EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUIT AND VEGETABLES.

SCREENING METHODS 15 (EUPT-FV-SM15)

Pesticide Residues in wheat kernels

Final Report

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CONTENT

EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUIT AND VEGETABLES. SCREENING METHODS 15

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 625/2017[2] 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 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, given that the EURL-FV already organises the Proficiency Tests for quantitative multi-residue pesticide analysis (EUPT-FVs) 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, are invited to take part.

DG-SANTE has 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.

[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 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 objective of the EURL-FV screening proficiency tests is for laboratories to be able to use mass-spectrometry-based screening methods routinely, following validation. This is in line with Document No SANTE/11312/2021 "Analytical quality control and method validation procedures for pesticide residues and analysis in food and feed".

This EUPT-FV-SM15 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 ITEM

2.1 Preparation of the treated test item.

This proficiency test is based on the pesticide-residue analysis of wheat kernels. Wheat kernels were supplied by EURL-CF (Copenhagen, Denmark).

The pesticides used to spike the wheat kernels were decided by the Quality Control Group. No target pesticide list was provided to participants.

For the preparation of the EUPT-SM15 test item, wheat kernels were spiked with the pesticides applied as analytical standards. The mixture was homogenised and packed in polyethylene bottles that had previously been coded. The bottles were sealed and stored in a freezer at about -20 °C prior to distribution to participants. Table 2.1.1 shows the pesticide residues present in the EUPT-SM15 test item.

Table 2.1.1 Pesticides present in the test item.

Spiked Pesticides	
Carbaryl	Flufenacet
Carbofuran	Fluroxypyr
Chlorpyrifos	Metazachlor
Chlorpyrifos methyl	Pirimiphos-methyl
Cypermethrin (sum)	Tebuconazole
Deltamethrin	Tetramethrin
Fipronil	Trinexapac-Ethyl

ONLY those compounds above 0.01 mg/kg have been considered for the evaluation of this proficiency test. All of them are above this concentration as can be seen in section 2.2.

2.2 Homogeneity and stability tests.

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 2.2.1 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. All the compounds passed the homogeneity test.

Table 2.2.1 shows the statistical analyses for each of the pesticides used to treat the sample. All pesticides passed this test.

Table 2.2.1 Statistical evaluation of the homogeneity test data (n = 20 analyses)

Pesticide	Mean Conc. (mg/Kg)	Ss^2	c	$Ss^2 < c$ Pass/Fail
Carbaryl	0.103	0.00005	0.00017	Pass
Carbofuran	0.182	0.00004	0.00023	Pass
Chlorpyrifos	0.189	0.00004	0.00021	Pass
Chlorpyrifos methyl	0.113	0	0.00006	Pass
Cypermethrin (sum)	0.064	0	0.00033	Pass
Deltamethrin	0.130	0.00001	0.00014	Pass
Fipronil	0.077	0.00009	0.00013	Pass
Flufenacet	0.067	0.00001	0.00005	Pass
Fluroxypyr	0.135	0	0.00019	Pass
Metazachlor	0.059	0	0.00024	Pass
Pirimiphos-methyl	0.085	0	0.00019	Pass
Tebuconazole	0.055	0	0.00018	Pass
Tetramethrin	0.093	0	0.00013	Pass
Trinexapac-Ethyl	0.057	0.00002	0.00022	Pass

The stability tests were also carried out by the EURL-FV laboratory at the University of Almería. The tests were performed according to ISO 13528:2015. Shortly before the test item shipment, three bottles that were stored in the freezer at -20 °C were chosen randomly and stored in a -80 °C freezer (Day 1). After the deadline for reporting results, those three bottles stored at -80 °C, together with three other bottles that were stored in the freezer at -20 °C and were chosen randomly (Day 2) were analysed by duplicate.

A pesticide was considered to be adequately stable if $|x1 - yi| \leq 0.3 \times \sigma$, where x1 is the mean value of the Day 1 stability test, yi the mean value of the Day 2 stability test and σ the standard deviation used for proficiency assessment (typically 25 % of the assigned value).

[3] ISO 13528:2015, Statistical methods for use in proficiency testing by interlaboratory comparison, International Organization for Standardization

The individual results are given in Table 2.2.2. This test did not show any significant decrease in the pesticide concentrations with time. This demonstrates that, for the duration of the proficiency test, and provided that the storage conditions prescribed were followed, the time elapsed until the participants performed the analysis would not have influenced their results.

Table 2.2.2
Statistical test for analytical precision and to demonstrate results stability after the interval of time-elapse between the shipment of the test item and the deadline for reporting of results.

Pesticide	Day 1(mg/Kg)							Day 2(mg/Kg)							(M2 – M1)	M2-M1 ≤ 0.3*σ
	Sample 18_A	Sample 18_B	Sample 33_A	Sample 33_B	Sample 50_A	Sample 50_B	Mean 1	Sample 13_A	Sample 13_B	Sample 06_A	Sample 06_B	Sample 12_A	Sample 12_B	Mean 2		
Carbaryl	0.103	0.110	0.082	0.080	0.096	0.096	0.095	0.100	0.101	0.105	0.103	0.090	0.089	0.098	0.004	Pass
Carbofuran	0.200	0.195	0.179	0.180	0.197	0.190	0.190	0.189	0.185	0.200	0.198	0.185	0.180	0.190	-0.001	Pass
Chlorpyrifos	0.185	0.185	0.195	0.192	0.189	0.190	0.189	0.175	0.178	0.180	0.182	0.190	0.192	0.183	-0.007	Pass
Chlorpyrifos methyl	0.110	0.108	0.112	0.110	0.108	0.108	0.109	0.119	0.118	0.105	0.106	0.100	0.120	0.111	0.002	Pass
Cypermethrin (sum)	0.065	0.068	0.070	0.070	0.068	0.065	0.068	0.070	0.068	0.074	0.072	0.070	0.075	0.072	0.004	Pass
Deltamethrin	0.125	0.128	0.132	0.133	0.127	0.127	0.129	0.132	0.134	0.129	0.128	0.135	0.140	0.133	0.004	Pass
Fipronil	0.080	0.081	0.079	0.082	0.073	0.080	0.079	0.082	0.080	0.076	0.075	0.088	0.086	0.081	0.002	Pass
Flufenacet	0.070	0.066	0.066	0.062	0.072	0.077	0.069	0.075	0.072	0.068	0.072	0.064	0.068	0.070	0.001	Pass
Fluroxypyr	0.150	0.155	0.145	0.144	0.135	0.138	0.145	0.158	0.150	0.136	0.135	0.129	0.137	0.141	-0.004	Pass
Metazachlor	0.065	0.060	0.068	0.070	0.066	0.064	0.066	0.060	0.062	0.066	0.064	0.060	0.060	0.062	-0.004	Pass
Pirimiphos-methyl	0.090	0.092	0.078	0.075	0.080	0.082	0.083	0.083	0.082	0.079	0.081	0.090	0.086	0.084	0.001	Pass
Tebuconazole	0.070	0.072	0.068	0.070	0.066	0.068	0.069	0.075	0.074	0.066	0.062	0.059	0.062	0.066	-0.003	Pass
Tetramethrin	0.100	0.095	0.103	0.108	0.098	0.099	0.100	0.099	0.104	0.104	0.107	0.102	0.100	0.103	0.002	Pass
Trinexapac-Ethyl	0.062	0.064	0.064	0.065	0.059	0.062	0.063	0.060	0.058	0.055	0.057	0.062	0.060	0.059	-0.004	Pass

Moreover, regarding the stability of the sample arriving not completely frozen, a duplicate analysis of three bottles reproducing the delivery conditions that the samples experienced for 48 hours was performed (Day 3). Laboratories could therefore be sufficiently confident in accepting the treated test item even if it was not completely frozen. All the pesticides passed this second stability test. Results for this 48-hour stability test are indicated in Table 2.2.3.

Table 2.2.3 Statistical test for analytical precision and to demonstrate stability for the 48-hour time-elapse interval.

Pesticide	Day 1(mg/Kg)							Day 2(mg/Kg)							(M2 – M1)	M2-M1 ≤ 0.3*σ
	Sample 18_A	Sample 18_B	Sample 33_A	Sample 33_B	Sample 50_A	Sample 50_B	Mean 1	Sample 07_A	Sample 07_B	Sample 20_A	Sample 20_B	Sample 55_A	Sample 55_B	Sample 18_A		
Carbaryl	0.103	0.110	0.082	0.080	0.096	0.096	0.095	0.089	0.092	0.100	0.101	0.095	0.099	0.096	0.002	Pass
Carbofuran	0.200	0.195	0.179	0.180	0.197	0.190	0.190	0.192	0.185	0.190	0.187	0.200	0.203	0.193	0.003	Pass
Chlorpyrifos	0.185	0.185	0.195	0.192	0.189	0.190	0.189	0.189	0.190	0.200	0.198	0.185	0.180	0.190	0.001	Pass
Chlorpyrifos methyl	0.110	0.108	0.112	0.110	0.108	0.108	0.109	0.109	0.110	0.130	0.129	0.105	0.107	0.115	0.006	Pass
Cypermethrin (sum)	0.065	0.068	0.070	0.070	0.068	0.065	0.068	0.075	0.072	0.072	0.064	0.072	0.077	0.072	0.004	Pass
Deltamethrin	0.125	0.128	0.132	0.133	0.127	0.127	0.129	0.119	0.118	0.122	0.124	0.120	0.120	0.121	-0.008	Pass
Fipronil	0.080	0.081	0.079	0.082	0.073	0.080	0.079	0.068	0.072	0.085	0.080	0.075	0.079	0.077	-0.003	Pass
Flufenacet	0.070	0.066	0.066	0.062	0.072	0.077	0.069	0.065	0.062	0.080	0.078	0.065	0.067	0.070	0.001	Pass
Fluroxypyr	0.150	0.155	0.145	0.144	0.135	0.138	0.145	0.148	0.150	0.129	0.130	0.132	0.130	0.137	-0.008	Pass
Metazachlor	0.065	0.060	0.068	0.070	0.066	0.064	0.066	0.062	0.060	0.070	0.069	0.055	0.060	0.063	-0.003	Pass
Pirimiphos-methyl	0.090	0.092	0.078	0.075	0.080	0.082	0.083	0.079	0.082	0.080	0.085	0.079	0.075	0.080	-0.003	Pass
Tebuconazole	0.070	0.072	0.068	0.070	0.066	0.068	0.069	0.060	0.056	0.069	0.070	0.070	0.075	0.067	-0.002	Pass
Tetramethrin	0.100	0.095	0.103	0.108	0.098	0.099	0.100	0.105	0.102	0.108	0.110	0.096	0.099	0.103	0.003	Pass
Trinexapac-Ethyl	0.062	0.064	0.064	0.065	0.059	0.062	0.063	0.055	0.062	0.060	0.064	0.058	0.055	0.059	-0.004	Pass

2.3 Distribution of test item and protocol to participants

Approximately 200 g of treated wheat kernels homogenate were shipped to participants on 6th March 2023. 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 HRMS (High Resolution Mass Spectrometry). 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-SM15 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 (on a voluntary basis) 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-five laboratories agreed to participate in this proficiency test on screening methods. Sixty laboratories submitted results on time. All results reported by the participants are given in Appendix 1. Graphical representations of the results reported are shown in Appendix 2. Details of the methods used are provided in Appendix 3 (available on the EUPT-FV-SM15 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 4.1.1.

Table 4.1.1 Summary of Reported Results.

Pesticide	Reported		Not Reported	
	No. of laboratories	% of Laboratories#	No. of laboratories	% of laboratories *
Carbaryl	56	93	4	7
Carbofuran	58	97	2	3
Chlorpyrifos	58	97	2	3
Chlorpyrifos methyl	59	98	1	2
Cypermethrin (sum)	55	92	5	8
Deltamethrin	57	95	3	5
Fipronil	56	93	4	7
Flufenacet	51	85	9	15
Fluroxypyr	29	48	31	52
Metazachlor	51	85	9	15
Pirimiphos-methyl	58	97	2	3
Tebuconazole	58	97	2	3
Tetramethrin	54	90	6	10
Trinexapac-Ethyl	30	50	30	50

#The % of laboratories is calculated based on the total number of laboratories submitting results (60 laboratories).

In this EUPT-FV-SM15, 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 4.1.2 shows the average concentration from the homogeneity test, the robust mean of the estimated concentrations reported by EU/EFTA laboratories, the number of concentration results reported and the dispersion of the concentration results reported.

NOTE: All compounds reported by the laboratories above 0.01 mg/kg and present in the sample are shown in Table 4.1.2.

Table 4.1.2 Robust mean values and CVs (%) for all present pesticides reported.

Pesticides°	No of Conc. Reported	No of Conc. Reported by EU/EFTA labs	Conc. Homogeneity Test (mg/kg)	Robust mean (mg/kg)	CV (%)
Carbaryl	56	53	0.103	0.101	27.0
Carbofuran	58	55	0.182	0.170	29.2
Chlorpyrifos	62	59	0.189	0.182	24.1
Chlorpyrifos methyl	62	58	0.113	0.109	32.2
Cypermethrin (sum)	55	52	0.064	0.058	33.5
Deltamethrin	59	55	0.130	0.116	33.1
Fipronil	61	57	0.077	0.066	32.9
Flufenacet	51	49	0.067	0.063	26.1
Fluroxypyr	27	27	0.135	0.110	37.4
Metazachlor	49	46	0.059	0.057	20.7
Pirimiphos-methyl	62	59	0.085	0.079	25.8
Tebuconazole	59	55	0.055	0.052	24.9
Tetramethrin	52	49	0.093	0.091	22.8
Trinexapac-Ethyl	26	25	0.057	0.059	32.4

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

Chlormequat was reported by 10 laboratories, and it was present in the sample but as it has to be analysed using single residue methods, the Advisory Group decided not to include it for the evaluation of the participating laboratories.

4.1.1 Other Reported Compounds

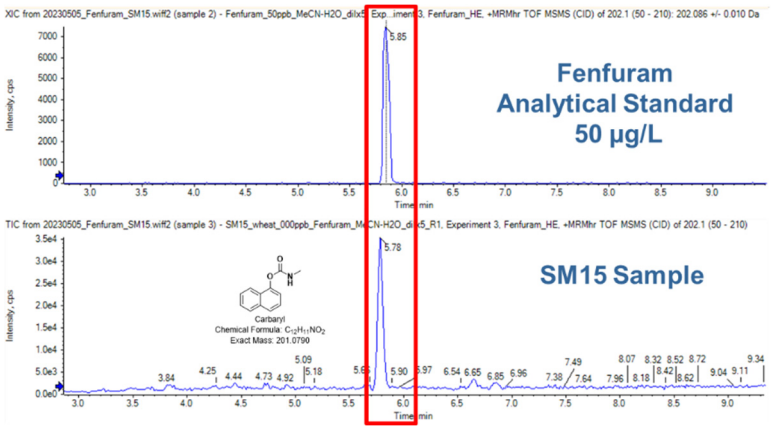
Some laboratories reported additional compounds to those present in the test item. Table 4.1.1.1 shows the reported compounds and the concentrations as reported by the laboratories. Some of them were reported below 0.01 mg/kg or not quantified. The reported compounds at or above 0.01 mg/kg are marked in light blue.

Table 4.1.1.1 Other reported compounds.

Laboratory Code	Other reported compounds	Concentration Reported (mg/kg)
LAB011	((8Z,11Z)-2-Heptadeca-8,11-dienyl)-4,5-dihydro-oxazole	
LAB058	4,4'-dichlorobenzophenone	0.001
LAB017	8-hydroxyquinoline	
Lab053		0.058
LAB004	Acephate	0.050
LAB041	Azobenzene	0.050
LAB011	Benzoic acid	
LAB011	Benzoic acid methyl ester	
LAB058	BHC	0.008
LAB058	Biphenyl	0.004
LAB051	Bromide	0.453
LAB011	Canolol	
LAB058	Captafol	0.240
LAB028	Captan	0.248
LAB017	Carvone	
LAB058	DDE	0.001
LAB058	DDT	0.001
LAB011	Dicyclohexylphtalate	
LAB019	Dinobuton	0.039
LAB058	Diphenamid	0.002
LAB017	Empenthrin	
LAB058	Endrin ketone	0.001
Lab053	Ethylene oxide	<0.01
LAB004	Fenbuconazole	0.013
LAB017	Fenclorim	
LAB004	Fenfuram	0.011
LAB007		0.062
LAB012		0.162
LAB011	Ferulic acid	
LAB035	Fluopyram	0.005
LAB033	Formetanate	0.130
LAB034		0.141 ± 0.071
LAB011	Glycerol tricaprylate (Tricaprylin)	
LAB018	Glyphosate	0.064
Lab053		0.059
Lab053	Indole-3-acetic acid	0.033
LAB056		
LAB011	Methoprene	
LAB011	Methyl ferulate	
Lab010	Parathion-methyl	0.071
LAB030		0.049
LAB058	Pendimethalin	0.002
LAB058	Permethrin	0.001
LAB017	Picaridin	
LAB058	Propisochlor	0.080
LAB011	Pyrimidine	
LAB056	Pyrimitate	
LAB044	Sulcotriona	
LAB011	TMA C16	
LAB011	Trans synapyl alcohol	
LAB058	Tryciclazole	0.300

Fenfuram was the only compound not present in the test item that was reported by 3 laboratories. This may be due to two circumstances: (i) carbaryl was present in the test item, with which fenfuram shares an identical chemical formula, and (ii) there may be natural products in the test material that, upon fragmentation, may give rise to fragment ions common to fenfuram. Given the proximity

of the elution times (and potentially, co-elution) for fenfuram and carbaryl, it is possible that participants may have observed a positive signal for fenfuram due to the aforementioned reasons.



4.2 Assessment of laboratory performance.

Laboratory performance was assessed with the number of results reported by each laboratory. Table 4.2.1 classifies the laboratories according to the number of pesticides reported.

Table 4.2.1 Classification of laboratories according to the number of pesticides reported.

Laboratory Code	Number of Present Pesticides Reported (14 Evaluated Pesticides)	% of Present Pesticides Reported (14 Evaluated Pesticides)	Other Reported Pesticides Not Confirmed by the Organiser
LAB016	14	100	
LAB020	14	100	
LAB005	14	100	
LAB006	14	100	
LAB015	14	100	
LAB021	14	100	
LAB038	14	100	
LAB047	14	100	
LAB048	14	100	
LAB054	14	100	
LAB064	14	100	
LAB007	14	100	1
LAB019	14	100	1
LAB018	14	100	1
LAB028	14	100	1
LAB051	14	100	1
LAB053	14	100	4
LAB029	13	93	
LAB008	13	93	
LAB001	13	93	
LAB002	13	93	
LAB003	13	93	
LAB013	13	93	
LAB014	13	93	
LAB036	13	93	
LAB040	13	93	

Laboratory Code	Number of Present Pesticides Reported (14 Evaluated Pesticides)	% of Present Pesticides Reported (14 Evaluated Pesticides)	Other Reported Pesticides Not Confirmed by the Organiser
LAB043	13	93	
LAB045	13	93	
LAB059	13	93	
LAB033	13	93	1
LAB010	13	93	1
LAB030	13	93	1
LAB035	13	93	1
LAB056	13	93	2
LAB017	13	93	5
LAB024	12	86	
LAB025	12	86	
LAB026	12	86	
LAB031	12	86	
LAB046	12	86	
LAB050	12	86	
LAB052	12	86	
LAB062	12	86	
LAB063	12	86	
LAB044	12	86	1
LAB012	12	86	1
LAB041	12	86	1
LAB004	12	86	3
LAB032	11	79	
LAB009	11	79	
LAB055	11	79	
LAB065	11	79	
LAB057	10	71	
LAB011	10	71	11
LAB037	8	57	
LAB039	8	57	
LAB049	6	43	
LAB058	6	43	12
LAB042	5	36	
LAB034	5	36	1

The extraction methods used by the laboratories, the chromatographic techniques, detectors, instrumentation, etc… are detailed in Appendix 3 (available only on the EUPT-FV-SM15 webpage).

Table 4.2.2 shows the same data shown in Table 4.2.1 but classified by laboratory code.

Table 4.2.2 Results classified by laboratory code

Laboratory Code	Number of Present Pesticides Reported (14 Evaluated Pesticides)	% of Present Pesticides Reported (14 Evaluated Pesticides)	Other Reported Pesticides Not Confirmed by the Organiser
LAB001	13	93	
LAB002	13	93	
LAB003	13	93	

Laboratory Code	Number of Present Pesticides Reported (14 Evaluated Pesticides)	% of Present Pesticides Reported (14 Evaluated Pesticides)	Other Reported Pesticides Not Confirmed by the Organiser
LAB004	12	86	3
LAB005	14	100	
LAB006	14	100	
LAB007	14	100	1
LAB008	13	93	
LAB009	11	79	
LAB010	13	93	1
LAB011	10	71	11
LAB012	12	86	1
LAB013	13	93	
LAB014	13	93	
LAB015	14	100	
LAB016	14	100	
LAB017	13	93	5
LAB018	14	100	1
LAB019	14	100	1
LAB020	14	100	
LAB021	14	100	
LAB024	12	86	
LAB025	12	86	
LAB026	12	86	
LAB028	14	100	1
LAB029	13	93	
LAB030	13	93	1
LAB031	12	86	
LAB032	11	79	
LAB033	13	93	1
LAB034	5	36	1
LAB035	13	93	1
LAB036	13	93	
LAB037	8	57	
LAB038	14	100	
LAB039	8	57	
LAB040	13	93	
LAB041	12	86	1
LAB042	5	36	
LAB043	13	93	
LAB044	12	86	1
LAB045	13	93	
LAB046	12	86	
LAB047	14	100	
LAB048	14	100	
LAB049	6	43	
LAB050	12	86	
LAB051	14	100	1
LAB052	12	86	
LAB053	14	100	4

Laboratory Code	Number of Present Pesticides Reported (14 Evaluated Pesticides)	% of Present Pesticides Reported (14 Evaluated Pesticides)	Other Reported Pesticides Not Confirmed by the Organiser
LAB054	14	100	
LAB055	11	79	
LAB056	13	93	2
LAB057	10	71	
LAB058	6	43	12
LAB059	13	93	
LAB062	12	86	
LAB063	12	86	
LAB064	14	100	
LAB065	11	79	

Table 4.2.3 is a summary of the chromatographic techniques used for each pesticide. A graphical representation of this information is shown in Appendix 2.

Table 4.2.3 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/AIF GC	LC	Full Scan/AIF LC
Carbaryl	56	62	7	4	55	11
Carbofuran	58	63	6	5	57	11
Chlorpyrifos	58	69	49	13	19	5
Chlorpyrifos methyl	59	67	52	15	15	4
Cypermethrin (sum)	55	61	56	12	5	2
Deltamethrin	57	64	50	11	14	5
Fipronil	56	66	32	9	34	8
Flufenacet	51	57	14	4	43	10
Fluroxypyr	29	29			29	5
Metazachlor	51	57	23	9	34	8
Pirimiphos-methyl	58	68	38	10	30	9
Tebuconazole	58	63	22	5	41	9
Tetramethrin	54	60	31	9	29	9
Trinexapac-Ethyl	30	31	4	3	27	7

*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.

5. CONCLUSIONS

Sixty-five laboratories agreed to participate in this proficiency test on screening methods. Sixty laboratories submitted results on time.

Twenty EU Member States, 2 EFTA countries (Norway and Switzerland) and four non-EU/EFTA countries (China, Costa Rica, Kenya and Peru) participated in this European Union Proficiency Test.

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-SM15 webpage, not in the printed version). The laboratories that agreed to participate are listed in Annex 1.

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

(without the other reported compounds) were 817; 384 were made by GC and 432 by LC. 26% of the detections were made using full-scan or all ion fragmentation (AIF)(109 by GC-full scan/AIF techniques and 103 by LC-full scan/AIF techniques). 27% of the laboratories reported their results using HRMS and 731 of the results were reported indicating a concentration value (89% of the total results).

Seventeen laboratories were able to detect all 14 pesticides present in the test item. Only four laboratories detected less than 50% of the pesticides present. Ninety percent of the laboratories (54 laboratories) that reported results were able to detect more than 70% of the evaluated pesticides.

Two other compounds different from the present pesticides were reported by three or more laboratories at concentrations above 0.01 mg/mg, Fenfuram and Chlormequat. Chlormequat was present but it was not evaluated because it is a SRM pesticide. Fenfuram was a potential false positive result due to the presence of carbaryl in the test item.

Twenty-four participants reported 43 different compounds not evaluated in this proficiency test.

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/11312/2021

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.

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8. ACKNOWLEDGEMENTS

The Organiser is grateful to the European Commission for funding this 15th 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 the University of Almería for the use of their facilities and to the EURL for Cereals and Feeding Stuff (DTU National Food Institute) for providing the wheat kernels and their knowledge about this matrix.

APPENDIX 1. Results

Table AP1.1. Reported pesticides

Laboratory Code Total No of Reporting Laboratories=60	Evaluated Pesticides (14) R: Reported Pesticide														Reported Pesticides by Laboratory	% Reported Pesticides by Laboratory
	Carbaryl	Carbofuran	Chlorpyrifos	Chlorpyrifos methyl	Cypermethrin	Deltamethrin	Fipronil	Flufenacet	Fluroxypyr	Metazachlor	Pirimiphos methyl	Tebuconazole	Tetramethrin	Trinexapac ethyl		
LAB001	R	R	R	R	R	R	R	R	R		R	R	R	R	13	93
LAB002	R	R	R	R	R	R	R	R	R		R	R	R	R	13	93
LAB003	R	R	R	R	R	R	R	R	R	R	R	R	R		13	93
LAB004	R	R	R	R	R	R	R	R		R	R	R	R		12	86
LAB005	R	R	R	R	R	R	R	R	R	R	R	R	R	R	14	100
LAB006	R	R	R	R	R	R	R	R	R	R	R	R	R	R	14	100
LAB007	R	R	R	R	R	R	R	R	R	R	R	R	R	R	14	100
LAB008	R	R	R	R	R	R	R	R	R	R	R	R	R		13	93
LAB009	R	R	R	R	R	R	R	R		R	R	R			11	79
LAB010	R	R	R	R	R	R	R	R		R	R	R	R	R	13	93
LAB011		R	R	R	R	R	R			R	R	R	R		10	71
LAB012	R	R	R	R	R	R	R	R		R	R	R	R		12	86
LAB013	R	R	R	R	R	R	R	R	R	R	R	R	R		13	93
LAB014	R	R	R	R	R	R	R	R	R	R	R	R	R		13	93
LAB015	R	R	R	R	R	R	R	R	R	R	R	R	R	R	14	100
LAB016	R	R	R	R	R	R	R	R	R	R	R	R	R	R	14	100
LAB017	R	R	R	R	R	R	R	R		R	R	R	R	R	13	93
LAB018	R	R	R	R	R	R	R	R	R	R	R	R	R	R	14	100
LAB019	R	R	R	R	R	R	R	R	R	R	R	R	R	R	14	100
LAB020	R	R	R	R	R	R	R	R	R	R	R	R	R	R	14	100
LAB021	R	R	R	R	R	R	R	R	R	R	R	R	R	R	14	100
LAB024	R	R	R	R	R	R	R	R		R	R	R	R		12	86
LAB025	R	R	R	R	R	R	R	R		R	R	R	R		12	86
LAB026	R	R	R	R	R	R	R	R		R	R	R	R		12	86
LAB028	R	R	R	R	R	R	R	R	R	R	R	R	R	R	14	100
LAB029	R	R	R	R	R	R	R	R		R	R	R	R	R	13	93
LAB030	R	R	R	R	R	R	R	R		R	R	R	R	R	13	93
LAB031	R	R	R	R	R	R		R		R	R	R	R	R	12	86
LAB032	R	R	R	R	R	R	R	R		R	R	R			11	79
LAB033	R	R	R	R	R	R	R	R	R	R	R	R	R		13	93
LAB034	R	R	R	R	R										5	36
LAB035	R	R	R	R	R	R	R	R	R	R	R	R	R		13	93
LAB036	R	R	R	R	R	R	R	R		R	R	R	R	R	13	93
LAB037	R	R	R	R	R		R				R	R			8	57
LAB038	R	R	R	R	R	R	R	R	R	R	R	R	R	R	14	100
LAB039	R	R	R	R		R	R				R	R			8	57
LAB040	R	R	R	R	R	R	R	R	R	R	R	R	R		13	93
LAB041	R	R	R	R	R	R	R	R		R	R	R	R		12	86
LAB042			R	R		R					R		R		5	36
LAB043	R	R	R	R	R	R	R	R		R	R	R	R	R	13	93
LAB044	R	R	R	R	R	R	R	R		R	R	R	R		12	86
LAB045	R	R	R	R	R	R	R	R		R	R	R	R	R	13	93
LAB046	R	R	R	R	R	R	R	R		R	R	R	R		12	86
LAB047	R	R	R	R	R	R	R	R	R	R	R	R	R	R	14	100
LAB048	R	R	R	R	R	R	R	R	R	R	R	R	R	R	14	100
LAB049		R					R	R			R	R	R		6	43
LAB050	R	R	R	R		R		R	R	R	R	R	R	R	12	86
LAB051	R	R	R	R	R	R	R	R	R	R	R	R	R	R	14	100
LAB052	R	R	R	R	R	R	R	R		R	R	R	R		12	86
LAB053	R	R	R	R	R	R	R	R	R	R	R	R	R	R	14	100
LAB054	R	R	R	R	R	R	R	R	R	R	R	R	R	R	14	100
LAB055	R	R	R	R		R	R	R		R	R	R	R		11	79
LAB056	R	R	R	R	R	R	R	R		R	R	R	R	R	13	93
LAB057	R	R	R	R	R	R	R				R	R	R		10	71
LAB058				R	R	R	R			R		R			6	43
LAB059	R	R	R	R	R	R	R	R		R	R	R	R	R	13	93
LAB062	R	R	R	R	R	R	R	R		R	R	R	R		12	86
LAB063	R	R	R	R	R	R	R		R	R	R	R	R		12	86
LAB064	R	R	R	R	R	R	R	R	R	R	R	R	R	R	14	100
LAB065	R	R	R	R	R	R	R		R		R	R	R		11	79
Reported Pesticides	**56**	**58**	**58**	**59**	**55**	**57**	**56**	**51**	**29**	**51**	**58**	**58**	**54**	**30**		
% of Reported Pesticides	**93**	**97**	**97**	**98**	**92**	**95**	**93**	**85**	**48**	**85**	**97**	**97**	**90**	**50**		

Table AP1.2. Estimated Concentrations Reported on a voluntary basis (only informative purposes)

NOTES: Not all the laboratories reporting results have reported estimated concentration values. Some Laboratories reported more than one result for the same pesticide.
All compounds reported by the laboratories above 0.01 mg/kg and present in the sample are shown in this table.

LABORATORY CODE	Reported Pesticide Concentratons													
	Carbaryl	Carbofuran	Chlorpyrifos	Chlorpyrifos methyl	Cypermethrin	Deltamethrin	Fipronil	Flufenacet	Fluroxypyr	Metazachlor	Pirimiphos methyl	Tebuconazole	Tetramethrin	Trinexapac ethyl
Concentration Homogeneity test (mg/kg)	0.103	0.182	0.189	0.113	0.064	0.13	0.077	0.067	0.135	0.059	0.085	0.055	0.093	0.057
Robust mean of estimated concentrations reported (mg/kg)	0.101	0.17	0.182	0.109	0.058	0.116	0.066	0.063	0.11	0.057	0.079	0.052	0.091	0.059
CV (%)	27.0	29.2	24.1	32.2	33.5	33.1	32.9	26.1	37.4	20.7	25.8	24.9	22.8	32.4
LAB001	0.113	0.165	0.157	0.089	0.057	0.085	0.049	0.057	0.061		0.064	0.066	0.092	0.059
LAB002	0.082	0.130	0.148	0.084	0.043	0.100	0.044	0.052	0.065		0.055	0.049	0.086	0.044
LAB003	0.122	0.184	0.204	0.169	0.037	0.133	0.090	0.072	0.174	0.062	0.081	0.062	0.120	
LAB004	0.016	0.366	0.142	0.076	0.042	0.069	0.060	0.042		0.051	0.059	0.049	0.079	
LAB005	0.109	0.258	0.219	0.110	0.055	0.120	0.097	0.112	0.124	0.063	0.086	0.051	0.096	
LAB006	0.086	0.148	0.191	0.089	0.062	0.141	0.073	0.065	0.148	0.049	0.087	0.051	0.092	0.070
LAB007	0.024	0.160 0.351	0.177 0.261	0.113 0.189	0.105 0.121	0.264 0.270	0.183 0.710	0.053 0.078	0.120	0.061 0.066	0.070 0.091	0.043 0.098	0.085 0.129 0.145	0.022 0.056
LAB008	0.088	0.170	0.200	0.096	0.070	0.130	0.060	0.060	0.080		0.079	0.052		
LAB009	0.110	0.180	0.180	0.091	0.045	0.110	0.045	0.073		0.063	0.077	0.060		
LAB010	0.077	0.098	0.189	0.111	0.065	0.101	0.096	0.058		0.043	0.074	0.046	0.087	0.085
LAB011		0.080	0.120	0.100	0.016	0.008	0.080				0.060	0.060		
LAB012	0.162	0.261	0.260	0.161		0.057	0.098				0.103	0.085		
LAB014	0.105	0.176	0.143	0.065	0.032	0.082	0.058	0.044	0.094	0.048	0.059	0.046	0.069	
LAB015	0.125	0.179	0.244	0.153	0.060	0.128	0.105	0.090	0.079	0.072	0.101	0.078	0.106	
LAB016	0.095	0.207	0.206 0.206	0.130 0.133	0.065 0.074	0.126 0.133	0.072	0.073	0.123	0.067	0.099	0.066	0.097 0.106	0.031
LAB017	0.094	0.145	0.221	0.122	0.044	0.155	0.075	0.077		0.059	0.097	0.049	0.090	
LAB018	0.150	0.232	0.209	0.114	0.073	0.136	0.096	0.061	0.081	0.063	0.088	0.054	0.093	0.075
LAB019	0.061 0.065	0.134	0.087 0.121	0.059 0.068	0.040	0.079	0.047 0.067	0.052	0.110	0.042 0.051	0.108	0.034	0.055 0.082	0.045
LAB020	0.087 0.122	0.147	0.174	0.099	0.071	0.131	0.057 0.063	0.055	0.139	0.052	0.065 0.076	0.048	0.082	0.029
LAB021	0.195	0.363	0.128	0.068	0.039	0.088	0.039	0.045	0.377	0.047	0.056	0.031	0.045	0.072

APPENDIX 1. Results

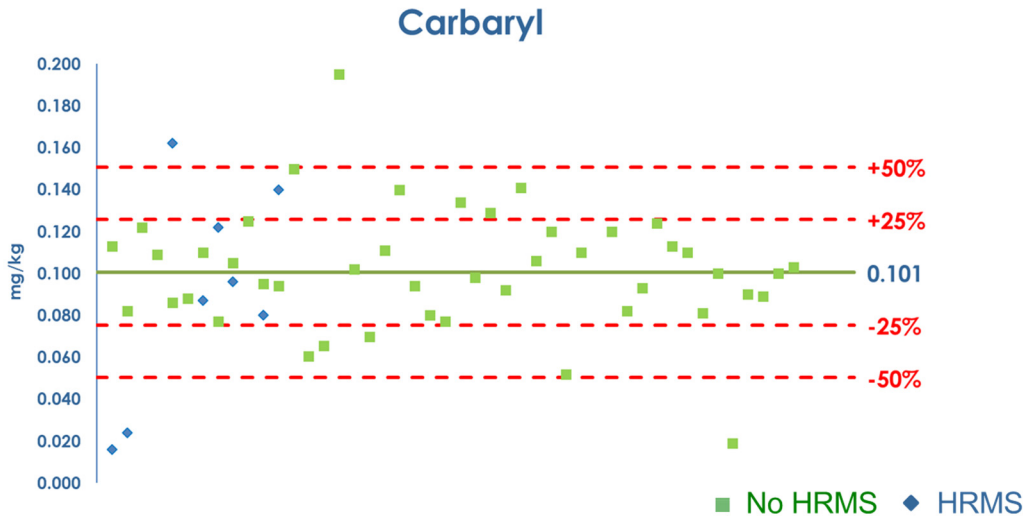
LABORATORY CODE	Reported Pesticide Concentratons													
	Carbaryl	Carbofuran	Chlorpyrifos	Chlorpyrifos methyl	Cypermethrin	Deltamethrin	Fipronil	Flufenacet	Fluroxypyr	Metazachlor	Pirimiphos methyl	Tebuconazole	Tetramethrin	Trinexapac ethyl
Concentration Homogeneity test (mg/kg)	0.103	0.182	0.189	0.113	0.064	0.13	0.077	0.067	0.135	0.059	0.085	0.055	0.093	0.057
Robust mean of estimated concentrations reported (mg/kg)	0.101	0.17	0.182	0.109	0.058	0.116	0.066	0.063	0.11	0.057	0.079	0.052	0.091	0.059
CV (%)	27.0	29.2	24.1	32.2	33.5	33.1	32.9	26.1	37.4	20.7	25.8	24.9	22.8	32.4
LAB024	0.102	0.137	0.213	0.162	0.069	0.100	0.089	0.065		0.059	0.085	0.051	0.087	
LAB025	0.070	0.135	0.134	0.072	0.027	0.093	0.057	0.038		0.036	0.060	0.031	0.083	
LAB026	0.111	0.214	0.269	0.160	0.080	0.156	0.088	0.078		0.078	0.102	0.063	0.108	
LAB028	0.140	0.191	0.110	0.090	0.045	0.149	0.080	0.089	0.078	0.081	0.093	0.068	0.140	0.035
LAB029	0.094 0.096	0.150 0.170	0.170 0.190	0.100 0.110	0.041 0.068	0.110 0.110	0.066 0.079	0.055 0.058			0.074 0.085	0.043 0.044	0.066 0.085	0.064
LAB030	0.080	0.150	0.160	0.051	0.043	0.086	0.043	0.056		0.045	0.059	0.051	0.071	0.061
LAB031	0.077	0.150	0.155	0.063	0.052	0.086		0.048		0.061	0.038	0.054	0.067	0.058
LAB032	0.134	0.195	0.180 0.182	0.114 0.116	0.042 0.051	0.093 0.099 0.103	0.063 0.062 0.066	0.066 0.072 0.083		0.060 0.063 0.066	0.079 0.085 0.091	0.052 0.058 0.067		
LAB033	0.098	0.120	0.175 0.180	0.083	0.066	0.139	0.022 0.069	0.072	0.120	0.057	0.047	0.042	0.084	
LAB034	0.129	0.124	0.142	0.150	0.086									
LAB035	0.080	0.130	0.200	0.080	0.050	0.100	0.030	0.050		0.050	0.050	0.040	0.060	
LAB036	0.092	0.185	0.184	0.096	0.053	0.096	0.053	0.071		0.057	0.079	0.043	0.081	0.070
LAB037	0.141	0.198	0.156	0.107	0.059		0.038				0.108	0.063		
LAB038	0.106	0.203	0.138	0.065	0.025	0.051	0.043	0.029	0.122	0.044	0.113	0.043	0.068	0.066
LAB039	0.120	0.570	0.227	0.138		0.154	0.093				0.140	0.064		
LAB040	0.052	0.120	0.160	0.140	0.068	0.160	0.071	0.047	0.110	0.036	0.090	0.056	0.110	
LAB041	0.110	0.130	0.180	0.110	0.060	0.064	0.064	0.083		0.059	0.082	0.058	0.091	
LAB042			0.249	0.173		0.126					0.082		0.095	
LAB044	0.120	0.260	0.230	0.125	0.070	0.120	0.086	0.730			0.088	0.057		
LAB045	0.082	0.203	0.184	0.110	0.040	0.060	0.063	0.070		0.063	0.082	0.052	0.090	0.079
LAB046	0.093	0.180	0.200	0.110	0.063	0.120	0.058	0.059		0.054	0.077	0.055	0.090	
LAB047	0.124	0.174	0.238	0.128	0.088	0.175	0.083	0.077	0.185	0.071	0.099	0.059	0.112	0.088
LAB048	0.113	0.205	0.182	0.109	0.077	0.149	0.058	0.060	0.253	0.055	0.094	0.047	0.112	0.071
LAB049		0.035					0.053	0.049			0.069	0.033	0.096	
LAB050	0.110	0.180	0.180	0.120		0.240		0.085	0.064	0.080	0.110	0.062	0.120	0.060
LAB051	0.081	0.093	0.177	0.110	0.055	0.120	0.052	0.049	0.083	0.057	0.069	0.033	0.087	0.058

LABORATORY CODE	Reported Pesticide Concentratons													
	Carbaryl	Carbofuran	Chlorpyrifos	Chlorpyrifos methyl	Cypermethrin	Deltamethrin	Fipronil	Flufenacet	Fluroxypyr	Metazachlor	Pirimiphos methyl	Tebuconazole	Tetramethrin	Trinexapac ethyl
Concentration Homogeneity test (mg/kg)	0.103	0.182	0.189	0.113	0.064	0.13	0.077	0.067	0.135	0.059	0.085	0.055	0.093	0.057
Robust mean of estimated concentrations reported (mg/kg)	0.101	0.17	0.182	0.109	0.058	0.116	0.066	0.063	0.11	0.057	0.079	0.052	0.091	0.059
CV (%)	27.0	29.2	24.1	32.2	33.5	33.1	32.9	26.1	37.4	20.7	25.8	24.9	22.8	32.4
LAB052	0.100	0.164	0.164	0.114	0.061	0.115	0.044	0.062		0.041	0.082	0.050	0.100	
LAB053	0.019	0.054	0.110	0.061	0.035	0.064	0.042	0.037	0.092	0.029	0.038	0.030	0.053	0.041
LAB054	0.140	0.210	0.250	0.170	0.079	0.119	0.130	0.110	0.044	0.089	0.120	0.074	0.146	0.050
LAB055	0.125	0.183	0.150	0.084		0.185	0.100	0.077		0.083	0.086	0.071	0.095	
LAB057	0.086	0.069	0.194	0.120	0.060	0.110	0.055				0.182	0.044	0.083	
LAB058				0.050	0.120	0.160	0.030			0.020		0.020		
LAB059	0.083	0.120	0.425	0.092	0.060	0.042	0.050	0.425		0.045	0.220	0.120	0.110	0.043
LAB062	0.090	0.170	0.190	0.100	0.060	0.090	0.060	0.060		0.060	0.070	0.040	0.075	
LAB063	0.089	0.160	0.120	0.069	0.088	0.180	0.044		0.100	0.049	0.058	0.035	0.073	
LAB064	0.100	0.140	0.244	0.148	0.088	0.170	0.070	0.074	0.130	0.064	0.104	0.073	0.114	0.070
LAB065	0.103	0.139	0.166	0.105	0.060	0.130	0.085		0.117		0.071	0.051	0.116	

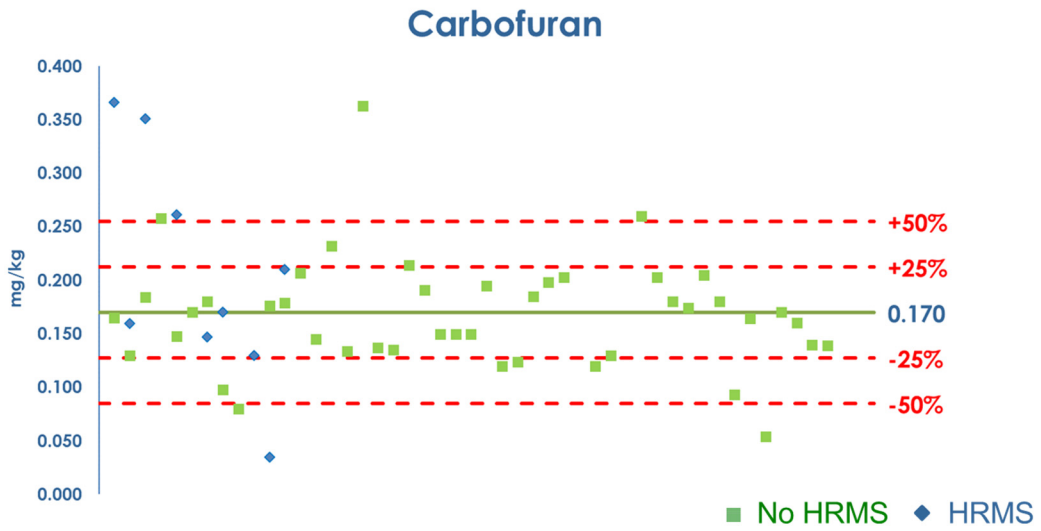
APPENDIX 2. Graphical Representations

The green line represents the robust mean

Pesticides	No of Detections	Nº of Labs detected the pesticide	% of Labs detected the pesticide	No of Conc. Reported	No of Conc. Reported by EU/EFTA labs	Conc. Homogeneity Test (mg/kg)	Robust mean (mg/kg)	CV (%)
Carbaryl	62	56	93	56	53	0.103	0.101	27.0



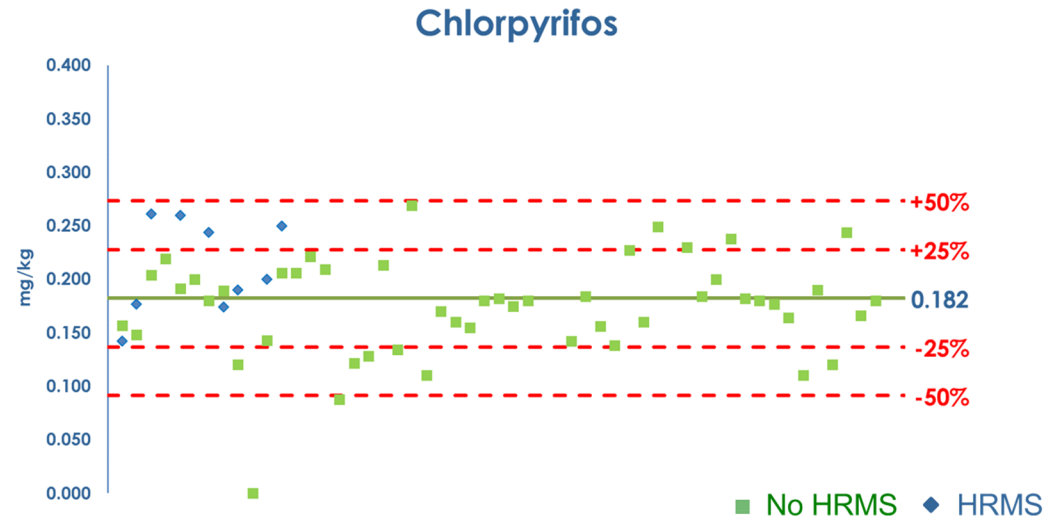
Pesticides	No of Detections	Nº of Labs detected the pesticide	% of Labs detected the pesticide	No of Conc. Reported	No of Conc. Reported by EU/EFTA labs	Conc. Homogeneity Test (mg/kg)	Robust mean (mg/kg)	CV (%)
Carbofuran	63	58	97	58	55	0.182	0.170	29.2



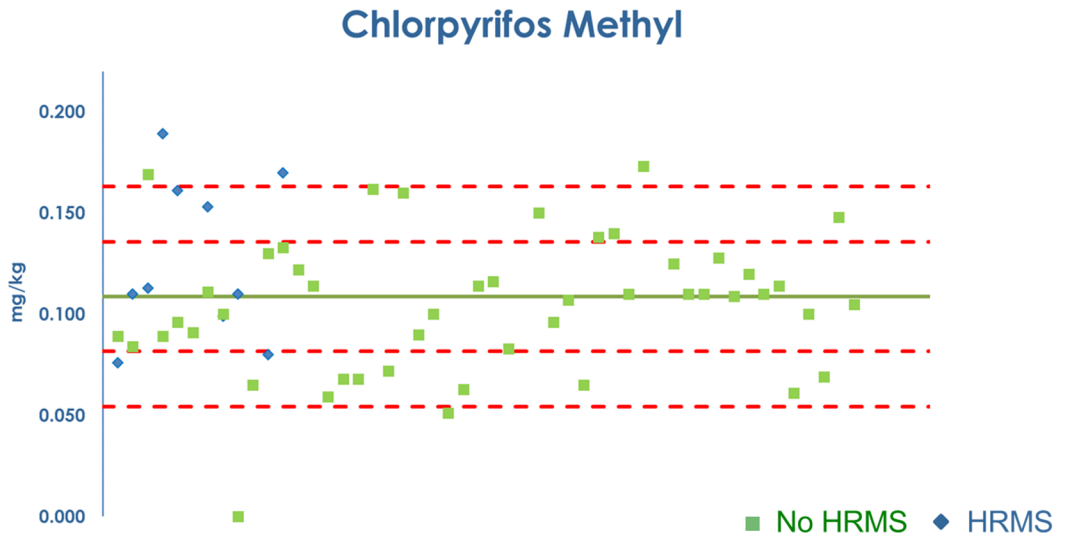
APPENDIX 2. Graphical Representations

The green line represents the robust mean

Pesticides	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of Conc. Reported	No of Conc. Reported by EU/EFTA labs	Conc. Homogeneity Test (mg/kg)	Robust mean (mg/kg)	CV (%)
Chlorpyrifos	69	58	97	62	59	0.189	0.182	24.1

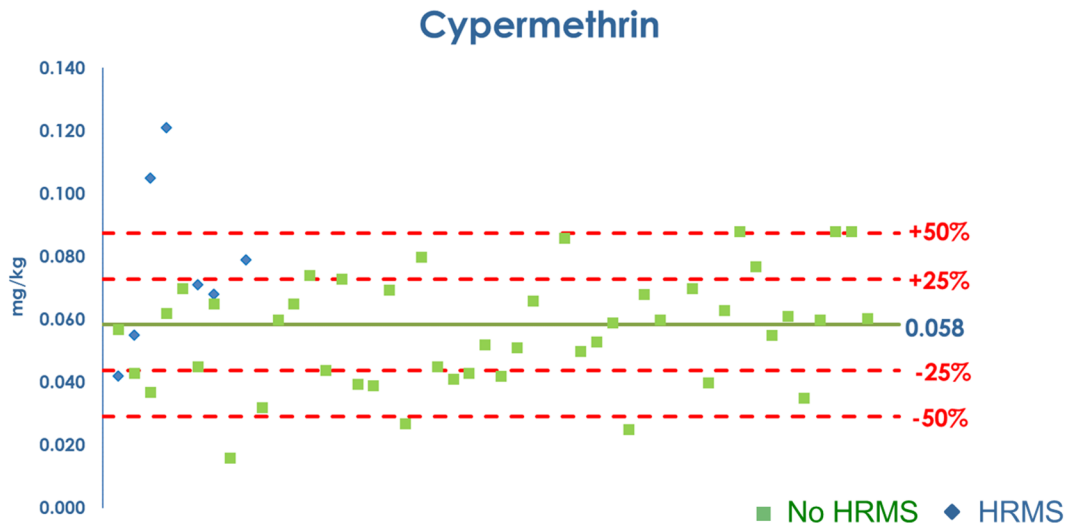


Pesticides	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of Conc. Reported	No of Conc. Reported by EU/EFTA labs	Conc. Homogeneity Test (mg/kg)	Robust mean (mg/kg)	CV (%)
Chlorpyrifos methyl	67	59	98	62	58	0.113	0.109	32.2

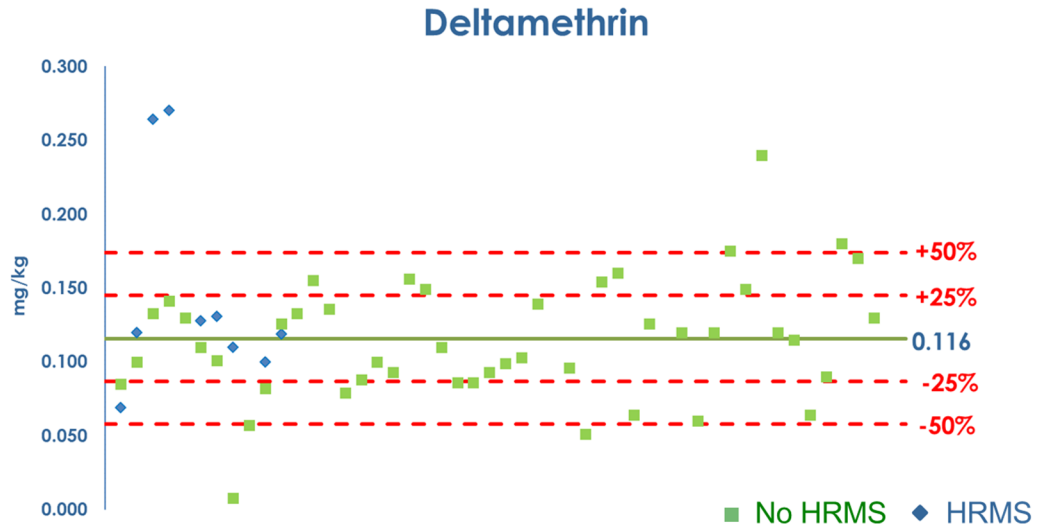


The green line represents the robust mean

Pesticides	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of Conc. Reported	No of Conc. Reported by EU/EFTA labs	Conc. Homogeneity Test (mg/kg)	Robust mean (mg/kg)	CV (%)
Cypermethrin (sum)	61	55	92	55	52	0.064	0.058	33.5

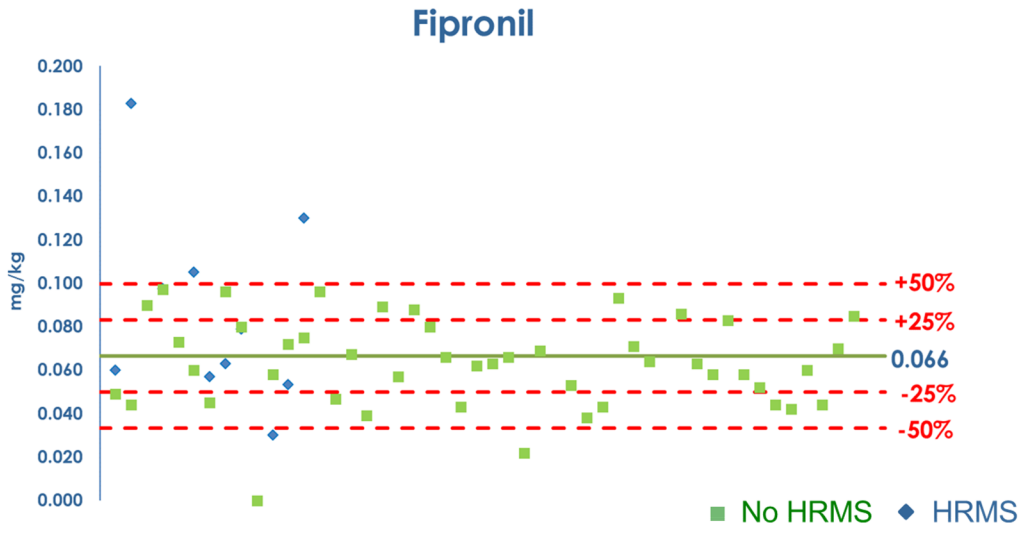


Pesticides	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of Conc. Reported	No of Conc. Reported by EU/EFTA labs	Conc. Homogeneity Test (mg/kg)	Robust mean (mg/kg)	CV (%)
Deltamethrin	64	57	95	59	55	0.130	0.116	33.1

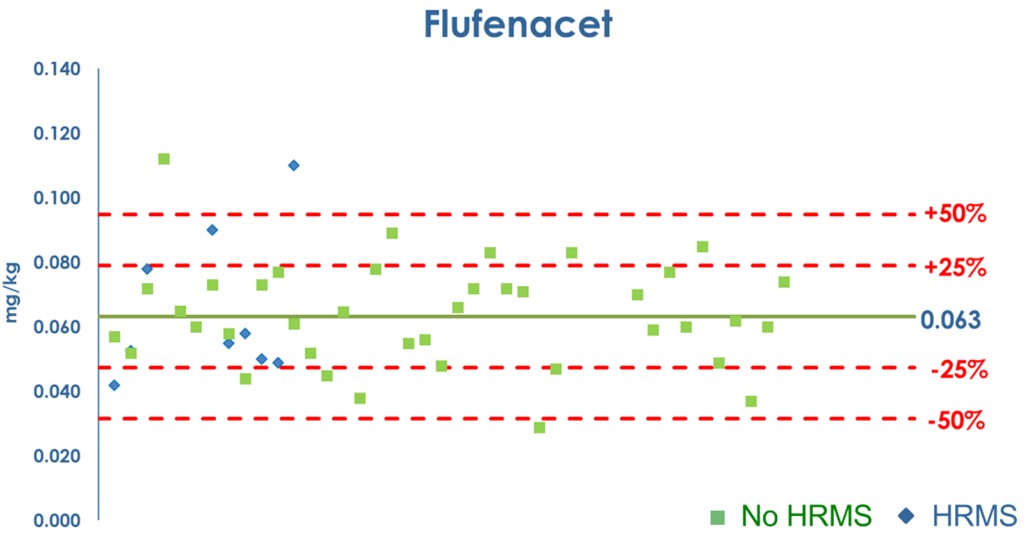


The green line represents the robust mean

Pesticides	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of Conc. Reported	No of Conc. Reported by EU/EFTA labs	Conc. Homogeneity Test (mg/kg)	Robust mean (mg/kg)	CV (%)
Fipronil	66	56	93	61	57	0.077	0.066	32.9

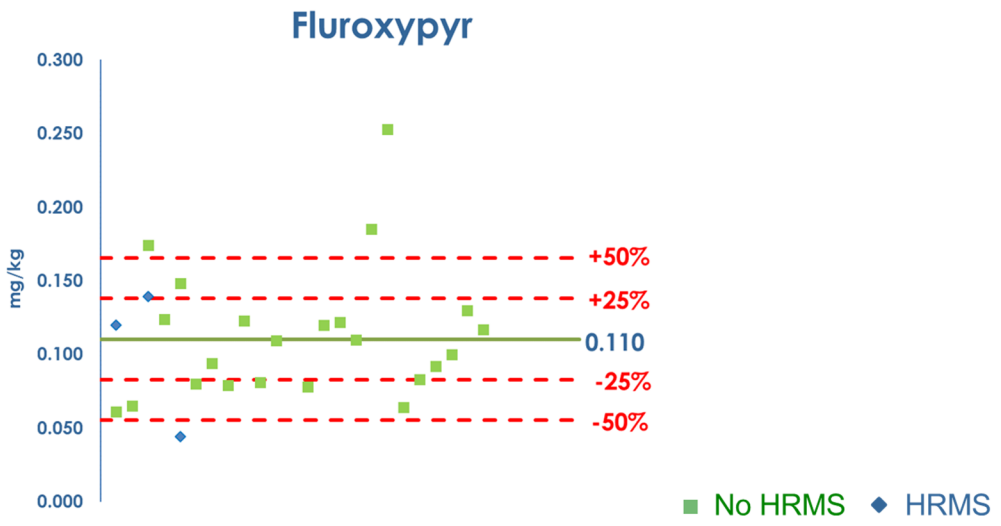


Pesticides	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of Conc. Reported	No of Conc. Reported by EU/EFTA labs	Conc. Homogeneity Test (mg/kg)	Robust mean (mg/kg)	CV (%)
Flufenacet	57	51	85	51	49	0.067	0.063	26.1

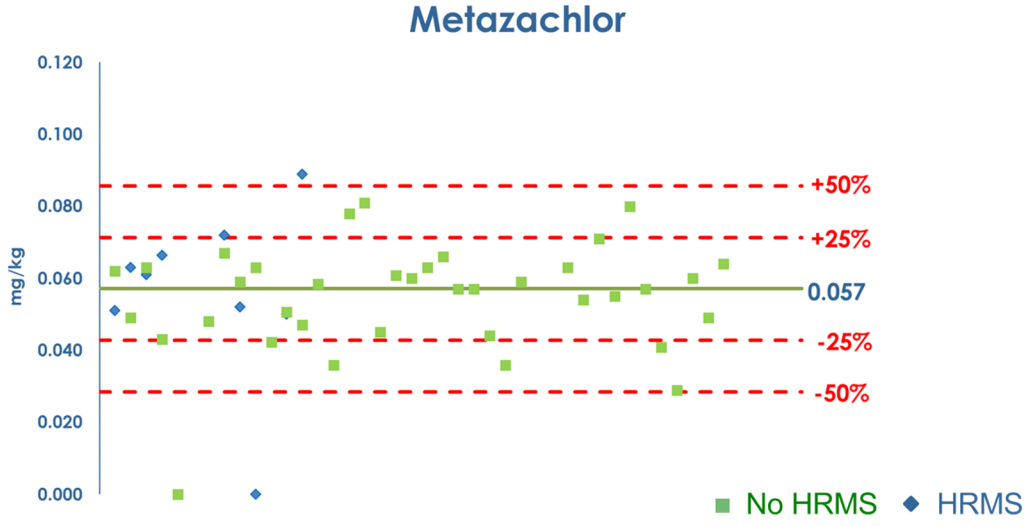


The green line represents the robust mean

Pesticides	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of Conc. Reported	No of Conc. Reported by EU/EFTA labs	Conc. Homogeneity Test (mg/kg)	Robust mean (mg/kg)	CV (%)
Fluroxypyr	29	29	48	27	27	0.135	0.110	37.4



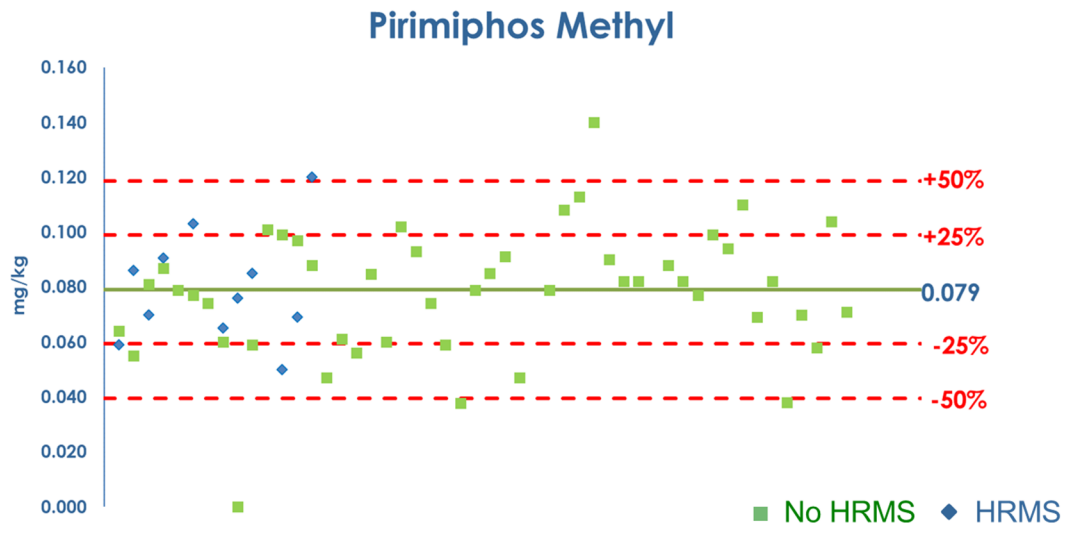
Pesticides	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of Conc. Reported	No of Conc. Reported by EU/EFTA labs	Conc. Homogeneity Test (mg/kg)	Robust mean (mg/kg)	CV (%)
Metazachlor	57	51	85	49	46	0.059	0.057	20.7



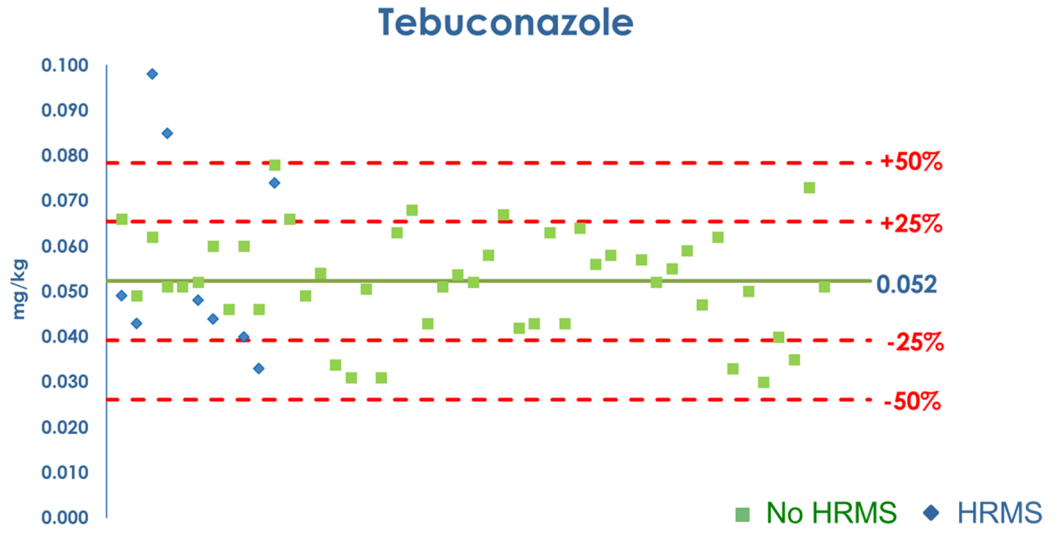
APPENDIX 2. Graphical Representations

The green line represents the robust mean

Pesticides	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of Conc. Reported	No of Conc. Reported by EU/EFTA labs	Conc. Homogeneity Test (mg/kg)	Robust mean (mg/kg)	CV (%)
Pirimiphos-methyl	68	58	97	62	59	0.085	0.079	25.8

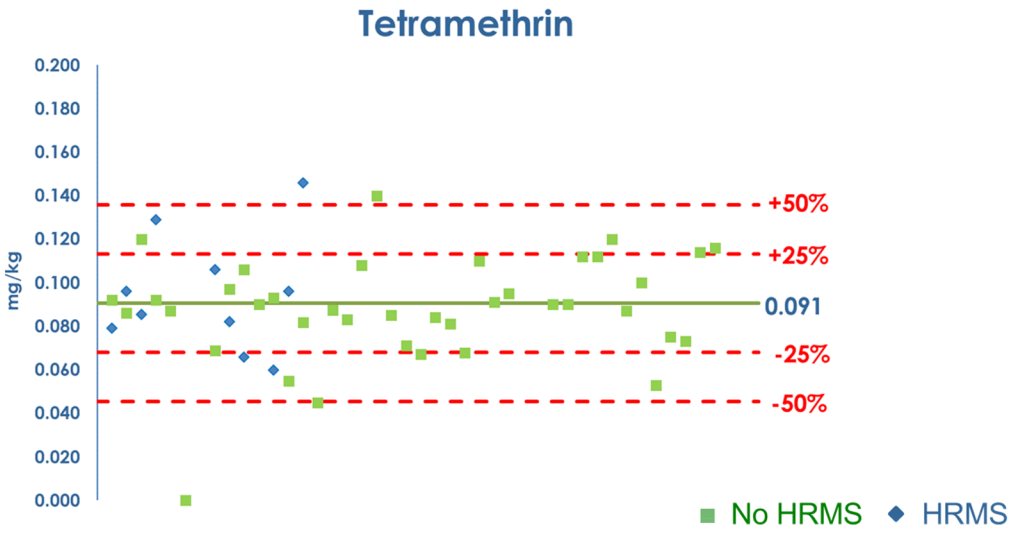


Pesticides	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of Conc. Reported	No of Conc. Reported by EU/EFTA labs	Conc. Homogeneity Test (mg/kg)	Robust mean (mg/kg)	CV (%)
Tebuconazole	63	58	97	59	55	0.055	0.052	24.9

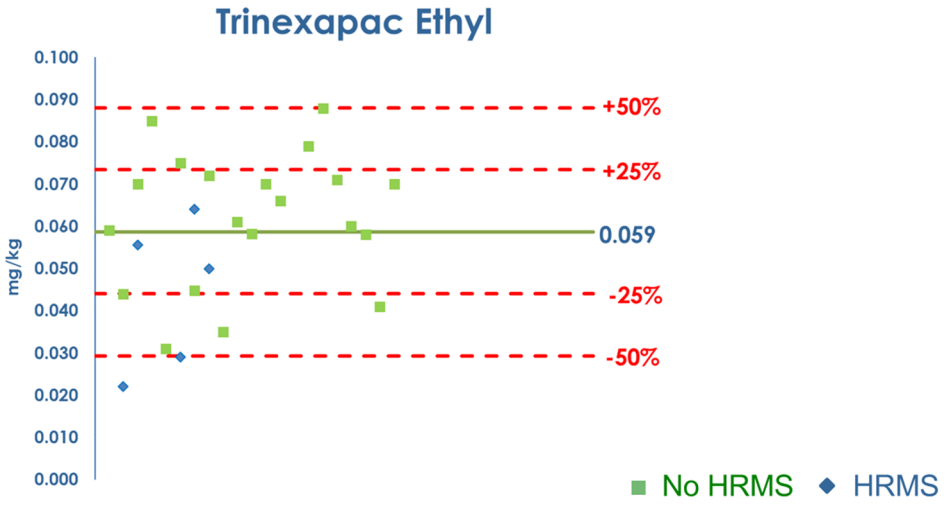


The green line represents the robust mean

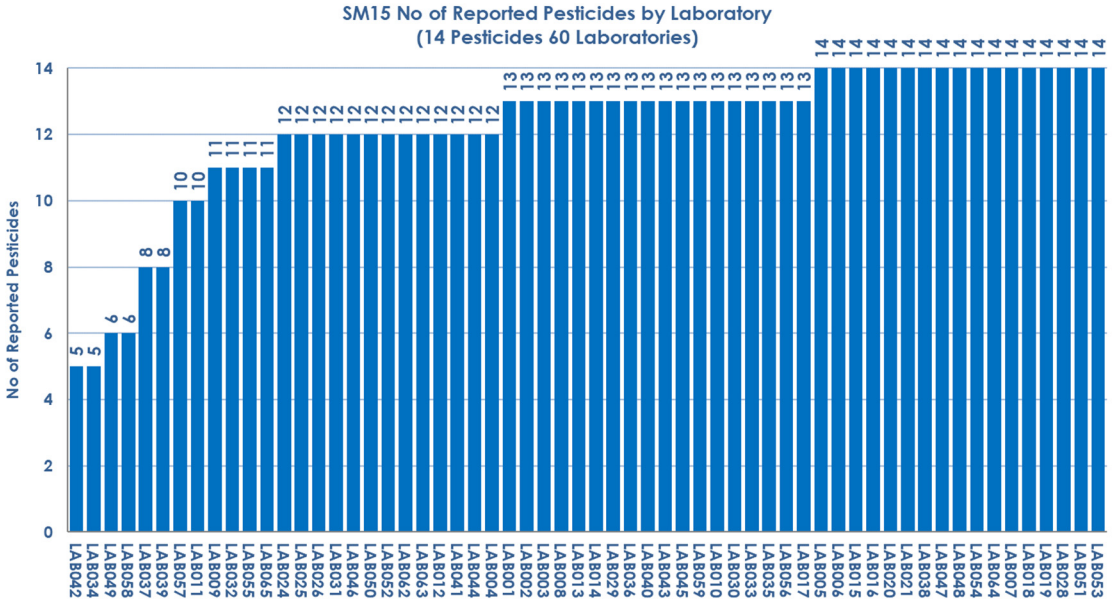
Pesticides	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of Conc. Reported	No of Conc. Reported by EU/EFTA labs	Conc. Homogeneity Test (mg/kg)	Robust mean (mg/kg)	CV (%)
Tetramethrin	60	54	90	52	49	0.093	0.091	22.8

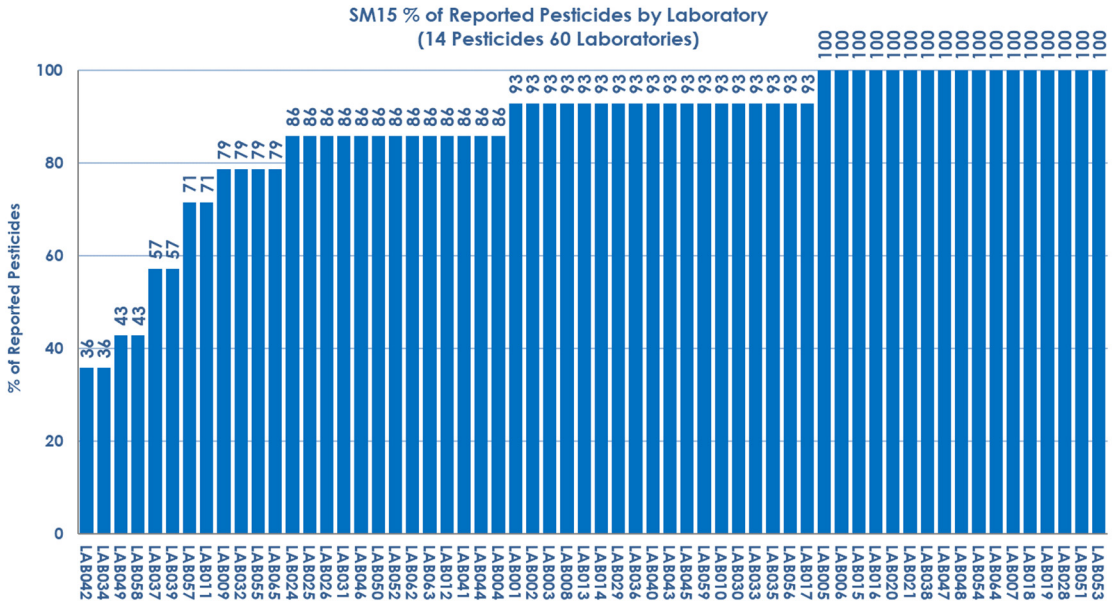


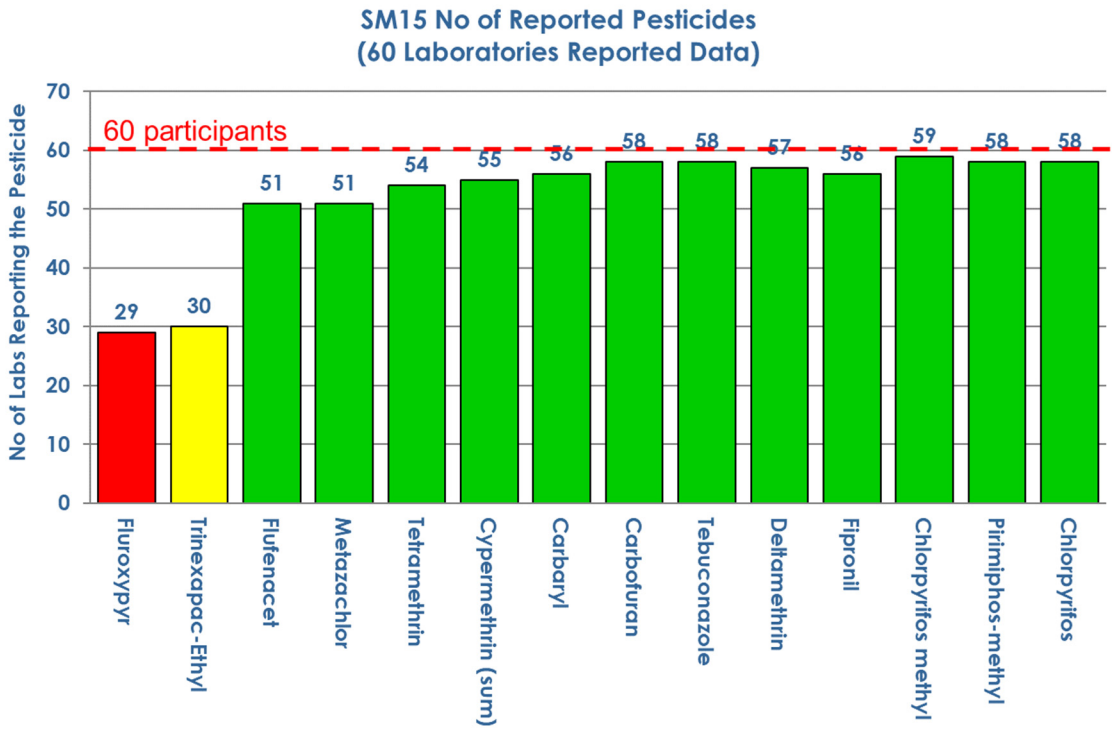
Pesticides	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of Conc. Reported	No of Conc. Reported by EU/EFTA labs	Conc. Homogeneity Test (mg/kg)	Robust mean (mg/kg)	CV (%)
Trinexapac-Ethyl	31	30	50	26	25	0.057	0.059	32.4

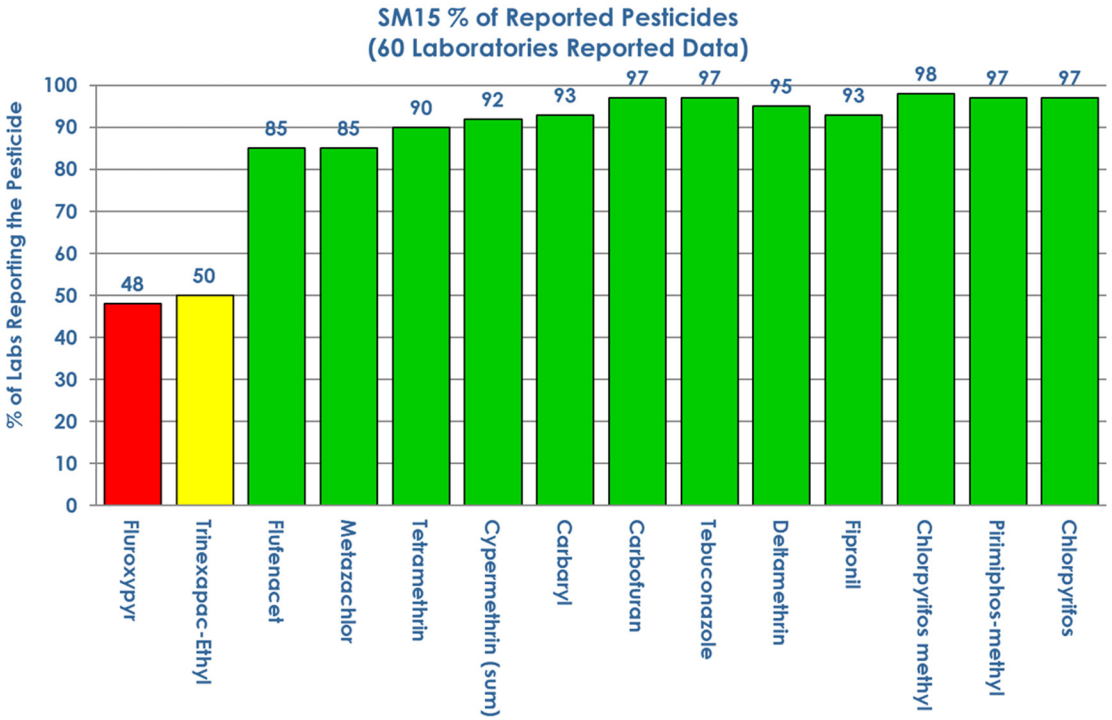


APPENDIX 2. Graphical Representations



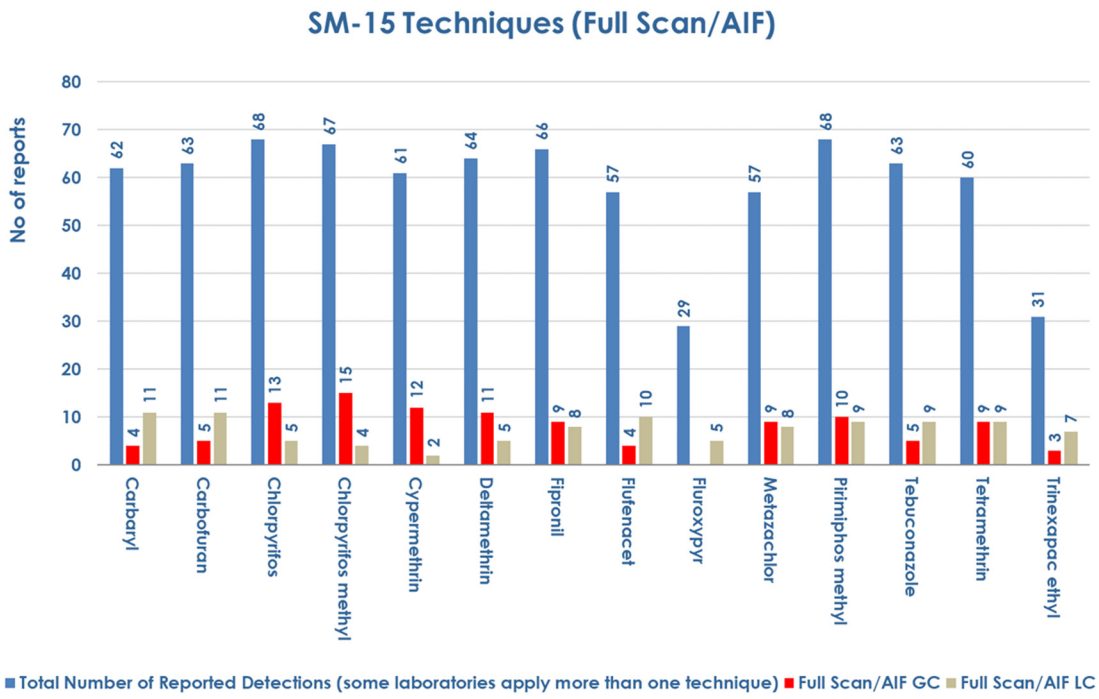



SM15 No of Reported Pesticides
(60 Laboratories Reported Data)
No of Labs Reporting the Pesticide
60 participants
0
10
20
30
40
50
60
70
29
30
51
51
54
55
56
58
58
57
56
59
58
58
Fluroxypyr
Trinexapac-Ethyl
Flufenacet
Metazachlor
Tetramethrin
Cypermethrin (sum)
Carbaryl
Carbofuran
Tebuconazole
Deltamethrin
Fipronil
Chlorpyrifos methyl
Pirimiphos-methyl
Chlorpyrifos

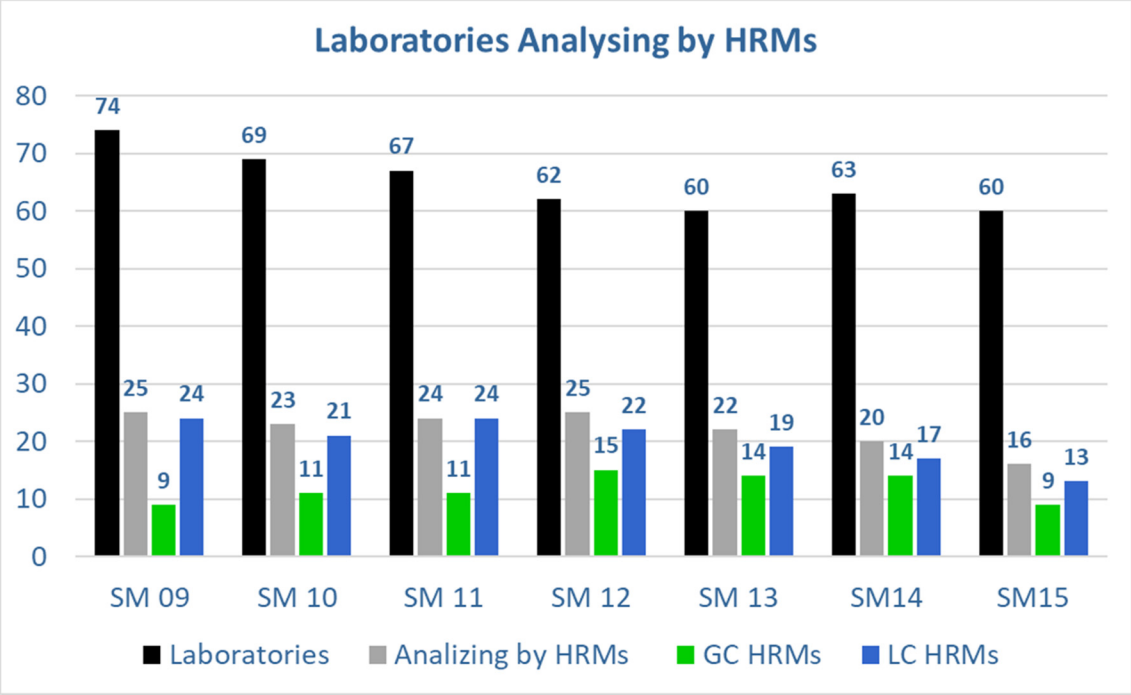

SM15 % of Reported Pesticides
(60 Laboratories Reported Data)
% of Labs Reporting the Pesticide
0
10
20
30
40
50
60
70
80
90
100
48
50
85
85
90
92
93
97
97
95
93
98
97
97
Fluroxypyr
Trinexapac-Ethyl
Flufenacet
Metazachlor
Tetramethrin
Cypermethrin (sum)
Carbaryl
Carbofuran
Tebuconazole
Deltamethrin
Fipronil
Chlorpyrifos methyl
Pirimiphos-methyl
Chlorpyrifos

APPENDIX 2. Graphical Representations

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



Number of laboratories analysing the test items by HRMs



ANNEX 1. List of Laboratories that reported results in EUPT-FV-SM15.

COUNTRY	LABORATORY NAME	CITY
AUSTRIA	AGES INNSBRUCK (LSI-PLMA)	INNSBRUCK
BELGIUM	PRIMORIS BELGIUM	ZWIJNAARDE
BULGARIA	PRIMORIS BULGARIA AD	PLOVDIV
CHINA	AGRO-PRODUCT SAFETY RESEARCH CENTER, CAIQ	BEIJING
CHINA	SHANGHAI MUNICIPAL CENTER FOR DISEASE CONTROL AND PREVENTION	SHANGHAI
COSTA RICA	LABORATORIO DE ANÁLISIS DE RESIDUOS DE PLAGUICIDAS	SAN JOSÉ
CROATIA	BIOINSTITUT D.O.O.	CAKOVEC
CROATIA	SAMPLE CONTROL D.O.O.	ZAGREB
CZECH REPUBLIC	CZECH AGRICULTURE AND FOOD INSPECTION AUTHORITY	PRAGUE
CZECH REPUBLIC	UCT PRAGUE, METROLOGICAL AND TESTING LABORATORY	PRAGUE
DENMARK	DTU NATIONAL FOOD INSTITUTE	LYNGBY
DENMARK	DANISH VETERINARY AND FOOD ADMINISTRATION	RINGSTED
FRANCE	LABORATOIRE CERECO	GARONS
FRANCE	INOVALYS	LE MANS
FRANCE	ANSES - LSAL - UNITÉ PBM	MAISONS-ALFORT CEDEX
FRANCE	SCL	MASSY
FRANCE	SERVICE COMMUN DES LABORATOIRES 34 (SCL 34)	MONTPELLIER
FRANCE	CAMP-LDA66	PERPIGNAN
GERMANY	NIEDERSÄCHSISCHES LANDESAMT FÜR VERBRAUCHERSCHUTZ UND LEBENSMITTELSICHERHEIT LEBENSMITTEL- UND VETERINÄRINSTITUT OLDENBURG	OLDENBURG
GERMANY	BAYER. LANDESAMT FÜR GESUNDHEIT UND LEBENSMITTELSICHERHEIT	ERLANGEN
GERMANY	BUNDESWEHR - PESTICIDE LAB	GARCHING
GERMANY	GALAB LABORATORIES	HAMBURG
GERMANY	EUROFINS DR. SPECHT LABORATORIEN GMBH	HAMBURG
GERMANY	CVUA RRW	KREFELD
GERMANY	LABOR FRIEDLE GMBH	TEGERNHEIM
HUNGARY	PLANT PROTECTION AND SOIL CONSERVATION NRL	VELENCE
HUNGARY	NFCSO FCSLD PLANT PROTECTION AND SOIL CONSERVATION NATIONAL REFERENCE LABORATORY	MISKOLC
IRELAND	DEPARTMENT OF AGRICULTURE, FOOD AND THE MARINE	CELBRIDGE
ITALY	APPA BOLZANO	BOLZANO
KENYA	KENYA PLANT HEALTH SERVICE (KEPHIS)	NAIROBI
LITHUANIA	NATIONAL FOOD AND VETERINARY RISK ASSESSMENT INSTITUTE	VILNIUS
LUXEMBOURG	LABORATOIRE NATIONAL DE SANTÉ DU LUXEMBOURG	DUDELANGE
NORWAY	NIBIO DEPARTMENT OF PESTICIDES AND NATURAL PRODUCTS CHEMISTRY	AAS
PERU	INSPECTORATE SERVICES PERU S.A.C	CHORRILLOS LIMA

ANNEX 1. List of Laboratories that participate in EUPT-FV-SM15

COUNTRY	LABORATORY NAME	CITY
POLAND	GBA POLSKA SP. Z O.O.	LAJSKI
POLAND	GBA POLSKA SP. Z O.O.	LUBLIN
POLAND	WOJEWÓDZKA STACJA SANITARNO-EPIDEMIOLOGICZNA W BYDGOSZCZY	BYDGOSZCZ
PORTUGAL	LABORATÓRIO REGIONAL DE VETERINÁRIA E SEGURANÇA ALIMENTAR	FUNCHAL
PORTUGAL	LABIAGRO	OEIRAS
ROMANIA	SVFSD - NON ANIMAL PESTICIDES LAB	CONSTANTA
SLOVENIA	NLZOH (NATIONAL LABORATORY FOR HEALTH, ENVIRONMENT AND FOOD	MARIBOR
SPAIN	LABORATORIO ANALITICO BIOCLINICO	ALMERIA
SPAIN	LABORATORIO SOIVRE ALMERIA	ALMERIA
SPAIN	ANALYTICA ALIMENTARIA ALMERÍA	ALMERÍA
SPAIN	LABORATORIO AGROALIMENTARIO DE EXTREMADURA	CÁCERES
SPAIN	CARM-LAYSA-LABORATORIO AGROALIMENTARIO	EL PALMAR
SPAIN	LABORATORIO SALUD PÚBLICA AYUNTAMIENTO DE MADRID-MADRIDSALUD	MADRID
SPAIN	LABORATORIO AGRARIO Y FITOPATOLOGICO DE GALICIA	ABEGONDO
SPAIN	LABORATORIO DE RESIDUOS. INSTITUTO TECNOLÓGICO DE CANARIAS, S. A. DEPARTAMENTO DE ANÁLISIS AMBIENTAL	AGÜIMES
SPAIN	LABORATORIO AGROALIMENTARIO GENERALITAT VALENCIANA	BURJASSOT
SPAIN	LABORATORIO KUDAM SLU	PILAR DE LA HORADADA
SPAIN	LABORATORIO AGROAMBIENTAL (GOBIERNO DE ARAGÓN)	ZARAGOZA
SWEDEN	EUROFINS FOOD AND FEED TESTING SWEDEN AB	LIDKÖPING
SWEDEN	SWEDISH FOOD AGENCY	UPPSALA
SWITZERLAND	LABORATORIUM DER URKANTONE	BRUNNEN
SWITZERLAND	SCAV - PESTICIDE LAB	GENEVA
SWITZERLAND	KANTONALES LABOR ZURICH	ZURICH
THE NETHERLANDS	NORMEC GROEN AGRO CONTROL	DELFGAUW
THE NETHERLANDS	NOFALAB B.V.	SCHIEDAM
THE NETHERLANDS	WAGENINGEN FOOD SAFETY RESEARCH (WFSR)	WAGENINGEN