600 pest. total (200 pesticides)									
(10 LC/MS/MS methods)									
196									

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/identification done?	MS Tolerance (ppm)	RT Deviation (s)	Other identification Details	Chromatographic Technique	Detector	Analyzer	Polarity	Routine Method?	Routine Scope?	Screening Detection Limit (SDL) (mg/kg)	Instrument Model	Extraction Solvent	pH adjusted	Agitation Mode	Injection Volume (µl)	Software	Compounds in library (pesticides Only)	Standard Solution Frequency
058 Tentatively detected	30 5 Acccurate mass	LC Orbitrap Orbitrap	ESI +	Yes Yes 0.01 - 0.05	Thermo Orbitrap	10	ACN	Yes	Mechanical	10	PSA	RP18	10	Xcalibur	>100	Every batch			
059 Fully identified (SANCO/1257/2013)	2 2 Transitions	LC MS/MS Q-QAPTOF	ESI +	Yes Yes 0.01	Varian-320	10	ACN	No	3	DSPE	C18 50x2mm	10	MS Workstation	162	Always				
060 Fully identified (SANCO/1257/2013)	2 Transitions	LC MS/MS Q-TOF	ESI +	Yes Yes	AP 3200 Qtrap	10	ACN		Manual	2	DSPE	Atlantis T3	5						
063 Tentatively detected	30 5 high resolution mass spectrum precursor ion	LC MS/MS Q-TOF	ESI + No	No 0.001	No 4600 AB Sciex	10	ACN	Yes	3	DSPE	C18	5	Analyst	500	10%				
064 Tentatively detected		LC MS QQQ	ESI + No	No	Yes Agilent 6410	10	ACN	Yes	Mechanical	2	GBC	ZORBAX XDB-C18	20	Mass Hunter	32				
065 Fully identified (SANCO/1257/2013)	60 2 Transitions	LC MS/MS QQQ	ESI + Yes	Yes 0.01	No AB-Sciex Qtrap 5500	10	ACN	No	30	DSPE	YMC-UltraHT 75x 2.0mm I.D.	5	Analyst	150	Daily				
066 Fully identified (SANCO/1257/2013)	2 Transitions	LC MS/MS QQQ	ESI + Yes	Yes 0.01	No Waters QD	10	ACN	Yes	Mechanical	16	None	Hydrosphere C18 1.7 mm 2.1 x 100 mm	14		200				
067 Fully identified (SANCO/1257/2013)	2 Transitions	LC MS/MS QQQ	ESI + Yes	Yes 0.01	No Agilent 6460	10	ACN	No	Mechanical	1	DSPE	C18	10	Mass Hunter	193	Daily			
068 Fully identified (SANCO/1257/2013)	0 2 Transitions	LC NS/MS QQQ	+ Yes	Yes 10	No AP4000	10	ACN	Yes	2	SPE	rp-amide	10	Analyst	170	Every batch				

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/differentiation was done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Detection Limit (SDL)? (mg/kg)	Instrument Model	Extraction Solvent	Agitation Time (min)	Injection Volume (μL)	Software	Standard Solution Frequency						
001	Fully identified (SANCO/1257/2013)		2 Transitions	LC	MS/MS	QQQ	ESI	-	Yes	Yes	0.01	No	Agilent 6460	10	AcN	No	1	None	Zorbax C18	5	Mass Hunter	200	Daily	
007	Fully identified (SANCO/1257/2013)	30	5 confirmed on TOF (negative & positive mode)	LC	TOF	Q-TOF	Jet Stream	-	No	No	NA	Yes	Agilent 6530 accurate mass Q-TOF LC/MS	10	AcN	No	0.5	None	Zorbax C18	5	Mass Hunter	200	Daily	
008	Fully identified (SANCO/1257/2013)			LC	MS/MS				Yes	Yes			13	AcN	No	Mechanical			C18	3	Mass Hunter	550	NA	
015	Fully identified (SANCO/1257/2013)	5	10 Accurate mass	LC	MS/MS	Q-TRAP	ESI	-	Yes	Yes	0.01	Yes	6490	10	AcN	No	Manual	3	DSPE	Poroshell 1	1	Mass Hunter	300	Daily
016	Fully identified (SANCO/1257/2013)	0		LC	MS/MS				Yes	Yes	0.01	No	ABSciex 4000 QTrap	10	AcN	Yes	1	None	Phenomenex Synergie Fusion RP 2.5 μm 50x2.0	55	MultiQuant	~250	Always	
018	Fully identified (SANCO/1257/2013)			LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.010	No	Qtrap3200	10	AcN	No	Manual	1	DSPE	C18	5	Analyst	205	Every batch
024	Fully identified (SANCO/1257/2013)	5	Match spectra with standard: 865	GC	MS	TOF	EI	Yes	Yes	0.01	Yes	LOQ: 0.01 mg/kg	10	AcN	No	Manual	1	QueChERS without PSA	DB5-MS (30m x 0.25mm x 0.25μm) in the 1st Dimension; BPX-50 (2m x 0.1mm x 0.1μm) in the 2nd Dimension	10	Chromatof	200	Before and after sample	
025	Fully identified (SANCO/1257/2013)	3	0 2 Transitions	LC	MS/MS	QQQ	ESI	-	Yes	Yes	0.05	No	Waters Quattro 1st	10	MeOH/water	No	Mechanical	1	Acuity BEH C18	5	Masslynx	224	Always	
027	Fully identified (SANCO/1257/2013)	0	1 Transition	LC	MS/MS	QQQ	ESI	-	Yes	Yes	0.10	No	Xevo	10	MeOH	No	Mechanical	1	None	C18	2	Waters	21	Every batch
028	Fully identified (SANCO/1257/2013)	0.1	0 2 Transitions	LC	MS/MS	QQQ	ESI	-	Yes	Yes	0.005	No	Agilent 6410 Triple Quadrupole	10	AcN	No	Manual	2	None	Zorbax Eclipse XDB-C18	10	Agilent ChemStation	144	No
029	Fully identified (SANCO/1257/2013)		2 Transitions	LC		QQQ	ESI	-	Yes	Yes	0.01	No	API 5500	10	AcN	Yes	Mechanical	15	None	Pursuit UPS 2.4	3	Both	550	Every batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/identification done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Detector	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Detection limit (mg/kg)	Instrument Model	Sample Weight (g)	Extraction Solvent	pH adjusted	Agitation Mode	Clean up Step	Column Type	Injection Volume (μl)	Compounds in method (pesticides Only)	Standard Solution	Frequency
032 Fully identified (SANCO/1257/2013)	2 Transitions	LC MS/MS	QQQ	EI -	Yes Yes 0.01	No	Agilent HPLC: 1290 Infinity, MS: G460 Triple Quad LC/MS, Model: G460A	ACN	No	0.01	Waters Aquity UPLC system, API 5000 Tripelkvaradrupole mass spectrometer Applied Biosystems	EIOAC	Yes Mechanical	18	Filtration	HSS T3	2	Analyst	15	Every batch	
035 Fully identified (SANCO/1257/2013)	2 Transitions	LC MS/MS	QQQ	EI -	Yes Yes 0.01	No	Agilent 6490	ACN	No	0.01	Waters Aquity UPLC system, API 5000 Tripelkvaradrupole mass spectrometer Applied Biosystems	EIOAC	Yes Mechanical	18	Filtration	HSS T3	2	Analyst	15	Every batch	
036 Fully identified (SANCO/1257/2013)		LC MS/MS	QQQ	EI +	Yes Yes 0.01	No	Agilent 6490	ACN	Yes	0.01	Agilent 6490	ACN	No	15	None	C18 50x2mm	5	Both	method 550	Always	
037 Tentatively detected <30	5 Isotope	LC MS Orbitrap	EI -	No No established	Yes	Not established	AcN/% acetic acid	No	Mechanical	30	Liquid-Liquid Partitioning	C18 Atlantis	5	ToxiD	600	Mass Hunter	550	Daily	Every batch (100+ pesticides)		
038 Fully identified (SANCO/1257/2013)	10 2 Transitions	LC MS/MS	QQQ	EI -	Yes Yes 0.01	No	Agilent 6490	ACN	No	0.01	Agilent 6490	ACN	No	4000	10	DSPE	C18	10	Analyst	Daily	
042 Fully identified (SANCO/1257/2013)	2 Transitions	LC NS/MS	QQQ	EI -	Yes Yes 0.01	No	Agilent 6490	ACN	No	0.01	Agilent 6490	ACN	No	20	None	HSS T3	2	Mass Hunter	265	Every batch	
046 Tentatively detected 0.04	2.2 Accurate mass	LC MS	TOF	EI +	Yes Yes 0.02	Yes	Brukermaxis 10	EIOAC	No	0.02	Brukermaxis 10	EIOAC	No	15	None	C18	2	Target Analysis	750	Always	
047 Fully identified (SANCO/1257/2013)	4 2 Transitions	LC MS/MS	QQQ	EI -	Yes Yes 0.01	No	Thermo Finnigan TSQ 10 Quantum	ACN	No	0.01	Thermo Finnigan TSQ 10 Quantum	ACN	No	20	DSPE	Agilent Eclipse C18 XDB	20	Xcalibur	282	Every batch	
048 Fully identified (SANCO/1257/2013)	1 2 Transitions	LC MS/MS	QQQ	EI -	Yes Yes 0.010	Yes	Waters Acquity UPLC 15 QD	ACN	Yes	0.010	Waters Acquity UPLC 15 QD	ACN	Yes	1	DSPE	UPLC BEH C18	10	Auto	293	Every batch	
051 Fully identified (SANCO/1257/2013)	0.3 2 Transitions	LC NS/MS	QQQ	EI -	Yes Yes 0.01	Yes	Varian Pro Star LC + 320-MS	ACN	No	0.01	Varian Pro Star LC + 320-MS	ACN	No	3	None	Rectek Ultra C18	10	Workstation	363	Every batch	
052 Fully identified (SANCO/1257/2013)	12 2 Transitions ratio 30%	LC NS/MS	QQQ	EI -	Yes Yes 0.010	Yes	AB SCIEX 4000 QTRAP	ACN	No	0.010	AB SCIEX 4000 QTRAP	ACN	No	20	Analyst	C18	20	Analyst	4	Every batch	
055 Fully identified (SANCO/1257/2013)	none	LC NS/MS	QQQ	EI -	Yes Yes 0.01	No	API 4000	ACN	Yes	0.01	API 4000	ACN	Manual	5	DSPE	Waters C18 2.1x50mmx 1.7μm	5	Analyst	600 pest total (10 LC/MS methods)	Weekly (200 pesticides)	
057 Fully identified (SANCO/1257/2013)	2 Transitions	LC NS/MS	QQQ	EI -	Yes Yes 0.01	No	Quattro Premier	ACN	No	0.01	Quattro Premier	ACN	No	2	DSPE	C18	5	Mastlynx	196		

APPENDIX 3. Methods used by participants for detecting pesticides.

060	Fully identified (SANCO/1/257/2013)	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Chromatographic Technique	Analyzer	Polarity	Routine Method?	Routine Scope?	Screening Detection Limit (SDL)? (mg/kg)	Instrument Model	Sample Weight (g)	Agitation Time (min)	Clean Up Step	Column Type	Injection Volume (μL)	Software	Compounds in method or library (pesticides Only)	Standard Solution Frequency	*NRL Laboratory Code
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APPENDIX 3. Methods used by participants for detecting pesticides.

Dodemorph											
Laboratory Code *NRL	How was the detection/differentiation?	RT Deviation (s)	MS Tolerance (ppm)	Other identification details	Analyzer	Chromatographic Technique	Detector	Ionization Mode	Sample Weight (g)	Injection Volume (μL)	Software
								pH adjusted			Standard Solution (pesticides Only)
001 Fully identified (SANCO/1/257/2013)	3 Transitions	GC MS/MS	QQQ	EI + Yes	0.01	No	Agilent 7000	10	AcN	No	HP5-MS
003 Fully identified (SANCO/1/257/2013)		LC NS/MS		EI + Yes	No			10	AcN	Yes	SPE
006 Tentatively detected		GC MS/MS	QQQ	EI + No	No	No	Agilent 15	Acetone/DCM/PE	No	Mechanical	DB5
008 Fully identified (SANCO/1/257/2013)		LC NS/MS		Yes Yes			13	AcN	No	Mechanical	None
009 Tentatively detected <5 min	2 Transitions	LC MS/MS	QQQ	EI + Yes	0.05 - 0.1	No	API 5500	10	AcN	No	5
010 Fully identified (SANCO/1/257/2013)		LC MS/MS	QQQ	EI + Yes	0.010		Waters Xevo TQ	10	AcN	No	DSPE
011 Tentatively detected		GC MS	TOF	EI	No	0.005-0.25	Yes	Pegasus 4D	10	AcN	No
012 Fully identified (SANCO/1/257/2013)	2 Transitions	LC MS/MS	Q-TOF	EI + No	0.005-0.1	Yes	TripletOF	10	AcN	No	2
013 Tentatively detected	2	GC NS/MS	QQQ	EI + No	No	10	TSQ QUANTUM XLS Thermo	10	AcN	No	Manual
014 Tentatively detected	5	GC MS/MS	QQQ	EI + Yes	0.01		Agilent7890/Agilent7000	10	AcN	Yes	5
015 Fully identified (SANCO/1/257/2013)	3 10 Accurate mass	GC NS/MS	QQQ	EI Yes	0.01 - 0.10	Yes	LCT PREMIER XE	15 Acetone/DCM/PE	No		1.5
016 Fully identified (SANCO/1/257/2013)	-0.3	GC MS/MS	QQQ	EI + Yes	0.01	No	Agilent 7000	10	AcN	Yes	1 DSPE
021 Fully identified (SANCO/1/257/2013)	2 Transitions	LC MS/MS	QQQ	EI + Yes	0.005	No	Waters Xevo TQ	15	AcN	No	Mechanical
024 Tentatively detected	5	Match spectra with library: 901	GC MS	TOF	EI	No	No	0.01-0.1	Yes LECO Pegasus 4D	No	Manual
026 Tentatively detected	5	Accurate mass	LC NS/MS	Q-Orbitrap	EI + No	No	Q Exactive	10	AcN	No	Mechanical
											10 DSPE BEH C18
											5 700

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/differentiation done?	MS Tolerance (ppm)	Other identification details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Routine Method?	Routine Scope?	Screening Detection Limit (SDL) (mg/Kg)	Sample Weight (g)	Full Scan	Instrument Model	Extraction Solvent	pH adjusted	Agitation Mode	Clean up Step	Column Type	Injection Volume (µl)	Compounds in library (pesticides Only)	Standard Solution Frequency				
027 (SANCO/1257/2013)	Fully identified	0	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	0.010	No	Xevo	10	Acetone/DCM/PE	No	Mechanical	1	None	C18	2	Waters	286	Every batch		
028 (SANCO/1257/2013)	Fully identified	0.1	no	GC	MS	Q	EI	+	Yes	0.01	No	Agilent 6890/5973N	10	ACN	No	Manual	2	None	HP5-MS	2	Agilent Chemstation	184	No		
029 (SANCO/1257/2013)	Fully identified		2 Transitions	LC		QQQ	ESI	+	Yes	0.01	No	API 5500	10	ACN	Yes	Mechanical	15	DSPE	Pursuit XRs Ultra	3	Both	550	Every batch		
031 (SANCO/1257/2013)	Fully identified	max 0.5%	1	3 ion ratios	GC	MS	Q	EI	+	Yes	0.01	Yes	Trace DSQ	15	ACN	Yes	Manual	1	SPE	DB5MS	0.8	Xcalibur	350	Every batch	
035	Fully identified (SANCO/1257/2013)	± 0.5%	1 Target, 3 Qualifier	GC	MS	Q	EI	+	Yes	0.01	No	GC-Shimadzu GC-2010, MS Shimadzu GCMS-QP2011	10	ACN	Yes		15	DSPE	HP5-MS	3	Both	method 550	Always		
037 (SANCO/1257/2013)	Fully identified	<20	spectrum	GC	MS	TOF	EI	+	Yes	0.05	Yes	Leco Pegasus IV		AcN/1% acetic acid	No	Mechanical	30	Liquid-Liquid Partitioning + DSPE	RTX-Chloroform	10	MetAlign + ChromatOF	560	Every batch (235 pesticides)		
038 (SANCO/1257/2013)	Fully identified	10	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	0.01	No	4000	10	ACN	No	Mechanical	5	DSPE	C18	10	Analyst		Daily		
039 (SANCO/1257/2013)	Fully identified	1.20	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	0.005	No	Waters Quattro Premier XE	15	Acetone/DCM/PE	No	Mechanical	0.5	None	Acquity BEH (100x2.1 mm i.d.)	5	TargetLynx	215	Every batch		
041	Tentatively detected	3	5	GC-QTOF	LC	MS	Q-TOF	ESI	+	No	Yes	0.02	Yes	Agilent 6550 iFunnel QTOF LC/MS	10	ACN	No		2	DSPE	C18	5	Mass Hunter	1700	Always
042 (SANCO/1257/2013)	Fully identified	-2.1	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	0.01	No	Agilent 6490	10	EtOAc	No	Mechanical	20	None	HSS13	2	Mass Hunter	265	Every batch		
044 (SANCO/1257/2013)	Fully identified	-1.6		GC	MS	Q	EI	+	Yes	0.02	Yes	AT5975 GC-MS	10	ACN/EtOAc	No		2		HP5-MS	10	DRS/Chemstation	-750	Always		
047 (SANCO/1257/2013)	Fully identified	3 ions		GC	MS	IT	EI	+	Yes	0.01	Yes	Variion Saturn 4000	10	ACN	No	Mechanical	20	None	Restek Rx1-5ms	3	Varian MS Workstation	282	Every batch		
050	Tentatively detected		2 Transitions	LC	MS	QQQ	ESI	+	No	No	No	TSQ QUANTUM	10	ACN	No	None			XBRIDGE	20	Xcalibur	25			
051 (SANCO/1257/2013)	Fully identified	0.3		GC	MS	IT	EI		Yes	0.01	Yes	Variion 3800 GC+Saturn 2000 MS	10	ACN	Yes	Mechanical	3	DSPE	RTX5-MS	3	Workstation	363	Every batch		
052 (SANCO/1257/2013)	Fully identified	12	2 Transitions ratio 30%	LC	MS/MS	QQQ	ESI	+	Yes	0.010	Yes	ABSCEX 4000 QTRAP	10	ACN	No	Mechanical	20	None	C18	20	Analyst	3	Every batch		

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/differentiation done?	MS Tolerance (ppm)	Other identification details	Chromatographic Technique	Detector	Analyzer	Polarity	Routine Method?	Routine Scope?	Screening Detection limit (DL) (mg/kg)	Instrument Model	Extraction Solvent	pH adjusted	Agitation time (min)	Injection Volume (μL)	Software	Standard Solution (pesticides Only)	Frequency						
053 (SANCO/1/237/2013)	Fully identified	1	Accurate mass	LC	MS/MS	TOF	ESI	+	Yes	0.01	XEVO G2-S QToF	10	ACN	Yes	Mechanical	15	DSPE	C18	1	Unity	900	Often		
055	Tentatively detected	none	2 Transitions	LC	MS/MS	QQQ	ESI	+	No	No	0.01	No	API 4000	10	ACN	Yes	Manual	5	DSPE	Waters-C18 2.1x50mmx 1.7,μm	600 pest total (10 LC/MS/MS methods)	Weekly (200 pesticides)		
056	Tentatively detected	1	1	GC	MS	Q	EI	Yes	No	0.01	Yes	Agilent 5973	10	ACN	No	Manual	1	PSA	HP-5MS UI	2	Auto	923	Every batch	
057	Tentatively detected			GC	MS	Q	EI	+	No	No	Yes	HP-5973	10	ACN	No	Manual	2	DSPE	SE54	5	DRS			
058	Tentatively detected	15	1 target + qualifier	GC	MS	Q	EI	+	Yes	Yes	0.01 - 0.05	Yes	Agilent GC-MSD 10	10	ACN	Yes	Mechanical	10	PSA	DB5	10	ChemStation	>600	Every batch
060 (SANCO/1/237/2013)	Fully identified	2 Transitions + GC/MS scan	LC	MS/MS	Q-TRAP	ESI	+	Yes	Yes			Ap 3200 QTrap	10	ACN		Manual	2	DSPE	Agilent T3	5				
062	Tentatively detected	-3.2		GC	MS	Q	EI	+	Yes	No	0.01	Yes	Agilent GC-MSD 10	10	ACN	Yes	Manual	5	DSPE	HP5-MS	1	Auto	927	Weekly
064	Tentatively detected			GC	MS	TOF	EI	+	No	No	Yes	Pegasus 4D	15	Acetone/PE/ DCM	No	Mechanical	1	None	TR5MS	1	Chroma TOF	76		
066 (SANCO/1/237/2013)	Fully identified	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.01	No	Waters iQD 10	10	ACN	Yes	Mechanical	16	Acuity UPLC BECH C18 1.7 mm 2.1x 100 mm	14	200				

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/denification done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification details	Chromatographic Technique	Detector	Analyzer	Routine Method?	Routine Scope?	Screening Detection Limit (SDL)? (mg/kg)	Instrument Model	Sample Weight (g)	Injection Volume (μL)	Software	Column Type	Agitation Time (min)	Clench Up Step	pH adjusted	Extraction Solvent	Instrument Scan	Agilent 7000	AcN	No	10	Agilent	Yes	Manual	1	None	HP5-MS	1	Mass Hunter	350	Daily	Standard Solution Frequency
001	Fully identified (SANCO/1/257/2013)				GC	MS/MS	QQQ	EI	+	Yes	0.01	No	Agilent	7000	10	AcN	No	1	None	HP5-MS	1	Mass Hunter	350	Daily											
003	Fully identified (SANCO/1/257/2013)				GC	MS/MS			+	Yes	No			10	AcN	Yes	Manual	1	SPE																
004	Fully identified (SANCO/1/257/2013)	0	2 Transitions		GC	MS/MS	QQQ	EI	+	Yes	Yes	No	Agilent	10	AcN	Yes	Mechanical	15	DSPE	HP5-MS	2	Mass Hunter	>300	Every batch											
006	Tentatively detected				GC	MS/MS	QQQ	EI	+	No	No	No	Agilent	15	Acetone/ DCM/PE	No	Mechanical	1	None	DB5	1	Mass Hunter	176	No screening methods											
007	Tentatively detected	30			GC	MS	Q	CI		No	No	No	Agilent	59/3ms 6890N	30	EtOAc		0.5	GPC	DB5-MS	2	Mass Hunter	150	NA											
008	Fully identified (SANCO/1/257/2013)				GC	MS/MS				Yes	Yes			13	AcN	No	Mechanical		None																
009	Fully identified (+/- 0.2 min) (SANCO/1/257/2013)	2 Transitions			GC	MS/MS	QQQ	EI	+	Yes	Yes	0.01	No	Bruker 450 Scion TQ	10	AcN	No	Mechanical	5	None	HP1 MS UI	5	Both	250	Every batch										
010	Fully identified (SANCO/1/257/2013)				GC	MS/MS	QQQ	EI	+	Yes	Yes	0.010		Bruker Scion TQ	10	AcN	No	Manual	2	DSPE	Zébron ZB	2	Bruker	210	Daily										
011	Fully identified (SANCO/1/257/2013)	2	2 Transitions		GC	MS/MS	QQQ	EI		Yes	Yes	0.005-0.25	No	Agilent 7000B	10	AcN	No	Manual	2	DSPE	HP5-MS	2	Mass Hunter	170	Every batch										
012	Fully identified (SANCO/1/257/2013)		2 Transitions		GC	MS/MS	QQQ	EI	+	Yes	Yes	10	Yes	10	TSQ QUANTUM XLS Thermo	10	AcN	No	Manual	5	SPE	C18	1												
013	Tentatively detected	10	2		GC	MS/MS	QQQ	EI	+	Yes	Yes	0.01		Agilent 7890/ Agilent 7000	10	AcN	Yes		5	DSPE	DB5-MS	5	Mass Hunter	~500											
015	Fully identified (SANCO/1/257/2013)	3			GC	MS/MS	QQQ	EI		Yes	Yes	0.01	Yes	5977	10	Cyclohexane/ EtOAc/Acetone	No		3	DSPE	HP5-MS	1	Mass Hunter	850	Daily										
016	Fully identified (-0.36) (SANCO/1/257/2013)				GC	MS/MS	QQQ	EI	+	Yes	Yes	0.01	No	Agilent 7000	10	AcN	Yes		1	DSPE	HPSMSUI	10	Mass Hunter	~300	Always										
017	Fully identified (SANCO/1/257/2013)	10	0.02		GC	MS	Q	EI	+	Yes	Yes	0.01	Yes	Thermo DSQII	10	AcN	Yes	Manual	2	QueChERS	ZB-5ms	1	Xcalibur	300	Weekly										
018	Fully identified (SANCO/1/257/2013)				LC	MS/MS	QQQ		+	Yes	Yes	0.010	No	Qtrap3200	10	AcN	No	Manual	1	SPE	C18	5	Analyst	205	Every batch										
019	Fully identified (SANCO/1/257/2013)				GC	MS/MS	QQQ	EI	+	Yes	Yes	0.01	No	Quantum	10	AcN	No	Manual	1	DSPE	ZB50	2	Xcalibur												
020	Fully identified (SANCO/1/257/2013)		2 Transitions		GC	MS/MS	QQQ	EI	+	Yes	Yes	0.005	No	Quantum XLS Ultra	10	AcN	No	Mechanical	30	DSPE	RTX5-MS	1	TraceFinder	300	Always										

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/differentiation done?	RT Differentiation(s)	MS Tolerance (ppm)	Other differentiation details	Detector	Ionization Mode	Polarity	Routine Method?	Screening Detection limit (SDL) (mg/kg)	Instrument Model	Sample Weight (g)	Extraction Solvent	pH adjusted	Agitation Mode	Agitation time (min)	Injection Volume (μl)	Software	Compounds in method (library or library pesticides Only)	Standard Solution Frequency		
022 Tentatively detected					GC MS/MS	ion trap	EI	Yes	0.005	Yes	saturno 2000	10	AcN	No		5	PSA/ MgSO ₄	factor four	10		
023 Fully identified (SANCO/1257/2013)					GC	MS	IT	No	No	Yes	Varian 240MS	15	Acetone/DCM/ PE	No		1	GPC	VF-5MS	3	Auto	
024 Fully identified (SANCO/1257/2013)	Match spectra with standard: 95%	5			GC	MS	TOF	Yes	Yes	LOG: 0.01 mg/kg	LECO Pegasus 4D	10	AcN	No	Manual	1	QueChERS without PSA	DB5-MS (30m x 0.25mm x 0.25μm in the 1st Dimension); BPX-50 (2m x 0.1mm x 0.1μm) in the 2nd Dimension	10	Chromatof	
025 Fully identified (SANCO/1257/2013)	2	0	2 Transitions		GC	MS/MS	QQQ	EI	+	Yes	0.02	No	varian 320	50	Acetone	No	Mechanical	2	Liquid-Liquid Partitioning	DB5	8
026 Tentatively detected	0.01				GC	MS/MS	QQQ	Cl	-	Yes	0.001	No	Varian 320MS	10	AcN	No	Mechanical	10	DSPE	DB5	1
027 Fully identified (SANCO/1257/2013)	0		2 Transitions		GC	MS/MS	QQQ	EI	+	Yes	0.010	No	Thermo 6890/5973N	10	Acetone/DCM/ PE	No		1	None	HP5-MS	1
028 Fully identified (SANCO/1257/2013)	0.1	0	no		GC	MS	Q	EI	+	Yes	0.01	No	Agilent 6890/5973N	10	ACN	No	Manual	2	None	HP5-MS	2
029 Fully identified (SANCO/1257/2013)			speculum		GC		TOF	EI		Yes	0.01	Yes	LECO Pegasus IV	10	ACN	Yes	Mechanical	15	DSPE	HP5-MS	3
030 Fully identified (SANCO/1257/2013)	2	5			GC	MS	IT	EI		Yes	0.01	Yes	Varian 4000	10	AcN	No	Mechanical	1	SPE	Rxi 5ms 5% diphenyl 95% dimethyl polysiloxane	1
031 Fully identified (SANCO/1257/2013)	max 0.5%	1	3 ion ratios		GC	MS	Q	EI	+	Yes	0.01	Yes	Trace DSQ	15	AcN	Yes	Manual	1	SPE	DB5MS	0.8
032 Fully identified (SANCO/1257/2013)			2 Transitions		GC	MS/MS	QQQ	EI	+	Yes	0.01	No	Waters Quattro Micro GC	10	EtOAc	Yes	Mechanical	18	Filtration	Rxi-5sil M/S/integra-guard Resek	10
034 Fully identified (SANCO/1257/2013)	<0.2 min		2 Transitions		GC	MS/MS	QQQ	EI	+	Yes	0.01	No	Agilent 7890A	10	AcN	Yes	Mechanical	20	DSPE	Pesticides2	4
035 Fully identified (SANCO/1257/2013)	± 0.5%		1 Target, 3 Qualifier		GC	MS	Q	Cl	-	Yes	0.01	No	GC Shimadzu GC-2010, MS Shimadzu GCMS-QP2011	10	ACN	Yes		15	DSPE	HP5-MS	3
036 Fully identified (SANCO/1257/2013)					GC	MS/MS	QQQ	EI	+	Yes	0.01	No	Agilent 7000B	5	ACN	Yes		3	None	HP-5MS UI	2
																			Daily		

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/denomination was done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Sample Weight (g)	Instrument Model	Extraction Solvent	pH adjusted	Agitation Mode	Agitation time (min)	Injection Volume (μL)	Compounds in method (pesticides Only)	Standard Solution Frequency	Endrin					
																		Column Type	Software				
037 (SANCO/1257/2013)	Fully identified	<20	spectrum	GC	MS	TOF	EI	+	Yes	Yes	0.05	Yes	Leco Pegasus IV	AcN/1% acetic acid	No	Mechanical	30	Liquid-Liquid Partitioning + DSPE	RTX-Clpesticides	10	MetAlign + ChromatOF	560	Every batch (235 pesticides)
038 (SANCO/1257/2013)	Fully identified	10	Accurate mass	GC	MS	Q	EI	+	Yes	Yes	0.01	No	Agilent	10	AcN	No	Mechanical	5	DSPE	DB5	5	ChemStation	Daily
039 (SANCO/1257/2013)	Fully identified	0.36	2 Transitions	GC	MS/MS	QQQ	EI	+	Yes	Yes	0.005	No	Bruker Scion GC-MS TQ	15 Acetone/DCM/PE	No	Mechanical	0.5	VFS-ms, 30mmx25 mm [0.25 μm film]	VF5-ms, 30mmx25 mm [0.25 μm film]	5	Workstation	336	Every batch
040 (SANCO/1257/2013)	Fully identified	0	2 Transitionss	GC	MS/MS	QQQ	EI	+	Yes	Yes	0.01	No	Waters Quattro Micro GC/MS	10 EtOAc	Yes		4	GPC	DB5 MS	1.5	Masslynx	80	Always
041 Tentatively detected	0	-2	MS/MS	GC	MS	TOF	EI	+	No	Yes	Yes	Yes	Agilent 7200 GC-QTOF	10 AcN	No		2	DSPE	DB5	5	Mass Hunter	Always	
042 (SANCO/1257/2013)	Fully identified	2 Transitions	GC	MS/MS	QQQ	EI	+	Yes	Yes	0.01	No	Varian 1200	10 EtOAc	No	Mechanical	20	DSPE	DB5-MS	5	Workstation	171	Every batch	
044 (SANCO/1257/2013)	Fully identified	1.0		GC	MS	Q	EI	+	Yes	Yes	0.02	Yes	AT5975 GC-MS	10 AcN/EtOAc	No		2	HP5-MS	10 Chemstation	DRS/	~750	Always	
045 (SANCO/1257/2013)	Fully identified	0.2	2 Transitions	GC	MS/MS	QQQ	EI		Yes	Yes	0.010	No	7000 Agilent	10 AcN	No	Mechanical	5	DSPE	HP5-MS	2	Mass Hunter	150	No screening methods
046 (SANCO/1257/2013)	Fully identified	2	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.01	No	Agilent 6460	10 EtOAc			15	None	C18	2	Mass Hunter	550	Always	
047 (SANCO/1257/2013)	Fully identified	0.04	3 Transitions	GC	MS	IT	EI	+	Yes	Yes	0.005	No	Varian Saturn 4000	10 AcN	No	Mechanical	20	None	Restek Rx-5ms	3 Varian Ms Workstation	282	Every batch	
048 (SANCO/1257/2013)	Fully identified	1		GC	MS/MS	QQQ	EI		Yes	Yes	0.010	Yes	Agilent 7890A GC 5975 MSD	15 Acetone/DCM/PE	No		1	DSPE	DB5-MS	10 Xcalibur	60	Every batch	
049 (SANCO/1257/2013)	Fully identified	0	3 Transitions	GC	MS/MS	QQQ	EI		Yes	Yes	0.01	Yes	Agilent 6490 LC/MS/MS	10 AcN	Yes	Mechanical	3	DSPE	Rxi-1ms	3 Workstation	363	Every batch	
050 (SANCO/1257/2013)	Fully identified	2 Transitions	GC	MS	IT	EI	+	Yes	Yes	0.010	Yes	POLARIS Q	10 AcN	No	None		DSPE	DB5-MS	20	CP Sil8	2 Analyst	2 Every batch	
051 (SANCO/1257/2013)	Fully identified	0	3 Transitions	GC	MS/MS	QQQ	EI		Yes	Yes	0.003	No	Varian 3800 GC + 320-MS	10 AcN	Yes	Mechanical	20	DSPE	CP Sil8	2 Chemstation	100	Often	
052 (SANCO/1257/2013)	Fully identified	12	2 Transitions ratio 30%	GC	MS/MS	IT	EI		Yes	Yes	0.010	Yes	Agilent 7000B	10 AcN	No	Mechanical		DSPE	DB5-MS	2 Chemstation	100	Often	
053 (SANCO/1257/2013)	Fully identified	0	1	GC	MS	Q	CI	-	Yes	Yes	0.01	Yes	5975C	10 AcN	Yes	Mechanical	15	DSPE	DB5-MS	2 Chemstation	100	Often	
054 (SANCO/1257/2013)	Fully identified	0.5	3 Transitions	GC	MS	Q	EI	+	Yes	Yes	0.01	Yes	Agilent 6890M	10 AcN	Yes		25	SPE	HP5-MS	2 Auto	169	Every batch	

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/differentiation was done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Detector	IonizationMode	Polarity	Routine Method?	Screening Detection limit (SDL) (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	pH adjusted	Agitation Mode	Clean up Step	Column Type	Injection Volume (μL)	Compounds in method or library (pesticides Only)	Standard Solution Frequency		
057 [SANCO/1257/2013]	Fully identified	2 Transitions	GC MS/MS	QQQ	EI	+	Yes	Yes	0.01	No	Quattro micro	10	ACN	No	Manual	2	DSPE	SE54	5	Mastlynx	110	
058 Tentatively detected	15 1 target + qualifier	GC	MS	Q	EI	+	Yes	Yes	0.01 - 0.05	Yes	Agilent GC - MSD	10	ACN	Yes	Mechanical	10	PSA	DB5	10	ChemStation	>600	
059 [SANCO/1257/2013]		2	GC	MS/MS	QQQ	EI	+	Yes	Yes	0.010	No	SCION	10	ACN	No	Manual	3	DSPE	BR5MS	2	Workstation	127
062 Tentatively detected	-1.5	GC	MS	Q	EI	+	Yes	No	0.01	Yes	Agilent GC-MSD 7890A/5975C	10	ACN	Yes	Manual	5	DSPE	HP5-MS	1	Auto	927	
065 [SANCO/1257/2013]	60 3 diagnostic ions	GC	MS	Q	EI	+	Yes	No	0.02	Yes	Shimadzu QP2010+	10	ACN	No		30	DSPE	VF-5-MS-30M*0.25* 0.25μm	10	GC-MSsolution	325	
067 [SANCO/1257/2013]		2 Transitions	GC	MS/MS	QQQ	EI	Yes	Yes	0.01	No	Agilent 7000	10	ACN	No	Mechanical	1	DSPE	DB-5MS UI	2	Mass Hunter	236	

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/differentiation done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Sample Weight (g)	Instrument Model	Full Scan	Screening Detection Limit (SDL)? (mg/kg)	Routine Method?	Routine Scope?	Agitation Time (min)	Column Type	Injection Volume (μL)	Software	Standard Solution Frequency			
Ethopropox																						
001	Fully identified (SANCO/1/257/2013)		3 Transitions	GC	MS/MS	QQQ	EI	+	Yes	Yes	0.01	No	Agilent 7000	10	AcN	No	1	None	HP5-MS	1		
002	Fully identified (SANCO/1/257/2013)			LC	MS/MS	QQQ	ESI	+	Yes	Yes			Agilent 6410	10	AcN/ACN		1	SPE	C18 4 micras	5		
003	Fully identified (SANCO/1/257/2013)			LC	MS/MS			+	Yes	No				10	AcN	Yes	Manual	1	SPE			
004	Tentatively detected	90	2.33	2 fragment ions	LC	MS/MS	Q-TOF	ESI	+	Yes	Yes	Yes	Agilent 6520	10	AcN	Yes	Mechanical	15	DSPE	Synergie Fusion RP80A,	8	
006	Fully identified (SANCO/1/257/2013)			GC	MS/MS	QQQ	EI	+	Yes	Yes	No	No	Agilent 15	Acetone/DCM/PE	No	Mechanical	1	None	DB5	1		
007	Fully identified (SANCO/1/257/2013)	30	5	confirmed on ToF, GC-MS	LC	ToF	Q-TOF	Jet Stream	+	No	No	NA	Agilent 6530 accurate mass Q-ToF LCMS	10	AcN		0.5	None	C18	3		
008	Fully identified (SANCO/1/257/2013)			GC	MS/MS				Yes	Yes				13	AcN	No	Mechanical					
009	Fully identified (SANCO/1/257/2013)	1/-0.2	<5 min	acc. m/z + isotope + fragment	LC	MS/MS	Q-TOF	ESI	+	No	No	0.05 - 0.1	Yes	API 5600 Q-ToF	10	AcN	No	Mechanical	5	None	C18 Aqua Phenomenex	20
010	Fully identified (SANCO/1/257/2013)			LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.010		Waters XEVO TQ	10	AcN	No	Manual	2	DSPE	Acuity BEH C18	10	
011	Fully identified (SANCO/1/257/2013)	3	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.005-0.1	No	Xevo TQS	10	AcN	No	2	None	C18	2.5		
012	Tentatively detected	3	1	2 Transitions	LC	MS/MS	Q-TOF	ESI	+	No	No	0.005-0.1	Yes	TripleTOF	10	AcN	No	2	None	C18	2	
013	Fully identified (SANCO/1/257/2013)		2 Transitions	GC	MS/MS	QQQ	EI	+	Yes	Yes	0.01		TSQ QUANTUM	10	AcN	No	Manual	5	SPE	C18	1	
014	Tentatively detected	5	5	LC	MS	TOF	ESI	+	Yes	No	0.01 - 0.10	Yes	LCT PREMIER	15	Acetone/DCM/PE	No		1.5	None	C18	5	
015	Fully identified (SANCO/1/257/2013)	2	10	Accurate mass	GC	MS/MS	QQQ	EI	+	Yes	Yes	0.01	Yes	Agilent 7890/7000	10	Cyclohexane/EtOAc/Acetone	No		DSPE	HP5-MS	1	
016	Fully identified (SANCO/1/257/2013)	-0.36		GC	MS	Q	EI	+	Yes	Yes	0.01	No	Agilent 7000	10	AcN	Yes	1	DSPE	HPSMSU	10		
017	Fully identified (SANCO/1/257/2013)	10	0.02	GC	MS	Q	EI	+	Yes	Yes	0.01	Yes	Thermo DSQ II	10	AcN	Yes	Manual	2	QueChERS	ZB-5ms	1	
																		Xcalibur	300			
																		Weekly				

APPENDIX 3. Methods used by participants for detecting pesticides.

Ethioprophos											
Laboratory Code *NRL	How was the detection/differentiation done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Chromatographic Technique	Detector	Analyser	Polarity	Routine Method?	Screening Detection limit (SDL) (mg/kg)	Instrument Model
018 Fully identified (SANCO/1257/2013)				LC MS/MS QQQ	+ Yes Yes	0.010 No	Qtrap3200 10	AcN	No Manual	1 SPE	C18 5 Analyst 205 Every batch
019 Fully identified (SANCO/1257/2013)				LC NS/MS Q-TRAP ESI	+ Yes Yes	0.01 No	5500 10	AcN	No Manual	1 DSPE	C18 10 Analyst
020 Fully identified (SANCO/1257/2013)	2 Transitions	GC MS/MS	QQQ EI + Yes Yes	0.005 No	TSQ Quantum XLS Ultra	10 AcN	No Mechanical	30 DSPE	RTX5-MS 1 TraceFinder	300 Always	
022 Tentatively detected		GC MS/MS	ion trap CI	Yes Yes	0.002 Yes	saturn 2000 10 varian	AcN	No	5 PSA/MgSO4	factor four	10
023 Fully identified (SANCO/1257/2013)		GC MS	IT	No No	Yes Varian 240MS	15 Acetone/DCM/PE	No	1	1 GPC	VF-5MS 3 Auto	127
024 Fully identified (SANCO/1257/2013)	3	LC MS/MS	QQQ ESI	+ Yes Yes	0.002 No	ABSciex 4000 QTRAP mg/kg	10 AcN	No	1 QueCHERS without PSA	Atlantis 3, 21x100 mm, 3µm C18	2 Analyst 250 Before and after sample
025 Fully identified (SANCO/1257/2013)	3 0	LC NS/MS	QQQ ESI	+ Yes Yes	0.01 No	Waters Quattro 1st	10 MeOH/Water	No	1 None	Accuity BEH C18	5 Masslynx 224 Always
026 Tentatively detected	0.01 5	LC MS/MS	Q-Orbitrap	+ Yes Yes	0.005 Yes	Q Exactive 10	AcN	No	10 DSPE	BEH C18	5 700
027 Fully identified (SANCO/1257/2013)	0	GC NS/MS	QQQ EI	+ Yes Yes	0.010 No	Thermo 10 Acetone/DCM/PE	No	1	None	HP5-MS 1 Thermo	201 Every batch
028 Fully identified (SANCO/1257/2013)	0.1 0 no	GC MS	Q EI + Yes Yes	0.01 No	Agilent 6890/5973N	10 AcN	No	2 None	HP5-MS 2 Agilent Chemstation	184 No	
029 Fully identified (SANCO/1257/2013)	2 Transitions	LC	QQQ ESI	+ Yes Yes	0.01 No	API 5500 10	AcN	Yes Mechanical	15 DSPE	Pursuit XRs Ultra	3 Both 550 Every batch
030 Fully identified (SANCO/1257/2013)	1 5	GC MS	IT EI	Yes Yes	0.01 Yes	Varian 4000 10	AcN	No Mechanical	1 SPE	Rxi 5ms 5% diphenyl/ 95% dimethyl polysiloxane	1 Varian Ms Workstation 196 Every batch
031 Fully identified (SANCO/1257/2013)	max 0.5%	GC MS	Q EI + Yes Yes	0.01 Yes	Trace DSQ 15	AcN	Yes	Manual	1 SPE	DB5MS 0.8 Xcalibur	350 Every batch
032 Fully identified (SANCO/1257/2013)	2 Transitions	LC MS/MS	QQQ EI	+ Yes Yes	0.01 No	Waters Aquity UPLC system, API 5000 Trippelkvadrupol mass spectrometer Applied Biosystems	EtOAc	Yes Mechanical	18 Filtration	HSS T3 2 Analyst	404 Every batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/differentiation done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Sample Weight (g)	Instrument Model	Full Scan	Acetone/DCM/PE	No	Varian 320	7.5	Acetone/DCM/PE	No	Manual	1	Liquid-Liquid Partitioning	DB5	1	Workstation	NS		Frequency Standard Solution (pesticides Only) or library	Software	Injection Volume (μL)	Compounds in method (pesticides Only)	Standard Solution (pesticides Only) or library	Frequency		
033	Tentatively detected	2 Transitions	GC	MS/MS	QQQ	EI	+	No	No	No	Varian 320	7.5	Acetone/DCM/PE	No	Manual	1	Liquid-Liquid Partitioning	DB5	1	Workstation													
034	Fully identified (SANCO/1/257/2013)	<0.2 min	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	No	0.01	No	API 3200	10	AcN	Yes	Mechanical	20	DSPE	Kinetex C18	20	Analyst	139	Every batch									
035	Fully identified (SANCO/1/257/2013)	2.5%	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.01	No	HPLC Agilent 1100 MS API	10	AcN	Yes		15	DSPE	C18 50x2mm	10	Both	method 550	Always									
036	Fully identified (SANCO/1/257/2013)		GC	MS/MS	QQQ	EI	+	Yes	Yes	0.01	No	Agilent 7000B	5	AcN	Yes		3	None	HP-5MS UI	2	Mass Hunter	550	Daily										
037	Tentatively detected	<30	5 isotope	LC	MS	Orbitrap	ESI	+	No	Not Established	Yes	Agitive	AcN/1% acetic acid	No	Mechanical	30	Liquid-Liquid Partitioning	C18 Atlantis	5	ToxiD	600	Every batch (100+ pesticides)											
038	Fully identified (SANCO/1/257/2013)	<20	spectrum	GC	MS	TOF	EI	+	Yes	Yes	0.2	Yes	Leco Pegasus IV	AcN/1% acetic acid	No	Mechanical	30	Liquid-Liquid Partitioning + DSPE	RTX-Capillaries	10	MetAlign + ChromatOf	560	Every batch (235 pesticides)										
039	Fully identified (SANCO/1/257/2013)	0.30	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.005	No	Waters Quattro Premier XE	15 Acetone/DCM/PE	No	Mechanical	0.5	None	Acquity BEH (100x2.1 mm i.d.)	5	TargetLynx	21.5	Every batch										
040	Tentatively detected	0 +18.1 ppm	accurate mass + MRM	LC	MS	Q-ToF	ESI	+	No	Yes	0.01	No	4000	10	AcN	No	Mechanical	5	DSPE	C18	10	Analyst	Daily										
041	Tentatively detected	-1	5 GC-QTOF	LC	MS	Q-ToF	ESI	+	No	Yes	0.01	Yes	Waters Xevo (gen)	EiOAc	Yes		4	None	Acquity BEH C18	3	TargetLynx + MassFragment tool	500	Always										
042	Fully identified (SANCO/1/257/2013)		2 Transitions	GC	MS/MS	QQQ	EI	+	Yes	Yes	0.01	No	Varian 1200	10	EiOAc	No	Mechanical	20	DSPE	DB5-MS	5	Workstation	17	Every batch									
043	Tentatively detected	0.5%	0.3 2 Transitions	GC	MS	Q	EI	Yes	Yes	0.01	No	Hewlett Packard 5870 & 5973 LC/MS	15 Acetone/DCM/PE	No	Mechanical	1	Liquid-Liquid Partitioning	HP5-MS	1	Chemstation	201	Day of run											
044	Fully identified (SANCO/1/257/2013)	0.6		GC	MS	Q	EI	+	Yes	Yes	0.02	Yes	AT5975 GC-MS	10 AcN/EtOAc	No		2	SPDE	HP5-MS	10	DRS/Chemstation	-750	Always										
045	Fully identified (SANCO/1/257/2013)	0.2	2 Transitions	GC	MS/MS	QQQ	EI	Yes	Yes	0.010	No	7000 Agilent	10 AcN	No	Mechanical	5	DSPE	HP5-MS	2	Mass Hunter	150	No screening methods											
046	Tentatively detected	0.01	2	LC	MS	TOF	ESI	+	Yes	Yes	0.02	Yes	Bruker maxis 10 EiOAc	10		15	None	C18	2	TargetAnalysis	750	Always											
047	Fully identified (SANCO/1/257/2013)	0.08	3 Transitions	GC	MS	IT	EI	+	Yes	Yes	0.01	No	Variation Saturn 4000	10 AcN	No	Mechanical	20	None	Restek Rx-5ms	3	Variation Ms Workstation	282	Every batch										

APPENDIX 3. Methods used by participants for detecting pesticides.

Ethiopropos											
Laboratory Code *NRL	How was the detection/differentiation done?	RT Deviation (s)	MS Tolerance (ppm)	Other differentiation Details	Chromatographic Technique	Detector	Analyzer	Polarity	Routine Method?	Screening Detection limit (mg/kg)	Injection Volume (μl)
048 Fully identified (SANCO/1257/2013)	1		GC	MS Q EI + Yes Yes	Agilent 7890A GC 5975 MSD	15	ACN	Yes	DSPE	HP 5 MS % 5 phenyl methyl siloxane	2 Auto 174 Every batch
049 Fully identified (SANCO/1257/2013)			GC	MS/MS QQQQ EI Yes Yes	Agilent 6490 LC/MS/MS	15	Acetone/DCM/PE	No	None	C18 10 Mass Hunter	250 Every batch
050 Fully identified (SANCO/1257/2013)	2 Transitions	LC	MS QQQQ ESI + Yes Yes	Agilent 3800 GC + 320-MS	10	ACN	No	None	XBRIDGE	20 Xcellibur	150 Every batch
051 Fully identified (SANCO/1257/2013)	3 Transitions	GC	MS/MS QQQQ EI Yes Yes	Agilent 7000B	10	ACN	Yes	Mechanical	DSPE	Rxi-1ms 3 Workstation	363 Every batch
052 Fully identified (SANCO/1257/2013)	2 Transitions ratio 30%	GC	MS/MS IT EI Yes Yes	Agilent 7000B	10	ACN	No	Mechanical	DSPE	CP Sil8 2 Analyst	2 Every batch
053 Fully identified (SANCO/1257/2013)	0 1 Accurate mass	LC	MS/MS TOF ESI + Yes Yes	XEVOL G2-S QTof	10	ACN	Yes	Mechanical	DSPE	C18 1 Unity	900 Often
054 Fully identified (SANCO/1257/2013)	2.5 2 Transitions	LC	MS/MS QQQQ ESI + Yes Yes	Agilent 6440A	10	ACN	Yes		SPE	ZORBAX ECLIPSE 20	Auto 143 Every batch
055 Fully identified (SANCO/1257/2013)	none	2 Transitions	LC	MS/MS QQQQ ESI + Yes Yes	API 4000	10	ACN	Yes	DSPE	Waters-C18 2.1x50mm 1.7,μm	600 pest/total (10 LC/MS/MS methods) weekly (200 pesticides)
056 Tentatively detected	1 1	GC	MS Q EI Yes No	Agilent 5973	10	ACN	No	Manual	PSA	HP-5MS UI 2 Analyst	923 Every batch
057 Fully identified (SANCO/1257/2013)	2 Transitions	GC	MS/MS QQQQ EI + Yes Yes	Quattro micro	10	ACN	No	Manual	DSPE	SE54 5 MassLynx	110
058 Tentatively detected	15 1 target + qualifier	GC	MS Q EI + Yes Yes	Agilent GC - MSD	10	ACN	Yes	Mechanical	PSA	DB5 10 ChemStation	>600 Every batch
059 Fully identified (SANCO/1257/2013)	2	GC	MS/MS QQQQ EI + Yes Yes	SCION	10	ACN	No	Manual	DSPE	BR5MS 2 Workstation	127 Always
060 Fully identified (SANCO/1257/2013)		GC	MS Q EI + No No	Yes Agilent	10	ACN		Manual	DSPE	HP5-MS 20 AMDIS	~550
061 Fully identified (SANCO/1257/2013)	0 0 2	GC	MS/MS QQQQ EI	No 0.005	TSQ Quantum XLS Thermo	10	ACN	Yes	DSPE	DB5MSUI 2 TraceFinder	50 Every batch
062 Tentatively detected -0.3		GC	MS Q EI + Yes No	Yes 0.05	Agilent GC-MSD 7890A/5975C	10	ACN	Yes	DSPE	HP5-MS 1	Auto 927 Weekly
063 Tentatively detected 30 5	high resolution mass spectrum precursor ion	LC	MS/MS Q-TOF ESI + No No	No 0.001	Triple TOF 4,000 AB Sciex	10	ACN	Yes	DSPE	C18 5 Analyst	500 10%
064 Tentatively detected		GC	MS TOF EI + No No	Yes Pegasus 4D	15 Acetone/PE/DCM	15	ACN	No Mechanical	None	TR5MS 1 Chroma TOF	76

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/denification done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Chromatographic Technique	Detector	Polarity	Routine Method?	Routine Scope?	Screening Detection Limit (SDL) (mg/kg)	Instrument Model	Sample Weight (g)	Extraction Solvent	pH adjusted	Agitation Mode	Agitation time (min)	Column Type	Injection Volume (μL)	Software	Standard Solution (pesticides Only)	Frequency	
065 (SANCO/1/257/2013)	Fully identified	45	3 diagnostic ions	GC	MS	Q	EI	+	Yes	0.01	Shimadzu QP2010+	10	ACN	No		30	DSPE	VF-5-MS-30M*125*0.25μm	10 GC-MS solution	112	Monthly	
066 (SANCO/1/257/2013)	Fully identified		2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	0.01	Waters TQD	10	ACN	Yes	Mechanical	16	None	Acuity UPLC BECH C18 1.7 mm 2.1 x 100 mm	14		200	
067 (SANCO/1/257/2013)	Fully identified		2 Transitions	LC	NS/MS	QQQ	ESI	+	Yes	0.01	No	Agilent 6460	10	ACN	No	Mechanical	1	DSPE	C18	10	Mass Hunter	193
068 (SANCO/1/257/2013)	Fully identified	0	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	10	No	API4000	10	ACN	Yes		2	DSPE	rp-amide	10	Analyst	170
																					Every batch	

APPENDIX 3. Methods used by participants for detecting pesticides.

Fenpropidin											
Laboratory *NRL Code	How was the detection/identification was done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Chromatographic Technique	Detector	Analyzer	Polarity	Routine Method?	Screening Scope?	Screening Detection Limit (SDL)? (mg/kg)
				Instrument Model	Extraction Solvent	pH adjusted	Agitation Mode	Agitation time (min)	Injection Volume (μL)	Compounds in method (pesticides Only)	Standard Solution Frequency
001	Fully identified (SANCO/1257/2013)	3 Transitions	GC MS/MS	QQQ	EI +	Yes	Yes	0.01	No	Agilent 7000	10
003	Fully identified (SANCO/1257/2013)		LC NS/MS	ESI +	Yes	No				ACN	No
004	Tentatively detected Fully identified (SANCO/1257/2013)	19.2 1.42	5 fragment ions 2 Transitions	LC MS/MS	Q-TOF	ESI +	Yes	0.01	Yes	Agilent 6520	10
006	Fully identified (SANCO/1257/2013)		GC MS/MS	QQQ	EI +	Yes	Yes		No	Agilent	10
007	Tentatively detected	30	LC ToF	Q-TOF	Jet Stream	+ No	No	NA	Yes	Agilent 6530 Q-ToF LC/MS	10
008	Fully identified (SANCO/1257/2013)		LC NS/MS			Yes	Yes			ACN	13
009	Fully identified (SANCO/1257/2013)	+/- 0.2 min <5 acc. m/z + isotope + fragment	LC NS/MS	Q-TOF	ESI +	No	No	0.05 - 0.1	Yes	AP 5600 Q-ToF	10
010	Fully identified (SANCO/1257/2013)		LC NS/MS	QQQ	ESI +	Yes	Yes	0.010		Waters Xevo TQ	10
011	Fully identified (SANCO/1257/2013)	3	LC NS/MS	QQQ	ESI +	Yes	Yes	0.005-0.1	No	Xevo TQS	10
012	Tentatively detected Fully identified (SANCO/1257/2013)	3 0.6 2 Transitions	LC NS/MS	Q-TOF	ESI +	No	No	0.005-0.1	Yes	TripleTOF	10
013	Tentatively detected	10	GC NS/MS	QQQ	EI +	Yes		0.01		Ultra Thermo Q Exactive	10
014	Tentatively detected	5 5	LC NS/MS	TOF	ESI +	Yes	No	0.01 - 0.10	Yes	LCT PREMIER XE	15
015	Fully identified (SANCO/1257/2013)	3 10	Accurate mass GC	NS/MS	QQQ	EI	Yes	0.01	Yes	5977	10
016	Fully identified (SANCO/1257/2013)	0.42	GC NS/MS	QQQ	EI +	Yes	Yes	0.01	No	Agilent 7000	10
018	Fully identified (SANCO/1257/2013)		LC NS/MS	QQQ	ESI +	Yes	Yes	0.010	No	Qtrap3200	10
020	Fully identified (SANCO/1257/2013)		GC NS/MS	QQQ	EI +	Yes	Yes	0.005	No	TSQ Quantum XL Ultra	10

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/identification done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Routine Method?	Routine Scope?	Screening Detection Limit (SDL)? (mg/kg)	Instrument Model	Sample Weight (g)	Fenpropidin		Standard Solution (pesticides Only) or library method		Frequency			
														Clean up Step	Column Type	Injection Volume (μL)	Software	Agitation time (min)	Compounds in method	Masslynx	400
021	Fully identified (SANCO/1257/2013)		2 Transitions	LC MS/MS QQQ	ESI + Yes	0.005	No	Waters Xevo TQ	15	ACN	No	Mechanical	5	None	HSS T3	10	Masslynx	400	Before and after sample	Before and after sample	
024	Fully identified (SANCO/1257/2013)	3	304.2>130.1, 304.2>146.9	LC MS/MS QQQ	ESI + Yes	0.002	No	ABSciex 4000 QTRAP	10	ACN	No	Manual	1	QueCHERS without PSA	Atlantis T3, 2.1× 100 mm, 3 μm	2	Analyst	250			
025	Fully identified (SANCO/1257/2013)	3	0	2 Transitions	LC MS/MS QQQ	ESI + Yes	0.01	No	Waters Quattro 1st	10	MeOH/Water	No	Mechanical	1	None	Acquity BEH C18	5	Masslynx	224	Always	Always
026	Tentatively detected	5	Accurate mass	LC NGS/MS Q-Orbitrap	ESI + No	0.010	Yes	Q Exactive	10	ACN	No	Mechanical	10	DSPE	BEH C18	5		700			
027	Fully identified (SANCO/1257/2013)	0	2 Transitions	LC MS/MS QQQ	ESI + Yes	0.010	No	Xevo PE	10	Acetone/DCM/ PE	No	Mechanical	1	None	C18	2	Waters	286	Every batch	Every batch	
028	Fully identified (SANCO/1257/2013)	0.1	0	no	GC MS Q	EI + Yes	0.01	No	Agilent 6890/5973N	10	ACN	No	Manual	2	None	HP-MS	2	Agilent ChemStation	184	No	No
029	Fully identified (SANCO/1257/2013)		2 Transitions	LC QQQ	ESI + Yes	0.01	No	API 5500	10	ACN	Yes	Mechanical	15	DSPE	Pursuit XR's Ultra	3	Both	550	Every batch	Every batch	
030	Fully identified (SANCO/1257/2013)	2	5	GC MS IT	EI Yes	0.01	Yes	Varian 4000	10	ACN	No	Mechanical	1	SPE	diphenyl/95% dimethyl polysiloxane	1	Varian MS Workstation	196	Every batch	Every batch	
031	Fully identified (SANCO/1257/2013)	Max 2.5%	1	2 Transitions	LC MS/MS QQQ	ESI + Yes	0.01	No	Quattro Premier XE	15	ACN	Yes	Manual	1	None	C18	3	Quanlynx	350	Every batch	Every batch
032	Fully identified (SANCO/1257/2013)		2 Transitions	LC MS/MS QQQ	EI + Yes	0.05	No	Waters UPLC system, API 5000 QQQ mass spectrometer Applied Biosystems	10	EtOAc	Yes	Mechanical	18	Filtration	HSS T3	2	Analyst	107	Every batch	Every batch	
033	Tentatively detected		2 Transitions	GC MS/MS QQQ	EI + No	No	No	Variian 320	7.5	Acetone/DCM/ PE	No	Manual	1	Liquid-Liquid Partitioning	DB5	1	MS Workstation				
034	Fully identified (SANCO/1257/2013)	< 0.2 min	2 Transitions	LC MS/MS QQQ	ESI + Yes	0.01	No	API 3200	10	ACN	Yes	Mechanical	20	DSPE	Kinetex C18	20	Analyst	139	Every batch	Every batch	
035	Fully identified (SANCO/1257/2013)	2.5%	2 Transitions	LC MS/MS QQQ	ESI + Yes	0.01	No	HPLC Agilent 1100, MS AP 13000	10	ACN	Yes	None	15	DSPE	C18 50x2mm	10	Both	method 550	Always	Always	
036	Fully identified (SANCO/1257/2013)			LC NGS/MS QQQ	ESI + Yes	0.01	No	Agilent 6490	5	ACN	Yes	None	3	None	RPI 8	5	Mass Hunter	550	Daily	Daily	
037	Fully identified (SANCO/1257/2013)	<20	spectrum	GC NS TOF	EI + Yes	Not Established	Yes	Leco Pegasus IV	1% acetic acid	No	Mechanical	30	Liquid-Liquid Partitioning + DSPE	RTX-Clipesticides	10	MetAlign + ChromatOF	560	Every batch (235 pesticides)	Every batch (235 pesticides)		
038	Fully identified (SANCO/1257/2013)	10	2 Transitions	LC MS/MS QQQ	ESI + Yes	0.01	No	4000	10	ACN	No	Mechanical	5	DSPE	C18	10	Analyst		Daily	Daily	
039	Fully identified (SANCO/1257/2013)	0.06	2 Transitions	LC MS/MS QQQ	ESI + Yes	0.005	No	Waters Quattro Premier XE	15	Acetone/DCM/ PE	No	Mechanical	0.5	Acquity BEH (100x2, 1 mm.i.d.)	Targetlynx	215	Every batch	Every batch	Every batch		

APPENDIX 3. Methods used by participants for detecting pesticides.

Fenpropidin											
Laboratory *NRL Code	How was the detection/identification was done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Chromatographic Technique	Detector	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Detection Limit (SDL)? (mg/kg)
				Instrument Model	Extraction Solvent						
040	Tentatively detected	N/A no standard	-2.0 Accurate mass	LC MS Q-TOF	ESI + No No	0.01 Yes	Waters Xevo Q-TOF (1st gen)	10 EtOAc	Yes Mechanical	4 None	Acquity BEH C18 3 Target Lynx + MassFragment tool
041	Tentatively detected	-4 5	GC-QTOF	LC MS	Q-TOF ESI	+ Yes	Yes	Agilent 6550 iFunnel QTOF LC/MS	10 AcN	No	2 DSPE C18 5 Mass Hunter
042	Fully identified (SANCO/1257/2013)		2 Transitions	LC MS/MS	QQQ ESI	+ Yes Yes	0.01 No	Agilent 6490	10 EtOAc	No Mechanical	20 None HSS13 2 Mass Hunter
043	Tentatively detected	2.5% 0.3	1 Transition	LC MS/MS	QQQ ESI	+ Yes Yes	0.01 No	TSQ Quantum discovery max thermo	10 AcN	No Mechanical	1 Liquid-Liquid Partitioning Altima C18 150 >2.1 mm (Alltech 88370) 25 LC quan 150 DRS/MS 10 ChemStation ~750
044	Fully identified (SANCO/1257/2013)	-2.2		GC MS	Q ESI + Yes Yes	0.02 Yes	Agilent 7975 GC-MS	10 AcN/EtOAc	No	2 DSPE C18 5 Mass Hunter	265 Day of run
046	Tentatively detected	0.15 1.0	Accurate mass	LC MS	TOF ESI	+ Yes Yes	0.02 Yes	Bruker maxis	10 EtOAc	No	15 None C18 2 TargetAnalysis 750
047	Fully identified (SANCO/1257/2013)	1	2 Transitions	LC MS/MS	QQQ ESI	+ Yes Yes	0.01 No	Thermo Finnigan TSQ Quantum	10 AcN	No Mechanical	20 DSPE Agilent Eclipse C18 XDB 20 Xcalibur 282
048	Fully identified (SANCO/1257/2013)	1		GC MS	Q ESI + Yes Yes	0.010 Yes	Agilent 7890A GC 5975 MSD	15 AcN	Yes	1 DSPE phenyl methyl siloxane	200 Every batch
049	Fully identified (SANCO/1257/2013)			LC MS/MS	QQQ ESI	+ Yes Yes	0.01 No	Agilent 6490 LC/MS/MS	15 Acetone/DCM/ PE	No	1 None C18 10 Mass Hunter 250 Every batch
050	Fully identified (SANCO/1257/2013)		2 Transitions	LC MS	QQQ ESI	+ Yes Yes	0.010 No	TSQ QUANTUM	10 AcN	No	None XBRIDGE 20 Xcalibur 150 Every batch
051	Fully identified (SANCO/1257/2013)	0.1	2 Transitions	LC MS/MS	QQQ ESI	+ Yes Yes	0.01 Yes	Varian ProStar LC+320-MS	10 AcN	No Mechanical	3 None Rectek Ultra C18 10 Workstation 363 Every batch
052	Fully identified (SANCO/1257/2013)	12	2 Transitions ratio 30%	GC MS/MS	IT ESI	+ Yes Yes	0.010 Yes	Agilent 7000B	10 AcN	No Mechanical	20 DSPE CP Sil8 2 Analyst 2 Every batch
053	Fully identified (SANCO/1257/2013)	0 1	Accurate mass	LC MS/MS	TOF ESI	+ Yes Yes	0.01 Yes	XEVO G2-S QTOF	10 AcN	Yes Mechanical	15 DSPE C18 1 Unity 900 Often
055	Fully identified (SANCO/1257/2013)	none	2 Transitions	LC MS/MS	QQQ ESI	+ Yes Yes	0.01 No	API 4000	10 AcN	Yes Manual	5 DSPE Waters-C18 2.1x50mmx 1.7μm 5 Analyst (10 LC/MS/MS methods) 600 past total weekly (200 pesticides)
056	Tentatively detected	1 1		GC MS	Q ESI		Yes No	Agilent 5973	10 AcN	No Manual	1 PSA HP-5MS UI 2 Auto 923 Every batch
057	Fully identified (SANCO/1257/2013)		2 Transitions	LC MS/MS	QQQ ESI	+ Yes Yes	0.01 No	Quattro Premier	10 AcN	No Manual	2 DSPE C18 5 MassLynx 196
058	Tentatively detected	15 1	target + qualifier	GC MS	Q ESI + Yes Yes	0.01 - 0.05 Yes	Agilent GC -MSD	10 AcN	Yes Mechanical	10 PSA DB5 10 ChemStation >600 Every batch	
060	Fully identified (SANCO/1257/2013)		2 Transitions + GC/MS scan	LC MS/MS	Q-TRAP ESI	+ Yes Yes	Apie3200 Qtrap	10 AcN	Manual	2 DSPE Atlantis T3 5	

APPENDIX 3. Methods used by participants for detecting pesticides.

Fenpropidin									
Laboratory Code *NRL	How was the detection/identification done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification details	Chromatographic Technique	Detector	Analyzer	Routine Method?	Routine Scope?
062	Tentatively detected	-2.7		GC MS Q EI + Yes No 0.05 Yes Agilent GC-MSD 7890A/5975C	Instrument Model	Sample Weight (g)	Extraction Solvent	Agitation Mode	Agitation time (min)
064	Tentatively detected			GC MS TOF EI + No No Yes Pegasus 4D	Instrument Model	Sample Weight (g)	Extraction Solvent	Agitation Mode	Agitation time (min)
065 [SANCO/12571/2013]	Fully identified	60	3 diagnostic ions	GC MS Q EI + Yes No 0.02 Yes Shimadzu QP2010+	Instrument Model	Sample Weight (g)	Extraction Solvent	Agitation Mode	Agitation time (min)
066 [SANCO/12571/2013]	Fully identified		2 Transitions	LC MS/MS QQQQ ESI + Yes Yes 0.01 No Waters TQD	Instrument Model	Sample Weight (g)	Extraction Solvent	Agitation Mode	Agitation time (min)
067 [SANCO/12571/2013]	Fully identified		3 Transitions	GC MS/MS QQQQ EI Yes Yes 0.01 No Agilent 7000	Instrument Model	Sample Weight (g)	Extraction Solvent	Agitation Mode	Agitation time (min)
Standard Solution Frequency									
Compounds in method (pesticides Only) or library (pesticides Only)									
Software									
Injection Volume(µL)									
Column Type									
Clean up Step									
DSPE									
HP5-MS									
1									
Auto									
927									
Weekly									

APPENDIX 3. Methods used by participants for detecting pesticides.

Fipronil																							
Laboratory *NRL Code	How was the detection/identification done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Detector	Analyzer	Polarity	Routine Method?	Routine Scope?	Screening Detection Limit (SDL)? (mg/kg)	Instrument Model	Sample Weight (g)	Extraction Solvent	Agitation Mode	Clean up Step	Software	Compounds in method or library (pesticides Only)	Standard Solution Frequency					
001 Fully identified (SANCO/12571/2013)	3 Transitions	GC	MS/MS	QQQ	E	+	Yes	Yes	0.01	No	Agilent 7000	10	ACN	No	I	None	HP5-MS	1	Mass Hunter	350	Daily		
002 Fully identified (SANCO/12571/2013)		LC	MS/MS	QQQ	ESI	+	Yes	Yes			Agilent 6410	10	ACN/ACN		1	SPE	C18 4 micras	5	Mass Hunter	221			
003 Fully identified (SANCO/12571/2013)		LC	MS/MS	QQQ	ESI	+	Yes	No				10	ACN	Yes	1	SPE							
004 Fully identified (SANCO/12571/2013)	2 Transitions	GC	MS/MS	QQQ	E	+	Yes	Yes		No	Agilent	10	ACN	Yes	Mechanical	15	DSPE	HP5-MS	2	Mass Hunter	>300	Every batch	
005 Tentatively detected	17.4	2 Transitions	LC	MS/MS	QQQ	ESI	-	Yes	Yes	0.01	No	ABSciex API550	10	ACN	Yes	Mechanical	15	DSPE	Zorbax C18	8	Analyst	>400	Every batch
006 Tentatively detected		GC	MS/MS	QQQ	E	+	No	No		No	Agilent	15	Acetone/DCM/PE	No	Mechanical	1	None	DB5	1	Mass Hunter	176	No screening methods	
007 Fully identified (SANCO/12571/2013)	30	5 Confirmed on Tof, LC-MS/MS	LC	ToF	Q-ToF	Ief	Stream	+	No	NA	Yes	10	ACN		0.5	None	C18	3	Mass Hunter	550	NA		
008 Fully identified (SANCO/12571/2013)		GC	MS/MS									13	ACN	No	Mechanical		None						
009 Fully identified (SANCO/12571/2013)	+/- 0.2 min	<5	acc. m/z + isotope + fragment	LC	MS/MS	Q-ToF	ESI	-	No	0.05 - 0.1	Yes	API 5000 Q-ToF	10	ACN	No	Mechanical	5	None	C18 Aqua Phenomenex	20	Both	600	2 Months
010 Fully identified (SANCO/12571/2013)		GC	MS/MS	QQQ	E	+	Yes	Yes	0.010		Bruker SCION	10	ACN	No	Manual	2	DSPE	Zerion ZB	2	Bruker	210	Daily	
011 Fully identified (SANCO/12571/2013)	3	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.0050-1	No	Xevo TQS	10	ACN	No		2	None	C18	2.5	MassLynx	300	Every batch
012 Fully identified (SANCO/12571/2013)		2 Transitions	LC	MS/MS	QQQ	ESI	-	Yes	Yes	10	No	UltraThermo	10	ACN	No	Manual	5	DSPE	C18	10			
013 Tentatively detected	10	2	GC	MS/MS	QQQ	E	+	Yes		0.01		Agilent 7890/Agilent 7000	10	ACN	Yes		5	DSPE	DB5-MS	5	Mass Hunter	~500	
014 Tentatively detected	0	0.08	2	LC	MS/MS	Q-Orbitrap	ESI	-	Yes	0.01		Thermo Q-Exactive	10	ACN	Yes		5	DSPE	C18	5	Xcalibur	~500	
015 Fully identified (SANCO/12571/2013)	5	10	Accurate mass	GC	MS/MS	QQQ	E		Yes	0.01	Yes	5977	10	Cyclohexane/EtOAc/Acetone	No		3	DSPE	HP5-MS	1	Mass Hunter	850	Daily
016 Fully identified (SANCO/12571/2013)	0			LC	MS/MS	Q-TRAP	ESI	+	Yes	0.01	No	ABSciex 4000 QTrap	10	ACN	Yes	1	None	Phenomenex Synergie Fusion RP 2.5 μ m 50x2.0	55	MultiQuant	~250	Always	

APPENDIX 3. Methods used by participants for detecting pesticides.

Fipronil												
Laboratory Code *NRL	How was the detection/identification done?	RT Deviation(s)	MS Tolerance (ppm)	Other identification details	Chromatographic Technique	Detector	Analyzer	Routine Method?	Routine Scope?	Screening Detection Limit (SDL)? (mg/Kg)	Instrument Model	
018 [SANCO/1257/2013]	Fully identified Fully identified [SANCO/1257/2013]	2 Transitions	GC	MS/MS	QQQ	LC	MS/MS	QQQ	+	Yes	Yes	
019 [SANCO/1257/2013]	Fully identified Fully identified [SANCO/1257/2013]	2 Transitions	GC	MS/MS	QQQ	E	+ Yes	Yes	0.010	No	Qtrap3200	
020 [SANCO/1257/2013]	Fully identified Fully identified [SANCO/1257/2013]	2 Transitions	GC	MS/MS	QQQ	E	+ Yes	Yes	0.005	No	Quantum XLS Ultra	
022 Tentatively detected			GC	MS/MS	ion trap	E	Yes	Yes	0.002	Yes	saturno 2000 variian	
024 Fully identified [SANCO/1257/2013]	3	434.7>329.8, 434.7>249.7	LC	MS/MS	QQQ	EI	- Yes	Yes	0.002	No	ABSciex 4000 QTRAP	
025 Fully identified [SANCO/1257/2013]	3	0	2 Transitions	LC	MS/MS	QQQ	EI	- Yes	0.01	No	Waters Quattro LST	
026 Tentatively detected	0	5	Acccurate mass	LC	MS/MS	Q-Orbitrap	EI	- Yes	0.005	Yes	Q Exactive	
027 Fully identified [SANCO/1257/2013]	0	2 Transitions	GC	MS/MS	QQQ	EI	+ Yes	Yes	0.010	No	Thermo 10 Acetone/DCM/PE	
028 Fully identified [SANCO/1257/2013]	0.1	0	no	GC	MS	Q	E	+ Yes	Yes	0.01	No	Agilent 6890/5973N
029 Fully identified [SANCO/1257/2013]			spectrum	GC	TOF	EI	Yes	Yes	0.01	Yes	LECO Pegasus IV	
030 Fully identified [SANCO/1257/2013]	0	5		GC	MS	IT	EI	Yes	0.01	Yes	Varian 4000	
031 Fully identified [SANCO/1257/2013]	max. 2.5%	1	2 Transitions	LC	MS/MS	QQQ	EI	- Yes	0.01	No	Quattro Premier XE	
032 Fully identified [SANCO/1257/2013]			2 Transitions	GC	MS/MS	QQQ	EI	+ Yes	Yes	0.01	No	Waters Quattro Micro GC
033 Tentatively detected			2 Transitions	GC	MS/MS	QQQ	EI	+ Yes	No	No	Variian 320 7.5 Acetone/DCM/PE	
034 Fully identified [SANCO/1257/2013]	< 0.2 min	2 Transitions	GC	MS/MS	QQQ	EI	+ Yes	No	0.01	No	Agilent 7890A	

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/identification was done?	RT Deviation (s)	MS Tolerance (ppm)	Other Identification Details	Detector	Analyzer	Polarity	Routine Method?	Routine Scope?	Screening Detection Limit (SDL)? (mg/kg)	Instrument Model	Sample Weight (g)	Extraction Solvent	pH adjusted	Agritation Mode	Clean up Step	Injection Volume (μL)	Compounds in method or library (pesticides Only)	Standard Solution Frequency
035 Fully identified (SANCO/1257/2013)	2.5%	2 Transitions	LC MS/MS QQQ	ESI - Yes Yes 0.01	No Agilent HPLC; 1290 Infinity, MS; 6460 Triple Quad LC/MS, Model: G6460A	10 ACN	No	15	None	C18 50x2mm	5	Both	method 550	Always					
036 Fully identified (SANCO/1257/2013)	<30	5	isotope	LC MS Orbitrap ESI + Yes Yes 0.01	No Agilent 7000B	5 ACN	Yes	3	None	HP-5MS UI	2	Mass Hunter	550	Daily					
037 Tentatively detected	<30	5	isotope	GC MS Orbitrap ESI - No No Not Established	Yes	Not Established	Exactive	ACN/1% acetic acid	No	Mechanical	30 Liquid-Liquid Partitioning	C18 Atlantis	5	ToxD	600	Every batch (100+ pesticides)			
Fully identified (SANCO/1257/2013)	<20		spectrum	GC MS TOF E + Yes Yes Not Established	Yes	Not Established	Exactive	ACN/1% acetic acid	No	Mechanical	30 Liquid-Liquid Partitioning	C18 Atlantis	5	ToxD	600	Every batch (235 pesticides)			
038 Fully identified (SANCO/1257/2013)	10	2 Transitions	LC MS/MS QQQ	ESI - Yes Yes 0.01	No 4000	10 ACN	No	Mechanical	5 DSPE	C18	10 Analyst	Daily							
039 Fully identified (SANCO/1257/2013)	0	2 Transitions	LC MS/MS QQQ	ESI - Yes Yes 0.005	No Waters Quattro Premier XE	15 Acetone/DCM/ PE	No	Mechanical	0.5 None	Acquity BEH (100x2.1 mm i.d.)	5 Targetlynx	215	Every batch						
040 Fully identified (SANCO/1257/2013)	0	0	accurate mass + MRM	LC Q-TOF ESI - No Yes 0.01	Yes Waters Xevo Q-TOF (1st gen)	10 EtOAc	Yes	4	None	Acquity BEH C18	3 Massfragment tool	500	Always						
041 Tentatively detected	-5	1	GC-QTOF	LC MS Q-TOF ESI + Yes Yes	Yes Agilent 65250 QTOF LC/MS	10 ACN	No	2 DSPE	C18	5	Mass Hunter	1700	Always						
042 Fully identified (SANCO/1257/2013)		2 Transitions	LC MS/MS QQQ	ESI - Yes Yes 0.01	No Agilent 6490 EtOAc	10	No	Mechanical	20 None	HSST3	2 Mass Hunter	265	Every batch						
043 Tentatively detected	0.5%	0.3	2 Transitions	GC MS Q E + Yes Yes 0.01	No Hewlett Packard 5870 GC-MS & 5973	15 Acetone/DCM/ PE	No	Mechanical	1 Liquid-Liquid Partitioning	HP5-MS	1 Chemstation	201	Day of run						
044 Fully identified (SANCO/1257/2013)	12.2		GC MS/MS QQQ	E Yes Yes 0.02	Yes A15975 GC-MS	10 ACN/EtOAc	No	2	HP5-MS	10 Chemstation	~750	Always							
045 Fully identified (SANCO/1257/2013)	0.2	2 Transitions	GC Accurate mass	MS TOF E + Yes Yes 0.005	No 7000 Agilent 10 ACN	No Mechanical	5 DSPE	2 Mass Hunter	150	No screening methods									
046 Tentatively detected	0.3	4.8	Accurate mass	LC MS TOF ESI + Yes Yes 0.02	Yes Bruker maxis 10 EtOAc	15 None	C18	2 TargetAnalysis	750	Always									

APPENDIX 3. Methods used by participants for detecting pesticides.

Fipronil											
Laboratory Code *NRL	How was the detection/identification done?	RT Deviation(s)	MS Tolerance (ppm)	Other identification Details	Chromatographic Technique	Detector	Analyzer	Routine Method?	Routine Scope?	Screening Detection Limit (SDL)? (mg/kg)	Instrument Model
047 (SANCO/1257/2013)	Fully identified	3 0.04	3 Transitions	GC	MS	IT	EI	+	Yes	0.01	No
048 (SANCO/1257/2013)	Fully identified	1		GC	MS	Q	EI	+	Yes	0.010	Yes
049 (SANCO/1257/2013)	Fully identified			LC	MS/MS	QQQ	EI	-	Yes	0.01	No
050 (SANCO/1257/2013)	Fully identified	2	Transitions	LC	MS	QQQ	EI	-	Yes	0.010	No
051 (SANCO/1257/2013)	Fully identified	0	3 Transitions	GC	MS/MS	QQQ	EI		Yes	0.003	No
052 (SANCO/1257/2013)	Fully identified	12	2 Transitions ratio 30%	GC	MS/MS	TOF	EI		Yes	0.010	Yes
053 (SANCO/1257/2013)	Fully identified	0	1 Accurate mass	LC	MS/MS	TOF	EI		Yes	0.01	Yes
055 (SANCO/1257/2013)	Fully identified	none	2 Transitions	LC	MS/MS	QQQ	EI	+	Yes	0.01	No
056 Tentatively detected	Tentatively detected	1	1	GC	MS	Q	EI		Yes	No	Yes
057 (SANCO/1257/2013)	Fully identified		2 Transitions	LC	MS/MS	QQQ	EI	-	Yes	0.01	No
058 Tentatively detected	Tentatively detected	15	1 target + qualifier	GC	MS	Q	EI	+	Yes	0.01 - 0.05	Yes
059 (SANCO/1257/2013)	Fully identified	2		GC	MS/MS	QQQ	EI	+	Yes	0.010	No
060 (SANCO/1257/2013)	Fully identified	0	0	2	GC	MS/MS	QQQ	EI	No	0.005	No
061 (SANCO/1257/2013)	Fully identified	0		GC	MS	Q	EI	+	No	Yes	Agilent 10
062 Tentatively detected	Tentatively detected	2.3		GC	MS	TOF	EI	+	Yes	0.01	Yes
064 Tentatively detected				GC	MS	Q	EI	+	No	Yes	Pegasus 4D
065 Fully identified (SANCO/1257/2013)	Fully identified	60	2 Transitions	LC	MS/MS	QQQ	EI	-	Yes	0.01	No

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/identification was done?	RT Deviation (s)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Polarity	Routine Method?	Routine Scope?	Screening Detection Limit (SDL) (mg/kg)	Instrument Model	Sample Weight (g)	Extraction Solvent	pH adjusted	Agitation Mode	Agitation time (min)	Injection Volume (μL)	Software	Standard Solution Frequency
066 Fully identified (SANCO/12571/2013)	Fully identified	2 Transitions	GC	MS/MS	QQQ	EI	Yes	Yes	0.005	No	Agilent 7000	15	EtOAC	No	Mechanical	I	None	DB-5MS Capilar	2	200
067 Fully identified (SANCO/12571/2013)	Fully identified	2 Transitions	GC	MS/MS	QQQ	EI	Yes	Yes	0.01	No	Agilent 7000	10	ACN	No	Mechanical	I	DSPE	DB-5MS UI	2	Mass Hunter 236
068 Fully identified (SANCO/12571/2013)	0	2 Transitions	LC	MS/MS	QQQ	+	Yes	Yes	10	No	API4000	10	ACN	Yes		2	SPE	tp-amide	10	Analyst 170
																				Every batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/differentiation done?	RT Deviation (s)	MS Tolerance (ppm)	Other detection/differentiation details	Chromatographic Technique	Detector	Analyzer	Polarity	Routine Method?	Routine Scope?	Screening Detection Limit (SDL)? (mg/kg)	Instrument Model	Extraction Solvent	pH adjusted	Agitation Mode	Agitation time (min)	Injection Volume (μL)	Software	Standard Solution Frequency	
001 Fully identified (SANCO/1257/2013) 003	2 Transitions	LC	MS/MS	QQQ	ESI + Yes	Yes	0.01	No	Agilent 6460	10	AcN	No	1	None	Zorbax C18	5	Mass Hunter	200	Daily	
007 Fully identified (SANCO/1257/2013)	5 confirmed on ToF, LC-MS/MS	LC	MS/MS	Q-TOF	Jet Stream	+ No	No	NA	Yes	Agilent 6530 accurate mass Q-ToF LC/MS	10	AcN	Yes	1	SPE				NA	
008 Fully identified (SANCO/1257/2013)		LC	MS/MS			Yes	Yes				13	AcN	No	Mechanical	None					
010 Fully identified (SANCO/1257/2013)		LC	MS/MS	QQQ	ESI - Yes	Yes	0.010		Waters Xevo TQ	10	AcN	No	Manual	2	DSPE	Acquity BEH C18	10	Waters	25	
011 Tentatively detected	3 2 Transitions	LC	MS/MS	QQQ	ESI + Yes	No	0.005-0.1	No	Xevo TQS	10	AcN	No	2	None	C18	2.5	MastLynx	300	Every batch	
013 Tentatively detected	0.05	LC	MS/MS	Q-Orbitrap	ESI + Yes	Yes	0.01		Thermo Q-Exactive	10	AcN	Yes		5	DSPE	C18	5	Xcalibur	~500	
015 Fully identified (SANCO/1257/2013)	10 Accurate mass	LC	MS/MS	QQQ	EI	Yes	Yes	0.01	Yes	6490	10	AcN	No	3	DSPE	Poroshell 1	1	Mass Hunter	300	Daily
016 Fully identified (SANCO/1257/2013)	0	LC	MS/MS	Q-TRAP	ESI + Yes	Yes	0.01	No	ABSciex 5500 QTrap	10	AcN	Yes	1	None	Waters UPLC BEH C18	10	MultiQuant	~250	Always	
024 Fully identified (SANCO/1257/2013)	3 681.0>253.7, 681.0>273.9	LC	MS/MS	QQQ	ESI - Yes	Yes	0.008 mg/kg	No	ABSciex 4000 QTrap	10	AcN	No	Manual	1	QueChERS without PSA	Athenis T3, 2.1x100 mm, 3μm	2	Analyst	250	
026 Tentatively detected	5 Accurate mass	LC	MS/MS	Q-Orbitrap	ESI + No	No	0.010	Yes	Q Exactive	10	AcN	No	Mechanical	10	DSPE	BEH C18	5		700	
027 Fully identified (SANCO/1257/2013)	0 2 Transitions	LC	MS/MS	QQQ	ESI + Yes	Yes	0.010	No	Xevo 10	Acetone/DCM/PE	No	Mechanical	1	None	C18	2	Waters	286	Every batch	
030 (SANCO/1257/2013)	5 2 Transitions	LC	MS/MS	QQQ	ESI + Yes	Yes	0.01	No	AbSciex 3200 QTrap	10	AcN	No	Mechanical	1	SPE	Acquity BEH RP 18	50	Analyst	153	Every batch
032 Fully identified (SANCO/1257/2013)	2 Transitions	LC	MS/MS	QQQ	EI + Yes	Yes	0.01	No	EtOAc							HSS T3	2	Analyst	15	Every batch
035 Fully identified (SANCO/1257/2013)	2.5%	LC	MS/MS	QQQ	ESI + Yes	No	0.01	No	HPIC Agilent 1100, MS API 3000	10	AcN	Yes		15	DSPE	C18 50x2mm	10	Both	method 550	
036 Fully identified (SANCO/1257/2013)		LC	MS/MS	QQQ	ESI + Yes	Yes	0.01	No	Agilent 6490	5	AcN	Yes	3	None	RPI 8	5	Mass Hunter	550	Daily	

APPENDIX 3. Methods used by participants for detecting pesticides.

Flubendiamide											
Laboratory Code *NRL	How was the detection/differentiation was done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Chromatographic Technique	Detector	Analyzer	Polarity	Routine Method?	Screening Detection Limit (SDL) (mg/kg)	Instrument Model
037 Fully identified (SANCO/1257/2013)	<30 5 Isotope	10 2 Transitions	LC MS Orbitrap	ESI -	No Not Established	Yes Excative	1% Acn/ 1% acetic acid	No Mechanical	30 Liquid-Liquid Partitioning	C18 Atlantis 5	ToxID 600 Every batch (100+ pesticides)
038 Fully identified (SANCO/1257/2013)	120 2 Transitions	LC MS/MS	QQQ	ESI	-	Yes Yes	0.01	No 4000 AcN	5 DSPE	C18 10 Analyst	Daily
039 Fully identified (SANCO/1257/2013)	0 2 Transitions	LC MS/MS	QQQ	ESI	+	Yes Yes	0.005	No Waters Quattro Premier XE	15 Acetone/DCM/ PE	No Mechanical	0.5 None Acuity BEH (100x2.1 mm i.d.)
040 Fully identified (SANCO/1257/2013)	0 2 Transitions	LC MS/MS	QQQ	ESI	+	Yes Yes	0.01	No Waters Quattro Premier XE	10 EtOAc	Yes No Mechanical	4 None Acuity BEH C18
042 Fully identified (SANCO/1257/2013)	2 Transitions	LC MS/MS	QQQ	ESI	-	Yes Yes	0.01	No Agilent 6490	10 EtOAc	No Mechanical	20 None HSS3
044 Fully identified (SANCO/1257/2013)	0.2 2 Transitions	LC MS/MS	QQQ	ESI	-	Yes Yes		No AB4000	10 AcN	No No 2 None	2 Mass Hunter 265 Every batch
045 Fully identified (SANCO/1257/2013)	0.2 2 Transitions	LC MS/MS	QQQ	ESI	+	Yes Yes	0.010	No 6460 Agilent	10 AcN	No Mechanical	5 DSPE C18 5 Mass Hunter 150 No screening methods
046 Tentatively detected	0 0.1 Accurate mass	LC MS	TOF	ESI	+	Yes Yes	0.02	Yes Bruker maxis 10 EtOAc	10 Bruker maxis 10 EtOAc		15 None C18 2 Target Analysis 750 Always
048 Fully identified (SANCO/1257/2013)	1 2 Transitions	LC MS/MS	QQQ	ESI	+	Yes Yes	0.010	Yes Waters Acuity UPLC TQD	15 Acetone/DCM/ PE	Yes Yes Mechanical	1 1 DSPE UPLC BEH C18 10 Auto 336 Every batch
049 Fully identified (SANCO/1257/2013)		LC MS/MS	QQQ	ESI	-	Yes Yes	0.01	No Agilent 6490 LC/MS/MS	15 Acetone/DCM/ PE	No Mechanical	1 None C18 10 Mass Hunter 250 Every batch
053 Fully identified (SANCO/1257/2013)	0 1 Accurate mass	LC MS/MS	TOF	ESI	+	Yes Yes	0.01	Yes XEVO G2-S QToF	10 AcN	Yes Yes Manual	15 DSPE C18 1 Unity 900 Often
055 Tentatively detected none	2 Transitions	LC MS/MS	QQQ	ESI	+	No No	0.01	No API 4000	10 AcN	Yes No Mechanical	5 DSPE Waters C18 2.1x50mm 1.7µm 600 pest total (10 LC/MS/MS methods)
057 Fully identified (SANCO/1257/2013)	2 Transitions	GC	MS/MS	QQQ	EI	+	Yes Yes	0.01 No Quattro micro	10 AcN	No Yes Manual	2 DSPE SE54 5 Mass Lynx 110 Every batch
058 Tentatively detected	30 5 Accurate mass	LC	Orbitrap	ESI	+	Yes Yes	0.01 - 0.05	Yes Thermo 10 Orbitrap Ap 3200 Qtrap	10 AcN	Yes Yes Mechanical Manual	10 PSA RPL8 10 Xcalibur >100 Every batch
060 Fully identified (SANCO/1257/2013)	2 Transitions	LC MS/MS	Q-TRAP	ESI	+	Yes Yes		No Waters QTOF	10 AcN	Yes Yes Mechanical Manual	2 DSPE Atlantis T3 5 Acuity UPLC BEH C18 14 1.7 mm 2.1 x 100 mm 200 Daily
066 Fully identified (SANCO/1257/2013)	2 Transitions	LC MS/MS	QQQ	ESI	+	Yes Yes	0.01	No Agilent 6460	10 AcN	No Mechanical	1 DSPE C18 10 Mass Hunter 193 Daily
067 Fully identified (SANCO/1257/2013)	2 Transitions	LC MS/MS	QQQ	ESI	-	Yes Yes	0.01	No Agilent 6460	10 AcN	No Mechanical	

APPENDIX 3. Methods used by participants for detecting pesticides.

APPENDIX 3. Methods used by participants for detecting pesticides.

Fluometuron										
Laboratory Code *NRL	How was the detection/differentiation	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Detector	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Detection limit (SDL) (mg/kg)
003 Fully identified (SANCO/1/257/2013)	Tentatively detected			GC MS/MS	LC	MS/MS	El	+	Yes	No
006 Fully identified (SANCO/1/257/2013)	5 confirmed on ToF, LC-MS/MS	30	<5	QQQ	GC	MS/MS	ToF	Q-TOF	No	No
007 Fully identified (SANCO/1/257/2013)				Jet Stream	LC	MS/MS	+	+	No	NA
008 Fully identified (SANCO/1/257/2013)				Agilent 6530 accurate mass Q-ToF LCMS	Agilent	MS/MS	10	10	AcN	Yes
009 Fully identified (SANCO/1/257/2013)	+/- 0.2 min	0.2 min	<5 acc. m/z + isotope + fragment	Q-ToF	LC	MS/MS	ESI	+	No	0.05 - 0.1
011 Tentatively detected	0.8 2 Transitions			Q-TOF	LC	MS/MS	ESI	+	No	0.005-0.1
013 Tentatively detected	0.13			Q-Orbitrap	LC	MS/MS	ESI	+	Yes	0.01
014 Tentatively detected	5 5			TOF	LC	MS	TOF	ESI	+	0.01 - 0.10
015 Fully identified (SANCO/1/257/2013)	10 Acccurate mass	6		QQQ	GC	MS/MS	El	YES	Yes	0.01
016 Fully identified (SANCO/1/257/2013)	0			Q-TRAP	LC	MS/MS	ESI	+	Yes	0.01
024 Tentatively detected	5 accurate mass: 232.0827, transition 1; 232.0827>72.0445, transition 2; 232.0827>46.0649	3		Q-ToF	LC	MS	Q-ToF	ESI	+	0.01-0.1
026 Tentatively detected	5 Acccurate mass	0.01		Q-Orbitrap	LC	MS/MS	ESI	+	Yes	0.010
029 Fully identified (SANCO/1/257/2013)	2 Transitions	2		QQQ	LC	MS/MS	ESI	+	Yes	0.01
032 Fully identified (SANCO/1/257/2013)	2 Transitions			QQQ	LC	MS/MS	El	+	Yes	0.05
Standard Solution										
Compounds in method or library (pesticides Only)										
Software										
Injection Volume (μl)										
Column Type										
Agrifaction time (min)										
Clean up Step										
Agitator										
Extraction Solvent										
Instrument Model										
Full Scan										
Sample Weight (g)										
Ph adjusted										
Agrifaction Mode										
DB5										
Mass Hunter										
176										
No screening methods										

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/differentiation done?	MS Tolerance (ppm)	Other identification details	Chromatographic Technique	Detector	Analyzer	Polarity	Routine Method?	Routine Scope?	Screening Detection Limit (SDL)? (mg/kg)	Instrument Model	Extraction Solvent	pH adjusted	Agitation Mode	Agitation time (min)	Injection Volume (μL)	Compounds in method (pesticides Only)	Standard Solution Frequency	Fluometuron			
																			Column Type	Software		
035 Fully identified (SANCO/1/257/2013)	2.5%	2 Transitions	LC MS/MS	QQQQ	ESI	+	No	0.01	No	HPLC Agilent 1100, MS API 3000	AcN	Yes		15	DSPE	C18 50x2mm	10	Both	method 550	Daily		
036 Fully identified (SANCO/1/257/2013)			LC MS/MS	QQQQ	ESI	+	Yes	0.01	No	Agilent 6490	5	AcN	Yes		3	None	RPLC	5	Mass Hunter	550	Every batch (100+ pesticides)	
037 Fully identified (SANCO/1/257/2013)	<30	5	isotope	LC MS	Orbitrap	ESI	+	No	0.01	Yes	Excative	1% acetic acid	No	Mechanical	30	Liquid-Liquid Partitioning	C18 Atlantis	5	ToxiD	600	Daily	
038 Fully identified (SANCO/1/257/2013)	10	2 Transitions	LC MS/MS	QQQQ	ESI	-	Yes	0.01	No	4000	10	AcN	No	Mechanical	5	DSPE	C18	10	Analyst			
040 Tentatively detected	0 +17.5 ppm	accurate mass + MRM	LC MS	Q-TOF	ESI	+	No	Yes	0.01	Yes	Waters Xevo Q-TOF (1st gen)	EtOAc	Yes	Mechanical	4	Acquity BEH C18	3	TargetLynx + MassFragment tool	500	Always		
041 Tentatively detected	-3	5		LC MS	Q-TOF	ESI	+	No	Yes	Yes	Agilent 6550 iFunnel QTOF LC/MS	AcN	No		2	DSPE	C18	5	Mass Hunter	1700	Always	
043 Tentatively detected	2.5%	0.3	1 Transition	LC MS/MS	QQQQ	ESI	+	Yes	0.005	No	TSQ Quantum discovery max thermo	10	AcN	No	Mechanical	1	Liquid-Liquid Partitioning	Alltima C18 150*2.1 mm (Alltech 88370)	25	LC quan	150	Day of run
044 Fully identified (SANCO/1/257/2013)	3.8			GC MS	Q	EI	+	Yes	0.02	Yes	AT5975 GC-MS	10	AcN/EtOAc	No		2		HP-MS	10	DRS/Chemstation	~750	Always
046 Tentatively detected	0	1.5	Accurate mass	LC MS	TOF	ESI	+	Yes	0.02	Yes	Bruker maxis 10	EtOAc			15	None	C18	2	TargetAnalysis	750	Always	
047 Tentatively detected			2 Transitions	LC MS/MS	QQQQ	ESI	+	Yes	0.01	No	Finnigan TSQ 10	AcN	No	Mechanical	20	DSPE	Agilent Eclipse C18 XDB	20	Xcellibur	282	Every batch	
048 Fully identified (SANCO/1/257/2013)	1	2	Transitions	LC MS/MS	QQQQ	ESI	+	Yes	0.010	Yes	Waters Acquity UPLC TQD	15	AcN	Yes		1	DSPE	UPLC BEH C18	10	Auto	340	Every batch
050 Tentatively detected			2 Transitions	LC MS	QQQQ	ESI	+	No	No	No	TSQ QUANTUM 10	AcN	No	None		None	XBRIDGE	20	Xcellibur	25		
051 Fully identified (SANCO/1/257/2013)	0.3	2 Transitions	LC MS/MS	QQQQ	ESI	+	Yes	0.01	No	Variian Pro Star LC + 320-MS	10	AcN	No	Mechanical	3	None	Rectek Ultra C18	10	Workstation	363	Every batch	
053 Fully identified (SANCO/1/257/2013)	0	1	Accurate mass	LC MS/MS	TOF	ESI	+	Yes	0.01	Yes	XEVO G2-S QToF	10	AcN	Yes	Mechanical	15	DSPE	C18	1	Unify	900	Often
054 Fully identified (SANCO/1/257/2013)	2.5	2 Transitions	LC MS/MS	QQQQ	ESI	+	Yes	0.01	Yes	Agilent G6410A	10	AcN	Yes		25	SPE	ZORBAX ECLIPSE	20	Auto	143	Every batch	

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/determination done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Chromatographic Technique	Detector	Polarity	Routine Method?	Routine Scope?	Screening Detection limit (SDL) (mg/kg)	Instrument Model	Sample Weight (g)	Extraction Solvent	pH adjusted	Agitation Mode	Clean up Step	Injection Volume (μL)	Compounds in method (library or library pesticides Only)	Standard Solution Frequency	
055	Tentatively detected	none	2 Transitions	LC MS/MS	QQQ	ESI	+	No	No	0.01	API 4000	10	ACN	Yes	Manual	5	DSPE	Waters C18 2,1x50mmx 1,7μm	600 pesi total (10 LC/MS/MS methods)	Weekly (200 pesticides)
057	Tentatively detected	2	Accurate mass	LC MS	Q-Orbitrap	ESI	+	No	No		Orbitrap Elite	10	ACN	No	Manual	2	DSPE	C18	5	TraceFinder
058	Tentatively detected	15	target + qualifier	GC	MS Q	EI	+	Yes	Yes	0.01 - 0.05	Agilent GC - MSD	10	ACN	Yes	Mechanical	10	PSA	DB5	10	ChemStation
059	Fully identified (SANCO/1257/2013)	2	LC	MS/MS	QQQQ	ESI	-	Yes	Yes	0.01	Varian-320	10	ACN	No		3	DSPE	C18 50x2mm	10	MS Workstation
060	Fully identified (SANCO/1257/2013)	2 Transitions	LC	MS/MS	Q-TRAP	ESI	+	Yes	Yes		Ap 3200 Qtrap	10	ACN		Manual	2	DSPE	Allantis T3	5	
066	Fully identified (SANCO/1257/2013)	2 Transitions	LC	MS/MS	QQQQ	ESI	+	Yes	Yes	0.01	Waters TQD	10	ACN	Yes	Mechanical	16	None	Acquity UPLC BECH C18 1.7 mm 2.1 x 100 mm	14	200

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/differentiation done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Sample Weight (g)	Instrument Model	Full Scan	Limit (SD)? (mg/kg)	Screening Detection Limit (SD)? (mg/kg)	Fuberidazole		Standard Solution Frequency							
														Agitation Mode	Clean up Step	Column Type	Injection Volume (μL)	Compounds in method (pesticides Only)	Software				
001 Fully identified (SANCO/1257/2013)	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	0.01	No	Agilent 6460	10	AcN	No	1	None	Zorbax C18	5	Mass Hunter	200	Daily			
003 Fully identified (SANCO/1257/2013)		LC	MS/MS	QQQ	El	+	No	No	No	Agilent	15	Acetone/DCM/PE	No	1	None	DB5	1	Mass Hunter	176	No screening methods			
006 Tentatively detected		GC	MS/MS	QQQ	El	+	No	No	No	Agilent	15	Acetone/DCM/PE	Mechanical	1	None	DB5	1	Mass Hunter	200	Daily			
007 Fully identified (SANCO/1257/2013)	30	5	confirmed on ToF (negative & positive mode)	LC	ToF	Q-ToF	Jet Stream	+	No	Agilent	6530	AcN	No	0.5	None	C18	3	Mass Hunter	550	NA			
008 Fully identified (SANCO/1257/2013)		LC	MS/MS			Yes	Yes			Agilent	6530	AcN	No	0.5	None	C18	3	Mass Hunter	550	NA			
009 Fully identified (SANCO/1257/2013)	+/-0.2 min	<5	acc. m/z + isotope + fragment	LC	MS/MS	Q-ToF	ESI	+	No	0.05 - 0.1	Yes	API 5600 G-Tof	10	AcN	No	Mechanical	5	None	C18 Aqua Phenomenex	20	Both		
011 Tentatively detected	0.1	2 Transitions	LC	MS/MS	Q-ToF	ESI	+	No	0.005-0.1	Yes	TripletOF	10	AcN	No	2	None	C18	2	Peak/Few	620	No standard		
013 Tentatively detected	10	2	GC	MS/MS	QQQ	El	+	Yes	0.01	Agilent7890/ Agilent7000	10	AcN	Yes	5	DSPE	DB5-MS	5	Mass Hunter	~500				
014 Tentatively detected	5	5	LC	MS/MS	Q-Orbitrap	ESI	+	Yes	0.01	Thermo Q-Exactive	10	AcN	Yes	5	DSPE	C18	5	Xcalibur	~500				
015 Fully identified (SANCO/1257/2013)	4	10	Acccurate mass	GC	MS/MS	QQQ	El	Yes	0.01	Yes	5977	Cyclohexane/EtOAc/Acetone	No	3	DSPE	HP5-MS	1	Mass Hunter	850	Daily			
016 Tentatively detected	0.72		GC	MS/MS	QQQ	El	+	Yes	0.01	No	Agilent 7000	10	AcN	Yes	1	DSPE	HP5MSU	10	Mass Hunter	~300	Always		
020 Fully identified (SANCO/1257/2013)		2 Transitions	GC	MS/MS	QQQ	El	+	Yes	0.005	No	TSQ Quantum XLS Ultra	10	AcN	No	Mechanical	30	DSPE	RTX5-MS	1	TraceFinder	300	Always	
021 Fully identified (SANCO/1257/2013)		2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	0.005	No	Waters Xevo TQ	15	AcN	No	Mechanical	5	None	HSS T3	10	Masslynx	400	Every batch	
024 Tentatively detected	3	5	accurate mass: 184.0639, transition 1; 184.0639>157.0760 184.0639>130.0649	LC	MS	Q-ToF	ESI	+	No	0.01-0.1	Yes	Agilent 6550 QTOF	10	AcN	No	Manual	1	QueCHERS without PSA	Altantis 13, 2.1x100 mm, 3um	2	Mass Hunter	1640	
026 Tentatively detected		5	Acccurate mass	LC	MS/MS	Q-Orbitrap	ESI	+	No	0.005	Yes	Q Exactive	10	AcN	No	Mechanical	10	DSPE	BEH C18	5		700	
027 Fully identified (SANCO/1257/2013)	0	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	0.010	No	Xevo	10	Acetone/DCM/PE	No	1	None	C18	2	Waters	286	Every batch		
028 Fully identified (SANCO/1257/2013)	0.1	0	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	0.005	No	Agilent 6410 Triple Quad	10	AcN	No	Manual	2	None	Zorbax Eclipse XDB-C18	4	Agilent ChemStation	144	No

APPENDIX 3. Methods used by participants for detecting pesticides.

Fuberidazole											
Laboratory Code *NRL	How was the detection/differentiation done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Detector	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Detection limit (SDL) (mg/kg)	Instrument Model
029 [SANCO/1/257/2013]	Fully identified	spectrum	GC	TOF	EI	Yes	No	0.01	Yes	IECC Pegasus IV	AcN
032 [SANCO/1/257/2013]	Fully identified	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.05	No
035 [SANCO/1/257/2013]	Fully identified	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.01	No
036 [SANCO/1/257/2013]	Fully identified		GC	MS/MS	QQQ	EI	+	Yes	Yes	0.01	No
037 [SANCO/1/257/2013]	Tentatively detected	<30	5	isotope spectrum	LC	MS	Orbitrap ESI	+	No	Not Established	Yes
038 [SANCO/1/257/2013]	Fully identified	10	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.01
039 [SANCO/1/257/2013]	Fully identified	0.18	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.005
040 [SANCO/1/257/2013]	Tentatively detected	N/A - no standard	-1.6	Accurate mass	LC	MS	Q-TOF ESI	+	No	No	0.01
041 [SANCO/1/257/2013]	Tentatively detected	0	4		LC	MS	Q-TOF	ESI	+	No	Yes
042 [SANCO/1/257/2013]	Fully identified		2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.01
044 [SANCO/1/257/2013]	Fully identified	7.6		GC	MS	Q	EI	+	Yes	Yes	0.02
046 [SANCO/1/257/2013]	Tentatively detected	0.15	1.9	Accurate mass	LC	MS	TOF	ESI	+	Yes	0.02
048 [SANCO/1/257/2013]	Fully identified	1	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.010
049 [SANCO/1/257/2013]	Fully identified			LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.01
Standard Solution (pessticides Only) or library											
Software											
Injection Volume (μl)											
Compounds in method (pesticides Only)											
Frequency											
Standard Solution											

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/identification done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Detection Limit (SDL) (mg/kg)	Instrument Model	Sample Weight (g)	Agitation Time (min)	Clean up Step	Column Type	Injection Volume (μL)	Compounds in method (pesticides Only)	Standard Solution Frequency				
050	Tentatively detected		2 Transitions	LC	MS	QQQ	ESI	+	No	No	No	No	TSQ QUANTUM	10	ACN	No	None	None	XBRIDGE	20	Xcalibur	25		
053	Fully identified (SANCO/1257/2013)	0	1 Accurate mass	LC	MS/MS	TOF	ESI	+	Yes	Yes	0.01	Yes	XEVO G2-S QTof	10	ACN	Yes	Mechanical	15	DSPE	C18	1	Unity	900	
055	Tentatively detected	none	2 Transitions	LC	MS/MS	QQQ	ESI	+	No	No	0.01	No	API 4000	10	ACN	Yes	Manual	5	DSPE	Waters-C18 2,1x50mmx 1,7,μm	5	Analyst	600 pest.total (10 LC/MS/MS methods)	
057	Tentatively detected		3 Transitions	LC	MS/MS	QQQ	ESI	+	No	No	No	No	Quattro Premier	10	ACN	No	Manual	2	DSPE	C18	5	Masslynx	Often	
058	Tentatively detected	15	1 target + qualifier	GC	MS	Q	EI	+	Yes	Yes	0.01 - 0.05	Yes	Agilent GC - MSD	10	ACN	Yes	Mechanical	10	PSA	DB5	10	ChemStation	>600	Every batch
064	Tentatively detected			LC	MS	QQQ	ESI	+	No	No	Yes	Agilent 6410	10	ACN	Yes	Mechanical	2	GBC	ZORBAX XDB-C18	20	Mass Hunter	32		
066	Fully identified (SANCO/1257/2013)		2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.01	No	Waters TQD	10	ACN	Yes	Mechanical	14	Acuity UPLC BECH C18 1,7 mm 2,1 x 100 mm	14		200		
068	Tentatively detected		full scan spectra	GC	MS	Q		+	No	No	Yes	Thermo Trace GC -DSQ	10	ACN	Yes		2	SPE	elite 5 ms	1	Xcalibur	100	Every year	

APPENDIX 3. Methods used by participants for detecting pesticides.

HCB											
Laboratory Code *NRL	How was the detection/identification was done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Chromatographic Technique	Detector	Analyzer	Polarity	Routine Method?	Screening Detection Limit (SDL) (mg/kg)	Injection Volume (μl)
001 Fully identified (SANCO/1257/2013)	3 Transitions	GC MS/MS	QQQ	EI + Yes	0.01	No	Agilent 7000	10 ACN	No	10 Agilent 5973n	1 HP-MS
003 Fully identified (SANCO/1257/2013)		GC NS/MS		+ Yes	No	Yes	Agilent 6890N	10 ACN	Yes	30 EtOAc	1 Mass Hunter
007 Fully identified (SANCO/1257/2013)	confirmed on GC-MS full spectra	GC MS	Q	CI No	No	NA	Agilent 5973n	13 ACN	Manual	30	350 Daily
008 Fully identified (SANCO/1257/2013)		GC MS/MS			Yes	Yes	Agilent 6890N	13 ACN	No	10 Acetone	1 Agilent ChemStation
009 Fully identified (SANCO/1257/2013)	+/- 0.2 min	GC MS/MS	QQQ	EI + Yes	0.01	No	Bruker 450 Scion TQ	10 ACN	No	10 MeOH	5 567 NA
010 Fully identified (SANCO/1257/2013)		GC MS/MS	QQQ	EI + Yes	0.010		Bruker SCION 10	10 ACN	No	10 Acetone	2 Agilent ZB
011 Fully identified (SANCO/1257/2013)	2 Transitions	GC NS/MS	QQQ	EI Yes	0.005-0.25	No	Agilent 7000B	10 ACN	No	10 Acetone	2 DSPE
012 Fully identified (SANCO/1257/2013)	2 Transitions	GC MS/MS	QQQ	EI + Yes	5	No	TSQ Quantum XLS Thermo	10 ACN	Manual	10 Acetone	2 HP-MS
013 Tentatively detected	2	GC MS/MS	QQQ	EI + Yes	0.01		Agilent 7890/ Agilent 7000	10 ACN	Yes	10 Acetone	5 Mass Hunter
015 Fully identified (SANCO/1257/2013)	4	GC MS/MS	QQQ	EI Yes	0.01	Yes	59770 Cyclonexane/EtOAc/Acetone	No		10 Acetone	3 DSPE
016 Fully identified (SANCO/1257/2013)	0	GC MS/MS	QQQ	EI + Yes	0.01	No	Agilent 7000	10 ACN	Yes	10 Acetone	10 Mass Hunter
017 Fully identified (SANCO/1257/2013)	0.02	GC MS	Q	EI + Yes	0.01	Yes	Thermo DSQ II	10 ACN	Yes	10 Acetone	2 QueChERS
018 Fully identified (SANCO/1257/2013)		LC NS/MS	QQQ	+ Yes	0.010	No	Qtrap3200	10 ACN	No	10 Acetone	1 DSPE
019 Fully identified (SANCO/1257/2013)		GC MS/MS	QQQ	EI + Yes	0.01	No	Quantum 10	10 ACN	No	10 Acetone	2 ZB-30
020 Fully identified (SANCO/1257/2013)	2 Transitions	GC MS/MS	QQQ	EI + Yes	0.005	No	TSQ Quantum XLS Ultra	10 ACN	No	10 Acetone	30 DSPE
022 Tentatively detected		GC NS/MS	Ion trap	EI Yes	0.005	Yes	Saturno 2000 Varian	10 ACN	No	10 Acetone/DCM/PE	1 RTX-MS
023 Fully identified (SANCO/1257/2013)		GC MS	T	No	No	Yes	Varian 240MS	15 Acetone/DCM/PE	No	1 GPC	3 VF-5MS
										10 factor four	10 Always
										1 GPC	3 Auto 127

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/differentiation done?	RT Deviation (s)	MS Tolerance (ppm)	Other differentiation details	Chromatographic Technique	Detector	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Detection Limit (SDL)? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Injection Volume(µL)	Software	Standard Solution (pesticides Only)	Frequency Before and after sample							
024	Fully identified (SANCO/1/257/2013)	5	Match spectra with standard: 908	GC	MS/MS	QQQ	EI	+	Yes	Yes	0.01	No	Varian 320MS	10	AcN	No	Mechanical	10	DB5-MS (30m x 0.25mm x 0.25µm) in the 1st Dimension; BPX-50 (2m x 0.1mm x 0.1µm) in the 2nd Dimension	10	Chromatof	200	Always		
025	Fully identified (SANCO/1/257/2013)	2	0	2 Transitions	GC	MS/MS	QQQ	Cl	-	Yes	Yes	0.010	No	Varian 320MS	10	Acetone	No	Mechanical	2	Liquid-Liquid Partitioning	DB5	8	Star	146	Always
026	Tentatively detected	0.01	5		GC	MS/MS	QQQ	Cl	-	Yes	Yes	0.001	No	Varian 320MS	10	AcN	No	Mechanical	10	DSPE	DB5	1		250	
027	Fully identified (SANCO/1/257/2013)	0		2 Transitions	GC	MS/MS	QQQ	EI	+	Yes	Yes	0.010	No	Thermo 10	Acetone/DCM/PE	No		1	None	HP5-MS	1	Thermo	201	Every batch	
028	Fully identified (SANCO/1/257/2013)	0.1	0	no	GC	MS	Q	EI	+	Yes	Yes	0.01	No	Agilent 6890/5973N	10	AcN	No	Manual	2	None	HP5-MS	2	Agilent ChemStation	184	No
029	Fully identified (SANCO/1/257/2013)			spectrum	GC			TOF	EI	Yes	Yes	0.01	Yes	LECO Pegasus IV	10	AcN	Yes	Mechanical	15	DSPE	HP5-MS	3	Both	750	Always
030	Fully identified (SANCO/1/257/2013)	0	5		GC	MS	IT	EI		Yes	Yes	0.01	Yes	Varian 4000	10	AcN	No	Mechanical	1	SPE	Rxi 5ms 5% diphenyl / 95% dimethyl polysiloxane	1	Varian MS Workstation	196	Every batch
031	Fully identified (SANCO/1/257/2013)	max. 0.5%	1	3 ion ratios	GC	MS	Q	EI	+	Yes	Yes	0.01	Yes	Trace DSQ	15	AcN	Yes	Manual	1	SPE	DB5MS	0.8	Xcalibur	350	Every batch
032	Fully identified (SANCO/1/257/2013)	< 0.2 min	2	2 Transitions	GC	MS/MS	QQQ	EI	+	Yes	Yes	0.01	No	Waters Quattro Micro GC	10	EtOAc	Yes	Mechanical	18	Filtration	Rxi 5sli	10	Mastlynx	404	Every batch
034	Fully identified (SANCO/1/257/2013)			1 Target, 3 Qualifier	GC	MS/MS	QQQ	Cl	-	Yes	Yes	0.01	No	Agilent 7890A	10	AcN	Yes	Manual	20	DSPE	Pesticides2	4	Mass Hunter	74	Every batch
035	Fully identified (SANCO/1/257/2013)	± 0.5%												GC Shimadzu GC-2010, MS Shimadzu GCMS-QP2011	10			15	DSPE	HP5-MS	3	Both	method 550	Always	
036	Fully identified (SANCO/1/257/2013)	<20		spectrum	GC	MS	TOF	EI	+	Yes	Yes	0.05	Yes	Agilent 7000B	5	AcN	Yes		3	None	HP-5MS UI	2	Mass Hunter	550	Daily
037	Fully identified (SANCO/1/257/2013)													1% acetic acid	No	Mechanical	30	Liquid-Liquid Partitioning + DSPE	RTX-Pesticides	10	MetAlign + Chromatof	560	Every batch (235 pesticides)		

APPENDIX 3. Methods used by participants for detecting pesticides.

HCB											
Laboratory Code *NRL	How was the detection/differentiation was done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Detector	IonizationMode	Polarity	Routine Method?	Screening Detection limit (SDL) (mg/kg)	Instrument Model	Extraction Solvent
038 (SANCO/1257/2013)	Fully identified	10	Accurate mass	GC	MS	Q	EI	+	Yes	0.01	No
039 (SANCO/1257/2013)	Fully identified	0.12	2 Transitions	GC	MS/MS	QQQ	EI	+	Yes	0.002	No
042 (SANCO/1257/2013)	Fully identified	2 Transitions	GC	MS/MS	QQQ	EI	+	Yes	0.01	No	Bruker Scion GC-MS TQ
044 (SANCO/1257/2013)	Fully identified	0.8	2 Transitions	GC	MS/MS	QQQ	EI	+	Yes	0.02	Yes
045 (SANCO/1257/2013)	Fully identified	0.2	2 Transitions	GC	MS/MS	QQQ	EI	+	Yes	0.010	No
046 (SANCO/1257/2013)	Fully identified	2	3 Transitions	GC	MS	IT	EI	+	Yes	0.01	No
047 (SANCO/1257/2013)	Fully identified	2.04	3 Transitions	GC	MS/MS	QQQ	EI	+	Yes	0.01	No
048 (SANCO/1257/2013)	Fully identified	1		GC	MS	Q	EI	+	Yes	0.010	Yes
049 (SANCO/1257/2013)	Fully identified			GC	MS/MS	QQQ	EI	Yes	Yes	0.01	No
050 (SANCO/1257/2013)	Fully identified	2 Transitions	GC	MS	IT	EI	+	Yes	0.010	Yes	POLARIS Q
051 (SANCO/1257/2013)	Fully identified	0.1	3 Transitions	GC	MS/MS	QQQ	EI	Yes	Yes	0.003	No
052 (SANCO/1257/2013)	Fully identified	12	2 Transitions ratio 30%	GC	MS/MS	IT	EI	Yes	Yes	0.010	Yes
053 (SANCO/1257/2013)	Fully identified	0	1	3	GC	MS	Q	CI	-	Yes	0.01
054 (SANCO/1257/2013)	Fully identified	0.5	3 Transitions	GC	MS	Q	EI	+	Yes	0.01	Yes
055 (SANCO/1257/2013)	Fully identified	none			GC	MS	Q	EI	+	Yes	Agilent 6890M
056 Tentatively detected	1	1		GC	MS	Q	EI	Yes	No	0.01	Yes
057 (SANCO/1257/2013)	Fully identified		2 Transitions	GC	MS/MS	QQQ	EI	+	Yes	0.01	Quattro micro
058 Tentatively detected	1.5	1 target + qualifier	GC	MS	Q	EI	+	Yes	0.01 - 0.05	Yes	Agilent GC - MSD
											DB5
											PSA
											DB5
											ChemStation
											5
											DSPE
											20
											Mechanical
											5
											None
											5
											Workstation
											5
											ChemStation
											10
											DRS
											2
											HP5-MS
											5
											Workstation
											NS
											171
											Every batch
											336
											Every batch
											Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/denification done?	RT Deviation (s)	MS Tolerance (ppm)	Other denification Details	Chromatographic Technique	Detector	Analyzer	Polarity	Routine Method?	Routine Scope?	Screening Detection Limit (SDL) (mg/kg)	Instrument Model	Sample Weight (g)	Extraction Solvent	pH adjusted	Agitation Mode	Agitation time (min)	Column Type	Injection Volume (μL)	Software	Compounds in method (pesticides Only)	Standard Solution Frequency		
060 (SANCO/1/257/2013)	Fully identified	60	3 diagnostic ions	GC	MS	Q	EI	+	No	No	Yes	Agilent	10	AcN		Manual	2	DSPE	HP5-MS	20	AMDIS	~550		
065 (SANCO/1/257/2013)	Fully identified			GC	MS	Q	EI	+	Yes	No	0.02	Shimadzu QP2010+	10	AcN	No		30	DSPE	VF-5-MS-30M*0.25*0.25μm	10	GC-MSSolution	325	3 Months	
067 (SANCO/1/257/2013)	Fully identified		2 Transitions	GC	MS/MS	QQQ	EI		Yes	Yes	0.01	No	Agilent 7000	10	AcN	No	Mechanical	1	DSPE	DB-5MS UI	2	Mass Hunter	236	Daily
068	Tentatively detected		full scan spectra	GC	MS	Q	EI	+	No	No	Yes	thermo trace gc-dsq	10	AcN	Yes		2	DSPE	elite 5 rms	1	Xcalibur	100	Every year	

APPENDIX 3. Methods used by participants for detecting pesticides.

Metosulam											
Laboratory Code *NRL	How was the detection/differentiation done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Detector	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Detection limit (SDL) (mg/kg)	Instrument Model
003 Fully identified (SANCO/1257/2013)	Fully identified	2.5	2.31	Accurate mass	LC MS/MS	Q-TOF	ESI +	Yes Yes	No	Yes Agilent 6520	10
004 Tentatively detected					LC MS/MS	QQQ	ESI +	No No	No	Agilent 6490	10
006 Tentatively detected				confirmed on LC-MS/MS, 2 transitions	LC MS/MS	QQQ	ESI +	No No	No	Agilent 6490	10
007 Fully identified (SANCO/1257/2013)	30				LC MS/MS	TOF	ESI +	No No	Yes	Agilent 6490	10
008 Fully identified (SANCO/1257/2013)					LC MS/MS	TOF	ESI +	Yes Yes			13
011 Fully identified (SANCO/1257/2013)	3	2 transitions		confirmed on LC-MS/MS, 2 transitions	LC MS/MS	QQQ	ESI +	Yes Yes	0.005-0.1	Xevo TQs	10
011 Tentatively detected	3	0.7	2 Transitions		LC MS/MS	Q-TOF	ESI +	No No	0.005-0.1	TripleTOF 10	10
014 Tentatively detected	5				LC MS	TOF	ESI +	Yes No	0.01 -0.10	PREMIER XE	15
015 Fully identified (SANCO/1257/2013)	6	10	Accurate mass		LC MS/MS	Q-TRAP	ESI +	Yes Yes	0.01	ABSciex 4000 QTrap	10
016 Fully identified (SANCO/1257/2013)	0				LC MS/MS	TOF	ESI +	Yes Yes	0.01	ABSciex 4000 QTrap	10
021 Fully identified (SANCO/1257/2013)		2 Transitions			LC MS/MS	QQQ	ESI +	Yes Yes	0.005	Waters Xevo TQ	15
024 Fully identified (SANCO/1257/2013)	3	418.0>174.9, 418.0>139.9			LC MS/MS	QQQ	ESI +	Yes Yes	LOQ: 0.002 mg/kg	ABSciex 4000 QTrap	10
026 Tentatively detected	5	Accurate mass			LC MS/MS	Q-Orbitrap	ESI +	No No	0.010	Q Exactive	10
027 Fully identified (SANCO/1257/2013)	0	2 Transitions			LC MS/MS	QQQ	ESI +	Yes Yes	0.010	Xevo 10	Acetone/DCM/PE
029 Fully identified (SANCO/1257/2013)		2 Transitions			LC	QQQ	ESI +	Yes Yes	0.01	API 5500	10
032 Fully identified (SANCO/1257/2013)					LC	MS/MS	QQQ	El +	Yes Yes	EtOAc	10
Standard Solution											
Software	Compounds in method or library (pesticides Only)	Injection Volume (μl)	Column Type	Clean up Step	Agitation time (min)	Agitation Mode	pH adjusted	Instrument Scan	Sample Weight (g)	Screening Detection limit (SDL) (mg/kg)	Frequency
											No screening methods
											3 Months
											>800
											No screening methods
											Every batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/differentiation done?	RT Deviation(s)	MS Tolerance (ppm)	Other identification details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Sample Weight (g)	Instrument Scan	Screening Detection Limit (SDL)? (mg/kg)	Routine Method?	Routine Scope?	Polarity	LC/MS/MS	MS/MS	QQQ	ESI	+ Yes	Yes 0.01	No	HPLC Agilent 1100 MS API 3000	AcN	Yes	15	DSPE	C18 50x2mm	10	Both	method 550	Always	Metosulam		
035	Fully identified (SANCO/1257/2013)	2.5%	2 Transitions	LC	MS/MS	QQQ	ESI	+ Yes	Yes 0.01	No	HPLC Agilent 1100 MS API 3000	AcN	Yes	15	DSPE	C18 50x2mm	10	Both	method 550	Always														
036	Fully identified (SANCO/1257/2013)			LC	MS/MS	QQQ	ESI	+ Yes	Yes 0.01	No	Agilent 6490 5	AcN	Yes	3	None	RPI 8	5	Mass Hunter	550	Daily														
037	Fully identified (SANCO/1257/2013)	<30	5	isotope	LC	MS	Orbitrap	ESI	+ No	No 0.01	Yes	AcN/1% acetic acid	No	Mechanical	30	Liquid-Liquid Partitioning	C18 Atlantis	5	ToxiD	600	Every batch (100+ pesticides)													
038	Fully identified (SANCO/1257/2013)	10	2 Transitions	LC	MS/MS	QQQ	ESI	- Yes	Yes 0.01	No	4000 10	AcN	No	Mechanical	5	DSPE	C18	10	Analyst		Daily													
040	Tentatively detected no standard	N/A -160	Accurate mass	LC	MS	Q-TOF	ESI	+ No	No 0.01	Yes	Waters Xevo Q-TOF (1st gen) LC/MS	EtOAc	Yes	4	None	Acquity BEH C18	3	TargetLynx + MassFragment tool	500	Always														
041	Tentatively detected	2	2	LC	MS	Q-TOF	ESI	+ No	Yes	Yes	Agilent 6550 iFunnel QTOF 10 LC/MS	AcN	No	2	DSPE	C18	5	Mass Hunter	1700	Always														
042	Fully identified (SANCO/1257/2013)		2 Transitions	LC	MS/MS	QQQ	ESI	+ Yes	Yes 0.01	No	Agilent 6490 10	EtOAc	No	Mechanical	20	None	HSST 3	2	Mass Hunter	265	Every batch													
043	Tentatively detected	2.5%	0.3	1 Transition	LC	MS/MS	QQQ	ESI	Yes Yes	Yes 0.005	No	TSQ Quantum discovery max thermo	10	AcN	No	Mechanical	1	Liquid-Liquid Partitioning	Alltech 150*2.1 mm (Alltech 88370)	25	LC quan	150	Day of run											
044	Fully identified (SANCO/1257/2013)	0.2	2 Transitions	LC	MS/MS	QQQ	ESI	+ Yes	Yes 0.01	No	AB4000 10	AcN	No	2	None	C18	10	Analyst	110															
046	Fully identified (SANCO/1257/2013)	2	LC	MS/MS	QQQ	ESI	- Yes	Yes 0.01	No	Agilent 6460 10	EtOAc			15	None	C18	2	Mass Hunter	550	Always														
048	Fully identified (SANCO/1257/2013)	1	2 Transitions	LC	MS/MS	QQQ	ESI	+ Yes	Yes 0.01	No	Waters UPLC 15 TQD LC/MS/MS	AcN	Yes	1	DSPE	UPLC BEH C18	10	Auto	40	Every batch														
049	Fully identified (SANCO/1257/2013)			LC	MS/MS	QQQ	ESI	+ Yes	Yes 0.01	No	Agilent 6490 15 Acetone/DCM/ PE LC/MS/MS	AcN	No	1	None	C18	10	Mass Hunter	250	Every batch														
050	Tentatively detected		2 Transitions	LC	MS	QQQ	ESI	+ No	No 0.01	No	TSQ 10 QUANTUM	AcN	No	None	None	XBRIDGE	20	Xcalibur	25															
052	Fully identified (SANCO/1257/2013)	12	2 Transitions ratio 30%	LC	MS/MS	QQQ	ESI	+ Yes	Yes 0.010	Yes	ABSCIEX 4000 QTRAP	AcN	No	Mechanical	20	None	C18	20	Analyst	1	Every batch													
053	Fully identified (SANCO/1257/2013)	0	1	Accurate mass	LC	MS/MS	TOF	ESI	+ Yes	Yes 0.01	Yes	XEVO G2-S QTOF	AcN	Yes	Mechanical	15	DSPE	C18	1	Unity	900	Often												
054	Fully identified (SANCO/1257/2013)	2.5	2 Transitions	LC	MS/MS	QQQ	ESI	+ Yes	Yes 0.01	Yes	Agilent G6410A	AcN	Yes	25	SPE	ZORBAX ECLIPSE	20	Auto	143	Every batch														

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/determination done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Chromatographic Technique	Detector	Polarity	Routine Method?	Routine Scope?	Screening Detection Limit (SDL) (mg/kg)	Instrument Model	Sample Weight (g)	Extraction Solvent	pH adjusted	Agitation Mode	Clean up Step	Column Type	Injection Volume (µL)	Compounds in method (library or library)	Standard Solution Frequency
055	Tentatively detected	none	2 Transitions	LC M/S/QQQ	M/S/QQQ	ESI	+	No	No	0.01	API 4000	10	ACN	Yes	Manual	5	DSPE	Waters-C18 2,1x50mmx 1,7µm	600 pes; total (10 LC/MS/MS methods)	600 pes; total (10 LC/MS/MS methods)
057	Tentatively detected		3 Transitions	LC M/S/QQQ	M/S/QQQ	ESI	+	No	No		Quattro Premier	10	ACN	No	Manual	2	DSPE	C18	5	Masslynx
060	Fully identified (SANCO/1257/2013)		2 Transitions	LC M/S/Q-TRAP	M/S/Q-TRAP	ESI	+	Yes	Yes		AP 3200 Qtrap	10	ACN		Manual	2	DSPE	Atlantis T3	5	

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/differentiation done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Sample Weight (g)	Instrument Model	Full Scan	Extraction Solvent	Agitation Mode	Column Type	Injection Volume (μL)	Software	Standard Solution Frequency				
001 Fully identified (SANCO/1257/2013)		3 Transitions	GC	MS/MS	QQQ	EI	+	Yes	0.01	No	Agilent 7000	10	AcN	No	1	None	HP5-MS	Daily			
002 Fully identified (SANCO/1257/2013)			LC	MS/MS	QQQ	ESI	+	Yes			Agilent 6410	10	AcN/ACN		1	SPE	C18 4 micras	350			
003 Fully identified (SANCO/1257/2013)			LC	MS/MS			+	Yes	No			10	AcN	Yes	Manual	1	SPE		221		
004 Fully identified (SANCO/1257/2013)		0	2 Transitions	GC	MS/MS	QQQ	EI	+	Yes		No	Agilent 10	AcN	Yes	Mechanical	15	DSPE	HP5-MS	>300		
006 Fully identified (SANCO/1257/2013)			GC	MS/MS	QQQ	EI	+	Yes		No	Agilent 15	Acetone/DCM/PE	No	Mechanical	1	None	DB5	1	Mass Hunter	176	
007 Fully identified (SANCO/1257/2013)		30	confirmed on LC-MS/MS, GC-MS	GC	MS	Q	CI	No	No	Yes	Agilent 5973MS 6890N	EtOAc			0.5	GPC	DB5-MS	2	Agilent ChemStation	567	
008 Fully identified (SANCO/1257/2013)			LC	MS/MS				Yes	Yes			13	AcN	No	Mechanical		None		NA		
009 Fully identified (SANCO/1257/2013)	+/- 0.2 min <5	acc. m/z + isotope + fragment	LC	MS/MS	Q-TOF	ESI	+	No	0.05 - 0.1	Yes	Agilent 5600 Q-ToF	10	AcN	No	Mechanical	5	None	C18 Aqua Phenomenex	20	Bruker	600
010 Fully identified (SANCO/1257/2013)			GC	MS/MS	QQQ	EI	+	Yes	0.010		Bruker SCION 10	AcN	No	Manual	2	DSPE	Zebtron ZB	2	Bruker	210	
011 Fully identified (SANCO/1257/2013)	3	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	0.005-0.1	No	Xevo TQs 10	AcN	No	2	None	C18	2.5	Masslynx	300		
012 Tentatively detected	3	0.9	2 Transitions	LC	MS/MS	Q-TOF	ESI	+	No	0.005-0.1	Yes	TripleTOF 10	AcN	No	2	None	C18	2	PeakView	620	
013 Tentatively detected	10	2	GC	MS/MS	QQQ	EI	+	Yes	0.01		Agilent 7890/ Agilent 7000	10	AcN	Yes	Mechanical	5	DSPE	DB5-MS	5	Mass Hunter	~500
014 Tentatively detected	5	5	LC	MS	TOF	ESI	+	Yes	0.01 - 0.10	Yes	LCT PREMIER XE	15 Acetone/DCM/PE	No		1.5	None	C18	10	Masslynx	350	
015 Fully identified (SANCO/1257/2013)	3	10	Accurate mass	GC	MS/MS	QQQ	EI	+	Yes	0.01	Yes	5977	Cyclohexane/EtOAc/Acetone	No	Manual	3	DSPE	HP5-MS	1	Mass Hunter	850
016 Fully identified (SANCO/1257/2013)	0		GC	MS/MS	QQQ	EI	+	Yes	0.01	No	Agilent 7000	10	AcN	Yes	1	DSPE	HP5MSUI	10	Mass Hunter	~300	
018 Fully identified (SANCO/1257/2013)			LC	MS/MS	QQQ		+	Yes	0.010	No	Qtrap3200	10	AcN	No	Manual	1	SPE	C18	5	Analyst	205
020 Fully identified (SANCO/1257/2013)		2 Transitions	GC	MS/MS	QQQ	EI	+	Yes	0.005	No	TQ Quantum XLS Ultra	10	AcN	No	Mechanical	30	DSPE	RTX5-MS	1	TraceFinder	300
023 Fully identified (SANCO/1257/2013)			GC	MS	IT			No	No	Yes	Varian 240MS	15 Acetone/DCM/PE	No		1	GPC	VF-5MS	3	Auto	127	

APPENDIX 3. Methods used by participants for detecting pesticides.

Phorate												
Laboratory Code *NRL	How was the detection/differentiation was done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Detector	IonizationMode	Polarity	Routine Method?	Routine Scope?	Screening Detection limit (SDL) (mg/kg)	Injection Volume (μl)	
024	Fully identified (SANCO/1/257/2013)	5	Match spectra with standard: 955	GC	MS	TOF	EI	Yes	Yes	LOQ: 0.01 mg/kg	LECO Pegasus 4D	
025	Fully identified (SANCO/1/257/2013)	2	0	2 Transitions	GC	MS/MS	QQQ	EI	+	Yes	0.010	No
026	Tentatively detected	0.01			GC	MS/MS	QQQ	EI	+	Yes	0.002	No
027	Fully identified (SANCO/1/257/2013)	0		2 Transitions	GC	MS/MS	QQQ	EI	+	Yes	0.010	No
028	Fully identified (SANCO/1/257/2013)	0.1	0	no	GC	MS	Q	EI	+	Yes	0.01	No
029	Fully identified (SANCO/1/257/2013)			spectrum	GC		TOF	EI	Yes	Yes	0.01	Agilent 6890/5973N
030	Fully identified (SANCO/1/257/2013)	1	5		GC	MS	IT	EI	Yes	Yes	0.01	Varian 4000
031	Fully identified (SANCO/1/257/2013)	max. 0.5% 1	3 ion ratios	GC	MS	Q	EI	+	Yes	No	0.01	Trace DSQ
032	Fully identified (SANCO/1/257/2013)				LC	MS/MS	QQQ	EI	+	Yes	0.01	No
034	Tentatively detected	<0.2 min	1 Transition	LC	MS/MS	QQQ	ESI	+	Yes	0.01	No	
035	Fully identified (SANCO/1/257/2013)	2.5%	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	0.01	No	
036	Fully identified (SANCO/1/257/2013)			LC	MS/MS	QQQ	ESI	+	Yes	0.01	No	
037	Tentatively detected	<30	5	isotope	LC	MS	Orbitrap	ESI	+	No	No	Exactive
												AcN/ 1% acetic acid
												Mechanical
												30
												Liquid-Liquid Partitioning
												C18 Atlantis
												ToxID
												600
												Every batch (100+ pesticides)
												Every batch
												404
												Every batch
												139
												Every batch
												139
												Always
												DSPE
												C18 50x2mm
												10
												Both
												method 550
												Final Report- EURL-Fv-SM-06, 2014

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/differentiation was done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Sample Weight (g)	Scanning Model	Extraction Solvent	pH adjusted	Agitation Mode	Injection Volume (μL)	Compounds in method (pesticides Only)	Standard Solution Frequency	Phorate								
																	Column Type								
																		Software	Injection Volume (μL)						
038	Fully identified (SANCO/1257/2013)	<20	spectrum	GC	MS	TOF	EI	+	Yes	Yes	0.05	Yes	Leco Pegasus IV	1% Acetic acid	No	Mechanical	30	Liquid-Liquid Partitioning + DSPE	RTX-Clpesticides	10	MetAlign + ChromatOF	560	Every batch (235 pesticides)		
039	Fully identified (SANCO/1257/2013)	10	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.01	No	4000	10	ACN	No	Mechanical	5	DSPE	C18	10	Analyst	Daily		
040	Fully identified (SANCO/1257/2013)	0.24	2 Transitions	GC	MS/MS	QQQ	ESI	+	Yes	Yes	0.002	No	Bruker Scion GC-MS TQ	15 Acetone/DCM/PE	No	Mechanical	0.5	None	VF5-ms, 30mmx25 mm (0.25 μm film)	5	Workstation	336	Every batch		
041	Tentatively detected		MS/MS	GC	MS	Q-TOF	ESI	+	No	Yes	Yes	Yes	Agilent GC QTof	10	ACN	No	None	2	DSPE	DB5	5	Mass Hunter	100	Always	
042	Fully identified (SANCO/1257/2013)		2 Transitions	GC	MS/MS	QQQ	EI	+	Yes	Yes	0.01	No	Varian 1200	10	EtOAc	No	Mechanical	20	DSPE	DB5-MS	5	Workstation	171	Every batch	
044	Fully identified (SANCO/1257/2013)	-0.6		GC	MS	Q	EI	+	Yes	Yes	0.02	Yes	AT5975 GC-MS	10 ACN/EtOAc	No		2		HP5-MS	10	Chemstation	~750	Always		
044	Fully identified (SANCO/1257/2013)	0.2	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes		No	AB4000	10	ACN	No		2	None	C18	10	Analyst	110		
045	Fully identified (SANCO/1257/2013)	0.2	2 Transitions	GC	MS/MS	QQQ	EI	Yes	Yes	Yes	0.010	No	7000 Agilent	10 EtOAc	No	Mechanical	5	DSPE	HP5-MS	2	Mass Hunter	150	No screening methods		
046	Fully identified (SANCO/1257/2013)	2	LC	MS/MS	QQQ	ESI	-	Yes	Yes	Yes	0.01	No	Agilent 6460	10	EtOAc	No	Mechanical	15	None	C18	2	Mass Hunter	550	Always	
047	Fully identified (SANCO/1257/2013)	0.06	2 Transitions	GC	MS	IT	EI	+	Yes	Yes	0.01	No	Varian Saturn 4000	10	ACN	No	Mechanical	20	None	Restek Rx-5ms	3	Varian Ms Workstation	282	Every batch	
051	Fully identified (SANCO/1257/2013)	0.1		GC	FPD				Yes	Yes	0.01		Varian 3800 GC	10	ACN	Yes	Mechanical	3	DSPE	Rtx-OPP	2	Galaxie	363	Every batch	
053	Fully identified (SANCO/1257/2013)	0	1	3	GC	MS/MS	QQQ	EI	+	Yes	Yes	0.01	Yes	7000A	10	ACN	Yes	Mechanical	15	DSPE	DB5-MS	3	Mass Hunter	350	Often
056	Tentatively detected	1	1	GC	MS	Q	EI	Yes	No	0.01	Yes	Agilent 5973	10	ACN	No	Manual	1	PSA	HP-5MS UI	2	Auto	923	Every batch		
057	Fully identified (SANCO/1257/2013)		2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.01	No	Quattro Premier	10	ACN	No	Manual	2	DSPE	C18	5	Mastlynx	196		
058	Tentatively detected	30	5	Accurate mass	LC	Orbitrap	Orbitrap	ESI	+	Yes	Yes	0.01 - 0.05	Thermo Orbitrap	10	ACN	Yes	Mechanical	10	PSA	RPI-8	10	Xcalibur	>100	Every batch	
060	Fully identified (SANCO/1257/2013)			GC	MS	Q	EI	+	No	No	Yes	Agilent	10	ACN	Manual	2	DSPE	HP5-MS	20	AMDIS	~550				

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/determination done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification details	Chromatographic Technique	Detector	Analyzer	Polarity	Routine Method?	Routine Scope?	Screening Detection limit (SDL) (mg/kg)	Instrument Model	Full Scan	Sample Weight (g)	Extraction Solvent	pH adjusted	Agitation Mode	Agitation time (min)	Column Type	Injection Volume (μL)	Compounds in method or library (pesticides Only)	Standard Solution Frequency	
062 Tentatively detected	-2.0		GC	MS	Q	EI	+	Yes	No	0.01	Yes	Agilent GC-MSD 7890A/5975C	10	AcN	Yes	Manual	5	DSPE	HP5-MS	1	Auto	927	Weekly
064 Fully identified (SANCO/1257/2013)			GC	MS	TOF	EI	+	Yes	Yes		Yes	Pegasus 4D	15	Acetone/PE/DCM	No	Mechanical	1	None	TR5-MS	1	Chroma TOF	76	
065 Fully identified (SANCO/1257/2013)	45		GC	MS	Q	EI	+	Yes	Yes	0.01	Yes	Shimadzu QP2010+	10	AcN	No		30	DSPE	VF-5-MS-30M ^{0.25*} 0.25μm	10	GC-MSolution	112	Monthly
066 Fully identified (SANCO/1257/2013)			GC	MS/MS	QQQ	EI		Yes	Yes	0.01	No	Agilent 7000	15	EtOAc	No	Mechanical	1	None	DB-5MS Capilar	2		200	
067 Fully identified (SANCO/1257/2013)			GC	MS/MS	QQQ	EI		Yes	Yes	0.01	No	Agilent 7000	10	AcN	No	Mechanical	1	DSPE	DB-5MS UI	2	Mass Hunter	236	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/identification done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Detector	Analyzer	Ionization Method	Routine Method?	Routine Scope?	Screening Detection Limit (SDL)? (mg/kg)	Full Scan	Instrument Model	Extraction Solvent	Column Type	Inj	Mass Hunter	350	Daily				
Prosulfocarb																						
001 Fully identified (SANCO/12571/2013)	3 Transitions	GC	MS/MS	QQQ	EI	+	Yes	Yes	0.01	No	Agilent 7000	10	AcN	No	1	None	HP5-MS	1	Mass Hunter			
003 Fully identified (SANCO/12571/2013)		LC	MS/MS			+	Yes	No				10	AcN	Yes	Manual	1	DSPE					
004 Fully identified (SANCO/12571/2013)	2 Transitions	GC	MS/MS	QQQ	EI	+	Yes	Yes		No	Agilent	10	AcN	Yes	Mechanical	15	DSPE	HP5-MS	2			
006 Tentatively detected		GC	MS/MS	QQQ	EI	+	No	No		No	Agilent	15	Acetone/DCM/ PE	No	Mechanical	1	None	DBS	1			
007 Fully identified (SANCO/12571/2013)	30 confirmed on Tof, GC-MS	LC	Tof	Q-Tof	Jet Stream	+	No	No	NA	Yes	Agilent 6530 accurate mass Q-ToF LC/MS	10	AcN		0.5	None	C18	3	Mass Hunter			
008 Fully identified (SANCO/12571/2013)		LC	MS/MS				Yes	Yes				13	AcN	No	Mechanical	None	C18 Aqua Phenomenex	20	Both			
009 Fully identified (SANCO/12571/2013) +/- 0.2 min <5 acc. m/z + isotope + fragment		LC	MS/MS	Q-Tof	ESI	+	No	No	0.05 - 0.1	Yes	API 5600 Q- Tof	10	AcN	No	Mechanical	5	None	Acuity BEH C18	10	Waters		
010 Fully identified (SANCO/12571/2013)		LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.010		Waters Xevo TQ	10	AcN	No	Manual	2	DSPE	Acuity BEH C18	10	Waters		
011 Fully identified (SANCO/12571/2013)	3 2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.005-0.1	No	Xevo TQ	10	AcN	No		2	None	C18	2.5	Masslynx		
011 Tentatively detected	3 0.5 2 Transitions	LC	MS/MS	Q-Tof	ESI	+	No	No	0.005-0.1	Yes	TripleTof	10	AcN	No		2	None	C18	2	PeakView		
012 Fully identified (SANCO/12571/2013)	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	10	Yes	Ultra Thermo	10	AcN	No	Manual	5	DSPE	C18	10			
013 Tentatively detected	10 2	GC	MS/MS	QQQ	EI	+	Yes	0.01		Agilent 7890/ Agilent 7000	10	AcN	Yes		5	DSPE	DBS-MS	5	Mass Hunter			
014 Tentatively detected	5 5	LC	MS	TOF	ESI	+	Yes	No	0.01 - 0.10	Yes	LCT PREMIER XE	15	Acetone/DCM/ PE	No		1.5	None	C18	10	Masslynx		
015 Fully identified (SANCO/12571/2013)	10 Accurate mass	GC	MS/MS	QQQ	EI	Yes	Yes	0.01	Yes	5977 e	Cyclotrihexane/ EtOAc/Aceton	No		3	DSPE	HP5-MS	1	Mass Hunter	850	Daily		
016 Fully identified (SANCO/12571/2013)	0	LC	MS/MS	Q-TRAP	ESI	+	Yes	Yes	0.01	No	ABSciex 4000 QTrap	10	AcN	Yes		1	None	Phenomenex Synergie Fusion RP 2.5 µm 50x2.0	-250	Always		
018 Fully identified (SANCO/12571/2013)		LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.010	No	Qtrap3200	10	AcN	No	Manual	1	DSPE	C18	5	Analyst		
020 (SANCO/12571/2013)	2 Transitions	GC	MS/MS	QQQ	EI	+	Yes	Yes	0.005	No	TSQ Quantum XLS Ultra	10	AcN	No	Mechanical	30	DSPE	RTX5-MS	1	TraceFinder	300	Always

APPENDIX 3. Methods used by participants for detecting pesticides.

Prosulfocarb												
Laboratory Code *NRL	How was the detection/identification was done?	RT Deviation (s)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Extraction Solvent	Agitation Mode	Software	
											Compounds in method (pesticides Only) or library	
021 (SANCO/12571/2013)	Fully identified	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	0.005	No	Waters Xevo TQ	
024	Tentatively detected	3	5	25.1,1346, transition 1: 25.1,1346>9.10543, transition 2: 25.1,1346>28.1072	LC	MS	Q-ToF	ESI	+ No	0.01-0.1	Yes	Agilent 6550 QTOF
025	Fully identified (SANCO/12571/2013)	3	0	2 Transitions	LC	MS/MS	QQQ	ESI	+ Yes	0.02	No	Waters Quattro Iist
026	Tentatively detected	5	5	Accurate mass	LC	MS/MS	Q-Orbitrap	ESI	+ No	0.010	Yes	Q Exactive 10
027	Fully identified (SANCO/12571/2013)	0	0	2 Transitions	LC	MS/MS	QQQ	ESI	+ Yes	0.010	No	Xevo 10 Acetone/DCM/PE
028	Fully identified (SANCO/12571/2013)	0.1	0	2 Transitions	LC	MS/MS	QQQ	ESI	+ Yes	0.005	No	Agilent 6410 Triple Quad
029	Fully identified (SANCO/12571/2013)			2 Transitions	LC		QQQ	ESI	+ Yes	0.01	No	API 5500 10 ACN
030	Fully identified (SANCO/12571/2013)	6	5	2 Transitions	LC	MS/MS	QQQ	ESI	+ Yes	0.01	No	AbSciex 3200 QTRAP
031	Fully identified max. 2.5% (SANCO/12571/2013)	1	2 Transitions	LC	MS/MS	QQQ	ESI	+ Yes	0.01	No	Premier Xe	
032	Fully identified (SANCO/12571/2013)										Waters Aquity UPLC system, API 5000 QQQ mass spectrometer Applied Biosystems	
033	Tentatively detected		2 Transitions	LC	MS/MS	QQQ	ESI	+ No	No	Varian 320	7.5 ACN/DCM	
035	Fully identified (SANCO/12571/2013)	2.5%	2 Transitions	LC	MS/MS	QQQ	ESI	+ Yes	0.01	No	HPLC Agilent 1100, MS API 3000	
036	Fully identified (SANCO/12571/2013)			LC	MS/MS	QQQ	ESI	+ Yes	0.01	No	Agilent 6490	
037	Tentatively detected	<30	5	isotope	LC	MS	Orbitrap	ESI	+ No	No	Not Established	
											1% acetic acid	
											Mechanical	
											30 Liquid-Liquid Partitioning	
											5 ToxID	
											600 Every batch (100+ pesticides)	
											MS Workstation	
											10 Both method 550	
											15 DSPE C18 50x2mm	
											2 Analyst 404	
											10 MassLynx	
											5 MassHunter 550	
											Daily	

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory/NRL Code	How was the detection/identification done?	RT Deviation (s)	MS Tolerance (ppm)	Other Identification Details	Detector	Analyzer	Ionization Mode	Routinely Method?	Routine Scope?	Screening Detection Limit (SDL)? (mg/kg)	Sample Weight (g)	Extraction Solvent	Column Type	Injektion Volume (µL)	Compounds in method (pesticides ONLY)	Standard Solution Method	Frequency							
037 (SANCO/1257/2013)	Fully identified	<20	spectrum	GC	MS	TOF	EI	+	Yes	Not established	Yes	Leco Regosys IV	1% acetic acid	No	Mechanical	30	Liquid-Liquid Partitioning + DSPE	10	MetAlign + ChromatOF	560	Every batch (235 pesticides)			
038 (SANCO/1257/2013)	Fully identified	10	2 Transitions	LC	MS/MS	QQQQ	ESI	+	Yes	Yes	0.01	No	4000	10	ACN	No	Mechanical	5	DSPE	C18	10	Analyst	Daily	
039 (SANCO/1257/2013)	Fully identified	0.18	2 Transitions	LC	MS/MS	QQQQ	ESI	+	Yes	Yes	0.005	No	Waters Quattro Premier XE	15 Acetone/DCM/PE	No	Mechanical	0.5	None	Acquity BEH (100x2.1 mm i.d.)	5	TargetLynx	215	Every batch	
040 Tentatively detected	0	+9.1	accurate mass + fragment + NRM	LC	MS	Q-TOF	ESI	+	No	Yes	0.01	Yes	Waters Xevo Q-TOF (1st gen)	25 ACN	No		2	None	Acquity BEH C18	3	Target Lynx + MassFragment tool	500	Always	
041 Tentatively detected	-9	5	GC-QTOF	LC	MS	Q-TOF	ESI	+	No	Yes		Yes	Agilent 6550 iFunnel QTOF LC/MS	10 ACN	No		2	DSPE	C18	5	Mass Hunter	1700	Always	
042 (SANCO/1257/2013)	Fully identified		2 Transitions	LC	MS/MS	QQQQ	ESI	+	Yes	Yes	0.01	No	Agilent 6490	10 EIOAC	No	Mechanical	20	None	HSST3	2	Mass Hunter	265	Every batch	
043 Tentatively detected	0.5%	0.3	2 Transitions	GC	MS	Q	EI	Yes	Yes	0.01	No	Hewlett Packard 5870 & 5973 GC-MS	15 Acetone/DCM/PE	No	Mechanical	1	Liquid-Liquid Partitioning	HP5-MS	1	Chemstation	201	Day of run		
044 (SANCO/1257/2013)	Fully identified	3.2		GC	MS	Q	EI	+	Yes	Yes	0.02	Yes	AT5975 GC-MS	10 ACN/EIOAC	No		2		HP5-MS	10	DPS/Chemstation	-750	Always	
046 Tentatively detected	0.04	0.4	Acccurate mass	LC	MS	TOF	ESI	+	Yes	Yes	0.02	Yes	Brucker Maxis 10 EIOAC	10			15	None	C18	2	TargetAnalysis	750	Always	
047 (SANCO/1257/2013)	Fully identified	3	0.04	3 Transitions	GC	MS	IT	EI	+	Yes	Yes	0.01	No	Varian Saturn 4000	10 ACN	No	Mechanical	20	None	Restek Rx-5ms	3	Variam Ms Workstation	282	Every batch
048 (SANCO/1257/2013)	Fully identified	1		2 Transitions	LC	MS/MS	QQQQ	ESI	+	Yes	Yes	0.010	Yes	Waters UPLC QD	15 ACN	Yes		1	DSPE	UPLC BEH C18	10	Auto	435	Every batch
049 (SANCO/1257/2013)	Fully identified			LC	MS/MS	QQQQ	ESI	+	Yes	Yes	0.05	No	Agilent 6490 LC/MS/MS	15 Acetone/DCM/PE	No		1	None	C18	10	Mass Hunter	250	Every batch	
051 (SANCO/1257/2013)	Fully identified	0.1		GC	MS	IT	EI		Yes	No	0.01	Yes	Variam 3800 GC + Saturn 2000 MS	10 ACN	Yes	Mechanical	3	DSPE	RTX5-MS	3	Workstation	363	Every batch	
052 (SANCO/1257/2013)	Fully identified	12	2 Transitions ratio 30%	LC	MS/MS	QQQQ	ESI	+	Yes	Yes	0.010	Yes	AB SCIEX 4000 QTRAP	10 ACN	No	Mechanical	20	SPE	C18	20	Analyst	1	Every batch	
053 (SANCO/1257/2013)	Fully identified	0	1	Acccurate mass	LC	MS/MS	TOF	ESI	+	Yes	Yes	0.01	Yes	XEV/O G2-S QTOF	10 ACN	Yes	Mechanical	15	DSPE	C18	1	Unify	900	Often
054 (SANCO/1257/2013)	Fully identified	2.5		2 Transitions	LC	MS/MS	QQQQ	ESI	+	Yes	Yes	0.01	Yes	Agilent G6410A	10 ACN	Yes		25	SPE	ZORBAX ECLIPSE	20	Auto	143	Every batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Prosulfocarb									
Laboratory Code *NRL	How was the detection/identification was done?	MS Tolerance (ppm)	RT Deviation (s)	Other Identification Details	Chromatographic Technique	Analyser	Ionization Mode	Routine Method?	Routine Scope?
055	Tentatively detected	none	2 Transitions	LC MS/MS QQQQ	ESI + No 0.01	No API 4000	10 ACN Yes	Manual	5 DSPE Waters C18 2.1x50mm 1.7µm
056	Tentatively detected	1 1	GC MS Q	MS Q	ESI Yes No 0.01	Yes Agent 5973	10 ACN No	Manual	1 PSA HP-5MS UI 2
057	Tentatively detected		GC MS Q	MS Q	ESI + No No	Yes HP-5973	10 ACN No	Manual	2 DSPE SE54 5 DRS
058	Tentatively detected	30 5	Accurate mass LC Orbitrap Orbitrap	Orbitrap Orbitrap	ESI + Yes 0.01 - 0.05	Yes Thermo Orbitrap	10 ACN Yes	Mechanical	10 PSA RP 18 10 Xcalibur > 100
059 (SANCO/1257/2013)	Fully identified	2	LC MS/MS QQQQ	ESI + Yes Yes 0.01	No Varian-320 10	ACN No	3 DSPE C18 50x2mm	10 MS Workstation	1/62 Always
060 (SANCO/1257/2013)	Fully identified	2 Transitions + GC/MS scan	LC MS/MS Q-TRAP	ESI + No No	ESI + No No	3200 Qtrap API 10 ACN	Manual	2 DSPE Atlantis T3 5	
062	Tentatively detected	2.0	GC MS Q	MS Q	ESI + Yes No 0.05	Yes Agilent 7890A/5975C	10 ACN Yes	Manual	5 DSPE HP-MS 1 Auto 927 Weekly
064	Tentatively detected		GC MS TOF	TOF	ESI + No No	Yes Pegasus 4D 15	Acetone/PE/DCM	No Mechanical	1 None TR5MS 1 Chroma TOF 76
065 (SANCO/1257/2013)	Fully identified	60	2 Transitions	LC MS/MS QQQQ	ESI + Yes Yes 0.01	No AB-Sciex Qtrap 5500	10 ACN No	30 DSPE YMC-UltrahydroSphere C18 75x 2.0mm I.D.	5 Analyst 150 Daily
066 (SANCO/1257/2013)	Fully identified		2 Transitions	LC MS/MS QQQQ	ESI + Yes Yes 0.01	No Waters TQD 10	ACN Yes	Mechanical 16 None Acuity UPC BECH C18 1.7 mm 2.1 x 100 mm	14 200
067 (SANCO/1257/2013)	Fully identified		2 Transitions	GC MS/MS QQQQ	ESI El Yes Yes 0.01	No Agilent 7000 10	ACN No	Mechanical 1 DSPE DB-5MS UI 2 Mass Hunter 236	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/differentiation done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification details	Chromatographic Technique	Detector	Analyzer	Instrument Model	Sample Weight (g)	Full Scan	Extraction Solvent	Agitation Mode	Column Type	Injection Volume (μL)	Compounds in method (pesticides Only)	Standard Solution Frequency						
006	Tentatively detected			GC	MS/MS	QQQ	EI	+	No	No	Agilent 15 Acetone/DCM/PE	No	Mechanical	1	None	DB5	1					
007	Tentatively detected	30		LC	ToF	Q-ToF	Jet Stream	+	No	No	Agilent 6530 accurate mass Q-ToF LCMS	10	AcN		0.5	None	C18	3				
009	Fully identified (SANCO/1/257/2013)	+/- 0.2 min	3 ions	GC	MS	Q	EI	+	Yes	No	0.01	Yes	Bruker 450 Scion TQ	10	AcN	No	Mechanical	5	None	HP 1 MS UI	5	
010	Fully identified (SANCO/1/257/2013)			LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.010	Waters Xevo TQ	10	AcN	No		2	DSPE	Acuity BEH C18	10		
011	Tentatively detected	0.1	2 Transitions	LC	MS/MS	Q-ToF	ESI	+	No	No	0.005-0.1	Yes	TripleTOF 10	10	AcN	No		2	None	C18	2	
012	Fully identified (SANCO/1/257/2013)			GC	MS/MS	QQQ	EI	+	No	No	0.01	Yes	TSQ Quantum XLS Thermo	10	AcN	No	Manual	5	SPE	C18	1	
013	Tentatively detected	10	2	GC	MS/MS	QQQ	EI	+	Yes		0.01	Yes	Agilent 7890/Agilent 7000	10	AcN	Yes		5	DSPE	DB5-MS	5	
015	Fully identified (SANCO/1/257/2013)	4	10	Accurate mass	GC	MS/MS	QQQ	EI	Yes	Yes	0.01	Yes	5977	10	Cyclonexane/EtOAc/Acetone	No		3	DSPE	HP5-MS	1	
016	Tentatively detected	-8.34		GC	MS/MS	QQQ	EI	+	Yes	Yes	0.01	No	Agilent 7000	10	AcN	Yes		1	DSPE	HP5MSUI	10	
018	Fully identified (SANCO/1/257/2013)			LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.010	No	Qtrap3200	10	AcN	No	Manual	1	DSPE	C18	5	
021	Fully identified (SANCO/1/257/2013)		2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.005	No	Waters Xevo TQ	15	AcN	No	Mechanical	5	None	HSS T3	10	
024	Tentatively detected	3	5	accurate mass: 225.1593, 225.1592>>70.1037, transition 1; 225.1593>>100.0507, transition 2.	LC	MS	Q-ToF	ESI	+	No	0.01-0.1	Yes	Agilent 6550 Q-ToF	10	AcN	No	Manual	1	QueChERS without PSA	Atlantis T3, 2.1x100 mm, 3μm	2	
026	Tentatively detected	0.01	5	Accurate mass	LC	MS/MS	Q-Orbitrap	ESI	+	Yes	Yes	0.010	Yes	Q Exactive 10	10	AcN	No	Mechanical	10	DSPE	BEH C18	5
029	Tentatively detected		spectrum	GC			TOF	EI	Yes	No		Yes	LECO Pegasus IV	10	AcN	Yes	Mechanical	15	DSPE	HP5-MS	3	
030	Tentatively detected		5	GC	MS	IT	EI	Yes	No		Yes	Varian 4000	10	AcN	No	Mechanical	1	SPE	Rxi 5ms 5% diphenyl/ 95% dimethyl polysiloxane	1		
031	Fully identified (SANCO/1/257/2013)	max. 0.5%	1	3 ion ratios	GC	MS	Q	EI	+	Yes	0.01	Yes	Trace DSQ	15	AcN	Yes	Manual	1	SPE	DB5MS	0.8	
																		Xcalibur	350	Every batch		

APPENDIX 3. Methods used by participants for detecting pesticides.

Secbumeton											
Laboratory Code *NRL	How was the detection/differentiation was done?	RT Deviation (s)	MS Tolerance (ppm)	Other Identification Details	Detector	Analyzer	Polarity	Routine Method?	Screening Detection Limit (SDL) (mg/kg)	Instrument Model	Sample Weight (g)
033 Tentatively detected		Accurate mass	GC MS/MS QQQ	EI + No	No	Varian 320	7.5 Acetone/DCM/ PE	Yes	Exactive	AcN/ 1% acetic acid	No
037 Tentatively detected	<30	5 Isotope spectrum	LC MS Orbitrap ESI	+ No	No	Not Established	Yes	Yes	1% acetic acid	Mechanical	Manual
038 Fully identified (SANCO/1257/2013)	<20		GC MS TOF	EI + Yes	Yes	Established	Yes	Yes	AcN/ 1% acetic acid	Liquid-Liquid Partitioning	C18 Atlantis 30
040 Tentatively detected	10	Accurate mass	GC MS Q	EI + Yes	Yes	0.01	No	Agilent 10	AcN	Mechanical	DB5
041 Tentatively detected	-7	GC-QTOF accurate mass + MS _e	LC MS Q-TOF ESI	+ No	No	0.01	Yes	Waters Xevo Q-TOF (1st gen)	EHOAC	Yes	AcqB/EI C18
044 Fully identified (SANCO/1257/2013)	1.1		GC MS Q	EI + Yes	Yes	0.02	Yes	Agilent 6550 iFunnel QTOF LC/MS	AcN/EHOAC	No	DSPE
046 Tentatively detected	0.22	2.3 Accurate mass	LC MS TOF ESI	+ Yes	Yes	0.02	Yes	Agilent 6575 GC-MS	AcN/EHOAC	No	C18
048 Fully identified (SANCO/1257/2013)	1	2 Transitions	LC MS/MS QQQ	ESI + Yes	Yes	0.010	Yes	Bruker maxis 10	EHOAC		Target Analysis
050 Tentatively detected		2 Transitions	LC MS QQQ	ESI + No	No			Waters Acquity UPLC TQD	AcN	None	15
053 Fully identified (SANCO/1257/2013)	0	1 Accurate mass	LC MS/MS TOF ESI	+ Yes	Yes	0.01	Yes	Waters Acquity UPLC TQD	AcN	Yes	DSPE
056 Tentatively detected	1		GC MS Q	EI + No	No	0.01	Yes	Agilent 59730	AcN	No	PSA
057 Tentatively detected			GC MS Q	EI + No	No		Yes	HP-59730	AcN	No	DSPE
058 Tentatively detected	15	1 target + qualifier	GC MS Q	EI + Yes	Yes	0.01 - 0.05	Yes	XEVO C2-S QToF	AcN	Yes	Mechanical
060 Fully identified (SANCO/1257/2013)			GC MS Q	EI + No	No		Yes	Agilent 59730	AcN	No	PSA
062 Tentatively detected	-0.7		GC MS Q	EI + Yes	Yes	0.01	Yes	Agilent GC-MSD 7890A/5975C	AcN	Yes	DSPE
064 Tentatively detected			GC MS TOF EI	+ No	No		Yes	Pegasus 4D 15	Acetone/PE/ DCM	No	Mechanical
Standard Solution (pesicides Only or library)											
Software											
Injection Volume (μl)											
Column Type											
Agrifaction time (min)											
Clean up Step											
Liquid-Liquid Partitioning											
Standard Solution											
Frequency											

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/identification done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification details	Chromatographic Technique	Detector	Analyzer	Polarity	Routine Method?	Routine Scope?	Screening Detection Limit (SDL) (mg/kg)	Instrument Model	Sample Weight (g)	Extraction Solvent	pH adjusted	Agitation Mode	Agitation time (min)	Injection Volume (μL)	Software	Standard Solution Frequency			
065 Fully identified (SANCO/1/257/2013)	60	3 diagnostic ions	GC	MS	Q	EI	+	Yes	No	0.02	Shimadzu QP2010+	10	ACN	No	30	DSPE	VF-5-MS-30M*×25* 0.25μm	10	GC-MSolution	325	3 Months		
067 Fully identified (SANCO/1/257/2013)		2 Transitions	GC	MS/MS	QQQ	EI		Yes	Yes	0.01	No	Agilent 7000	10	ACN	No	Mechanical	1	DSPE	DB-SqMS UI	2	Mass Hunter	236	Daily
068 Tentatively detected		full scan spectra	GC	MS	Q		+	No	No	Yes	thermo trace gc-dsq	10	ACN	Yes		2	SPE	elite 5 ms	1	Xcalibur	100	Every year	

APPENDIX 3. Methods used by participants for detecting pesticides.

Spiromesifen												
Laboratory Code *NRL	How was the detection/differentiation done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification Details	Detector	IonizationMode	Polarity	Routine Method?	Screening Detection Limit (SDL) (mg/kg)	Injection Volume (μl)	Standard Solution (pesticides Only) or library	
001 Fully identified (SANCO/1257/2013)	3 Transitions	GC MS/MS	QQQ	EI + Yes	0.01	No	Agilent 7000	10	ACN	No	1	None HP5-MS
003 Fully identified (SANCO/1257/2013)		GC NS/MS		+ Yes	No			10	ACN	Yes	Manual	1 SPE
006 Fully identified (SANCO/1257/2013)		GC NS/MS	QQQ	EI + Yes	Yes	No	Agilent	15	Acetone/DCM/PE	No	Mechanical	1 DB5
007 Fully identified (SANCO/1257/2013)	30 5 confirmed on ToF, LC-MS/MS acc. m/z + isotope + fragment	LC ToF	Q-TOF	Jet Stream + No	No	Yes	Agilent 6530 accurate mass Q-ToF LC/MS	10	ACN	0.5	None	C18 3 Mass Hunter 550
008 Fully identified (SANCO/1257/2013)		GC NS/MS				Yes	Yes			13	ACN	No Mechanical
009 Fully identified (SANCO/1257/2013) +/- 0.2 min <5		LC NS/MS	Q-TOF	ESI + No	No	0.05 - 0.1	Yes	API 5600 Q-ToF	10	ACN	5	None C18 Aqua Phenomenex 20 Both 600
010 Fully identified (SANCO/1257/2013)		GC NS/MS	QQQ	EI + Yes	Yes	0.010	Bruker SCION	10	ACN	No	Manual	2 DSPE Zebtron ZB 2 Bruker 210
011 Fully identified (SANCO/1257/2013)	3 2 Transitions	LC MS/MS	QQQ	ESI + Yes	Yes	0.005-0.1	No	Xevo TQS	10	ACN	2 None C18 2.5 Masslynx 300	
Tentatively detected	3 0.4 2 Transitions	LC MS/MS	Q-TOF	ESI + No	No	0.005-0.1	Yes	TripletOF	10	ACN	2 None C18 2 PeakView 620	
012 Fully identified (SANCO/1257/2013)		GC NS/MS	QQQ	EI + No	No	10	Yes	TSQ QUANTUM XLS Thermo	10	ACN	5 SPE C18 1	
Tentatively detected	10 2	GC NS/MS	QOrbitrap	ESI + Yes	Yes	0.01	Agilent7890/Agilent7000	10	ACN	Yes	5 DSPE C18 5 DB5-MS 5 Mass Hunter ~500	
013 Tentatively detected 0.02	2	LC NS/MS	QOrbitrap	ESI + Yes	Yes	0.01	Thermo Q-Exactive	10	ACN	Yes	5 DSPE C18 5 Xcalibur ~500	
Fully identified (SANCO/1257/2013)	5 10 Accurate mass	GC NS/MS	QQQ	EI + Yes	Yes	0.01	Yes	5977	10 Cyclohexane/EtOAc/Acetone	No	3 DSPE HP5-MS 1 Mass Hunter 850	
Fully identified (SANCO/1257/2013)	0.9	GC NS/MS	QQQ	EI + Yes	Yes	0.01	No	Agilent 7000	10	ACN	1 DSPE HPSMSU 10 Mass Hunter ~300	
Fully identified (SANCO/1257/2013)		LC NS/MS	QQQ	ESI + Yes	Yes	0.010	No	Qtrap3200	10	ACN	1 Manual DSPE C18 5 Analyst 205	
Fully identified (SANCO/1257/2013)		LC NS/MS	Q-JRAP	ESI + Yes	Yes	0.01	No	5500	10	ACN	1 Manual DSPE C18 10 Analyst	
Fully identified (SANCO/1257/2013)	5 Accurate mass	LC MS	Q-Orbitrap	ESI + No	No	0.005	Yes	Excutive	10	ACN	30 DSPE Kinetex C18 10 Exactifinder 400 Often	

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/differentiation done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Sample Weight (g)	Instrument Model	Full Scan	Screening Detection Limit (SDL)7 (mg/kg)	Agitation Scope?	Routine Method?	Polarity	Sample Weight (g)	Extraction Solvent	Cleant up Step	Column Type	Injection Volume (μL)	Compounds in method or library (pesticides Only)	Standard Solution Frequency	Spiromesifen			
024	Fully identified (SANCO/1/257/2013)	5	Nanochip spectra with standard: 941	GC	MS/MS	QQQ	EI	+ Yes	Yes	LECO Pegasus 4D	10	AcN	No	Manual	1	QueCHERS without PSA	DB5-MS (30m x 0.25mm x 0.25μm) in the 1st Dimension; BPX-50 (2m x 0.1mm x 0.1μm) in the 2nd Dimension	10	Chromatof	200	Before and after sample	Always				
025	Fully identified (SANCO/1/257/2013)	2	0	2 Transitions	GC	MS/MS	QQQ	EI	+ Yes	Yes	0.010	No	Varian 320	50	Acetone	No	Mechanical	2	Liquid-Liquid Partitioning	DB5	8	Star	146	Always		
026	Tentatively detected	5	Accurate mass	LC	MS/MS	Q Orbitrap	ESI	+ No	No	0.002	Yes	Q Exactive	10	AcN	No	Mechanical	10	DSPE	BEH C18	5		700				
027	Fully identified (SANCO/1/257/2013)	0	2 Transitions	GC	MS/MS	QQQ	EI	+ Yes	Yes	0.010	No	Thermo 10	Acetone/DCM/ PE	No		1	None	HP5-MS	1	Thermo	201	Every batch				
029	Fully identified (SANCO/1/257/2013)		spectrum	GC			TOF	EI	Yes	Yes	0.01	Yes	LECO Pegasus IV	10	AcN	Yes	Mechanical	15	DSPE	HP5-MS	3	Both	750	Always		
030	Fully identified (SANCO/1/257/2013)	0	5	GC	MS	IT	EI	Yes	Yes	0.01	Yes	Varian 4000	10	AcN	No	Mechanical	1	SPE	Rxi 5ms 5% diphenyl / 95% dimethyl polysiloxane	1	Varian MS Workstation	196	Every batch			
031	Fully identified (SANCO/1/257/2013)	max 2.5%	1	2 Transitions	LC	MS/MS	QQQ	ESI	+ Yes	Yes	0.01	No	Quattro Premier Xe	15	AcN	Yes	Manual	1	None	C18	3	Quanlynx	350	Every batch		
032	Fully identified (SANCO/1/257/2013)		2 Transitions	GC	MS/MS	QQQ	EI	+ Yes	Yes	0.01	No	Waters Quattro Micro GC	10	EtOAc	Yes	Mechanical	18	Filtration	Rxi-5sil MS/Integra-guard Restek	10	Masslynx	404	Every batch			
033	Tentatively detected		2 Transitions	LC	MS/MS	QQQ	ESI	+ No	No		No	Varian 320	7.5	AcN/DCM	No	Manual	1	Liquid-Liquid Partitioning	Polaris C-18	10	Workstation					
035	Fully identified (SANCO/1/257/2013)	2.5%	2 Transitions	LC	MS/MS	QQQ	ESI	+ Yes	Yes	0.01	No	HPLC Agilent 1100, 10 MS API 3000	10	AcN	Yes		15	DSPE	C18 50x2mm	10	Both	method 550	Always			
036	Fully identified (SANCO/1/257/2013)		GC	MS/MS	QQQ	EI	+ Yes	Yes	0.01	No	Agilent 7000B	5	AcN	Yes		3	None	HP-5MS UI	2	Mass Hunter	550	Daily				
037	Fully identified (SANCO/1/257/2013)	<30	5	isotope	LC	MS	Orbitrap	ESI	+ No	No	Not Established	Yes	1% acetic acid	No	Mechanical	30	Liquid-Liquid Partitioning	C18 Atlantis	5	ToxID	600	Every batch (100+ pesticides)				
038	Fully identified (SANCO/1/257/2013)	10	2 Transitions	LC	MS/MS	QQQ	ESI	+ Yes	Yes	0.01	No	4000	10	AcN	No	Mechanical	5	DSPE	C18	10	Analyt	Daily				

APPENDIX 3. Methods used by participants for detecting pesticides.

Spiromesifen											
Laboratory Code *NRL	How was the detection/differentiation was done?	RT Deviation (s)	MS Tolerance (ppm)	Other Identification Details	Detector	IonizationMode	Polarity	Routine Method?	Screening Detection Limit (SDL) (mg/kg)	Instrument Model	Extraction Solvent
039	Fully identified (SANCO/1/257/2013)	0	2 Transitions	LC MS/MS QQQ	ESI +	Yes	0.005	No	Waters Quattro Premier XE	15 Acetone/DCM/PE	No
040	Fully identified (SANCO/1/257/2013)	0	3+3 + fragment + MRM	LC MS-Q-TOF	ESI	+ No	Yes	0.01	Yes	Waters Xevo Q-TOF (1st gen)	10 EHOAc
041	Tentatively detected	-4	GC-QTOF	IC MS	Q-TOF	ESI +	No	Yes	Yes	Agilent 6550 iFunnel	10 AcN
042	Fully identified (SANCO/1/257/2013)		2 Transitions	GC MS/MS	QQQ	EI +	Yes	Yes	0.01	No	Varian 1200
043	Tentatively detected	0.5%	0.3	2 Transitions	GC MS	Q	EI	Yes	Yes	Hewlett Packard 5870 & 5973	15 Acetone/DCM/PE
044	Fully identified (SANCO/1/257/2013)	3.5		GC MS	Q	EI +	Yes	Yes	0.02	Yes	AT5975 GC-MS
046	Fully identified (SANCO/1/257/2013)	2		GC MS/MS	QQQ	EI +	Yes	Yes	0.01	No	Thermo TSQ 10 EHOAc
047	Fully identified (SANCO/1/257/2013)	3	0.04	2 ions	GC MS	IT	EI +	Yes	No	Varian Saturn 4000	10 AcN
048	Fully identified (SANCO/1/257/2013)	1		GC MS	Q	EI +	Yes	Yes	0.010	Yes	Agilent 7890A GC 5975 MSD
049	Fully identified (SANCO/1/257/2013)			LC MS/MS	QQQQ	ESI +	Yes	Yes	0.01	No	Agilent 6490 LC/MS/MS
050	Tentatively detected		2 Transitions	LC MS	QQQQ	ESI +	No	No	No	TSQ QUANTUM 10	15 AcN
051	Fully identified (SANCO/1/257/2013)	0.2		GC				Yes	Yes	Varian 3800 GC	10 AcN
052	Fully identified (SANCO/1/257/2013)	12	2 Transitions ratio 30%	GC MS/MS	IT	EI	Yes	Yes	0.010	Yes	Agilent 7000B
053	Fully identified (SANCO/1/257/2013)	0	1	Accurate mass	LC MS/MS	TOF	ESI +	Yes	Yes	XEV/O G2S QTof	10 AcN
054	Fully identified (SANCO/1/257/2013)	2.5		2 Transitions	LC MS/MS	QQQQ	ESI +	Yes	Yes	Agilent 6410A	10 AcN
055	Tentatively detected	none		2 Transitions	LC MS/MS	QQQQ	ESI +	No	No	API 4000	10 AcN
056	Tentatively detected	1	1	GC MS	Q	EI	Yes	No	0.01	Yes	Agilent 5973

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the detection/identification done?	RT Deviation (s)	MS Tolerance (ppm)	Other identification details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Limit (SD)? (mg/kg)	Scanning Detection	Instrument Model	Sample Weight (g)	Injection Volume (μL)	Software	Standard Solution Frequency							
057 (SANCO/1257/2013)	Fully identified	15	1	target + qualifier	GC	MS	QQQ	ESI	+	Yes	Yes	0.01	No	Quattro Premier	10	AcN	No	Manual	2	DSPE	C18	5	Masslynx	196	Every batch
058 Tentatively detected		2 Transitions	LC	MS/MS	QQQ	Q	EI	+	Yes	Yes	0.01 - 0.05	Yes	Agilent GC - MSD Qtrap	10	AcN	Yes	Mechanical	10	PSA	DB5	10	ChemStation	>600		
060 Fully identified (SANCO/1257/2013)		2 Transitions + GC/MS scan	LC	MS/MS	Q-TRAP	ESI	+	Yes	Yes				AP 3200 Qtrap	10	AcN		Manual	2	DSPE	Atlantis T3	5				
062 Tentatively detected	0.8		GC	MS	Q	EI	+	Yes	No	0.01	Yes	7890A/5975C	Agilent GC-MSD 10	10	AcN	Yes	Manual	5	DSPE	HP5-MS	1	Auto	927	Weekly	
064 Tentatively detected			GC	MS	TOF	EI	+	No	No			Yes	Pegasus 4D 15	15	Acetone/PE/ DCM	No	Mechanical	1	None	TR5MS	1	Chroma TOF	76		
065 Fully identified (SANCO/1257/2013)	60	2 Transitions	LC	MS/MS	QQQ	ESI	+	Yes	Yes	0.01	No	ABSciex Qtrap 5500	10	AcN	No		30	DSPE	YMC-Ultrachrom Hydrosphere C18 75x 2.0mm I.D.	5	Analyst	150	Daily		
067 Fully identified (SANCO/1257/2013)		3 Transitions	GC	MS/MS	QQQ	EI	Yes	Yes	0.01	No	Agilent 7000	10	AcN	No	Mechanical	1	DSPE	DB-SMS UI	2	Mass Hunter	236	Daily			

APPENDIX 3. Methods used by participants for detecting pesticides.

Labbordatory Code *NRL	How was the identification done?	RT Deviation (s)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	IonizationMode	Routine Scope?	Screening Detection Limit (mg/kg)	Instrument Model	Extraction Solvent	pH adjusted	Agitation Mode	Injection Volume (µl)	Compounds in library (pesticides Only)	Standard Solution Frequency			
001	Fully identified (SANCO/1257/2013)	3 Transitions	GC MS/MS	QQQ	EI +	Yes	0.01	No	Agilent 7000	10	AcN	No	None	HP5-MS	1	Mass Hunter	350	Daily		
002	Fully identified (SANCO/1257/2013)		LC NS/MS	QQQ	ESI +	Yes			Agilent 6410	10	ACN/ACN		1	SPE	C18 4 micras	5	Mass Hunter	221		
003	Fully identified (SANCO/1257/2013)		LC	MS/MS		+	Yes	No		10	AcN	Yes	Manual	1	SPE					
004	Fully identified (SANCO/1257/2013)	0	GC	MS/MS	QQQ	EI +	Yes	Yes	No	Agilent	10	AcN	Yes	Mechanical	15	DSPE	HP5-MS	2	Mass Hunter >300	
006	Tentatively detected		GC	MS/MS	QQQ	EI +	No	No	No	Agilent	15	Acetone/DCM/PE	No	Mechanical	1	None	DB5	1	Mass Hunter 176	
007	Fully identified (SANCO/1257/2013)	30	5	confirmed on ToF, LC-MS/MS, GC-MS	LC	ToF	Q-TOF	Jet Stream	+ No	No	NA	Yes	Agilent 6530 accurate mass Q-ToF LC/MS	10	AcN		0.5	None	C18 3 Mass Hunter	
008	Fully identified (SANCO/1257/2013)				GC	MS/MS			Yes	Yes			13	AcN	No	Mechanical	None			
009	Fully identified (SANCO/1257/2013)	+/- 0.2 min	3 ions	GC	MS	Q	EI +	Yes	No	0.01	Yes	Bruker 450 Scion TQ	10	AcN	No	Mechanical	5	None	HP1 MS UI 5 Both	
010	Fully identified (SANCO/1257/2013)				GC	MS/MS	QQQ	EI +	Yes	Yes	0.010		Bruker SCION 10	AcN	No	Manual	2	DSPE	Zébron ZB 2 Bruker	
011	Fully identified (SANCO/1257/2013)	3	2 Transitions	LC	MS/MS	QQQ	EI +	Yes	Yes	0.005-0.1	No	Xevo TQS 10	AcN	No		2	None	C18 2.5 Masslynx		
013	Tentatively detected	10	2		GC	MS/MS	QQQ	EI +	Yes	Yes	0.005-0.1	Yes	TripleTOF 10	AcN	No		2	None	C18 2 PeakView	
014	Tentatively detected	5			LC	MS	TOF	ESI +	No	No	0.005-0.1	Yes	LCT PREMIER XE	15	Acetone/DCM/PE	No		1.5	None	DB5-MS 5 Mass Hunter ~500
015	Fully identified (SANCO/1257/2013)	2	10	Accurate mass	GC	MS/MS	QQQ	EI	Yes	Yes	0.01	Yes	Agilent 7890/Agilent 7000	10	Cyclohexane/EtOAc/Acetone	No	3	DSPE	HP5-MS 1 Mass Hunter	
016	Fully identified (SANCO/1257/2013)	-0.24			GC	MS/MS	QQQ	EI +	Yes	Yes	0.01	No	Agilent 7000	10	AcN	Yes	1	DSPE	HP5MSUI 10 Mass Hunter	
017	Fully identified (SANCO/1257/2013)	10	0.02		GC	MS	Q	EI +	Yes	Yes	0.01	Yes	Thermo DSQ II	10	AcN	Yes	2	QueCHERS	ZB-5ms 1 Xcalibur	
018	Fully identified (SANCO/1257/2013)				GC	MS/MS	QQQ		+	Yes	0.010	No	Agilent 7890B	10	AcN	No	1	SPF	HP5-MS 1 Mass Hunter	
020	Fully identified (SANCO/1257/2013)		2 Transitions	GC	MS/MS	QQQ	EI +	Yes	Yes	0.005	No	TSQ Quantum XLS Ultra	10	AcN	No	Mechanical	30	DSPE	RTX5-MS 1 TraceFinder	
023	Fully identified (SANCO/1257/2013)				GC	MS	T		No	No	Yes	Varian - 240MS	15	Acetone/DCM/PE	No	1	GPC	VF-5MS 3 Auto		

APPENDIX 3. Methods used by participants for detecting pesticides.

Terbufos											
Laboratory Code *NRL	How was the identification done?	RT Deviation (s)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	IonizationMode	Polarity	Routine Method?	Routine Scope?
024 Fully identified (SANCO/1/257/2013)	5	Match spectra with standard.	GC	MS TOF	QQQ	EI +	Yes	Yes	Yes	LOQ: 0.01	LOQ: 0.01
025 Fully identified (SANCO/1/257/2013)	2	0	2 Transitions	GC MS/MS	MS/MS	QQQ	El	Yes	Yes	0.010	No
026 Tentatively detected	0.01			GC NS/MS	MS/MS	QQQ	EI +	Yes	Yes	0.002	No
027 Fully identified (SANCO/1/257/2013)	0	2 Transitions	LC	MS/MS	QQQ	ESI +	Yes	Yes	Yes	0.010	No
028 Fully identified (SANCO/1/257/2013)	0.1	0	no	GC	MS Q	ESI	El +	Yes	Yes	0.01	No
029 Fully identified (SANCO/1/257/2013)		2 Transitions	LC	MS/MS	QQQ	ESI +	Yes	Yes	Yes	0.01	No
030 Fully identified (SANCO/1/257/2013)	1	5		GC	MS IT	ESI	El	Yes	Yes	0.01	Yes
031 Fully identified (SANCO/1/257/2013)	max 2.5%	1	2 Transitions	LC	MS/MS	QQQ	ESI +	Yes	Yes	0.01	No
032 Fully identified (SANCO/1/257/2013)				LC	MS/MS	QQQ	ESI	El +	Yes	Yes	0.01
035 Fully identified (SANCO/1/257/2013)	2.5%		2 Transitions	LC	MS/MS	QQQ	ESI +	Yes	Yes	0.01	No
036 Fully identified (SANCO/1/257/2013)				LC	MS/MS	QQQ	ESI +	Yes	Yes	0.01	No
037 Tentatively detected	<30	5	isotope	LC	MS	Orbitrap	ESI +	No	No	Not Established	Yes

APPENDIX 3. Methods used by participants for detecting pesticides.

Terburos											
Laboratory Code *NRL	How was the identification done?	RT Deviation (s)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Instrument Model	Sample Weight (g)	Injection Volume (μL)
038 Fully identified (SANCO/1257/2013)	<20	spectrum	GC	MS	TOF	EI +	Yes	0.05	Yes	Leco Pegasus IV	1% acetic acid
039 Fully identified (SANCO/1257/2013)	0.60	2 Transitions	LC	MS/MS	QQQQ	EI	+ Yes	0.01	No	4000	10
042 Fully identified (SANCO/1257/2013)	0.60	2 Transitions	LC	MS/MS	QQQQ	EI	+ Yes	0.005	No	Waters Quattro Premier XE	15 Acetone/DCM/PE
044 Fully identified (SANCO/1257/2013)	-1.8	2 Transitions	GC	MS/MS	QQQQ	EI +	Yes	0.01	No	Varian 1200 GC-MS	10 EIOAC
045 Fully identified (SANCO/1257/2013)	0.2	2 Transitions	GC	MS/MS	QQQQ	EI	+ Yes	0.02	Yes	AT5975 GC-MS	10 AcN/EIOAC
046 Fully identified (SANCO/1257/2013)	2	LC	MS/MS	QQQQ	EI	+ Yes	Yes	0.01	No	7000 Agilent 10	10 EIOAC
047 Fully identified (SANCO/1257/2013)	0.04	3 Transitions	GC	MS	IT	EI +	Yes	0.01	No	Agilent 6460	10 EIOAC
048 Fully identified (SANCO/1257/2013)	1	2 Transitions	LC	MS/MS	QQQQ	EI	+ Yes	0.01	Yes	Varian 4000 Saturn	10 ACN
049 Fully identified (SANCO/1257/2013)		LC	MS/MS	QQQQ	EI	+ Yes	Yes	0.01	Yes	Agilent 6490 LC/MS/MS	15 Acetone/DCM/PE
050 Fully identified (SANCO/1257/2013)		2 Transitions	GC	MS	IT	EI +	Yes	0.01	Yes	POLARIS Q	10 ACN
051 Fully identified (SANCO/1257/2013)	0.1	3 Transitions	GC	MS/MS	QQQQ	EI	+ Yes	0.003	No	Varian 3800 GC + 320-QTRAP	10 ACN
052 Fully identified (SANCO/1257/2013)	12	2 Transitions ratio 30%	LC	MS/MS	QQQQ	EI +	Yes	0.01	Yes	AB SCIEX 4000 QTRAP	10 ACN
053 Fully identified (SANCO/1257/2013)	0	3	GC	MS/MS	QQQQ	EI +	Yes	0.01	Yes	7000A	10 ACN
054 Fully identified (SANCO/1257/2013)	0.5	3 Transitions	GC	MS	Q	EI +	Yes	0.01	Yes	Agilent 6890M	10 ACN
055 Tentatively detected	none		LC	MS/MS	QQQQ	EI +	No	0.01	No	API 4000	10 ACN
056 Tentatively detected	1	1	GC	MS	Q	EI	Yes	0.01	Yes	Agilent 5973	10 ACN
057 Tentatively detected			GC	MS	Q	EI +	No	No	Yes	HP-5973	10 ACN
										Manual	2
										DSPE	SE54
										DS	

APPENDIX 3. Methods used by participants for detecting pesticides.

Laboratory Code *NRL	How was the identification done?	RT Deviation (s)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Detection Limit (SDL)? (mg/kg)	Instrument Model	Full Scan	Sample Weight (g)	Extraction Solvent	pH adjusted	Agitation Time (min)	Injection Volume (μL)	Compounds in library (pesticides Only)	Standard Solution Frequency	Terbufos			
058	Tentatively detected	15	1	target + qualifier	GC	MS	Q	EI	+	Yes	Yes	0.01 - 0.05	Yes	Agilent GC - MSD	10	AcN	Yes	Mechanical	10	PSA	DB5	10	ChemStation	>600	Every batch
060 [SANCO/1/257/2013]	Fully identified	-2.6			GC	MS	Q	EI	+	No	No		Yes	Agilent 7890A/5975C	10	AcN	Yes	Manual	2	DSPE	HP5-MS	20	AMDIS	~550	
062	Tentatively detected				GC	MS	Q	EI	+	Yes	No	0.01	Yes	Agilent GC-MSD	10	AcN	Yes	Manual	5	DSPE	HP5-MS	1	Auto	927	Weekly
064	Tentatively detected				GC	MS	TOF	EI	+	No	No		Yes	Pegasus 4D	15	Acetone/PE/DCM	No		1	None	TR5MS	1	Chroma TOF	76	
065 [SANCO/1/257/2013]	Fully identified	45		3 diagnostic ions	GC	MS	Q	EI	+	Yes	Yes	0.01	Yes	Shimadzu QP2010+	10	AcN	No		30	DSPE	VF-5-MS-30M*0.25*0.25μm	10	GC-MSsolution	112	Monthly
067 [SANCO/1/257/2013]	Fully identified		3 Transitions	GC	MS/MS	QQQ	EI		Yes	Yes	0.01	No	Agilent 7000	10	AcN	No	Mechanical	1	DSPE	DB-SMS UI	2	Mass Hunter	236	Daily	

ANNEX 1. List of Laboratories that participate in EUPT-FV-SM06.

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COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
AUSTRIA	AUSTRIAN AGENCY FOR HEALTH AND FOOD SAFETY (AGES), INSTITUTE FOR FOOD SAFETY INNSBRUCK, DEPARTMENT FOR PESTICIDE AND FOOD ANALYTICS (PLMA)	INNSBRUCK	YES
BELGIUM	FYTOLAB CVBA	ZWIJNAARDE	YES
BELGIUM	LOVAP	GEEL	YES
BELGIUM	SCIENTIFIC INSTITUTE OF PUBLIC HEALTH	BRUXELLES	YES
CHINA	KEY LABORATORY OF FOOD SAFETY RISK ASSESSMENT	BEIJING	YES
CHINA	SHANGHAI MUNICIPAL CENTER FOR DISEASE CONTROL AND PREVENTION	SHANGHAI	YES
CROATIA	FOOD CONTROL CENTER	ZAGREB	YES
CZECH REPUBLIC	CZECH AGRICULTURE AND FOOD INSPECTION AUTHORITY	PRAHA	YES
CZECH REPUBLIC	INSTITUTE OF CHEMICAL TECHNOLOGY FOOD ANALYSIS AND NUTRITION DEPT.	PRAGUE	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	ANSES - LSAL - UNITÉ PESTICIDES ET BIOTOXINES MARINES	MAISONS-ALFORT	YES
FRANCE	CERECO SUD	GARONS	YES
FRANCE	GIRPA	BEAUCOUZE	YES
FRANCE	IDAC	NANTES	YES
FRANCE	LABORATOIRE DEPARTEMENTAL DE LA SARTHE	LE MANS	YES
FRANCE	LABORATOIRE DU SCL DE MONTPELLIER	MONTPELLIER	YES
FRANCE	LABORATOIRE DU SCL MASSY	MASSY CEDEX	YES
GERMANY	BAYERISCHES LANDESAMT FUER GESUNDHEIT UND LEBENSMITTELSECHEIT	ERLANGEN	YES
GERMANY	CHEMISCHES UND VETERINÄRUNTERSUCHUNGSAKT OSTWESTFALEN-LIPPE (CVUA-OWL)	DETMOLD	YES
GERMANY	CHEMISCHES UND VETERINÄRUNTERSUCHUNGSAKT RHEIN-RUHR-WUPPER (CVUA-RRW)	KREFELD	YES
GERMANY	CVUA-MEL CHEMISCHES UND VETERINÄRUNTERSUCHUNGSAKT MÜNSTERLAND-EMSCHER-LIPPE	MÜNSTER	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	HAMBURG	YES
GERMANY	LABOR FRIEDEL GMBH	TEGERNHEIM	YES
GERMANY	NIEDERSAECHSISCHES LANDESAMT FUER VERBRAUCHERSCHUTZ UND LEBENSMITELSICHERHEIT, LEBENSMITTEL- UND VETERINAERINSTITUT OLDENBURG	OLDENBURG	YES
GREECE	PESTICIDE RESIDUES LABORATORY, D CHEMICAL DIVISION OF ATHENS, GENERAL CHEMICAL STATE LABORATORY	ATHENS	YES

ANNEX 1. List of Laboratories that participate in EUPT-FV-SM06.

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
HUNGARY	NATIONAL FOOD CHAIN SAFETY OFFICE, DPPSCA PESTICIDE RESIDUE ANALYTICAL LABORATORY, MISKOLC	MISKOLC	YES
HUNGARY	NFCSO PESTICIDE ANALYTICAL LABORATORY, VELENCE	VELENCE	YES
IRELAND	THE PESTICIDE CONTROL LABORATORY	CELBIDGE	YES
ITALIA	ISTITUTO ZOOPROFILATTICO SPERIMENTALE DELLA SICILIA	PALERMO	YES
ITALIA	LABORATORIO DI PREVENZIONE ASL MILANO	MILANO	YES
ITALY	A.R.P.A. VENETO - SERVIZIO LABORATORI VERONA	VERONA	YES
ITALY	ARPA EMILIA ROMAGNA, AREA FITOFARMACI	FERRARA	NO
ITALY	ARPA FVG LABORATORIO UNICO MULTISITO SEDE DI PORDENONE	PORDENONE	YES
ITALY	ISTITUTO ZOOPROFILATTICO SPERIMENTALE UMBRIA MARCHE LABORATORIO CONTAMINANTI AMBIENTALI	PERUGIA	YES
ITALY	LANDESAGENTUR FÜR UMWELT LABOR FÜR CHROMATOGRAPHIE	BOZEN	YES
LATVIA	INSITUTE OF FOOD SAFETY, ANIMAL HEALTH AND ENVIROMENT "BIOR"	RIGA	YES
ROMANIA	CENTRAL LABORATORY FOR PESTICIDES RESIDUES CONTROL	BUCHAREST	YES
ROMANIA	REGIONAL LABORATORY FOR DETERMINATION OF PESTICIDE RESIDUES IN PLANT AND PLANT PRODUCTS MURES	TARGU MURES	YES
ROMANIA	SANITARY VETERINARY AND FOOD SAFETY DIRECTORATE	BUCHAREST	YES
SERBIA	CENTER FOR FOOD ANALYSIS	BELGRADE	YES
SLOVENIA	NATIONAL LABORATORY OF HEALTH, ENVIRONMENT AND FOODSTUFFS (DEPARTMENT FOT CHEMICAL ANALYSIS MERIBOR)	MARIBOR	YES
SPAIN	AGRICULTURAL AND PHYTOPATHOLOGICAL LABORATORY OF GALICIA	ABEGONDO A CORUÑA	YES
SPAIN	ANALYTICA ALIMENTARIA GMBH - SUCURSAL EN ESPAÑA	SPAIN	YES
SPAIN	LABORATORIO AGROALIMENTARIO DE EXTREMADURA	CÁCERES	YES
SPAIN	LABORATORIO AGROALIMENTARIO DE GRANADA	ATARFE	YES
SPAIN	LABORATORIO AGROALIMENTARIO DE VALENCIA	BURJASSOT	YES
SPAIN	LABORATORIO AGROALIMENTARIO Y DE SANIDAD ANIMAL	EL PALMAR MURCIA	YES
SPAIN	LABORATORIO DE PRODUCCIÓN Y SANIDAD VEGETAL DE ALMERÍA	LA MOJONERA, ALMERÍA	YES
SPAIN	LABORATORIO DE SALUD PUBLICA DE CUENCA	CUENCA	YES
SPAIN	LABORATORIO PRODUCCION Y SANIDAD VEGETAL JAEN. AGAPA	MENGIBAR JAEN	YES
SPAIN	LABORATORIO QUÍMICO MICROBIOLÓGICO, S.A	MURCIA	YES
SPAIN	LABORATORIOS ECOSUR, S.A.	LORQUI MURCIA	YES
SPAIN	LABORATORY OF BARCELONA PUBLIC HEALTH AGENCY	BARCELONA	YES
SWEDEN	EUROFINS FOOD & AGRO TESTING SWEDEN AB	LIDKÖPING	YES
SWEDEN	SWEDISH NATIONAL FOOD AGENCY, SCIENCE DEPARTMENT CHEMISTRY DIVISION 1	UPPSALA	
SWITZERLAND	KANTONALES LABOR ZÜRICH	ZURICH	YES
SWITZERLAND	SERVICE DE LA CONSOMMATION ET DES AFFAIRES VETERINAIRES (SCAV)	GENEVE	YES

ANNEX 1. List of Laboratories that participate in EUPT-FV-SM06.

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
THE NETHERLANDS	NVWA - NETHERLANDS FOOD AND CONSUMER PRODUCT SAFETY AUTHORITY	WAGENINGEN	YES
THE NETHERLANDS	RIKILT - INSTITUTE OF FOOD SAFETY	WAGENINGEN	YES
TURKEY	OZEL MSM GIDA KONTROL LABORATUVARI VE DAN. HIZ. TIC. A.S.	MERSIN	YES
UK	EUROFINS FOOD TESTING UK LTD	WOLVERHAMPTON	YES
UK	SASA (SCIENCE AND ADVICE FOR SCOTTISH AGRICULTURE)	EDINBURGH	YES
UK	THE FOOD AND ENVIRONMENT RESEARCH AGENCY (FERA)	YORK	YES

ANNEX 2. Specific Protocol.



EUPT-FV-SM06 SPECIFIC PROTOCOL
European Union Proficiency Test for Pesticide Residues in Fruits and Vegetables
Screening Multiresidue Methods (2014)

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 EUR-LIN 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 item

This proficiency test is based on the pesticide-residue analysis of pepper.

The pesticide treatments will be carried out post-harvest using either commercial formulation in micro-spray solutions or using standard solutions. The test item 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 item will be chosen randomly and analysed to check for homogeneity.

The test item 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 item (firstly, when the test items 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 item.

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 EUR-LIN 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-FV16. For those who are not EUPT-FV16 participants, please see **Cost for shipment of the test item** for further details. The payment procedure must have started before 24th February 2014. 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 item (and not before), they must enter the restricted area and submit **Form 0 - Test Item Receipt** to inform the Organiser that they have accepted the test item. If no test item has been received by 27th 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 EUR-LIN 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.

ANNEX 2. Specific Protocol.

Amount of Test Item

Participants will receive:

- Approximately 300 g of pepper test item treated with pesticides.
- Approximately 300 g of 'blank' pepper test item.

Shipment of Test Items

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

The shipment of the test items will start on 24th February 2014. 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 Item Handling

Once received, the test item should be stored deeply frozen (-18°C or less) prior to analysis to avoid any possible deterioration/spoilage. The test item 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 Item Receipt

Once the laboratory has received the test item, the Organiser must be notified using Form 0 in the restricted area; filling in the date of receipt, the condition of the test item, and acknowledging its acceptance. If the laboratory does not inform the Organiser by 28th February 2014 (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 item 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.	15 th November 2013
Submission of Application Form by invited laboratories.	23 rd Dec 2013 – 31 st Jan 2014
Sample distribution.	24 th February 2014
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.	End of April 2014
Final Report distributed to the Laboratories.	November 2014

Cost for shipment of the test item

Only those laboratories not participating in EUPT-FV15 will have to pay the following fee for sample shipment: **EU and EFTA** laboratories will be charged **175€** and other laboratories will be charged **250 €**. 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 Almería

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:

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

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

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Mr. Octavio Malato Rodríguez	EURL-FV	omalato@ual.es	+34 950214423
Ms. Noelia Belmonte	EURL-FV	nbel143@ual.es	+34 950015645
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Ms. Samanta Uclés.	EURL-FV	samantaucles@ual.es	+34 950015645
Ms. Ana Uclés.	EURL-FV	anauclesm@ual.es	+34 950015645
Ms. Sonia Herrera.	EURL-FV	shl183@ual.es	+34 950015645
Mr. Łukasz Rajski	EURL-FV	154303@edu.p.lodz.pl	+34 950015645

Quality Control Group

Dr. Antonio Valverde, Senior Chemist	University of Almería, Spain.
Mr. Stewart Reynolds, Senior Chemist	FERA, York, United Kingdom.

Statistical Group

Dr. Carmelo Rodriguez, Senior Mathematician	University of Almeria, Spain.
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Advisory Group

Dr. Michelangelo Anastassiades, Senior Chemist	CVUA, Stuttgart, Germany.
Mr. Richard Fussell, Senior Chemist	FERA, York, United Kingdom.
Dr. Miguel Gamón, Senior Chemist	Laboratorio Agroalimentario, Valencia, Spain.
Dr. Magnus Jezussek, Senior Chemist	LGL, Erlangen, Germany.
Dr. André de Kok, Senior Chemist	NVWA, Wageningen, The Netherlands.
Mr. Ralf Lippold, Senior Chemist	CVUA, Freiburg, Germany.
Dr. Sonja Masselter, Senior Chemist	AGES, Institute for Food Safety, Innsbruck, Austria.
Dr. Tuija Pihlström, Senior Chemist	NFA, Uppsala, Sweden.
Dr. Mette Erecius Poulsen, Senior Chemist	NFI, Copenhagen, Denmark.
Dr. Darinka Štajnbaher, Senior Chemist	IPH, Maribor, Slovenia.