

EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUIT AND VEGETABLES.

## SCREENING METHODS 11

### (EUPT-FV-SM11)

## Pesticide Residues in Red Cabbage Homogenate

### Final Report

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# EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUIT AND VEGETABLES. SCREENING METHODS 11

## BACKGROUND

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

Regulation (EU) No 625/2017<sup>2</sup> lays down the responsibilities and tasks, of European Union Reference Laboratories (EURLs) for Food, Feed and Animal Health. Among these tasks is the provision for regular inter-laboratory comparative testing or proficiency tests. This is the ninth time that the EURL for pesticides in fruit and vegetables at the University of Almería, Spain, has organised a proficiency test on qualitative screening methods for pesticides in fruits 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 mass spectrometry (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-FV21) 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 Phytopharmaceuticals – Pesticides Residues section of the Plants, Animals, Food and Feed Committee.

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<sup>1</sup>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.

<sup>2</sup>Regulation (EU) No 625/2017 of the European Parliament and of the Council on official controls and other official activities performed to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection products. Published in the OJ of the EU L95/1 of 07/04/2017



## 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.

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 or all ion fragmentation 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 No SANTE/11813/2017 "Guidance document on analytical quality control and method validation procedures for pesticide residues and analysis in food and feed".

This EUPT-FV-SM11 is aimed at all NRLs and all OfLs for fruits and vegetables in EU Member States. Laboratories outside this EURL/NRL/OfL-Network were also invited to participate.

The evaluation of this PT was based on qualitative information, although an estimated concentration was requested for those pesticides that were detected, only for informative purposes. 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 red cabbage. The red cabbages were cultivated in a farm in Almería, Spain.

The pesticides used to spike the red cabbage were decided upon by the Quality Control Group. No target pesticide list was provided to participants. The pesticides selected for treating the test item for this EUPT-FV-SM11 were mainly chosen taking into account the following considerations:

- That they were not included in the EU-Coordinated Multiannual Control Programme of the Union for 2019, 2020 and 2021.
- That they had particularly acute toxicity and/or had low ARfD values.

Table 2.1 shows the 16 used to spike present in the red cabbage 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 red cabbages were mixed in a constantly-spinning container until a homogeneous item was obtained. Finally, 200 g portions of the well-mixed homogenate were weighed out into screw-capped polyethylene plastic bottles, sealed and stored in a freezer at about -20 °C prior to distribution to participants.

Table 2.1 Pesticides used to spike in the sample.

Pesticides			
Bifenazate	Flufenacet	Metrafenone	Pyridalil
Etoxazole	Fluopicolide	Orthosulfamuron	Spinetoram
Fenpyrazamine	Isoprothiolane	Penthiopyrad	Tricyclazole
Flubendiamide	Isopyrazam	Propoxur	Valifenalate

### 2.2 Preparation of “blank” test item.

Red cabbages used to produce the blank test item were grown in the same field as the test item. The homogenate was prepared in the same way as the treated test item described previously.

### 2.3 Homogeneity and stability tests.

Homogeneity and stability tests associated with ‘quantitative’ PTs were conducted by the Organisers 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 0.01 mg/kg.

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<sup>3</sup>. The results of the homogeneity tests are given in Table 2.3a. The acceptance criteria for the test item to be sufficiently homogenous for the proficiency test were that:  $S_s^2 < c$ , where  $S_s$  is the between-bottle sampling standard deviation and  $c = F_1\sigma_{all}^2 + F_2s_{an}^2$ ;  $F_1$  and  $F_2$  being constant values of 1.88 and 1.01, respectively, from the ten samples taken, and  $\sigma_{all}^2 = 0.3 \times \text{FFP RSD}(25\%) \times \text{the analytical sampling mean for all the pesticides}$ . This was used to demonstrate that the between-bottle variance was not higher than the within-bottle variance.

<sup>3</sup> ISO 13528:2015, Statistical methods for use in proficiency testing by interlaboratory comparison, International Organization for Standardization

Table 2.3a shows the results of these tests, together with the average concentration values for each of the pesticides used to treat the sample.

Table 2.3a Homogeneity tests

Test item No.	129 A	129 B	015 A	015 B	162 A	162 B	036 A	036 B	003 A	003 B	058 A	058 B	066 A	066 B	081 A	081 B	096 A	096 B	118 A	118 B	R. Cc (mg/kg)	Ss² < C Pass/Fail	
Bifenazate	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.045	Pass
Etoxazole	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.068	Pass
Fenpyrazamine	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.073	Pass
Flubendiamide	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.093	Pass
Flufenacet	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.113	Pass
Fluopicolide	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.116	Pass
Isoprothiolane	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.064	Pass
Isopyrazam	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.051	Pass
Metrafenone	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.086	Pass
Orthosulfamuron	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.070	Pass
Penthiopyrad	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.077	Pass
Propoxur	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.099	Pass
Pyridalil	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.067	Pass
Spinetoram	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.091	Pass
Tricyclazole	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.107	Pass
Valifenalate	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.061	Pass

I: Identified

R. Cc: Robust mean Concentration

Nine bottles, again chosen randomly, were analysed by duplicate over a period of time to confirm the stability of the pesticides in the test item. Three when the test items were shipped, three after 48 hours reproducing the sample shipment conditions and then, other three bottles a few days after the deadline for submitting results to see if there was any degradation of any of the pesticides present in the test item. The results are given in table 2.3b.

Table 2.3b Stability tests performed.

Date	Shipment Day (25 <sup>th</sup> February 2019)			48h later Shipment Day (27 <sup>th</sup> February 2019)			Few days after deadline (13 <sup>th</sup> March 2019)		
	165	093	161	149	099	147	136	070	068
Bifenazate	I	I	I	I	I	I	I	I	I
Etoxazole	I	I	I	I	I	I	I	I	I
Fenpyrazamine	I	I	I	I	I	I	I	I	I
Flubendiamide	I	I	I	I	I	I	I	I	I
Flufenacet	I	I	I	I	I	I	I	I	I
Fluopicolide	I	I	I	I	I	I	I	I	I
Isoprothiolane	I	I	I	I	I	I	I	I	I
Isopyrazam	I	I	I	I	I	I	I	I	I
Metrafenone	I	I	I	I	I	I	I	I	I
Orthosulfamuron	I	I	I	I	I	I	I	I	I
Penthiopyrad	I	I	I	I	I	I	I	I	I
Propoxur	I	I	I	I	I	I	I	I	I
Pyridalil	I	I	I	I	I	I	I	I	I
Spinetoram	I	I	I	I	I	I	I	I	I
Tricyclazole	I	I	I	I	I	I	I	I	I
Valifenalate	I	I	I	I	I	I	I	I	I

I: Identified

NI: Not identified

## **2.4 Distribution of test items and protocol to participants**

Approximately 200 g of treated red cabbage homogenate together with another 200 g of 'blank' red cabbage homogenate were shipped to participants on 25<sup>th</sup> February 2019. 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 or all ion fragmentation with HRAMS. However, extended targeted methods using LC-MS/MS (triple quadrupole, Q-trap, Q-ToF) or GC-MS/MS (triple quadrupole, ion trap, Q-trap, Q-ToF) could also be used.

Before shipment, the laboratories received full instructions for the receipt and analysis of the test item, and 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-SM11 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 the Sample Receipt and the results forms. 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 evaluation of this PT was based on qualitative information, although an estimated concentration was requested for those pesticides that were detected, only for informative purposes.

The robust mean of the estimated concentrations reported was calculated using robust statistics as described in ISO 13528:2015, considering the results reported by EU and EFTA countries laboratories only.

##### 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 several 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.*

##### 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.

## 4. RESULTS

### 4.1 Summary of reported results

Sixty-nine laboratories agreed to participate in this eleventh proficiency test on screening methods. Sixty-seven laboratories submitted results on time (two laboratories cancelled their participation). 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 methods used are provided in Appendix 3 (available on the EUPT-FV-SM11 webpage, not in the printed version). The laboratories that agreed to participate are listed in Annex 1.

Fifteen laboratories reported bifenazate-diazene, which appeared in the test item as a result of the treatment with bifenazate. Bifenazate and bifenazate-diazene interconvert depending on the environmental conditions. Bifenazate-diazene is the reduced form, present at high pH. At low pH values, bifenazate-diazene is converted to bifenazate. However, at medium pH conditions, both forms are present. This interconversion takes place also in the analytical standard, which restrains the individual quantification of the compounds, unless the total conversion is achieved. For this reason, the Scientific Committee decided not to use the results of bifenazate-diazene for the evaluation of the participant laboratories. However, information of bifenazate-diazene will be shown for informative purposes.

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

Table 4.1a Summary of Reported Results.

Pesticide	Reported		Not Reported	
	No. of laboratories	% of Laboratories <sup>#</sup>	No. of laboratories	% of laboratories *
Bifenazate	53	79	14	21
Bifenazate-diazene*	15	22	52	78
Etoxazole	62	93	5	7
Fenpyrazamine	52	78	15	22
Flubendiamide	52	78	15	22
Flufenacet	57	85	10	15
Fluopicolide	60	90	7	10
Isoprothiolane	58	87	9	13
Isopyrazam	51	76	16	24
Metrafenone	59	88	8	12
Orthosulfamuron	26	39	41	61
Penthiopyrad	51	76	16	24
Propoxur	62	93	5	7
Pyridalil	50	75	17	25
Spinetoram	54	81	13	19
Tricyclazole	57	85	10	15
Valifenalate	42	63	25	37

<sup>#</sup>The % of laboratories is calculated based on the total number of laboratories submitting results (67 laboratories).

\* This pesticide appears only with informative purposes, it is not included in the evaluation of the laboratories.

In this EUPT-FV-SM11, the estimated concentration was requested for those pesticides that were detected, only for informative purposes. However, not all the laboratories reported concentration results (Appendix 1 – Estimated Concentrations Reported). Table 4.1b shows the robust mean of the estimated concentrations reported by EU/EFTA laboratories, the average concentration from the homogeneity test and the dispersion of the concentration results reported.

Table 4.1b Robust mean values and CVs (%) for all pesticides evaluated.

Pesticide	Robust mean of estimated concentrations reported (mg/kg)	Average concentration Homogeneity test (mg/kg)	CV (%)
Bifenazate	0.037	0.045	44.9
Etoxazole	0.060	0.068	20.7
Fenpyrazamine	0.052	0.073	17.2
Flubendiamide	0.065	0.093	22.8
Flufenacet	0.085	0.113	21.1
Fluopicolide	0.084	0.116	19.5
Isoprothiolane	0.067	0.064	18.5
Isopyrazam	0.057	0.051	22.0
Metrafenone	0.064	0.086	20.8
Orthosulfamuron	0.119	0.070	79.8
Penthiopyrad	0.054	0.077	17.9
Propoxur	0.081	0.099	18.1
Pyridalil	0.041	0.067	12.9
Spinetoram	0.052	0.091	35.1
Tricyclazole	0.082	0.107	15.2
Valifenalate	0.043	0.061	20.2

No other compounds were identified and quantified by the organizer at concentrations above 0.010 mg/kg.

#### 4.1.1 Other Reported Compounds

Some laboratories reported additional compounds to those present in the test item. Some of them are reported below 0.01 mg/kg or not quantified, the reported compounds at or above 0.01 mg/kg are marked in grey.

Table 4.1.1. Other reported pesticides bellow 0.01 mg/kg or not quantified.

Laboratory Code	Other Reported Compounds	Concentration Reported (mg/kg)
Lab007	Isoxaflutol	0.06
Lab015	Buprofezin	
	Diuron	0.014
	Permethrin	0.008
Lab017	Flupyradifurone	
	Tembotrione	
Lab022	Tetraconazol	
Lab031	Spirotetramat	
	Tetraconazol	
	Thiametoxam	
Lab040	Goitrine	
	Paraquat	
Lab045	Isocarbophos	0.06
Lab046	Triticonazole	0.06
Lab050	Propiconazole (sum of isomers)	0.028
Lab057	Mapenterol	
Lab059	Aldimorph	0.05
	Cymiazol	0.01
	Fenoxycarb	0.005

Laboratory Code	Other Reported Compounds	Concentration Reported (mg/kg)
	Metconazol	0.01
	Spiroxamin	0.05-0.1
	Sulfamethoxazol	0.01
	Trifluralin	0.005
Lab062	Spinosad	0.011
Lab069	Phenylphenol	
	Sulphur	

None of the other reported pesticides was reported by three or more laboratories.

#### 4.1.2 Non-Reported Pesticides

Table 4.1a shows for each specific pesticide, the number and percentage of laboratories that did not report them. The individual results for each laboratory are given in Appendix 1. Graphical representations can be seen in Appendix 2

#### 4.2 Concentration levels.

Sixteen pesticides were used to spike the red cabbage test item at different concentration levels, in a range of 0.01-0.1 mg/kg.

#### 4.3 Assessment of laboratory performance.

Laboratory performance was assessed with the number of results reported by each laboratory. Table 4.3.a classifies the laboratories according to the number of present pesticides reported.

Table 4.3.a 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
Lab002	16	100	0
Lab004	16	100	0
Lab013	16	100	0
Lab016	16	100	0
Lab019	16	100	0
Lab020	16	100	0
Lab025	16	100	0
Lab030	16	100	0
Lab032	16	100	0
Lab034	16	100	0
Lab039	16	100	0
Lab043	16	100	0
Lab052	16	100	0
Lab056	16	100	0
Lab065	16	100	0
Lab006	16	100	0
Lab009	16	100	0
Lab014	16	100	0
Lab024	16	100	0
Lab047	16	100	0

Laboratory Code	No of Reported Pesticides	% of Reported Pesticides	Other Reported Pesticides Not Confirmed by the Organiser
Lab007	16	100	1
Lab017	16	100	2
Lab001	15	94	0
Lab005	15	94	0
Lab018	15	94	0
Lab023	15	94	0
Lab035	15	94	0
Lab037	15	94	0
Lab060	15	94	0
Lab066	15	94	0
Lab067	15	94	0
Lab010	15	94	0
Lab011	15	94	0
Lab026	15	94	0
Lab049	15	94	0
Lab053	15	94	0
Lab063	15	94	0
Lab057	15	94	1
Lab015	15	94	4
Lab021	14	88	0
Lab054	14	88	0
Lab058	14	88	0
Lab069	14	88	2
Lab029	13	81	0
Lab061	13	81	0
Lab068	13	81	0
Lab045	13	81	1
Lab031	12	75	3
Lab033	11	69	0
Lab050	11	69	1
Lab062	11	69	1
Lab027	10	63	0
Lab036	10	63	0
Lab051	10	63	0
Lab046	10	63	1
Lab041	7	44	0
Lab055	7	44	0
Lab028	6	38	0
Lab042	6	38	0
Lab059	6	38	7
Lab003	3	19	0
Lab044	3	19	0
Lab040	3	19	2
Lab008	2	13	0
Lab022	2	13	1
Lab048	1	6	0

Laboratory Code	No of Reported Pesticides	% of Reported Pesticides	Other Reported Pesticides Not Confirmed by the Organiser
Lab038	0	0	0

The extraction methods used by the laboratories, the chromatographic techniques, detectors, instrumentation, etc... are detailed in Appendix 3 (available only on the EUPT-FV-SM11 webpage, not in the printed version).

Table 4.3.b shows the number and percentage of the pesticides present in the sample which were reported by each laboratory.

Table 4.3.b Number and Percentage of Present Pesticides Reported by Laboratory

Laboratory Code	Number of Present Pesticides Reported (16 Evaluated Pesticides)	% of Present Pesticides Reported (16 Evaluated Pesticides)
Lab001	15	94
Lab002	16	100
Lab003	3	19
Lab004	16	100
Lab005	15	94
Lab006	16	100
Lab007	16	100
Lab008	2	13
Lab009	16	100
Lab010	15	94
Lab011	15	94
Lab013	16	100
Lab014	16	100
Lab015	15	94
Lab016	16	100
Lab017	16	100
Lab018	15	94
Lab019	16	100
Lab020	16	100
Lab021	14	88
Lab022	2	13
Lab023	15	94
Lab024	16	100
Lab025	16	100
Lab026	15	94
Lab027	10	63
Lab028	6	38
Lab029	13	81
Lab030	16	100
Lab031	12	75
Lab032	16	100
Lab033	11	69
Lab034	16	100

Laboratory Code	Number of Present Pesticides Reported (16 Evaluated Pesticides)	% of Present Pesticides Reported (16 Evaluated Pesticides)
Lab035	15	94
Lab036	10	63
Lab037	15	94
Lab038	0	0
Lab039	16	100
Lab040	3	19
Lab041	7	44
Lab042	6	38
Lab043	16	100
Lab044	3	19
Lab045	13	81
Lab046	10	63
Lab047	16	100
Lab048	1	6
Lab049	15	94
Lab050	11	69
Lab051	10	63
Lab052	16	100
Lab053	15	94
Lab054	14	88
Lab055	7	44
Lab056	16	100
Lab057	15	94
Lab058	14	88
Lab059	6	38
Lab060	15	94
Lab061	13	81
Lab062	11	69
Lab063	15	94
Lab065	16	100
Lab066	15	94
Lab067	15	94
Lab068	13	81
Lab069	14	88

Table 4.3.c is a summary of the chromatographic techniques used for each pesticide. Graphical representation is shown in Appendix 2.

Table 4.3.c Chromatographic techniques used to determine each pesticide in the test item

Pesticide	Total Number of Laboratories Reporting Data	*Total Number of Reported Detections	GC	Full Scan GC	LC	Full Scan LC
Bifenazate	53	58	13	13	21	11
Bifenazate-diazene*	15	15			8	7
Etoazole	62	69	14	7	30	18
Fenpyrazamine	52	58	2	3	33	20

Pesticide	Total Number of Laboratories Reporting Data	*Total Number of Reported Detections	GC	Full Scan GC	LC	Full Scan LC
Flubendiamide	52	56	1		36	19
Flufenacet	57	62	10	7	28	17
Flupicolide	60	64	13	5	29	17
Isoprothiolane	58	65	16	4	27	18
Isopyrazam	51	55	6	3	27	19
Metrafenone	59	65	21	6	21	17
Orthosulfamuron	26	28			14	14
Penthiopyrad	51	56	9	4	23	20
Propoxur	62	68	10	4	35	19
Pyridalil	50	55	10	8	24	13
Spinetoram	54	71			46	25
Tricyclazole	57	62	4	5	35	18
Vallifenalate	42	44	2	1	22	19

\*Note: the number of reported detections for each of the pesticides could be different to the number of laboratories reporting the pesticide because a particular laboratory might have analysed one pesticide with more than one technique.

\* This pesticide appears only with informative purposes; it is not used for the evaluation of the laboratories.

## 5. CONCLUSIONS

Sixty-nine laboratories agreed to participate in this proficiency test on screening methods. Sixty-seven laboratories submitted results on time (two laboratories cancelled their participation). Eighteen EU Member States, 2 EFTA countries (Norway and Switzerland) and four non-EU/EFTA countries (China, Costa Rica, Kenya, 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. The total amount of detections were 951, 201 were made by GC and 750 by LC; 361 were made using full-scan, meaning 38% of detections (70 by GC-full scan techniques and 291 by LC-full scan techniques); 33% of the laboratories reported their results using HRMS (high resolution accurate mass spectrometry); 809 of the results were reported indicating a concentration value (85% of the total results).

Twenty-two of the 67 laboratories were able to detect all 16 present pesticides in the test item. Twelve laboratories detected less than 50 % of the pesticides present. Seventy-two percent of the laboratories (48 laboratories) that reported results were able to detect more than 70 % of the evaluated pesticides.

Fifteen laboratories reported bifenazate-diazene, which appeared in the test item as a result of the treatment with bifenazate. Bifenazate and bifenazate-diazene interconvert depending on the environmental conditions. Bifenazate-diazene is the reduced form, present at high pH. At low pH values, bifenazate-diazene is converted to bifenazate. However, at medium pH conditions, both forms are present. This interconversion takes place also in the analytical standard, which restrains the individual quantification of the compounds, unless the total conversion is achieved. For this reason, the Scientific Committee decided not to use the results of bifenazate-diazene for the evaluation of the participant laboratories. However, information of bifenazate-diazene has been shown only for informative purposes.

Thirteen participants reported 25 different compounds which were not present in the 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 increase 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 Document SANTE/11813/2017

Next year, once again, 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.

From now on, no blank test item will be sent to the participants.

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

The Organiser is grateful to the European Commission for funding this 11<sup>th</sup> 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

Table AP1a. Reported pesticides

Laboratory Code Total No of Reporting Laboratories = 67	Evaluated Pesticides (16)															R: Reported Pesticide		
	Bifentazate	Etoazazole	Fenpyrazamine	Flubendiamide	Flufenacet	Flupicolide	Isoprothiolane	Isopyrazam	Metrafenone	Orthosulfamuron	Penthiopyrad	Propoxur	Pyridalil	Spinetoram	Tricyclazole	Vailfenalate	Reported Pesticides by Laboratory	% Reported Pesticides by Laboratory
Lab001	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	15	94
Lab002	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	100
Lab003						R			R		R						3	19
Lab004	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	100
Lab005	R	R	R	R	R	R	R	R		R	R	R	R	R	R	R	15	94
Lab006	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	100
Lab007	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	100
Lab008	R								R								2	13
Lab009	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	100
Lab010	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	15	94
Lab011	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	15	94
Lab013	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	100
Lab014	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	100
Lab015	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	15	94
Lab016	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	100
Lab017	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	100
Lab018	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	15	94
Lab019	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	100
Lab020	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	100
Lab021		R	R	R	R	R	R	R	R		R	R	R	R	R	R	14	88
Lab022											R		R				2	13
Lab023	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	15	94
Lab024	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	100
Lab025	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	100
Lab026	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	15	94
Lab027	R	R	R		R	R	R	R	R		R		R				10	63
Lab028		R		R		R	R		R					R			6	38
Lab029	R	R	R	R	R	R	R		R		R	R	R	R	R		13	81
Lab030	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	100
Lab031	R	R			R	R	R	R	R		R	R	R		R	R	12	75
Lab032	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	100
Lab033	R	R		R	R	R	R		R		R	R		R	R		11	69
Lab034	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	100
Lab035	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	15	94
Lab036	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	10	63
Lab037	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	15	94
Lab038																	0	0
Lab039	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	100
Lab040		R			R						R						3	19
Lab041		R	R			R	R		R		R			R			7	44
Lab042	R	R			R						R		R	R			6	38
Lab043	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	100
Lab044		R					R				R						3	19
Lab045		R	R	R	R	R	R	R	R		R	R	R	R	R		13	81
Lab046		R	R	R	R	R	R		R		R	R		R			10	63
Lab047	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	100
Lab048						R											1	6
Lab049	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	15	94
Lab050	R	R		R	R	R	R		R		R		R	R			11	69
Lab051		R	R		R	R	R	R	R	R	R			R			10	63
Lab052	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	100
Lab053	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	15	94
Lab054	R	R	R	R	R	R	R	R	R		R	R	R	R	R		14	88
Lab055	R	R		R	R						R		R	R			7	44
Lab056	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	100
Lab057	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	15	94
Lab058	R	R	R	R	R	R	R	R	R		R	R	R	R	R		14	88
Lab059		R		R		R	R				R		R				6	38
Lab060	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	15	94

**APPENDIX 1**

Laboratory Code Total No. of Reporting Laboratories = 67	Evaluated Pesticides (16)															R: Reported Pesticide		
	Bifenazate	Etoazale	Fenpyrazamine	Flubendiamide	Flufenacet	Fluopicolide	Isoprothiolane	Isopyrazam	Metrafenone	Orthosulfamuron	Penthiopyrad	Propoxur	Pyridalil	Spinetoram	Tricyclazole	Vallifenalate	Reported Pesticides by Laboratory	% Reported Pesticides by Laboratory
Lab061	R	R	R	R	R	R	R	R	R			R	R	R	R		<b>13</b>	<b>81</b>
Lab062	R	R	R	R		R		R	R			R	R	R	R		<b>11</b>	<b>69</b>
Lab063	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	<b>15</b>	<b>94</b>
Lab065	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	<b>16</b>	<b>100</b>
Lab066		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	<b>15</b>	<b>94</b>
Lab067	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	<b>15</b>	<b>94</b>
Lab068	R	R	R		R	R	R	R	R		R	R	R	R	R		<b>13</b>	<b>81</b>
Lab069	R	R	R	R	R	R	R	R	R		R	R		R	R	R	<b>14</b>	<b>88</b>
<b>Reported Pesticides</b>	<b>53</b>	<b>62</b>	<b>52</b>	<b>52</b>	<b>57</b>	<b>60</b>	<b>58</b>	<b>51</b>	<b>59</b>	<b>26</b>	<b>51</b>	<b>62</b>	<b>50</b>	<b>54</b>	<b>57</b>	<b>42</b>		
<b>% of Reported Pesticides</b>	<b>79</b>	<b>93</b>	<b>78</b>	<b>78</b>	<b>85</b>	<b>90</b>	<b>87</b>	<b>76</b>	<b>88</b>	<b>39</b>	<b>76</b>	<b>93</b>	<b>75</b>	<b>81</b>	<b>85</b>	<b>63</b>		

Table AP1b. Estimated Concentrations Reported on a voluntary basis (only informative purposes)  
 Not all the laboratories reporting results have reported estimated concentration values  
 Results reported without concentration values are expressed as R.

Evaluated Pesticides (16)																
LABORATORY CODE Total No. of Reporting Laboratories = 67	Bifenazate	Etoxazole	Fenpyrazamine	Flubendiamide	Flufenacet	Fluopicolide	Isoprothiolane	Isopyrazam	Metrafenone	Orthosulfamuron	Penthiopyrad	Propoxur	Pyridalil	Spinetoram	Tricyclazole	Valifenalate
Robust mean of estimated concentrations reported (mg/kg)	0.037	0.060	0.052	0.065	0.085	0.084	0.067	0.057	0.064	0.119	0.054	0.081	0.041	0.052	0.082	0.043
Average concentration Homogeneity test (mg/kg)	0.045	0.068	0.073	0.093	0.113	0.116	0.064	0.051	0.086	0.070	0.077	0.099	0.067	0.091	0.107	0.061
CV (%)	44.9	20.7	17.2	22.8	21.1	19.5	18.5	22.0	20.8	79.8	17.9	18.1	12.9	35.1	15.2	20.2
Lab001	0.046	0.055	0.047	0.054	0.078	0.076	0.06	0.052	0.062		0.055	0.072	0.038	0.041	0.076	0.022
Lab002	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R
Lab003						0.08			0.07			0.09				
Lab004	0.03	0.0419	0.0528	0.0533	0.0836	0.0698	0.0656	0.048	0.0436	0.0704	0.0466	0.0955	0.0371	0.0172	0.0962	0.0435
Lab005	0.019	0.049	0.038	0.045	0.066	0.059	0.052	0.04	0.049		0.04	0.074	0.042	0.034	0.086	0.033
Lab006	R	0.05845	0.0543	0.069	0.0798	0.077	0.0632	0.0552	0.0695	0.1	0.0506	0.0702	0.044	R	0.0778	0.0384
Lab007	0.03	0.06	0.05	0.07	0.12	0.11	0.09	0.06	0.1	0.15	0.07	0.2	0.04	0.06	0.1	0.05
Lab008	0.026								0.063							
Lab009	0.0374	0.04	0.0434	0.0488	0.0819	0.0698	0.0542	0.0491	0.054	0.0862	0.0507	0.0753	0.0398	0.0079	0.0838	0.0313
Lab010	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R
Lab011	0.04	0.07	0.06	0.075	0.09	0.09	R	0.07	0.075		0.06	R	R	R	R	R
Lab013	0.059	0.057	0.05	0.132	0.077	0.099	0.084	0.073	0.069	0.063	0.07	0.079	0.046	0.053	0.076	0.049
Lab014	0.09	0.07	0.07	0.07	0.1	0.1	0.06	0.07	0.08	R	0.06	0.09	0.04	0.07	0.1	0.05
Lab015	0.017	0.085	0.054	0.081	0.097	R	R	R	R		0.045	0.079	R	0.063	0.085	R
Lab016	0.042	0.087	0.078	0.012	0.1	0.13	0.1	0.076	0.093	0.08	0.08	0.14	R	0.093	0.12	0.058
Lab017	R	0.051	R	R	0.952	R	0.05	R	0.054	R	R	0.07	0.042	R	0.074	R
Lab018	0.027	0.054	0.057	0.09	0.082	0.081	0.067	0.057	0.069		0.052	0.085	0.038	0.045	0.083	0.048
Lab019	0.0226	0.0725	0.0485	0.0524	0.1368	0.1057	0.0805	0.0807	0.0796	0.774	0.0641	0.0996	0.042	0.0684	0.0902	0.0339
Lab020	0.025	0.0661	0.0494	0.0526	0.1328	0.0902	0.0758	0.0681	0.0639	0.1487	0.0603	0.0951	0.0421	0.046	0.112	0.0409
Lab021		R		R		R	R	R	R			R		R	R	R
Lab022												0.073		0.048		
Lab023	0.033	0.058	0.053	0.054	0.069	0.077	0.066	0.036	0.07		0.054	0.086	0.037	0.051	0.036	0.04
Lab024	R	0.067	0.065	0.071	0.09	0.079	0.079	0.068	0.08	0.05	0.061	0.09	0.043	0.07	0.097	0.05
Lab025	R	0.07	0.06	0.07	0.07	0.08	0.07	0.06	0.07	R	0.06	0.08	0.04	0.06	0.08	R
Lab026	0.022	0.0673	0.048		0.07	0.073	0.068	0.054	0.0593	0.55	0.054	0.091	0.04	0.0637	0.0893	0.04
Lab027	0.03	0.068	0.049		0.094	0.073	0.07	0.023	0.06		0.046		0.04			

APPENDIX 2. Graphical Representations

Evaluated Pesticides (16)																
LABORATORY CODE Total No of Reporting Laboratories = 67	Bifentazate	Etoazazole	Fenpyrazamine	Flubendiamide	Flufenacet	Fluopicolide	Isoprothiolane	Isopyrazam	Metrafenone	Orthosulfamuron	Penthiopyrad	Propoxur	Pyridalil	Spinetoram	Tricyclazole	Valifenalate
Robust mean of estimated concentrations reported (mg/kg)	0.037	0.060	0.052	0.065	0.085	0.084	0.067	0.057	0.064	0.119	0.054	0.081	0.041	0.052	0.082	0.043
Average concentration Homogeneity test (mg/kg)	0.045	0.068	0.073	0.093	0.113	0.116	0.064	0.051	0.086	0.070	0.077	0.099	0.067	0.091	0.107	0.061
CV (%)	44.9	20.7	17.2	22.8	21.1	19.5	18.5	22.0	20.8	79.8	17.9	18.1	12.9	35.1	15.2	20.2
Lab028		0.0575		0.0695		0.0845	0.0625		0.0549						0.0695	
Lab029	0.038	0.13	0.025	0.102	0.215	0.166	0.12		0.05		0.04	0.21	0.028	0.025	0.162	
Lab030	0.096	0.058	0.057	0.028	R	0.072	0.057	0.051	0.06	R	0.046	0.081	0.042	R	0.078	R
Lab031	R	R			R	R	R	R	R		R	R	R	R	R	R
Lab032	0.05	0.075	0.05	0.05	0.1	0.1	0.05	0.05	0.075	0.05	0.05	0.075	0.05	0.05	0.075	0.05
Lab033	0.07	0.05		0.07	0.1	0.07	0.07		0.07		0.04	0.07		0.007	0.07	
Lab034	0.057	0.068	0.06	0.11	0.14	0.12	0.084	0.08	0.061	0.38	0.056	0.11	0.04	0.065	0.1	0.049
Lab035	0.023	0.053	0.044	0.065	0.084	0.085	0.078	0.054	0.041		0.054	0.075	0.036	0.053	0.081	0.033
Lab036	R	R				0.07	0.052	0.056	R			0.057	R		R	
Lab037	0.026	0.051	0.053	0.061	0.085	0.079	0.062	0.059	0.061		0.053	0.077	0.032	0.052	0.081	R
Lab039	R	0.07	0.07	0.1	R	0.12	0.09	0.07	0.09	0.27	0.08	0.11	0.02	0.05	0.09	0.03
Lab040		R			R							0.244				
Lab041		0.05	0.06			0.1	0.04		0.06			0.08			0.08	
Lab042	0.0177	0.0146			0.0328							0.0251		0.0109	0.0521	
Lab043	0.033	0.057	0.041	0.063	0.086	0.075	0.064	0.054	0.063	0.059	0.049	0.067	0.04	0.055	0.07	0.046
Lab044		0.05					0.07					0.09				
Lab045		0.057	0.038	0.08	0.077	0.053	0.044	0.055	0.05		0.05	0.07	0.034	0.062	0.074	
Lab046		0.15	0.04	0.05	0.06	0.04	0.04		0.04		0.08	0.06		0.04		
Lab047	0.0358	0.0531	0.0415	0.0763	0.053	0.0475	0.0641	0.0439	0.0453	0.0503	0.0413	0.112	0.0748	0.0573	0.0731	0.047
Lab048						0.083										
Lab049	0.059	0.062	0.047	0.068	0.075	0.077	0.064	0.05	0.062		0.045	0.074	0.042	0.043	0.057	0.05
Lab050	0.045	0.05		0.057	0.069	0.07	0.07		0.052		0.059	0.044		0.053	0.087	
Lab051		0.049	0.05		0.081	0.087	0.087	0.055	0.062	0.055		0.072			0.092	
Lab052	0.025	0.053	0.05	0.11	0.069	0.071	0.07	0.046	0.069	0.052	0.085	0.055	0.068	0.033	0.066	0.051
Lab053	R	0.08	R		0.08	0.1	0.08	R	0.09		R	R	R	R	R	0.04
Lab054	0.023	0.073	0.054	0.074	0.07	0.085	0.065	0.064	0.069		0.05	0.089	0.043	0.053	0.087	
Lab055	0.04	0.05		0.06	0.08							0.87		0.07	0.09	
Lab056	0.036	0.072	0.06	0.076	0.108	0.087	0.073	0.065	0.076	R	0.061	0.067	0.052	0.134	0.082	0.044
Lab057	R	0.054	R	0.058	0.085	0.092	0.068	R	0.06		R	0.077	0.043	R	0.082	R
Lab058	0.0720	0.0626	0.0582	0.0608	0.0910	0.0914	0.0682	0.0612	0.0664		0.0566	0.0832	0.0440	0.07	0.0846	

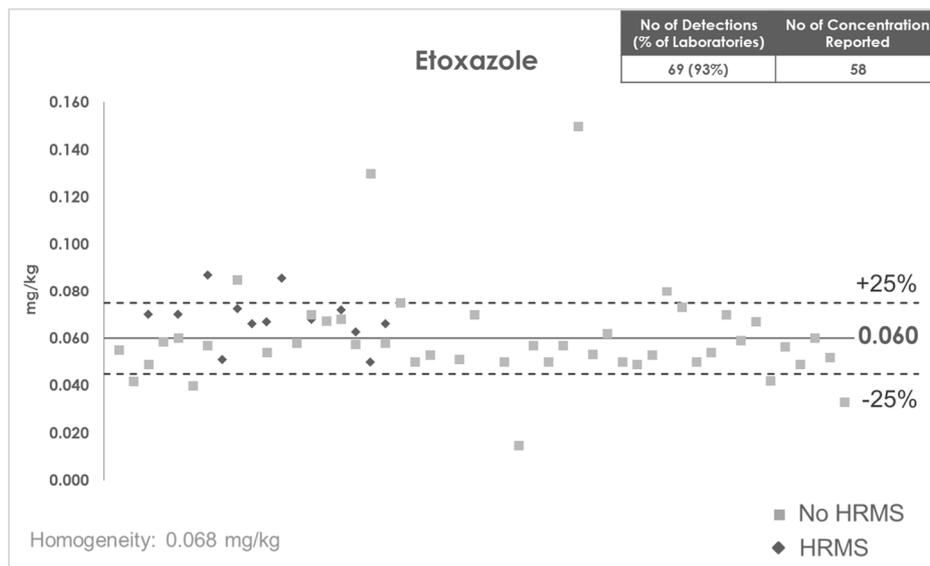
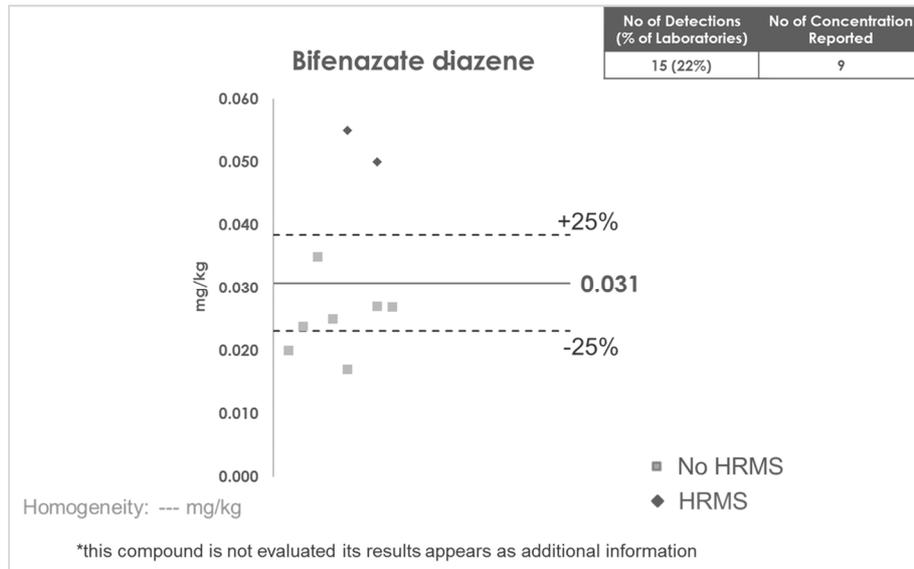
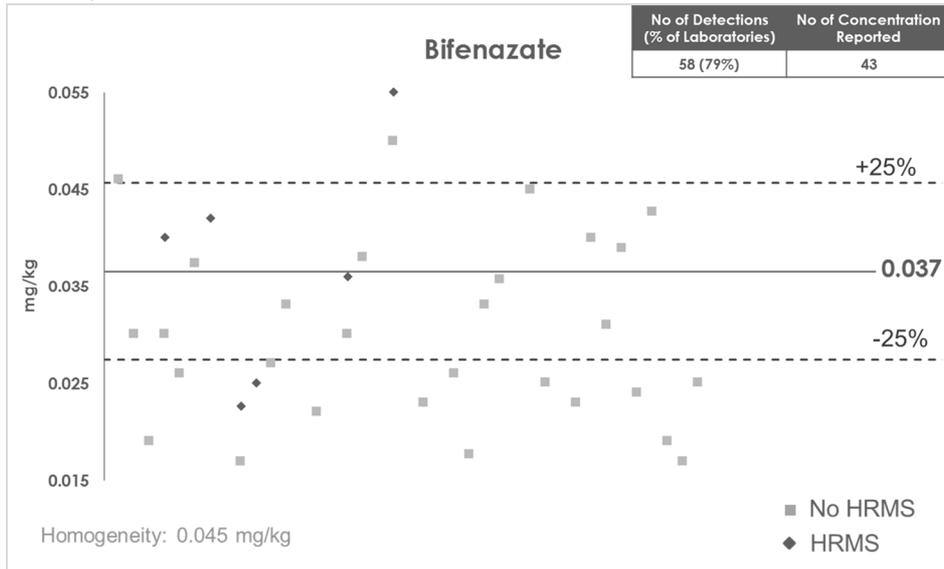
APPENDIX 2. Graphical Representations

Evaluated Pesticides (16)																
LABORATORY CODE Total No of Reporting Laboratories = 67	Bifenazate	Etoxazole	Fenpyrazamine	Flubendiamide	Flufenacet	Fluopicolide	Isoprotiolane	Isopyrazam	Metrafenone	Orthosulfamuron	Penthiopyrad	Propoxur	Pyridalil	Spinetoram	Tricyclazole	Valifenalate
Robust mean of estimated concentrations reported (mg/kg)	0.037	0.060	0.052	0.065	0.085	0.084	0.067	0.057	0.064	0.119	0.054	0.081	0.041	0.052	0.082	0.043
Average concentration Homogeneity test (mg/kg)	0.045	0.068	0.073	0.093	0.113	0.116	0.064	0.051	0.086	0.070	0.077	0.099	0.067	0.091	0.107	0.061
CV (%)	44.9	20.7	17.2	22.8	21.1	19.5	18.5	22.0	20.8	79.8	17.9	18.1	12.9	35.1	15.2	20.2
Lab059		0.07		0.07		0.09	0.08					0.08		0.06		
Lab060	0.031	0.059	0.053	0.071	0.079	0.079	0.063	0.085	0.066		0.051	0.073	0.051	0.045	0.069	0.017
Lab061	0.039	0.067	0.056	0.064	0.067	0.124	0.07	0.069	0.067			0.09	0.045	0.068	0.083	
Lab062	0.024	0.042	0.042	0.051		0.088		0.056	0.044			0.08	0.044	0.04	0.082	
Lab063	0.0427	0.0563	0.0562	0.0517	0.0713	0.0833	0.0653	0.0518	0.0646		0.0530	0.0687	0.0390	0.0496	0.0852	0.0486
Lab065	0.019	0.049	0.061	0.061	0.081	0.073	0.061	0.048	0.054	0.085	0.051	0.079	0.048	0.024	0.066	0.047
Lab066		0.06	0.06	0.05	0.1	0.1	0.07	0.05	0.08	R	0.06	0.08	0.04	0.03	0.08	R
Lab067	0.017	0.052	0.043	0.055	0.09	0.074	0.06	0.053	0.058		0.052	0.073	0.035	0.05	0.074	0.04
Lab068	0.025	0.033	0.043		0.038	0.056	0.037	0.035	0.038		0.023	0.071	0.013	0.044	0.036	
Lab069	0.055	0.066	0.06	0.071	0.096	0.087	0.077	0.038	0.062		0.05	0.081		0.055	0.094	0.053



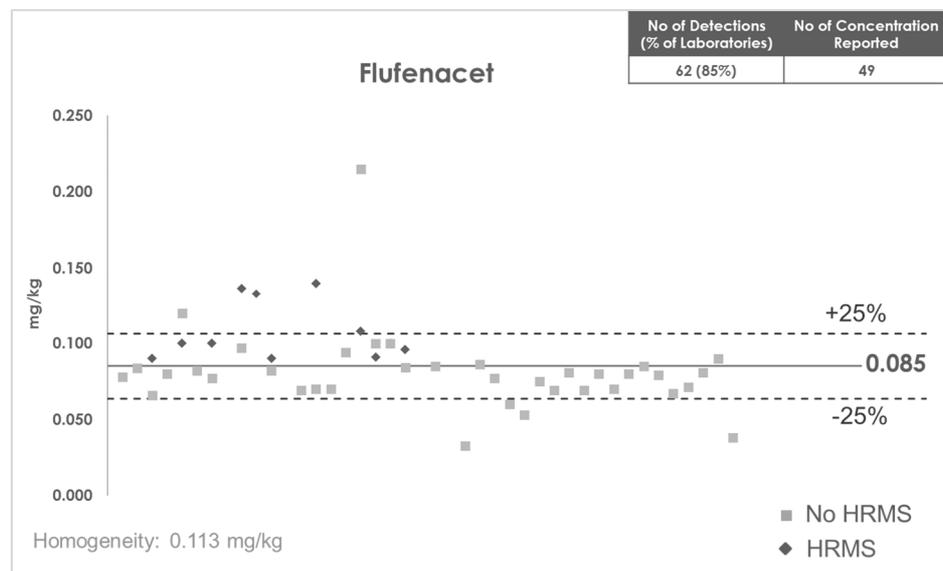
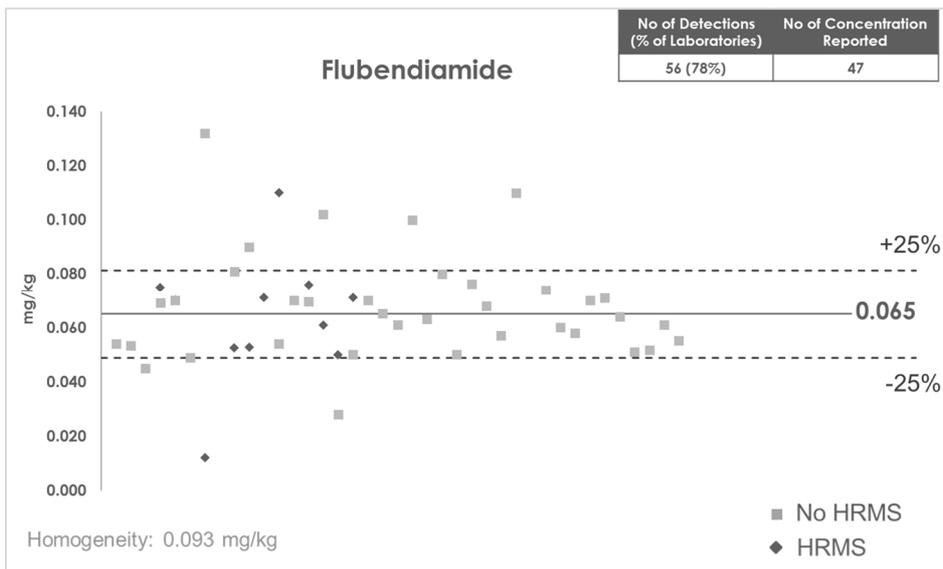
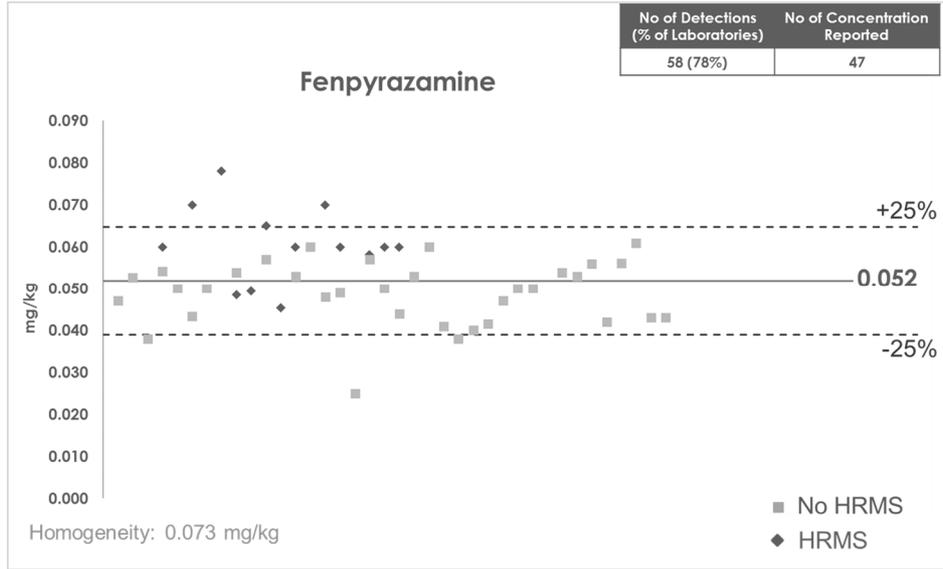
APPENDIX 2. Graphical Representations

The bold line represents the robust mean

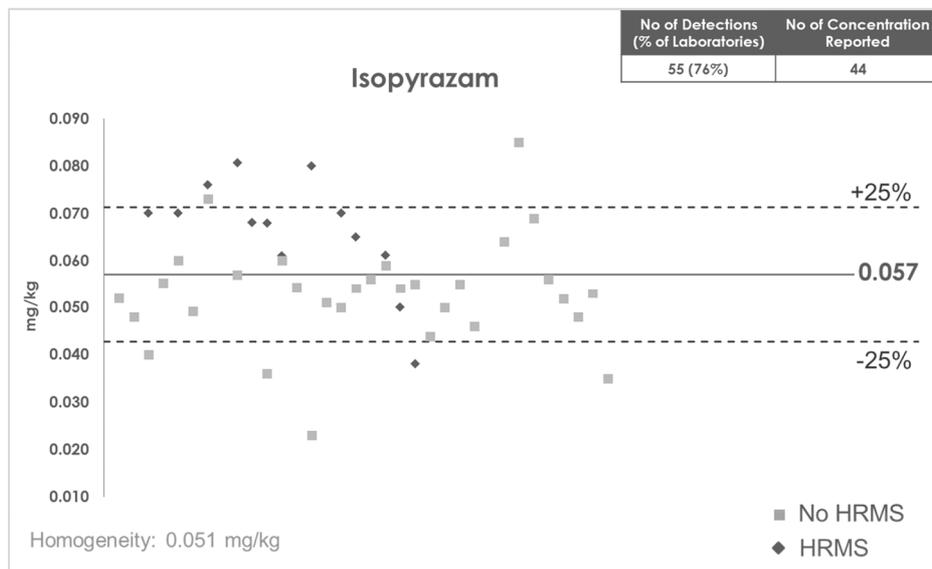
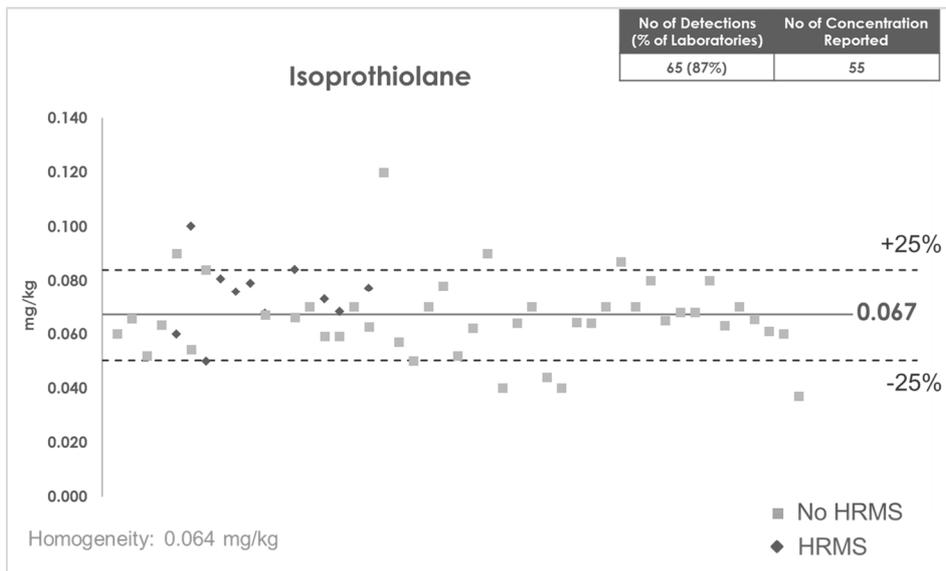
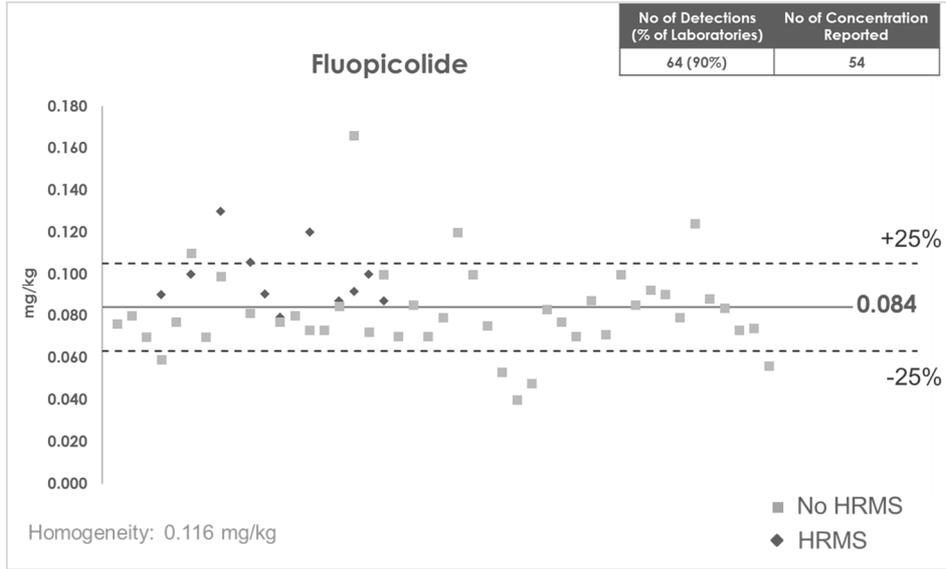


## APPENDIX 2. Graphical Representations

The bold line represents the robust mean

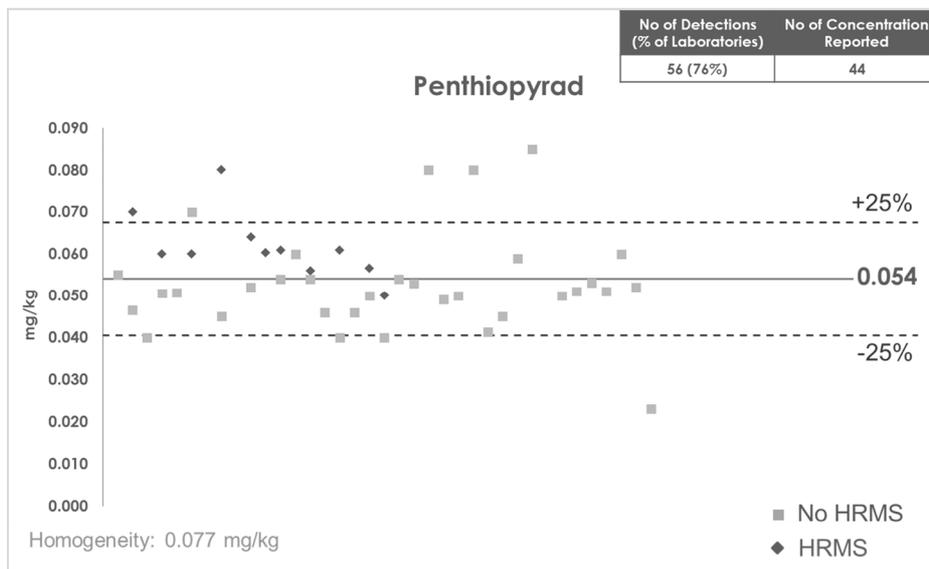
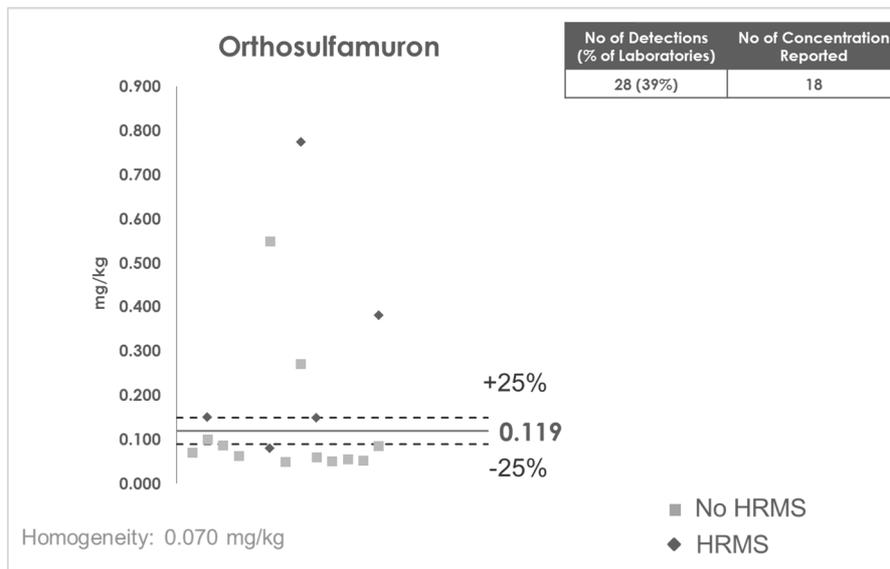
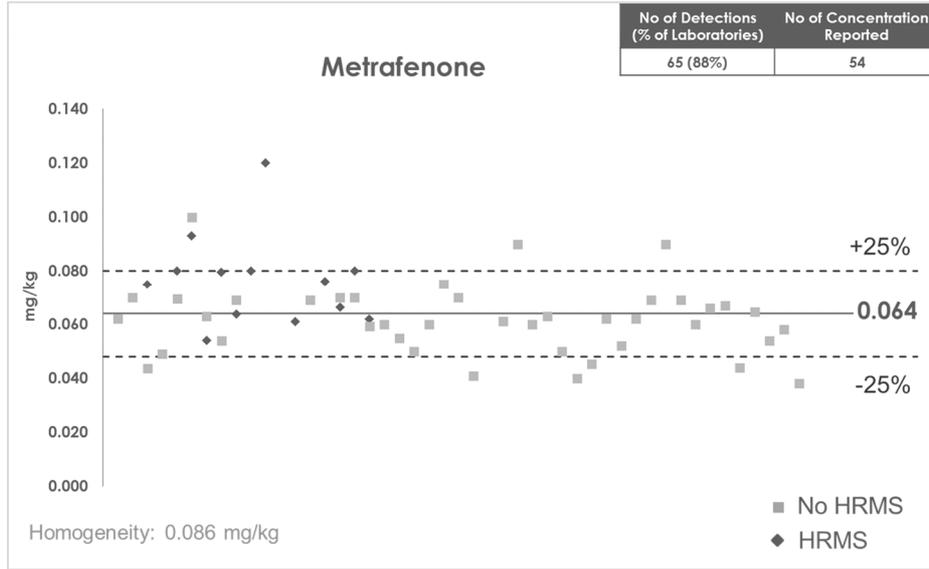


The bold line represents the robust mean

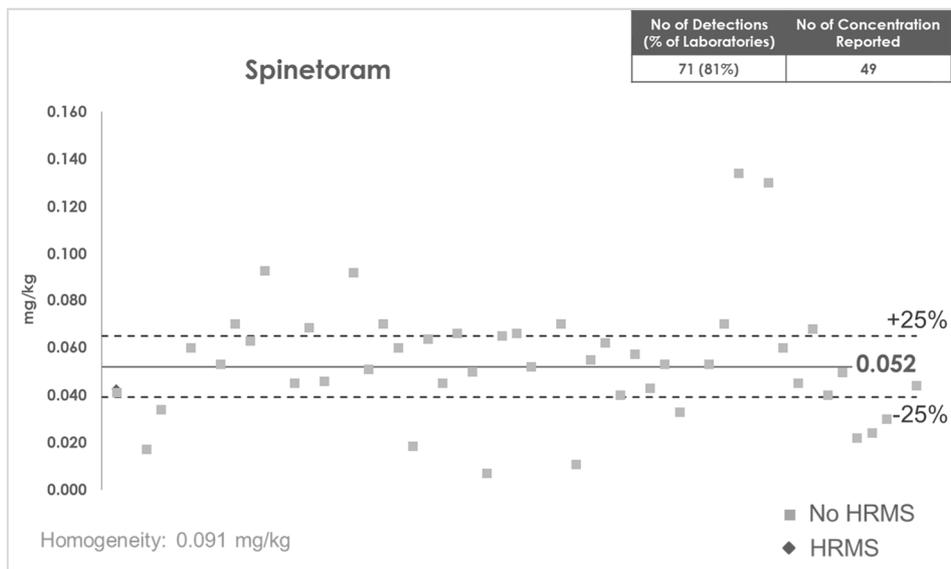
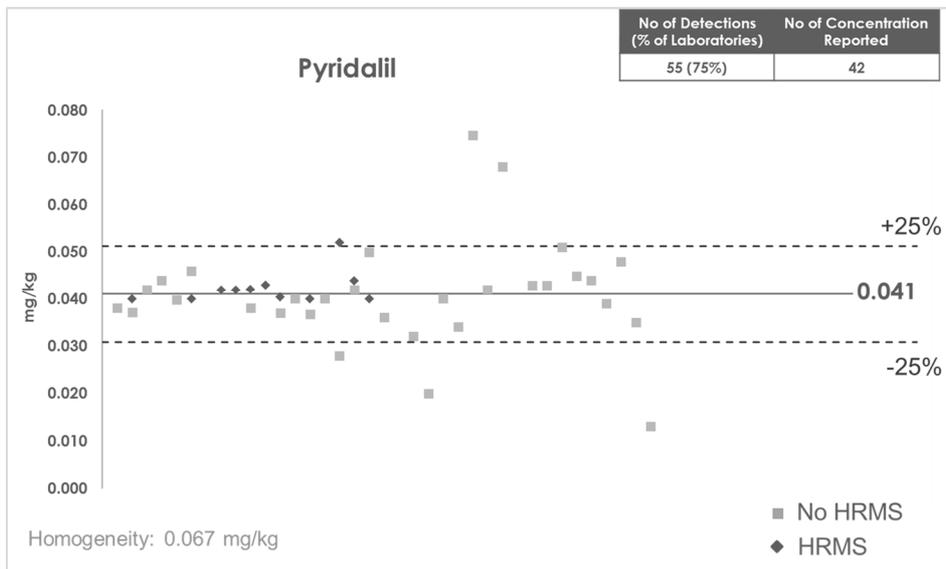
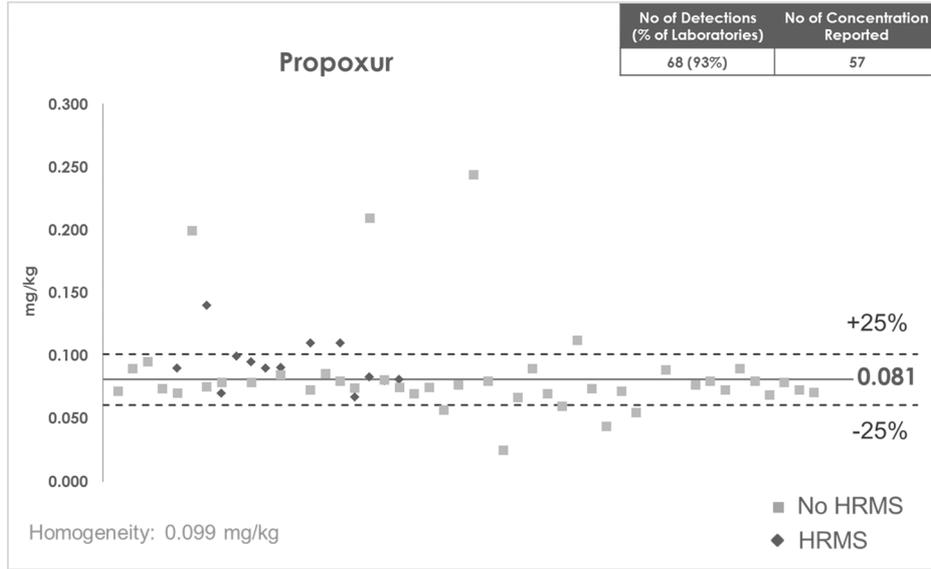


## APPENDIX 2. Graphical Representations

The bold line represents the robust mean

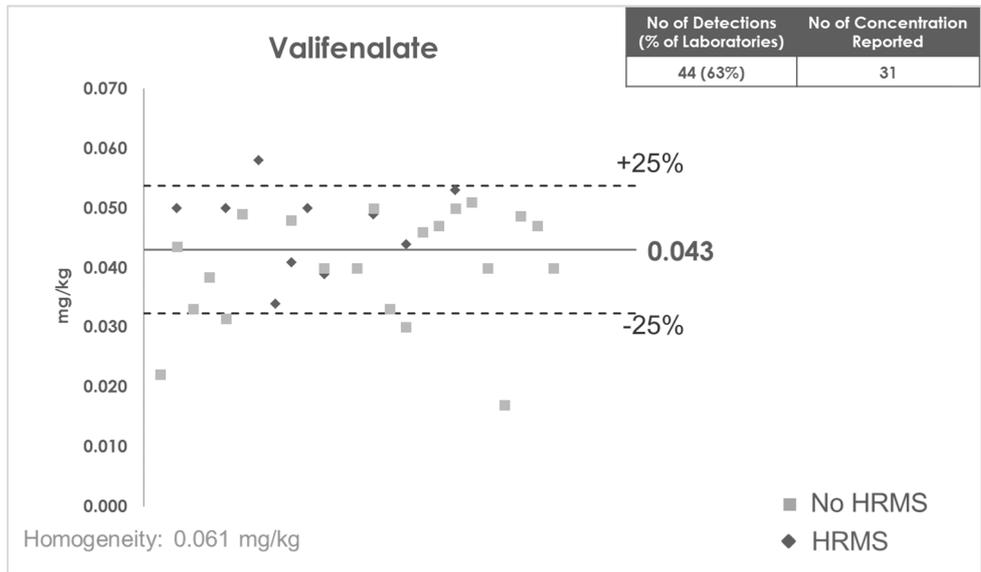
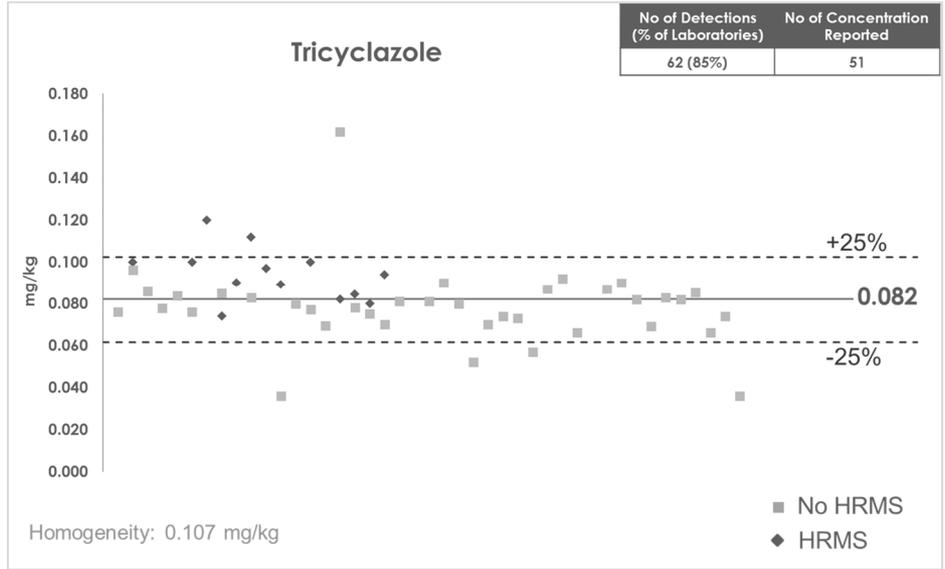


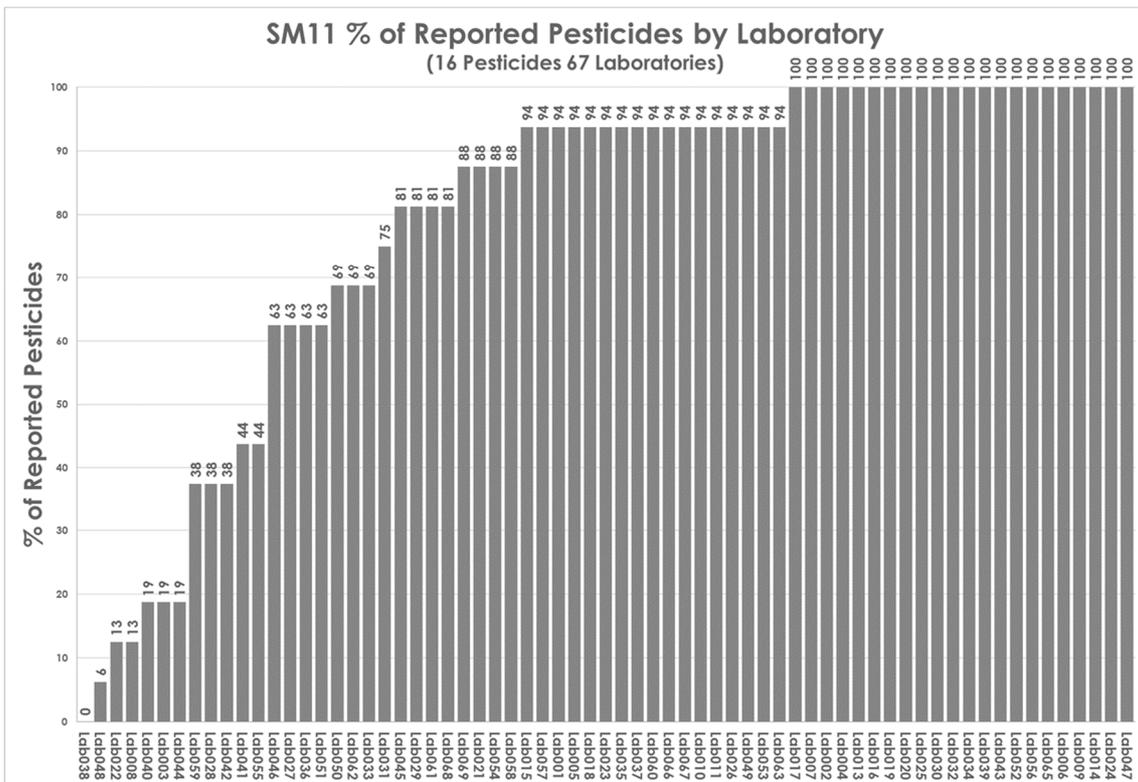
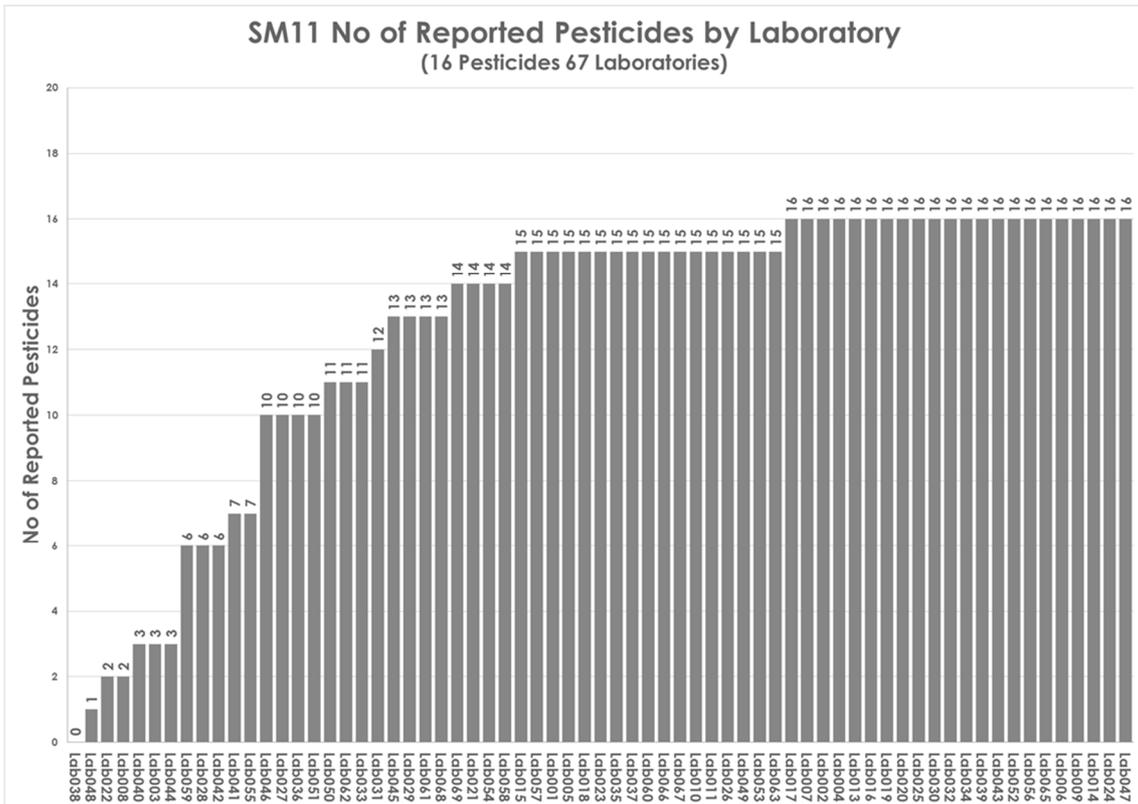
The bold line represents the robust mean



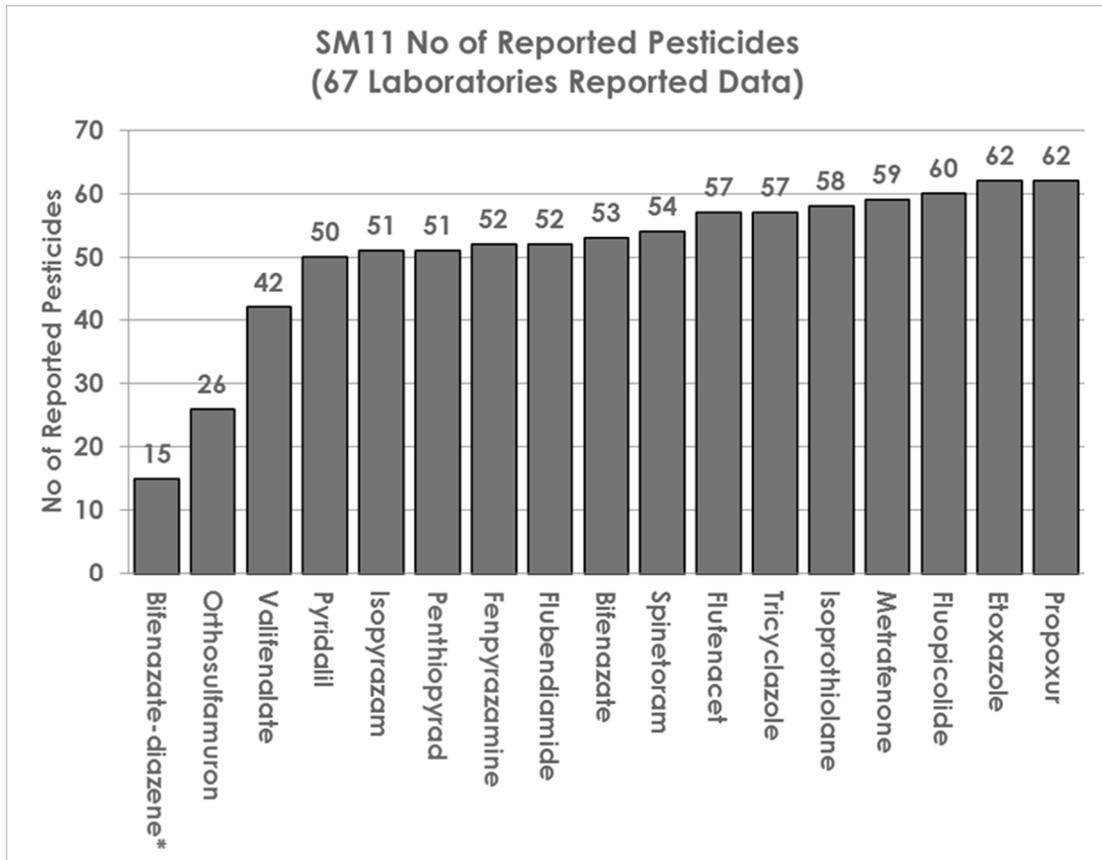
**APPENDIX 2. Graphical Representations**

The bold line represents the robust mean

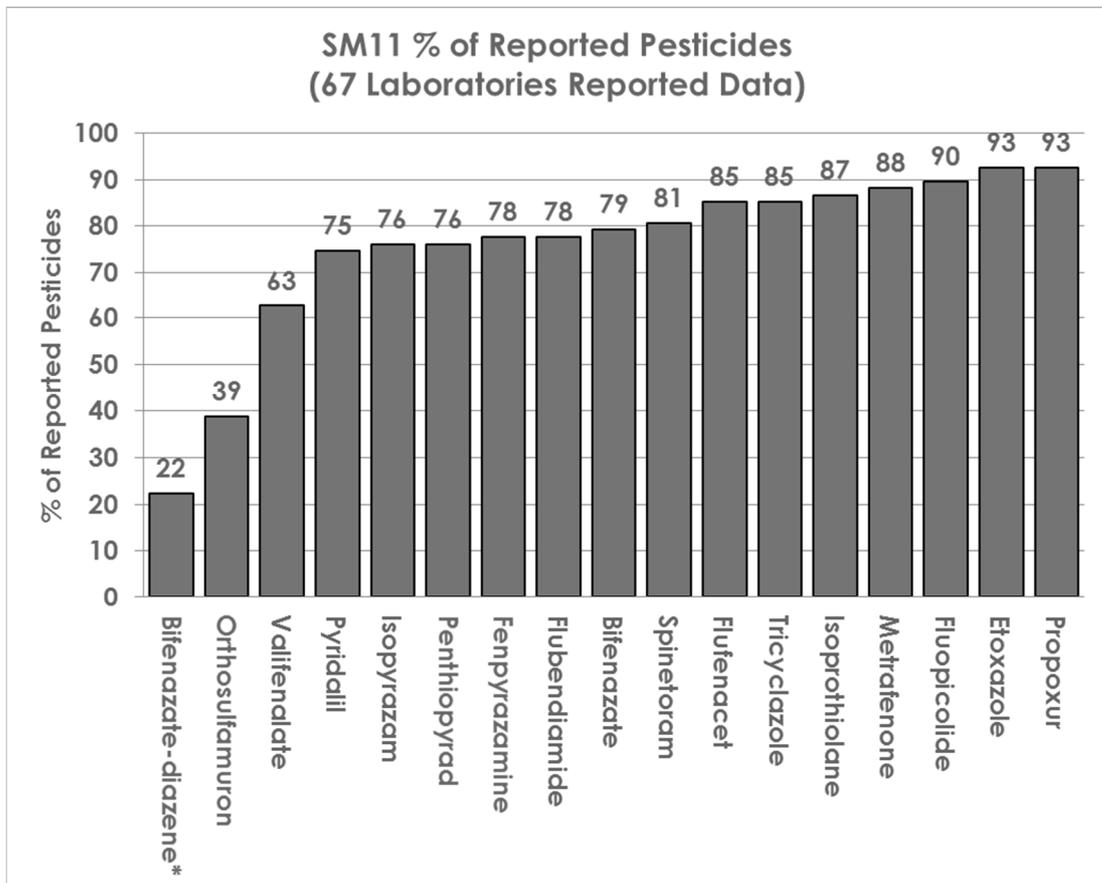




APPENDIX 2. Graphical Representations

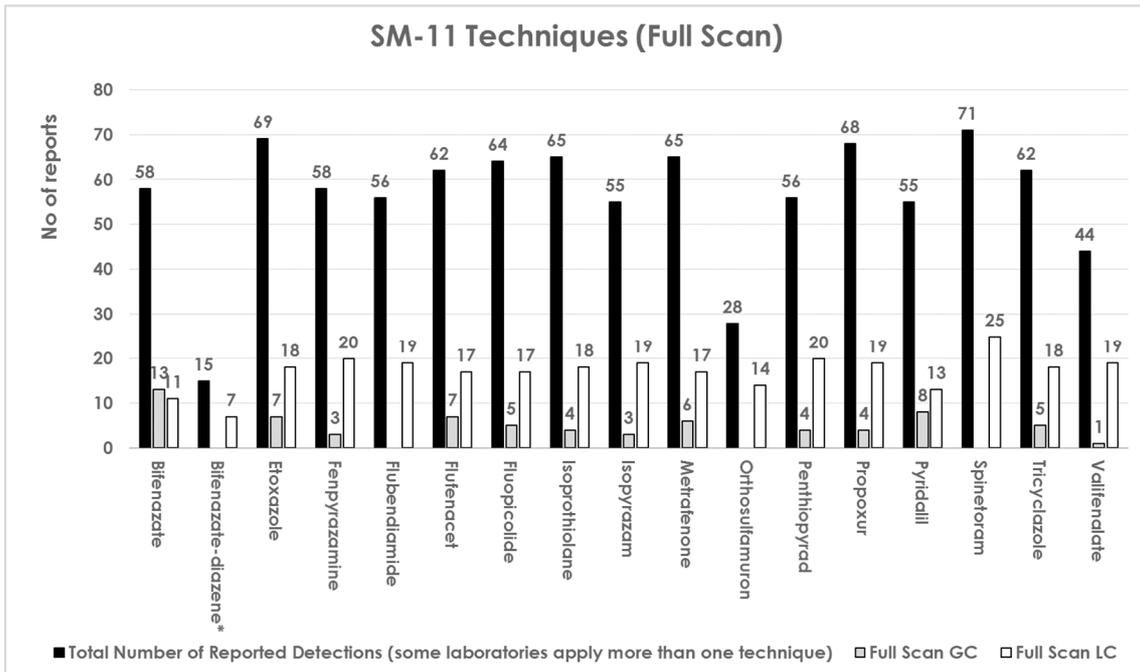


\*only informative purpose



\*only informative purpose

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



\*only informative purpose



**ANNEX 1. List of Laboratories that participate in EUPT-FV-SM11.**

**ANNEX 1. List of Laboratories that reported results in EUPT-FV-SM11.**

<b>COUNTRY</b>	<b>LABORATORY NAME</b>	<b>CITY</b>
AUSTRIA	DEPARTMENT FOR PESTICIDE AND FOOD ANALYTICS (PLMA)	INNSBRUCK
BELGIUM	LOVAP NV	GEEL
BELGIUM	PRIMORIS (PHYTO LAB)	GENT - ZWIJNAARDE
BELGIUM	SCIENSANO - PESTICIDE LAB	BRUSSELS
CHINA	AGRO-PRODUCT SAFETY RESEARCH CENTER - GUOFANG PANG	BEIJING
CHINA	BEIJING UNI-STAR INSPECTION - PESTICIDE LAB	BEIJING
CHINA	SCDC - PESTICIDE LAB	SHANGHAI
COSTA RICA	PESTICIDE LAB	SAN JOSÉ
CROATIA	SAMPLE CONTROL - PESTICIDE LAB	ZAGREB
CZECH REPUBLIC	CAFIA - PESTICIDE LAB (PRAHA)	PRAHA
CZECH REPUBLIC	VSCHT - PESTICIDE LAB	PRAHA
DENMARK	DTU, NATIONAL FOOD INSTITUTE	LYNGBY
EL EJIDO	LABORATORIO ALHONDIGA LA UNIÓN	ALMERIA
FINLAND	FINNISH CUSTOMS LABORATORY	ESPOO
FRANCE	ANSES -LSAL	MAISONS-ALFORT CEDEX
FRANCE	INOVALYS LE MANS - PESTICIDE LAB	LE MANS
FRANCE	SCL	MASSY CEDEX
FRANCE	SCL	MONTPELLIER
GERMANY	ANALYTICA ALIMENTARIA GMBH - LAB	KLEINMACHNOW
GERMANY	BUNDESWEHR - PESTICIDE LAB	GARCHING-HOCHBRÜCK
GERMANY	BVL UNIT 504 NRL FOR PESTICIDE RESIDUES	BERLIN
GERMANY	CVUA RRW - PESTICIDE LAB	KREFELD
GERMANY	CVUA STUTTGART - PESTICIDE LAB	FELLBACH
GERMANY	EUROFINS	HAMBURG
GERMANY	GALAB LABORATORIES GMBH	HAMBURG
GERMANY	LABOR FRIEDLE	TEGERNHEIM
GERMANY	LABOR MANG - PESTICIDE LAB	FRANKFURT
GERMANY	LALLF - PESTICIDE LAB	ROSTOCK
GERMANY	LAVES - PESTICIDE LAB	OLDENBURG
GERMANY	LGL ERLANGEN - PESTICIDE LAB	ERLANGEN
GERMANY	LTZ AUGUSTENBERG - ORGANIC ANALYSIS	KARLSRUHE
GERMANY	LUA SACHSEN - PESTICIDE LAB	DRESDEN
HUNGARY	NFC SO - PESTICIDE LAB	VELENCE
HUNGARY	NFC SO PESTICIDE LAB	HÓDMEZOVÁSÁRHELY
HUNGARY	NFC SO PESTICIDE LAB	MISKOLC
HUNGARY	NFC SO PESTICIDE LAB	SZOLNOK

**ANNEX 1. List of Laboratories that participate in EUPT-FV-SM11**

<b>COUNTRY</b>	<b>LABORATORY NAME</b>	<b>CITY</b>
IRELAND	PESTICIDE LAB	CO. KILDARE
ITALY	APPA BOLZANO - PESTICIDE LAB	BOLZANO
ITALY	ARPA FVG - PESTICIDE LAB	UDINE
ITALY	ARPA LAZIO (SEZ. LATINA) - PESTICIDE LAB	LATINA
ITALY	ASF - PESTICIDE LAB	FIRENZE
ITALY	IZS LT (SEZIONE FIRENZE) - PESTICIDE LAB	SAN MARTINO ALLA PALMA SCANDICCI
ITALY	IZS SICILIA - PESTICIDE LAB	PALERMO
ITALY	S.A.MER. SERVIZIO ANALISI CHIMICO MERCEOLOGICHE	BARI
KENYA	SGS KENYA LTD	MOMBASA
LITHUANIA	NMVRVI - PESTICIDE LAB	VILNIUS
NORWAY	NIBIO - DEPARTMENT OF PESTICIDE CHEMISTRY	ÅS
SLOVENIJA	PESTICIDE LAB	MARIBOR
SPAIN	AGRICULTURAL AND PHYTOPATHOLOGICAL LAB. OF GALICIA	ABEGONDO
SPAIN	ANALYTICA ALIMENTARIA GMBH	ALMERIA
SPAIN	EUROFINS ECOSUR - PESTICIDE LAB	LORQUÍ
SPAIN	EUROFINS SICA AGRIQ S.L.U. - PESTICIDE LAB	ALMERIA
SPAIN	LABORATORIO AGROALIMENTARIO DE EXTREMADURA	CÁCERES
SPAIN	LABORATORIO AGROALIMENTARIO Y DE SANIDAD ANIMAL	MURCIA
SPAIN	LABORATORIO AGROAMBIENTAL DE ZARAGOZA	ZARAGOZA
SPAIN	LABORATORIO DEL SOIVRE	ALMERÍA
SPAIN	LARAGA - PESTICIDE LAB	TOLEDO
SWEDEN	EUROFINS FOOD & FEED - PESTICIDE LAB	LIDKÖPING
SWEDEN	SCIENCE DEPARTMENT - CHEMISTRY DIVISION 1	UPPSALA
SWITZERLAND	PESTICIDE LAB	ZÜRICH
SWITZERLAND	SCAV - PESTICIDE LAB	GENEVE
THE NETHERLANDS	GROEN AGRO CONTROL - PESTICIDE LAB	DELFGAUW
THE NETHERLANDS	NOFALAB B.V.	SCHIEDAM
THE NETHERLANDS	NVWA - NRL FOR PESTICIDES	
THE NETHERLANDS	RIKILT - WAGENINGEN UNIVERSITY & RESEARCH	WAGENINGEN
TURKEY	SGS - FOOD CONTROL LABORATORY	MERSIN
UNITED KINGDOM	FERA - PESTICIDE LAB	YORK