EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUIT AND VEGETABLES.

SCREENING METHODS 07
(EUPT-FV-SM07)

Pesticide Residues in Broccoli Homogenate

Final Report
(15th December 2015)

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 (University of Almería)

Dr. Milagros Mezcua, Chemist.	University of Almería
Ms. Carmen Ferrer, Chemist.	University of Almería
Mr. Octavio Malato, Chemist.	University of Almería
Ms. Ana Lozano, Chemist.	University of Almería
Ms. Mª del Mar Gómez, Agronomist.	University of Almería
Ms. Samanta Uclés, Chemist.	University of Almería
Ms. Ana Uclés, Chemist.	University of Almería
Mr. Łukasz Rajski, Chemist.	University of Almería
Dr. Sonia Herrera, Chemist.	University of Almería

Quality Control Group

Antonio Valverde, Senior Chemist	University of Almería, Spain.
Stewart Reynolds, Senior Chemist	Fera Science Ltd, York, United Kingdom.

Statistical Group

Carmelo Rodríguez, Senior Mathematician	University of Almería, Spain.

Advisory Group

Michelangelo Anastassiades, Senior Chemist	CVUA, Stuttgart, Germany.
Miguel Gamón, Senior Chemist	Laboratorio Agroalimentario, Valencia, Spain.
Magnus Jezussek, Senior Chemist	LGL, Erlangen, Germany.
André de Kok, Senior Chemist	NVWA, Wageningen, The Netherlands.
Ralf Lippold, Senior Chemist	CVUA, Freiburg, Germany.
Sonja Masselter, Senior Chemist	AGES, Institute for Food Safety, Innsbruck, Austria.
Tuija Pihlström, Senior Chemist	NFA, Uppsala, Sweden.
Mette Erecius Poulsen, Senior Chemist	NFI, Copenhagen, Denmark.
Darinka Štajnbaher, Senior Chemist	IPH, Maribor, Slovenia.
Finbarr O'Regan	The Pesticide Control Laboratory, Celbridge, Ireland
Philippe Gros	Laboratoire du SCL de Montpellier, France

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

CONTENT

EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUIT AND VEGETABLES. SCREENING METHODS 07

BACKGROUND

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

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

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

Participation in this PT remains on a voluntary basis. Besides this one, official laboratories have a significant number of mandatory PTs annually, given that the EURL-FV already organises the PT for quantitative multi-residue pesticide analysis (EUPT-FV17) over the same time period. Nevertheless, all FV- National Reference Laboratories (FV-NRLs) and FV-Official Laboratories (FV-OfLs) involved in the determination of pesticide residues in fruit and vegetables for the EU-coordinated monitoring programme, or for their own national programmes, were invited to take part.

DG-SANTE will have full access to all EUPT data including the individual lab-codes/lab-name keys. This report may be presented to the European Union Standing Committee on Plants, Animals, Food and Feed.

[1]Regulation (EC) No 396/2005, published in the OJ of the EU L70 of 16.03.2005, as last amended by Regulation 839/2008 published in the OJ of the EU L234 of 30.08.2008.
[2]Regulation (EC) No 882/2004 of the European Parliament and of the Council on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules. Published in the OJ of the EU L191 of 28.05.2004
[3]Commission Regulation (EC) No 776/2006 of 23 May 2006 - amending Annex VII of Regulation (EC) No 882/2004 of the European Parliament and of the Council as regards Community Reference Laboratories.

1. INTRODUCTION

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

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

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

The aim of the EURL-FV is for laboratories to be able to use mass-spectrometry-based screening methods routinely, following validation. This is in line with Document N° SANCO/12571/2013 (which supersedes Document No. SANCO/12495/2011) - Analytical quality control and method validation procedures for pesticide residues analysis in food and feed.

This EUPT-FV-SM07 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 SANTE.

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

2. TEST ITEMS

2.1 Preparation of the treated test item.

This proficiency test is based on the pesticide-residue analysis of broccoli. The broccoli plants were organically grown in Almería by the organisers in a greenhouse with passive ventilation - typically used for horticulture in the Almeria region ('raspa y amagado' type), in south-eastern Spain.

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

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

The pesticides used to spike the broccoli heads 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-SM07 were mainly chosen taking into account the following considerations:

- That they were not included in the EU-Coordinated Multiannual Control Programme for 2014 (Regulation (EC) 788/2012).
- That they had particularly acute toxicity and/or had low ARfD values.

Table 2.1 shows the 14 pesticides present in the broccoli sample. The pesticide treatments were carried out post-harvest using standard solutions. The test item was frozen (using liquid nitrogen) and chopped. The frozen minced broccoli was mixed in a constantly-spinning container until a homogeneous item was obtained. Finally, 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 present in the sample.

Pesticides			
Carbetamide	Chlorothalonil	Deltamethrin	Diafenthiuron
Dicrotophos	Dinocap	Fluazifop-P-butyl	Flubendiamide
Imidacloprid	Metosulam	Pencycuron	Prochloraz
	Promecarb	Spirotetramat	

2.2 Preparation of “blank” test item.

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

2.3 Homogeneity and stability tests.

The Organiser's homogeneity and stability tests associated with ‘quantitative’ PTs were conducted with a further acceptance criterion to those in the classical EUPT-FVs - the PT test item was analysed in order to identify the present pesticides, which were consistently confirmed to be above the Organiser's LODs.

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

The injection sequence of the 20 analyses by GC and LC was determined from a table of randomly-generated numbers. The statistical evaluation was performed according to the International Harmonized Protocol published by IUPAC, ISO and AOAC. The individual residues data from the homogeneity tests are given in table 2.3.1 and the stability tests in table 2.3.2. The acceptance criteria for the test item to be sufficiently homogenous for the proficiency test were that: $Ss^2 < c$, where Ss is the between-bottle sampling standard deviation and $c = F_1\sigma^2_{all} + F_2S^2_{an}$; F_1 and F_2 being constant values of 1.88 and 1.01, respectively, from the ten samples taken, and σ^2_{all} = 0.3 x FFP RSD(25 %) x the analytical sampling mean for all the pesticides. This was used to demonstrate that the between-bottle variance was not higher than the within-bottle variance.

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.	093 A	093 B	033 A	033 B	140 A	140 B	086 A	086 B	057 A	057 B	013 A	013 B	137 A	137 B	022 A	022 B	006 A	006 B	116 A	116 B	A. Cc (mg/kg)	$Ss^2 < c$ Pass/Fail
Carbetamide	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0,085	Pass
Chlorothalonil	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0,095	Pass
Deltamethrin	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0,079	Pass
Diafenthiuron	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0,012	Pass
Dicrotophos	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0,056	Pass
Dinocap	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0,105	Pass
Fluazifop-P-butyl	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.092	Pass
Flubendiamide	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0,147	Pass
Imidacloprid	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0,108	Pass
Metosulam	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0,076	Pass
Pencycuron	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0,061	Pass
Prochloraz	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0,049	Pass
Promecarb	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0,023	Pass
Spirotetramat*	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0,060	**Fail**

I: Identified A. Cc: Average Concentration *Only for informative purposes

Spirotetramat did not pass the homogeneity test, and for that reason, it will not be considered for the evaluation of the laboratories.

Six bottles, again chosen randomly, were analysed over a period of time to confirm the stability of the pesticides in the test item (three when the test items were shipped, then other three bottles a few days after the deadline for submitting results).

There was one further analysis of three bottles reproducing the sample shipment to see if there was any degradation of any of the pesticides present in the test item.

Table 2.3.2 Stability tests performed.

Test item No.	075 Shipment Day (16th March)	048 Shipment Day (16th March)	149 Shipment Day (16th March)	043 48h later Shipment Day (18th March)	064 48h later Shipment Day (18th March)	120 48h later Shipment Day (18th March)	034 Few days after deadline (20th March)	095 Few days after deadline (20th March)	138 Few days after deadline (20th March)
Carbetamide	I	I	I	I	I	I	I	I	I
Chlorothalonil	I	I	I	I	I	I	I	I	I
Deltamethrin	I	I	I	I	I	I	I	I	I
Diafenthiuron*	I	I	I	I	I	**NI**	I	I	**NI**
Dicrotophos	I	I	I	I	I	I	I	I	I
Dinocap	I	I	I	I	I	I	I	I	I
Fluazifop-P-butyl	I	I	I	I	I	I	I	I	I
Flubendiamide	I	I	I	I	I	I	I	I	I
Imidacloprid	I	I	I	I	I	I	I	I	I
Metosulam	I	I	I	I	I	I	I	I	I
Pencycuron	I	I	I	I	I	I	I	I	I
Prochloraz	I	I	I	I	I	I	I	I	I
Promecarb	I	I	I	I	I	I	I	I	I
Spirotetramat*	I	I	I	I	I	I	I	I	I

I: Identified NI: Not identified *Only for informative purposes

Diafenthuron did not pass the stability test, so it is excluded from the rest of the report and it will not be taken into account for the evaluation of the laboratories.

2.4 Distribution of test items and protocol to participants

Approximately 300 g of treated broccoli homogenate together with another 300 g of 'blank' broccoli homogenate were shipped to participants on 16th March 2015. 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 (single quadrupole, ion trap, ToF) and/or LC-MS (ToF and Orbitrap). However, extended targeted methods using LC-MS/MS (triple quadrupole, Q-trap, Q-ToF) or GC-MS/MS (triple quadrupole and ion trap) could also be used.

Before shipment, the laboratories received full instructions for the receipt and analysis of the test item although they were encouraged to use their own screening methods. These instructions, laid out as the General and Specific Protocols, were uploaded onto the EUPT-FV-SM07 web page, designed especially for this Proficiency Test. This information was also sent by e-mail to all participant laboratories. The Application Form was uploaded onto this same web site together with Form 0 (Sample Receipt) and Form 1 (Results). These allowed the evaluation of the mass-spectrometric screening methods that each of the participants used.

3. STATISTICAL METHODS

3.1 Type of results reported

The results evaluation is concerned with the results themselves matching the pesticides that the Organiser used to treat the sample; or otherwise stating a 'non-reported pesticide' or 'other reported pesticide' from those used to treat the sample. After receiving the results, the Organiser may consider further evaluation highlighted by important information received.

3.1.1 Other Reported Pesticides

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

> *Organiser's Note: Not all screening methods immediately provide sufficient information to allow full identification. In such cases, when they detect a pesticide in real life, laboratories normally do a follow-up confirmatory analysis: using, for example, LC-MS/MS and based on two transitions.*

3.1.2 Non-Reported Pesticides

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

4. RESULTS

4.1 Summary of reported results

Seventy-six laboratories agreed to participate in this seventh proficiency test on screening methods. Seventy laboratories submitted results on time. All results reported by the participants are given in Appendix 1. Graphical representations of the results reported are shown in Appendix 2. Details of the screening methods used are provided in Appendix 3 (available on the EUPT-SM07 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 Reported Results.

Pesticide	Reported		Not Reported	
	No. of laboratories	% of laboratories *	No. of laboratories	% of laboratories *
Carbetamide	47	67	23	33
Chlorothalonil	53	76	17	24
Deltamethrin	67	96	3	4
Dicrotophos	53	76	17	24
Dinocap	31	44	39	56
Fluazifop-P-butyl	57	81	13	19
Flubendiamide	44	63	26	37
Imidacloprid	63	90	7	10
Metosulam	43	61	27	39
Pencycuron	61	87	9	13
Prochloraz	62	89	8	11
Promecarb	47	67	23	33

* The % of laboratories is calculated based on the total number of laboratories submitting results (70 laboratories).

No other compounds were identified and quantified by the organizer.

4.1.1 Other Reported Pesticides

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

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

LABORATORY CODE	OTHER REPORTED PESTICIDES
Lab001*	Biphenyl
Lab007	Clopyralid, Difenoconazole
Lab009*	Binapacryl, Ethiofencarb-sulfone, Fluquinconazole, Phosmet oxon
Lab012	Dimefuron, Fludioxonil, Metamitron, Monocrotophos
Lab014	Fluoroglycofen, Trichlamide
Lab015	Fluquinconazole, Terbacil

LABORATORY CODE	OTHER REPORTED PESTICIDES
Lab017	Terbucarb, Tralomethrin
Lab020	Benoxacor, Ditalimfos
Lab025*	2.4 DMF, Aldicarb sulfone, Allethrin, Biphenyl, Monocrotophos, Orthophenylphenol, Parathion methyl, Propham
Lab027*	Benzthiazuron
Lab032	Fluoroglycofen, Trichlamide
Lab033*	Bipyridyl, Fenobucarb, Mesosulfuron
Lab037	Difenoconazole, Thiabendazole
Lab042*	Dimefuron
Lab045*	Fenobucarb, Fenpyrazamine
Lab052	Dieldrin
Lab055	Orthophenylphenol
Lab058*	Isobornyl thiocyanoacetate, Isoprocarb, Metazachlor, Spiromesifen
Lab060	Tebuconazole
Lab061	Benomil, Chlorpyrifos, o-hydroxibiphenyl
Lab063	Acrinathrin, Azoxystrobin, Bitertanol, Boscalid, Buprofezin, Carbaril, Chlorpyrifos, Chlorpyrifos methyl, Cyprodinil, Diazinon, Dichlorvos, Dimethoate, Endosulfan alfa, Endosulfan beta, Endosulfan sulfate, Fenitrothion, Fenthion, Isophenfos methyl, Kresoxim methyl, Malathion, Metalaxyl, Methidathion, Myclobutanil, Oxadixyl, Pirimicarb, Procymidone, Tolyfluanid
Lab066	Endosulfan sulfate, Fipronil, Fludioxonil
Lab071	Fenpyrazamine, Hydramethylnon
Lab072	Acephate, Chlorfenapyr, Crotoxyphos, Fenuron, Metamitron, Monocrotophos, Oxamyl, Quinoxyfen, Spiroxamine

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

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

4.1.2 Non-Reported Pesticides

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

4.2 Concentration levels.

Fourteen pesticides were used to spike the broccoli test item at different levels, in the range between 0.020 mg/kg and 0.200 mg/kg according to the homogeneity table 2.3.1, most of them in concentrations lower than 0.100 mg/kg (spirotetramat did not pass the homogeneity test and diafenthuron did not pass the stability test, so they were excluded from the rest of the report).

No other compounds have been identified and quantified by the organizer at concentrations below 0.010 mg/kg.

No compounds present in the blank were identified and quantified by the organizer at concentrations below 0.010 mg/kg.

4.3 Assessment of laboratory performance.

No z-score values were calculated as no numerical results were reported by the participants. However, classification was considered important, based on the number of results each laboratory reported. Table 4.3.1 classifies the laboratories according to the number of present pesticides reported.

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

Laboratory Code	No of Reported Pesticides	% of Reported Pesticides	Other Reported Pesticides Not Confirmed by the Organiser
Lab005*	12	100	
Lab011*	12	100	
Lab016	12	100	
Lab021	12	100	
Lab024	12	100	
Lab030	12	100	
Lab036	12	100	
Lab040	12	100	
Lab059	12	100	
Lab074	12	100	
Lab076	12	100	
Lab027*	12	100	1
Lab014	12	100	2
Lab032	12	100	2
Lab004	11	92	
Lab006	11	92	
Lab013	11	92	
Lab019	11	92	
Lab022*	11	92	
Lab023	11	92	
Lab028	11	92	
Lab044	11	92	
Lab046*	11	92	
Lab047	11	92	
Lab049	11	92	
Lab067	11	92	
Lab070	11	92	
Lab078	11	92	
Lab001*	11	92	1
Lab045*	11	92	2
Lab071	11	92	2
Lab012	11	92	4
Lab058*	11	92	4
Lab072	11	92	9
Lab003*	10	83	

Laboratory Code	No of Reported Pesticides	% of Reported Pesticides	Other Reported Pesticides Not Confirmed by the Organiser
Lab035	10	83	
Lab043	10	83	
Lab042*	10	83	1
Lab061	10	83	3
Lab009*	10	83	4
Lab010	9	75	
Lab026	9	75	
Lab056*	9	75	
Lab057	9	75	
Lab069	9	75	
Lab007	9	75	2
Lab020	9	75	2
Lab029	8	67	
Lab031	8	67	
Lab050*	8	67	
Lab033*	8	67	3
Lab065	7	58	
Lab068*	7	58	
Lab017	7	58	2
Lab025*	7	58	8
Lab051	6	50	
Lab075*	6	50	
Lab015	6	50	2
Lab053	5	42	
Lab062	5	42	
Lab073	5	42	
Lab077	5	42	
Lab055	4	33	1
Lab060	3	25	1
Lab037	3	25	2
Lab002	2	17	
Lab048	2	17	
Lab052	2	17	1
Lab066	2	17	3
Lab063	1	8	27

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

The extraction methods used by the laboratories, the chromatographic techniques, detectors, instrumentation, etc… are detailed in Appendix 3 (available only on the EUPT-SM07 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
Carbetamide	52	4	4	48	15
Chlorothalonil	55	51	17	4	3
Deltamethrin	75	57	15	18	9
Dicrotophos	67	22	8	45	16
Dinocap	34	1	1	33	10
Fluazifop-P-butyl	69	25	11	44	15
Flubendiamide	48	2	1	46	16
Imidacloprid	68	1	1	67	18
Metosulam	48	1	1	47	17
Pencycuron	67	5	2	62	18
Prochloraz	74	18	7	56	17
Promecarb	52	11	4	41	12

Note: The number of reports for each of the pesticides could be different to the reports shown in Table 4.1.1 because a particular laboratory might have analysed one pesticide with more than one technique.

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

Table 4.3.3 shows the number and percentage of the pesticides present in the sample which were reported by each laboratory. National Reference Laboratories are marked with an asterisk.

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

Laboratory Code	Number of Present Pesticides Reported	% of Present Pesticides Reported
Lab001*	11	92
Lab002	2	17
Lab003*	10	83
Lab004	11	92
Lab005*	12	100
Lab006	11	92
Lab007	9	75
Lab009*	10	83
Lab010	9	75
Lab011*	12	100
Lab012	11	92
Lab013	11	92
Lab014	12	100
Lab015	6	50
Lab016	12	100

Laboratory Code	Number of Present Pesticides Reported	% of Present Pesticides Reported
Lab017	7	58
Lab019	11	92
Lab020	9	75
Lab021	12	100
Lab022*	11	92
Lab023	11	92
Lab024	12	100
Lab025*	7	58
Lab026	9	75
Lab027*	12	100
Lab028	11	92
Lab029	8	67
Lab030	12	100
Lab031	8	67
Lab032	12	100
Lab033*	8	67
Lab035	10	83
Lab036	12	100
Lab037	3	25
Lab040	12	100
Lab042*	10	83
Lab043	10	83
Lab044	11	92
Lab045*	11	92
Lab046*	11	92
Lab047	11	92
Lab048	2	17
Lab049	11	92
Lab050*	8	67
Lab051	6	50
Lab052	2	17
Lab053	5	42
Lab055	4	33
Lab056*	9	75
Lab057	9	75
Lab058*	11	92
Lab059	12	100
Lab060	3	25
Lab061	10	83
Lab062	5	42
Lab063	1	8
Lab065	7	58
Lab066	2	17
Lab067	11	92
Lab068*	7	58

Laboratory Code	Number of Present Pesticides Reported	% of Present Pesticides Reported
Lab069	9	75
Lab070	11	92
Lab071	11	92
Lab072	11	92
Lab073	5	42
Lab074	12	100
Lab075*	6	50
Lab076	12	100
Lab077	5	42
Lab078	11	92

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

5. CONCLUSIONS

Seventy-six laboratories agreed to participate in this seventh proficiency test on screening methods. Seventy laboratories submitted results on time. Seventeen of the laboratories were National Reference Laboratories for Fruit and Vegetables (marked with an asterisk on the graphs and tables) representing twenty one EU Member States. In addition to these, 1 EFTA country (Switzerland) and five non-EU/EFTA countries (China, Egypt, Kenya, Serbia and Turkey) participated in this European Union Proficiency Test.

Most laboratories analysed the test item using methods based on both gas and liquid chromatography, combined with mass spectrometric detection. In the case of GC-MS analysis, full-scan acquisition with associated target-library software (covering a large number of pesticides) was used by the majority of the laboratories. In the case of LC-MS analysis, targeted acquisition methods using triple quadrupole instruments were the most widely used. Of 709 detections, 238 were made using full-scan, meaning 33.6 % of detections (166 by full scan LC techniques and 72 by full scan GC techniques).

Fourteen of the 70 laboratories were able to detect all 12 present pesticides in the broccoli test item. Twelve laboratories detected less than 50 % of the pesticides present.

Sixty seven percent of the laboratories (47 laboratories) that reported results were able to find more than 70 % of the evaluated pesticides.

Twenty-four participants reported 73 different pesticides which were not present in the broccoli test items. Whether this should be judged as poor performance, or not, depends on how each participant would act on these positive findings in routine analysis. If the reported pesticide was reported as positive with no further identifying confirmation, then the result would be a false positive and hence erroneous monitoring data would be reported. If the reported pesticide is regarded simply as 'suspect' or 'indicatively present', leading to additional analysis to confirm identity before reporting the result, then those pesticides indicated as 'other reported pesticides' in this report are not really an issue.

As in previous years, EUPT-SM interlaboratory tests on wide-scope screening methods showed that such an approach can substantially expand the scope of pesticide residue analysis. This is especially useful for pesticides not frequently found in food and feed, or not monitored by the laboratories because they are not part of the EU-Coordinated Programme. The use of screening methods can greatly increase the chance of detecting less commonly found pesticides. However, the test also revealed that improvements in scope (both in number and the choice of pesticides included) and verification of the screening methods performance (i.e. validation) are necessary to improve the reliability of such methods.

6. SUGGESTIONS FOR FUTURE WORK

The Organiser and the Scientific Committee consider that screening methods have provided additional value to the current quantitative multiresidue methods routinely used for monitoring purposes. The results of this test are most encouraging, but also indicate the need for continued evaluation of screening methods. Therefore, further proficiency tests will be organised to provide support to those laboratories using screening methods in order to extend their use and improve their reliability. These methods will be used more and more as screens/filters, to make routine laboratory work easier and faster. The need for screening method validation has been recognised and guidelines for such validation have been prepared and included in the SANCO Document/11945/2015.

Next year, the matrix of the test item will be spinach homogenate. For the first time in this type of PTs of screening methods, participants will be invited to report the estimated concentration of the pesticides identified. The concentration value will be used for informative purposes only, and not for the evaluation of the laboratories.

7. BIBLIOGRAPHIC 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.
- 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 7th 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 University of Almería for the use of their facilities.

APPENDIX 1. Results

Laboratory Code Total No of Reporting Laboratories = 70	Evaluated Pesticides (12)												Reported Pesticides by Laboratory	% of Reported Pesticides by Laboratory
	Carbetamide	Chlorothalonil	Deltamethrin	Dicrotophos	Dinocap	Fluazifop-P-butyl	Flubendiamide	Imidacloprid	Metosulam	Pencycuron	Prochloraz	Promecarb		
Lab001*	R	R	R	R	R	R	R	R	R	R	R		**11**	**92**
Lab002		R		R									**2**	**17**
Lab003*	R	R	R	R		R	R	R	R	R	R		**10**	**83**
Lab004	R		R	R	R	R	R	R	R	R	R	R	**11**	**92**
Lab005*	R	R	R	R	R	R	R	R	R	R	R	R	**12**	**100**
Lab006		R	R	R	R	R	R	R	R	R	R	R	**11**	**92**
Lab007	R	R	R		R	R		R	R	R	R		**9**	**75**
Lab009*	R	R	R	R		R		R	R	R	R	R	**10**	**83**
Lab010	R	R	R		R	R	R	R		R	R		**9**	**75**
Lab011*	R	R	R	R	R	R	R	R	R	R	R	R	**12**	**100**
Lab012	R	R	R	R	R	R	R	R	R	R	R		**11**	**92**
Lab013	R	R	R	R		R	R	R	R	R	R	R	**11**	**92**
Lab014	R	R	R	R	R	R	R	R	R	R	R	R	**12**	**100**
Lab015	R					R	R		R	R	R		**6**	**50**
Lab016	R	R	R	R	R	R	R	R	R	R	R	R	**12**	**100**
Lab017	R		R			R		R		R	R	R	**7**	**58**
Lab019		R	R	R	R	R	R	R	R	R	R	R	**11**	**92**
Lab020	R		R	R		R		R	R	R	R	R	**9**	**75**
Lab021	R	R	R	R	R	R	R	R	R	R	R	R	**12**	**100**
Lab022*	R	R	R	R		R	R	R	R	R	R	R	**11**	**92**
Lab023	R		R	R	R	R	R	R	R	R	R	R	**11**	**92**
Lab024	R	R	R	R	R	R	R	R	R	R	R	R	**12**	**100**
Lab025*		R	R	R		R		R			R	R	**7**	**58**
Lab026	R	R	R	R			R	R		R	R	R	**9**	**75**
Lab027*	R	R	R	R	R	R	R	R	R	R	R	R	**12**	**100**
Lab028	R	R	R	R		R	R	R	R	R	R	R	**11**	**92**
Lab029		R	R	R			R	R		R	R	R	**8**	**67**
Lab030	R	R	R	R	R	R	R	R	R	R	R	R	**12**	**100**
Lab031		R	R			R		R	R	R	R	R	**8**	**67**
Lab032	R	R	R	R	R	R	R	R	R	R	R	R	**12**	**100**
Lab033*	R		R	R		R		R	R	R	R		**8**	**67**
Lab035		R	R	R	R	R	R	R		R	R	R	**10**	**83**
Lab036	R	R	R	R	R	R	R	R	R	R	R	R	**12**	**100**
Lab037			R			R				R			**3**	**25**
Lab040	R	R	R	R	R	R	R	R	R	R	R	R	**12**	**100**
Lab042*	R		R	R		R	R	R	R	R	R	R	**10**	**83**
Lab043	R		R	R		R	R	R	R	R	R	R	**10**	**83**
Lab044	R	R	R	R	R	R	R	R		R	R	R	**11**	**92**
Lab045*	R		R	R	R	R	R	R	R	R	R	R	**11**	**92**
Lab046*	R	R	R	R		R	R	R	R	R	R	R	**11**	**92**
Lab047	R	R	R	R		R	R	R	R	R	R	R	**11**	**92**
Lab048			R					R					**2**	**17**
Lab049	R	R	R		R	R	R	R	R	R	R	R	**11**	**92**
Lab050*		R	R	R	R	R		R		R	R		**8**	**67**
Lab051		R	R					R	R	R	R		**6**	**50**
Lab052		R	R										**2**	**17**
Lab053		R	R					R		R	R		**5**	**42**
Lab055			R			R		R		R			**4**	**33**
Lab056*	R		R	R		R	R	R		R	R	R	**9**	**75**

APPENDIX 1. Results

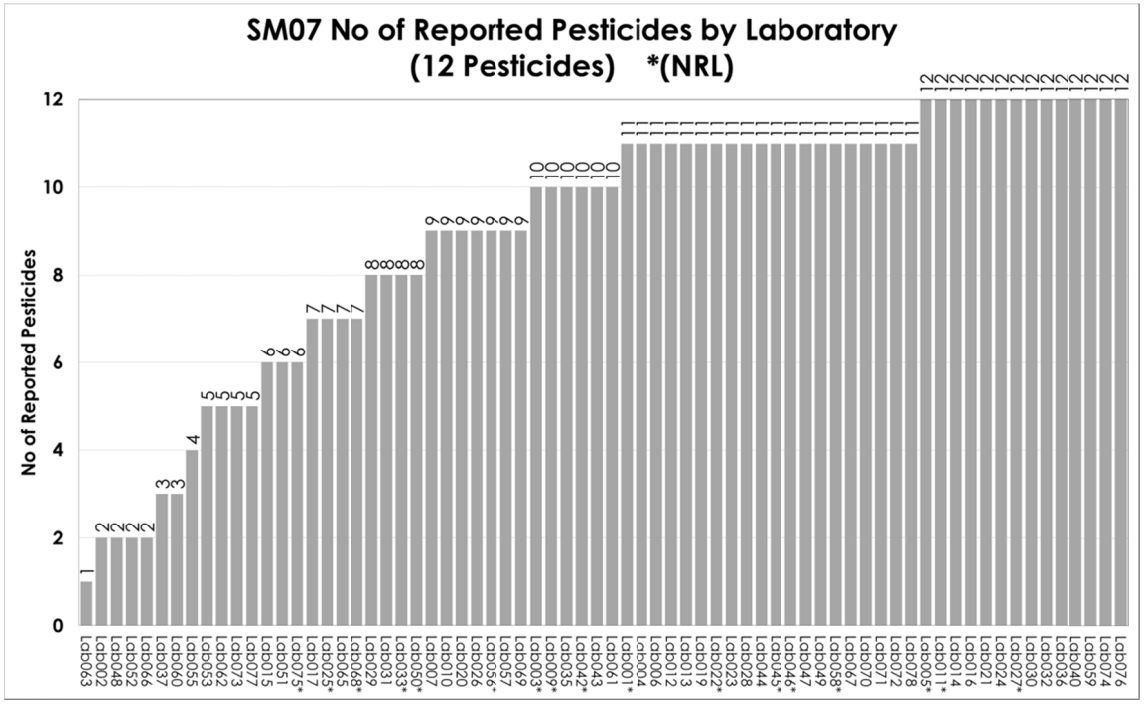
Laboratory Code Total No of Reporting Laboratories = 70	Evaluated Pesticides (12)												Reported Pesticides by Laboratory	% of Reported Pesticides by Laboratory
	Carbetamide	Chlorothalonil	Deltamethrin	Dicrotophos	Dinocap	Fluazifop-P-butyl	Flubendiamide	Imidacloprid	Metosulam	Pencycuron	Prochloraz	Promecarb		
Lab057	R	R	R		R	R	R	R		R	R		**9**	**75**
Lab058*	R	R	R	R		R	R	R	R	R	R	R	**11**	**92**
Lab059	R	R	R	R	R	R	R	R	R	R	R	R	**12**	**100**
Lab060		R	R					R					**3**	**25**
Lab061	R	R	R	R		R		R	R	R	R	R	**10**	**83**
Lab062			R	R		R					R	R	**5**	**42**
Lab063		R											**1**	**8**
Lab065			R	R		R		R		R	R	R	**7**	**58**
Lab066		R	R										**2**	**17**
Lab067	R	R	R	R		R	R	R	R	R	R	R	**11**	**92**
Lab068*	R		R	R	R			R		R	R		**7**	**58**
Lab069		R	R	R	R	R		R		R	R	R	**9**	**75**
Lab070	R	R	R	R	R	R	R	R		R	R	R	**11**	**92**
Lab071	R	R	R	R		R	R	R	R	R	R	R	**11**	**92**
Lab072	R	R	R	R		R	R	R	R	R	R	R	**11**	**92**
Lab073		R	R			R		R			R		**5**	**42**
Lab074	R	R	R	R	R	R	R	R	R	R	R	R	**12**	**100**
Lab075*		R	R	R				R		R	R		**6**	**50**
Lab076	R	R	R	R	R	R	R	R	R	R	R	R	**12**	**100**
Lab077			R	R				R		R	R		**5**	**42**
Lab078	R	R	R	R		R	R	R	R	R	R	R	**11**	**92**
Reported Pesticides	**47**	**53**	**67**	**53**	**31**	**57**	**44**	**63**	**43**	**61**	**62**	**47**		
% of Reported Pesticides	**67**	**76**	**96**	**76**	**44**	**81**	**63**	**90**	**61**	**87**	**89**	**67**		

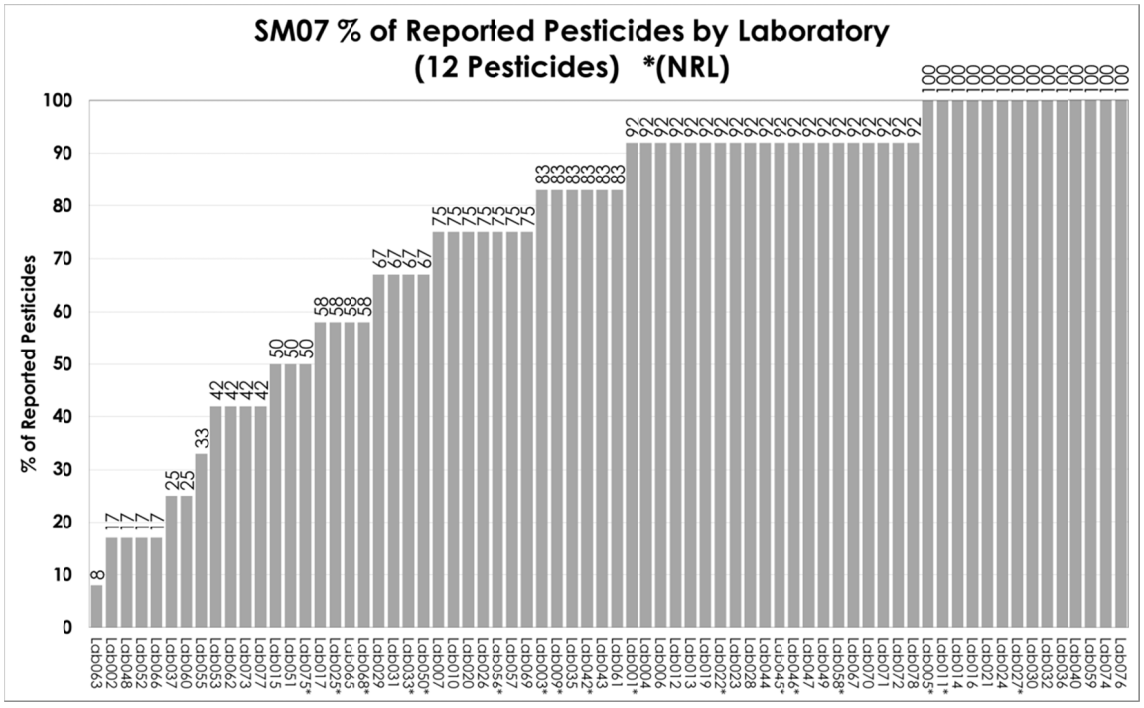
R: Reported pesticide

*NRLs from EU

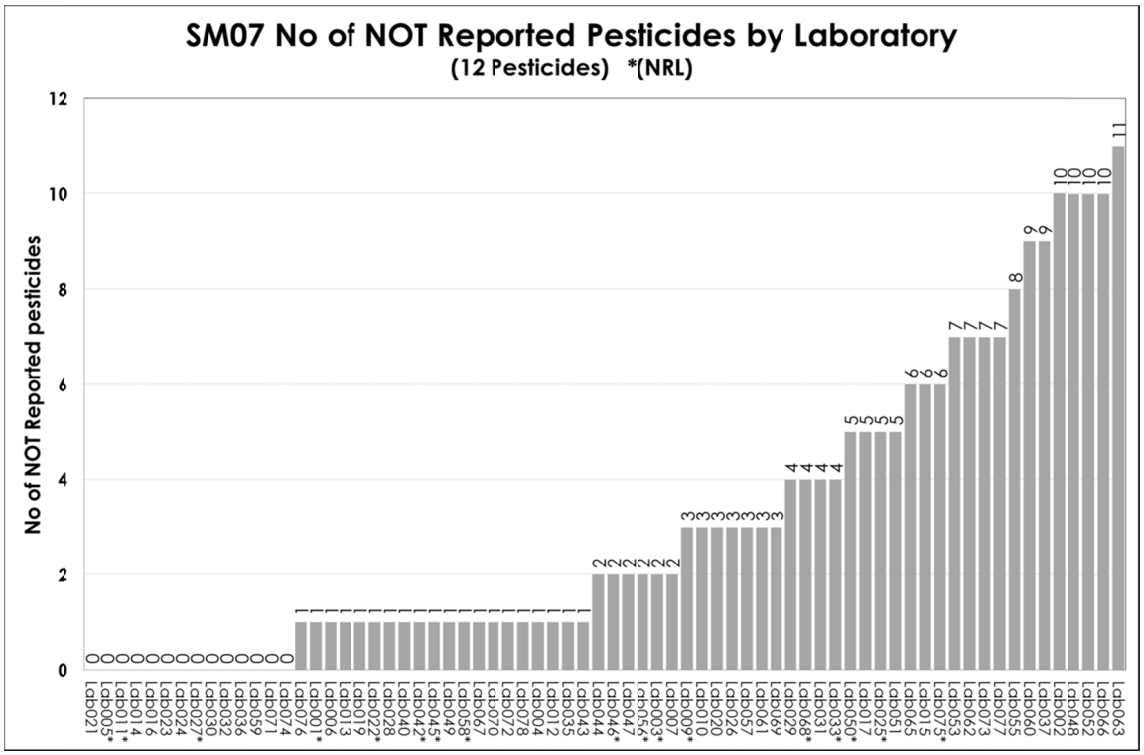
No other compounds have been reported or identified and quantified by the organizer.

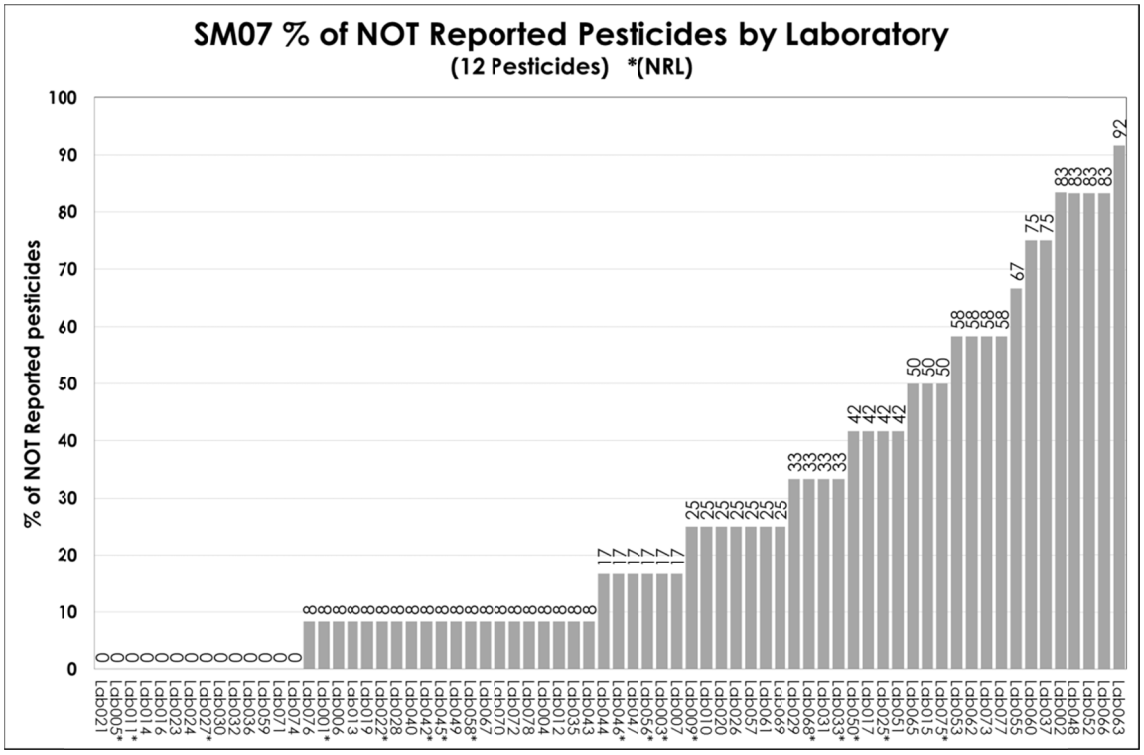
APPENDIX 2. Graphical Representations

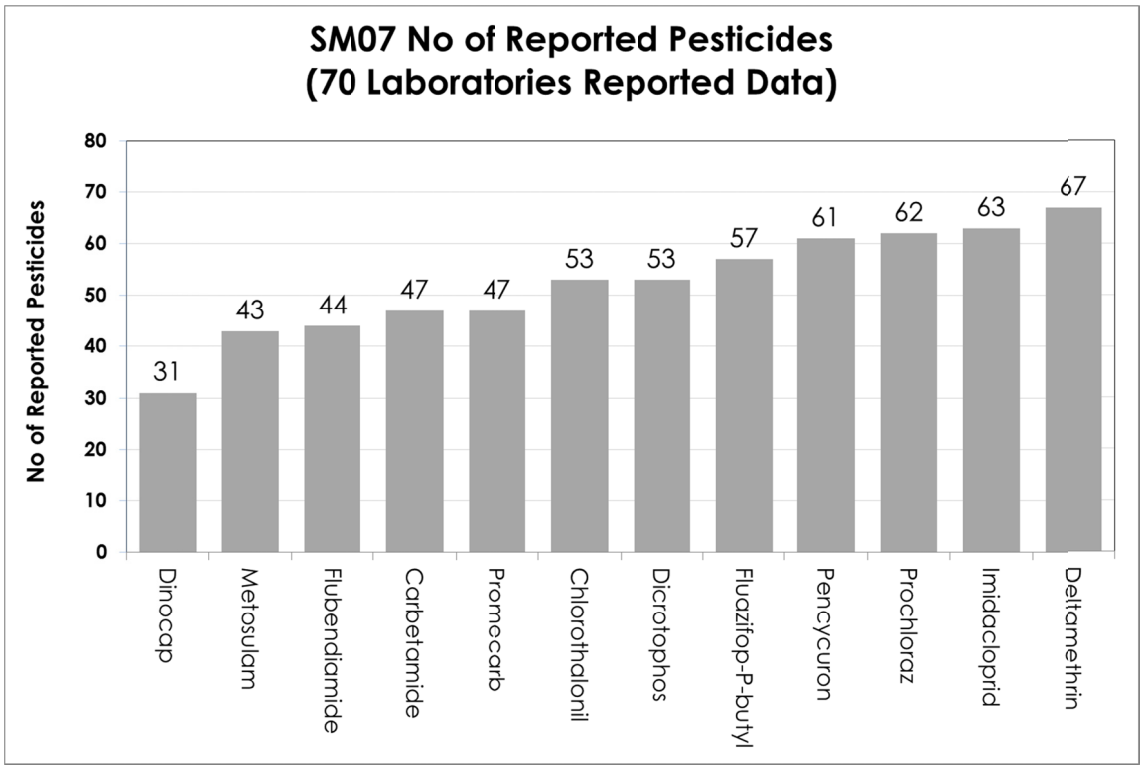


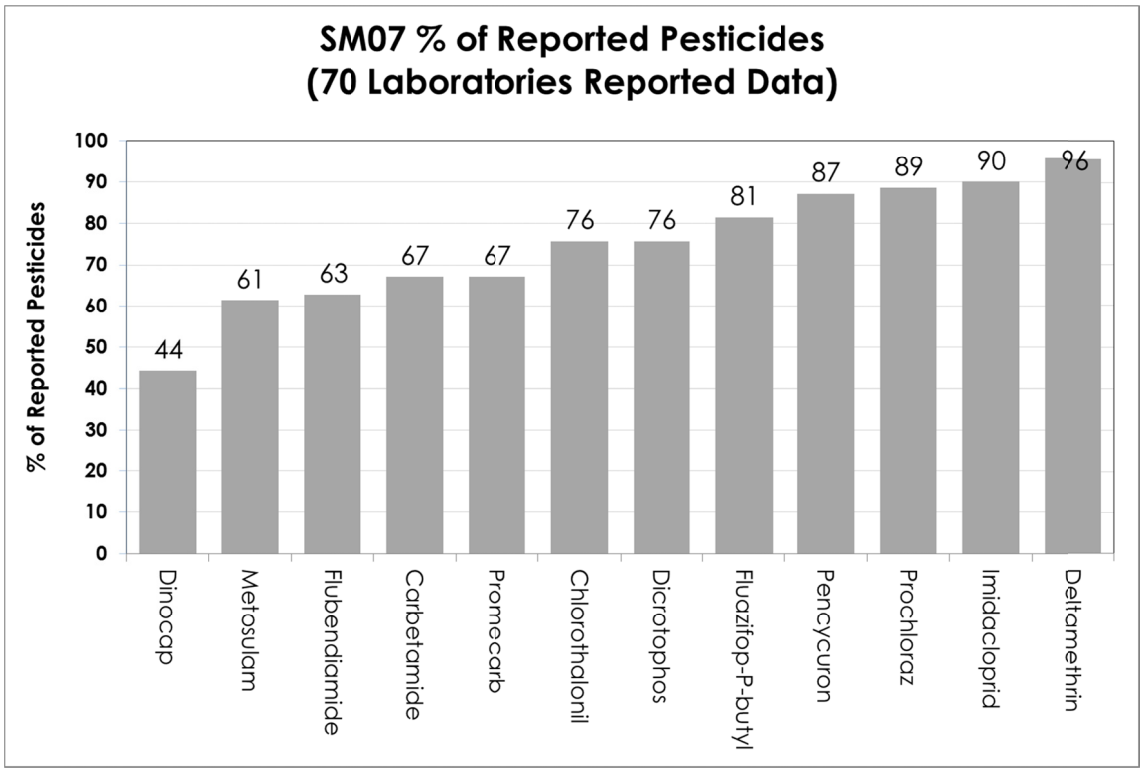


APPENDIX 2. Graphical Representations



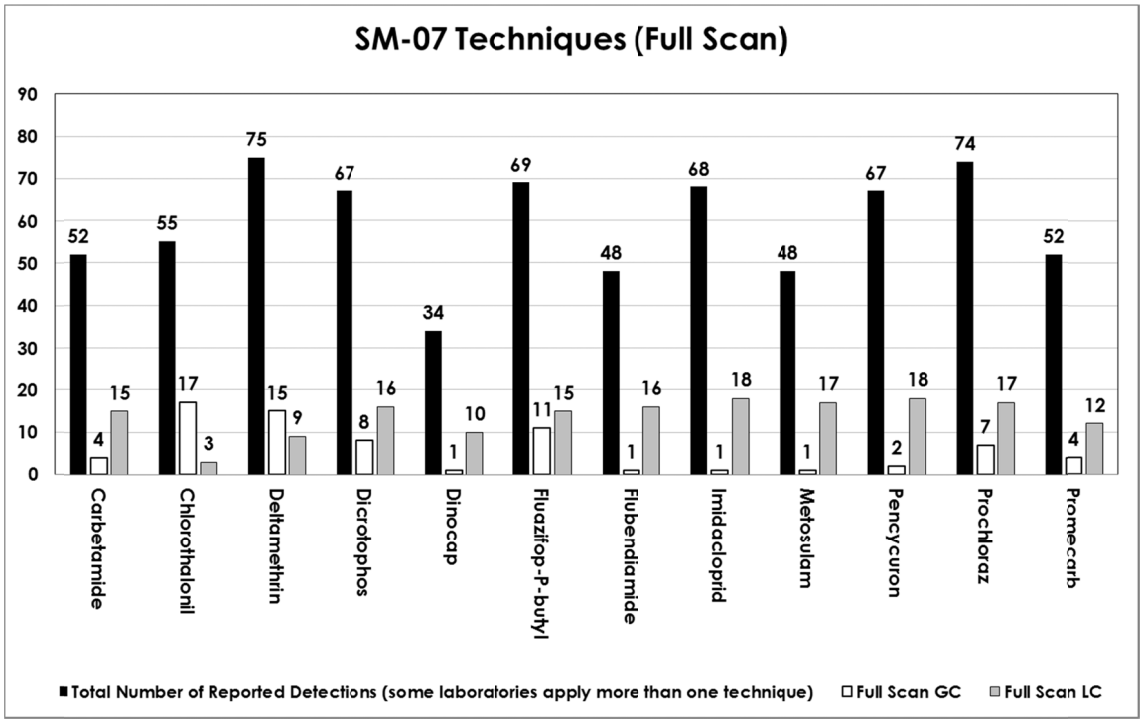



SM07 No of Reported Pesticides
(70 Laboratories Reported Data)
No of Reported Pesticides
0
10
20
30
40
50
60
70
80
31
43
44
47
47
53
53
57
61
62
63
67
Dinocap
Metosulam
Flubendiamide
Carbetamide
Promecarb
Chlorothalonil
Dicrotophos
Fluazifop-P-butyl
Pencycuron
Prochloraz
Imidacloprid
Deltamethrin


SM07 % of Reported Pesticides
(70 Laboratories Reported Data)
% of Reported Pesticides
0
10
20
30
40
50
60
70
80
90
100
44
61
63
67
67
76
76
81
87
89
90
96
Dinocap
Metosulam
Flubendiamide
Carbetamide
Promecarb
Chlorothalonil
Dicrotophos
Fluazifop-P-butyl
Pencycuron
Prochloraz
Imidacloprid
Deltamethrin

APPENDIX 2. Graphical Representations

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



ANNEX 1. List of Laboratories that participate in EUPT-FV-SM07.

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
AUSTRIA	AUSTRIAN AGENCY FOR HEALTH AND FOOD SAFETY (AGES GMBH), DEPARTMENT FOR PESTICIDE AND FOOD ANALYTICS (PLMA)	INNSBRUCK	YES
BELGIUM	LOVAP NV	GEEL	YES
BELGIUM	FYTOLAB BELGIUM CVBA	ZWIJNAARDE	YES
CHINA	SHANGHAI MUNICIPAL CENTER FOR DISEASE CONTROL AND PREVENTION	SHANGHAI	YES
CROATIA	EUROINSPEKT-CROATIAKONTROLA	ZAGREB	YES
CROATIA	FACULTY OF FOOD TECHNOLOGY AND BIOTECHNOLOGY, FOOD CONTROL CENTER, ZAGREB, CROATIA	ZAGREB	NO
CYPRUS	PESTICIDES RESIDUES LABORATORY OF THE STATE GENERAL LABORATORY OF THE MINISTRY OF HEALTH	NICOSIA	YES
CZECH REPUBLIC	CZECH AGRICULTURE AND FOOD INSPECTION AUTHORITY	BRNO	YES
CZECH REPUBLIC	UNIVERSITY OF CHEMISTRY AND TECHNOLOGY, DEPARTMENT OF FOOD ANALYSIS AND NUTRITION	PRAGUE 6	YES
CZECH REPUBLIC	CENTRAL INSTITUTE FOR SUPERVISING AND TESTING IN AGRICULTURE	BRNO	YES
DENMARK	NATIONAL FOOD INSTITUTE, TECHNICAL UNIVERSITY OF DENMARK	LYNGBY	YES
EGYPT	CENTRAL LAB OF RESIDUE ANALYSIS OF PESTICIDES AND HEAVY METALS IN FOODS	GIZA	YES
ESTONIA	LABORATORY FOR RESIDUES AND CONTAMINANTS, AGRICULTURAL RESEARCH CENTRE	SAKU	YES
FINLAND	FINNISH CUSTOMS LABORATORY	ESPOO	YES
FRANCE	INOVALYS LE MANS	LE MANS	YES
FRANCE	LABORATOIRE DU SCL DE MONTPELLIER	MONTPELLIER	YES
FRANCE	CERECO SUD	LIEU SAINT AMAND	YES
FRANCE	ANSES- LSAL	MAISONS-ALFORT CEDEX	YES
FRANCE	SCL - LABORATOIRE DE MASSY	MASSY CEDEX	YES
GERMANY	EUROFINS DR. SPECHT LABORATORIEN GMBH	DÜSSELDORF	YES
GERMANY	LUFA-ITL GMBH	KIEL	YES
GERMANY	NDS. LANDESAMT FUER VERBRAUCHERSCHUTZ UND LEBENSMITTELSICHERHEIT, LVI OL	BERLIN	YES
GERMANY	FEDERAL OFFICE OF CONSUMER PROTECTION AND FOOD SAFETY (BVL)	BERLIN	YES
GERMANY	LABOR FRIEDLE GMBH	TEGERNHEIM	YES
GERMANY	LTZ AUGUSTENBERG	BERLIN	YES
GERMANY	BAYERISCHES LANDESAMT FÜR GESUNDHEIT UND LEBENSMITTELSICHERHEIT	BERLIN	YES
GERMANY	CHEMICAL AND VETERINARY ANALYTICAL INSTITUTE RHINE-RUHR-WUPPER	BERLIN	YES
GERMANY	GALAB LABORATORIES GMBH	HAMBURG	YES
GREECE	GENERAL CHEMICAL STATE LABORATORY	ATHENS	YES
GREECE	BENAKI PHYTOPATHOLOGICAL INSTITUTE, PESTICIDE RESIDUES LABORATORY	KIPHISSIA (ATHENS)	YES

ANNEX 1. List of Laboratories that participate in EUPT-FV-SM07

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
HUNGARY	NATIONAL FOOD CHAIN OFFICE DIRECTORAT OF PLANT PROTECTION, SOIL CONSERVATION AND AGRI-ENVIRONMENT PESTICIDE RESIDUE ANALYTICAL LABORATORY, SZOLNOK	BUDAPEST	YES
HUNGARY	NATIONAL FOOD CHAIN SAFETY OFFICE, DPPSCA, PESTICIDE RESIDUE ANALYTICAL LABORATORY, MISKOLC	BUDAPEST	YES
HUNGARY	NATIONAL FOOD CHAIN SAFETY OFFICE, PESTICIDE RESIDUE ANALYTICAL LABORATORY OF HODMEZOVASARHELY	BUDAPEST	YES
HUNGARY	NFCSO PESTICIDE ANALYTICAL LABORATORY, VELENCE	BUDAPEST	YES
IRELAND	THE PESTICIDE CONTROL LABORATORY	CELBRIDGE	NO
ITALY	LABORATORIO DI SANITA' PUBBLICA AZIENDA SANITARIA DI FIRENZE	FIRENZE	YES
ITALY	ARPALAZIO SEZIONE DI LATINA	RIETI	NO
ITALY	ASL MILANO - LABORATORIO DI PREVENZIONE	MILANO	YES
ITALY	APPA TRENTO	TRENTO	YES
ITALY	ISTITUTO SUPERIORE DI SANITA' - REPARTO ANTIPARASSITARI	ROME	YES
ITALY	LANDESAGENTUR FÜR UMWELT-LABOR FÜR CHROMATOGRAPHIE	BOLZANO	YES
ITALY	ARPA FVG LABORATORIO UNICO MULTISITO - SEDE DI PORDENONE	PALMANOVA	YES
ITALY	ARPACAMPANIA LABORATORIO REGIONALE MICOTOSSINE E FITOFARMACI	NAPOLI	YES
ITALY	ARPA VENETO-SL VERONA	PADOVA	YES
ITALY	ISTITUTO ZOOPROFILATTICO SPERIMENTALE DEL LAZIO E DELLA TOSCANA	ROMA	YES
KENYA	AGRIQ-QUEST	NAIROBI	YES
LATVIA	INSTITUTE OF FOOD SAFETY, ANIMAL HEALTH AND ENVIRONMENT BIOR	RIGA	YES
NORWAY	BIOFORSK, PLANT HEALTH AND PLANT PROTECTION, PESTICIDE CHEMISTRY SECTION	AAS	NO
POLAND	SGS POLSKA SP. Z O.O. LAB. SRODOWISKOWE	WARSZAWA	YES
POLAND	RESEARCH INSTITUTE OF HORTICULTURE, FOOD SAFETY LABORATORY	SKIERNIEWICE	YES
ROMANIA	LABORATORY FOR PESTICIDES RESIDUES CONTROL IN PLANTS AND VEGETABLES	VOLUNTARI	YES
ROMANIA	REGIONAL LABORATORY FOR DETERMINATION OF PESTICIDE RESIDUES IN PLANT AND PLANT PRODUCTS MURES	TARGU MURES	YES
SERBIA	CENTER FOR FOOD ANALYSIS	BELGRADE	YES
SLOVENIA	NATIONAL LABORATORY FOR HEALTH, ENVIRONMENT AND FOOD (DEP. FOR CHEM. ANAL. MARIBOR)	MARIBOR	YES
SPAIN	LABORATORIO KUDAM,S.L	PILAR DE LA HORADADA	YES
SPAIN	LABORATORIO AGROALIMENTARIO Y DE SANIDAD ANIMAL	EL PALMAR (MURCIA)	YES
SPAIN	LABORATORIO AGROALIMENTARIO Y AMBIENTAL DE CASTILLA LA MANCHA	TOLEDO	NO
SPAIN	LABORATORIO PRODUCCIÓN Y SANIDAD VEGETAL	MENGIBAR (JAÉN)	YES
SPAIN	CNTA	SAN ADRIAN (NAVARRA)	YES
SPAIN	AGRICULTURAL AND PHYTOPATHOLOGICAL LABORATORY OF GALICIA	ABEGONDO. A CORUÑA	NO
SPAIN	LABORATORIO AGROALIMENTARIO VALENCIA	BURJASSOT. VALENCIA	YES

ANNEX 1. List of Laboratories that participate in EUPT-FV-SM07.

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
SPAIN	LABORATORIO AGROALIMENTARIO DE GRANADA	ATARFE (GRANADA)	YES
SPAIN	LABORATORIOS ECOSUR, S.A.	LORQUI MURCIA	YES
SPAIN	LABORATORIO DE PRODUCCIÓN Y SANIDAD VEGETAL DE ALMERÍA	LA MOJONERA	YES
SPAIN	ANALYTICA ALIMENTARIA GMBH, SUCURSAL EN ESPAÑA	ALMERIA	YES
SPAIN	SERVICIOS PERIFERICOS DE LA CONSEJERIA DE SANIDAD Y ASUNTOS SOCIALES, LABORATORIO DE SALUD PUBLICA	CUENCA	YES
SPAIN	LABORATORI AGROALIMANTARI-DAAM	VILASSAR DE MAR	YES
SPAIN	LABORATORIO AGROALIMENTARIO DE EXTREMADURA	CÁCERES	YES
SPAIN	LABORATORIO QUÍMICO MICROBIOLÓGICO, S.A	MURCIA	YES
SWEDEN	SWEDISH NATIONAL FOOD AGENCY, SCIENCE DEPARTMENT, CHEMISTRY DIVISION	STRÖMSUND	YES
SWEDEN	EUROFINS FOOD & FEED TESTING SWEDEN AB	FAGERSTA	YES
SWITZERLAND	KANTONALES LABOR ZÜRICH	ZÜRICH	YES
THE NETHERLANDS	RIKILT - INSTITUTE OF FOOD SAFETY	WAGENINGEN	YES
THE NETHERLANDS	NVWA - NETHERLANDS FOOD AND CONSUMER PRODUCT SAFETY AUTHORITY	UTRECHT	YES
TURKEY	ÖZEL MSM GIDA KONTROL LABORATUVARI VE DAN. HIZ. TIC. A.S.	MERSIN	YES
UNITED KINGDOM	FOOD AND ENVIRONMENT RESEARCH AGENCY	YORK	YES