EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUIT AND VEGETABLES.

SCREENING METHODS 10

(EUPT-FV-SM10)

Pesticide Residues in Green Bean Homogenate

Final Report

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CONTENT

EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUIT AND VEGETABLES. SCREENING METHODS 10

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 (EU) No 628/2017[2] lays down the responsabilities 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 nineth 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 Proficiency Test (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-FV20) 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 in the Standing Committee on Plants, Animals, Food and Feed-Phytopharmaceuticals – Pesticides Residues section.

[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 (EU) No 625/2017 of 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 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 "Analytical quality control and method validation procedures for pesticide residues and analysis in food and feed".

This EUPT-FV-SM10 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 green beans with pods. The green beans were cultivated in a farm in Almería, Spain.

The pesticides used to spike the green beans 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-SM10 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 2018, 2019 and 2020 (Regulation (EU) 2017/660).
- That they had particularly acute toxicity and/or had low ARfD values.

Seventeen pesticides were selected for the preparation of the test item (benalaxyl, clomazone, cyfluthrin, emamectin, etoxazole, fenpyrazamine, isopyrazam, metrafenone, penflufen, pentachloroaniline, penthiopyrad, proquinazid, prosulfocarb, spinetoram, spirotetramat, sulfoxaflor, tetramethrin). The pesticide treatments were carried out post-harvest using standard solutions. After the treatment, spirotetramat metabolites were also present in the test item. Spirotetramat-enol will be evaluated, but not spirotetramat–ketohydroxy, because it was present below 0.01 mg/kg. The test item was frozen (using liquid nitrogen) and chopped. The frozen minced green beans were mixed in a constantly-spinning container until a homogeneous item was obtained. Finally, 200 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.

2.2 Preparation of "blank" test item.

The green beans used to produce the blank item were 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.

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[3]. 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: $Ss^2 < c$, 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.

[3] 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.	084 A	084 B	052 A	052 B	143 A	143 B	066 A	066 B	014 A	014 B	096 A	096 B	108 A	108 B	011 A	011 B	122 A	122 B	137 A	137 B	A. Cc (mg/kg)	$Ss^2 < c$ Pass/Fail
Benalaxyl	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.050	Pass
Clomazone	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.011	Pass
Cyfluthrin	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.071	Pass
Emamectin	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.011	Pass
Etoxazole	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.012	Pass
Fenpyrazamine	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.010	Pass
Isopyrazam	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.014	Pass
Metrafenone	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.017	Pass
Penflufen	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.012	Pass
Pentachloroaniline	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.017	Pass
Penthiopyrad	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.094	Pass
Proquinazid	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.021	Pass
Prosulfocarb	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.015	Pass
Spinetoram	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.054	Pass
Spirotetramat	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.059	Pass
Spirotetramat-enol	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.025	Pass
Sulfoxaflor	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.011	Pass
Tetramethrin	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.015	Pass

I: Identified A. Cc: Average 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 (5th March 2018)			48h later Shipment Day (7th March 2018)			Few days after deadline (13th March 2018)		
Test item No.	049	103	135	034	82	147	015	092	123
Benalaxyl	I	I	I	I	I	I	I	I	I
Clomazone	I	I	I	I	I	I	I	I	I
Cyfluthrin	I	I	I	I	I	I	I	I	I
Emamectin	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
Isopyrazam	I	I	I	I	I	I	I	I	I
Metrafenone	I	I	I	I	I	I	I	I	I
Penflufen	I	I	I	I	I	I	I	I	I
Pentachloroaniline	I	I	I	I	I	I	I	I	I
Penthiopyrad	I	I	I	I	I	I	I	I	I
Proquinazid	I	I	I	I	I	I	I	I	I
Prosulfocarb	I	I	I	I	I	I	I	I	I
Spinetoram	I	I	I	I	I	I	I	I	I
Spirotetramat	I	I	I	I	I	I	I	I	I
Spirotetramat-enol	I	I	I	I	I	I	I	I	I
Sulfoxaflor	I	I	I	I	I	I	I	I	I
Tetramethrin	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 green beens homogenate together with another 200 g of 'blank' green beens homogenate were shipped to participants on 5th March 2018. 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, 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-SM10 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, taking into account 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 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- or GC-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

Seventy-three laboratories agreed to participate in this tenth proficiency test on screening methods. Sixty-nine laboratories submitted results on time (four 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 screening methods used are provided in Appendix 3 (available on the EUPT-FV-SM10 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 can be seen in Table 4.1a.

Table 4.1a Summary of Reported Results.

Pesticide	Reported		Not Reported	
	No. of laboratories	% of laboratories *	No. of laboratories	% of laboratories *
Benalaxyl	62	90	7	10
Clomazone	68	99	1	1
Cyfluthrin	62	90	7	10
Emamectin	54	78	15	22
Etoxazole	66	96	3	4
Fenpyrazamine	46	67	23	33
Isopyrazam	52	75	17	25
Metrafenone	67	97	2	3
Penflufen	39	57	30	43
Pentachloroaniline	47	68	22	32
Penthiopyrad	51	74	18	26
Proquinazid	59	86	10	14
Prosulfocarb	68	99	1	1
Spinetoram	53	77	16	23
Spirotetramat	59	86	10	14
Spirotetramat-enol	40	58	29	42
Sulfoxaflor	36	52	33	48
Tetramethrin	63	91	6	9

* The % of laboratories is calculated based on the total number of laboratories submitting results (69 laboratories).

In this EUPT-FV-SM10 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, the average concentrarion from the homogeneity test and the dispersion of the concentration results reported.

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

Pesticide	Average concentration Homogeneity test (mg/kg)	Robust mean of estimated concentrations reported (mg/kg)	CV (%)
Benalaxyl	0.050	0.041	22.0
Clomazone	0.011	0.010	21.0
Cyfluthrin	0.071	0.068	32.0
Emamectin	0.011	0.009	34.4
Etoxazole	0.012	0.013	26.0
Fenpyrazamine	0.010	0.008	26.8
Isopyrazam	0.014	0.009	29.3
Metrafenone	0.017	0.013	21.1
Penflufen	0.012	0.010	15.2
Pentachloroaniline	0.017	0.012	20.2
Penthiopyrad	0.094	0.067	23.4
Proquinazid	0.021	0.017	18.9
Prosulfocarb	0.015	0.011	22.1
Spinetoram	0.054	0.035	26.1
Spirotetramat	0.059	0.072	38.2
Spirotetramat–enol	0.025	0.036	69.0
Sulfoxaflor	0.011	0.009	24.2
Tetramethrin	0.015	0.011	15.7

No other compounds were identified and quantified by the organizer at concentrations above 0.010 mg/kg. Spirotetramat-enol was detected below 0.010 mg/kg.

4.1.1 Other Reported Pesticides

Some laboratories reported additional pesticides to those present in the test item. Some of them are reported below 0.01 mg/kg or not quantified.

Table 4.1.1 Other reported pesticides.

Laboratory Code	Other Reported Pesticides	Concentration Reported (mg/kg)
Lab002	TFNA	<< 0.01
Lab010	TFNA	0.01
Lab012	Chlorothalonil	0.018
Lab022	Chlorantraniliprole	
	Flufenoxuron	
Lab025	Triadimenol	<0.01
Lab027	Chlorothalonil	0.01
Lab029	Amitraz	0.037
Lab033	Jasmonic acid	
Lab040	TFNA	<0.005
Lab044	(Z)-13-hexadecen-11-yn-1-yl acetate	
	Binapacryl	
	Cinerin	
	Cycloxydim	0.035
	Hexachlorobenzene	0.001
	Kinoprene	
	Pyranocoumarin	
Lab047	Methacrifos	0.0096
Lab048	Chlorate	0.019 (no blank correction)
	Perchlorate	0.021 (no blank correction)

Laboratory Code	Other Reported Pesticides	Concentration Reported (mg/kg)
Lab049	Terbufos-oxon-sulfoxide	
Lab051	Tolclofos-methyl	0.004
Lab052	Pyrethrins	0.01
Lab054	Jasmonic acid	
Lab055	Kinoprene	
Lab056	Hexachlorobenzene	trace
Lab058	Isoxaben	> 0.01
	Methoxyfenozide	> 0.01
Lab070	TFNA	0.0044
Lab073	Spinosad	0.005

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

4.1.2 Non-Reported Pesticides

Table 4.1a shows for each pesticide present in the sample, 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.

Seventeen pesticides were used to spike the green beans at different levels between 0.010 mg/kg and 0.100 mg/kg (benalaxyl, clomazone, cyfluthrin, emamectin, etoxazole, fenpyrazamine, isopyrazam, metrafenone, penflufen, pentachloroaniline, penthiopyrad, proquinazid, prosulfocarb, spinetoram, spirotetramat, sulfoxaflor, tetramethrin). Spirotetramat metabolites, spirotetramat-enol and spirotetramat-ketoyhydroxy, were not spiked but they were also present in the test item. However, spirotetramat–ketohydroxy was present below 0.01 mg/kg, and thus, it will not be used for the evaluation of the laboratories.

4.3 Assessment of laboratory performance.

No z score values were calculated. Classification was based on 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
Lab003	18	100	0
Lab018	18	100	0
Lab019	18	100	0
Lab021	18	100	0
Lab024	18	100	0
Lab026	18	100	0
Lab032	18	100	0
Lab036	18	100	0
Lab038	18	100	0
Lab041	18	100	0
Lab043	18	100	0

Laboratory Code	No of Reported Pesticides	% of Reported Pesticides	Other Reported Pesticides Not Confirmed by the Organiser
Lab045	18	100	0
Lab059	18	100	0
Lab060	18	100	0
Lab064	18	100	0
Lab066	18	100	0
Lab002	18	100	1
Lab033	18	100	1
Lab049	18	100	1
Lab054	18	100	1
Lab070	18	100	1
Lab042	17	94	0
Lab025	17	94	1
Lab056	17	94	1
Lab022	17	94	2
lab048	17	94	2
Lab001	16	89	0
Lab046	16	89	0
Lab068	16	89	0
Lab069	16	89	0
Lab071	16	89	0
Lab004	15	83	0
Lab017	15	83	0
Lab031	15	83	0
Lab039	15	83	0
Lab010	15	83	1
Lab012	15	83	1
Lab073	15	83	1
Lab067	14	78	0
Lab005	13	72	0
Lab040	13	72	1
Lab051	13	72	1
Lab055	13	72	1
Lab030	12	67	0
Lab061	12	67	0
Lab065	12	67	0
Lab044	12	67	7
Lab015	11	61	0
Lab034	11	61	0
Lab050	11	61	0
Lab027	11	61	1
Lab007	10	56	0
Lab035	10	56	0
Lab014	9	50	0
Lab006	8	44	0
Lab008	7	39	0
Lab057	7	39	0

Laboratory Code	No of Reported Pesticides	% of Reported Pesticides	Other Reported Pesticides Not Confirmed by the Organiser
Lab047	7	39	1
Lab029	4	22	1
Lab052	4	22	1
Lab058	4	22	2
Lab009	3	17	0
Lab011	3	17	0
Lab062	3	17	0
Lab037	2	11	0
Lab016	1	6	0
Lab020	1	6	0
Lab072	1	6	0
Lab028	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-SM10 webpage, not in the printed version).

In Table 4.3.b there is a summary of the chromatographic techniques used for each pesticide, and a graphical representation is shown in Appendix 2.

Table 4.3.b 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
Benalaxyl	62	74	21	9	26	18
Clomazone	68	68	14	8	27	19
Cyfluthrin	62	62	45	13	2	2
Emamectin	54	54	0	0	37	17
Etoxazole	66	66	12	6	31	17
Fenpyrazamine	46	46	0	3	30	13
Isopyrazam	52	52	4	3	32	13
Metrafenone	67	67	20	9	22	16
Penflufen	39	39	5	4	20	10
Pentachloroaniline	47	47	32	15	0	0
Penthiopyrad	51	51	6	6	26	13
Proquinazid	59	59	5	6	31	17
Prosulfocarb	68	68	6	8	35	19
Spinetoram	53	56	0	0	38	18
Spirotetramat	59	59	0	0	43	16
Spirotetramat-enol	40	40	0	0	27	13
Sulfoxaflor	36	36	0	0	26	10
Tetramethrin	63	63	27	8	16	12

*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.

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

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

Laboratory Code	Number of Present Pesticides Reported (18 Evaluated Pesticides)	% of Present Pesticides Reported (18 Evaluated Pesticides)
Lab001	16	89
Lab002	18	100
Lab003	18	100
Lab004	15	83
Lab005	13	72
Lab006	8	44
Lab007	10	56
Lab008	7	39
Lab009	3	17
Lab010	15	83
Lab011	3	17
Lab012	15	83
Lab014	9	50
Lab015	11	61
Lab016	1	6
Lab017	15	83
Lab018	18	100
Lab019	18	100
Lab020	1	6
Lab021	18	100
Lab022	17	94
Lab024	18	100
Lab025	17	94
Lab026	18	100
Lab027	11	61
Lab028	0	0
Lab029	4	22
Lab030	12	67
Lab031	15	83
Lab032	18	100
Lab033	18	100
Lab034	11	61
Lab035	10	56
Lab036	18	100
Lab037	2	11
Lab038	18	100
Lab039	15	83
Lab040	13	72
Lab041	18	100
Lab042	17	94
Lab043	18	100
Lab044	12	67
Lab045	18	100
Lab046	16	89

Laboratory Code	Number of Present Pesticides Reported (18 Evaluated Pesticides)	% of Present Pesticides Reported (18 Evaluated Pesticides)
Lab047	7	39
lab048	17	94
Lab049	18	100
Lab050	11	61
Lab051	13	72
Lab052	4	22
Lab054	18	100
Lab055	13	72
Lab056	17	94
Lab057	7	39
Lab058	4	22
Lab059	18	100
Lab060	18	100
Lab061	12	67
Lab062	3	17
Lab064	18	100
Lab065	12	67
Lab066	18	100
Lab067	14	78
Lab068	16	89
Lab069	16	89
Lab070	18	100
Lab071	16	89
Lab072	1	6
Lab073	15	83

5. CONCLUSIONS

Seventy-three laboratories agreed to participate in this tenth proficiency test on screening methods. Sixty-nine laboratories submitted results on time (four laboratories cancelled their participation). Twenty EU Member States and in addition to these, 2 EFTA countries (Norway and Switzerland) as well as five non-EU/EFTA countries (China, Costa Rica, Kenya, Serbia and Turkey) participated in this European Union Proficiency Test.

Seventeen pesticides were used to spike the green beans test item at different levels between 0.010 mg/kg and 0.100 mg/kg (benalaxyl, clomazone, cyfluthrin, emamectin, etoxazole, fenpyrazamine, isopyrazam, metrafenone, penflufen, pentachloroaniline, penthiopyrad, proquinazid, prosulfocarb, spinetoram, spirotetramat, sulfoxaflor, tetramethrin). Spirotetramat-enol was also present in the test item in this range of concentration and it was evaluated, but not spirotetramat–ketohydroxy, because it was present below 0.01 mg/kg. Spirotetramat-ketohydroxy was reported by these laboratories:

- Lab001
- Lab002
- Lab004
- Lab024
- Lab025
- Lab026
- Lab027
- Lab033
- Lab043
- Lab049
- Lab054
- Lab059
- Lab060
- Lab067
- Lab069
- Lab070

Most laboratories analysed the test item using methods based on both gas and liquid chromatography combined with mass spectrometric detection. Of 1029 detections, 295 were made by GC and 734 by LC, 346 were made using full-scan, meaning 34% of detections (98 by GC-full scan techniques and 248 by LC-full scan techniques); 33% of the laboratories reported their results using HRMS (high resolution accurate mass spectrometry); 855 of the results were reported indicating a concentration value (83% of the total results).

Twenty-one of the 69 laboratories were able to detect 100% of the present pesticides in the test item (18 pesticides). Fifteen laboratories detected less than 50% of the pesticides present. Forty-three laboratories that reported results were able to find more than 70% of the evaluated pesticides.

Twenty-one participants reported 23 different pesticides 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 the 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.

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.
- Guidance document on analytical quality control and method validation procedures for pesticide residues and analysis in food and feed. (SANTE/11813/2017 Supersedes SANTE/11945/2015).

8. ACKNOWLEDGEMENTS

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

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APPENDIX 1. Results

Table AP1a. Reported pesticides

Laboratory Code Total No of Reporting Laboratories 69	Evaluated Pesticides (18) R: Reported Pesticide																		
	Benalaxyl	Clomazone	Cyfluthrin	Emamectin	Etoxazole	Fenpyrazamine	Isopyrazam	Metrafenone	Penflufen	Pentachloroaniline	Penthiopyrad	Proquinazid	Prosulfocarb	Spinetoram	Spirotetramat	Spirotetramat Metabolite BYI08330-enol	Sulfoxaflor	Tetramethrin	Reported Pesticides by Laboratory	% Reported Pesticides by Laboratory
Lab001	R	R	R		R	R	R	R	R		R	R	R	R	R	R	R	R	**16**	**89**
Lab002	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**18**	**100**
Lab003	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**18**	**100**
Lab004	R	R		R	R	R	R	R			R	R	R	R	R	R	R	R	**15**	**83**
Lab005	R	R	R	R	R	R	R	R		R		R	R		R			R	**13**	**72**
Lab006	R	R	R			R		R			R		R					R	**8**	**44**
Lab007	R	R			R	R	R	R				R	R		R			R	**10**	**56**
Lab008	R	R	R		R			R			R							R	**7**	**39**
Lab009	R		R											R					**3**	**17**
Lab010	R	R	R	R	R			R		R	R	R	R	R	R	R	R	R	**15**	**83**
Lab011			R		R										R				**3**	**17**
Lab012	R	R		R	R	R	R	R	R	R	R	R	R	R	R			R	**15**	**83**
Lab014	R	R	R					R		R		R	R		R			R	**9**	**50**
Lab015	R	R	R	R	R			R		R	R	R			R			R	**11**	**61**
Lab016			R																**1**	**6**
Lab017	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R			**15**	**83**
Lab018	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**18**	**100**
Lab019	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**18**	**100**
Lab020													R						**1**	**6**
Lab021	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**18**	**100**
Lab022	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	R	**17**	**94**
Lab024	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**18**	**100**
Lab025	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	**17**	**94**
Lab026	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**18**	**100**
Lab027	R	R	R	R	R			R		R		R	R		R	R			**11**	**61**
Lab028																			**0**	**0**
Lab029	R				R									R	R				**4**	**22**
Lab030	R	R	R	R	R			R		R		R	R	R	R			R	**12**	**67**
Lab031	R	R		R	R	R	R	R	R		R	R	R	R	R	R	R		**15**	**83**
Lab032	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**18**	**100**
Lab033	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**18**	**100**
Lab034		R	R		R	R	R	R				R	R	R	R			R	**11**	**61**
Lab035	R	R	R	R	R			R		R		R	R					R	**10**	**56**
Lab036	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**18**	**100**
Lab037	R		R																**2**	**11**
Lab038	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**18**	**100**
Lab039	R	R		R	R		R	R	R		R	R	R	R	R	R	R	R	**15**	**83**
Lab040	R	R	R	R	R		R	R		R		R	R		R	R		R	**13**	**72**
Lab041	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**18**	**100**
Lab042	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	**17**	**94**
Lab043	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**18**	**100**
Lab044	R	R	R	R	R					R	R	R	R	R	R			R	**12**	**67**
Lab045	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**18**	**100**
Lab046	R	R	R	R	R	R	R	R		R	R	R	R	R	R	R		R	**16**	**89**
Lab047	R	R	R		R			R				R	R						**7**	**39**
lab048	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	**17**	**94**
Lab049	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**18**	**100**
Lab050	R	R	R	R	R			R				R	R	R	R			R	**11**	**61**
Lab051	R	R	R	R	R		R	R		R		R	R		R	R		R	**13**	**72**
Lab052			R					R					R					R	**4**	**22**
Lab054	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**18**	**100**

APPENDIX 1

Laboratory Code Total No of Reporting Laboratories 69	Evaluated Pesticides (18) R: Reported Pesticide																		Reported Pesticides by Laboratory	% Reported Pesticides by Laboratory
	Benalaxyl	Clomazone	Cyfluthrin	Emamectin	Etoxazole	Fenpyrazamine	Isopyrazam	Metrafenone	Penflufen	Pentachloroaniline	Penthiopyrad	Proquinazid	Prosulfocarb	Spinetoram	Spirotetramat	Spirotetramat Metabolite BYI08330-enol	Sulfoxaflor	Tetramethrin		
Lab055	R	R	R		R	R	R	R	R	R	R	R	R	R					**13**	**72**
Lab056	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	**17**	**94**
Lab057	R		R	R	R									R	R			R	**7**	**39**
Lab058	R	R											R					R	**4**	**22**
Lab059	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**18**	**100**
Lab060	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**18**	**100**
Lab061	R	R	R		R	R	R	R		R	R		R	R				R	**12**	**67**
Lab062	R							R										R	**3**	**17**
Lab064	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**18**	**100**
Lab065	R	R	R	R	R			R		R		R	R	R	R			R	**12**	**67**
Lab066	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**18**	**100**
Lab067	R	R		R	R	R	R	R	R		R		R	R	R		R	R	**14**	**78**
Lab068	R	R	R	R	R	R	R	R		R	R	R	R	R	R	R		R	**16**	**89**
Lab069	R	R	R	R	R	R	R	R		R	R	R	R	R	R	R		R	**16**	**89**
Lab070	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**18**	**100**
Lab071	R	R	R	R	R	R	R	R	R		R	R	R	R	R		R	R	**16**	**89**
Lab072			R																**1**	**6**
Lab073	R	R	R	R		R	R	R		R		R	R	R	R	R	R	R	**15**	**83**
Reported Pesticides	**62**	**68**	**62**	**54**	**66**	**46**	**52**	**67**	**39**	**47**	**51**	**59**	**68**	**53**	**59**	**40**	**36**	**63**		
% of Reported Pesticides	**90**	**99**	**90**	**78**	**96**	**67**	**75**	**97**	**57**	**68**	**74**	**86**	**99**	**77**	**86**	**58**	**52**	**91**		

Table AP1b. Estimated Concentrations Reported
Not all the laboratories reporting results have reported estimated concentration values
Results reported without concentration values are expressed as R.

Laboratory Code Total No of Reporting Laboratories 69	Evaluated Pesticides (18)																	
	Benalaxyl	Clomazone	Cyfluthrin	Emamectin	Etoxazole	Fenpyrazamine	Isopyrazam	Metrafenone	Penflufen	Pentachloroaniline	Penthiopyrad	Proquinazid	Prosulfocarb	Spinetoram	Spirotetramat	Spirotetramat Metabolite BYI08330-enol*	Sulfoxaflor	Tetramethrin
Homogeneity (mg/kg)	**0.050**	**0.011**	**0.071**	**0.011**	**0.012**	**0.010**	**0.014**	**0.017**	**0.012**	**0.017**	**0.094**	**0.021**	**0.015**	**0.054**	**0.059**	**0.025**	**0.011**	**0.015**
Robust Mean (mg/kg)	**0.041**	**0.010**	**0.068**	**0.009**	**0.013**	**0.008**	**0.009**	**0.013**	**0.010**	**0.012**	**0.067**	**0.017**	**0.011**	**0.035**	**0.072**	**0.036**	**0.009**	**0.011**
CV (%)	**22.0**	**21.0**	**32.0**	**34.4**	**26.0**	**26.8**	**29.3**	**21.1**	**15.2**	**20.2**	**23.4**	**18.9**	**22.1**	**26.1**	**38.2**	**69.0**	**24.2**	**15.7**
001	0.042	0.011	0.08		0.03	0.007	0.01	0.013	0.01		0.076	0.019	0.012	0.08	0.083	0.036	0.011	0.01
002	0.05	0.01	0.08	R	0.015	R	R	0.015	0.01	0.015	0.05	0.02	0.01	0.035	0.1	0.015	R	0.01
003	0.03664	0.0082586	0.0548	0.0077511	4.3181862	0.0067501	0.0068457	0.0093146	0.0088912	0.0126	0.0610248	0.0200609	0.0091431	0.0297576	0.0673128	0.0710167	0.0087377	0.0092929
004	0.045	0.01		0.01	0.01	0.01	0.01	0.012			0.07	0.015	0.01	0.032	0.038	0.04	0.01	0.01
005	0.08	0.007	0.1	0.011	0.013	0.008	0.012	0.016		0.01		0.02	0.01		0.09			0.01
006	0.043	0.01	0.095			0.009		0.015			0.079		0.011					0.011
007	0.053	0.013			0.014	0.008	0.011	0.013				0.015	0.014		0.082			0.011
008	0.044	0.009	0.058		0.012			0.013			0.08							0.012
009	0.0344		0.0793											0.027				
010	0.036	0.008	0.076	0.007	0.012			R		0.011	0.062	0.019	0.011	0.042	0.079	0.02	0.006	0.009
011			0.088		0.012										0.045			
012	0.0447	0.0105		0.0103	0.013	0.0102	0.0106	0.0149	0.0116	0.014	0.0801	0.0202	0.0127	0.0394	0.057			0.0116
014	0.03	0.01	0.05					0.01		0.012		0.015	0.01		0.06			0.01
015	0.051	0.013	0.068	0.008	0.015			0.015		0.015	R	0.036			0.12			0.013
016			0.066															
017	0.035	0.011	0.044		0.025	0.007	0.01	0.013	0.012	0.016	0.008	0.002	0.002	0.036	0.081	0.022		
018	0.056	0.01	0.07	0.009	0.01	0.008	0.007	0.013	0.007	0.05	0.092	0.035	0.013	0.046	0.089	0.036	0.012	0.02
019	0.04	0.009	0.07	0.014	0.011	0.008	0.009	0.014	0.01	0.015	0.074	0.018	0.011		0.053	0.023	0.007	0.011
020													0.015					
021	0.043	0.01	0.076	0.0004	0.03	0.007	0.008	0.019	0.009	0.014	0.074	0.018	0.011	0.036	0.079	0.038	0.01	0.007
022	0.043	0.011	0.066		0.021	0.011		0.012	0.016	0.014	0.051	0.014	0.014					0.011
024	0.034	0.008	0.0433	0.0006	0.0144	0.006	0.007	0.0123	0.007	0.0127	0.06	0.015	0.009	0.006	0.056	0.012	0.007	0.008
025	0.053	0.011	0.085	0.011	0.014		R	0.013	0.01	0.013	0.081	0.017	0.013	0.036	0.084	0.027	0.01	0.016
026	0.042	0.01	0.074	0.01	0.013	0.01	0.01	0.015	0.012	0.011	0.089	0.019	0.01	0.054	0.082	0.038	0.01	0.01
027	0.027	0.01	0.044	0.01	0.01			0.01		0.01		0.02	0.01		0.16	0.094		
028	0.055				0.012									0.041	0.076			
029	0.0628	0.0157	0.125	0.0061	0.0101			0.0207		0.015		0.0209	0.0226	0.0287	0.0024			0.0116
030																		
031	0.05	0.012	0.12	0.01	0.013	0.01	0.01	0.013	0.01	0.01	0.08	0.02	0.012	0.04	0.1	0.02	0.01	0.01

APPENDIX 1

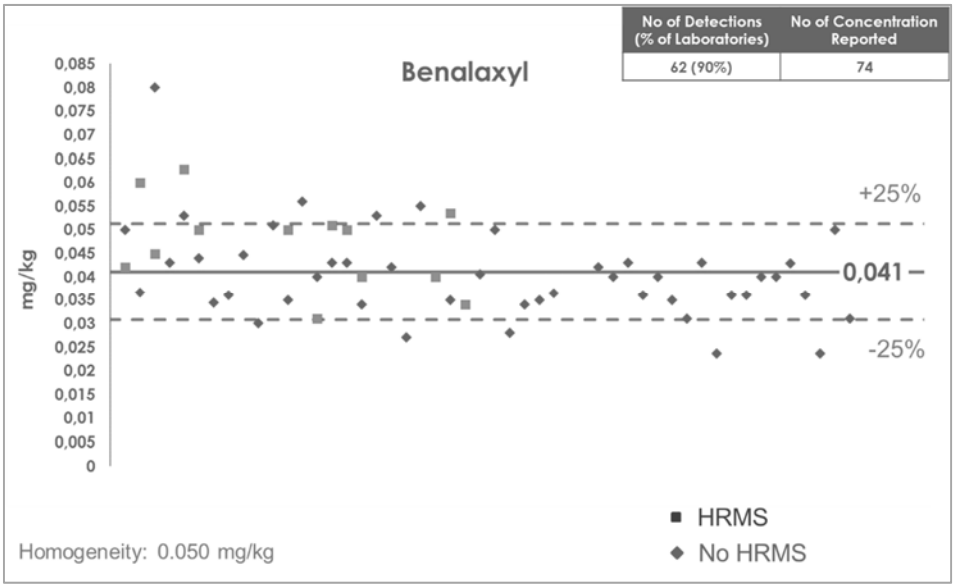
Laboratory Code Total No of Reporting Laboratories 69	Evaluated Pesticides (18)																	
	Benalaxyl	Clomazone	Cyfluthrin	Emamectin	Etoxazole	Fenpyrazamine	Isopyrazam	Metrafenone	Penflufen	Pentachloroaniline	Penthiopyrad	Proquinazid	Prosulfocarb	Spinetoram	Spirotetramat	Spirotetramat Metabolite BYI08330-enol*	Sulfoxaflor	Tetramethrin
Homogeneity (mg/kg)	**0.050**	**0.011**	**0.071**	**0.011**	**0.012**	**0.010**	**0.014**	**0.017**	**0.012**	**0.017**	**0.094**	**0.021**	**0.015**	**0.054**	**0.059**	**0.025**	**0.011**	**0.015**
Robust Mean (mg/kg)	**0.041**	**0.010**	**0.068**	**0.009**	**0.013**	**0.008**	**0.009**	**0.013**	**0.010**	**0.012**	**0.067**	**0.017**	**0.011**	**0.035**	**0.072**	**0.036**	**0.009**	**0.011**
CV (%)	**22.0**	**21.0**	**32.0**	**34.4**	**26.0**	**26.8**	**29.3**	**21.1**	**15.2**	**20.2**	**23.4**	**18.9**	**22.1**	**26.1**	**38.2**	**69.0**	**24.2**	**15.7**
032	0.035	0.01	0.052		0.064	0.007	0.007	0.012	0.01	0.005	0.065	0.018	0.012		0.072	0.105	0.007	0.01
033		0.012	0.048		0.012	0.011	0.011	0.012				0.022	0.021	0.071	0.072			0.011
034		0.025						0.03				0.05	0.025					
035	0.0405	0.0075	0.054	0.0062	0.0097	0.005	0.0052	0.021	0.0057	0.0118	0.058	0.0167	0.0083	0.0326	0.0517	0.0179	0.0123	0.0097
036	0.05		0.11															
037	0.05	0.01	0.09	0.01	0.02	0.01	0.01	0.01	0.01	0.01	0.09	0.02	0.01	0.03	0.1	0.01	0.01	0.01
038	R	R		R	R		R	R	R		R	R	R	R	R	R	R	R
039	0.028	0.007	0.067	0.006	0.009		0.005	0.011		0.009		0.012	0.009		0.055	0.029		0.009
040	0.031	0.01	0.063	0.01	0.01	0.01	0.01	0.011	0.01	0.012	0.058	0.016	0.01	0.029	0.098	0.043	0.01	0.01
041	0.035	0.01	0.18	0.01	0.015	0.01	0.01	0.01	0.01	0.01	0.075	0.015	0.01	0.04	0.07		0.01	0.01
042	0.03646281	0.01	0.048922178	0.01	0.01	0.0105	0.01	0.011507244	0.013	0.011004007	0.076	0.015183288	0.01	0.0335	0.0925	0.0185	0.01	0.010102827
043	0.051	0.015	0.025	0.004	0.011					R	R	R	R	0.052	0.072			0.022
044	0.042	0.01	0.063	0.0075	0.01	0.0071	0.0089	0.01	0.01	0.011	0.056	0.012	0.01	0.048	0.047	0.061	0.01	0.018
045	0.04	0.012	0.065	0.011	0.013	0.012	0.013	0.019		0.011	0.12	0.021	0.015	0.032	0.069	0.071		0.011
046	0.043	0.0088	0.069		0.026			0.011				0.019	0.0059					
047	0.036	R	0.036	0.013	R		R	0.012	R	0.01	0.063	0.017	R	0.025	0.084	R	R	R
048	0.05	0.013	0.051	0.004	0.051	0.006	0.006	0.08	0.01	0.012	0.057	0.016	0.014	0.02	0.07	0.01	0.0065	0.01
049	0.04	0.01	0.068	0.009	0.021			0.014				0.016	0.013	0.033	0.071			0.01
050	0.035	0.01	0.168	0.02	0.012		0.06	0.012		0.012		0.012	0.012		0.09	0.06		0.01
051			0.071					0.013					0.01					0.01
052	0.04	0.001	0.075	0.01	0.01	0.005	0.006	0.013	0.012	0.015	0.07	0.02	0.007	0.035	0.17	0.01	0.004	0.011
054																		
055						R												
056	0.031		0.082	0.012	0.015									0.081	0.101			0.018
057	R	R											R					R
058	0.043	0.011	0.061	0.008	0.011	0.008	0.008	0.013	0.01	0.013	0.068	0.015	0.013	0.037	0.021	0.063	0.008	0.012
059	0.0236	0.0043	0.0435	0.005	0.0098	0.0046	0.0055	0.0066	0.0044	0.0088	0.0466	0.0137	0.0084	0.0249	0.0483	0.0108	0.0053	0.0075
060	0.036	0.011	0.054		0.012	0.0087	0.012	0.013		0.037	0.052		0.013	0.026				0.012
061	0.04							0.02										0.01
062	0.04	0.018	0.068	R	0.012	0.015	0.02	0.015	0.01	0.012	0.067	0.017	0.03	0.025	0.11	0.072	0.012	0.017
064	0.0535	0.01677	0.237	0.006	0.01205			0.02182		0.013		0.01659	0.02334	0.031	0.00419			0.01287
065	0.04	0.009	0.082	0.009	0.011	0.007	0.007	0.012	0.01	0.011	0.071	0.018	0.01	0.037	0.063	0.032	0.008	0.016
066	0.034	0.0085		0.009	0.0098	0.0085	0.012	0.011	0.011		0.067		0.011	0.029	0.012		0.011	0.01

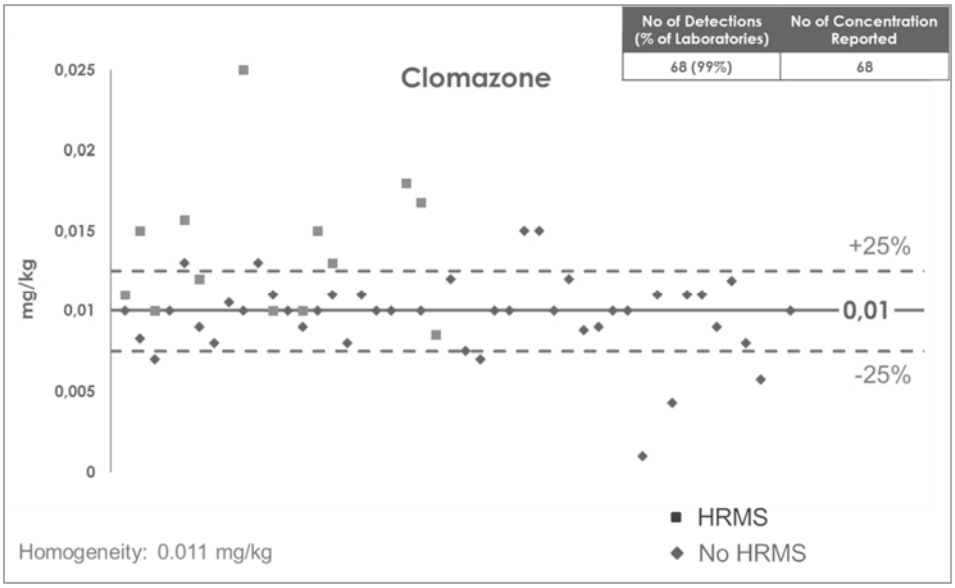
Laboratory Code Total No of Reporting Laboratories 69	**Evaluated Pesticides (18)**																	
	Benalaxyl	**Clomazone**	**Cyfluthrin**	**Emamectin**	**Etoxazole**	**Fenpyrazamine**	**Isopyrazam**	**Metrafenone**	**Penflufen**	**Pentachloroaniline**	**Penthiopyrad**	**Proquinazid**	**Prosulfocarb**	**Spinetoram**	**Spirotetramat**	**Spirotetramat Metabolite BYI08330-enol***	**Sulfoxaflor**	**Tetramethrin**
Homogeneity (mg/kg)	**0.050**	**0.011**	**0.071**	**0.011**	**0.012**	**0.010**	**0.014**	**0.017**	**0.012**	**0.017**	**0.094**	**0.021**	**0.015**	**0.054**	**0.059**	**0.025**	**0.011**	**0.015**
Robust Mean (mg/kg)	**0.041**	**0.010**	**0.068**	**0.009**	**0.013**	**0.008**	**0.009**	**0.013**	**0.010**	**0.012**	**0.067**	**0.017**	**0.011**	**0.035**	**0.072**	**0.036**	**0.009**	**0.011**
CV (%)	**22.0**	**21.0**	**32.0**	**34.4**	**26.0**	**26.8**	**29.3**	**21.1**	**15.2**	**20.2**	**23.4**	**18.9**	**22.1**	**26.1**	**38.2**	**69.0**	**24.2**	**15.7**
067	0.0429	0.0119	0.0615	0.0064	0.0113	R	R	0.0122		0.0142	R	0.018	0.0119	0.0311	0.0691	R		0.0119
068	0.036	0.008	0.06	0.008	0.012	0.008	0.01	0.01		0.012	0.025	0.021	0.012	0.028	0.026	0.04		0.009
069	0.0236	0.00575	0.0322	0.0044	0.0086	0.0054	0.006	0.0078	0.0088	0.0082	0.0502	0.0104	0.0102	0.0254	0.124	0.0082	0.008	0.0044
070	0.05	R	0.046	R	0.012	R	R	0.015	0.011		0.046	0.016	R	0.044	0.055		R	0.011
071			0.039															
072	0.031	0.01	0.082	0.01		0.01	0.01	0.013		0.01		0.016	0.01	0.035	0.041	0.049	0.01	0.01
073	0.042	0.011	0.08		0.03	0.007	0.01	0.013	0.01		0.076	0.019	0.012	0.08	0.083	0.036	0.011	0.01

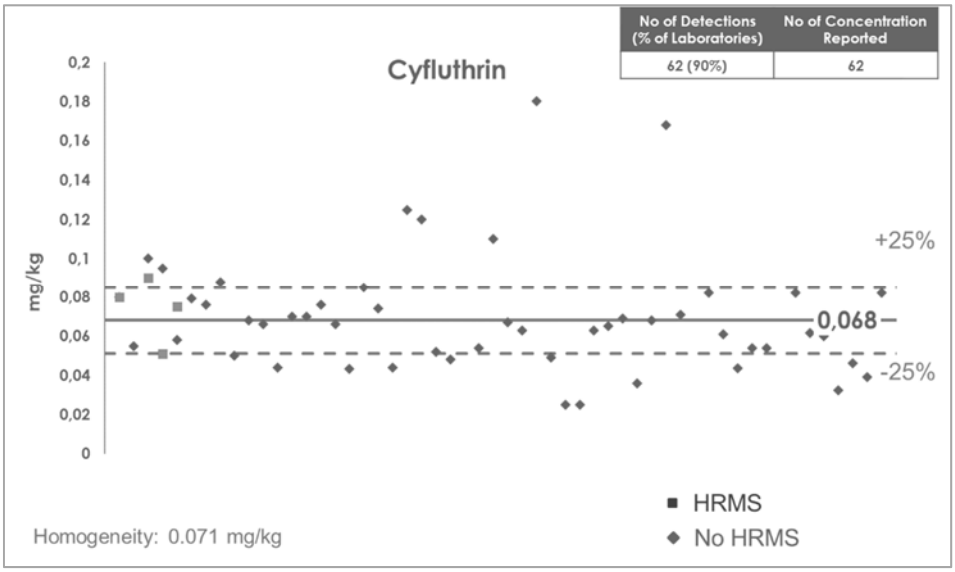
* These two compounds were not spiked, thus homogeneity and stability tests were not performed

APPENDIX 2. Graphical Representations

The bold line represents the robust mean

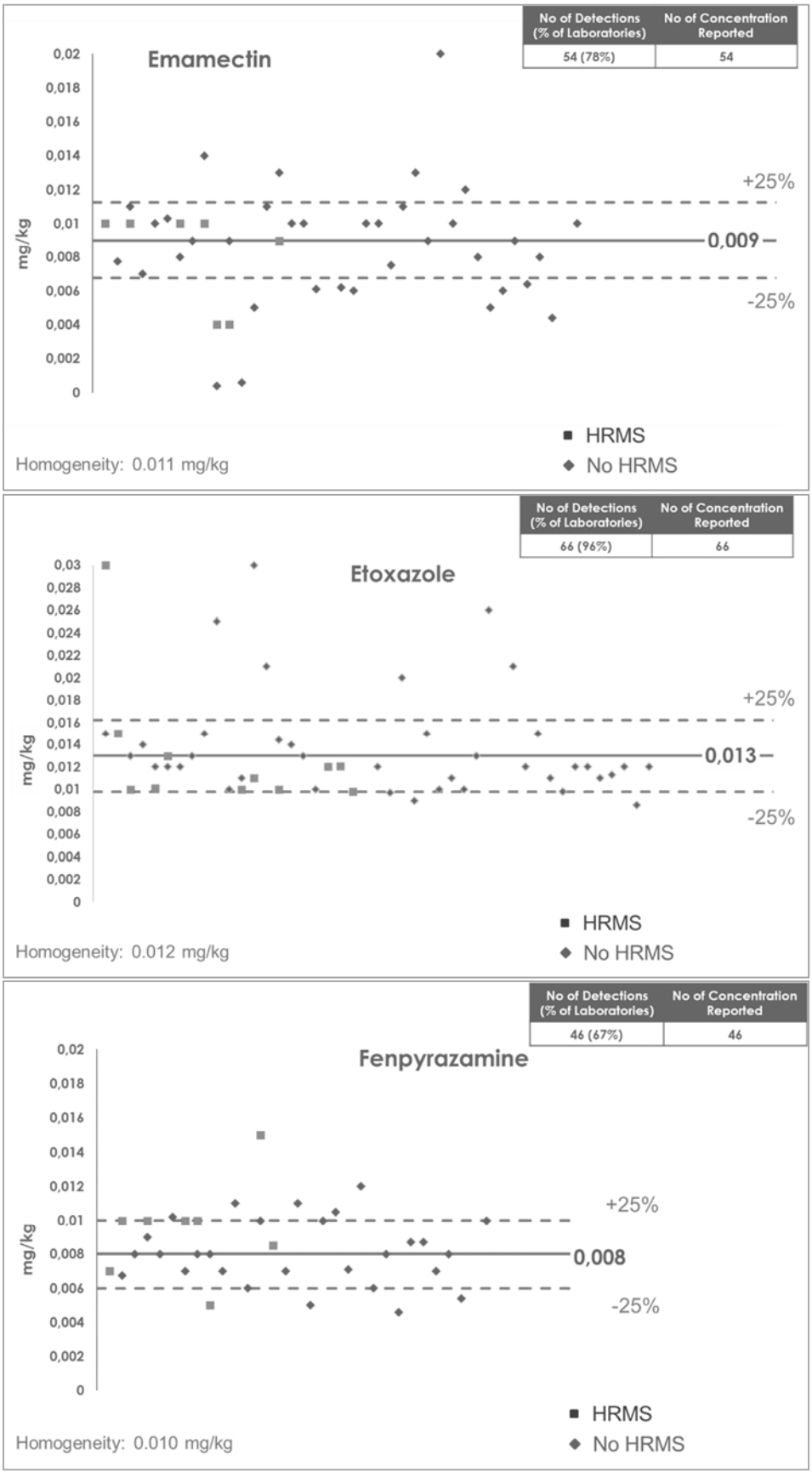




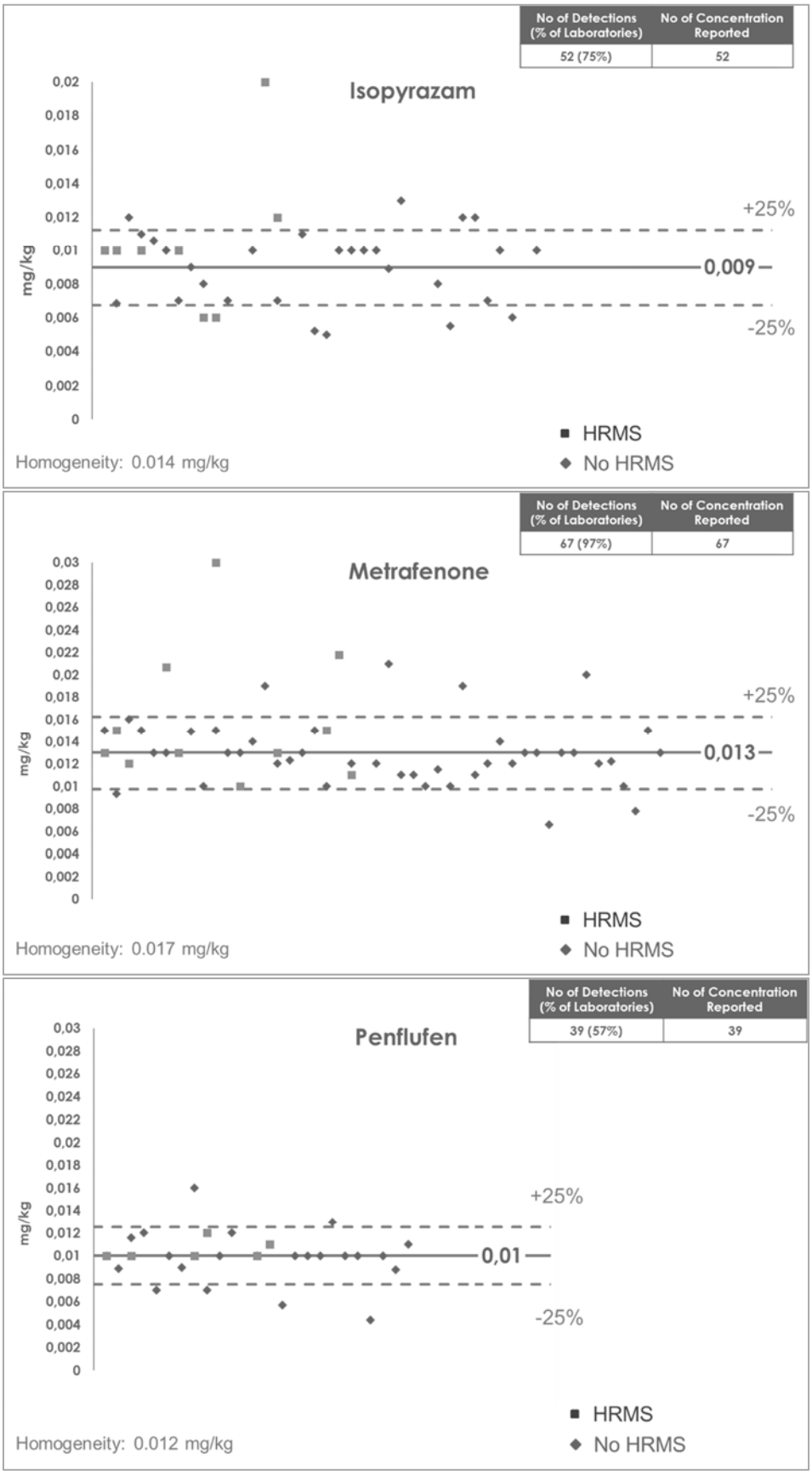


APPENDIX 2. Graphical Representations

The bold line represents the robust mean

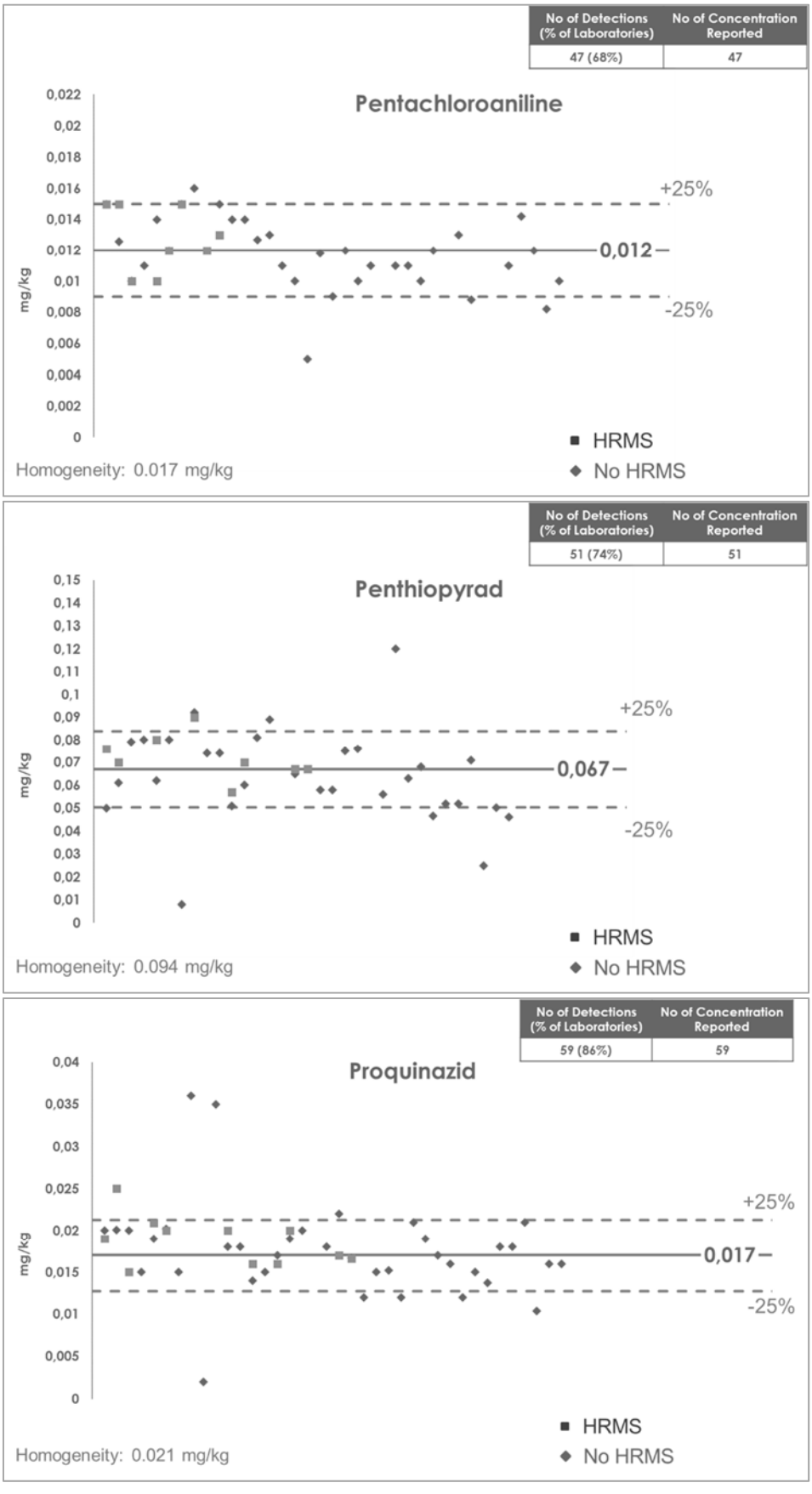


The bold line represents the robust mean

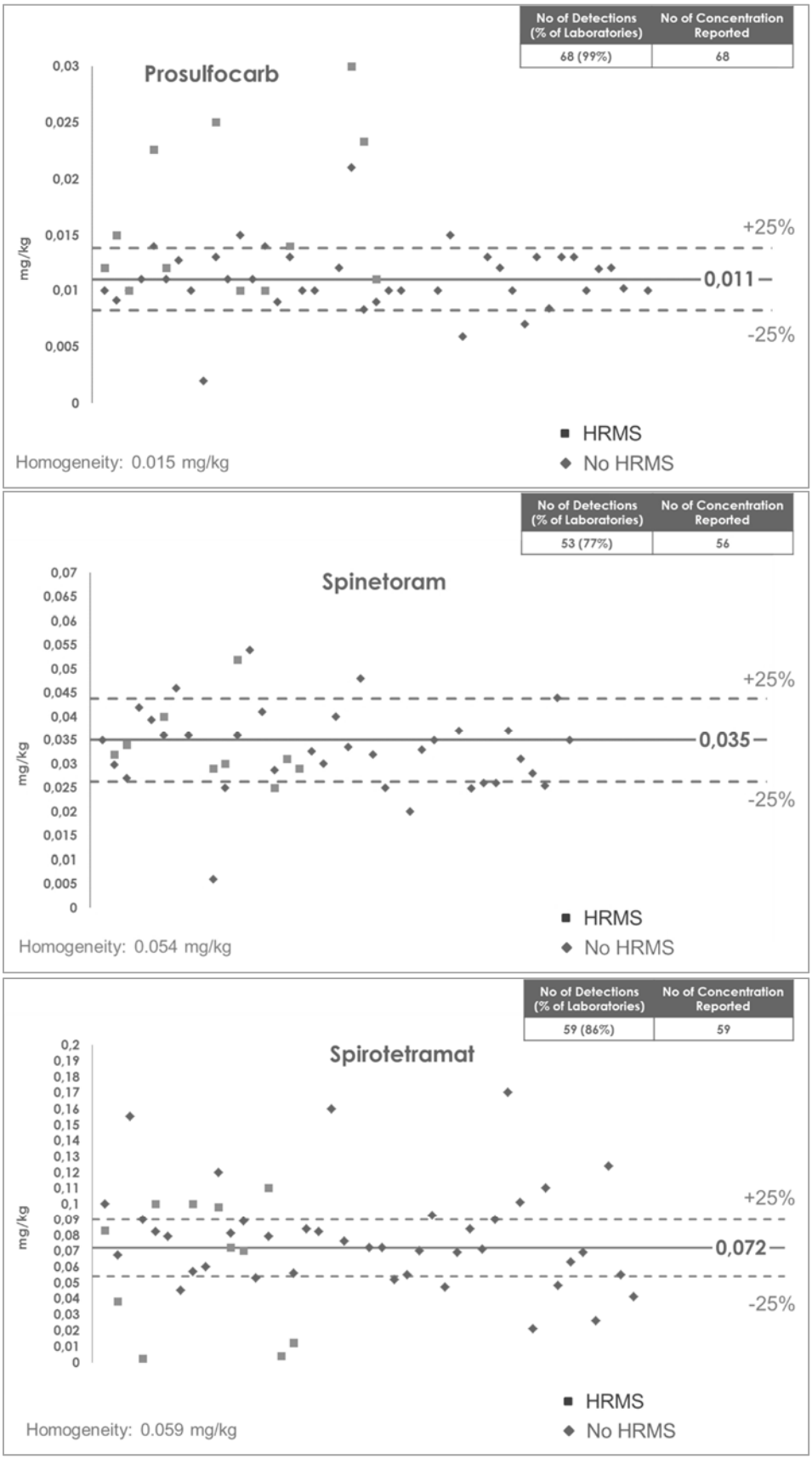


APPENDIX 2. Graphical Representations

The bold line represents the robust mean

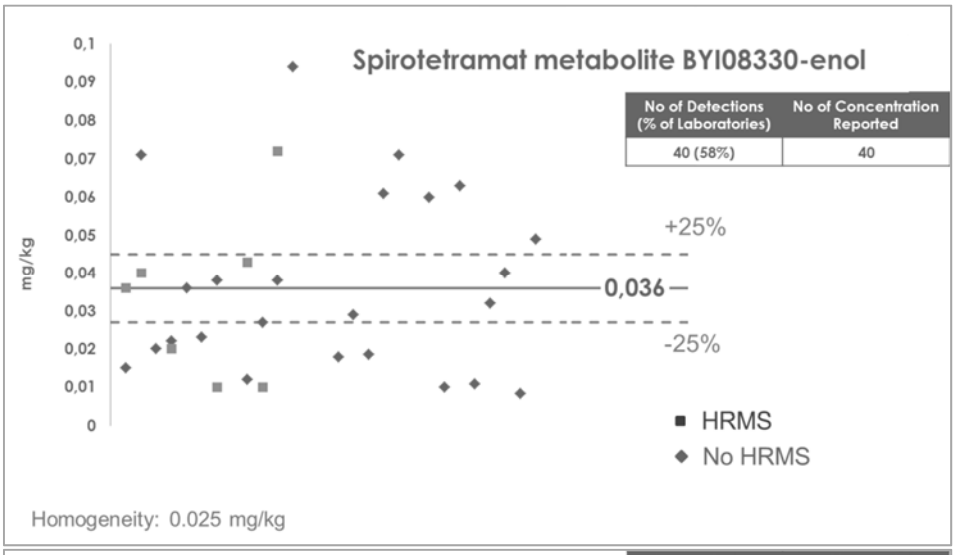


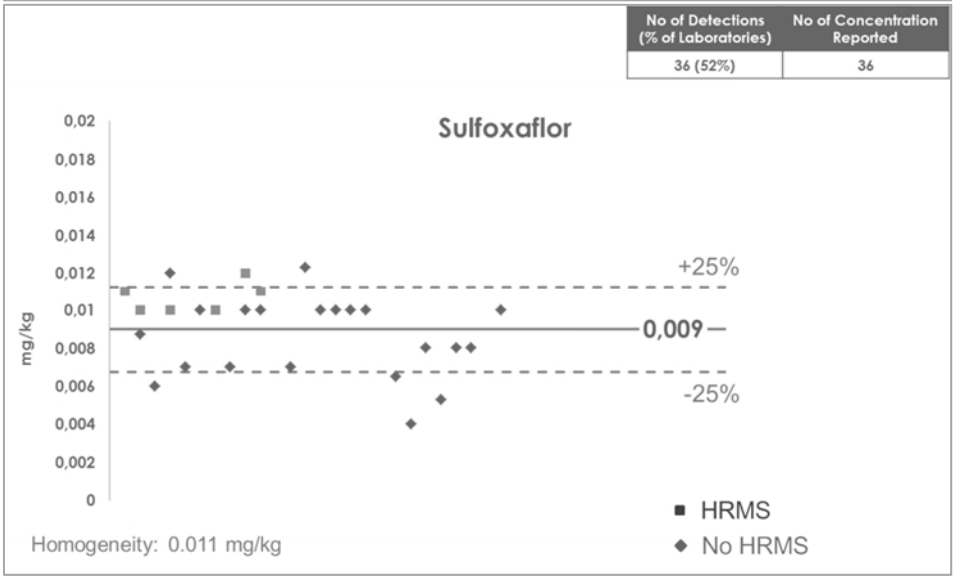
The bold line represents the robust mean

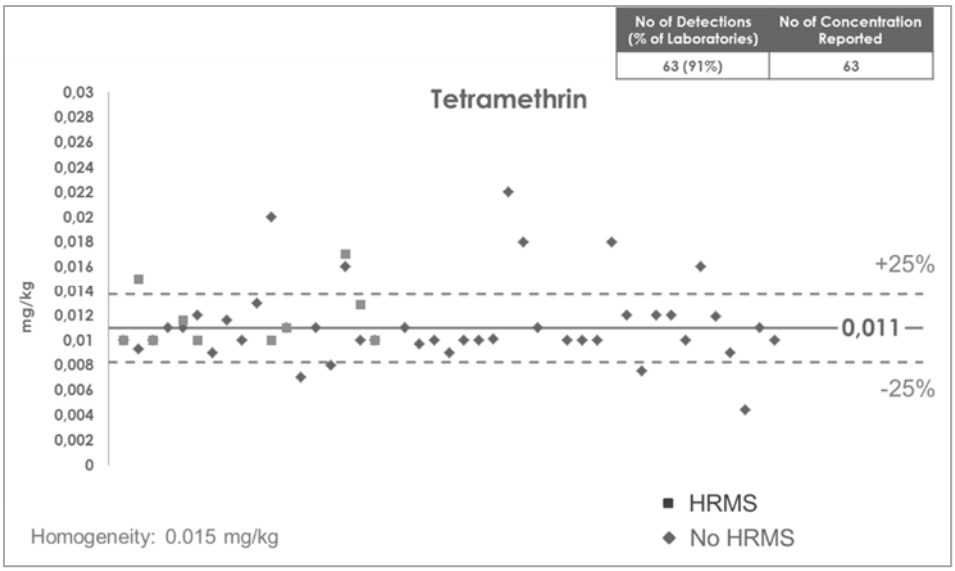


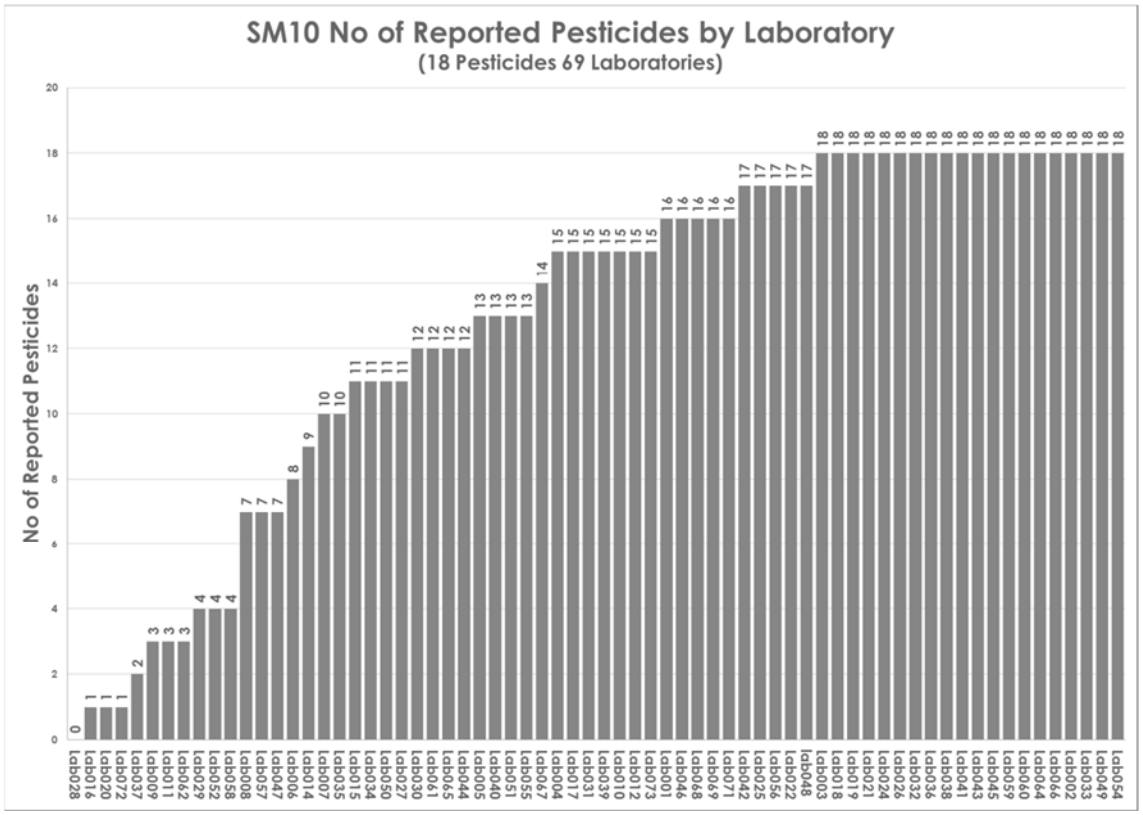
APPENDIX 2. Graphical Representations

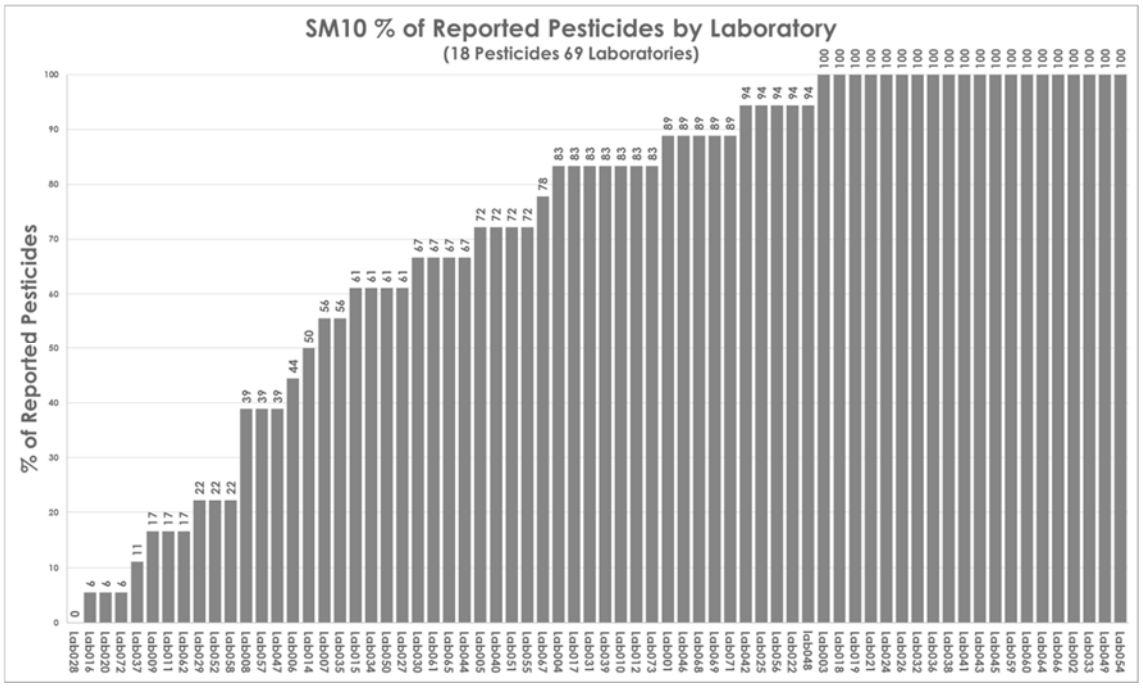
The bold line represents the robust mean



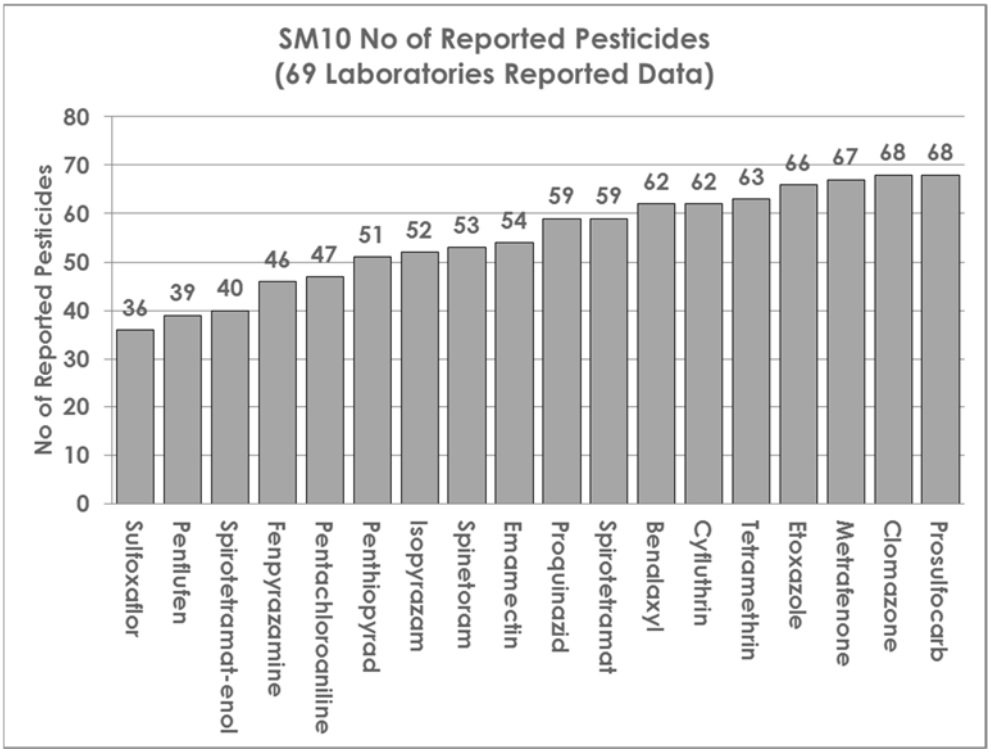


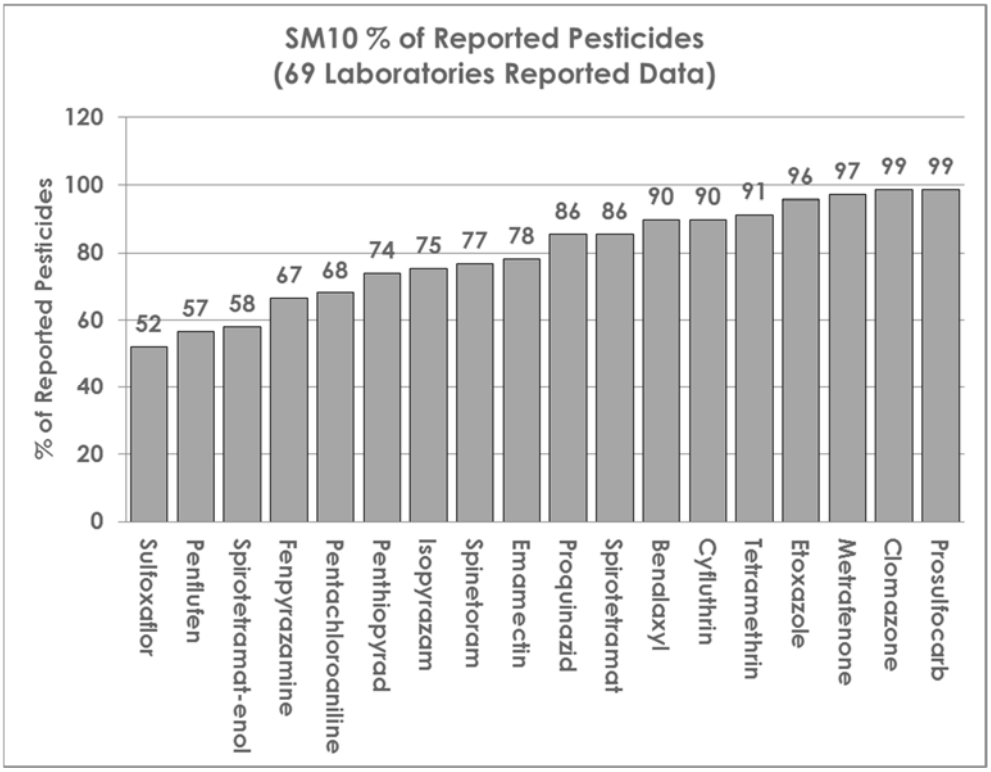



SM10 No of Reported Pesticides by Laboratory
(18 Pesticides 69 Laboratories)
No of Reported Pesticides

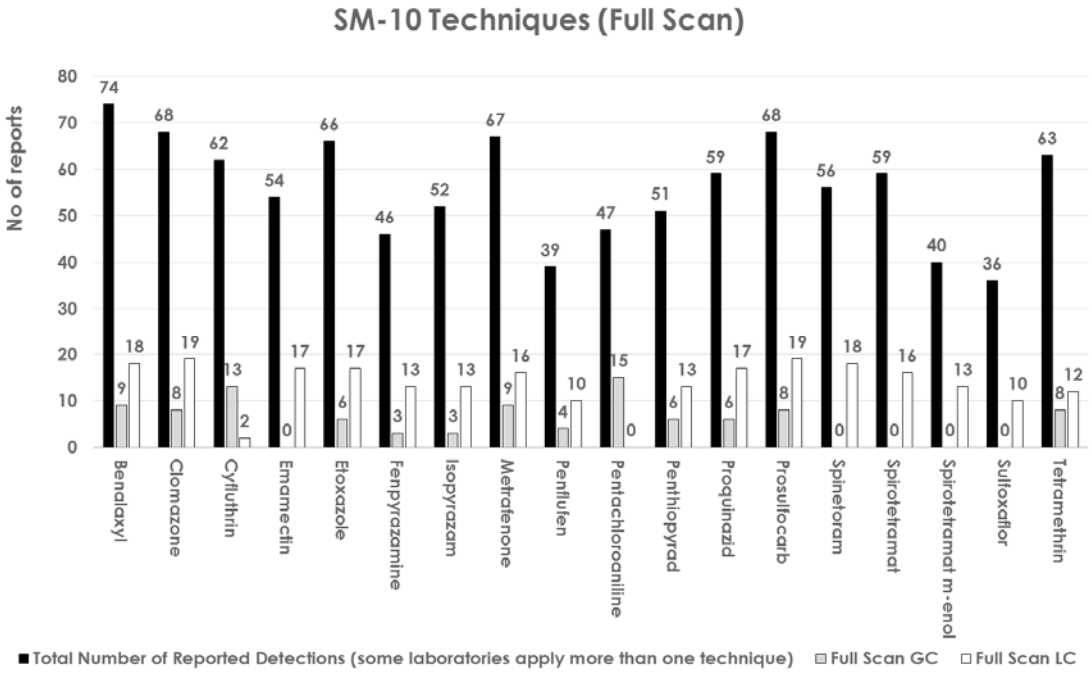

SM10 % of Reported Pesticides by Laboratory
(18 Pesticides 69 Laboratories)
% of Reported Pesticides

APPENDIX 2. Graphical Representations





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



ANNEX 1. List of Laboratories that reported results in EUPT-FV-SM10.

COUNTRY	LABORATORY NAME	CITY
AUSTRIA	DEPARTMENT FOR PESTICIDE AND FOOD ANALYTICS (PLMA)	INNSBRUCK
BELGIUM	BELGIAN SCIENTIFIC INSTITUTE OF PUBLIC HEALTH	BRUSSELS
BELGIUM	LOVAP	GEEL
BELGIUM	PRIMORIS (PHYTOLAB)	GENT - ZWIJNAARDE
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)	SAN JOSÉ
CROATIA	SAMPLE CONTROL - PESTICIDE LAB	ZAGREB
CZECH REPUBLIC	CAFIA - PESTICIDE LAB	PRAHA
CZECH REPUBLIC	VSCHT (PRAHA) - PESTICIDE LAB	PRAHA
DENMARK	DTU, NATIONAL FOOD INSTITUTE	LYNGBY
ESTONIA	PESTICIDE LAB (SAKU)	SAKU
FINLAND	FINNISH CUSTOMS LABORATORY	ESPOO
FRANCE	ANSES -LSAL	MAISONS ALFORT CEDEX
FRANCE	CERECO	GARONS
FRANCE	INOVALYS LE MANS - PESTICIDE LAB	LE MANS
FRANCE	SCL - MASSY CEDEX	MASSY CEDEX
FRANCE	SCL (MONTPELLIER)	MONTPELLIER
GERMANY	ANALYTICA ALIMENTARIA KLEINMACHNOW	KLEINMANCHONW
GERMANY	EUROFINS - PESTICIDE LAB	HAMBURG
GERMANY	GALAB LABORATORIES GMBH	HAMBURG
GERMANY	ILAU GMBH - PESTICIDE LAB	ANZING
GERMANY	LABOR MANG - PESTICIDE LAB	FRANKFURT
GERMANY	LAVES - PESTICIDE LAB	OLDENBURG
GERMANY	LTZ AUGUSTENBERG - ORGANIC CHEMISTRY	KARLSRUHE
GERMANY	LUA SACHSEN - PESTICIDE LAB	DRESDEN
GERMANY	PESTICIDE LAB	ERLANGEN
GERMANY	PESTICIDE LAB	TEGERNHEIM
GREECE	BPI - PESTICIDE LAB	KIFISSIA
HUNGARY	NFCSO - PESTICIDE LAB	VELENCE
HUNGARY	NFCSO PESTICIDE LAB	HÓDMEZOVÁSÁRHELY

ANNEX 1. List of Laboratories that participate in EUPT-FV-SM10

COUNTRY	LABORATORY NAME	CITY
HUNGARY	NFCSO PESTICIDE LAB	MISKOLC
HUNGARY	NFCSO PESTICIDE LAB	SZOLNOK
IRELAND	PESTICIDE LAB (CO. KILDARE)	CO. KILDARE
ITALY	APPA BOLZANO - PESTICIDE LAB	BOLZANO
ITALY	APPA-SL TRENTO - PESTICIDE LAB	TRENTO
ITALY	ARPA FVG - PESTICIDE LAB	UDINE
ITALY	ARPA VENETO	VERONA
ITALY	ATS MILANO - LABORATORIO DI PREVENZIONE	MILANO
ITALY	IZSAM - PESTICIDE LAB	TERAMO
KENYA	SGS KENYA LTD	MOMBASA
LATVIA	BIOR (RIGA) - PESTICIDE LAB	RIGA
LITHUANIA	NMVRVI - PESTICIDE LAB	VILNIUS
NORWAY	NIBIO - DEPARTMENT OF PESTICIDE CHEMISTRY	ÅS
SERBIA	CENTER FOR FOOD ANALYSIS	BELGRADE
SLOVENIJA	PESTICIDE LAB - MARIBOR	MARIBOR
SPAIN	ANALYTICA ALIMENTARIA GMBH - PESTICIDE LAB	ALMERIA
SPAIN	ECOSUR - PESTICIDE LAB	LORQUÍ (MURCIA)
SPAIN	EUROFINS AGRIQ SPAIN - PESTICIDE LAB	ALMERIA
SPAIN	LAB. AGROALIMENTARIO-LAYSA	EL PALMAR (MURCIA)
SPAIN	LABORATORI AGROALIMENTARI DE CABRILS - LAC	CABRILS
SPAIN	LABORATORIO AGROALIMENTARIO	VALENCIA
SPAIN	LABORATORIO AGROALIMENTARIO DE EXTREMADURA	CÁCERES
SPAIN	LABORATORIO AGROALIMENTARIO DE GRANADA	GRANADA
SPAIN	LABORATORIO ALHÓNDIGA LA UNIÓN	EL EJIDO
SPAIN	LABORATORIO DE SALUD PÚBLICA DE CUENCA	CUENCA
SPAIN	LABORATORIO DE SALUD PÚBLICA DE GALICIA	LUGO
SPAIN	LARAGA CASTILLA LA MANCHA	TOLEDO
SPAIN	NATIONAL CENTER FOR TECHNOLOGY AND FOOD SAFETY	SAN ADRIÁN (NAVARRA)
SWEDEN	EUROFINS FOOD & FEED - PESTICIDE LAB	LIDKÖPING
SWEDEN	SCIENCE DEPARTMENT - CHEMISTRY DIVISION 1	UPPSALA
SWITZERLAND	PESTICIDE LAB (ZÜRICH)	ZÜRICH
SWITZERLAND	SCAV - PESTICIDE LAB	GENEVE
THE NETHERLANDS	GROEN AGRO CONTROL - PESTICIDE LAB	DELFGAUW
THE NETHERLANDS	NVWA - PESTICIDE LAB	WAGENINGEN

ANNEX 1. List of Laboratories that participate in EUPT-FV-SM10.

COUNTRY	LABORATORY NAME	CITY
THE NETHERLANDS	RIKILT – WAGENINGEN UNIVERSITY & RESEARCH	WAGENINGEN
TURKEY	ÖZEL MSM GIDA KONTROL LABORATUVARI VE DAN. HIZ. TIC. A.S.	MERSIN
UNITED KINGDOM	FERA - PESTICIDE LAB	YORK