

EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUIT AND VEGETABLES. SCREENING METHODS 05 (EUPT-FV-SM-05)

Pesticide Residues in Potato Homogenate

Final Report (29th November 2013)

Organiser:

Dr. Amadeo R. Fernández-Alba

Co-Head of EURL-FV

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

Phone: +34 950015034; Fax: +34 950015008

E-mail: amadeo@ual.es

www.eurl-pesticides.eu

Organising team at the University of Almería, Spain:

Mr. Octavio Malato, Chemist.

Ms. Carmen Ferrer, Chemist.

Ms. Noelia Belmonte, Chemist.

Ms. Ana Lozano, Chemist.

Ms. M^o del Mar Gómez, Chemist.

Ms. Samanta Uclés, Chemist.

Ms. Ana Uclés, Chemist.

Scientific Committee

Mr. Stewart Reynolds, Senior Chemist (QCG)

Dr. Antonio Valverde, Senior Chemist (QCG),

Dr. Michelangelo Anastassiades, Senior Chemist (AG),

Mr. Richard Fussell (AG),

Dr. Miguel Gamón, Senior Chemist (AG),

Dr. Magnus Jezussek, Senior Chemist (AG),

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

Mr. Ralf Lippold, Senior Chemist (AG),

Dr. Sonja Masselter, Senior Chemist (AG),

Dr. Tuija Pihlström, Senior Chemist (AG),

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

Dr. Carmelo Rodriguez, Senior Mathematician (AG),

Dr. Darinka Štajnbaher, Senior Chemist (AG),

FERA, York, United Kingdom.

University of Almería, Spain.

CVUA, Stuttgart, Germany.

FERA, York, United Kingdom.

Lab. Agroalimentario, Valencia,
Spain.

LGL, Erlangen, Germany.

NVWA, Wageningen,
The Netherlands.

CVUA, Freiburg, Germany.

AGES, Innsbruck, Austria.

NFA, Uppsala, Sweden.

DTU, Copenhagen, Denmark.

University of Almeria, Spain.

IPH, Maribor, Slovenia.

QCG: Quality Control Group

AG: Advisory Group

*Authorized by: Dr. Amadeo R. Fernández-Alba
Co-Head of EURL-FV*

CONTENTS

1. INTRODUCTION.	1
2. TEST ITEMS.	2
2.1 Analytical methods.	
2.2 Preparation of the test item.	
2.3 Preparation of "blank" test item.	
2.4 Homogeneity and stability tests.	
2.5 Distribution of test item and protocol of participants.	
3. STATISTICAL METHODS.	5
3.1 Type of Results Reported.	
4. RESULTS.	6
4.1 Summary of reported results	
4.2 Concentration levels.	
4.3 Assessment of laboratory performance.	
5. CONCLUSIONS.	12
6. SUGGESTIONS FOR FUTURE WORK.	13
7. REFERENCES.	14
8. ACKNOWLEDGEMENTS.	15
APPENDIX 1.Results.	17
APPENDIX 2.Graphical Representations.	19
APPENDIX 3. Methods used by participants for detecting pesticides. (available only as a pdf document uploaded to the EUPT-SM05 webpage, not in the printed version).	23
ANNEX 1. List of laboratories that participate in EUPT-FV-SM-05.	103
ANNEX 2. Specific Protocol. (available only as a pdf document uploaded to the EUPT-SM05 webpage, not in the printed version).	105

EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUITS AND VEGETABLES. SCREENING METHODS 05

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 fifth 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, official laboratories have a significant number of mandatory PTs annually, given that the EURL-FV already organises the PT for quantitative multi-residue pesticide analysis (EUPT-FV15) 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.

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

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

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

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

Mass spectrometry plays an essential role in the everyday work carried out by laboratories. Technological improvements in modern MS systems offer new possibilities for greatly increasing the scope of MRM (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.

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

This EUPT-FV-SM05 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.

2. TEST ITEMS

2.1 Analytical methods

The two analytical methods described briefly below were performed in order to conduct the homogeneity and stability tests.

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

2.2 Preparation of the treated test item.

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

The pesticides used to spike the potato 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-SM05 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 used to spike the potato 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 potatoes were mixed in a constantly-spinning container until a homogeneous item was obtained. 300 g portions of the well-mixed homogeneate 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 used to spike the sample.

Spiked Pesticides (in bold pesticides with concentration <100 ppb)			
Aldicarb Sulfone	Dieldrin	Metribuzin	Prosulfocarb
Aldicarb Sulfoxide	Diuron	Molinate	Quinoclamine
Atrazine	Fluazinam	Oxamyl	Thiodicarb
Beta-Cyfluthrin	Flufenacet	Picloram	Triflumizole
Bromoxynil	Metamitron	Picolinafen	
Clomazone	Methomyl #	Propamocarb	

Thiodicarb degradation product (not spiked)

2.3 Preparation of "blank" test item.

The potatoes 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.

2.4 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 detect the presence of the spiked pesticides, which were consistently confirmed to be above the Organiser's LODs.

To confirm the homogeneity of the test item sent, ten spiked test samples were randomly chosen from those stored in the freezer and analysed in duplicate so as to check for the presence of the pesticides.

The injection sequence of the 10 analyses by GC and LC was determined from a table of randomly-generated numbers. The relative standard deviation (RSD) of these 20 analyses needed to be at 15%, or below, to consider the 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.	004 a	004 b	007 a	007 b	023 a	023 b	064 a	064 b	066 a	066 b	076 a	076 b	101 a	101 b	122 a	122 b	144 a	144 b	148 a	148 b	A. Cc (mg/kg)	RSD (%)	
Aldicarb sulfone	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.210	14.7
Aldicarb Sulfoxide	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.426	9.0
Atrazine	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.301	1.2
Beta-Cyfluthrin	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.201	9.1
Bromoxynil	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.386	7.8
Clomazone	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.083	6.1
Dieldrin	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.400	6.4
Diuron	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.935	4.5
Fluazinam	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.030	13.6
Flufenacet	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.126	7.3
Metamitron	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.184	6.8
Methomyl#	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.037	9.4
Metribuzin	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.225	4.4
Molinate	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.060	12.3
Oxamyl	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.092	7.4
Picloram	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.098	13.1
Picolinafen	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.488	13.7
Propamocarb	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.082	7.7
Prosulfocarb	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.247	9.3
Quinoclamine	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.073	9.6
Thiodicarb#	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.068	10.1
Triflumizole	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.022	14.9

D: Detected A. Cc: Average Concentration RSD: Relative Standard Deviation

Thiodicarb degradation product (not spiked)

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

The three occasions were:

- Day 1: the day of the sample shipment on 21st January 2013.
- Day 2: on 23rd January 2013 after reproducing the sample shipment conditions.
- Day 3: soon after the deadline for reporting results on 25th January 2013.

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

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

Table 2.3.2 Stability tests performed.

Stability tests						
Test item Number	142 Day 1	142 Day 1	025 Day 2	025 Day 2	107 Day 3	107 Day 3
Aldicarb sulfone	D	D	D	D	D	D
Aldicarb Sulfoxide	D	D	D	D	D	D
Atrazine	D	D	D	D	D	D
Beta-Cyfluthrin	D	D	D	D	D	D
Bromoxynil	D	D	D	D	D	D
Clomazone	D	D	D	D	D	D
Dieldrin	D	D	D	D	D	D
Diuron	D	D	D	D	D	D
Fluazinam	D	D	D	D	D	D
Flufenacet	D	D	D	D	D	D
Metamitron	D	D	D	D	D	D
Methomyl#	D	D	D	D	D	D
Metribuzin	D	D	D	D	D	D
Molinate	D	D	D	D	D	D
Oxamyl	D	D	D	D	D	D
Picloram	D	D	D	D	D	D
Picolinafen	D	D	D	D	D	D
Propamocarb	D	D	D	D	D	D
Prosulfocarb	D	D	D	D	D	D
Quinoclamine	D	D	D	D	D	D
Thiodicarb	D	D	D	D	D	D
Triflumizole	D	D	D	D	D	D

D: Detected

Thiodicarb degradation product (not spiked)

2.5 Distribution of test items and protocol to participants

Approximately 300 g of treated potato homogenate together with another 300 g of 'blank' potato homogenate were shipped to participants on 21st January 2013. 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 spiked 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-SM05 web page, designed especially for this Proficiency Test. This information was also sent by e-mail to all participant laboratories. The Application Form was uploaded onto this same web site together with Form 0 (Sample Receipt) and Form 1 (Results). These allowed the evaluation of the mass-spectrometric screening methods that each of the participants used.

3. STATISTICAL METHODS

3.1 Type of results reported

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

3.1.1 Other Reported Pesticides

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

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

3.1.2 Not-Reported Pesticides

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

4. RESULTS

4.1 Summary of reported results

Fifty-eight laboratories agreed to participate in this fifth proficiency test on screening methods. Fifty-seven laboratories submitted results on time and only one submitted results after the deadline, so those data are not included in this report. All results reported by the participants are given in Appendix 1. Graphical representations of the results reported are shown in Appendix 2. Details of the screening methods used are provided in Appendix 3 (available only as a pdf document uploaded to the EUPT-SM05 webpage, not in the printed version). The laboratories that agreed to participate are listed in Annex 1.

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

Table 4.1 Summary of Results Reported.

Pesticide	No. of Reported	% of Reported*	No. of Not Reported	% of Not Reported*
Aldicarb sulfone	50	88	7	12
Aldicarb Sulfoxide	50	88	7	12
Atrazine	53	93	4	7
Beta-Cyfluthrin	51	89	6	11
Bromoxynil	40	70	17	30
Clomazone	49	86	8	14
Dieldrin	53	93	4	7
Diuron	46	81	11	19
Fluazinam	31	54	26	46
Flufenacet	45	79	12	21
Metamitron	39	68	18	32
Methomyl#	50	88	7	12
Metribuzin	51	89	6	11
Molinate	37	65	20	35
Oxamyl	52	91	5	9
Picloram	10	18	47	82
Picolinafen	39	68	18	32
Propamocarb	47	82	10	18
Prosulfocarb	46	81	11	19
Quinoclamine	22	39	35	61
Thiodicarb	50	88	7	12
Triflumizole	42	74	15	26

* The % of laboratories is calculated based on the total number of laboratories submitting results (57).

Thiodicarb degradation product (not spiked)

4.1.1 Other Reported Pesticides

Many laboratories reported additional pesticides to those used to spike 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
Lab004*	Asulam
Lab006*	Flutolanil
Lab009	Daminozide
Lab010*	Fenuron, Hymexazol
Lab011	Isoxaflutole
Lab012*	2-Phenylphenole, DDT p,p', Diflubenzuron, p,p-DDE
Lab014*	Fluometuron, Pyroquilon
Lab015*	Isoproturon, Thiacloprid
Lab016*	Chloroxynil
Lab017	Byphenyl
Lab019	Isoprocarb, Spinosad
Lab021	Chlorpropham, Cypermethrin, Fenuron, Thiacloprid
Lab023	Dodine, Ethoxyquin
Lab024*	Dikegulac
Lab025*	Ethoxyquin
Lab037	Asulam
Lab040	p,p-DDE
Lab044	Chloroxuron, Ethoxyquin
Lab047	Dazomet
Lab053	Benzalkonium BAC-12, BAC-14, Didecyldimethyl ammonium (DDAC), Monuron
Lab054	Bufencarb
Lab055*	Flumethrin, Prothiophos
Lab056	Cadusafos, Pencycuron, Prothiophos
Lab057	Pirimicarb-desmethyl
Lab058*	Isoprocarb, Oryzalin, Sebuthylazine

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

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

4.1.2 Not-Reported Pesticides

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

4.2 Concentration levels.

Twenty-one pesticides were used to spike the potato test item at different levels, in the range between 0.013 mg/kg and 0.935 mg/kg according to the homogeneity table 2.3.1.

The test item was spiked/treated with thiodicarb, which degrades to methomyl. The test item was not spiked/treated with methomyl.

Propazine, simazine and thiabendazole were not intentionally used to spike/treat the test item, but they were detected by the organisers at concentrations below 0.01 mg/kg due to impurities of the commercial formulations.

4.3 Assessment of laboratory performance.

No z-score values were carried out for laboratories to assess their performance as no numerical results were reported by the participants. However, classification was considered important, based on the number of detected results each laboratory reported and also on which methods they used. Table 4.3.1 classifies the laboratories according to the number of spiked pesticides reported.

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

Laboratory Code	No of Reported Pesticides	% of Reported Pesticides	Other Reported Pesticides	
			Not Confirmed by the Organiser	Confirmed by the Organiser
Lab002	22	100		
Lab033*	22	100		
Lab042	22	100		
Lab053	22	100	4	0
Lab005*	21	95		
Lab030	21	95		
Lab034*	21	95		
Lab038	21	95		
Lab039	21	95		
Lab041	21	95		
Lab043	21	95		
Lab011	21	95	1	0
Lab056	21	95	3	0
Lab058*	21	95	3	0
Lab013	20	91		
Lab026	20	91		
Lab046*	20	91		
Lab017	20	91	1	0
Lab029	19	86		
Lab035	19	86		
Lab049	19	86		
Lab059	19	86		
Lab016*	19	86	1	0
Lab024*	19	86	1	0
Lab037	19	86	1	0
Lab047	19	86	1	0
Lab014*	19	86	2	0
Lab001	18	82		
Lab020*	18	82		
Lab057	18	82	1	0
Lab023	18	82	2	0
Lab055*	18	82	2	0

Laboratory Code	No of Reported Pesticides	% of Reported Pesticides	Other Reported Pesticides	
			Not Confirmed by the Organiser	Confirmed by the Organiser
Lab021	18	82	4	0
Lab018	17	77		
Lab044	17	77	2	0
Lab008	16	73		
Lab004*	16	73	1	0
Lab040	16	73	1	0
Lab022	15	68		
Lab031	15	68		
Lab006*	15	68	1	0
Lab009	15	68	1	0
Lab010*	15	68	2	0
Lab007	14	64		
Lab019	14	64	2	0
Lab061	13	59		
Lab012*	13	59	4	0
Lab048	12	55		
Lab025*	12	55	1	0
Lab054	12	55	1	0
Lab036	11	50		
Lab015*	11	50	2	0
Lab060	9	41		
Lab050	7	32		
Lab051	5	23		
Lab028	4	18		
Lab032	2	9		
Lab003*	Results submitted after deadline			

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

The methods used by the laboratories, the chromatographic techniques, detectors, instrumentation, etc... are detailed in Appendix 3 (available only as a pdf document uploaded to the EUPT-SM05 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
Aldicarb sulfone	50	0	0	50	11
Aldicarb sulfoxide	53	0	0	53	11
Atrazine	56	23	11	33	12
Beta-Cyfluthrin	55	52	19	3	2
Bromoxynil	42	10	8	32	7
Clomazone	52	24	13	28	7
Dieldrin	54	53	22	1	0
Diuron	48	3	2	45	10
Fluazinam	32	8	3	24	4

Pesticide	Total no. of Reports	GC	Full-scan GC	LC	Full-scan LC
Flufenacet	48	14	8	34	10
Metamitron	41	5	3	36	7
Methomyl#	51	1	0	50	10
Metribuzin	54	27	11	27	9
Molinate	40	22	16	18	5
Oxamyl	57	1	0	56	11
Picloram	10	3	1	7	1
Picolinafen	41	22	13	19	5
Propamocarb	49	1	1	48	9
Prosulfocarb	49	15	8	34	8
Quinoclamine	24	10	10	14	3
Thiodicarb	51	0	0	51	11
Triflumizole	45	19	8	26	7

Thiodicarb degradation product (not spiked)

Note: The number of reports for each of the pesticides could be different to the reports shown in Table 4.1 because a particular laboratory might analyze each of the pesticides with more than one technique.

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

Table 4.3.3 shows the number and percentage of the pesticides used by the Organiser to spike the sample and methomyl, 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	18	82
Lab002	22	100
Lab003*	0	0
Lab004*	16	73
Lab005*	21	95
Lab006*	15	68
Lab007	14	64
Lab008	16	73
Lab009	15	68
Lab010*	15	68
Lab011	21	95
Lab012*	13	59
Lab013	20	91
Lab014*	19	86
Lab015*	11	50
Lab016*	19	86
Lab017	20	91
Lab018	17	77
Lab019	14	64

Laboratory Code	Number of Present Pesticides Reported	% of Present Pesticides Reported
Lab020*	18	82
Lab021	18	82
Lab022	15	68
Lab023	18	82
Lab024*	19	86
Lab025*	12	55
Lab026	20	91
Lab028	4	18
Lab029	19	86
Lab030	21	95
Lab031	15	68
Lab032	2	9
Lab033*	22	100
Lab034*	21	95
Lab035	19	86
Lab036	11	50
Lab037	19	86
Lab038	21	95
Lab039	21	95
Lab040	16	73
Lab041	21	95
Lab042	22	100
Lab043	21	95
Lab044	17	77
Lab046*	20	91
Lab047	19	86
Lab048	12	55
Lab049	19	86
Lab050	7	32
Lab051	5	23
Lab053	22	100
Lab054	12	55
Lab055*	18	82
Lab056	21	95
Lab057	18	82
Lab058*	21	95
Lab059	19	86
Lab060	9	41
Lab061	13	59

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

5. CONCLUSIONS

Fifty-eight laboratories applied to participate in this test and fifty-seven laboratories submitted results on time, last one submitted results after the deadline and its results are not present in this report. Seventeen of the laboratories which applied were National Reference Laboratories for Fruit and Vegetables (marked with an asterisk on the graphs and tables) representing twenty Member States. In addition to these, 1 EFTA country (Switzerland) and two non-EU/EFTA countries (Latvia and China) 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 1002 detections, 317 were made using full-scan, meaning 32% of detections (160 by LC techniques and 157 by GC techniques).

Four of the 58 laboratories were able to detect all 22 pesticides in the spiked potato test item (21 spiked plus methomyl). Five laboratories failed to detect less than 50% of the pesticides present. And only one laboratory sent its results after the deadline.

Sixty-seven percent of the laboratories (38 laboratories) that reported results were able to find more than 70% of the pesticides used to spike the sample.

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

This fifth 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 fifth test are most encouraging, but also indicate the need for continued evaluation of screening methods. Therefore, further proficiency tests will be organised to provide support to those laboratories using screening methods in order to extend their use and improve their reliability. These methods will be used more and more as screens/filters, to make routine laboratory work easier and faster. The need for screening method validation has been recognised and guidelines for such validation have been prepared and included in the updated version of the SANCO Document 12495/2011 ("Method validation and quality control procedures for pesticide residue analysis in food and feed") with the new title: Analytical Quality Control and Method Validation Procedures for Pesticide Residues Analysis in Food and Feed (SANCO/12571/2013).

Next year, pepper 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

- Malato O., Lozano, A., Mezcua M., Agüera, A., and Fernandez-Alba A. R. Benefits and pitfalls of the application of screening methods for the analysis of pesticide residues in fruits and vegetables. *Journal of Chromatography A*, 2011, 1218(42), 7615-7626.
- Mezcua M., Malato O., Martinez-Uroz M. A., Lozano, A., Agüera, A., and Fernandez-Alba A. R. Evaluation of Relevant Time-of-Flight-MS Parameters Used in HPLC/MS Full-Scan Screening Methods for Pesticides Residues. *Journal of AOAC Int.*, 2011, 94 (6), 1674-1684.
- Mezcua M., Martinez-Uroz M. A., Wylie P. L. and Fernandez-Alba A.R. Simultaneous screening and target analytical approach by GC-q-MS for pesticide residues in fruits and vegetables. *Journal of AOAC Int.*, 2009, 92 (6).
- Mezcua M., Malato O., Garcia-Reyes J. F., Molina-Diaz A., and Fernandez-Alba A. R. Accurate-Mass Databases for Comprehensive Screening of Pesticide Residues in Food by Fast Liquid Chromatography Time-of-Flight Mass Spectrometry. *Anal. Chem.*; 2009, 81, 913–929.
- Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed, European Commission, Document No. SANCO/12495/2011. Supersedes Document No. SANCO/10684/2009
- ISO/IEC 17043:2010 Conformity assessment - General requirements for proficiency testing.
- Analytical Quality Control and Method Validation Procedures for Pesticide Residues Analysis in Food and Feed (SANCO/12571/2013. Supersedes Document No. SANCO/12495/2011.

8. ACKNOWLEDGEMENTS

The Organiser is grateful to the European Commission for funding this 5th European Proficiency Test for Screening Methods in Fruit and Vegetables.

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

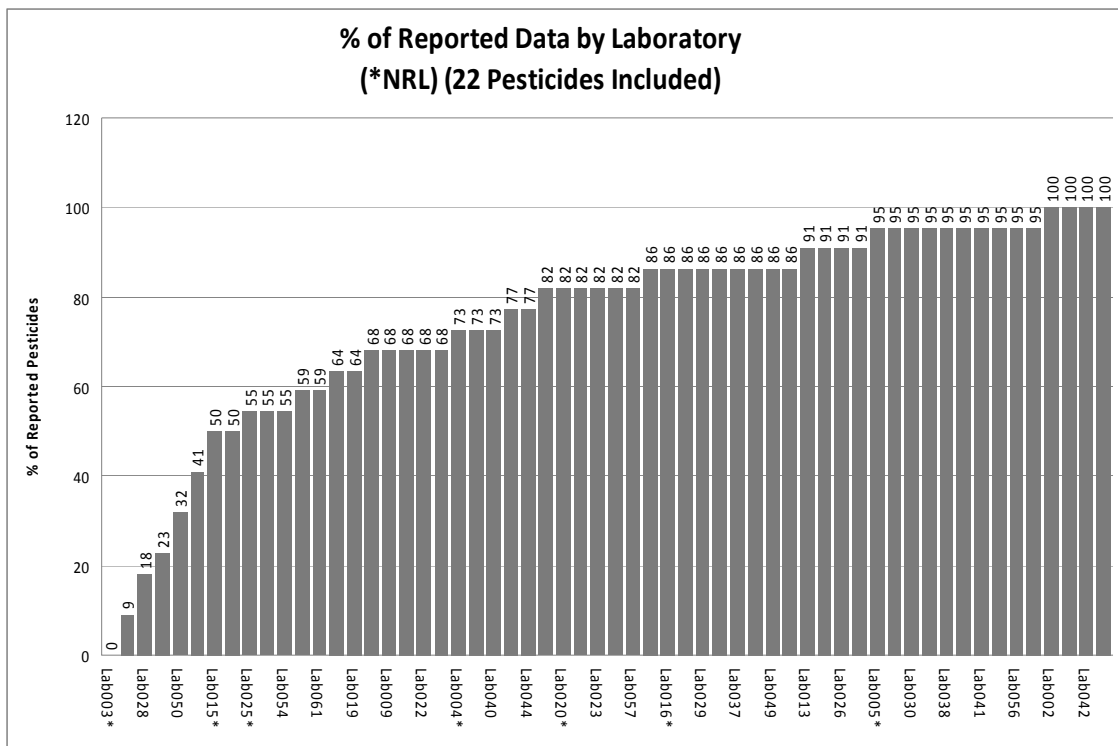
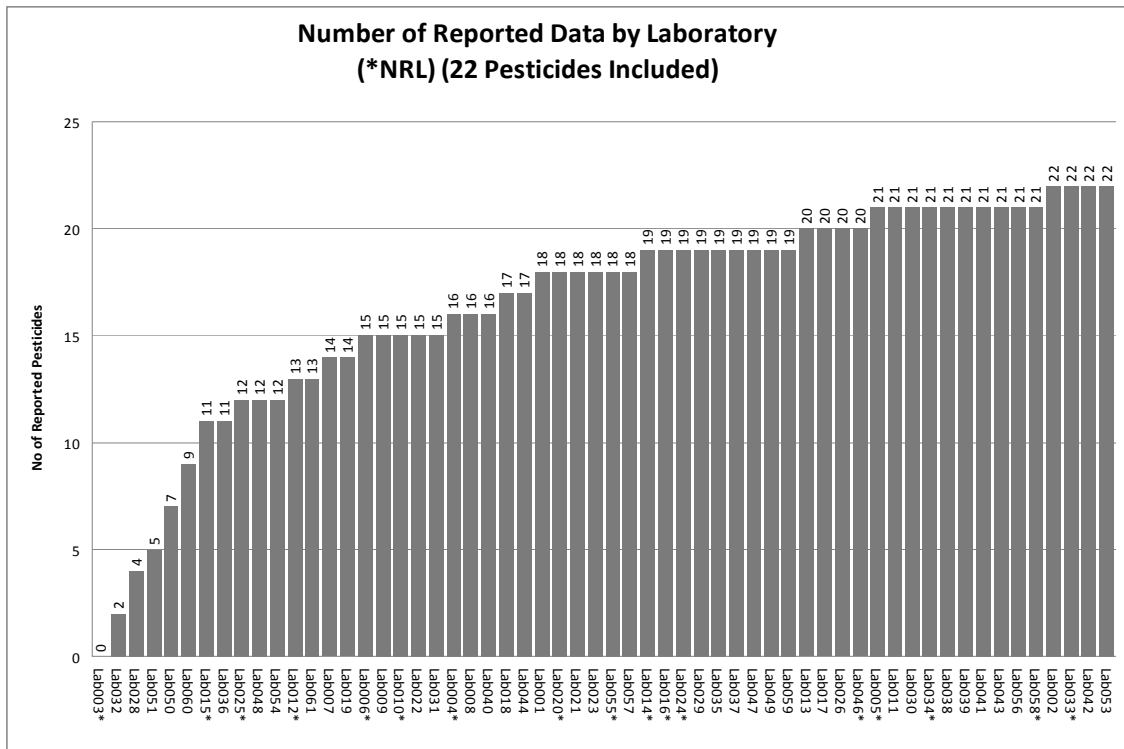
The Organiser wishes to give a special thank-you to Almeria University for the use of their facilities.

APPENDIX 1. Results

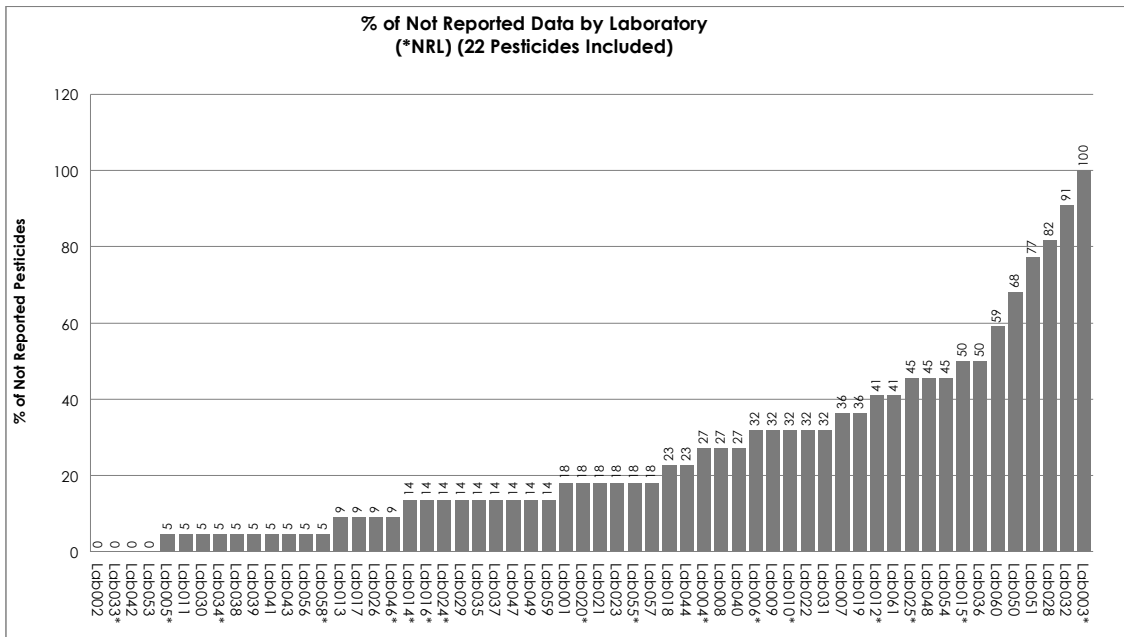
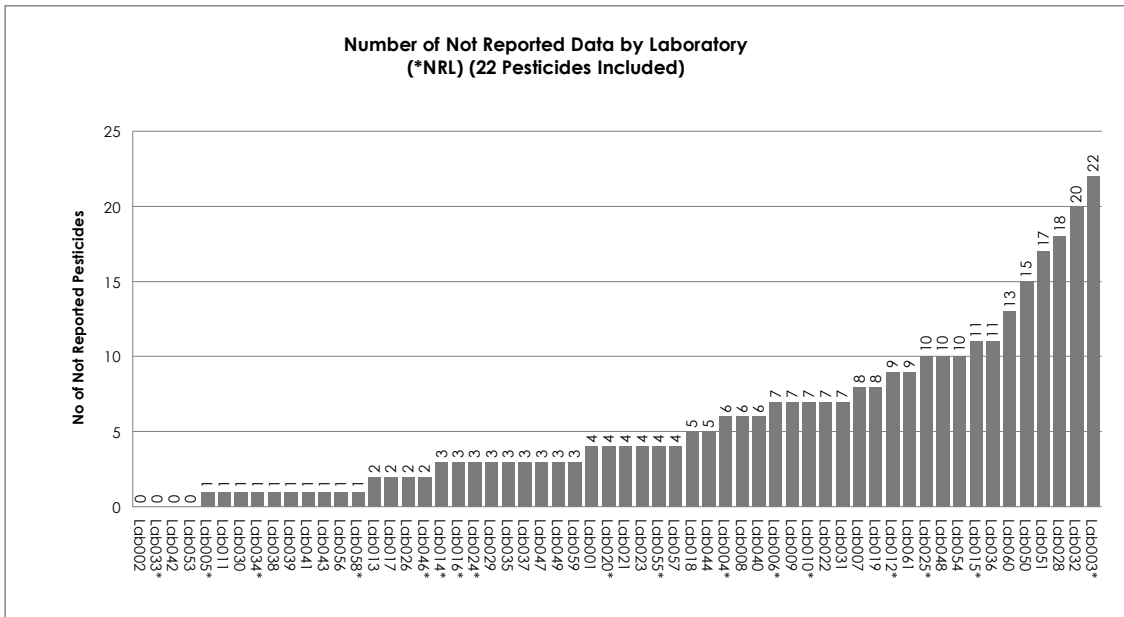
Laboratory Code Total No of Reporting Laboratories = 58	Evaluated Pesticides (22) *(pesticides with concentration <100 ppb)																				Reported Pesticides by Laboratory	% of Reported Pesticides by Laboratory		
	Aldicarb sulfone	Aldicarb sulfoxide	Altrazine	Beta-Cyfluthrin	Bromoxynil	*Clomazone	Dieldrin	Diuron	*Fluzinam	Flufenacet	Metamitron	Methomyl#	Meitibuzin	*Melinate	*Oxamyl	*Picolram	Picolinaten	*Propamocarb	Prosulfacarb	*Quinoclamine			Thiodicarb#	*Triflumizole
Lab001	R	R	R	R	R	R	R	R		R	R	R	R	R	R			R	R	R	R	R	18	82
Lab002	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	22	100
Lab003*																							0	0
Lab004*	R	R				R	R	R		R	R	R	R		R		R	R	R	R	R	R	16	73
Lab005*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	21	95
Lab006*	R	R	R	R		R	R		R		R	R	R		R		R	R	R		R		15	68
Lab007	R	R		R			R		R	R		R	R	R			R	R	R	R	R	R	14	64
Lab008	R	R	R	R	R	R	R	R				R	R	R			R	R	R	R	R	R	16	73
Lab009	R	R	R		R	R		R	R	R		R		R		R	R	R		R			15	68
Lab010*		R	R	R	R	R	R	R		R		R	R	R			R		R				15	68
Lab011	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R		R	R	21	95
Lab012*	R	R	R	R		R	R		R			R		R		R					R	R	13	59
Lab013	R	R	R	R	R	R	R	R	R	R	R	R		R			R	R	R	R	R	R	20	91
Lab014*	R	R	R	R	R	R	R	R		R	R	R		R	R		R	R	R	R	R	R	19	86
Lab015*	R	R	R	R			R	R				R	R		R			R			R		11	50
Lab016*	R	R	R	R	R	R	R	R	R	R	R	R	R		R		R	R	R		R	R	19	86
Lab017	R	R	R	R	R	R	R	R		R	R	R	R	R			R	R	R	R	R	R	20	91
Lab018		R	R	R	R	R	R	R	R	R	R	R	R	R				R	R	R	R	R	17	77
Lab019	R	R	R	R		R		R		R	R		R		R		R		R		R	R	14	64
Lab020*	R	R	R	R	R	R	R	R	R	R	R	R	R		R			R	R		R	R	18	82
Lab021	R		R	R		R	R	R		R	R	R	R	R			R	R	R	R	R	R	18	82
Lab022	R	R	R	R		R	R	R		R		R	R	R			R	R			R		15	68
Lab023	R	R	R	R		R	R	R	R	R	R	R	R		R		R	R	R	R	R	R	18	82
Lab024*	R	R	R	R	R	R	R	R		R	R	R	R	R			R	R	R		R	R	19	86
Lab025*	R	R	R		R		R	R					R	R	R			R	R		R		12	55
Lab026	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R			R	20	91
Lab028			R	R									R	R									4	18
Lab029	R	R	R	R	R	R	R	R			R	R	R	R	R		R	R	R	R	R	R	19	86
Lab030	R	R	R	R	R	R	R	R	R	R	R	R	R	R			R	R	R	R	R	R	21	95
Lab031	R	R	R	R		R	R	R		R		R	R	R			R	R			R		15	68
Lab032			R				R																2	9
Lab033*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	22	100
Lab034*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	21	95
Lab035	R	R	R	R	R	R	R	R	R	R	R	R	R		R		R	R	R		R	R	19	86
Lab036			R	R	R	R	R	R		R	R	R	R						R				11	50
Lab037	R	R	R	R	R	R	R	R		R	R	R	R	R	R		R	R	R	R	R	R	19	86
Lab038	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R		R	R	21	95
Lab039	R	R	R	R	R	R	R	R	R	R	R	R	R	R			R	R	R	R	R	R	21	95
Lab040	R	R	R	R		R	R	R		R	R	R	R	R			R	R	R	R	R	R	16	73
Lab041	R	R	R	R	R	R	R	R	R	R	R	R	R	R			R	R	R	R	R	R	21	95
Lab042	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	22	100
Lab043	R	R	R	R	R	R	R	R	R	R	R	R	R	R			R	R	R	R		R	21	95
Lab044	R	R	R		R	R	R	R		R	R	R	R	R	R		R	R		R	R	R	17	77
Lab046*	R	R	R	R	R	R	R	R	R	R	R	R	R	R			R	R	R	R	R	R	20	91
Lab047	R		R	R	R	R	R	R		R	R	R	R	R			R	R	R	R	R	R	19	86
Lab048			R	R	R	R	R		R	R		R					R		R	R		R	12	55
Lab049	R	R	R	R		R	R	R	R	R	R	R	R	R			R	R		R	R	R	19	86
Lab050	R	R		R								R		R				R			R		7	32
Lab051	R	R					R					R		R									5	23
Lab053	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	22	100
Lab054			R	R	R	R	R			R			R	R			R	R	R		R		12	55
Lab055*	R	R	R	R	R	R	R	R	R	R		R	R		R		R	R	R		R	R	18	82
Lab056	R	R	R	R	R	R	R	R	R	R	R	R	R	R			R	R	R	R	R	R	21	95
Lab057	R	R	R	R	R	R	R	R	R	R	R	R	R	R			R	R	R	R	R	R	18	82
Lab058*	R	R	R	R	R	R	R	R	R	R	R	R	R	R			R	R	R	R	R	R	21	95
Lab059	R	R	R	R	R	R	R	R	R	R	R	R	R	R			R	R			R	R	19	86
Lab060	R	R	R	R			R					R		R							R		9	41
Lab061	R	R	R	R	R	R	R					R	R		R			R		R	R		13	59
Reported Pesticides	50	50	53	51	40	49	53	46	31	45	39	50	51	37	52	10	39	47	46	22	50	42		
% of Reported Pesticides	85	85	90	86	68	83	90	78	53	76	66	85	86	63	88	17	66	80	78	37	85	71		

R: Reported pesticide
 *NRLs from EU
 #The test item was spiked/treated with thiodicarb, which degrades to methomyl.
 The test item was not spiked/treated with methomyl.

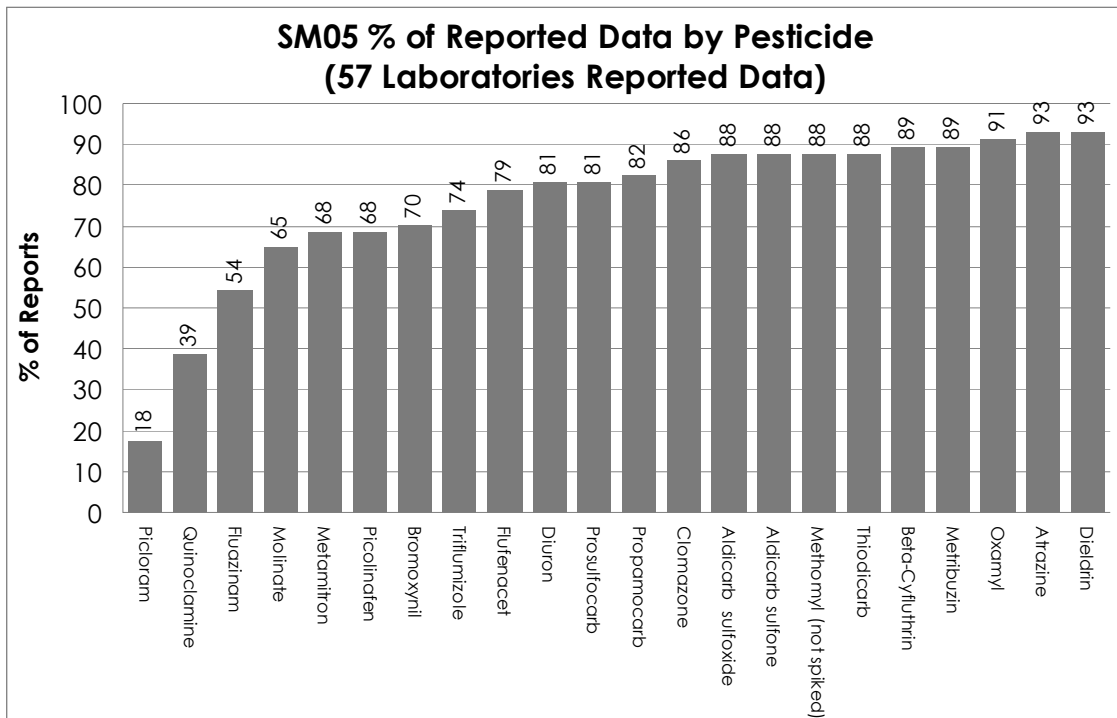
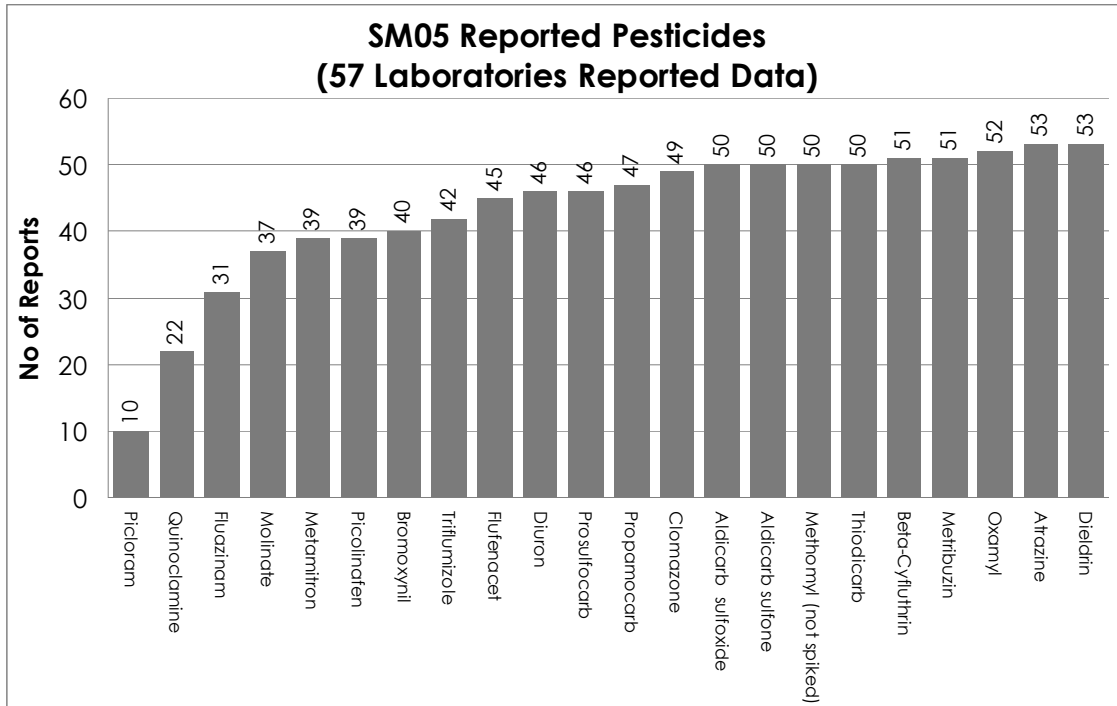
APPENDIX 2. Graphical Representations



APPENDIX 2. Graphical Representations

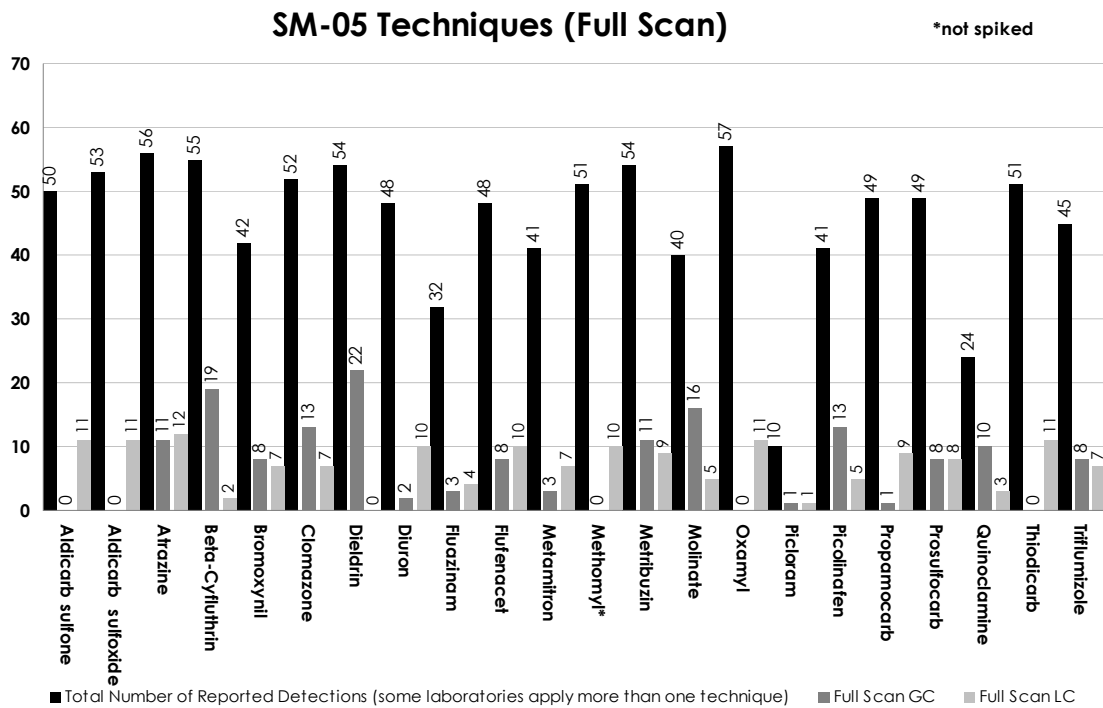


APPENDIX 2. Graphical Representations



APPENDIX 2. Graphical Representations

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



APPENDIX 3. Methods used by participants for detecting pesticides.

Aldicarb Sulfone																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume(µL)	Software	Compounds in (pesticides or library method or library)	Standard Solution Frequency
001	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	ABSCIEX	10	AcN	DSPE	C18	5	Automatic	200	Always
002	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.002	no	Xevo TQ-S	10	AcN	no	reversed phase	2.5	Automatic	300	Each batch
004*	Tentative detected [SANCO/12495/2011 p.74-80]	0	0	2 transitions	LC	MSMS	QQQ	ESI	+	yes	yes	0.01	no	Xevo TQ	15	AcN	LLE	C18	10	Both	400	Always
005*	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 6460	10	20	none	C18	2	Automatic	520	Always
005*	Confirmed with second method			accurate mass + isotope	LC	MSD	TOF	ESI	+	no	no	0.05	no	Bruker Maxis	10	10	none	C18	2	Automatic	760	Always
006*	Fully identified [SANCO/12495/2011 p.74-80]	1	none	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes		no	Agilent 6410	10	AcN	none	Eclipse XDB-C18	4	Manual	143	Each batch
007	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes		no	Agilent 6490	15	EIOAc	Dilution	Waters HSS T3	1	Manual	256	Each batch
008	Fully identified [SANCO/12495/2011 p.74-80]	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	AGILENT G-6410A	10	AcN	PSA	ZORBAX-ECLIPSE XDB-C8	20	Automatic	143	Each batch
009	Tentative detected	0.1	<10	accurate mass	LC	MSD	TOF	ESI	+	yes	yes	0.01	yes	Xevo Q-ToF	10	EfAc	none	BEH C18	3	Manual	500	Each batch
011	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Waters Xevo TQ	10	Acetone/PE/DCM	Na ₂ SO ₄	C18	2	Both	289	Each batch
012*	Tentative detected	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	Varian 1200L	25	EIOAc	no	C18	5	Both	50	
013	Fully identified [SANCO/12495/2011 p.74-80]	<2	0	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	>0.010	no	Waters Quattro Premier	15	AcN	no	BEH C18	3	Both		
014*	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01		ABI 4000 QTRAP	10	AcN	no	C18	55	Both	approx. 660	Always
015*	Fully identified [SANCO/12495/2011 p.74-80]				LC	MSD	Q-TRAP	ESI	+	no	no		no	AB Sciex 5500	10	AcN	PSA		10	Manual		
016*	Fully identified [SANCO/12495/2011 p.74-80]	30		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	xevo TQ	10	AcN	DSPE	C18	10	Automatic	162	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

Aldicarb Sulfone																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume(µl)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
017	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API-3200 QTrap	10	AcN	PSA	Athlantis T3	5	Both	180	Each batch
019	Tentative detected	5	0.005	None	LC	MSD	TOF	ESI	+	yes	yes	0.1	yes	LCT premier XE	15	Acetone; PE; DCM	No	BEH C18	7	Automatic	345	Daily
020*	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.010	no	Quattro Premier	10	MeOH		C18 BEH	5	Both	220	Each batch
021	Fully identified (SANCO/12495/2011 p.74-80)			2 MRM	LC	MSD	QQQ	ESI	+	yes	yes	0.001-0.01	no	Agilent 6460	10	AcN	dispersive SPE (Q)	YMC Triart C18 2*100 mm 3µm	5	Both	400	Each batch
022	Confirmed with second method	0.3	1.2	2 transitions	LC	MSD	Trap and Q-Orbitrap	ESI	+	yes	yes	0.01	yes	Thermo Fisher LTQ and Q-Exactive	10	AcN	PSA and SPE with NH ₂ /Carb. seperately	Shiseido CR 1.4 and BEH C18	10	Both	574	Every 12 injections
023	Fully identified (SANCO/12495/2011 p.74-80)	<2.5%		2 transitions	LC																	
024*	Confirmed with second method	3	0.8	accurate mass + isotope	LC		Q-TOF	ESI	+	no	yes		yes	Bruker Maxis	10	AcN	MgSO ₄ , PSA	C18	5	Both	450	often
025*	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	yes	yes		no		7.5	AcN, DCM	no	C18	5	Automatic		
026	Fully identified (SANCO/12495/2011 p.74-80)	0.04	450	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 1290/6460	10	AcN	25 mg PSA-150 mg MgSO ₄	HP 5 MS	5	Both	142	Daily
029	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	Waters Aquity UPLC system, API 5000 QQQ, Sciex	10	EIOAc	Filter	HSS T3	10	Both	387	Each batch
030	Fully identified (SANCO/12495/2011 p.74-80)	< ± 2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	HPLC Agilent 1100, MSD API 3000	10	AcN	DSPE	C18 3µ 50x2mm	10	Both	method. 550	Always
031	Confirmed with second method	0.2	1.1	2 transitions	LC	MSD	Trap and Q-Orbitrap	ESI	+	yes	yes	0.01	yes	Thermo fisher LTQ and Q-Exactive	10	AcN	QuEChERS and NH ₂ /Carb	SHISEIDO CR 1.4- BEH C18	10	Both	512	Every 12 injections
033*	Fully identified (SANCO/12495/2011 p.74-80)			223.1>86.1, 223.1>76.1	LC	MSD	QQQ	ESI	+	yes	yes	0.002		LC: Agilent 1200, MS: API 4000 QTRAP	10	AcN	GuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Automatic	591	
034*	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions	LC		QQQ	ESI	+	yes	yes	0.01	no	Quattro Premier	10	AcN	DSPE (PSA/MgSO ₄)	C18	5	Both	197	
035	Confirmed with second method			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	API 3200QTrap	50	Acetone	SLE	C18	20	Both	300	Each batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Aldicarb Sulfone																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume(µL)	Software	Compounds in (pesticides or library)	Standard Solution Frequency
037	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MS/MS	QQQ	ESI	+	yes	yes	from 0.01	no	4000 QTRAP	10	AcN	DSPE	C18 hydro Phenomenex	20	Both		Always
038	Confirmed with second method Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 6490	5	AcN		RP-18	5	Automatic	>500	Daily
039	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API 5500	10	AcN		Luna 5µm Pfp	3	Both	550	Each batch
041	Tentative detected	30	5	accurate mass	LC	Orbitrap	Orbitrap	ESI	+	yes	yes	0.01-0.05	yes	Thermo Orbitrap	10	AcN	PSA	RP18	10	Both	>100	Each batch
042	Fully identified [SANCO/12495/2011 p.74-80]	10		2*MRM	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	AB Sciex API 4000	10	AcN	PSA	Phenomenex C18	10	Both	500	Daily
044	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Thermo TSQ Quantum	10	AcN	GuEChERS	Xterra-MSC18	20	Both		Daily
046*	Fully identified [SANCO/12495/2011 p.74-80]	0.8	0	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Varian Pro Star LC + 320-MS	10	AcN	no clean up	Restek Ultra C18	10	Both	363	Each batch
047	Fully identified [SANCO/12495/2011 p.74-80]	5		2 transitions	LC	MSD	QQQ	ESI	+	no	no	0.1 - 1	no	API 4000	10	AcN	DSPE	C18	5	Automatic	500	when we find a + result
049	Fully identified [SANCO/12495/2011 p.74-80]	6		3 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01-0.5	no	API 4000	10	AcN	no	RP-Amide	10	Both	170	once a month
050	Tentative detected	268		2 transitions	LC	MSMS	QQQ	ESI	+	no	no	0.01	no	Waters Premier XE	10	AcN	DSPE	HSST3	4	Both	87	Always
051	Fully identified [SANCO/12495/2011 p.74-80]	± 2.5%	n.a.	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.010	no	API 3200 QTRAP	10	AcN	GuEChERS	C18	20	Both	102	Each batch
053	Fully identified [SANCO/12495/2011 p.74-80]	<30	<5	acc. m/z + isotope+fragment	LC	MSD	Orbitrap	ESI	+	no	no	0.01-0.1	yes	Exactive	10	AcN/1% HAC	none	C18 Atlantis	5	Both	650	Every batch ±100 pesticides
055*	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions + ion ratio	LC	MS	QQQ	ESI	+	yes	yes	0.005	no	Waters Acquity-Guattro Premier XE	15	Acetone/DCM/PE	No	UPLC Acquity BEH	5	Both	240	Every quarter
056	Fully identified [SANCO/12495/2011 p.74-80]	0.02		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	Waters Acquity TQD	15	AcN	DSPE	UPLC-BEH C18	10	Both	327	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

Aldicarb Sulfone																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume(µl)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
057	Fully identified [SANCO/12495/2011 p.74-80]	na	na	2 transitions	LC		QQQ	ESI	+	yes	yes	0.5 - 0.01	no	Agilent 1290-6490	10	AcN	PSA	XB-C18	10	Both	220	Daily
058*	Confirmed with second method	30	10	accurate mass + isotope	LC		TOF	ESI	+	no	yes		yes	Agilent 6230 TOF	10	AcN		C18	3	Automatic	510	
059	Fully identified [SANCO/12495/2011 p.74-80]	0.6		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Thermo Finnigan TSQ Quantum	10	AcN	DSPE (PSA)	Agilent Eclipse C18 XDB	20	Both	170	Each batch
060	Confirmed with second method	12		3 transitions	LC		QQQ	ESI	+	yes	yes	0.01	no	Varian-320	10	AcN	PSA	C-18	10	Automatic	72	Daily
061	Fully identified [SANCO/12495/2011 p.74-80]	120		2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	Waters XEVO TQMS	10	AcN	PSA	UPLC C18	25	Automatic	120	Each batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Aldicarb Sulfoxide																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	ABSCIEX	10	AcN	DSPE	C18	5	Automatic	200	Always
002	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.002	no	Xevo TQ-S	10	AcN	no	reversed phase	2.5	Automatic	300	Each batch
004*	Tentative detected	0	0	2 transitions	LC	MSMS	QQQ	ESI	+	yes	yes	0.005	no	Xevo TQ	15	AcN	LLE	C18	10	Both	400	Always
005*	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 6460	10	20	none	C18	2	Automatic	520	Always
	Confirmed with second method	0.06	0.7	accurate mass + isotope	LC	MSD	TOF	ESI	+	no	no	0.05	no	Bruker Maxis	10	10	none	C18	2	Automatic	760	Always
006*	Fully identified [SANCO/12495/2011 p.74-80]	1	none	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes		no	Agilent 6410	10	AcN	none	Eclipse XDB-C18	4	Manual	143	Each batch
007	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes		no	Agilent 6490	15	EIOAc	Dilution	Waters HSS T3	1	Manual	256	Each batch
008	Fully identified [SANCO/12495/2011 p.74-80]	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	AGILENT G-6410A	10	AcN	PSA	ZORBAX-ECLIPSE XDB-C8	20	Automatic	143	Each batch
009	Tentative detected	0.1	<10	accurate mass	LC	MSD	TOF	ESI	+	yes	yes	0.01	yes	Xevo Q-ToF	10	EIAc	none	BEH C18	3	Manual	500	Each batch
010*	Fully identified [SANCO/12495/2011 p.74-80]				LC	MSD	Q	ESI	+	no	yes		yes	Agilent	15	Acetone, DCM, PE	none		1	Automatic		
011	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Waters Xevo TQ	10	Acetone/PE/DCM	NasSO ₄	C18	2	Both	289	Each batch
012*	Tentative detected	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	Varian 1200L	25	EIOAc	no	C18	5	Both	50	
013	Fully identified [SANCO/12495/2011 p.74-80]	<2	0	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	>0.010	no	Waters Quattro Premier	15	AcN	no	BEH C18	3	Both		
014*	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01		ABI 4000 QTRAP	10	AcN	no	C18	55	Both	approx. 660	Always

APPENDIX 3. Methods used by participants for detecting pesticides.

Aldicarb Sulfoxide																							
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µl)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency	
015*	Fully identified [SANCO/12495/2011 p.74-80]				LC	MSD	Q-TRAP	ESI	+	no	no		no	AB Sciex 5500	10	AcN	PSA			10	Manual		
016*	Fully identified [SANCO/12495/2011 p.74-80]	30		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	xevo TQ	10	AcN	DSPE	C18	10	Automatic	162	Daily	
017	Fully identified [SANCO/12495/2011 p.74-80]				LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API-3200 QTrap	10	AcN	PSA	Athlantis T3	5	Both	180	Each batch	
018	Fully identified [SANCO/12495/2011 p.74-80]				LC	MSD	QQQ	ESI	+	yes	yes	0.01-1	no	Ultra Thermo Fishers	10	AcN	PSA	C18	10	Both			
019	Tentative detected	5	0.005	None	LC	MSD	TOF	ESI	+	yes	yes	0.01	yes	LCT premier XE	15	Acetone:PE:DCM	No	BEH C18	7	Automatic	345	Daily	
020*	Fully identified [SANCO/12495/2011 p.74-80]	3		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.010	no	Quattro Premier	10	MeOH		C18 BEH	5	Both	220	Each batch	
022	Confirmed with second method	0.2	1.4	2 transitions	LC	MSD	Trap and Q-Orbitrap	ESI	+	yes	yes	0.01	yes	Thermo Fisher LTQ and Q-Exactive	10	AcN	PSA and SPE with NH ₂ /Carb. separately	Shiseido CR 1.4 and BEH C18	10	Both	574	Every 12 injections	
023	Fully identified [SANCO/12495/2011 p.74-80]	<2.5%		2 transitions	LC																		
024*	Confirmed with second method	1	0.1	accurate mass + isotope	LC		Q-TOF	ESI	+	no	yes		yes	Bruker Maxis	10	AcN	MgSO ₄ , PSA	C18	5	Both	450	often	
025*	Fully identified [SANCO/12495/2011 p.74-80]				LC	MSD	QQQ	ESI	+	yes	yes		no		7.5	AcN, DCM	no	C18	5	Automatic			
026	Fully identified [SANCO/12495/2011 p.74-80]	0.04	485	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 1290/6460	10	AcN	25 mg PSA-150 mg MgSO ₄	HP 5 MS	5	Both	142	Daily	
029	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	Waters Aquity IJPLC system, API 5000 GQG, Sciex	10	EtOAc	Filter	HSS T3	10	Both	387	Each batch	
030	Fully identified [SANCO/12495/2011 p.74-80]	< ± 2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	HPLC Agilent 1100, MSD API 3000	10	AcN	DSPE	C18 3µ, 50x2mm	10	Both	method: 550	Always	
031	Confirmed with second method	0.05	1.0	2 transitions	LC	MSD	Trap and Q-Orbitrap	ESI	+	yes	yes	0.01	yes	Thermo Fisher LTQ and Q-Exactive	10	AcN	QuEChERS and NH ₂ /Carb	SHISEIDO CR 1.4, BEH C18	10	Both	512	Every 12 injections	

APPENDIX 3. Methods used by participants for detecting pesticides.

Aldicarb Sulfoxide																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µl)	Software	Compounds in (pesticides ONLY)	Standard Solution Frequency
033*	Fully identified [SANCO/12495/2011 p.74-80]	x	x	207.2>132.0, 207.2>68.9	LC	MSD	QQQ	ESI	+	yes	yes	0.008		LC: Agilent 1200, MS: API 4000 QTRAP	10	AcN	GuEChERS without PSA	Supelco Discovery C18, 150 mm x 2,1 mm, 5 µm	2	Automatic	591	x
034*	Fully identified [SANCO/12495/2011 p.74-80]	2		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Quattro Premier	10	AcN	DSPE (PSA/MgSO ₄)	C18	5	Both	197	
035	Confirmed with second method			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	API 3200QTrap	50	Acetone	SLE	C18	20	Both	300	Each batch
037	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MS/MS	QQQ	ESI	+	yes	yes	From 0.01	no	4000 QTRAP	10	AcN	DSPE	C18 hydro Phenomenex	20	Both		Always
038	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 6490	5	AcN		RP-18	5	Automatic	>500	Daily
039	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API 5500	10	AcN		Luna 5µm PIP	3	Both	550	Each batch
040	Fully identified [SANCO/12495/2011 p.74-80]	0	-	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	API 3200QTrap	10	AcN	DSPE	Aqua 5µ C18	10	Both	205	Always
041	Tentative detected	3	-	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	API 3200QTrap	10	AcN	DSPE	Aqua 5µ C18	10	Both	205	Always
042	Tentative detected	30	5	accurate mass	LC	Orbitrap	Orbitrap	ESI	+	yes	yes	0.01-0.05	yes	Thermo Orbitrap	10	AcN	PSA	RP18	10	Both	>100	Each batch
043	Fully identified [SANCO/12495/2011 p.74-80]	10		2*MRM	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	AB Sciex API 4000	10	AcN	PSA	Phenomenex C18	10	Both	500	Daily
043	Fully identified [SANCO/12495/2011 p.74-80]	0	1	3	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	XEVEO TQD	10	AcN		C18	3	Both	250	often
044	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Thermo TSQ Quantum	10	AcN	GuEChERS	Xterra-MSC18	20	Both		Daily
046*	Fully identified [SANCO/12495/2011 p.74-80]	0.6	0	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Varian Pro Star LC + 320-MS	10	AcN	no clean up	Restek Ultra C18	10	Both	363	Each batch
049	Fully identified [SANCO/12495/2011 p.74-80]	6		3 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01-0.5	no	API 4000	10	AcN	no	RP-Amide	10	Both	170	once a month
050	Tentative detected	258		2 transitions	LC	MSMS	QQQ	ESI	+	no	no	0.01	no	Waters Premier XE	10	AcN	DSPE	HS33	4	Both	87	Always

APPENDIX 3. Methods used by participants for detecting pesticides.

Aldicarb Sulfoxide																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µl)	Software	Compounds in (pesticides ONLY) method or library	Standard Solution Frequency
051	Fully identified [SANCO/12495/2011 p.74-80]	± 2.5%	n.a.	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.010	no	API 3200 QTRAP	10	AcN	QUECHERS	C18	20	Both	102	Each batch
053	Fully identified [SANCO/12495/2011 p.74-80]	<30	<5	acc. m/z + isotope + fragment	LC	MSD	Orbitrap	ESI	+	no	no	0.01-0.1	yes	Exactive	10	AcN/1% HAC	none	C18 Allantis	5	Both	650	Every batch ±100 pesticides
055*	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions + ion ratio	LC	MS	QQQ	ESI	+	yes	yes	0.005	no	Waters Acquity-Quattro Premier XE	15	Acetone/DCM/PE	No	UPLC Acquity BEH	5	Both	240	Every quarter
056	Fully identified [SANCO/12495/2011 p.74-80]	0.02		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	Waters Acquity TQD	15	AcN	DSPE	UPLC-BEH C18	10	Both	327	Daily
057	Fully identified [SANCO/12495/2011 p.74-80]	na	na	2 transitions	LC		QQQ	ESI	+	yes	yes	0.5-0.01	no	Agilent 1290-6490	10	AcN	PSA	XB-C18	10	Both	220	Daily
058*	Confirmed with second method	30	10	accurate mass + isotope	LC		TOF	ESI	+	no	yes		yes	Agilent 6230 ToF	10	AcN		C18	3	Automatic	510	
059	Fully identified [SANCO/12495/2011 p.74-80]	0.6		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Thermo Finnigan TSQ Quantum	10	AcN	DSPE (PSA)	Agilent Eclipse C18 XDB	20	Both	170	Each batch
060	Confirmed with second method	12		3 transitions	LC		QQQ	ESI	+	yes	yes	0.01	no	Varian-320	10	AcN	PSA	C-18	10	Automatic	72	Daily
061	Fully identified [SANCO/12495/2011 p.74-80]	120		2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	Waters XEVO TQMS	10	AcN	PSA	UPLC C18	25	Automatic	120	Each batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Atrazine																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
001	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	ABSCIEX	10	AcN	DSPE	C18	5	Automatic	200	Always
002	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.001	no	Xevo TQ-S	10	AcN	no	reversed phase	2.5	Automatic	300	Each batch
005*	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 6460	10	20	none	C18	2	Automatic	520	Always
	Confirmed with second method	-0.04	1.1	accurate mass + isotope	LC	MSD	TOF	ESI	+	no	no	0.05	no	Bruker Maxis	10	10	none	C18	2	Automatic	760	Always
006*	Fully identified [SANCO/12495/2011 p.74-80]	1	none	2 transitions	GC	MSD	Q	EI	+	yes	yes		no	Agilent 6890/5973	10	AcN	none	HP-5MS	2	Manual	182	Each batch
008	Fully identified [SANCO/12495/2011 p.74-80]	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	AGILENT G-6410A	10	AcN	PSA	ZORBAX-ECLIPSE XDB-C8	20	Automatic	143	Each batch
009	Tentative detected	0.1	<10	accurate mass	LC	MSD	TOF	ESI	+	yes	yes	0.01	yes	Xevo Q-ToF	10	EtAc	none	BEH C18	3	Manual	500	Each batch
010*	Fully identified [SANCO/12495/2011 p.74-80]				GC	MSD	Q	EI	+	no	yes		yes	Agilent	15	Acetone, DCM, PE	none	DB-5	1	Automatic		
011	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Waters Xevo TQ	10	Acetone/PE/DCM	Na ₂ SO ₄	C18	2	Both	289	Each batch
012*	Tentative detected	0		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.005	no	Agilent7890/Agilent7000	25	EIOAc	no	HP5MS	2	Both	310	
013	Fully identified [SANCO/12495/2011 p.74-80]	<2	<1	3 diagnostic ions	GC	MSD	Q	EI	+	yes	yes	>0.010	yes	Trace DSQ	15	AcN	PSA/C18	DB5MS	0.8	Both		
014*	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01		ABI 4000 QTRAP	10	AcN	no	C18	55	Both	approx. 660	Always
015*	Tentative detected				LC	MSD	Orbitrap	ESI	+	no	no		yes	Q-Exactive	10	AcN	PSA		10	Manual	510	
016*	Confirmed with second method	30		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	xevo TQ	10	AcN	DSPE	C18	10	Automatic	82	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

Atrazine																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µl)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
017	Confirmed with second method				LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API-3200 QTrap	10	AcN	PSA	Altamnis T3	5	Both	180	Each batch
018	Fully identified [SANCO/12495/2011 p.74-80]				LC	MSD	QQQ	ESI	+	yes	yes	0.01-1	yes	Ultra Thermo Fichers	10	AcN	PSA	C18	10	Both		
019	Tentative detected	5	0.005	None	LC	MSD	TOF	ESI	+	yes	yes	0.1	yes	LCT premier XE	15	Acetone; PE; DCM	No	BEH C18	7	Automatic	345	Daily
020*	Fully identified [SANCO/12495/2011 p.74-80]	3		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.010	no	VARIAN 320	50	Acetone	L/L	DB5	8	Both	150	Each batch
021	Fully identified [SANCO/12495/2011 p.74-80]			2 MRM	LC	MSD	QQQ	ESI	+	yes	yes	0.001-0.01	no	Agilent 6460	10	AcN	Dispersive SPE (Q)	YMC Triart C18 2*100 mm 3µm	5	Both	400	Each batch
022	Confirmed with second method	0.2	1.4	2 transitions	LC	MSD	Trap and Q-Orbitrap	ESI	+	yes	yes	0.05	yes	Thermo Fisher LTQ and Q-Exactive	10	AcN	PSA and SPE with NH ₂ /Carb. separately	Shiseido CR 1.4 and BEH C18	10	Both	574	Every 12 injections
023	Fully identified [SANCO/12495/2011 p.74-80]	<0.5%	<5ppm	full scan	GC																	
024*	Confirmed with second method	1	0.1	accurate mass + isotope	LC		Q-TOF	ESI	+	no	yes		yes	Bruker Maxis	10	AcN	MgSO ₄ , PSA	C18	5	Both	450	often
025*	Fully identified [SANCO/12495/2011 p.74-80]				GC	MSD	QQQ	EI	+	yes	yes		no		7.5	Acetone.DCM,PE	no	VF-5	1		86	
026	Fully identified [SANCO/12495/2011 p.74-80]	0.02	470	3 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7890/7000	10	AcN	25 mg PSA-150 mg MgSO ₄	HP 5 MS	4	Both	355	Daily
028	Fully identified [SANCO/12495/2011 p.74-80]				GC	MSD	IT	EI		no	no		yes	VARIAN	3	Acetone/PE/DCM		VF-5MS	3	Automatic		
029	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	Waters Aquity UPLC system, API 5000 QQQ, Sciex	10	EIOAc	Filter	HSS T3	10	Both	387	Each batch
030	Fully identified [SANCO/12495/2011 p.74-80]	< ± 2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	HPLC Agilent 1100, MSD API 3000	10	AcN	DSPE	C18 3µ 50x2mm	10	Both	method: 550	Always
031	Confirmed with second method	0.2	1.8	2 transitions	LC	MSD	Trap and Q-Orbitrap	ESI	+	yes	yes	0.05	yes	Thermo Fisher LTQ and Q-Exactive	10	AcN	QuEChERS and NH ₂ /Carb	SHISEIDO CR 1.4, BEH C18	10	Both	512	Every 12 injections

APPENDIX 3. Methods used by participants for detecting pesticides.

Atrazine																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
032	Fully identified [SANCO/12495/2011 p.74-80]	0.5	10	3	GC	MSD	Q	EI	+	yes	yes	0.001	yes	AGILENT 5975 inert MSD	102.31	Acetone/EtOAc-cyclohexane	0.2 mcm PTFE filter	HP-5MS	1 mcl	Automatic	108	once a week
033*	Confirmed with second method			216.1>174.0, 216.1>104.1	LC	MSD	QQQ	ESI	+	no	no	0.01-0.1		LC: Agilent 1200, MS: API 4000 QTRAP	10	AcN	GuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Automatic	591	
034*	Fully identified [SANCO/12495/2011 p.74-80]	1		2 transitions	GC		QQQ	EI	+	yes	yes	0.01	no	Quattro micro	10	AcN	DSPE (PSA/MgSO ₄)	HP5	5	Both	110	
035	Confirmed with second method			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	API 3200QTrap	50	Acetone	SLE	C18	20	Both	300	Each batch
036	Fully identified [SANCO/12495/2011 p.74-80]				LC	MSD	QQQ	EI		yes	yes			AB SCIEX API 4000	2	ACN/MeOH	GuEChERS					all time
037	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MS/MS	QQQ	ESI	+	yes	yes	from 0.01	no	4000 QTRAP	10	AcN	DSPE	C18 hydro Phenomenex	20	Both		Always
038	Confirmed with second method			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7000B	5	AcN	DSPE	HP5-MS-UI	2	Automatic	>500	Daily
039	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API 5500	10	AcN	DSPE	Pursuit XRs Ultra	3	Both	550	Each batch
040	Fully identified [SANCO/12495/2011 p.74-80]	1		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.005	no	TSQ Quantum Ultra	10	AcN	DSPE	RX1 5 Sil MS	2	Both	290	Always
041	Tentative detected	15	unit mass	target + qualifier	GC	MSD	Q	EI	+	yes	yes	0.01-0.05	yes	Agilent GC-MSD	10	AcN	PSA	DB5	10	Both	>600	Each batch
042	Fully identified [SANCO/12495/2011 p.74-80]	10		2 masses	GC	MSD	IT	EI	+	yes	yes	0.01	yes	Varian Saturn 2000	10	AcN	PSA	VactorFour SMS	5	Automatic	500	Daily
043	Fully identified [SANCO/12495/2011 p.74-80]	0	1	3	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	XEVEO TGD	10	AcN		C18	3	Both	250	often
044	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Thermo TSQ Quantum	10	AcN	GuEChERS	Xterra-MSC18	20	Both		Daily
046*	Fully identified [SANCO/12495/2011 p.74-80]	0	0	3 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Varian 3800 GC + 320-MS	10	AcN	DSPE (PSA)	VF-1 ms	3	Both	363	Each batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Atrazine																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µl)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
047	Fully identified [SANCO/12495/2011 p.74-80]	5		2 transitions	LC	MSD	QQQ	ESI	+	no	no	0.1 -1	no	API 4000	10	AcN	DSPE	C18	5	Automatic	500	when we find a result
048	Tentative detected	2			GC	MSD	Q	EI		yes	no		yes	agilent 5973	10	AcN	PSA	HP 5 MS UI	2	Both	approx 600	Each batch
049	Fully identified [SANCO/12495/2011 p.74-80]	6		3 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01-0.5	no	API 4000	10	AcN	no	RP-Amide	10	Both	170	once a month
053	Fully identified [SANCO/12495/2011 p.74-80]	<20		spectrum	GC	MSD	TOF	EI	+	yes	yes	0.01-0.2	yes	Leco Pegasus IV	10	AcN/1% HAC	DSPE (PSA)	RTX-Cibesticides	10	Automatic	560	Every batch 235 pesticides
	Confirmed with second method	<30	<5	accurate mass + isotope	LC	MSD	Orbitrap	ESI	+	no	no	0.01-0.1	yes	Exactive	10	AcN/1% HAC	none	C18 Atlantis	5	Both	650	Every batch ±100 pesticides
054	Tentative detected	-0.5	20%	* 3 ions	GC	MSD	Q	EI	+	yes	no	0.05	yes	Agilent GC-MSD 7890A/5975c	10	AcN	PSA	HP-5MS	1	Automatic	927	weekly
055*	Fully identified [SANCO/12495/2011 p.74-80]	0.12		full scan	LC	MS	QQQ	ESI	+	yes	yes	0.01	yes	Varian 3800 + Saturn 2000	15	Acetone/DCM/PE	No	VF-ms5	5	Both	550	Every quarter
056	Fully identified [SANCO/12495/2011 p.74-80]	0.01		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	Waters Acquity TQD	15	AcN	DSPE	UPLC-BEH C18	10	Both	327	Daily
057	Fully identified [SANCO/12495/2011 p.74-80]	na	na	2 transitions	LC		QQQ	ESI	+	yes	yes	0.5 - 0.01	no	Agilent 1290-6490	10	AcN	PSA	XB-C18	10	Both	220	Daily
058*	Confirmed with second method	30	10	accurate mass + isotope	LC		TOF	ESI	+	no	yes		yes	Agilent 6230 TOF	10	AcN		C18	3	Automatic	510	
059	Fully identified [SANCO/12495/2011 p.74-80]	0.96	0	4 transitions	GC	MSD	IT	EI	+	yes	yes	0.01	no	Varian Saturn 4000	10	AcN	DSPE (PSA)	Restek Rxi-5ms	3	Both	202	Each batch
060	Confirmed with second method	12			GC		IT	EI	+	yes	yes	0.01	yes	VARIAN 4000	10	AcN	PSA	5MS	10	Automatic	121	Daily
061	Tentative detected			qualifiers	GC	MSD	IT	EI	+	yes	no		yes	Varian 4000	10	AcN	PSA	capillary VF5-pesticides	5	Manual		

APPENDIX 3. Methods used by participants for detecting pesticides.

Beta-Cyfluthrin																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
001	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	yes	AGILENT	10	AcN	DSPE	HP5MS	1	Automatic	200	Always
002	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC	MSD	QQQ	EI		yes	yes		no	Agilent 7000 Triple quadrupole	10	AcN	yes	HP-5ms	2	Automatic	170	Each batch
005*	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Thermo ISQ Quantum	10	20	none	TG-5SILMS	2	Automatic	520	Always
006*	Fully identified (SANCO/12495/2011 p.74-80)	1	none	2 transitions	GC	MSD	Q	EI	+	yes	yes		no	Agilent 6890/5973	10	AcN	none	HP-5MS	2	Manual	182	Each batch
007	Confirmed with second method			2 transitions	GC	MSD	QQQ	EI	+	yes	yes		no	Varian 1200	15	EIOAc	PSA	db-5ms	5	Manual	214	Each batch
008	Fully identified (SANCO/12495/2011 p.74-80)	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	AGILENT G-6410A	10	AcN	PSA	ZORBAX-ECLIPSE XDB-C8	20	Automatic	143	Each batch
010*	Tentative detected				GC	MSD	Q	EI	+	no	no		yes	Agilent	15	Acetone, DCM, PE	none		1	Automatic		
011	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Thermo ISQ Quantum GC	10	Acetone/PE/DCM	Na ₂ SO ₄	RXI-5SIL MS	1	Both	201	Each batch
012*	Tentative detected	0		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.005	no	Agilent 7890/Agilent 7000	25	EIOAc	no	HP5MS	2	Both	310	
013	Fully identified (SANCO/12495/2011 p.74-80)	<2	<1	3 diagnostic ions	GC	MSD	Q	EI	+	yes	yes	>0.01	yes	Trace DSQ	15	AcN	PSA/C18	DB5MS	0.8	Both		
014*	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01		Agilent 7000 QQQ	10	AcN	DSPE	HP5MS	10	Both	approx. 1100	Always
015*	Tentative detected				GC	MSD		EI		no	no		yes	Agilent	10	AcN	PSA		2	Manual		
016*	Fully identified (SANCO/12495/2011 p.74-80)	40		3 ions	GC	MSD	IT	EI		yes	yes	0.01	yes	Varian 4000 MS	10	AcN	DSPE	VF 5ms 20M*0.15mm	1	Automatic	239	Daily
017	Fully identified (SANCO/12495/2011 p.74-80)			AMDIS match 50-79	GC	MSD	Q	EI	+	no	no	NA	yes	AGILENT TECHNOLOGIES 7890A // G3440A	10	AcN	PSA	HP-5MS	20	Automatic	560	
018	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	QQQ	EI	+	yes	yes	0.01-1	yes	ISQ Thermo Fishers	10	AcN	PSA	C18	1	Both		
019	Tentative detected	5	0.005	None	LC	MSD	TOF	ESI	+	yes	yes	0.1	yes	LCT premier XE	15	Acetone; PE; DCM	No	BEH C18	7	Automatic	345	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

Beta-Cyfluthrin																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µl)	Software	Compounds in method (pesticides ONLY) or library or library	Standard Solution Frequency
020*	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.010	no	VARIAN 320	50	Acetone	L/L	DB5	8	Both	1.50	Each batch
021	Fully identified (SANCO/12495/2011 p.74-80)			SIM. 3 ions	GC	MSD	Q	EI	+	yes	yes	0.001-0.01	yes	Agilent HP 5973N	10	AcN	DSPE (Q)	HP 5MS 30 m x 0.25 mm x 0.25 µm	50	Both	cc. 100	Each batch
022	Confirmed with second method	0	1.2	2 transitions	GC	MSD	Q and Q-Orbitrap	CI	-	yes	yes	0.001	no	Bruker 320MS and Q-Exactive	10	AcN	PSA and SPE with NH ₂ /Carb. separately	1(GC) and 10(LC)	Both	208 for GC-MS and 574 for LC-HRMS	Every 12 injections	
023	Fully identified (SANCO/12495/2011 p.74-80)	<0.5%	<5ppm	full scan	GC																	
024*	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions	GC		QQQ	EI	+	yes	yes		no	Waters Micro	10	AcN	MgSO ₄ , PSA	DB-5MS	5	Automatic		
026	Fully identified (SANCO/12495/2011 p.74-80)	0.04	230	3 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7890/7000	10	AcN	25 mg PSA-150 mg MgSO ₄	4	Both	355	Daily	
028	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	IT	EI		no	no		yes	VARIAN	3	Acetone/PE/DCM		VF-5MS	3	Automatic		
029	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Waters Quattro Micro GC	10	EtOAc	Filter	Rxi-5sil MS/integra-guard Restek	10	Both	387	Each batch
030	Fully identified (SANCO/12495/2011 p.74-80)	< ± 0.5%		1 Target, 3 Qualifier	GC	MSD	Q	CI	-	yes	yes	0.01	no	GC Shimadzu GC-2010, MS Shimadzu GCMS-QP2010	10	AcN	DSPE	HP-5MS	3	Both	method: 550	Always
031	Confirmed with second method	0	1.5	4 transitions	GC	MSD	Q and Q-Orbitrap	ESI	+	yes	yes	0.005	yes	Varian 320 and Thermo Fisher Q-Exactive	10	AcN	QuEChERS and NH ₂ /Carb	DB5-ms, BEH C18	1(GC) and 10(LC)	Both	512	Every 12 injections
033*	Fully identified (SANCO/12495/2011 p.74-80)			Match with analytical standard (Match 910, (range 0-1000))	GC	MSD	TOF	EI		yes	yes	0.01	yes	GCxGC-TOF MS Pegasus 4D	10	AcN	QuEChERS without PSA	DB5-MS (30 m x 0.25 mm x 0.25 µm) in the 1st Dimension, BPX-50 (2 m x 0.1 mm x 0.1 µm) in the 2nd Dimension	10	Automatic		
034*	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC		QQQ	EI	+	yes	yes	0.01	no	Quattro micro	10	AcN	DSPE (PSA/MgSO ₄)	HP5	5	Both	110	Each batch
035	Confirmed with second method			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	AGILENT	50	Acetone	SLE	DB17-MS	1	Both	120	Each batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Beta-Cyfluthrin																							
Laboratory Code	*NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
036		Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	Q	EI		yes	yes			BRUKER SCION TQ	2	ACN/MeOH	QueChERS					Always
037		Fully identified (SANCO/12495/2011 p.74-80)			MS/MS spectrum	GC	MS/MS	IT	EI		yes	yes	from 0.01	no	Varian Saturn 2000	10	AcN	DSPE	CP Sil 8	2	Both		Always
038		Confirmed with second method			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7000B	5	AcN		HP-5-MS-UI	2	Automatic	>500	Daily
039		Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	Q	CI	-	yes	yes	0.01	no	Agilent 6890	10	AcN	DSPE	Phenomenex ZB-5MS	1	Both		Always
040		Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.005	no	TSQ Quantum Ultra	10	AcN	DSPE	RXI 5 Sil MS	2	Both	290	Always
041		Tentative detected	15	unit mass	target + qualifier	GC	MSD	Q	EI	+	yes	yes	0.01-0.05	yes	Agilent GC-MSD	10	AcN	PSA	DB5	10	Both	>600	Each batch
042		Fully identified (SANCO/12495/2011 p.74-80)	10		2 masses	GC	MSD	Q	EI	+	yes	yes	0.01	no	Agilent 5975	10	AcN	PSA	GsbP-5MS	5	Both	500	Daily
043		Fully identified (SANCO/12495/2011 p.74-80)	0	1	4	GC	MSD	Q	CI	-	yes	yes	0.01	no	5975C	10	AcN	PSA	DB5-MS	2	Both	100	often
046*		Fully identified (SANCO/12495/2011 p.74-80)	-1	0	3 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Varian 3800 GC + 320-MS	10	AcN	DSPE (PSA)	VF-1 ms	3	Both	363	Each batch
047		Fully identified (SANCO/12495/2011 p.74-80)	10		2 transitions	GC	MSD	Q	EI	+	no	no	0.1 -1	no	Agilent 5973	10	AcN	DSPE	C18	5	Automatic	500	when we find a + result
048		Tentative detected	2			GC	MSD	Q	EI		yes	no		yes	agilent 5973	10	AcN	PSA	HP 5 MS UI	2	Both	approx 600	Each batch
049		Fully identified (SANCO/12495/2011 p.74-80)	15		4 sim ions	GC	MSD	Q	EI	+	no	no	0.05-1.0	yes	Thermo DSQ	10	AcN	no	DB-5ms	1	Both	800	once a month
050		Tentative detected	1137		2 transitions	GC	MSMS	QQQ	EI	-	no	no	0.01	no	Thermo ISQ Quantum	10	AcN	DSPE	5MS - 30mx0.25x0.25	1	Both	33	Always
050		Tentative detected	1145		2 transitions	GC	MSMS	QQQ	EI	-	no	no	0.01	no	Thermo ISQ Quantum	10	AcN	DSPE	5MS - 30mx0.25x0.25	1	Both	33	Always
053		Fully identified (SANCO/12495/2011 p.74-80)	<20		spectrum	GC	MSD	TOF	EI	+	yes	yes	0.01-0.2	yes	Leco Pegasus IV	10	AcN/1% HAC	DSPE (PSA)	RTX-CIPesticides	10	Automatic	560	Every batch 236 pesticides

APPENDIX 3. Methods used by participants for detecting pesticides.

Beta-Cyfluthrin																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
054	Tentative detected	-8.3 0.2 3.8	20%	3 ions	GC	MSD	Q	EI	+	yes	no	0.01	yes	Agilent GC-MSD 7890A/5975C	10	AcN	PSA	HP-5MS	1	Automatic	927	weekly
055*	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions + ion ratio	GC	MS	QQQ	EI	+	yes	yes	0.005	no	Brüker 451 Scion TQ	15	Acetone/DCM/PE	No	UPLC Acquity BEH	5	Both	340	Every quarter
056	Fully identified (SANCO/12495/2011 p.74-80)	0.02		3 transitions	GC	MSD	Q	EI	+	yes	yes	0.01	yes	Agilent 7890 A-5975C	15	AcN	DSPE	HP-5MS	10	Both	220	Daily
057	Fully identified (SANCO/12495/2011 p.74-80)	na	na	2 transitions	GC		QQQ	EI	+	yes	yes	0.5 - 0.01	no	Agilent 7890-7000B	10	AcN	PSA	DB-5ms UI	2	Both	330	Daily
058*	Confirmed with second method	30	10	accurate mass + isotope	LC		TOF	ESI	-	no	yes		yes	Agilent 6230 TOF	10	AcN		C18	3	Automatic	510	
059	Fully identified (SANCO/12495/2011 p.74-80)	0.84	0.08	3 transitions	GC	MSD	IT	EI	+	yes	yes	0.01	no	Varian Saturn 4000	10	AcN	DSPE (PSA)	Restek Rxi-5ms	3	Both	202	Each batch
060	Confirmed with second method	24			GC		IT	EI	+	yes	yes	0.010	yes	VARIAN 4000	10	AcN	PSA	5MS	10	Automatic	121	Daily
061	Fully identified (SANCO/12495/2011 p.74-80)	0.25		qualifiers	GC	MSD	IT	EI	+	yes	yes	0.05	yes	Varian 4000	10	AcN	PSA	capillary VF5-pesticides	5	Automatic	121	Each batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Bromoxynil																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	-	yes	yes	0.01	yes	ABSCIEX	10	AcN	DSPE	C18	5	Automatic	200	Always
002	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC	MSD	QQQ	ESI	-	yes	yes	0.005	no	Xevo TQ-S	10	AcN	no	reversed phase	3	Automatic	20	Each batch
005*	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	-	yes	no	0.05	no	Agilent 6460	10	20	none	C18	2	Automatic	520	Always
	Confirmed with second method	-0.25	0.2	accurate mass + isotope	LC	MSD	TOF	ESI	-	no	no	0.05	no	Bruker Maxis	10	10	none	C18	2	Automatic	760	Always
008	Fully identified (SANCO/12495/2011 p.74-80)	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	AGILENT G-6410A	10	AcN	PSA	ZORBAX-ECLIPSE XDB-C8	20	Automatic	143	Each batch
009	Tentative detected	0.1	<10	accurate mass	LC	MSD	TOF	ESI	-	yes	yes	0.01	yes	Xevo Q-TOF	10	EtAc	none	BEH C18	3	Manual	500	Each batch
010*	Tentative detected				GC	MSD	Q	EI	+	no	no		yes	Agilent	15	Acetone, DCM, PE	none		1	Automatic		
011	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC	MSD	QQQ	ESI	-	yes	yes	0.01	no	Waters Xevo TQ	10	MeOH	Na ₂ SO ₄	UPLC HSS C18	5	Both	22	Each batch
013	Fully identified (SANCO/12495/2011 p.74-80)	<2	0	2 transitions	LC	MSD	QQQ	ESI	-	yes	yes	>0.01	no	Waters Quattro Premier	15	AcN	no	BEH C18	3	Both		
014*	Tentative detected	1.08		2 transitions	GC	MSD	Q	EI	+	yes	yes	0.01	yes	HP5975C	10	AcN	DSPE	HP5MS	10	Both	approx. 1100	Always
016*	Fully identified (SANCO/12495/2011 p.74-80)	30		2 transitions	LC	MSD	Q-TRAP	ESI	-	yes	yes	0.01	no	AB sciex 3200Qtrap	10	AcN	DSPE	C18	10	Automatic	15	Each batch
017	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	-	yes	yes	0.01	no	API-3200 Qtrap	10	AcN		Athlantis T3	5	Both	180	Each batch
018	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	-	yes	yes	0.01 - 1	yes	Ultra Thermo Fichers	10	AcN	PSA	C18	10	Both		
020*	Fully identified (SANCO/12495/2011 p.74-80)	3		full mass	GC	MSD	Q	EI	+	yes	yes	0.01	no	VARIAN 320	50	Acetone	L/L	DB5	8	Both	150	never
024*	Confirmed with second method	3	0.2	accurate mass + isotope	LC		Q-TOF	ESI	-	no	yes		yes	Bruker Maxis	10	AcN	MgSO ₄ , PSA	C18	5	Both	450	often
025*	Tentative detected				GC	MSD	QQQ	EI	+	no	no		no		7.5	Acetone, DCM, PE	no	VF-5	1			
026	Fully identified (SANCO/12495/2011 p.74-80)	0.04	365	2 transitions	LC	MSD	QQQ	ESI	-	yes	yes	0.01	no	Agilent 1290/6460	10	AcN	25 mg PSA-150 mg MgSO ₄	HP 5 MS	5	Both	142	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

Bromoxynil																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
029	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	EI	-	yes	yes	0.01	no	Waters Acquity UPLC system, API 5000 QQQ, Sciex	5	EIOAC	Filter	HSS T3	5	Both	20	Each batch
030	Fully identified (SANCO/12495/2011 p.74-80)	< ± 2.5%		2 transitions	LC	MSD	QQQ	ESI	-	yes	yes	0.01	no	HP LC Agilent 1100, MSD API 3000	10	AcN	none	C18 3µ 50x2mm	10	Both	method: 550	Always
033*	Fully identified (SANCO/12495/2011 p.74-80)			273.7>78.9, 275.4>81.0	LC	MSD	QQQ	ESI	-	yes	yes	0.02		LC: Agilent 1200, MS: API 4000 QTRAP	10	AcN	QUECHERS without PSA	Supelco Discovery C18, 150 mm x 2,1 mm, 5 µm	2	Automatic	591	
034*	Tentative detected			4 transitions	LC		QQQ	ESI	-	no	no	0.01	no	Quattro Premier	10	AcN	DSPE (PSA/MgSO ₄)	C18	5	Both		
035	Confirmed with second method			2 transitions	LC	MSD	QQQ	EI	-	yes	yes	0.005	no	API 2000	50	Acetone	SPE	C18	100	Both	18	Each batch
036	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	EI		yes	yes			AB SCIEX API 4000	2	AcN/MeOH	QUECHERS					all time
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	-	yes	yes	From 0.01	no	4000 QTRAP	10	AcN	DSPE	C18 hydro Phenomenex	20	Both		Always
038	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	-	yes	yes	0.01	no	Agilent 6490	5	AcN	none	RP-18	5	Automatic	>500	Daily
039	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	-	yes	yes	0.01	no	API 5500	10	AcN	none	Pursuit UPS 2.4	3	Both	550	Each batch
041	Tentative detected	15	unit mass	target + qualifier	GC	MSD	Q	EI	+	yes	yes	0.01-0.05	yes	Agilent GC-MSD	10	AcN	PSA	DB5	10	Both	>600	Each batch
042	Fully identified (SANCO/12495/2011 p.74-80)	10		2*MRM	LC	MSD	QQQ	EI	-	yes	yes	0.01	no	AB Sciex API 4000	10	AcN	PSA	Phenomenex C18	10	Both	500	Daily
043	Fully identified (SANCO/12495/2011 p.74-80)	0	1	3	LC	MSD	QQQ	ESI	-	yes	yes	0.01	no	XEVEO TQD	10	AcN		C18	3	Both	250	often
044	Tentative detected				GC	MSD	IT	EI	+	no	no		yes	Thermo Polaris Q	10	AcN	QUECHERS	DB-5MS	5	Both		
046*	Fully identified (SANCO/12495/2011 p.74-80)	-0,6	0	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Variation Pro Star LC + 320-MS	10	AcN	no clean up	Restek Ultra C18	10	Both	363	Each batch
047	Fully identified (SANCO/12495/2011 p.74-80)	5		2 transitions	LC	MSD	QQQ	ESI	-	no	no	0.1 -1	no	API 4000	10	AcN	DSPE	C18	5	Automatic	500	when we find a + result
048	Tentative detected	2			GC	MSD	Q	EI		yes	no		yes	agilent 5973	10	AcN	PSA	HP 5 MS UI	2	Both	approx 600	Each batch
053	Fully identified (SANCO/12495/2011 p.74-80)	<20		spectrum	GC	MSD	TOF	EI	+	yes	yes	0.01-0.2	yes	Leco Pegasus IV	10	AcN/1% HAc	DSPE (PSA)	RTX-Cipesticides	10	Automatic	560	Every batch 235 pesticides

APPENDIX 3. Methods used by participants for detecting pesticides.

Bromoxynil																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µL)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
	Confirmed with second method	<30	<5	accurate mass + isotope	LC	MSD	Orbitrap	ESI	-	no	no	0.01-0.1	yes	Exactive	10	AcN/1% HAc	none	C18 Atlantis	5	Both	650	Every batch ±100 pesticides
054	Tentative detected	2	20%	3 ions	GC	MSD	Q	EI	+	yes	no	0.05	yes	Agilent GC-MSD 7890A/5975c	10	AcN	PSA	HP-5MS	1	Automatic	927	weekly
055*	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions + ion ratio	LC	MS	QQQ	ESI	-	yes	yes	0.005	no	Waters Acquity-Quattro Premier XE	15	Acetone/DCM/PE	No	UPLC Acquity BEH	5	Both	27	Each batch
056	Fully identified (SANCO/12495/2011 p.74-80)	0.01		2 transitions	LC	MSD	QQQ	ESI	-	yes	yes	0.01	yes	Waters Acquity TQD	15	AcN	DSPE	UPLC-BEH C18	10	Both	327	Daily
057	Fully identified (SANCO/12495/2011 p.74-80)	na	na	2 transitions	LC		QQQ	ESI	-	yes	yes	0.5 - 0.01	no	Agilent 1290-6490	10	AcN	PSA	XB-C18	10	Both	220	Daily
058*	Confirmed with second method	30	10	accurate mass + isotope	LC		TOF	ESI	-	no	yes		yes	Agilent 6230 TOF	10	AcN		C18	3	Automatic	510	
059	Fully identified (SANCO/12495/2011 p.74-80)	0.4		3 transitions	LC	MSD	QQQ	ESI	-	yes	yes	0.01	no	Thermo Finnigan TSQ Quantum	10	AcN	DSPE (PSA)	Agilent Eclipse C18 XDB	20	Both	170	Each batch
061	Tentative detected			qualifiers	GC	MSD	IT	EI	+	yes	no		yes	Varian 4000	10	AcN	PSA	capillary VF5-pesticides	5	Manual		

APPENDIX 3. Methods used by participants for detecting pesticides.

Clomazone																							
Laboratory Code	*NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method (pesticides ONLY)	Standard Solution Frequency
001		Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	ABSCIEX	10	AcN	DSPE	C18	5	Automatic	200	Always
002		Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes		no	Xevo TQ-S	10	AcN	no	reversed phase	2.5	Automatic	300	Each batch
004*		Tentative detected	0	0	2 transitions	LC	MSMS	QQQ	ESI	+	yes	yes	0.005	no	Xevo TQ	15	AcN	LLE	C18	10	Both	400	Always
005*		Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 6460	10	20	none	C18	2	Automatic	520	Always
		Confirmed with second method	-0.01	0.2	accurate mass + isotope	LC	MSD	TOF	ESI	+	no	no	0.05	no	Bruker Maxis	10	10	none	C18	2	Automatic	760	Always
006*		Fully identified (SANCO/12495/2011 p.74-80)	1	none	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes		no	Agilent 6410	10	AcN	none	Eclipse XDB-C18	4	Manual	143	Each batch
008		Fully identified (SANCO/12495/2011 p.74-80)	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	AGILENT G-6410A	10	AcN	PSA	ZORBAX-ECLIPSE XDB-C8	20	Automatic	143	Each batch
009		Tentative detected	0.1	<10	accurate mass	LC	MSD	TOF	ESI	+	yes	no	0.01	yes	Xevo Q-ToF	10	EtAc	none	BEH C18	3	Manual	500	Each batch
010*		Tentative detected				GC	MSD	Q	EI	+	no	no		yes	Agilent	15	Acetone. DCM. PE	none		1	Automatic		
011		Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Waters Xevo TQ	10	Acetone/PE/DCM	Na ₂ SO ₄	C18	2	Both	289	Each batch
012*		Tentative detected	0		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.005	no	Agilent7890/Agilent7000	25	EIOAc	no	HP5MS	2	Both	310	
013		Tentative detected	<2	<1	3 diagnostic ions	GC	MSD	Q	EI	+	yes	no		yes	Trace DSQ	15	AcN	PSA/C18	DB5MS	0.8	Both		
014*		Fully identified (SANCO/12495/2011 p.74-80)	2.2		2 transitions	GC	MSD	Q	EI	+	yes	yes	0.01	yes	HP5975C	10	AcN	DSPE	HP5MS	10	Both	approx. 1100	Always
016*		Confirmed with second method	30		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	xevo TQ	10	AcN	DSPE	C18	10	Automatic	82	Daily
017		Tentative detected	-2.0		82	GC	MSD	Q	EI	+	no	no	NA	yes	AGILENT TECHNOLOGIES 7890A // G3440A	10	AcN	PSA	HP-5MS	20	Automatic	560	
018		Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	yes	yes	0.01-1	no	Ultra Thermo Fichers	10	AcN	PSA	C18	10	Both		

APPENDIX 3. Methods used by participants for detecting pesticides.

Clomazone																							
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µl)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency	
019	Tentative detected Fully identified (SANCO/12495/2011 p.74-80)	5	0.005	None	LC	MSD	TOF	ESI	+	yes	yes	0.1	yes	LCT premier XE	15	Acetone; PE; DCM	No	BEH C18	7	Automatic	345	Daily	
020*	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.010	no	VARIAN 320	50	Acetone	L/L	D85	8	Both	150	Each batch	
021	Fully identified (SANCO/12495/2011 p.74-80)			2 MRM	LC	MSD	QQQ	ESI	+	yes	yes	0.001-0.01	no	Agilent 6460	10	AcN	DSPE (Q)	YMC Triart C18 2*100 mm 3µm	5	Both	400	Each batch	
022	Confirmed with second method	0.2	1.7	2 transitions	LC	MSD	Q and Q-Orbitrap	EI	+	yes	yes	0.005	no	Bruker 320MS and Q-Exactive	10	AcN	PSA and SPE with NH ₂ /Carb. separately	DB-5 ms and BEH C18	1(GC) and 10(LC)	Both	208 for GC-MS and 574 for LC-HRMS	Every 12 injections	
023	Fully identified (SANCO/12495/2011 p.74-80)	<0.5%	<5ppm	full scan	GC																		
024*	Confirmed with second method	1	0.2	accurate mass + isotope	LC		Q-TOF	ESI	+	no	yes		yes	Bruker Maxis	10	AcN	MgSO ₄ , PSA	C18	5	Both	450	often	
026	Fully identified (SANCO/12495/2011 p.74-80)	0.02	420	3 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7890/7000	10	AcN	25 mg PSA-150 mg MgSO ₄	HP 5 MS	4	Both	355	Daily	
029	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	Waters Aquity UPLC system. API 5000 QQQ. Sciex	10	EIOAc	Filter	HSS T3	10	Both	387	Each batch	
030	Fully identified (SANCO/12495/2011 p.74-80)	< ± 2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	HPLC Agilent 1100. MSD API 3000	10	AcN	DSPE	C18 3µ 50x2mm	10	Both	method: 550	Always	
031	Confirmed with second method	0	1.6	4 transitions	GC	MSD	Q and Q-Orbitrap	ESI	+	yes	yes	0.005	yes	Varian 320 and Thermo Fisher Q-Exactive	10	AcN	QuEChERS and NH ₂ /Carb	DB5-ms. BEH C18	1(GC) and 10(LC)	Both	512	Every 12 injections	
033*	Confirmed with second method			240.1>125.0, 240.1>89.1	LC	MSD	QQQ	ESI	+	no	no	0.01-0.1		LC: Agilent 1200. MS: API 4000 QTRAP	10	AcN	QuEChERS without PSA	Supelco Discovery C18. 150 mm x 2.1 mm. 5 µm	2	Automatic	591		
034*	Confirmed with second method			full scan	GC	MSD	Q	EI	+	no	no	0.05	yes	Agilent 5973	10	AcN	DSPE (PSA/MgSO ₄)	HP5	5	Both	800		
035	Confirmed with second method			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	API 3200QTrap	50	Acetone	SLE	C18	20	Both	300	Each batch	
036	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	EI		yes	yes			AB SCIEX API 4000	2	AcN/MeOH	QuEChERS					all time	
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	yes	yes	from 0.01	no	4000 QTRAP	10	AcN	DSPE	C18 hydro Phenomenex	20	Both		Always	

APPENDIX 3. Methods used by participants for detecting pesticides.

Clomazone																							
Laboratory Code	*NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µl)	Software	Compounds in method (pesticides ONLY)	Standard Solution Frequency
038		Confirmed with second method			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7000B	5	AcN		HP-5-MS-UI	2	Automatic	>500	Daily
039		Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API 5500	10	AcN	DSPE	Pursuit XRs Ultra	3	Both	550	Each batch
040		Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.005	no	TSQ Quantum Ultra	10	AcN	DSPE	RXI 5 SII MS	2	Both	290	Always
041		Tentative detected	15	unit mass	target + qualifier	GC	MSD	Q	EI	+	yes	yes	0.01-0.05	yes	Agilent GC-MSD	10	AcN	PSA	DB5	10	Both	>600	Each batch
042		Fully identified (SANCO/12495/2011 p.74-80)	10		2 masses	GC	MSD	Q	EI	+	yes	yes	0.01	no	Agilent 5975	10	AcN	PSA	GsbP-5MS	5	Both	500	Daily
043		Fully identified (SANCO/12495/2011 p.74-80)	0	1	3	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	XEVEO TQD	10	AcN		C 18	3	Both	250	often
044		Tentative detected				GC	MSD	IT	EI	+	no	no		yes	Thermo Polaris Q	10	AcN	GuChERS	DB-5MS	5	Both		
046*		Fully identified (SANCO/12495/2011 p.74-80)	0.1	0	3 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Varian 3800 GC + 320-MS	10	AcN	DSPE (PSA)	VF-1 ms	3	Both	363	Each batch
047		Fully identified (SANCO/12495/2011 p.74-80)	5		2 transitions	LC	MSD	QQQ	ESI	+	no	no	0.1 -1	no	API 4000	10	AcN	DSPE	C 18	5	Automatic	500	when we find a + result
048		Tentative detected	2			GC	MSD	Q	EI		yes	no		yes	agilent 5973	10	AcN	PSA	HP 5 MS UI	2	Both	approx 600	Each batch
049		Tentative detected	15		3 sim ions	GC	MSD	Q	EI	+	no	no	0.05-1.0	yes	Thermo Polaris	10	AcN	no	Elite-5ms	1	Both	800	once a year
		Fully identified (SANCO/12495/2011 p.74-80)	<20		spectrum	GC	MSD	TOF	EI	+	yes	yes	0.01-0.2	yes	Leco Pegasus IV	10	AcN/1% HAC	DSPE (PSA)	RTX-C pesticides	10	Automatic	560	Every batch 235 pesticides
053		Confirmed with second method	<30	<5	accurate mass + isotope	LC	MSD	Orbitrap	ESI	+	no	no	0.01-0.1	yes	Exactive	10	AcN/1% HAC	none	C 18 Atlantis	5	Both	650	Every batch ±100 pesticides
054		Tentative detected	-2	20%	3 ions	GC	MSD	Q	EI	+	yes	no	0.01	yes	Agilent GC-MSD 7890A/5975C	10	AcN	PSA	HP-5MS	1	Automatic	927	weekly
055*		Fully identified (SANCO/12495/2011 p.74-80)	0.24		2 transitions + ion ratio	GC	MS	QQQ	EI	+	yes	yes	0.005	no	Bruker 451 Scion TQ	15	Acetone/DCM/PE	No	UPLC-Acquity BEH	5	Both	340	Every quarter
056		Fully identified (SANCO/12495/2011 p.74-80)	0.01		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	Waters Acquity TQD	15	AcN	DSPE	UPLC-BEH C18	10	Both	327	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

Clomazone																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	M5 Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
057	Fully identified (SANCO/12495/2011 p.74-80)	na	na	2 transitions	LC		QQQ	ESI	+	yes	yes	0.5 - 0.01	no	Agilent 1290-6490	10	AcN	PSA	XB-C18	10	Both	220	Daily
058*	Confirmed with second method	30	10	accurate mass + isotope	LC		TOF	ESI	+	no	yes		yes	Agilent 6230 TOF	10	AcN		C18	3	Automatic	510	
059	Fully identified (SANCO/12495/2011 p.74-80)	0.18	0	2 transitions	GC	MSD	IT	EI	+	yes	yes	0.01	no	Varian Saturn 4000	10	AcN	DSPE (PSA)	Restek Rxi-5ms	3	Both	202	Each batch
061	Tentative detected			qualifiers	GC	MSD	IT	EI	+	yes	no		yes	Varian 4000	10	AcN	PSA	capillary VF5-pesticides	5	Manual		

APPENDIX 3. Methods used by participants for detecting pesticides.

Dieldrin																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µl)	Software	Compounds in method or library (pesticides ONLY)	Standard Solution Frequency
001	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	yes	AGILENT	10	AcN	DSPE	HP5MS	1	Automatic	200	Always
002	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC	MSD	QQQ	EI		yes	yes		no	Agilent 7000 Triple quadrupole	10	AcN	yes	HP-5ms	2	Automatic	170	Each batch
004*	Tentative detected	0	0	2 transitions	GC	MSMS	QQQ	EI	+	yes	yes		no	Agilent GC-MS/MS 7000	15	AcN	LLE	DB-5MS UI	1	Both	130	Always
005*	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Thermo ISQ Quantum	10	20	none	TC-5SILMS	2	Automatic	520	Always
006*	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions	GC	MSD	Q	EI	+	yes	yes		no	Agilent 6890/5973	10	AcN	none	HP-5MS	2	Manual	182	Each batch
007	Confirmed with second method			2 transitions	GC	MSD	QQQ	EI	+	yes	yes		no	Varian 1200	15	EIOAc	PSA	db-5ms	5	Manual	214	Each batch
008	Fully identified (SANCO/12495/2011 p.74-80)	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	AGILENT G-6410A	10	AcN	PSA	ZORBAX-ECLIPSE XDB-C8	20	Automatic	143	Each batch
010*	Tentative detected				GC	MSD	Q	EI	+	no	no		yes	Agilent	15	Acetone, DCM, PE	none		1	Automatic		
011	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Thermo ISQ Quantum GC	10	Acetone/PE/DCM	Na ₂ SO ₄	RXI-5SII MS	1	Both	201	Each batch
012*	Tentative detected	0		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.005	no	Agilent 7890/Agilent 7000	25	EIOAc	no	HP5MS	2	Both	310	
013	Fully identified (SANCO/12495/2011 p.74-80)	<2	<1	3 diagnostic ions	GC	MSD	Q	EI	+	yes	yes	>0.010	yes	Trace DSQ	15	AcN	PSA/C18	DB5MS	0.8	Both		
014*	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01		Agilent 7000 QQQ	10	AcN	DSPE	HP5MS	10	Both	approx. 1100	Always
015*	Tentative detected				GC	MSD		EI		no	no		yes	Agilent	10	AcN	PSA		2	Manual		
016*	Fully identified (SANCO/12495/2011 p.74-80)	40		3 ions	GC	MSD	IT	EI		yes	yes	0.01	yes	Varian 4000 MS	10	AcN	DSPE	VF 5ms 20M*0.15mm	1	Automatic	239	Daily
017	Fully identified (SANCO/12495/2011 p.74-80)	-2.8		AMDIS Match 98	GC	MSD	Q	EI	+	no	no	NA	yes	AGILENT TECHNOLOGIES 7890A / G3440A	10	AcN	PSA	HP-5MS	20	Automatic	560	

APPENDIX 3. Methods used by participants for detecting pesticides.

Dieldrin																							
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency	
018	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	QQQ	EI	+	yes	yes	0.01-1	yes	TSQ Thermo Fichers	10	AcN	PSA	C18	1	Both			
020*	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.010	no	VARIAN 320	50	Acetone	L/L	DB5	8	Both	150	Each batch	
021	Fully identified (SANCO/12495/2011 p.74-80)			SIM, 3 ions	GC	MSD	Q	EI	+	yes	yes	0.001-0.01	yes	Agilent HP 5973N	10	AcN	DSPE (Q)	HP 5MS 30 m x 0.25 mm x 0.25 µm	50	Both	cc. 100	Each batch	
022	Tentative detected	0	0	2 transitions	GC	MSD	Q	CI	-	yes	yes	0.001	no	Bruker 320MS	10	AcN	PSA and SPE with NH ₂ /Carb. separately	DB-5 ms	1	Both	208	Every 12 injections	
023	Fully identified (SANCO/12495/2011 p.74-80)	<0.5%	<5	full scan	GC																		
024*	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	GC		QQQ	EI	+	yes	yes		no	Waters Micro	10	AcN	MgSO ₄ , PSA	DB-5MS	5	Automatic			
025*	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	QQQ	EI	+	yes	yes		no		7.5	Acetone,DCM,PE		VF-5	1		86		
026	Fully identified (SANCO/12495/2011 p.74-80)	0.02	270	3 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7890/7000	10	AcN	25 mg PSA-150 mg MgSO ₄	HP 5 MS	4	Both	355	Daily	
029	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Waters Quattro Micro GC	10	EIOAC	Filter	Rxi-5sil MS/integra-guard Restek	10	Both	387	Each batch	
030	Fully identified (SANCO/12495/2011 p.74-80)	< ± 0.5%		1 Target, 3 Qualifier	GC	MSD	Q	CI		yes	yes	0.01	no	GC Shimadzu GC-2010, MS Shimadzu GCMS-QP2010	10	AcN	DSPE	HP-5MS	3	Both	550	Always	
031	Tentative detected	0	1.3	4 transitions	GC	MSD	Q	CI		yes	yes	0.001	yes	Varian 320	10	AcN	GuEChERS and NH ₂ /Carb	DB5-ms, BEH C18	1	Both	512	Every 12 injections	
032	Fully identified (SANCO/12495/2011 p.74-80)	0.5	10	3	GC	MSD	Q	EI	+	yes	yes	0.001	yes	AGILENT 5975 inert MSD	102.31	Acetone/EIOAC-cyclohexane	0.2 µm PTFE filter	HP-5MS	1	Automatic	108	once a week	
033*	Fully identified (SANCO/12495/2011 p.74-80)			Match with analytical standard (Match 941, range 0-1000)	GC	MSD	TOF	EI		yes	yes	0.008	yes	GCxGC-TOF MS Pegasus 4D	10	AcN	GuEChERS without PSA	DB5-MS (30 m x 0.25 mm x 0.25 µm) in the 1 st Dimension, BPX-50 (2 m x 0.1 mm x 0.1 µm) in the 2 nd Dimension	10	Automatic			

APPENDIX 3. Methods used by participants for detecting pesticides.

Dieldrin																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µl)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
034*	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions	GC		QQQ	EI	+	yes	yes	0.01	no	Quattro micro	10	AcN	DSPE (PSA/MgSO ₄)	HP5	5	Both	110	
035	Confirmed with second method			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	AGILENT	50	Acetone	SLE	DB17-MS	1	Both	120	Each batch
036	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	QQQ	EI		yes	yes			BRUKER SCION TQ	2	ACN/MeOH	QuEChERS					Always
037	Fully identified (SANCO/12495/2011 p.74-80)			MS/MS spectrum	GC	MS/MS	IT	EI		yes	yes	from 0.01	no	Varian Saturn 2000	10	AcN	DSPE	CP Sil 8	2	Both		Always
038	Confirmed with second method			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7000B	5	AcN		HP5-MS-UI	2	Automatic	>500	Daily
039	Fully identified (SANCO/12495/2011 p.74-80)				GC		TOF	EI		yes	yes	0.01	yes	Leco Pegasus IV	10	AcN	DSPE	HP5-MS	3	Both	750	Always
040	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.005	no	TSQ Quantum Ultra	10	AcN	DSPE	RXI 5 Sil MS	2	Both	290	Always
041	Tentative detected	15	unit mass	target + qualifier	GC	MSD	Q	EI	+	yes	yes	0.01-0.05	yes	Agilent GC-MSD	10	AcN	PSA	DB5	10	Both	>600	Each batch
042	Fully identified (SANCO/12495/2011 p.74-80)	10		2 masses	GC	MSD	IT	EI	+	yes	yes	0.01	yes	Varian Saturn 2000	10	AcN	PSA	VactorFour 5MS	5	Automatic	500	Daily
043	Fully identified (SANCO/12495/2011 p.74-80)	0	1	4	GC	MSD	Q	CI	-	yes	yes	0.01	no	5975C	10	AcN	PSA	DB5-MS	2	Both	100	often
044	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MSD	IT	EI	+	yes	yes	0.01	no	Thermo PolariscQ	10	AcN	QuEChERS	DB-5MS	5	Both		Daily
046*	Fully identified (SANCO/12495/2011 p.74-80)	0	0	4 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Varian 3800 GC + 320-MS	10	AcN	DSPE (PSA)	VF-1 ms	3	Both	363	Each batch
047	Fully identified (SANCO/12495/2011 p.74-80)	10		2 transitions	GC	MSD	Q	EI	+	no	no	0.1 -1	no	Agilent 5973	10	AcN	DSPE	C18	5	Automatic	500	when we find a + result
048	Tentative detected	2			GC	MSD	Q	EI		yes	no		yes	agilent 5973	10	AcN	PSA	HP 5 MS UI	2	Both	approx 600	Each batch
049	Fully identified (SANCO/12495/2011 p.74-80)	15		4 sim ions	GC	MSD	Q	EI	+	no	no	0.05-1.0	yes	Thermo DSQ	10	AcN	no	DB-5ms	1	Both	800	once a month

APPENDIX 3. Methods used by participants for detecting pesticides.

Dieldrin																							
Laboratory Code	*NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
051		Confirmed with second method	± 0.5%			GC	ECD				no	no	0.010		GC-Agilent 5890	10	AcN	QuEChERS	Rtx Pesticide2	1	Manual		
053		Fully identified (SANCO/12495/2011 p.74-80)	<20		spectrum	GC	MSD	TOF	EI	+	yes	yes	0.01-0.2	yes	Leco Pegasus IV	10	AcN/1% HAC	DSPE (PSA)	RTX-Cpesticides	10	Automatic	560	Every batch 235 pesticides
054		Tentative detected	-2.4	20%	3 ions	GC	MSD	Q	EI	+	yes	no	0.01	yes	Agilent GC-MSD 7890A/5975C	10	AcN	PSA	HP-5MS	1	Automatic	927	weekly
055*		Fully identified (SANCO/12495/2011 p.74-80)	0.12		2 transitions + ion ratio	GC	MS	QQQ	EI	+	yes	yes	0.005	no	Bruker 451 Scion IQ	15	Acetone/DCM/PE	No	UPLC Acquity BEH	5	Both	340	Every quarter
056		Fully identified (SANCO/12495/2011 p.74-80)	0.01		3 transitions	GC	MSD	Q	EI	+	yes	yes	0.01	yes	Agilent 7890A-5975C	15	AcN	DSPE	HP-5MS	10	Both	220	Daily
057		Fully identified (SANCO/12495/2011 p.74-80)	na	na	2 transitions	GC		QQQ	EI	+	yes	yes	0.5-0.01	no	Agilent 7890-7000B	10	AcN	PSA	DB-5ms UI	2	Both	330	Daily
058*		Confirmed with second method			full scan	GC	MSD	Q	EI	+	no	yes		yes	Agilent 5973	30	EIOAc	HFGPC	DB5-MS	2	Automatic	500	
059		Fully identified (SANCO/12495/2011 p.74-80)	0.84	0	2 transitions	GC	MSD	IT	EI	+	yes	yes	0.01	no	Varian Saturn 4000	10	AcN	DSPE (PSA)	Restek Rxi-5ms	3	Both	202	Each batch
060		Confirmed with second method	12			GC		IT	EI	+	yes	yes	0.01	yes	VARIAN 4000	10	AcN	PSA	5MS	10	Automatic	121	Daily
061		Fully identified (SANCO/12495/2011 p.74-80)	0.25		qualifiers	GC	MSD	IT	EI	+	yes	yes	0.05	yes	Varian 4000	10	AcN	PSA	capillary VF5-pesticides	5	Automatic	121	Each batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Diuron																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µl)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
001	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	ABSCIEX	10	AcN	DSPE	C18	5	Automatic	200	Always
002	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.002	no	Xevo TQ-S	10	AcN	no	reversed phase	2.5	Automatic	300	Each batch
004*	Tentative detected	0	0	2 transitions	LC	MSMS	QQQ	ESI	+	yes	yes	0.005	no	Xevo TQ	15	AcN	LLE	C18	10	Both	400	Always
005*	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 6460	10	20	none	C18	2	Automatic	520	Always
	Confirmed with second method	-0.05	0.4	accurate mass + isotope	LC	MSD	TOF	ESI	+	no	no	0.05	no	Bruker Maxis	10	10	none	C18	2	Automatic	760	Always
008	Fully identified (SANCO/12495/2011 p.74-80)	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	AGILENT G-6410A	10	AcN	PSA	ZORBAX-ECLIPSE XDB-C8	20	Automatic	143	Each batch
009	Tentative detected	0.1	<10	accurate mass	LC	MSD	TOF	ESI	+	yes	yes	0.01	yes	Xevo Q-ToF	10	EtAc	none	BEH C18	3	Manual	500	Each batch
010*	Tentative detected				GC	MSD	Q	EI	+	no	no		yes	Agilent	15	Acetone, DCM, PE	none		1	Automatic		
011	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Waters Xevo TQ	10	Acetone/PE/DCM	Na ₂ SO ₄	C18	2	Both	289	Each batch
013	Tentative detected	<2	0	1 transition	LC	MSD	QQQ	ESI	+	yes	yes	>0.010	no	Waters Quattro Premier	15	AcN	no	BEH C18	3	Both		
014*	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01		ABI 4000 QTRAP	10	AcN	no	C18	55	Both	approx. 660	Always
015*	Tentative detected				LC	MSD	Orbitrap	ESI	+	no	no		yes	Q-Exactive	10	AcN	PSA		10	Manual	510	
016*	Fully identified (SANCO/12495/2011 p.74-80)	30		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	xevo TQ	10	AcN	DSPE	C18	10	Automatic	162	Daily
017	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API-3200 Qtrap	10	AcN	PSA	Athlantis T3	5	Both	180	Each batch
018	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	yes	yes	0.01-1	no	Ultra Thermo Fichers	10	AcN	PSA	C18	10	Both		
019	Tentative detected	5	0.005	None	LC	MSD	TOF	ESI	+	yes	yes	0.1	yes	LCT premier XE	15	Acetone, PE, DCM	No	BEH C18	7	Automatic	345	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

Diuron																							
Laboratory Code	*NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
020*		Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.010	no	Quattro Premier	10	MeOH		C18 BEH	5	Both	220	Each batch
021		Fully identified (SANCO/12495/2011 p.74-80)			2 MRM	LC	MSD	QQQ	ESI	+	yes	yes	0.001-0.01	no	Agilent 6460	10	AcN	DSPE (Q)	YMC Triart C18 2*100 mm 3µm	5	Both	400	Each batch
022		Confirmed with second method	0.3	1.6	2 transitions	LC	MSD	Q-Orbitrap	ESI	+	yes	yes	0.005	yes	Q-Exactive	10	AcN	PSA and SPE with NH ₂ /Carb. separately	BEH C18	10	Both	574	Every 12 injections
023		Fully identified (SANCO/12495/2011 p.74-80)	<2.5%		2 transitions	LC																	
024*		Confirmed with second method	1	0.4	accurate mass + isotope	LC		Q-TOF	ESI	+	no	yes		yes	Bruker Maxis	10	AcN	MgSO ₄ . PSA	C18	5	Both	450	often
025*		Tentative detected				GC	MSD	QQQ	EI	+	no	no		no		7.5	Acetone.DCM.IPE	no	VF-5	1			
026		Fully identified (SANCO/12495/2011 p.74-80)	0.04	430	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 1290/6460	10	AcN	25 mg PSA-150 mg MgSO ₄	HP 5 MS	5	Both	142	Daily
029		Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.05	no	Waters Acquity UPLC system, API 5000 QQQ, Sciex	10	EIOAc	Filter	HSS T3	10	Both	123	Each batch
030		Fully identified (SANCO/12495/2011 p.74-80)	< ± 2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	HPLC Agilent 1100, MSD API 3000	10	AcN	DSPE	C18 3µ 50x2mm	10	Both	method: 550	Always
031		Tentative detected	0.4	2.1	2 transitions	LC	MSD	Q-Orbitrap	ESI	+	yes	yes	0.005	yes	Q-Exactive	10	AcN	QuEChERS and NH ₂ /Carb	BEH C18	10	Both	512	Every 12 injections
033*		Tentative detected	x	x	133.1>72.0, 235.1>72.1	LC	MSD	QQQ	ESI	+	no	no	0.01-0.1		LC: Agilent 1200, MS: API 4000 QTRAP	10	AcN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5µm	2	Automatic	591	x
034*		Tentative detected			3 transitions	LC		QQQ	ESI	+	no	no	0.01	no	Quattro Premier	10	AcN	DSPE (PSA/MgSO ₄)	C18	5	Both		
035		Confirmed with second method			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	API 3200QTrap	50	Acetone	SLE	C18	20	Both	300	Each batch
036		Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	EI		yes	yes			AB SCIEX API 4000	2	AcN/MeOH	QuEChERS					all time
037		Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	yes	yes	from 0.01	no	4000 QTRAP	10	AcN	DSPE	C18 hydro Phenomenex	20	Both		Always

APPENDIX 3. Methods used by participants for detecting pesticides.

Diuron																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
038	Confirmed with second method (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 6490	5	AcN		RP-18	5	Automatic	>500	Daily
039	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API 5500	10	AcN	DSPE	Pursuit XRs Ultra	3	Both	550	Each batch
040	Fully identified (SANCO/12495/2011 p.74-80)	2		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	API 3200Qtrap	10	AcN	DSPE	Aqua 5u C18	10	Both	205	Always
041	Tentative detected	30	5	accurate mass	LC	Orbitrap	Orbitrap	ESI	+	yes	yes	0.01-0.05	yes	Thermo Orbitrap	10	AcN	PSA	RP18	10	Both	>100	Each batch
042	Fully identified (SANCO/12495/2011 p.74-80)	10		2*MRM	LC	MSD	QQQ	EI	-	yes	yes	0.01	no	AB Sciex API 4000	10	AcN	PSA	Phenomenex C18	10	Both	500	Daily
043	Fully identified (SANCO/12495/2011 p.74-80)	0	1	3	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	XEVEO TQD	10	AcN		C18	3	Both	250	often
044	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Thermo TSQ Quantum	10	AcN	GuEChERS	Xterra-MS-C18	20	Both		Daily
046*	Fully identified (SANCO/12495/2011 p.74-80)	-0.6	0	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Varian Pro Star LC + 320-MS	10	AcN	no clean up	Restek Ultra C18	10	Both	363	Each batch
047	Fully identified (SANCO/12495/2011 p.74-80)	2.06		2 transitions	LC	MSD	QQQ	ESI	+	no	no	0.1 -1	no	API 4000	10	AcN	DSPE	C18	5	Automatic	500	when we find a + result
049	Fully identified (SANCO/12495/2011 p.74-80)	6		3 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01-0.5	no	API 4000	10	AcN	no	RP-Amide	10	Both	170	once a month
	Confirmed with second method	<20		spectrum	GC	MSD	TOF	EI	+	yes	yes	0.01-0.2	yes	Leco Pegasus IV	10	AcN/1% HAC	DSPE (PSA)	RTX-Cipesticides	10	Automatic	560	Every batch 235 pesticides
053	Fully identified (SANCO/12495/2011 p.74-80)	<30	<5	acc. m/z + isotope+fragment	LC	MSD	Orbitrap	ESI	+	no	no	0.01-0.1	yes	Exactive	10	AcN/1% HAC	none	C18 Atlantis	5	Both	650	Every batch ±100 pesticides
055*	Fully identified (SANCO/12495/2011 p.74-80)	1.2		2 transitions + ion ratio	LC	MS	QQQ	ESI	+	yes	yes	0.005	no	Waters Acquity-Guadatro Premier XE	15	Acetone/DCM/PE	No	UPLC-Aquity BEH	5	Both	240	Every quarter
056	Fully identified (SANCO/12495/2011 p.74-80)	<0.01		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	Waters Acquity TQD	15	AcN	DSPE	UPLC-BEH C18	10	Both	327	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

Diuron																							
Laboratory Code	*NRL	How was the identification done?	RT Deviation (in seconds)	M5 Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
057		Fully identified (SANCO/12495/2011 p.74-80)	na	na	2 transitions	LC		QQQ	ESI	+	yes	yes	0.5-0.01	no	Agilent 1290-6490	10	AcN	PSA	XB-C18	10	Both	220	Daily
058*		Confirmed with second method	30	10	accurate mass + isotope	LC		TOF	ESI	+	no	yes		yes	Agilent 6230 ToF	10	AcN		C18	3	Automatic	510	
059		Fully identified (SANCO/12495/2011 p.74-80)	0.5		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Thermo Finnigan TSQ Quantum	10	AcN	DSPE (PSA)	Agilent Eclipse C18 XDB	20	Both	170	Each batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Fluazinam																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µl)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
002	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions	LC	MSD	QQQ	ESI	-	yes	yes	0.002	no	Xevo TQ-S	10	AcN	no	reversed phase	3	Automatic	20	Each batch
005*	Confirmed with second method Fully identified [SANCO/12495/2011 p.74-80]	n/a	0.7	accurate mass + isotope 2 transitions	LC	MSD	TOF	ESI	+	no	no	0.05	no	Bruker Maxis	10	10	none	C18	2	Automatic	760	Always
006*	Fully identified [SANCO/12495/2011 p.74-80]	1	none	2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.05	no	Thermo ISQ Quantum	10	20	none	TG-SILMS	2	Automatic	520	Always
007	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	-	yes	yes		no	Agilent 6410	10	AcN	none	Eclipse XDB-C18	4	Manual	143	Each batch
009	Tentative detected	0.1	<10	accurate mass	LC	MSD	TOF	ESI	-	yes	yes	0.01	yes	Xevo Q-ToF	10	EIAC	none	BEH C18	3	Manual	500	Each batch
011	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.02	no	Waters Xevo IQ	10	Acetone/PE/DCM	Na ₂ SO ₄	C18	2	Both	289	Each batch
012*	Tentative detected	0		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.005	no	Agilent7890/Agilent7000	25	EIOAc	no	HP5MS	2	Both	310	
013	Fully identified [SANCO/12495/2011 p.74-80]	<2	0	2 transitions	LC	MSD	QQQ	ESI	-	yes	yes	>0.01	no	Waters Quattro Premier	15	AcN	no	BEH C18	3	Both		
016*	Fully identified [SANCO/12495/2011 p.74-80]	30		2 transitions	LC	MSD	Q-TRAP	ESI	-	yes	yes	0.01	no	AB sciex 3200Qtrap	10	AcN	DSPE	C18	10	Automatic	15	Each batch
018	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	-	yes	yes	0.01-1	no	Ultra Thermo Fichers	10	AcN	PSA	C18	10	Both		
020*	Fully identified [SANCO/12495/2011 p.74-80]	3		2 transitions	LC	MSD	QQQ	ESI	-	yes	yes	0.010	no	Quattro Premier	10	MeOH		C18 BEH	5	Both	220	Each batch
023	Fully identified [SANCO/12495/2011 p.74-80]	<2.5%		2 transitions	LC																	
026	Fully identified [SANCO/12495/2011 p.74-80]	0.02	21.5	3 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7890/7000	10	AcN	25 mg PSA-150 mg MgSO ₄	HP 5 MS	4	Both	355	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

Fluazinam																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY)	Standard Solution Frequency
030	Fully identified [SANCO/12495/2011 p.74-80]	< ± 2.5%		2 transitions	LC	MSD	QQQ	ESI	-	yes	yes	0.01	no	HPIC Agilent 1100. MSD API 3000	10	AcN	none	C18 3µ. 50x2mm	10	Both	method: 550	Always
033*	Tentative detected			462.9>415.8, 462.9>397.9	LC	MSD	QQQ	ESI	-	no	no	0.01-0.1		LC: Agilent 1200. MS: API 4000 QTRAP	10	AcN	GuEChERS without PSA	Supelco Discovery C18. 150 mm x 2.1 mm. 5 µm	2	Automatic	591	
034*	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions	LC	MSD	QQQ	ESI	-	yes	yes	0.01	no	Quattro Premier	10	AcN	DSPE (PSA/MgSO ₄)	C18	5	Both	197	
035	Confirmed with second method			2 transitions	LC	MSD	QQQ	EI	-	yes	yes	0.01	no	API 3200QTrap	50	Acetone	SLE	C18	40	Both	15	Each batch
038	Confirmed with second method			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7000B	5	AcN		HP-5-MS-UI	2	Automatic	>500	Daily
039	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	-	yes	yes	0.01	no	API 5500	10	AcN	none	Pursuit UPS 2.4	3	Both	550	Each batch
041	Tentative detected	15	unit mass	target + qualifier	GC	MSD	Q	EI	+	yes	yes	0.01-0.05	yes	Agilent GC-MSD	10	AcN	PSA	DB5	10	Both	>600	Each batch
042	Fully identified [SANCO/12495/2011 p.74-80]	10		2*MRM	LC	MSD	QQQ	EI	-	yes	yes	0.01	no	AB Sciex API 4000	10	AcN	PSA	Phenomenex C18	10	Both	500	Daily
043	Fully identified [SANCO/12495/2011 p.74-80]	0	1	3	LC	MSD	QQQ	ESI	-	yes	yes	0.01	no	XEVEO TQD	10	AcN		C18	3	Both	250	often
046*	Fully identified [SANCO/12495/2011 p.74-80]	-1.8	0	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Varian Pro Star LC + 320-MS	10	AcN	no clean up	Restek Ultra C18	10	Both	363	Each batch
048	Tentative detected	2			GC	MSD	Q	EI		yes	no		yes	agilent 5973	10	AcN	PSA	HP 5 MS UI	2	Both	approx 600	Each batch
049	Tentative detected	15		3 sim ions	GC	MSD	Q	EI	+	no	no	0.05-1.0	yes	Thermo Polaris	10	AcN	no	Elite-5ms	1	Both	800	once a year
053	Tentative detected	<30	<5	accurate mass + isotope	LC	MSD	Orbitrap	ESI	-	no	no	0.01-0.1	yes	Exactive	10	AcN/1% Hac	none	C18 Atlantis	5	Both	650	Every batch ±100 pesticides
055*	Fully identified [SANCO/12495/2011 p.74-80]	1.2		2 transitions + ion ratio	LC	MS	QQQ	ESI	-	yes	yes	0.005	no	Waters Acquity-Quattro Premier XE	15	Acetone/DCM/PE	No	UPLC Acquity BEH	5	Both	27	Each batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Fluazinam																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
056	Fully identified [SANCO/12495/2011 p.74-80]	<0.01		2 transitions	LC	MSD	QQQ	ESI	-	yes	yes	0.01	yes	Waters Acquity TQD	15	AcN	DSPE	UPLC-BEH C18	10	Both	327	Daily
057	Fully identified [SANCO/12495/2011 p.74-80]	na	na	3 transitions	GC		QQQ	EI	+	yes	yes	0.5 - 0.01	no	Agilent 7890-7000B	10	AcN	PSA	DB-5ms UI	2	Both	330	Daily
058*	Confirmed with second method	30	10	accurate mass + isotope	LC		TOF	ESI	-	no	yes		yes	Agilent 6230 ToF	10	AcN		C18	3	Automatic	510	
059	Fully identified [SANCO/12495/2011 p.74-80]	0.6		2 transitions	LC	MSD	QQQ	ESI	-	yes	yes	0.01	no	Thermo Finnigan TSQ Quantum	10	AcN	DSPE (PSA)	Agilent Eclipse C18 XDB	20	Both	170	Each batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Flufenacet																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
001	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	ABSCIEX	10	AcN	DSPE	C18	5	Automatic	200	Always
002	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.001	no	Xevo TQ-S	10	AcN	no	reversed phase	2.5	Automatic	300	Each batch
004*	Tentative detected	0	0	2 transitions	LC	MSMS	QQQ	ESI	+	yes	yes	0.005	no	Xevo TQ	15	AcN	LLE	C18	10	Both	400	Always
005*	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	no	0.05	no	Agilent 6460	10	20	none	C18	2	Automatic	520	Always
	Confirmed with second method	-0.01	0.5	accurate mass + isotope	LC	MSD	TOF	ESI	+	no	no	0.05	no	Bruker Maxis	10	10	none	C18	2	Automatic	760	Always
007	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes		no	Agilent 6490	15	EIOAc	Dilution	Waters HSS T3	1	Manual	256	Each batch
009	Tentative detected	0.1	<10	accurate mass	LC	MSD	TOF	ESI	+	yes	yes	0.01	yes	Xevo Q-ToF	10	EIAc	none	BEH C18	3	Manual	500	Each batch
010*	Tentative detected				GC	MSD	Q	EI	+	no	no		yes	Agilent	15	Acetone, DCM, PE	none		1	Automatic		Each batch
011	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Waters Xevo TQ	10	Acetone/PE/DCM	Na2SO4	C18	2	Both	289	Each batch
013	Tentative detected	<2	<1	3 diagnostic ions	GC	MSD	Q	EI	+	yes	no		yes	Trace DSQ	15	AcN	PSA/C18	DB5MS	0.8	Both		
014*	Fully identified (SANCO/12495/2011 p.74-80)	2.2		2 transitions	GC	MSD	Q	EI	+	yes	yes	0.01	yes	HP5975C	10	AcN	DSPE	HP5MS	10	Both	approx. 1100	Always
016*	Fully identified (SANCO/12495/2011 p.74-80)	30		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	xevo TQ	10	AcN	DSPE	C18	10	Automatic	82	Daily
017	Confirmed with second method				LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API-3200 QITrap	10	AcN	PSA	Athlantis T3	5	Both	180	Each batch
018	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	yes	yes	0.01-1	no	Ultra Thermo Fichers	10	AcN	PSA	C18	10	Both		Each batch
019	Tentative detected	5	0.005	None	LC	MSD	TOF	ESI	+	yes	yes	0.1	yes	LCT premier XE	15	Acetone, PE, DCM	No	BEH C18	7	Automatic	345	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

Flufenacet																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µl)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
020*	Fully identified [SANCO/12495/2011 p.74-80]	3		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.010	no	Quattro Premier	10	MeOH		C18 BEH	5	Both	220	Each batch
021	Fully identified [SANCO/12495/2011 p.74-80]			2 MRM	LC	MSD	QQQ	ESI	+	yes	yes	0.001-0.01	no	Agilent 6460	10	AcN	DSPE (Q)	YMC Triart C18 2*100 mm 3µm	5	Both	400	Each batch
022	Tentative detected	0.8	0.9	2 transitions	LC	MSD	Q-Orbitrap	ESI	+	yes	yes	no standard solution	yes	Q-Exactive	10	AcN	PSA and SPE with NH ₂ /Carb. separately	BEH C18	10	Both	574	no standard solution. identified by MS database
023	Fully identified [SANCO/12495/2011 p.74-80]	<0.5%	<5 ppm	full scan	GC																	
024*	Confirmed with second method	1	0.0	accurate mass + isotope	LC		Q-TOF	ESI	+	no	yes		yes	Bruker Maxis	10	AcN	MgSO ₄ , PSA	C18	5	Both	450	often
026	Fully identified [SANCO/12495/2011 p.74-80]	0.02	280	3 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7890/7000	10	AcN	25 mg PSA-150 mg MgSO ₄	HP 5 MS	4	Both	355	Daily
030	Fully identified [SANCO/12495/2011 p.74-80]	<±2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	HPLC Agilent 1100, MSD API 3000	10	AcN	DSPE	C18 3µ 50x2mm	10	Both	550	Always
031	Tentative detected	0.4	1.3	2 transitions	LC	MSD	Q-Orbitrap	ESI	+	yes	yes	0.005	yes	Thermo Fisher Q-Exactive	10	AcN	QuEChERS and NH ₂ /Carb	BEH C18	10	Both	512	no standard solution
033*	Tentative detected			364.1>152.0, 364.1>194.2	LC	MSD	QQQ	ESI	+	no	no	0.01-0.1		LC: Agilent 1200, MS: API 4000 QTRAP	10	AcN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Automatic	591	x
034*	Confirmed with second method			full scan	GC	MSD	Q	EI	+	no	no	0.05	yes	Agilent 5973	10	AcN	DSPE (PSA/MgSO ₄)	HP5	5	Both	800	
035	Confirmed with second method			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	API 3200QTrap	50	Acetone	SLE	C18	20	Both	300	Each batch
036	Fully identified [SANCO/12495/2011 p.74-80]				LC	MSD	QQQ	EI		yes	yes			AB SCIEX API 4000	2	ACN/MeOH	QuEChERS					all time
037	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MS/MS	QQQ	ESI	+	yes	yes	from 0.01	no	4000 QTRAP	10	AcN	DSPE	C18 hydro Phenomenex	20	Both		Always

APPENDIX 3. Methods used by participants for detecting pesticides.

Flufenacet																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
038	Confirmed with second method			2 transitions	GC	MSD	QQQ	El	+	yes	yes	0.01	no	Agilent 7000B	5	AcN		HP5-MS-UI	2	Automatic	>500	Daily
039	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API 5500	10	AcN	DSPE	Pursuit XRs Ultra	3	Both	550	Each batch
040	Fully identified [SANCO/12495/2011 p.74-80]	1		2 transitions	GC	MSD	QQQ	El	+	yes	yes	0.005	no	TSQ Quantum Ultra	10	AcN	PSA	RXI 5 Sil MS	2	Both	290	Always
041	Tentative detected	15	unit mass	target + qualifier	GC	MSD	Q	El	+	yes	yes	0.01-0.05	yes	Agilent GC-MSD	10	AcN	PSA	DB5	10	Both	>600	Each batch
042	Fully identified [SANCO/12495/2011 p.74-80]	10		2*MRM	LC	MSD	QQQ	El	+	yes	yes	0.01	no	AB Sciex API 4000	10	AcN	PSA	Phenomenex C18	10	Both	500	Daily
043	Fully identified [SANCO/12495/2011 p.74-80]	0	1	4	GC	MSD	QQQ	El	+	yes	yes	0.01	no	Agilent 7000A	10	AcN	PSA	DB5-MS	2	Both	350	often
044	Tentative detected			2 transitions	LC	MSD	QQQ	ESI	+	no	no		no	Thermo ISQ Quantum	10	AcN	QuEChERS	Xterra-MS-C18	20	Both		
046*	Fully identified [SANCO/12495/2011 p.74-80]	1.2	0	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Varian Pro Star LC + 320-MS	10	AcN	no clean up	Restek Ultra C18	10	Both	363	Each batch
047	Fully identified [SANCO/12495/2011 p.74-80]	5		2 transitions	LC	MSD	QQQ	ESI	+	no	no	0.1 -1	no	API 4000	10	AcN	DSPE	C18	5	Automatic	500	when we find a + result
048	Tentative detected	2			GC	MSD	Q	El		yes	no		yes	Agilent 5973	10	AcN	PSA	HP 5 MS UI	2	Both	approx 600	Each batch
049	Fully identified [SANCO/12495/2011 p.74-80]	6		3 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01-0.5	no	API 4000	10	AcN	no	RP-Amide	10	Both	170	once a month
053	Confirmed with second method	<30	<5	accurate mass + isotope	LC	MSD	Orbitrap	ESI	+	no	no	0.01-0.1	yes	Exactive	10	AcN/1% HAC	none	C18 Atlantis	5	Both	650	Every batch ±100 pesticides
054	Tentative detected	-1.8	20%	3 ions	GC	MSD	Q	El	+	yes	no	0.01	yes	Agilent GC-MSD 7890A/5975C	10	AcN	PSA	HP-5MS	1	Automatic	927	weekly
055*	Fully identified [SANCO/12495/2011 p.74-80]	0.36		full scan	GC	MS	IT	El	+	yes	yes	0.01	yes	Varian 3800 + Saturn 2000	15	Acetone/DCM/PE	No	VF-ms5	5	Both	550	Every quarter

APPENDIX 3. Methods used by participants for detecting pesticides.

Flufenacet																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	M5 Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
056	Fully identified [SANCO/12495/2011 p.74-80]	<0.01		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	Waters Acquity TQD	15	AcN	DSPE	UPLC-BEH C18	10	Both	327	Daily
057	Fully identified [SANCO/12495/2011 p.74-80]	na	na	2 transitions	LC		QQQ	ESI	+	yes	yes	0.5 - 0.01	no	Agilent 1290-6490	10	AcN	PSA	XB-C18	10	Both	220	Daily
058*	Confirmed with second method	30	10	accurate mass + isotope	LC		TOF	ESI	+	no	yes		yes	Agilent 6230 TOF	10	AcN		C18	3	Automatic	510	
059	Fully identified [SANCO/12495/2011 p.74-80]	4.2		3 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Thermo Finnigan TSQ Quantum	10	AcN	DSPE (PSA)	Agilent Eclipse C18 XDB	20	Both	170	Each batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Metamitron																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
001	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	ABSCIEX	10	AcN	DSPE	C18	5	Automatic	200	Always
002	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.001	no	Xevo TQ-S	10	AcN	no	reversed phase	2.5	Automatic	300	Each batch
004*	Tentative detected	0	0	2 transitions	LC	MSMS	QQQ	ESI	+	yes	yes	0.02	no	Xevo TQ	15	AcN	LLE	C18	10	Both	400	Always
005*	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	no	0.05	no	Agilent 6460	10	20	none	C18	2	Automatic	520	Always
	Confirmed with second method	-0.03	0.5	accurate mass + isotope	LC	MSD	TOF	ESI	+	no	no	0.05	no	Bruker Maxis	10	10	none	C18	2	Automatic	760	Always
006*	Fully identified [SANCO/12495/2011 p.74-80]	1	none	2 transitions	GC	MSD	Q	EI	+	yes	yes		no	Agilent 6890/5973	10	AcN	none	HP-5MS	2	Manual	182	Each batch
009	Tentative detected	0.1	<10	accurate mass	LC	MSD	TOF	ESI	+	yes	no	0.01	yes	Xevo Q-ToF	10	EIAC	none	BEH C18	3	Manual	500	Each batch
011	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Waters Xevo TQ	10	Acetone/PE/DCM	Na ₂ SO ₄	C18	2	Both	289	Each batch
013	Tentative detected	<2	0	1 transition	LC	MSD	QQQ	ESI	+	yes	yes	>0.010	no	Waters Quattro Premier	15	AcN	no	BEH C18	3	Both		
014*	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01		ABI 4000 QTRAP	10	AcN	no	C18	55	Both	approx. 660	Always
016*	Fully identified [SANCO/12495/2011 p.74-80]	30		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	xevo TQ	10	AcN	DSPE	C18	10	Automatic	162	Daily
017	Confirmed with second method				LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API-3200 QTrap	10	AcN	PSA	Athlantis T3	5	Both	180	Each batch
019	Tentative detected	5	0.005	None	LC	MSD	TOF	ESI	+	yes	yes	0.1	yes	LCT premier XE	15	Acetone; PE; DCM	No	BEH C18	7	Automatic	345	Daily
020*	Fully identified [SANCO/12495/2011 p.74-80]	3		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.050	no	Quattro Premier	10	MeOH		C18 BEH	5	Both	220	Each batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Metamitron																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µl)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
021	Fully identified [SANCO/12495/2011 p.74-80]			2 MRM	LC	MSD	QQQ	ESI	+	yes	yes	0.001-0.01	no	Agilent 6460	10	AcN	DSPE (Q)	YMC Triart C18 2*100 mm 3µm	5	Both	400	Each batch
023	Fully identified [SANCO/12495/2011 p.74-80]	<2.5%		2 transitions	LC																	
024*	Confirmed with second method	1	0.1	accurate mass + isotope	LC		Q-TOF	ESI	+	no	yes		yes	Bruker Maxis	10	AcN	MgSO ₄ , PSA	C18	5	Both	450	often
026	Fully identified [SANCO/12495/2011 p.74-80]	0.02	495	3 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7890/7000	10	AcN	25 mg PSA-150 mg MgSO ₄	HP 5 MS	4	Both	355	Daily
029	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.05	no	Waters Aquity UPLC system, API 5000 QQQ, Sciex	10	EIOAc	Filter	HSS T3	10	Both	123	Each batch
030	Fully identified [SANCO/12495/2011 p.74-80]	< ± 2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	HPLC Agilent 1100, MSD API 3000	10	AcN	DSPE	C18 3µ 50x2mm	10	Both	method: 550	Always
033*	Fully identified [SANCO/12495/2011 p.74-80]			203.1>103.9, 203.1>175.1	LC	MSD	QQQ	ESI	+	yes	yes	0.008		LC: Agilent 1200, MS: API 4000 QTRAP	10	AcN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Automatic	591	
034*	Fully identified [SANCO/12495/2011 p.74-80]	2		2 transitions	LC		QQQ	ESI	+	yes	yes	0.01	no	Quattro Premier	10	AcN	DSPE (PSA/MgSO ₄)	C18	5	Both	197	
035	Confirmed with second method			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	API 3200QTrap	50	Acetone	SLE	C18	20	Both	300	Each batch
036	Fully identified [SANCO/12495/2011 p.74-80]				LC	MSD	QQQ	EI		yes	yes			AB SCIEX API 4000	2	ACN/MeOH	QuEChERS					all time
037	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MS/MS	QQQ	ESI	+	yes	yes	from 0.01	no	4000 QTRAP	10	AcN	DSPE	C18 hydra Phenomenex	20	Both		Always
038	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 6490	5	AcN		RP-18	5	Automatic	>500	Daily
039	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API 5500	10	AcN	DSPE	Pursuit XRs Ultra	3	Both	550	Each batch
040	Fully identified [SANCO/12495/2011 p.74-80]	6		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	API 3200QTrap	10	AcN	DSPE	Aqua 5u C18	10	Both	205	Always

APPENDIX 3. Methods used by participants for detecting pesticides.

Metamitron																							
Laboratory Code	*NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY)	Standard Solution Frequency
041		Tentative detected	15	unit mass	target + qualifier	GC	MSD	Q	EI	+	yes	yes	0.01-0.05	yes	Agilent GC-MSD	10	AcN	PSA	D85	10	Both	>600	Each batch
042		Fully identified [SANCO/12495/2011 p.74-80]	10		2*MRM	LC	MSD	QQQ	EI	-	yes	yes	0.01	no	AB Sciex API 4000	10	AcN	PSA	Phenomenex C18	10	Both	500	Daily
043		Fully identified [SANCO/12495/2011 p.74-80]	0	1	3	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	XEVEO TQD	10	AcN		C18	3	Both	250	often
044		Tentative detected			2 transitions	LC	MSD	QQQ	ESI	+	no	no		no	Thermo TSQ Quantum	10	AcN	QuEChERS	Xterra-MS C18	20	Both		
046*		Fully identified [SANCO/12495/2011 p.74-80]	-0.6	0	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Varian Pro Star LC + 320-MS	10	AcN	no clean up	Restek Ultra C18	10	Both	363	Each batch
047		Fully identified [SANCO/12495/2011 p.74-80]	5		2 transitions	LC	MSD	QQQ	ESI	+	no	no	0.1 - 1	no	API 4000	10	AcN	DSPE	C18	5	Automatic	500	when we find a + result
049		Tentative detected	15		3 sim ions	GC	MSD	Q	EI	+	no	no	0.05-1.0	yes	Thermo Polaris	10	AcN	no	Elite-5ms	1	Both	800	once a year
053		Fully identified [SANCO/12495/2011 p.74-80]	<20		spectrum	GC	MSD	TOF	EI	+	yes	yes	0.01-0.2	yes	Leco Pegasus IV	10	AcN/1% HAC	DSPE (PSA)	RTX-Cipesticides	10	Automatic	560	Every batch 235 pesticides
053		Confirmed with second method	<30	<5	accurate mass + isotope	LC	MSD	Orbitrap	ESI	+	no	no	0.01-0.1	yes	Exactive	10	AcN/1% HAC	none	C18 Atlantis	5	Both	650	Every batch ±100 pesticides
056		Fully identified [SANCO/12495/2011 p.74-80]	0.01		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	Waters Acquity TQD	15	AcN	DSPE	UPLC-BEH C18	10	Both	327	Daily
057		Fully identified [SANCO/12495/2011 p.74-80]	na	na	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.5 - 0.01	no	Agilent 1290-6490	10	AcN	PSA	XB-C18	10	Both	220	Daily
058*		Confirmed with second method	30	10	accurate mass + isotope	LC		TOF	ESI	+	no	yes		yes	Agilent 6230 ToF	10	AcN		C18	3	Automatic	510	
059		Fully identified [SANCO/12495/2011 p.74-80]	0.2		3 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Thermo Finnigan TSQ Quantum	10	AcN	DSPE (PSA)	Agilent Eclipse C18 XDB	20	Both	170	Each batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Methomyl																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µl)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
001	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	ABSCIEX	10	AcN	DSPE	C18	5	Automatic	200	Always
002	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.002	no	Xevo TQ-S	10	AcN	no	reversed phase	2.5	Automatic	300	Each batch
	Tentative detected		-1		LC	MSD	Q-TOF	ESI	+	no	no		yes	AB Sciex TripleTOF 5600	10	AcN	no	reversed phase	2	Automatic	500	we have not standard
004*	Tentative detected	0	0	2 transitions	LC	MSMS	QQQ	ESI	+	yes	yes	0.005	no	Xevo TQ	15	AcN	LLE	C18	10	Both	400	Always
005*	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 6460	10	20	none	C18	2	Automatic	520	Always
006*	Fully identified [SANCO/12495/2011 p.74-80]	1	none	2 transitions	GC	MSD	Q	EI	+	yes	yes		no	Agilent 6890/5973	10	AcN	none	HP-5MS	2	Manual	182	Each batch
007	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes		no	Agilent 6490	5	EIOAc / Cyclohexan 1/1	solvent exchange	Waters HSS T3	1	Manual	256	Each batch
008	Fully identified [SANCO/12495/2011 p.74-80]	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	AGILENT G-6410A	10	AcN	PSA	ZORBAX-ECLIPSE XDB-C8	20	Automatic	143	Each batch
010*	Tentative detected				LC	MSD	Q	ESI	+	no	yes		yes	Agilent	15	Acetone, DCM, PE	none		1	Automatic		
011	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Waters Xevo TQ	10	Acetone/PE/DCM	Na ₂ SO ₄	C18	2	Both	289	Each batch
012*	Tentative detected	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	Varian 1200L	25	EIOAc	no	C18	5	Both	50	
013	Fully identified [SANCO/12495/2011 p.74-80]	<2	0	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	>0.01	no	Waters Quattro Premier	15	AcN	no	BEH C18	3	Both		
014*	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01		ABI 4000 QTRAP	10	AcN	no	C18	55	Both	approx. 660	Always
015*	Fully identified [SANCO/12495/2011 p.74-80]				LC	MSD	Q-TRAP	ESI	+	no	no		no	AB Sciex 5500	10	AcN	PSA		10	Manual		
016*	Fully identified [SANCO/12495/2011 p.74-80]	30		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	xevo TQ	10	AcN	DSPE	C18	10	Automatic	162	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

Methomyl																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY)	Standard Solution Frequency
017	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API-3200 QTrap	10	AcN	PSA	Athlantis T3	5	Both	180	Each batch
018	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	yes	yes	0.01-1	no	Ultra Thermo Fichers	10	AcN	PSA	C18	10	Both		
020*	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.010	no	Quattro Premier	10	MeOH		C18 BEH	5	Both	220	Each batch
021	Fully identified (SANCO/12495/2011 p.74-80)			2 MRM	LC	MSD	QQQ	ESI	+	yes	yes	0.001-0.01	no	Agilent 6460	10	AcN	DSPE (Q)	YMC Triart C18 2*100 mm 3µm	5	Both	400	Each batch
022	Confirmed with second method	0.3	1.5	2 transitions	LC	MSD	Trap and Q-Orbitrap	ESI	+	yes	yes	0.005	yes	Thermo Fisher LTQ and Q-Exactive	10	AcN	PSA and SPE with NH ₂ /Carb. separately	Shiseido CR 1.4 and BEH C18	10	Both	574	Every 12 injections
023	Fully identified (SANCO/12495/2011 p.74-80)	<2.5%		2 transitions	LC																	
024*	Confirmed with second method	0	1.4	accurate mass + isotope	LC		Q-TOF	ESI	+	no	yes		yes	Bruker Maxis	10	AcN	MgSO ₄ , PSA	C18	5	Both	450	often
026	Fully identified (SANCO/12495/2011 p.74-80)	0.05	620	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 1290/6460	10	AcN	25 mg PSA-150 mg MgSO ₄	HP 5 MS	5	Both	142	Daily
029	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	Waters Aquity UPLC system, API 5000 QQQ, Sciex	10	EIOAc	Filter	HSS T3	10	Both	387	Each batch
030	Fully identified (SANCO/12495/2011 p.74-80)	< ± 2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	HPLC Agilent 1100, MSD API 3000	10	AcN	DSPE	C18 3µ 50x2mm	10	Both	method. 550	Always
031	Confirmed with second method	0.2	0.8	2 transitions	LC	MSD	Trap and Q-Orbitrap	ESI	+	yes	yes	0.005	yes	Thermo fisher LTQ and Q-Exactive	10	AcN	QuEChERS and NH ₂ /Carb	SHISEIDO CR 1.4- BEH C18	10	Both	512	Every 12 injections
033*	Fully identified (SANCO/12495/2011 p.74-80)			1.63.0>88.1, 1.63.0>106.0	LC	MSD	QQQ	ESI	+	yes	yes	0.008		LC, Agilent 1200, MS, API 4000 QTRAP	10	AcN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Automatic	591	
034*	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions	LC		QQQ	ESI	+	yes	yes	0.01	no	Quattro Premier	10	AcN	DSPE (PSA/MgSO ₄)	C18	5	Both	197	

APPENDIX 3. Methods used by participants for detecting pesticides.

Methomyl																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
035	Confirmed with second method			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	API 3200QTrap	50	Acetone	SLE	C18	20	Both	300	Each batch
036	Fully identified [SANCO/12495/2011 p.74-80]				LC	MSD	QQQ	EI		yes	yes			AB SCIEX API 4000	2	ACN/MeOH	QuEChERS					Always
037	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MS/MS	QQQ	ESI	+	yes	yes	from 0.01	no	4000 QTRAP	10	ACN	DSPE	C18 hydro Phenomenex	20	Both		Always
038	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 6490	5	AcN		RP-18	5	Automatic	>500	Daily
039	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API 5500	10	ACN		Luna 5µm PIP	3	Both	550	Each batch
040	Fully identified [SANCO/12495/2011 p.74-80]	4		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	API 3200QTrap	10	ACN	DSPE	Aqua 5µ C18	10	Both	205	Always
041	Tentative detected	30	5	accurate mass	LC	Orbitrap	Orbitrap	ESI	+	yes	yes	0.01-0.05	yes	Thermo Orbitrap	10	ACN	PSA	RP18	10	Both	> 100	Each batch
042	Fully identified [SANCO/12495/2011 p.74-80]	10		2*MRM	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	AB Sciex API 4000	10	ACN	PSA	Phenomenex C18	10	Both	500	Daily
043	Fully identified [SANCO/12495/2011 p.74-80]	0	1	3	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	XEVEO TQD	10	ACN		C18	3	Both	250	often
044	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Thermo ISQ Quantum	10	ACN	QuEChERS	Xttra-MSC18	20	Both		Daily
046*	Fully identified [SANCO/12495/2011 p.74-80]	-1.2	0	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Varian Pro Star LC + 320-MS	10	ACN	no clean up	Restek Ultra C18	10	Both	363	Each batch
047	Fully identified [SANCO/12495/2011 p.74-80]	5		2 transitions	LC	MSD	QQQ	ESI	+	no	no	0.1 -1	no	API 4000	10	ACN	DSPE	C18	5	Automatic	500	when we find a + result
049	Fully identified [SANCO/12495/2011 p.74-80]	6		3 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01-0.5	no	API 4000	10	ACN	no	RP-Amide	10	Both	170	once a month
050	Tentative detected	1308		2 transitions	LC	MSMS	QQQ	ESI	+	no	no	0.01	no	Waters Premier XE	10	ACN	DSPE	HSST3	4	Both	87	Always

APPENDIX 3. Methods used by participants for detecting pesticides.

Methomyl																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
051	Fully identified (SANCO/12495/2011 p.74-80)	± 2.5%	n.a.	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.010	no	API 3200 QTRAP	10	AcN	QuEChERS	C18	20	Both	102	Each batch
053	Fully identified (SANCO/12495/2011 p.74-80)	<30	<5	acc. m/z + isotope+fragment	LC	MSD	Orbitrap	ESI	+	no	no	0.01-0.1	yes	Exactive	10	AcN/1% HAC	none	C18 Atlantis	5	Both	650	Every batch ±100 pesticides
055*	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions + ion ratio	LC	MS	QQQ	ESI	+	yes	yes	0.005	no	Waters Acquity-Quattro Premier XE	15	Acetone/DCM/PE	No	UPLC Acquity BEH	5	Both	240	Each batch
056	Fully identified (SANCO/12495/2011 p.74-80)	0.01		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	Waters Acquity TQD	15	AcN	DSPE	UPLC-BEH C18	10	Both	327	Daily
057	Fully identified (SANCO/12495/2011 p.74-80)	na	na	2 transitions	LC		QQQ	ESI	+	yes	yes	0.5 - 0.01	no	Agilent 1290-6490	10	AcN	PSA	XB-C18	10	Both	220	Daily
058*	Confirmed with second method	30	10	accurate mass + isotope	LC		TOF	ESI	+	no	yes		yes	Agilent 6230 TOF	10	AcN		C18	3	Manual	510	
059	Fully identified (SANCO/12495/2011 p.74-80)	0.6		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Thermo Finnigan TSQ Quantum	10	AcN	DSPE (PSA)	Agilent Eclipse C18 XDB	20	Both	170	Each batch
060	Confirmed with second method	12		3 transitions	LC		QQQ	ESI	+	yes	yes	0.01	no	Varian-320	10	AcN	PSA	C-18	10	Automatic	72	Daily
061	Fully identified (SANCO/12495/2011 p.74-80)	120		2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	Waters XEVO TQMS	10	AcN	PSA	UPLC C18	25	Automatic	120	Each batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Metribuzin																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µL)	Software	Compounds in method (pesticides ONLY)	Standard Solution Frequency
001	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	ABSCIEX	10	AcN	DSPE	C18	5	Automatic	200	Always
002	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.002	no	Xevo TQ-S	10	AcN	no	reversed phase	2.5	Automatic	300	Each batch
004*	Tentative detected	0	0	2 transitions	LC	MSMS	QQQ	ESI	+	yes	yes	0.005	no	Xevo TQ	15	AcN	LLE	C18	10	Both	400	Always
005*	Confirmed with second method	-0.03	0.2	accurate mass + isotope	LC	MSD	TOF	ESI	+	no	no	0.05	no	Bruker Maxis	10	10	none	C18	2	Automatic	760	Always
	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Thermo TSQ Quantum	10	20	none	TG-SSILMS	2	Automatic	520	Always
006*	Fully identified (SANCO/12495/2011 p.74-80)	1	none	2 transitions	GC	MSD	Q	EI	+	yes	yes		no	Agilent 6890/5973	10	AcN	none	HP-5MS	2	Manual	182	Each batch
007	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes		no	Agilent 6490	15	EtOAc	Dilution	Waters HSS T3	1	Manual	256	Each batch
008	Fully identified (SANCO/12495/2011 p.74-80)	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	AGILENT G-6410A	10	AcN	PSA	ZORBAX-ECLIPSE XDB-C8	20	Automatic	143	Each batch
009	Tentative detected	0.1	<10	accurate mass	LC	MSD	TOF	ESI	+	yes	yes	0.01	yes	Xevo Q-ToF	10	EtAc	none	BEH C18	3	Manual	500	Each batch
010*	Tentative detected				GC	MSD	Q	EI	+	no	yes		yes	Agilent	15	Acetone, DCM, PE	none	DB-5	1	Automatic		
011	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Thermo TSQ Quantum GC	10	Acetone/PE/DCM	Na ₂ SO ₄	RXi-5Sil MS	1	Both	201	Each batch
013	Fully identified (SANCO/12495/2011 p.74-80)	<2	0	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	>0.01	no	Waters Quattro Premier	15	AcN	no	BEH C18	3	Both		
015*	Tentative detected				LC	MSD	Orbitrap	ESI	+	no	no		yes	Q-Exactive	10	AcN	PSA		10	Manual	510	
016*	Confirmed with second method	40		3 ions	GC	MSD	IT	EI		yes	yes	0.02	yes	Varian 4000 MS	10	AcN	DSPE	VF 5ms 20M*0.15mm	1	Automatic	239	Daily
017	Confirmed with second method				LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API-3200 QTrap	10	AcN	PSA	Athlantis T3	5	Both	180	Each batch
018	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	yes	yes	0.01-1	yes	Ultra Thermo Fichers	10	AcN	PSA	C18	10	Both		
019	Tentative detected	5	0.005	None	LC	MSD	TOF	ESI	+	yes	yes	0.1	yes	LCT premier XE	1.5	Acetone, PE, DCM	No	BEH C18	7	Automatic	345	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

Metribuzin																							
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency	
020*	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.010	no	Quattro Premier	10	MeOH		C18 BEH	5	Both	220	Each batch	
021	Fully identified (SANCO/12495/2011 p.74-80)			2 MRM	LC	MSD	QQQ	ESI	+	yes	yes	0.001-0.01	no	Agilent 6460	10	AcN	DSPE (Q)	YMC Triart C18 2*100 mm 3µm	5	Both	400	Each batch	
022	Confirmed with second method	0.2	1.2	2 transitions	LC	MSD	Q and Q-Orbitrap	EI	+	yes	yes	0.005	no	Bruker 320MS and Q-Exactive	10	AcN	PSA and SPE with NH ₂ /Carb. separately	DB-5 ms and BEH C18	1(GC) and 10(LC)	Both	208 for GC-MS and 574 for LC-HRMS	Every 12 injections	
023	Fully identified (SANCO/12495/2011 p.74-80)	<0.5%	<5ppm	full scan	GC																		
024*	Confirmed with second method	0	0.5	accurate mass + isotope	LC		Q-TOF	ESI	+	no	yes		yes	Bruker Maxis	10	AcN	MgSO ₄ , PSA	C18	5	Both	450	often	
025*	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	QQQ	EI	+	yes	yes		no		7.5	Acetone,DCM,PE		VF-5	1		86		
026	Fully identified (SANCO/12495/2011 p.74-80)	0.02	470	3 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7890/7000	10	AcN	25 mg PSA-150 mg MgSO ₄	HP 5 MS	4	Both	355	Daily	
028	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	IT	EI		no	no		yes	VARIAN	3	Acetone/PE/DCM		VF-5MS	3	Automatic			
029	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Waters Quattro Micro GC	10	EtOAc	Filter	Rxi-5sil MS/integral-guard Restek	10	Both	387	Each batch	
030	Fully identified (SANCO/12495/2011 p.74-80)	< ± 2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	HPLC Agilent 1100, MSD API 3000	10	AcN	DSPE	C18 3µ 50x2mm	10	Both	method: 550	Always	
031	Confirmed with second method	0.09	1.5	4 transitions	GC	MSD	Q and Q-Orbitrap	ESI	+	yes	yes	0.005	yes	Vaiaan 320 and Q-Exactive	10	AcN	QUECHERS and NH ₂ /Carb	DB5-ms, BEH C18	1(GC) and 10(LC)	Both	512	Every 12 injections	
033*	Fully identified (SANCO/12495/2011 p.74-80)			Match with analytical standard (Match 868, range 0-1000)	GC	MSD	TOF	EI		yes	yes	0.008	yes	GCxGC-TOF MS Pegasus 4D	10	AcN	QUECHERS without PSA	DB5-MS (30 m x 0.25 mm x 0.25 µm) in the 1 st Dimension, BPX-50 (2 m x 0.1 mm x 0.1 µm) in the 2 nd Dimension	10	Automatic			
034*	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC		QQQ	EI	+	yes	yes	0.01	no	Quattro micro	10	AcN	DSPE (PSA/MgSO ₄)	HP5	5	Both	110		

APPENDIX 3. Methods used by participants for detecting pesticides.

Metribuzin																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method (pesticides ONLY)	Standard Solution Frequency
035	Confirmed with second method			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	API 3200QTrap	50	Acetone	SLE	C18	20	Both	300	Each batch
036	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	EI		yes	yes			AB SCIEX API 4000	2	AcN/MeOH	QUECHERS					Always
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	yes	yes	from 0.01	no	4000 QTRAP	10	AcN	DSPE	C18 hydro Phenomenex	20	Both		Always
038	Confirmed with second method			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7000B	5	AcN		HP5-MS-UI	2	Automatic	>500	Daily
039	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API 5500	10	AcN	DSPE	Pursuit XR5 Ultra	3	Both	550	Each batch
040	Fully identified (SANCO/12495/2011 p.74-80)	0	-	2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.005	no	TSQ Quantum Ultra	10	AcN	DSPE	RX1 5 SILMS	2	Both	290	Always
041	Tentative detected	15	unit mass	target + qualifier	GC	MSD	Q	EI	+	yes	yes	0.01-0.05	yes	Agilent GC-MSD	10	AcN	PSA	DB5	10	Both	>600	Each batch
042	Fully identified (SANCO/12495/2011 p.74-80)	10		2 masses	GC	MSD	IT	EI	+	yes	yes	0.01	yes	Varian Saturn 2000	10	AcN	PSA	Vactorfour 5MS	5	Automatic	500	Daily
043	Fully identified (SANCO/12495/2011 p.74-80)	0	1	4	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7000A	10	AcN	PSA	DB5-MS	2	Both	350	often
044	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Thermo TSQ Quantum	10	AcN	QUECHERS	Xterra-MSC18	20	Both		Daily
046*	Fully identified (SANCO/12495/2011 p.74-80)	0.2	0	3 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Varian 3800 GC + 320-MS	10	AcN	DSPE (PSA)	VF-1 ms	3	Both	363	Each batch
047	Fully identified (SANCO/12495/2011 p.74-80)	5		2 transitions	LC	MSD	QQQ	ESI	+	no	no	0.1 - 1	no	API 4000	10	AcN	DSPE	C18	5	Automatic	500	when we find a + result
048	Tentative detected	2			GC	MSD	Q	EI		yes	no		yes	agilent 5973	10	AcN	PSA	HP 5 MS UI	2	Both	approx 600	Each batch
049	Fully identified (SANCO/12495/2011 p.74-80)	6		3 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01-0.5	no	API 4000	10	AcN	no	RP-Amide	10	Both	170	once a month
053	Fully identified (SANCO/12495/2011 p.74-80)	<20		spectrum	GC	MSD	TOF	EI	+	yes	yes	0.01-0.2	yes	Leco Pegasus IV	10	AcN/1% HAC	DSPE (PSA)	RTX-Cipesticides	10	Automatic	560	Every batch 235 pesticides

APPENDIX 3. Methods used by participants for detecting pesticides.

Metribuzin																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method (pesticides ONLY)	Standard Solution Frequency
	Confirmed with second method	<30	<5	accurate mass + isotope	LC	MSD	Orbitrap	ESI	+	no	no	0.01-0.1	yes	Exactive	10	ACN/1% HAC	none	C18 Atlantis	5	Both	650	Every batch ±100 pesticides
054	Tentative detected	-1.3	20%	3 ions	GC	MSD	Q	EI	+	yes	no	0.1	yes	Agilent GC-MSD 7890A/5975c	10	AcN	PSA	HP-5MS	1 µL	Automatic	927	weekly
055*	Fully identified (SANCO/12495/2011 p.74-80)	0.18		2 transitions + ion ratio	GC	MS	QQQ	EI	+	yes	yes	0.005	no	Bruker 451 Scion TQ	1.5	Acetone/DCM/PE	No	UPLC Acquity BEH	5	Both	340	Every quarter
056	Fully identified (SANCO/12495/2011 p.74-80)	<0.01		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	Waters Acquity TQD	1.5	AcN	DSPE	UPLC-BEH C18	10	Both	327	Daily
057	Fully identified (SANCO/12495/2011 p.74-80)	na	na	2 transitions	GC		QQQ	EI	+	yes	yes	0.5 - 0.01	no	Agilent 7890-7000B	10	AcN	PSA	DB-5ms UI	2	Both	330	Daily
058*	Confirmed with second method	30	10	accurate mass + isotope	LC		TOF	ESI	+	no	yes		yes	Agilent 6230 TOF	10	AcN		C18	3	Automatic	510	
059	Fully identified (SANCO/12495/2011 p.74-80)	1.14	0	2 transitions	GC	MSD	IT	EI	+	yes	yes	0.01	no	Varian Saturn 4000	10	AcN	DSPE (PSA)	Restek Rx1-5ms	3	Both	202	Each batch
061	Tentative detected			qualifiers	GC	MSD	IT	EI	+	yes	no		yes	Varian 4000	10	AcN	PSA	capillary VF5-pesticides	5	Manual		

APPENDIX 3. Methods used by participants for detecting pesticides.

Molinate																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µl)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
001	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	yes	AGILENT	10	AcN	DSPE	HP5MS	1	Automatic	200	Always
002	Tentative detected		-1.1		LC	MSD	Q-TOF	ESI	+	no	no		yes	AB Sciex TripleTOF 5600	10	AcN	no	reversed phase	2	Automatic	500	we have not standard
005*	Confirmed with second method	0.01	0.4	accurate mass + isotope	LC	MSD	TOF	ESI	+	no	no	0.05	no	Bruker Maxis	10	10	none	C18	2	Automatic	760	Always
	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MSD	QQQ	EI	+	yes	no	0.05	no	Thermo TSQ Quantum	10	20	none	TG-5SILMS	2	Automatic	520	Always
007	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes		no	Agilent 6490	15	EIOAc	Dilution	Waters HSS T3	1	Manual	256	Each batch
008	Fully identified (SANCO/12495/2011 p.74-80)	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	AGILENT G-6410A	10	AcN	PSA	ZORBAX-ECLIPSE XDB-C8	20	Automatic	143	Each batch
010*	Tentative detected				GC	MSD	Q	EI	+	no	no		yes	Agilent	15	Acetone, DCM, PE	none	DB-5	1	Automatic		
011	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Waters Xevo TQ	10	Acetone/PE/DCM	Na ₂ SO ₄	C18	2	Both	289	Each batch
012*	Tentative detected	0		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.005	no	Agilent7890/Agilent7000	25	EIOAc	no	HP5MS	2	Both	310	
014*	Fully identified (SANCO/12495/2011 p.74-80)	2.5		2 transitions	GC	MSD	Q	EI	+	yes	yes	0.01	yes	HP5975C	10	AcN	DSPE	HP5MS	10	Both	approx. 1100	Always
017	Fully identified (SANCO/12495/2011 p.74-80)	-1		AMDIS Match 94	GC	MSD	Q	EI	+	no	no	NA	yes	AGILENT TECHNOLOGIES 7890A // G3440A	10	AcN	PSA	HP-5MS	20	Automatic	560	
018	Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	QQQ	EI	+	yes	yes	0.01-1 mg/kg	yes	TSQ Thermo Fichers	10	AcN	PSA	C18	1	Both		
021	Fully identified (SANCO/12495/2011 p.74-80)			2 MRM	LC	MSD	QQQ	ESI	+	yes	yes	0.001-0.01	no	Agilent 6460	10	AcN	DSPE (Q)	YMC Triart C18 2*100 mm 3µm	5	Both	400	Each batch
022	Confirmed with second method	0.4	1.4	2 transitions	LC	MSD	Q and Q-Orbitrap	EI	+	yes	yes	0.005	no	Bruker 320MS and Q-Exactive	10	AcN	PSA and SPE with NH ₂ /Carb. separately	DB-5 ms and BEH C18	1(GC) and 10(LC)	Both	208 for GC-MS and 574 for LC-HRMS	Every 12 injections

APPENDIX 3. Methods used by participants for detecting pesticides.

Molinate																							
Laboratory Code	*NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
024*		Confirmed with second method	3	0.3	accurate mass + isotope	LC		Q-TOF	ESI	+	no	yes		yes	Bruker Maxis	10	AcN	MgSO ₄ , PSA	C18	5	Both	450	often
025*		Tentative detected				GC	MSD	QQQ	EI	+	no	no		no		7.5	Acetone/DCM/PE	no	VF-5	1			
026		Fully identified (SANCO/12495/2011 p.74-80)	0.02	535	3 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7890/7000	10	AcN	25 mg PSA-150 mg MgSO ₄	HP 5 MS	4	Both	355	Daily
028		Fully identified (SANCO/12495/2011 p.74-80)				GC	MSD	IT	EI		no	no		yes	VARIAN	3	Acetone/PE/DCM		VF-5MS	3	Automatic		
029		Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.05	no	Waters Quattro Micro GC	10	EIOAC	Filter	Rxi-5sil MS/integra-guard Restek	10	Both	123	Each batch
030		Fully identified (SANCO/12495/2011 p.74-80)	< ± 2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	no	0.01	no	HPLC Agilent 1100, MSD API 3000	10	AcN	DSPE	C18 3µ 50x2mm	10	Both	method: 550	Always
031		Confirmed with second method	0	1.0	4 transitions	GC	MSD	Q and Q-Orbitrap	ESI	+	yes	yes	0.005	yes	Varian 320 and Q-Exactive	10	AcN	QuEChERS and NH ₂ /Carb	DB5-ms, BEH C18	1(GC) and 10(LC)	Both	512	Every 12 injections
033*		Tentative detected			Match with NIST Library (Match 884, range 0-1000)	GC	MSD	TOF	EI		no	no	0.01-0.1	yes	GCXGC-TOF MS Pegasus 4D	10	AcN	QuEChERS without PSA	DB5-MS (30 m x 0.25 mm x 0.25 µm) in the 1 st Dimension, BPX-50 (2 m x 0.1 mm x 0.1 µm) in the 2 nd Dimension	10	Automatic		
034*		Confirmed with second method			3 transitions	LC	MSD	QQQ	ESI	+	no	no	0.01	no	Quattro Premier	10	AcN	DSPE (PSA/MgSO ₄)	C18	5	Both		
037		Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	yes	yes	from 0.01	no	4000 QTRAP	10	AcN	DSPE	C18 hydro Phenomenex	20	Both		Always
038		Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 6490	5	AcN		RP-18	5	Automatic	>500	Daily
039		Confirmed with second method			2 transitions	LC	MSD	TOF	ESI	+	no	no	NA	yes	Bruker Maxis	10	AcN		Acclaim RSLC 120	5	Both	700	Each batch
041		Tentative detected	15		target + qualifier	GC	MSD	Q	EI	+	yes	yes	0.01-0.05	yes	Agilent GC-MSD	10	AcN	PSA	DB5	10	Both	>600	Each batch
042		Fully identified (SANCO/12495/2011 p.74-80)	10		2*MRM	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	AB Sciex API 4000	10	AcN	PSA	Phenomenex C18	10	Both	500	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

Molinate																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µl)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
043	Fully identified (SANCO/12495/2011 p.74-80)	0	1	3	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	XEVEO TOQ	10	AcN		C18	3	Both	250	often
046*	Fully identified (SANCO/12495/2011 p.74-80)	0.2	0	3 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Varian 3800 GC + 320-MS	10	AcN	DSPE (PSA)	VF-1 ms	3	Both	363	Each batch
047	Fully identified (SANCO/12495/2011 p.74-80)	5		2 transitions	LC	MSD	QQQ	ESI	+	no	no	0.1 -1	no	API 4000	10	AcN	DSPE	C18	5	Automatic	500	when we find a + result
049	Fully identified (SANCO/12495/2011 p.74-80)	15		4 sim ions	GC	MSD	Q	EI	+	no	no	0.05-1.0	yes	Thermo DSQ	10	AcN	no	DB-5ms	1	Both	800	once a month
053	Fully identified (SANCO/12495/2011 p.74-80)	<20		spectrum	GC	MSD	TOF	EI	+	yes	yes	0.01-0.2	yes	Leco Pegasus IV	10	AcN/1% HAC	DSPE (PSA)	RTX-C18pesticides	10	Automatic	560	Every batch 235 pesticides
	Confirmed with second method	no RT	<5	acc. m/z + isotope (only peak in chrom)	LC	MSD	Orbitrap	ESI	+	no	no	0.01-0.1	yes	Exactive	10	AcN/1% HAC	none	C18 Allantis	5	Both	650	Every batch ±100 pesticides
054	Tentative detected	-1.5	20%	3 ions	GC	MSD	Q	EI	+	yes	no	0.05	yes	Agilent GC-MSD 7890A/5975C	10	AcN	PSA	HP-5MS	1	Automatic	927	weekly
056	Fully identified (SANCO/12495/2011 p.74-80)	0.02		3 transitions	GC	MSD	Q	EI	+	yes	yes	0.01	yes	Agilent 7890A-5975C	15	AcN	DSPE	HP-5MS	10	Both	220	Daily
058*	Confirmed with second method	30	10	accurate mass + isotope	LC		TOF	ESI	+	no	yes		yes	Agilent 6230 TOF	10	AcN		C18	3	Automatic	510	
059	Tentative detected	1.0	0		GC	MSD	IT	EI	+	yes	yes	0.01	yes	Varian Saturn 4000	10	AcN	DSPE (PSA)	Restek Rxi-5ms	3	Both	202	Each batch
060	Confirmed with second method	12			GC		IT	CI	+	yes	yes	0.01	yes	VARIAN 4000	10	AcN	PSA	5MS	10	Automatic	121	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

Oxamyl																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
001	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	ABSCIEX	10	AcN	DSPE	C18	5	Automatic	200	Always
002	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.001	no	Xevo TQ-S	10	AcN	no	reversed phase	2.5	Automatic	300	Each batch
004*	Tentative detected	0	0	2 transitions	LC	MSMS	QQQ	ESI	+	yes	yes	0.01	no	Xevo TQ	15	AcN	LLE	C18	10	Both	400	Always
005*	Fully identified (SANCO/12495/2011 p.74-80) Confirmed with second method	0.01	1.5	2 transitions accurate mass + isotope	LC	MSD	TOF	ESI	+	no	no	0.05	no	Bruker Maxis	10	20	none	C18	2	Automatic	520	Always
006*	Fully identified (SANCO/12495/2011 p.74-80)	1	none	2 transitions	GC	MSD	Q	EI	+	yes	yes		no	Agilent 6890/5973	10	AcN	none	HP-5MS	2	Manual	182	Each batch
007	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes		no	Agilent 6490	5	EIOAc / Cyclohexan 1/1	solvent exchange	Waters HSS T3	1	Manual	256	Each batch
008	Fully identified (SANCO/12495/2011 p.74-80)	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	AGILENT G-6410A	10	AcN	PSA	ZORBAX-ECLIPSE XDB-C8	20	Automatic	143	Each batch
009	Tentative detected	0.1	<10	accurate mass	LC	MSD	TOF	ESI	+	yes	yes	0.01	yes	Xevo Q-ToF	10	EIAc	none	BEH C18	3	Manual	500	Each batch
010*	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	Q	ESI	+	no	no		yes	Agilent	15	Acetone, DCM, PE	none		1	Automatic		
011	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Waters Xevo TQ	10	Acetone/PE/DCM	Na ₂ SO ₄	C18	2	Both	289	Each batch
012*	Tentative detected	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	Varian 1200L	25	EIOAc	no	C18	5	Both	50	
013	Fully identified (SANCO/12495/2011 p.74-80)	<2	0	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	>0.010	no	Waters Quattro Premier	15	AcN	no	BEH C18	3	Both		
014*	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01		ABI 4000 QTRAP	10	AcN	no	C18	55	Both	approx. 660	Always
015*	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	Q-TRAP	ESI	+	no	no		no	AB Sciex 5500	10	AcN	PSA		10	Manual		

APPENDIX 3. Methods used by participants for detecting pesticides.

Oxamyl																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µl)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
016*	Fully identified (SANCO/12495/2011 p.74-80)	30		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	xevo TQ	10	AcN	DSPE	C18	10	Automatic	142	Daily
017	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API-3200 Qtrap	10	AcN	PSA	Athlantis T3	5	Both	180	Each batch
018	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	yes	yes	0.01-1	no	Ultra Thermo Fishers	10	AcN	PSA	C18	10	Both		
019	Tentative detected	5	0.005	None	LC	MSD	TOF	ESI	+	yes	yes	0.01	yes	LCT premier XE	15	Acetone; PE; DCM	No	BEH C18	7	Automatic	345	Daily
020*	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.010	no	Quattro Premier	10	MeOH		C18 BEH	5	Both	220	Each batch
021	Fully identified (SANCO/12495/2011 p.74-80)			2 MRM	LC	MSD	QQQ	ESI	+	yes	yes	0.001-0.01	no	Agilent 6460	10	AcN	DSPE (Q)	YMC Triart C18 2*100 mm-3µm	5	Both	400	Each batch
022	Confirmed with second method	0.4	1.8	2 transitions	LC	MSD	Trap and Q-Orbitrap	ESI	+	yes	yes	0.01	yes	Thermo fisher LTQ and Q-Exactive	10	AcN	PSA and SPE with NH ₃ /Carb. separately	Shiseido CR 1.4 and BEH C18	10	Both	574	Every 12 injections
023	Fully identified (SANCO/12495/2011 p.74-80)	<2.5%		2 transitions	LC																	
024*	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC		QQQ	ESI	+	yes	yes		yes	Waters Ultima	10	AcN	MgSO ₄ , PSA	C18	10	Automatic		
025*	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	yes	yes		no		7.5	AcN, DCM	no	C18	5	Automatic		
026	Fully identified (SANCO/12495/2011 p.74-80)	0.05	460	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 1290/6460	10	AcN	25 mg PSA-150 mg MgSO ₄	HP 5 MS	5	Both	142	Daily
029	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	Waters Aquity UPLC system, API 500 QQQ, Sciex	10	EIOAc	Filter	HSS T3	10	Both	387	Each batch
030	Fully identified (SANCO/12495/2011 p.74-80)	< ± 2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	HPLC Agilent 1100, MSD API 3000	10	AcN	DSPE	C18 3µ 50x2mm	10	Both	550	Always
031	Confirmed with second method	0.07	0.9	2 transitions	LC	MSD	Trap and Q-Orbitrap	ESI	+	yes	yes	0.01	yes	Thermo fisher LTQ and Q-Exactive	10	AcN	GuEChERS and NH ₃ /Carb	SHISEIDO CR 1.4; BEH C18	10	Both	512	Every 12 injections
033*	Fully identified (SANCO/12495/2011 p.74-80)			237.0>71.9, 237.0>90.0	LC	MSD	QQQ	ESI	+	yes	yes	0.002		LC; Agilent 1200; MS; API 4000 QTRAP	10	AcN	GuEChERS without PSA	Supelco Discovery C18; 150 µm x 2.1 mm; 5 µm	2	Automatic	591	

APPENDIX 3. Methods used by participants for detecting pesticides.

Oxamyl																							
Laboratory Code	*NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
034*		Fully identified (SANCO/12495/2011 p.74-80)	2		2 transitions	LC		QQQ	ESI	+	yes	yes	0.01	no	Quattro Premier	10	AcN	DSPE (PSA/MgSO ₄)	C18	5	Both	197	
035		Confirmed with second method			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	API 3200QITrap	50	Acetone	SLE	C18	20	Both	300	Each batch
037		Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	yes	yes	from 0.01	no	4000 QITRAP	10	AcN	DSPE	C18 hydro Phenomenex	20	Both		Always
038		Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 6490	5	AcN		RP-18	5	Automatic	>500	Daily
039		Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API 5500	10	AcN		Luna 5µm Pfp	3	Both	550	Each batch
040		Fully identified (SANCO/12495/2011 p.74-80)	2		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	API 3200QITrap	10	AcN	DSPE	Aqua 5u C18	10	Both	205	Always
041		Tentative detected	30	5	accurate mass	LC	Orbitrap	Orbitrap	ESI	+	yes	yes	0.01-0.05	yes	Thermo Orbitrap	10	AcN	PSA	RP18	10	Both	>100	Each batch
042		Fully identified (SANCO/12495/2011 p.74-80)	10		2*MRM	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	AB Sciex API 4000	10	AcN	PSA	Phenomenex C18	10	Both	500	Daily
043		Fully identified (SANCO/12495/2011 p.74-80)	0	1	3	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	XEVEO TQD	10	AcN		C18	3	Both	250	often
044		Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Thermo TSQ Quantum	10	AcN	GuEChERS	Xterra-MSC18	20	Both		Daily
046*		Fully identified (SANCO/12495/2011 p.74-80)	-1.8	0	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Varian Pro Star LC + 320-MS	10	AcN	no clean up	Restek Ultra C18	10	Both	363	Each batch
047		Fully identified (SANCO/12495/2011 p.74-80)	5		2 transitions	LC	MSD	QQQ	ESI	+	no	no	0.1 -1	no	API 4000	10	AcN	DSPE	C18	5	Automatic	500	when we find a + result
049		Fully identified (SANCO/12495/2011 p.74-80)	6		3 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01-0.5	no	API 4000	10	AcN	no	RP-Amide	10	Both	170	once a month
050		Tentative detected	290		2 transitions	LC	MSMS	QQQ	ESI	+	no	no	0.01	no	Waters Premier XE	10	AcN	DSPE	HSS13	4	Both	87	Always
051		Fully identified (SANCO/12495/2011 p.74-80)	± 2.5%	n.a.	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.010	no	API 3200 QITRAP	10	AcN	GuEChERS	C18	20	Both	102	Each batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Oxamyl																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µl)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
053	Fully identified (SANCO/12495/2011 p.74-80)	<30	<5	acc. m/z + isotope+fragment	LC	MSD	Orbitrap	ESI	+	no	no	0.01-0.1	yes	Exactive	10	AcN/1% HAC	none	C18 Atlantis	5	Both	650	Every batch ±100 pesticides
055*	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions + ion ratio	LC	MS	QQQ	ESI	+	yes	yes	0.005	no	Waters Acquity-Quattro Premier XE	15	Acetone/DCM/PE	No	UPLC Acquity BEH	5	Both	240	Every quarter
056	Fully identified (SANCO/12495/2011 p.74-80)	0.02		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	Waters Acquity TOF	15	AcN	DSPE	UPLC-BEH C18	10	Both	327	Daily
057	Fully identified (SANCO/12495/2011 p.74-80)	na	na	2 transitions	LC		QQQ	ESI	+	yes	yes	0.5 - 0.01	no	Agilent 1290-6490	10	AcN	PSA	XB-C18	10	Both	220	Daily
058*	Confirmed with second method	30	10	accurate mass + isotope	LC		TOF	ESI	+	no	yes		yes	Agilent 6230 TOF	10	AcN		C18	3	Automatic	510	
059	Fully identified (SANCO/12495/2011 p.74-80)	0.5		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Thermo Finnigan TSQ Quantum	10	AcN	DSPE (PSA)	Agilent Eclipse C18 XDB	20	Both	170	Each batch
060	Confirmed with second method	12		3 transitions	LC		QQQ	ESI	+	yes	yes	0.01	no	Varian-320	10	AcN	PSA	C-18	10	Automatic	72	Daily
061	Fully identified (SANCO/12495/2011 p.74-80)	120		2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	Waters XEVO TOIMS	10	AcN	PSA	UPLC C18	25	Automatic	120	Each batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Picloram																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyser	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
002	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	Xevo TQ-S	10	AcN	no	reversed phase	2.5	Automatic	300	Each batch
011	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions	LC	MSD	QQQ	ESI	-	yes	yes	0.05	no	Waters Xevo TQ	10	MeOH	Na2SO4	UPLC HSS C18	5	Both	22	Each batch
026	Fully identified [SANCO/12495/2011 p.74-80]	0.02	41.5	3 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7890/7000	10	AcN	25 mg PSA-150 mg MgSO4	HP 5 MS	4	Both	355	Daily
033*	Fully identified [SANCO/12495/2011 p.74-80]			238.7>194.7, 240.7>196.6	LC	MSD	QQQ	ESI	-	yes	yes	0.01		LC: Agilent 1200, MS: API 4000 QTRAP	10	AcN	GuChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Automatic	591	x
035	Confirmed with second method			2 transitions	LC	MSD	QQQ	EI	-	yes	yes	0.005	no	API 2000	50	Acetone	SPE	C18	100	Both	18	Each batch
038	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	-	yes	yes	0.01	no	Agilent 6490	5	AcN		RP-18	5	Automatic	>500	Daily
042	Fully identified [SANCO/12495/2011 p.74-80]	10		2*MRM	LC	MSD	QQQ	EI	-	yes	yes	0.01	no	AB Sciex API 4000	10	AcN	PSA	Phenomenex C18	10	Both	500	Daily
043	Fully identified [SANCO/12495/2011 p.74-80]	0	1	3	GC	MSD	Q	CI	-	no	no	0.01	no	5975C	10	AcN		DB5-MS	2	Both	100	often
044	Tentative detected				GC	MSD	IT	EI	+	no	no		yes	Thermo Polaris Q	10	AcN	GuChERS	DB-5MS	5	Both		
053	Fully identified [SANCO/12495/2011 p.74-80]	<30	<5	acc. m/z + isotope+fragment	LC	MSD	Orbitrap	ESI	+	no	no	0.01-0.1	yes	Exactive	10	AcN/1% HAC	none	C18 Atlantis	5	Both	650	Every batch ±100 pesticides

APPENDIX 3. Methods used by participants for detecting pesticides.

Picolinafen																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µl)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
002	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.001	no	Xevo TQ-S	10	AcN	no	reversed phase	2.5	Automatic	300	Each batch
004*	Tentative detected	0	0	2 transitions	LC	MSMS	QQQ	ESI	+	yes	yes		no	Xevo TQ	15	AcN	LLE	C18	10	Both	400	Always
005*	Confirmed with second method	0.03	0.2	accurate mass + isotope	LC	MSD	TOF	ESI	+	no	no	0.05	no	Bruker Maxis	10	10	none	C18	2	Automatic	760	Always
	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MSD	QQQ	EI	+	yes	no	0.05	no	Thermo TSQ Quantum	10	20	none	TG-5SILMS	2	Automatic	520	Always
006*	Fully identified (SANCO/12495/2011 p.74-80)	1	none	2 transitions	GC	MSD	Q	EI	+	yes	yes		no	Agilent 6890/5973	10	AcN	none	HP-5MS	2	Manual	182	Each batch
009	Tentative detected	0.1	<10	accurate mass	LC	MSD	TOF	ESI	+	yes	no	0.01	yes	Xevo Q-ToF	10	EtAc	none	BEH C18	3	Manual	500	Each batch
010*	Tentative detected				GC	MSD	Q	EI	+	no	no		yes	Agilent	15	Acetone-DCM:PE	none		1	Automatic		Each batch
011	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Waters Xevo TQ	10	Acetone/PE/DCM	Na2SO4	C18	2	Both	289	Each batch
012*	Tentative detected	0		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.005	no	Agilent 7890/Agilent 7000	25	EtOAc	no	HP5MS	2	Both	310	
013	Tentative detected	<2	<1	3 diagnostic ions	GC	MSD	Q	EI	+	yes	no		yes	Trace DSQ	15	AcN	PSA/C18	DB5MS	0.8	Both		
014*	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01		Agilent 7000 QQQ	10	AcN	DSPE	HP-5MS	10	Both	approx. 1100	Always
016*	Fully identified (SANCO/12495/2011 p.74-80)	30			LC	MSD	QQQ	ESI	+	yes	no		no	xevo TQ	10	AcN	DSPE	C18	10	Automatic	162	
017	Fully identified (SANCO/12495/2011 p.74-80)	-0.3		AMDIS match 79	GC	MSD	Q	EI	+	no	no	NA	yes	AGILENT TECHNOLOGIES 7890A // G3440A	10	AcN	PSA	HP-5MS	20	Automatic	560	
019	Tentative detected	5	0.005	None	LC	MSD	TOF	ESI	+	yes	yes	0.1	yes	LCT premier XE	15	Acetone: PE: DCM	No	BEH C18	7	Automatic	345	Daily
021	Fully identified (SANCO/12495/2011 p.74-80)			2 MRM	LC	MSD	QQQ	ESI	+	yes	yes	0.001-0.01	no	Agilent 6460	10	AcN	DSPE (Q)	YMC Triaq C18 2*100 mm 3µm	5	Both	400	Each batch
022	Confirmed with second method	0.2	1.1	2 transitions	LC	MSD	Q and Q-Orbitrap	EI	+	yes	yes	0.005	no	Bruker 320MS and Q-Exactive	10	AcN	PSA and SPE with NH ₂ /Carb. separately	DB-5 ms and BEH C18	1(GC) and 10(LC)	Both	208 for GC-MS and 574 for LC-HRMS	Every 12 injections

APPENDIX 3. Methods used by participants for detecting pesticides.

Picolinafen																								
Laboratory Code	*NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency	
023		Fully identified (SANCO/12495/2011 p.74-80)	<0.5%	<5ppm	full scan	GC																		
024*		Confirmed with second method	0	0.2	accurate mass + isotope	LC	Q-TOF	ESI	+	+	no	yes		yes	Bruker Maxis	10	AcN	MgSO ₄ , PSA	C18	5	Both	450	often	
026		Fully identified (SANCO/12495/2011 p.74-80)	0.02	270	3 transitions	GC	QQQ	EI	+	+	yes	yes	0.01	no	Agilent 7890/7000	10	AcN	25 mg PSA-150 mg MgSO ₄	HP 5 MS	4	Both	355	Daily	
029		Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	GC	QQQ	EI	+	+	yes	yes	0.05	no	Waters Quattro Micro GC	10	EIOAC	Filter	Rxi-5sil MS/integra-guard Restek	10	Both	123	Each batch	
030		Fully identified (SANCO/12495/2011 p.74-80)	< ± 2.5%		2 transitions	LC	QQQ	ESI	+	+	yes	yes	0.01	no	HPLC Agilent 1100, MSD API 3000	10	AcN	DSPE	C18 3µ 50x2mm	10	Both	550	Always	
031		Confirmed with second method	0	1.5	4 transitions	GC	G and Q-Orbitrap	ESI	+	+	yes	yes	0.005	yes	Varian 320 and Thermo fisher Q-Exactive	10	AcN	QuEChERS and NH ₂ /Carb	DB5-ms, BEH C18	1(GC) and 10(LC)	Both	512	Every 12 injections	
033*		Tentative detected			377.1>145.0, 377.1>238.1	LC	QQQ	ESI	+	+	no	no	0.01-0.1		LC: Agilent 1200, MS: API 4000 QTRAP	10	AcN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Automatic	591		
034*		Confirmed with second method			3 transitions	LC	QQQ	ESI	+	+	no	no	0.01	no	Quattro Premier	10	AcN	DSPE (PSA/MgSO ₄)	C18	5	Both			
037		Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	yes	yes	from 0.01	no	4000 QTRAP	10	AcN	DSPE	C18 hydra Phenomenex	20	Both		Always	
038		Confirmed with second method			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7000B	5	AcN		HP5-MS-UI	2	Automatic	>500	Daily	
039		Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API 5500	10	AcN	DSPE	Pursuit XRs Ultra	3	Both	550	Each batch	
041		Tentative detected	15	unit mass	target + qualifier	GC	MSD	Q	EI	+	yes	yes	0.01-0.05	yes	Agilent GC-MSD	10	AcN	PSA	DB5	10	Both	>600	Each batch	
042		Fully identified (SANCO/12495/2011 p.74-80)	10		2masses	GC	MSD	IT	EI	+	yes	yes	0.01	yes	Varian Saturn 2000	10	AcN	PSA	VactorFour 5MS	5	Automatic	500	Daily	
043		Fully identified (SANCO/12495/2011 p.74-80)	0	1	4	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7000A	10	AcN	PSA	DB5-MS	2	Both	350	often	
044		Tentative detected			2 transitions	LC	MSD	QQQ	ESI	+	no	no		no	Thermo ISQ Quantum	10	AcN	QuEChERS	Xterra-MS-C18	20	Both			

APPENDIX 3. Methods used by participants for detecting pesticides.

Picolinafen																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
046*	Fully identified (SANCO/12495/2011 p.74-80)	0	0		GC	MSD	QQQ	EI	+	yes	yes	0.01	yes	Varian 3800 GC + 320-MS	10	AcN	DSPE (PSA)	VF-1 ms	3	Both	363	Each batch
047	Fully identified (SANCO/12495/2011 p.74-80)	5		2 transitions	LC	MSD	QQQ	ESI	+	no	no	0.1 -1	no	API 4000	10	AcN	DSPE	C18	5	Automatic	500	when we find a + result
048	Tentative detected	2			GC	MSD	Q	EI		yes	no		yes	agilent 5973	10	AcN	PSA	HP 5 MS UI	2	Both	approx 600	Each batch
049	Tentative detected	15		3 sim ions	GC	MSD	Q	EI	+	no	no	0.05-1.0	yes	Thermo Polaris	10	AcN	no	Elite-5ms	1	Both	800	once a year
053	Fully identified (SANCO/12495/2011 p.74-80)	<20		spectrum	GC	MSD	TOF	EI	+	yes	yes	0.01-0.2	yes	Leco Pegasus IV	10	AcN/1% HAC	DSPE (PSA)	RTX-CIpesticides	10	Automatic	560	Every batch 235 pesticides
	Confirmed with second method	<30	<5	accurate mass + isotope	LC	MSD	Orbitrap	ESI	+	no	no	0.01-0.1	yes	Exactive	10	AcN/1% HAC	none	C18 Atlantis	5	Both	650	Every batch ±100 pesticides
054	Tentative detected	-2	20%	3 ions	GC	MSD	Q	EI	+	yes	no	0.05	yes	Agilent GC-MSD 7890A/5975C	10	AcN	PSA	HP-5MS	1	Automatic	927	weekly
055*	Confirmed with second method			full scan	GC	MS	IT	EI	+	yes	yes	0.01	yes	Varian 3800 + Saturn 2000	15	Acetone/DCM/PE	No	VF-ms5	5	Both	550	Every quarter
056	Fully identified (SANCO/12495/2011 p.74-80)	0.02		3 transitions	GC	MSD	Q	EI	+	yes	yes	0.01	yes	Agilent 7890A-5975C	15	AcN	DSPE	HP-5MS	10	Both	220	Daily
058*	Confirmed with second method	30	10	accurate mass + isotope	LC		TOF	ESI	+	no	yes		yes	Agilent 6230 TOF	10	AcN		C18	3	Automatic	510	

APPENDIX 3. Methods used by participants for detecting pesticides.

Propamocarb																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
001	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	ABSCIEX	10	AcN	DSPE	C18	5	Automatic	200	Always
002	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.001	no	Xevo TQ-S	10	AcN	no	reversed phase	2.5	Automatic	300	Each batch
004*	Tentative detected	0	0	2 transitions	LC	MSMS	QQQ	ESI	+	yes	yes	0.005	no	Xevo TQ	15	AcN	LLE	C18	10	Both	400	Always
005*	Fully identified (SANCO/12495/2011 p.74-80) Confirmed with second method	0.08	0.3	2 transitions accurate mass + isotope	LC	MSD	TOF	ESI	+	no	no	0.01	no	Agilent 6460	10	20	none	C18	2	Automatic	520	Always
006*	Fully identified (SANCO/12495/2011 p.74-80)	1	none	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes		no	Agilent 6410	10	AcN	none	Eclipse XDB-C18	4	Manual	143	Each batch
007	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes		no	Agilent 6490	15	EIOAC	Dilution	Waters HSS T3	1	Manual	256	Each batch
008	Fully identified (SANCO/12495/2011 p.74-80)	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	AGILENT G-6410A	10	AcN	PSA	ZORBAX-ECLIPSE XDB-C8	20	Automatic	143	Each batch
009	Tentative detected	0.1	<10	accurate mass	LC	MSD	TOF	ESI	+	yes	yes	0.01	yes	Xevo Q-ToF	10	EtAc	none	BEH C18	3	Manual	500	Each batch
011	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Waters Xevo TQ	10	MeOH	Na ₂ SO ₄	UPLC HSS T3	2	Both	50	Each batch
013	Fully identified (SANCO/12495/2011 p.74-80)	<2	0	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	>0.01	no	Waters Quattro Premier	15	AcN	no	BEH C18	3	Both		
014*	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01		ABI 4000 QTRAP	10	AcN	no	C18	55	Both	approx. 660	Always
015*	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	Q-TRAP	ESI	+	no	no		no	AB Sciex 5500	10	AcN	PSA		10	Manual		
016*	Fully identified (SANCO/12495/2011 p.74-80)	30		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	xevo TQ	10	AcN	DSPE	C18	10	Automatic	162	Daily
017	Confirmed with second method				LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API-3200 QTrap	10	AcN	PSA	Athlantis T3	5	Both	180	Each batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Propamocarb																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injecton Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
018	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	yes	yes	0.01-1	no	Ultra Thermo Fichers	10	AcN	PSA	C18	10	Both		
020*	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.010	no	Quattro Premier	10	MeOH		C18 BEH	5	Both	220	Each batch
021	Fully identified (SANCO/12495/2011 p.74-80)			2 MRM	LC	MSD	QQQ	ESI	+	yes	yes	0.001-0.01	no	Agilent 6460	10	AcN	DSPE (Q)	YMC Triart C18 2*100 mm 3µm	5	Both	400	Each batch
022	Confirmed with second method	0.3	1.2	2 transitions	LC	MSD	Trap and Q-Orbitrap	ESI	+	yes	yes	0.01	yes	Thermo fisher LTQ and Q-Exactive	10	AcN	PSA and SPE with NH ₂ /Carb. separately	Shiseido CR 1.4 and BEH C18	10	Both	574	Every 12 injections
023	Fully identified (SANCO/12495/2011 p.74-80)	<2.5%		2 transitions	LC																	
024*	Confirmed with second method	8	0.6	accurate mass + isotope	LC		Q-TOF	ESI	+	no	yes		yes	Bruker Maxis	10	AcN	MgSO ₄ , PSA	C18	5	Both	450	often
025*	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	yes	yes		no		7.5	AcN, DCM	no	C18	5	Automatic		
026	Fully identified (SANCO/12495/2011 p.74-80)	0.06	535	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 1290/6460	10	AcN	25 mg PSA-150 mg MgSO ₄	HP 5 MS	5	Both	142	Daily
029	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	Waters Aquity UPLC system, API 5000 QQQ, Sciex	10	EIOAC	Filter	HSS T3	10	Both	387	Each batch
030	Fully identified (SANCO/12495/2011 p.74-80)	< ± 2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	HPLC Agilent 1100, MSD API 3000	10	AcN	DSPE	C18 3µ 50x2mm	10	Both	550	Always
031	Confirmed with second method	0.7	1.4	2 transitions	LC	MSD	Trap and Q-Orbitrap	ESI	+	yes	yes	0.01	yes	Thermo fisher LTQ and Q-Exactive	10	AcN	QuEChERS and NH ₂ /Carb	SHISEIDO CR 1.4- BEH C18	10	Both	512	Every 12 injections
033*	Fully identified (SANCO/12495/2011 p.74-80)			188.9>102.0, 188.9>144.1	LC	MSD	QQQ	ESI	+	yes	yes	0.002		LC: Agilent 1200, MS: API 4000 QTRAP	10	AcN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Automatic	591	
034*	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Quattro Premier	10	AcN	DSPE (PSA/MgSO ₄)	C18	5	Both	197	
035	Confirmed with second method			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	API 3200QTrap	50	Acetone	SLE	C18	20	Both	300	Each batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Propamocarb																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	yes	yes	from 0.01	no	4000 QTRAP	10	AcN	DSPE	C18 hydra Phenomenex	20	Both		Always
038	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 6490	5	AcN		RP-18	5	Automatic	>500	Daily
039	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API 5500	10	AcN	none	Luna 5µm PIP	3	Both	550	Each batch
040	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	API 3200Qtrap	10	AcN	DSPE	Aqua 5µ C18	10	Both	205	Always
041	Tentative detected	30	5	accurate mass	LC	Orbitrap	Orbitrap	ESI	+	yes	yes	0.01-0.05	yes	Thermo Orbitrap	10	AcN	PSA	RP18	10	Both	>100	Each batch
042	Fully identified (SANCO/12495/2011 p.74-80)	10		2*MRM	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	AB Sciex API 4000	10	AcN	PSA	Phenomenex C18	10	Both	500	Daily
043	Fully identified (SANCO/12495/2011 p.74-80)	0	1	3	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	XEVEO TQD	10	AcN		C18	3	Both	250	often
046*	Fully identified (SANCO/12495/2011 p.74-80)	-3	0	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Varian Pro Star LC + 320-MS	10	AcN	no clean up	Restek Ultra C18	10	Both	363	Each batch
047	Fully identified (SANCO/12495/2011 p.74-80)	5		2 transitions	LC	MSD	QQQ	ESI	+	no	no	0.1 -1	no	API 4000	10	AcN	DSPE	C18	5	Automatic	500	when we find a + result
049	Fully identified (SANCO/12495/2011 p.74-80)	6		3 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01-0.5	no	API 4000	10	AcN	no	RP-Amide	10	Both	170	once a month
050	Tentative detected	304		2 transitions	LC	MSMS	QQQ	ESI	+	no	no	0.01	no	Waters Premier XE	10	AcN	DSPE	HSST3	4	Both	87	Always
053	Fully identified (SANCO/12495/2011 p.74-80)	<30	<5	acc. m/z + isotope+fragment	LC	MSD	Orbitrap	ESI	+	no	no	0.01-0.1	yes	Exactive	10	AcN/1% HAC	none	C18 Atlantis	5	Both	650	Every batch ±100 pesticides
054	Tentative detected	1.6	20%	3 ions	GC	MSD	Q	EI	+	yes	no	0.01	yes	Agilent GC-MSD 7890A/5975C	10	AcN	PSA	HP-5MS	1	Automatic	927	weekly
055*	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions + ion ratio	LC	MS	QQQ	ESI	+	yes	yes	0.005	no	Waters Acquity-Guattro Premier XE	15	Acetone/DCM/PE	No	UPLC-Acquity BEH	5	Both	240	Each batch
056	Fully identified (SANCO/12495/2011 p.74-80)	0.02		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	Waters Acquity TQD	15	AcN	DSPE	UPLC-BEH C18	10	Both	327	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

Propamocarb																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
057	Fully identified (SANCO/12495/2011 p.74-80)	na	na	2 transitions	LC		QQQ	ESI	+	yes	yes	0.5 - 0.01	no	Agilent 1290-6490	10	AcN	PSA	XB-C18	10	Both	220	Daily
058*	Confirmed with second method	30	10	accurate mass + isotope	LC		TOF	ESI	+	no	yes		yes	Agilent 6230 TOF	10	AcN		C18	3	Automatic	510	
059	Fully identified (SANCO/12495/2011 p.74-80)	0.4		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Thermo Finnigan TSQ Quantum	10	AcN	DSPE (PSA)	Agilent Eclipse C18 XDB	20	Both	170	Each batch
061	Fully identified (SANCO/12495/2011 p.74-80)	120		2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	Waters XEVO TQMS	10	AcN	PSA	UPLC C18	25	Automatic	120	Each batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Prosulfocarb																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
001	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	ABSCIEX	10	AcN	DSPE	C18	5	Automatic	200	Always
002	Fully identified (SANCO/12495/2011 p.74-80)	1		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.001	no	Xevo TQ-S	10	AcN	no	reversed phase	2.5	Automatic	300	Each batch
004*	Tentative detected	0	0	2 transitions	LC	MSMS	QQQ	ESI	+	yes	yes	0.005	no	Xevo TQ	15	AcN	LLE	C18	10	Both	400	Always
005*	Fully identified (SANCO/12495/2011 p.74-80) Confirmed with second method	0.03	0.1	2 transitions accurate mass + isotope	LC	MSD	TOF	ESI	+	no	no	0.01	no	Agilent 6460	10	20	none	C18	2	Automatic	520	Always
006*	Fully identified (SANCO/12495/2011 p.74-80)	1	none	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes		no	Agilent 6410	10	AcN	none	Eclipse XDB-C18	4	Manual	143	Each batch
007	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes		no	Agilent 6490	15	EIOAc	Dilution	Waters HSS T3	1	Manual	256	Each batch
008	Fully identified (SANCO/12495/2011 p.74-80)	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	AGILENT G-6410A	10	AcN	PSA	ZORBAX-ECLIPSE XDB-C8	20	Automatic	143	Each batch
009	Tentative detected	0.1	<10	accurate mass	LC	MSD	TOF	ESI	+	yes	no	0.01	yes	Xevo Q-TOF	10	EIAc	none	BEH C18	3	Manual	500	Each batch
010*	Tentative detected				GC	MSD	Q	EI	+	no	no		yes	Agilent	15	Acetone, DCM, PE	none		1	Automatic		
011	Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Waters Xevo TQ	10	Acetone/PE/DCM	Na ₂ SO ₄	C18	2	Both	289	Each batch
013	Fully identified (SANCO/12495/2011 p.74-80)	<2	0	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	>0.01	no	Waters Quattro Premier	15	AcN	no	BEH C18	3	Both		
014*	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01		ABI 4000 QTRAP	10	AcN	no	C18	55	Both	approx. 660	Always
016*	Fully identified (SANCO/12495/2011 p.74-80)	30		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	xevo TQ	10	AcN	DSPE	C18	10	Automatic	1.62	Daily
017	Fully identified (SANCO/12495/2011 p.74-80)	1.5		AMDIS Match ₉₄	GC	MSD	Q	EI	+	no	no	NA	yes	AGILENT TECHNOLOGIES 7890A // G3440A	10	AcN	PSA	HP-5MS	20	Automatic	560	

APPENDIX 3. Methods used by participants for detecting pesticides.

Prosulfocarb																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µl)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
018	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	ESI	+	yes	yes	0.01-1	no	Ultra Thermo Fishers	10	AcN	PSA	C18	10	Both		
019	Tentative detected	5	0.005	None	LC	MSD	TOF	ESI	+	yes	yes	0.1	yes	LCT premier XE	15	Acetone; PE; DCM	No	BEH C18	7	Automatic	345	Daily
020*	Fully identified (SANCO/12495/2011 p.74-80)	3		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.02	no	Quattro Premier	10	MeOH		C18 BEH	5	Both	220	Each batch
021	Fully identified (SANCO/12495/2011 p.74-80)			2 MRM	LC	MSD	QQQ	ESI	+	yes	yes	0.001-0.01	no	Agilent 6460	10	AcN	DSPE (Q)	YMC Triart C18 2*100 mm 3µm	5	Both	400	Each batch
023	Fully identified (SANCO/12495/2011 p.74-80)	<2.5%		2 transitions	LC																	
024*	Confirmed with second method	1	0.1	accurate mass + isotope	LC	MSD	Q-TOF	ESI	+	no	yes		yes	Bruker Maxis	10	AcN	MgSO ₄ , PSA	C18	5	Both	450	often
025*	Tentative detected				GC	MSD	QQQ	EI	+	no	no		no	7.5 Acetone; DCM; PE	7.5		no	VF-5	1			
026	Fully identified (SANCO/12495/2011 p.74-80)	0.02	400	3 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7890/7000	10	AcN	25 mg PSA-150 mg MgSO ₄	HP 5 MS	4	Both	355	Daily
029	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	Waters Aquity UPLC system, API 5000 QQQ, Sciex	10	EIOAc	Filter	HSS T3	10	Both	387	Each batch
030	Fully identified (SANCO/12495/2011 p.74-80)	< ± 2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	HPLC Agilent 1100, MSD API 3000	10	AcN	DSPE	C18 3µ 50x2mm	10	Both	550	Always
033*	Tentative detected			252.3>91.1, 252.3>128.1	LC	MSD	QQQ	ESI	+	no	no	0.01-0.1		LC; Agilent 1200, MS; API 4000 QTRAP	10	AcN	GuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Automatic	591	
034*	Confirmed with second method			full scan	GC	MSD	Q	EI	+	no	no	0.05	yes	Agilent 5973	10	AcN	DSPE (PSA/MgSO ₄)	HP5	5	Both	800	
035	Confirmed with second method			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	API 3200QITrap	50	Acetone	SLE	C18	20	Both	300	Each batch
036	Fully identified (SANCO/12495/2011 p.74-80)				LC	MSD	QQQ	EI		yes	yes			AB SCIEX API 4000	2	ACN/MeOH	GuEChERS					Always
037	Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MS/MS	QQQ	ESI	+	yes	yes	from 0.01	no	4000 QTRAP	10	AcN	DSPE	C18 hydro Phenomenex	20	Both		Always
038	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 6490	5	AcN		RP-18	5	Automatic	>500	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

Prosulfocarb																							
Laboratory Code	*NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
039		Fully identified (SANCO/12495/2011 p.74-80)			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API 5500	10	AcN	DSPE	Pursuit XRs Ultra	3	Both	550	Each batch
040		Fully identified (SANCO/12495/2011 p.74-80)	0		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.005	no	TSQ Quantum Ultra	10	AcN	DSPE	RX1 5 SII MS	2	Both	290	Always
041		Tentative detected	30	5	accurate mass	LC	Orbitrap	Orbitrap	ESI	+	yes	yes	0.01-0.05	yes	Thermo Orbitrap	10	AcN	PSA	RP18	10	Both	> 100	Each batch
042		Fully identified (SANCO/12495/2011 p.74-80)	10		2*MRM	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	AB Sciex API 4000	10	AcN	PSA	Phenomenex C18	10	Both	500	Daily
043		Fully identified (SANCO/12495/2011 p.74-80)	0	1	3	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	XEVEO TQD	10	AcN		C18	3	Both	250	often
044		Tentative detected				GC	MSD	IT	EI	+	no	no		yes	Thermo Polaris Q	10	AcN	QuEChERS	DB-5MS	5	Both		
046*		Fully identified (SANCO/12495/2011 p.74-80)	0	0		GC	MSD	QQQ	EI	+	yes	yes	0.01	yes	Varian 3800 GC + 320-MS	10	AcN	DSPE (PSA)	VF-1 ms	3	Both	363	Each batch
047		Fully identified (SANCO/12495/2011 p.74-80)	5		2 transitions	LC	MSD	QQQ	ESI	+	no	no	0.1 - 1	no	API 4000	10	AcN	DSPE	C18	5	Automatic	500	when we find a + result
048		Tentative detected	2			GC	MSD	Q	EI		yes	no		yes	Agilent 5973	10	AcN	PSA	HP 5 MS UI	2	Both	approx 600	Each batch
		Fully identified (SANCO/12495/2011 p.74-80)	<20		spectrum	GC	MSD	TOF	EI	+	yes	yes	0.01-0.2	yes	Leco Pegasus IV	10	AcN/1% HAC	DSPE (PSA)	RTX-C18pesticides	10	Automatic	560	Every batch 235 pesticides
053		Confirmed with second method	<30	<5	accurate mass + isotope	LC	MSD	Orbitrap	ESI	+	no	no	0.01-0.1	yes	Exactive	10	AcN/1% HAC	none	C18 Atlantis	5	Both	650	Every batch ±100 pesticides
054		Tentative detected	2	20%	3 ions	GC	MSD	Q	EI	+	yes	no	0.05	yes	Agilent GC-MSD 7890A/5975C	10	AcN	PSA	HP-5MS	1	Automatic	927	weekly
055*		Fully identified (SANCO/12495/2011 p.74-80)	0.3		2 transitions + ion ratio	GC	MS	QQQ	EI	+	yes	yes	0.005	no	Bruker 451 Scion TQ	15	Acetone/DCM/PE	No	VF-ms5	5	Both	340	Every quarter
056		Fully identified (SANCO/12495/2011 p.74-80)	<0.01		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	Waters Acquity TQD	15	AcN	DSPE	UPLC-BEH C18	10	Both	327	Daily
057		Fully identified (SANCO/12495/2011 p.74-80)	na	na	2 transitions	GC		QQQ	EI	+	yes	yes	0.5 - 0.01	no	Agilent 7890-7000B	10	AcN	PSA	DB-5ms UI	2	Both	330	Daily

APPENDIX 3. Methods used by participants for detecting pesticides.

Prosulfocarb																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
058*	Confirmed with second method	30	10	accurate mass + isotope	LC		TOF	ESI	+	no	yes		yes	Agilent 6230 TOF	10	AcN		C18	3	Automatic	510	Each batch
059	Fully identified (SANCO/12495/2011 P.74-80)	0.66	0	3 transitions	GC	MSD	IT	EI	+	yes	yes	0.01	no	Varian Saturn 4000	10	AcN	DSPE (PSA)	Restek Rxi-5ms	3	Both	202	

APPENDIX 3. Methods used by participants for detecting pesticides.

Quinoclamine																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
002	Fully identified [SANCO/12495/2011 p.74-80]	1		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.001	no	Xevo TQ-S	10	AcN	no	reversed phase	2.5	Automatic	300	Each batch
004*	Tentative detected	0	0	2 transitions	LC	MSMS	QQQ	ESI	+	yes	yes		no	Xevo TQ	15	AcN	LLE	C18	10	Both	400	Always
005*	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	no	0.01	no	Agilent 6460	10	20	none	C18	2	Automatic	520	Always
	Confirmed with second method	-0.02	1.2	accurate mass + isotope	LC	MSD	TOF	ESI	+	no	no	0.05	no	Bruker Maxis	10	10	none	C18	2	Automatic	760	Always
013	Tentative detected	<2	<1	3 diagnostic ions	GC	MSD	Q	EI	+	yes	no		yes	Trace DSQ	15	AcN	PSA/C18	DB5MS	0.8	Both		
014*	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01		ABI 4000 QTRAP	10	AcN	no	C18	55	Both	approx. 660	Always
017	Tentative detected	10		AMDIS Match 90	GC	MSD	Q	EI	+	no	no	NA	yes	AGILENT TECHNOLOGIES 7890A / G3440A	10	AcN	PSA	HP-5MS	20	Automatic	560	
021	Fully identified [SANCO/12495/2011 p.74-80]			2 MRM	LC	MSD	QQQ	ESI	+	yes	yes	0.001-0.01	no	Agilent 6460	10	AcN	DSPE (Q)	YMC Triart C18 2*100 mm 3µm	5	Both	400	Each batch
029	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.05	no	Waters Aquity UPLC system, API 5000 QQQ, Sciex	10	EIOAc	Filter	HSS T3	10	Both	123	Each batch
030	Fully identified [SANCO/12495/2011 p.74-80]	< ± 2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	HPLC Agilent 1100, MSD API 3000	10	AcN	DSPE	C18 3µ 50x2mm	10	Both	550	Always
033*	Tentative detected			Match with NIST Library (Match 832, range 0-1000)	GC	MSD	TOF	EI		no	no	0.01-0.1	yes	GCxGC-TOF MS Pegasus 4D	10	AcN	QuEChERS without PSA	DB5-MS (30 m x 0.25 mm x 0.25 µm) in the 1 st Dimension, BPX-50 (2 m x 0.1 mm x 0.1 µm) in the 2 nd Dimension	10	Automatic		
034*	Confirmed with second method			full scan	GC	MSD	Q	EI	+	no	no	0.05	yes	Agilent 5973	10	AcN	DSPE (PSA/MgSO ₄)	HP5	5	Both	800	
037	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MS/MS	QQQ	ESI	+	yes	yes	from 0.01	no	4000 QTRAP	10	AcN	DSPE	C18 hydro Phenomenex	20	Both		Always

APPENDIX 3. Methods used by participants for detecting pesticides.

Quinoclamine																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
039	Fully identified [SANCO/12495/2011 p.74-80]				GC		TOF	EI		yes	yes	0.01	yes	Leco Pegasus IV	10	AcN	DSPE	HP5-MS	3	Both	750	Always
041	Tentative detected	15	unit mass	target + qualifier	GC	MSD	Q	EI	+	yes	yes	0.01-0.05	yes	Agilent GC-MSD	10	AcN	PSA	DB5	10	Both	>600	Each batch
042	Fully identified [SANCO/12495/2011 p.74-80]	10		2*MRM	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	AB Sciex API 4000	10	AcN	PSA	Phenomenex C18	10	Both	500	Daily
047	Fully identified [SANCO/12495/2011 p.74-80]	5		2 transitions	LC	MSD	QQQ	ESI	+	no	no	0.1 -1	no	API 4000	10	AcN	DSPE	C18	5	Automatic	500	when we find a + result
048	Tentative detected	2			GC	MSD	Q	EI		yes	no		yes	agilent 5973	10	AcN	PSA	HP 5 MS UI	2	Both	approx 600	Each batch
049	Tentative detected	15		3 sim ions	GC	MSD	Q	EI	+	no	no	0.05-1.0	yes	Thermo Polaris	10	AcN	no	Elite-5ms	1	Both	800	once a year
053	Fully identified [SANCO/12495/2011 p.74-80]	<20		spectrum	GC	MSD	TOF	EI	+	yes	yes	0.01-0.2	yes	Leco Pegasus IV	10	AcN/1% HAC	DSPE (PSA)	RTX-C18pesticides	10	Automatic	540	Every batch 235 pesticides
	Confirmed with second method	<30	<5	accurate mass + isotope	LC	MSD	Orbitrap	ESI	+	no	no	0.01-0.1	yes	Exactive	10	AcN/1% HAC	none	C18 Atlantis	5	Both	650	Every batch ±100 pesticides
056	Fully identified [SANCO/12495/2011 p.74-80]	<0.01		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	Waters Acquity TQD	15	AcN	DSPE	UPLC-BEH C18	10	Both	327	Daily
058*	Confirmed with second method	30	10	accurate mass + isotope	LC		TOF	ESI	+	no	yes		yes	Agilent 6230 TOF	10	AcN		C18	3	Automatic	510	
061	Tentative detected			qualifiers	GC	MSD	IT	EI	+	yes	no		yes	Varian 4000	10	AcN	PSA	capillary VF5-pesticides	5	Manual		

APPENDIX 3. Methods used by participants for detecting pesticides.

Thiodicarb																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
001	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	ABSCIEX	10	AcN	DSPE	C18	5	Automatic	200	Always
002	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.002	no	Xevo TQ-S	10	AcN	no	reversed phase	2.5	Automatic	300	Each batch
004*	Tentative detected	0	0	2 transitions	LC	MSMS	QQQ	ESI	+	yes	yes	0.005	no	Xevo TQ	15	AcN	LLE	C18	10	Both	400	Always
005*	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 6460	10	20	none	C18	2	Automatic	520	Always
005*	Confirmed with second method	-0.06	1.1	accurate mass + isotope	LC	MSD	TOF	ESI	+	no	no	0.05	no	Bruker Maxis	10	10	none	C18	2	Automatic	760	Always
006*	Fully identified [SANCO/12495/2011 p.74-80]	1	none	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes		no	Agilent 6410	10	AcN	none	Eclipse XDB-C18	4	Manual	143	Each batch
007	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes		no	Agilent 6490	15	EIOAc	Dilution	Waters HSS T3	1	Manual	256	Each batch
008	Fully identified [SANCO/12495/2011 p.74-80]	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	AGILENT G-6410A	10	AcN	PSA	ZORBAX-ECLIPSE XDB-C8	20	Automatic	143	Each batch
009	Tentative detected	0.1	<10	accurate mass	LC	MSD	TOF	ESI	+	yes	yes	0.01	yes	Xevo Q-TOF	10	EIAC	none	BEH C18	3	Manual	500	Each batch
010*	Fully identified [SANCO/12495/2011 p.74-80]				LC	MSD	Q	ESI	+	no	yes		yes	Agilent	15	Acetone, DCM, PE	none		1	Automatic		
011	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Waters Xevo IQ	10	Acetone/PE/DCM	Na2SO4	C18	2	Both	289	Each batch
012*	Tentative detected	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	Varian 1200L	25	EIOAc	no	C18	5	Both	50	
013	Fully identified [SANCO/12495/2011 p.74-80]	<2	0	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	>0.010	no	Waters Quattro Premier	15	AcN	no	BEH C18	3	Both		
014*	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01		ABI 4000 QTRAP	10	AcN	no	C18	55	Both	approx. 660	Always

APPENDIX 3. Methods used by participants for detecting pesticides.

Thiodicarb																							
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µl)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency	
015*	Fully identified [SANCO/12495/2011 p.74-80]				LC	MSD	Q-TRAP	ESI	+	no	no		no	AB Sciex 5500	10	AcN	PSA			10	Manual		
016*	Fully identified [SANCO/12495/2011 p.74-80]	30		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	xevo IQ	10	AcN	DSPE	C18	10	Automatic	162	Daily	
017	Fully identified [SANCO/12495/2011 p.74-80]				LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API-3200 QTrap	10	AcN	PSA	Athlantis T3	5	Both	180	Each batch	
018	Fully identified [SANCO/12495/2011 p.74-80]				LC	MSD	QQQ	ESI	+	yes	yes	0.01-1	no	Ultra Thermo Fishers	10	AcN	PSA	C18	10	Both			
019	Tentative detected	5	0.005	None	LC	MSD	TOF	ESI	+	yes	yes	0.01	yes	LCT premier XE	15	Acetone:PE:DCM	No	BEH C18	7	Automatic	345	Daily	
020*	Fully identified [SANCO/12495/2011 p.74-80]	3		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.010	no	Quattro Premier	10	MeOH		C18 BEH	5	Both	220	Each batch	
021	Fully identified [SANCO/12495/2011 p.74-80]			2 MRM	LC	MSD	QQQ	ESI	+	yes	yes	0.001-0.01	no	Agilent 6460	10	AcN	DSPE (Q)	YMC Triart C18 2*100 mm 3µm	5	Both	400	Each batch	
022	Confirmed with second method	0.5	1.6	2 transitions	LC	MSD	Trap and Q-Orbitrap	ESI	+	yes	yes	0.005	yes	Thermo Fisher LTQ and Q-Exactive	10	AcN	PSA and SPE with NH ₂ /Carb. separately	Shiseido CR 1:4 and BEH C18	10	Both	574	Every 12 injections	
023	Fully identified [SANCO/12495/2011 p.74-80]	<2.5%		2 transitions	LC																		
024*	Confirmed with second method	0	0.4	accurate mass + isotope	LC	MSD	Q-TOF	ESI	+	no	yes		yes	Bruker Maxis	10	AcN	MgSO ₄ , PSA	C18	5	Both	450	often	
025*	Fully identified [SANCO/12495/2011 p.74-80]				LC	MSD	QQQ	ESI	+	yes	yes		no		7.5	AcN, DCM	no	C18	5	Automatic			
029	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	Waters Aquity UPLC system, API 5000 QQQ, Sciex	10	EIOAc	Filter	HSS T3	10	Both	387	Each batch	
030	Fully identified [SANCO/12495/2011 p.74-80]	< ± 2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	HPLC Agilent 1100, MSD API 3000	10	AcN	DSPE	C18 3µ 50x2mm	10	Both	550	Always	
031	Confirmed with second method	0.2	1.7	2 transitions	LC	MSD	Trap and Q-Orbitrap	ESI	+	yes	yes	0.005	yes	Thermo Fisher LTQ and Q-Exactive	10	AcN	QueChERS and NH ₂ /Carb	SHISEIDO CR 1:4, BEH C18	10	Both	512	Every 12 injections	

APPENDIX 3. Methods used by participants for detecting pesticides.

Thiodicarb																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
033*	Fully identified [SANCO/12495/2011 p.74-80]			355.0>108.0, 355.0>88.1	LC	MSD	QQQ	ESI	+	yes	yes	0.002		LC: Agilent 1200, MS: API 4000 QTRAP	10	AcN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Automatic	591	
034*	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions	LC		QQQ	ESI	+	yes	yes	0.01	no	Quattro Premier	10	AcN	DSPE (PSA/MgSO ₄)	C18	5	Both	197	
035	Confirmed with second method			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	API 3200QTrap	50	Acetone	SLE	C18	20	Both	300	Each batch
037	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MS/MS	QQQ	ESI	+	yes	yes	from 0.01	no	4000 QTRAP	10	AcN	DSPE	C18 hydro Phenomenex	20	Both		Always
038	Confirmed with second method			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Agilent 6490	5	AcN		RP-18	5	Automatic	>500	Daily
039	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API 5500	10	AcN	DSPE	Pursuit XRs Ultra	3	Both	550	Each batch
040	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	API 3200 Qtrap	10	AcN	DSPE	Aqua 5u C18	10	Both	205	Always
041	Tentative detected	30	5	accurate mass	LC	Orbitrap	Orbitrap	ESI	+	yes	yes	0.01-0.05	yes	Thermo Orbitrap	10	AcN	PSA	RP18	10	Both	>100	Each batch
042	Fully identified [SANCO/12495/2011 p.74-80]	10		2*MRM	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	AB Sciex API 4000	10	AcN	PSA	Phenomenex C18	10	Both	500	Daily
043	Fully identified [SANCO/12495/2011 p.74-80]	0	1	3	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	XEVEO TQD	10	AcN		C18	3	Both	250	often
044	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Thermo ISQ Quantum	10	AcN	QuEChERS	Xterra-MSC18	20	Both		Daily
046*	Fully identified [SANCO/12495/2011 p.74-80]	-0.6	0	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Varian Pro Star LC + 320-MS	10	AcN	no clean up	Restek Ultra C18	10	Both	363	Each batch
047	Fully identified [SANCO/12495/2011 p.74-80]	5		2 transitions	LC	MSD	QQQ	ESI	+	no	no	0.1 - 1	no	API 4000	10	AcN	DSPE	C18	5	Automatic	500	when we find a result
049	Fully identified [SANCO/12495/2011 p.74-80]	6		3 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01-0.5	no	API 4000	10	AcN	no	RP-Amide	10	Both	170	once a month

APPENDIX 3. Methods used by participants for detecting pesticides.

Thiodicarb																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µl)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
050	Tentative detected	487		2 transitions	LC	MSMS	QQQ	ESI	+	no	no	0.01	no	Waters Premier XE	10	AcN	DSPE	HS3	4	Both	87	Always
053	Fully identified [SANCO/12495/2011 p.74-80]	<30	<5	acc. m/z + isotope+fragment	LC	MSD	Orbitrap	ESI	+	no	no	0.01-0.1	yes	Exactive	10	AcN/1% HAC	none	C18 Atlantis	5	Both	650	Every batch ±100 pesticides
055*	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions + ion ratio	LC	MS	QQQ	ESI	+	yes	yes	0.005	no	Waters Acquity-Guardia Premier XE	15	Acetone/DCM/PE	No	UPLC-Aquity BEH	5	Both	240	Every quarter
056	Fully identified [SANCO/12495/2011 p.74-80]	<0.01		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	Waters Acquity TQD	15	AcN	DSPE	UPLC-BEH C18	10	Both	327	Daily
057	Fully identified [SANCO/12495/2011 p.74-80]	na	na	2 transitions	LC		QQQ	ESI	+	yes	yes	0.5 - 0.01	no	Agilent 1290-6490	10	AcN	PSA	XB-C18	10	Both	220	Daily
058*	Confirmed with second method	30	10	accurate mass + isotope	LC		TOF	ESI	+	no	yes		yes	Agilent 6230 TOF	10	AcN		C18	3	Automatic	510	
059	Fully identified [SANCO/12495/2011 p.74-80]	0.4		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Thermo Finnigan TSQ Quantum	10	AcN	DSPE (PSA)	Agilent Eclipse C18 XDB	20	Both	170	Each batch
060	Confirmed with second method	12		3 transitions	LC		QQQ	ESI	+	yes	yes	0.01	no	Varian-320	10	AcN	PSA	C-18	10	Automatic	72	Daily
061	Fully identified [SANCO/12495/2011 p.74-80]	120		2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	Waters XEVO TQMS	10	AcN	PSA	UPLC C18	25	Automatic	120	Each batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Triflumizole																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY)	Standard Solution Frequency
001	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	ABSCIEX	10	AcN	DSPE	C18	5	Automatic	200	Always
002	Tentative detected		3	confirmed by ESI- (OTOF) and GC-MS (QTOF)	LC	MSD	Q-TOF	ESI	+	no	no		yes	AB Sciex TripleTOF 5400	10	AcN	no	reversed phase	2	Automatic	500	we have not standard
004*	Tentative detected	0	0	2 transitions	LC	MSMS	QQQ	ESI	+	yes	yes	0.005	no	Xevo TQ	15	AcN	LLE	C18	10	Both	400	Always
005*	Confirmed with second method	0.03	0.1	accurate mass + isotope	LC	MSD	TOF	ESI	+	no	no	0.05	no	Bruker Maxis	10	10	none	C18	2	Automatic	760	Always
007	Confirmed with second method			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Thermo TSQ Quantum	10	20	none	TG-5SILMS	2	Automatic	520	Always
008	Fully identified [SANCO/12495/2011 p.74-80]	2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.005	no	Agilent 6490	15	EIOAc	Dilution	Waters HSS T3	1	Manual	256	Each batch
011	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Waters Xevo TQ	10	Acetone/PE/DCM	Na ₂ SO ₄	C18	2	Both	289	Each batch
012*	Tentative detected	0		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.005	no	Agilent7890/ Agilent7000	25	EIOAc	no	HP5MS	2	Both	310	
013	Fully identified [SANCO/12495/2011 p.74-80]	<2	0	2 transitions	LC	MSD	Q-TRAP	ESI	+	yes	yes	>0.01	no	Waters Quattro Premier	15	AcN	no	BEH C18	3	Both		
014*	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01		ABI 4000 QTRAP	10	AcN	no	C18	55	Both	approx. 660	Always
016*	Fully identified [SANCO/12495/2011 p.74-80]	30			LC	MSD	QQQ	ESI	+	yes	no		no	xevo TQ	10	AcN	DSPE	C18	10	Automatic	162	
017	Confirmed with second method				LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API-3200 Qtrap	10	AcN	PSA	Athlantis T3	5	Both	180	Each batch
018	Fully identified [SANCO/12495/2011 p.74-80]				GC	MSD	QQQ	EI	+	no	no	0.01-1	yes	Ultra Thermo Fichers	10	AcN	PSA	C18	1	Both		

APPENDIX 3. Methods used by participants for detecting pesticides.

Triflumizole																						
Laboratory Code *NRL	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Injection Volume (µl)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
019	Tentative detected	5	0.005	None	LC	MSD	TOF	ESI	+	yes	yes	0.01	yes	LCT premier XE	15	Acetone; PE; DCM	No	BEH C18	7	Automatic	345	Daily
020*	Fully identified [SANCO/12495/2011 p.74-80]	3		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.010	no	VARIAN 320	50	Acetone	L/L	DB5	8	Both	1.50	Each batch
021	Fully identified [SANCO/12495/2011 p.74-80]			2 MRM	LC	MSD	QQQ	ESI	+	yes	yes	0.001-0.01	no	Agilent 6460	10	AcN	DSPE (Q)	YMC Triart C18 2*100 mm 3µm	5	Both	400	Each batch
023	Fully identified [SANCO/12495/2011 p.74-80]	<0.5%	<5ppm	full scan	GC																	
024*	Confirmed with second method	5	0.3	accurate mass + isotope	LC		Q-TOF	ESI	+	no	yes		yes	Bruker Maxis	10	AcN	MgSO ₄ , PSA	C18	5	Both	450	often
026	Fully identified [SANCO/12495/2011 p.74-80]	0.02	290	3 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7890/7000	10	AcN	25 mg PSA-150 mg MgSO ₄	HP 5 MS	4	Both	355	Daily
029	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Waters Quattro Micro GC	10	EIOAc	Filter	Rxi-5sil MS/integra-guard Restek	10	Both	387	Each batch
030	Fully identified [SANCO/12495/2011 p.74-80]	< ± 2.5%		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	HPLC Agilent 1100, MSD API 3000	10	AcN	DSPE	C18 3µ 50x2mm	10	Both	550	Always
033*	Confirmed with second method			346>73.0, 346.0>156.2	LC	MSD	QQQ	ESI	+	no	no	0.01-0.1		LC: Agilent 1200, MS: API 4000 QTRAP	10	AcN	QuEChERS without PSA	Supelco Discovery C18, 150 mm x 2.1 mm, 5 µm	2	Automatic	591	
034*	Confirmed with second method			full scan	GC	MSD	Q	EI	+	no	no	0.05	yes	Agilent 5973	10	AcN	DSPE (PSA/MgSO ₄)	HP5	5	Both	800	
035	Confirmed with second method			2 transitions	LC	MSD	QQQ	EI	+	yes	yes	0.01	no	API 3200QTrap	50	Acetone	SLE	C18	20	Both	300	Each batch
038	Confirmed with second method			2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7000B	5	AcN		HP5-MS-UI	2	Automatic	>500	Daily
039	Fully identified [SANCO/12495/2011 p.74-80]			2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	API 5500	10	AcN	DSPE	Pursuit XRs Ultra	3	Both	550	Each batch
040	Fully identified [SANCO/12495/2011 p.74-80]	0		2 transitions	GC	MSD	QQQ	EI	+	yes	yes	0.005	no	TSQ Quantum Ultra	10	AcN	DSPE	RXI 5 Sil MS	2	Both	290	Always
041	Tentative detected	15	unit mass	target + qualifier	GC	MSD	Q	EI	+	yes	yes	0.01-0.05	yes	Agilent GC-MSD	10	AcN	PSA	DB5	10	Both	>600	Each batch

APPENDIX 3. Methods used by participants for detecting pesticides.

Triflumizole																						
Laboratory Code	How was the identification done?	RT Deviation (in seconds)	MS Tolerance (ppm)	Other Identification Details	Chromatographic Technique	Detector	Analyzer	Ionization Mode	Polarity	Routine Method?	Routine Scope?	Screening Reporting Level (SRL) Range? (mg/kg)	Full Scan	Instrument Model	Sample Weight (g)	Extraction Solvent	Clean up Step	Column Type	Inject Volume (µL)	Software	Compounds in method (pesticides ONLY) or library	Standard Solution Frequency
042	Fully identified (SANCO/12495/2011 p.74-80)	10		2 masses	GC	MSD	IT	EI	+	yes	yes	0.01	yes	Varian Saturn 2000	10	AcN	PSA	VactorFour 5MS	5	Automatic	500	Daily
043	Fully identified (SANCO/12495/2011 p.74-80)	0	1	4	GC	MSD	QQQ	EI	+	yes	yes	0.01	no	Agilent 7000A	10	AcN	PSA	DB5-MS	2	Both	350	often
044	Tentative detected			2 transitions	LC	MSD	QQQ	ESI	+	no	no		no	Thermo ISQ Quantum	10	AcN	QuEChERS	Xterra-MS-C18	20	Both		
046*	Fully identified (SANCO/12495/2011 p.74-80)	-2.4	0	2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	no	Varian Pro Star LC + 320-MS	10	AcN	no clean up	Restek Ultra C18	10	Both	343	Each batch
047	Fully identified (SANCO/12495/2011 p.74-80)	5		2 transitions	LC	MSD	QQQ	ESI	+	no	no	0.1-1	no	API 4000	10	AcN	DSPE	C18	5	Automatic	500	when we find a + result
048	Tentative detected	2			GC	MSD	Q	EI		yes	no		yes	Agilent 5973	10	AcN	PSA	HP 5 MS UI	2	Both	approx 600	Each batch
049	Tentative detected	15		3 sim ions	GC	MSD	Q	EI	+	no	no	0.05-1.0	yes	Thermo Polaris	10	AcN	no	Elite-5ms	1	Both	800	once a year
053	Fully identified (SANCO/12495/2011 p.74-80)	<20		spectrum	GC	MSD	TOF	EI	+	yes	yes	0.01-0.2	yes	Leco Pegasus IV	10	AcN/1% HAC	DSPE (PSA)	RTX-C18pesticides	10	Automatic	560	Every batch 235 pesticides
	Confirmed with second method	<30	<5	accurate mass + isotope	LC	MSD	Orbitrap	ESI	+	no	no	0.01-0.1	yes	Exactive	10	AcN/1% HAC	none	C18 Atlantis	5	Both	650	Every batch ±100 pesticides
054	Tentative detected	-1.3	20%	* 3 ions	GC	MSD	Q	EI	+	yes	no	0.1	yes	Agilent GC-MSD 7890A/5975c	10	AcN	PSA	HP-5MS	1	Automatic	927	weekly
055*	Fully identified (SANCO/12495/2011 p.74-80)	1.2		2 transitions + ion ratio	LC	MS	QQQ	ESI	+	yes	yes	0.005	no	Waters Acquity-Quattro Premier XE	15	Acetone/DCM/PE	No	UPLC Acquity BEH	5	Both	240	Each batch
056	Fully identified (SANCO/12495/2011 p.74-80)	<0.01		2 transitions	LC	MSD	QQQ	ESI	+	yes	yes	0.01	yes	Waters Acquity TQD	15	AcN	DSPE	UPLC-BEH C18	10	Both	327	Daily
057	Fully identified (SANCO/12495/2011 p.74-80)	na	na	2 transitions	LC		QQQ	ESI	+	yes	yes	0.5 - 0.01	no	Agilent 1290-6490	10	AcN	PSA	XB-C18	10	Both	220	Daily
058*	Confirmed with second method	30	10	accurate mass + isotope	LC		TOF	ESI	+	no	yes		yes	Agilent 6230 TOF	10	AcN		C18	3	Automatic	510	

APPENDIX 3. Methods used by participants for detecting pesticides.

Triflumizole																																													
Laboratory Code *NRL	059	How was the identification done?	Fully identified [SANCO/12495/2011 p.74-80]	RT Deviation (in seconds)	1.38	M5 Tolerance (ppm)	0	Other Identification Details	2 transitions	Chromatographic Technique	GC	Detector	MSD	Analyzer	IT	Ionization Mode	EI	Polarity	+	Routine Method?	yes	Routine Scope?	yes	Screening Reporting Level (SRL) Range? (mg/kg)	0.01	Full Scan	no	Instrument Model	Varian Saturn 4000	Sample Weight (g)	10	Extraction Solvent	AcN	Clean up Step	DSPE (PSA)	Column Type	Restek Rx-5ms	Injection Volume (µL)	3	Software	Both	Compounds in method (pesticides ONLY) or library	202	Standard Solution Frequency	Each batch

ANNEX 1. List of Laboratories that participate in EUPT-FV-SM05.

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
AUSTRIA	AUSTRIAN AGENCY FOR HEALTH AND FOOD SAFETY, INSTITUTE FOR FOOD SAFETY, PESTICIDE AND FOOD ANALYTICS (PLMA)	INNSBRUCK	YES
BELGIUM	FYTOLAB CVBA	ZWIJNAARDE	YES
BELGIUM	LOVAP NV	GEEL	YES
BELGIUM	SCIENTIFIC INSTITUTE OF PUBLIC HEALTH	BRUXELLES	YES
CHINA	KEY LAB OF CHEMICAL SAFETY AND HEALTH, CHINESE CENTER OF PREVENTION AND DISEASE CONTROL	BEIJING	YES
CHINA	LABORATORY OF FOOD CHEMISTRY, CHINA NATIONAL CENTER FOR FOOD SAFETY RISK ASSESSMENT	BEIJING	YES
CROATIA	CROATIAN NATIONAL INSTITUTE OF PUBLIC HEALTH, HEALTH ECOLOGY SERVICE, PESTICIDES UNIT	ZAGREB	YES
CZECH REPUBLIC	CZECH AGRICULTURE AND FOOD INSPECTION AUTHORITY	PRAHA	YES
CZECH REPUBLIC	INSTITUTE OF CHEMICAL TECHNOLOGY PRAGUE, DEPT. OF FOOD ANALYSIS	PRAGUE	YES
DENMARK	DTU NATIONAL FOOD INSTITUTE	SOEBORG	YES
ESTONIA	AGRICULTURAL RESEARCH CENTRE (ARC), LABORATORY FOR RESIDUES AND CONTAMINANTS (LRC)	SAKU	YES
FINLAND	FINNISH CUSTOMS LABORATORY	ESPOO	YES
FRANCE	CERECO SUD	GARONS	YES
FRANCE	GIRPA	BEAUCOUZE FRANCE	YES
FRANCE	LABORATOIRE DÉPARTEMENTAL DE LA SARTHE	LE MANS	YES
FRANCE	LABORATOIRE DU SCL DE MONTPELLIER	MONTPELLIER	YES
FRANCE	LABORATOIRE DU SCL-MASSY	MASSY CEDEX	YES
FRANCE	LABORATOIRE SCL - RENNES	RENNES	YES
FRANCE	LDA 22	PLOUFRAGAN	YES
FRANCE	TRISKALIA - CAPINOV	LANDERNEAU	YES
GERMANY	BAYERISCHES LANDESAMT FUER GESUNDHEIT UND LEBENSMITTELSICHERHEIT	ERLANGEN	YES
GERMANY	CHEMISCHES UND VETERINÄRUNTERSUCHUNGSAMT RHEIN-RUHR WUPPER	KREFELD	YES
GERMANY	CVUA-MEL CHEMISCHES UND VETERINÄRUNTERSUCHUNGSAMT MUENSTERLAND-EMSCHER-LIPPE	MUENSTER	YES
GERMANY	EUROFINS DR. SPECHT LABORATORIEN GMBH	HAMBURG	YES
GERMANY	GALAB LABORATORIES GMBH	GEESTHACHT	YES
GERMANY	LABOR FRIEDLE GMBH	TEGERNHEIM	YES
GERMANY	NIEDERSAECHSISCHES LANDESAMT FUER VERBRAUCHERSCHUTZ UND LEBENSMITTELSICHERHEIT, LVI OLDENBURG	OLDENBURG	YES
GREECE	PESTICIDE RESIDUES LABORATORY, D CHEMICAL DIVISION OF ATHENS, GENERAL CHEMICAL STATE LABORATORY	ATHENS	YES
HUNGARY	NATIONAL FOOD CHAIN SAFETY OFFICE, DPPSCA PESTICIDE ANALYTICAL LABORATORY, VELENCE	VELENCE	YES
HUNGARY	NATIONAL FOOD CHAIN SAFETY OFFICE, DPPSCA PESTICIDE RESIDUE ANALYTICAL LABORATORY, MISKOLC	MISKOLC	YES

ANNEX 1. List of Laboratories that participate in EUPT-FV-SM05.

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
HUNGARY	WESSLING HUNGRAY LDT.	BUDAPEST	YES
ITALY	A.R.P.A. VENETO - SERVIZIO LABORATORI VERONA	VERONA	YES
ITALY	ARPA FVG LABORATORIO UNICO MULTISITO - SEDE DI PORDENONE	PORDENONE	YES
ITALY	ISTITUTO SUPERIORE DI SANITÀ - DIP. AMPP - REP. ANTIPARASSITARI	ROME	YES
ITALY	ISTITUTO ZOOPROFILATTICO SPERIMENTALE DELLE VENEZIE	LEGNARO	YES
ITALY	LABORATORIO CONTAMINANTI AMBIENTALI	PERUGIA	YES
ITALY	LANDESAGENTUR FÜR UMWELT - LABOR FÜR CHROMATOGRAPHIE	BOZEN	YES
LATVIA	INSTITUTE OF FOOD SAFETY, ANIMAL HEALTH AND ENVIRONMENT "BIOR"	RIGA	YES
ROMANIA	CENTRAL LABORATORY FOR PESTICIDES RESIDUES CONTROL	BUCHAREST	YES
ROMANIA	SANITARY VETERINARY AND FOOD SAFETY DIRECTORATE	BUCHAREST	YES
SERBIA	CENTER FOR FOOD ANALYSIS	BELGRADE	YES
SLOVENIA	INSTITUTE OF PUBLIC HEALTH MARBOR (ZAVOD ZA ZDRAVSTVENO VARSTVO MARIBOR)	MARIBOR	NO
SPAIN	ANALYTICA ALIMENTARIA GMBH, SUCURSAL EN ESPAÑA	ALMERIA	YES
SPAIN	LABORATORIO AGRARIO Y FITOPATOLOGICO DE GALICIA	ABEGONDO. A CORUÑA	YES
SPAIN	LABORATORIO AGROALIMENTARIO DE GRANADA	ATARFE (GRANADA)	YES
SPAIN	LABORATORIO AGROALIMENTARIO DE VALENCIA	BURJASSOT	YES
SPAIN	LABORATORIO AGROALIMENTARIO Y DE SANIDAD ANIMAL	EL PALMAR (MURCIA)	YES
SPAIN	LABORATORIO DE PRODUCCION Y SANIDAD VEGETAL	MENGIBAR (JAEN)	YES
SWEDEN	EUROFINS FOOD & AGRO TESTING SWEDEN AB	LIDKÖPING	YES
SWEDEN	NATIONAL FOOD AGENCY (NFA), CHEMICAL UNIT 1	UPPSALA	YES
SWITZERLAND	KANTONALES LABOR ZÜRICH	ZURICH	YES
SWITZERLAND (CH)	SERVICE DE LA CONSOMMATION ET DES AFFAIRES VÉTÉRINAIRES (SCAV)	GENEVE	YES
THE NETHERLANDS	NVWA - NETHERLANDS FOOD AND CONSUMER PRODUCT SAFETY AUTHORITY	WAGENINGEN	YES
THE NETHERLANDS	RIKILT - INSTITUTE OF FOOD SAFETY	WAGENINGEN	YES
TURKEY	SGS MERSIN FOOD CONTROL LABORATORY (FORMER MSM)	MERSIN	YES
UNITED KINGDOM	EUROFINS LABORATORIES FOOD TESTING UK LTD	WOLVERHAMPTON	YES
UNITED KINGDOM	SASA (SCIENCE AND ADVICE FOR SCOTTISH AGRICULTURE)	EDINBURGH	YES
UNITED KINGDOM	THE FOOD AND ENVIRONMENT RESEARCH AGENCY	YORK	YES



EUPF-FV-SM05 SPECIFIC PROTOCOL
European Union Proficiency Test for
Pesticide Residues in Fruits and Vegetables
Screening Multiresidue Methods
(2013)

Introduction

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

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

Test material

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

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

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

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

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

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

Steps to follow

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

1. To participate, each laboratory must complete the Application Form online, available on the EURL-FV Web page, before the deadline stipulated on the Calendar.
2. Laboratories will then receive an e-mail confirming their participation in this exercise, and assigning them a unique Laboratory Code. With this code, laboratories will be able to access the restricted area containing the reply forms using their login information - consisting of their **USER NAME**, which is the Laboratory Code expressed as **Labxxx** (three digits with no spaces between them) and their **PASSWORD**, as chosen on the application form.
3. The sample delivery will be free of charge to those laboratories already participating in EUPF-FV15. For those who are not EUPF-FV15 participants, please see **Cost for shipment of the test material** for further details. The payment procedure must have started before 18th January. An e-mail showing the bank transfer confirmation, or similar, must have been sent beforehand, or may be requested by the Organiser at any time. **Payments without a Laboratory Code or Invoice Number to identify them will not be considered paid.**
4. When the participant laboratories receive the test material (and not before), they must enter the restricted area and submit **Form 0 - Test Material Receipt** to inform the Organiser that they have accepted the test material. If no test material has been received by 24th January, please contact the Organiser by e-mail (cferrer@ual.es and omalato@ual.es).
5. The participating laboratories must respect the deadline for submitting the results. Results must be reported using **Form 1- Results** within 72 hours after the arrival of the test item.
6. The Organiser will evaluate the results at the end of the proficiency test, once the deadline for the receipt of results has passed. The Organiser will prepare a Preliminary Report that will be sent to the participants and uploaded to the website to show the pesticides reported, after the revision of all the data by the Scientific Panel a Final Report will be done and the organiser will upload an electronic version on the EURL-FV website and, afterwards, send a hard copy to each participant laboratory. This report will include information regarding the design of the test, the homogeneity and stability test results, an evaluation of the participant's results as well as graphical displays of the results and any conclusions. Any other relevant information considered of value may also be included.

ANNEX 2. Protocols.

Amount of Test Material

Participants will receive:

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

Shipment of Test Materials

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

The shipment of the test materials will start on 21st January 2013. An information message will be sent out by e-mail before shipment. Laboratories must make their own arrangements for the receipt of the package. They must inform the Organiser of any public holidays in their country/city during the delivery period given in the calendar as well as making the necessary arrangements to receive the shipment, even if the laboratory is closed.

Advice on Test Material Handling

Once received, the test material should be stored deeply frozen (-18°C or less) prior to analysis to avoid any possible deterioration/spoilage. The test material should be mixed thoroughly before taking the analytical portion(s).

All participants should use their own routine standard operating procedures for extraction, clean-up and analytical measurement and their own reference standards for identification.

Form 0 - Test Material Receipt

Once the laboratory has received the test material, the Organiser must be notified using Form 0 in the restricted area; filling in the date of receipt, the condition of the test material, and acknowledging its acceptance. If the laboratory does not inform the Organiser by 25th February 2013 (at the latest) via email (to cferrer@ucl.es and omalato@ucl.es), stating that no sample has been received, the Organiser will assume that the test material has been received and accepted.

Form 1 - Results

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

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

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

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

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

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

Calendar

ACTIVITY	DATE
Publishing the Calendar and Matrix on the EURL-FV Web page.	23 rd October 2012
Submission of Application Form by invited laboratories.	3 rd Dec. 2012-7th Jan 2013
Sample distribution.	21 st January 2013
Submission of sample receipt and acceptance - Form 0.	As soon as sample is received
Deadline for receiving results - Form1	72 hours after receiving the sample
Preliminary Report.	March 2013
Final Report distributed to the Laboratories.	December 2013

Cost for shipment of the test material

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 **200 €**. For the payment procedures, each laboratory can specify their details and requests for invoices when applying for the test. Payment details are as follows:

BANK NAME: CAJAMAR - Caja Rural Sociedad Corporativa de Crédito

BANK ACCOUNT OWNER: Universidad de Almería

BANK ADDRESS: Office Number 990. Universidad de Almería. 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.):

Dr. Amadeo R. Fernández-Alba	EURL-FV amadeo@ual.es	+34 950015034
Ms. Carmen Ferrer Amate	EURL-FV cferrer@ual.es	+34 950015645
Mr. Octavio Malato Rodriguez	EURL-FV omalato@ual.es	+34 950214423

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 Almería, Spain.

Advisory Group

Dr. André de Kok, Senior Chemist, NVWA, Wageningen, The Netherlands.
Dr. Tuija Pihlström, Senior Chemist NFA, Uppsala, Sweden.
Dr. Sonja Masselter, Senior Chemist, AGES, Innsbruck, Austria.
Dr. Darinka Stajnbaher, Senior Chemist, IPH, Maribor, Slovenia.
Dr. Magnus Jezussek, Senior Chemist, LGL, Erlangen, Germany.
Dr. Miguel Gamón, Senior Chemist, Laboratorio Agroalimentario, Valencia, Spain.
Dr. Mette Erecius Poulsen, Senior Chemist, DTU, Copenhagen, Denmark.
Mr. Ralf Lippold, Senior Chemist, CVUA, Freiburg, Germany.
Dr. Michelangelo Anastassiades, Senior Chemist, CVUA, Stuttgart, Germany.