EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUIT AND VEGETABLES.

SCREENING METHODS 12 (EUPT-FV-SM12)

Pesticide Residues in Onion Homogenate

Final Report

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CONTENT

EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUIT AND VEGETABLES. SCREENING METHODS 12

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 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 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 PT for quantitative multi-residue pesticide analysis (EUPT-FV22) 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/12682/2019 "Guidance document on analytical quality control and method validation procedures for pesticide residues and analysis in food and feed".

This EUPT-FV-SM12 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 onion homogenate. The onions were cultivated in a greenhouse in Almería, Spain.

The pesticides used to spike the onion 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-SM12 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 2020, 2021 and 2022.
- That they had particularly acute toxicity and/or had low ARfD values.

Table 2.1 shows the 17 pesticides used to spike the onion sample.

The pesticide treatments were carried out post-harvest using analytical standard solutions. The test item was frozen (using liquid nitrogen) and chopped. The frozen minced onion was mixed in a constantly-spinning container until an homogeneous item was obtained. Finally, 200 g portions of the well-mixed homogenate were weighed out into screw-capped polyethylene plastic bottles, sealed and stored in a freezer at about -20 °C prior to distribution to participants.

Table 2.1 Pesticides used to spike the sample.

Pesticides			
Alachlor	Cyanofenphos	Diuron	Dodemorph
Endrin	Fluacrypyrim	Fonofos	Isoprocarb
Metamitron	Metazachlor	Metobromuron	Monolinuron
Prometryn	Propazine	Propoxur	Simazine
Tetrachlorvinphos			

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 2.3a. 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.

Table 2.2a shows the statistical analyses for each of the pesticides used to treat the sample. All pesticides passed this test.

Table 2.2a Statistical evaluation of the homogeneity test data (n = 20 analyses)

Pesticide	Mean Conc. (mg/Kg)	Ss^2	c	$Ss^2 < c$ Pass/Fail
Alachlor	0.039	7.2E-07	1.7E-05	Pass
Cyanofenphos	0.060	0	4.0E-05	Pass
Diuron	0.079	3.1E-07	6.7E-05	Pass
Dodemorph	0.037	7.8E-08	1.6E-05	Pass
Endrin	0.052	0	3.0E-05	Pass
Fluacrypyrim	0.032	0	1.3E-05	Pass
Fonofos	0.059	6.7E-08	3.8E-05	Pass
Isoprocarb	0.031	5.6E-08	1.1E-05	Pass
Metamitron	0.042	5.6E-08	2.0E-05	Pass
Metazachlor	0.044	3.3E-08	2.1E-05	Pass
Metobromuron	0.057	0	3.6E-05	Pass
Monolinuron	0.059	0	3.9E-05	Pass
Prometryne	0.038	3.3E-08	1.6E-05	Pass
Propazine	0.044	0	2.2E-05	Pass
Propoxur	0.065	2.2E-07	4.5E-05	Pass
Simazine	0.037	0	1.6E-05	Pass
Tetrachlorvinphos	0.054	3.3E-08	3.2E-05	Pass

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

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).

The individual results are given in Table 2.2b. 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 2.2b 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.

(mg/Kg)	Day 1							Day 2								
	Sample 56_A	Sample 56_B	Sample 91_A	Sample 91_B	Sample 121_A	Sample 121_B	Mean 1	Sample 47_A	Sample 47_B	Sample 115_A	Sample 115_B	Sample 71_A	Sample 71_B	Mean2	(M2 – M1)	M2-M1 ≤ 0.3*σ
Alachlor	0.038	0.040	0.039	0.038	0.038	0.039	0.039	0.038	0.039	0.040	0.039	0.041	0.040	0.040	0.001	Pass
Cyanofenphos	0.059	0.060	0.061	0.062	0.060	0.060	0.060	0.061	0.062	0.060	0.060	0.061	0.060	0.061	0.000	Pass
Diuron	0.079	0.081	0.080	0.080	0.078	0.081	0.080	0.080	0.080	0.078	0.081	0.078	0.079	0.079	-0.001	Pass
Dodemorph	0.036	0.037	0.035	0.037	0.041	0.038	0.037	0.037	0.035	0.037	0.038	0.037	0.037	0.037	-0.001	Pass
Endrin	0.051	0.052	0.052	0.052	0.054	0.053	0.052	0.052	0.051	0.052	0.054	0.050	0.052	0.052	-0.001	Pass
Fluacrypyrim	0.031	0.032	0.032	0.030	0.034	0.033	0.032	0.032	0.034	0.032	0.034	0.032	0.032	0.033	0.001	Pass
Fonofos	0.058	0.059	0.057	0.059	0.062	0.060	0.059	0.059	0.057	0.059	0.061	0.059	0.059	0.059	0.000	Pass
Isoprocarb	0.030	0.031	0.031	0.031	0.029	0.032	0.031	0.031	0.033	0.031	0.032	0.031	0.031	0.032	0.001	Pass
Metamitron	0.041	0.042	0.042	0.042	0.040	0.043	0.042	0.042	0.040	0.043	0.042	0.040	0.042	0.042	0.000	Pass
Metazachlor	0.043	0.044	0.042	0.044	0.044	0.045	0.044	0.044	0.042	0.044	0.047	0.044	0.044	0.044	0.001	Pass
Metobromuron	0.056	0.057	0.057	0.055	0.059	0.058	0.057	0.057	0.059	0.057	0.059	0.057	0.057	0.058	0.001	Pass
Monolinuron	0.058	0.059	0.059	0.059	0.061	0.060	0.059	0.059	0.058	0.059	0.061	0.057	0.059	0.059	-0.001	Pass
Prometryne	0.037	0.038	0.038	0.038	0.038	0.039	0.038	0.038	0.040	0.038	0.039	0.038	0.038	0.039	0.001	Pass
Propazine	0.043	0.044	0.044	0.042	0.046	0.045	0.044	0.044	0.046	0.044	0.046	0.044	0.044	0.045	0.001	Pass
Propoxur	0.064	0.065	0.063	0.065	0.065	0.066	0.065	0.065	0.063	0.065	0.067	0.065	0.065	0.065	0.000	Pass
Simazine	0.036	0.037	0.037	0.037	0.039	0.038	0.037	0.037	0.036	0.037	0.039	0.035	0.037	0.037	-0.001	Pass
Tetrachlorvinphos	0.053	0.054	0.052	0.054	0.056	0.055	0.054	0.054	0.053	0.054	0.056	0.054	0.054	0.054	0.000	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 2.2c.

Table 2.2c Statistical test for analytical precision and to demonstrate stability for the 48-hour time-elapse interval.

(mg/Kg)	Day 1							Day 3							(M2 – M1)	M2-M1 ≤ 0.3*σ
	Sample 56_A	Sample 56_B	Sample 91_A	Sample 91_B	Sample 121_A	Sample 121_B	Mean 1	Sample 236_A	Sample 236_B	Sample 42_A	Sample 42_B	Sample128_A	Sample 128_B	Mean2		
Alachlor	0.038	0.040	0.039	0.038	0.038	0.039	0.039	0.039	0.040	0.039	0.041	0.040	0.041	0.040	0.001	Pass
Cyanofenphos	0.059	0.060	0.061	0.062	0.060	0.060	0.060	0.061	0.060	0.059	0.059	0.060	0.060	0.060	-0.001	Pass
Diuron	0.079	0.081	0.080	0.080	0.078	0.081	0.080	0.078	0.079	0.080	0.079	0.079	0.080	0.079	-0.001	Pass
Dodemorph	0.036	0.037	0.035	0.037	0.041	0.038	0.037	0.037	0.037	0.037	0.037	0.037	0.036	0.037	-0.001	Pass
Endrin	0.051	0.052	0.052	0.052	0.054	0.053	0.052	0.050	0.052	0.050	0.052	0.052	0.051	0.051	-0.001	Pass
Fluacrypyrim	0.031	0.032	0.032	0.030	0.034	0.033	0.032	0.032	0.032	0.030	0.032	0.032	0.031	0.032	-0.001	Pass
Fonofos	0.058	0.059	0.057	0.059	0.062	0.060	0.059	0.059	0.059	0.059	0.059	0.059	0.058	0.059	0.000	Pass
Isoprocarb	0.030	0.031	0.031	0.031	0.029	0.032	0.031	0.031	0.031	0.033	0.031	0.031	0.030	0.031	0.001	Pass
Metamitron	0.041	0.042	0.042	0.042	0.040	0.043	0.042	0.042	0.042	0.044	0.042	0.042	0.041	0.042	0.001	Pass
Metazachlor	0.043	0.044	0.042	0.044	0.044	0.045	0.044	0.044	0.044	0.044	0.044	0.044	0.043	0.044	0.000	Pass
Metobromuron	0.056	0.057	0.057	0.055	0.059	0.058	0.057	0.057	0.057	0.055	0.057	0.057	0.056	0.057	-0.001	Pass
Monolinuron	0.058	0.059	0.059	0.059	0.061	0.060	0.059	0.057	0.059	0.057	0.059	0.059	0.058	0.058	-0.001	Pass
Prometryne	0.037	0.038	0.038	0.038	0.038	0.039	0.038	0.038	0.038	0.039	0.038	0.038	0.037	0.038	0.000	Pass
Propazine	0.043	0.044	0.044	0.042	0.046	0.045	0.044	0.044	0.044	0.042	0.044	0.044	0.043	0.044	-0.001	Pass
Propoxur	0.064	0.065	0.063	0.065	0.065	0.066	0.065	0.065	0.065	0.065	0.065	0.065	0.064	0.065	0.000	Pass
Simazine	0.036	0.037	0.037	0.037	0.039	0.038	0.037	0.035	0.037	0.035	0.037	0.037	0.036	0.036	-0.001	Pass
Tetrachlorvinphos	0.053	0.054	0.052	0.054	0.056	0.055	0.054	0.054	0.054	0.054	0.054	0.054	0.053	0.054	0.000	Pass

2.3 Distribution of test item and protocol to participants

Approximately 200 g of treated onion homogenate were shipped to participants on 2nd March 2020. 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 Resulution 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-SM12 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-four laboratories agreed to participate in this proficiency test on screening methods. Sixty-two 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-SM12 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 *
Alachlor	44	71	18	82
Cyanofenphos	37	60	25	75
Diuron	53	85	9	91
Dodemorph	39	63	23	77
Endrin	53	85	9	91
Fluacrypyrim	20	32	42	58
Fonofos	57	92	5	95
Isoprocarb	50	81	12	88
Metamitron	47	76	15	85
Metazachlor	56	90	6	94
Metobromuron	55	89	7	93
Monolinuron	47	76	15	85
Prometryn	55	89	7	93
Propazine	50	81	12	88
Propoxur	60	97	2	98
Simazine	53	85	9	91
Tetrachlorvinphos	58	94	4	96

#The % of laboratories is calculated based on the total number of laboratories submitting results (62 laboratories).

In this EUPT-FV-SM12, 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 average concentrarion 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.

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

Pesticide	Average concentration Homogeneity test (mg/kg)	Robust mean of estimated concentrations reported (mg/kg)	Number of concentrations reported	CV (%)
Alachlor	0.039	0.041	37	20.5
Cyanofenphos	0.060	0.057	29	25.2
Diuron	0.079	0.078	43	35.3
Dodemorph	0.037	0.037	28	16.0
Endrin	0.052	0.054	51	22.1
Fluacrypyrim	0.032	0.030	12	22.6
Fonofos	0.059	0.062	53	23.2
Isoprocarb	0.031	0.033	47	35.9
Metamitron	0.042	0.038	40	32.0
Metazachlor	0.044	0.042	50	27.5
Metobromuron	0.057	0.057	49	34.5
Monolinuron	0.059	0.054	39	36.1
Prometryn	0.038	0.038	52	19.5
Propazine	0.044	0.043	41	25.0
Propoxur	0.065	0.059	57	37.2
Simazine	0.037	0.033	49	40.2
Tetrachlorvinphos	0.054	0.051	46	36.3

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

4.1.1 Other Reported Compounds

Some laboratories reported additional compounds to those present in the test item. Some of them 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. Other reported compounds bellow 0.01 mg/kg or not quantified.

Laboratory Code	Other Reported Compounds	Concentration Reported (mg/kg)
Lab002	3,5-Dichloroaniline	0.01
Lab005	3,4-Dichloroaniline	
Lab006	3,4-Dichloroaniline	0.026
	Fluometuron	0.04
Lab015	Fenfuram	
Lab016	Chlorfenapyr	
Lab023	Chlorbufam	
	3,4-Dichlorophenyl isocyanate	
	Jasmonic Acid	
	Naphthol, 1-	
	Pyroquilon	
Lab025	Azoxystrobin	0.023
	Fenhexamid	0.010
Lab031	Proxan	
	Quinacetol	
	Terbutryn	0.02

Laboratory Code	Other Reported Compounds	Concentration Reported (mg/kg)
	Vitamin D3/Colecalciferol	
Lab032	3,4-Dichloraniline	0.13
	3-Chloraniline	0.012
Lab040	Sulphur	
Lab042	Acetochlor	
Lab047	Buturon	0.007
	Monuron	0.007
Lab048	Chlorbufam	
	Dinotefuran	
Lab052	Captan	0.073
	Tetrahydrophtalimide	0.037
Lab053	Jasmonic acid	
Lab055	3,4-Dichloraniline	
Lab057	Phenisopham	
	Sebuthylazine	
	Sebuthylazine desethyl	
	Trietazine	
Lab058	Chlorbufam	
Lab060	Dithiocarbamates (det. as CS2)	0.46
	Sulphur	0.86
Lab064	Dieldrin	0.048

Three laboratories reported Chlorbufam and four laboratories reported 3,4-Dichloroaniline, but none of them where detected by the Organiser.

4.2 Assessment of laboratory performance.

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

Table 4.3.a Classification of laboratories according to the number of pesticides reported.

Laboratory Code	No of Reported Pesticides	% of Reported Pesticides	Other Reported Pesticides Not Confirmed by the Organiser
Lab009	17	100	0
Lab018	17	100	0
Lab020	17	100	0
Lab035	17	100	0
Lab049	17	100	0
Lab062	17	100	0
Lab042	17	100	1
Lab025	17	100	2
Lab032	17	100	2
Lab047	17	100	2
Lab023	17	100	5
Lab003	16	94	0
Lab013	16	94	0
Lab021	16	94	0
Lab030	16	94	0
Lab034	16	94	0

Laboratory Code	No of Reported Pesticides	% of Reported Pesticides	Other Reported Pesticides Not Confirmed by the Organiser
Lab044	16	94	0
Lab045	16	94	0
Lab046	16	94	0
Lab050	16	94	0
Lab051	16	94	0
Lab056	16	94	0
Lab059	16	94	0
Lab002	16	94	1
Lab015	16	94	1
Lab016	16	94	1
Lab058	16	94	1
Lab010	15	88	0
Lab017	15	88	0
Lab005	15	88	1
Lab052	15	88	2
Lab057	15	88	4
Lab001	14	82	0
Lab004	14	82	0
Lab014	14	82	0
Lab029	14	82	0
Lab040	14	82	1
Lab011	13	76	0
Lab022	13	76	0
Lab028	13	76	0
Lab033	13	76	0
Lab037	13	76	0
Lab039	13	76	0
Lab053	13	76	1
Lab055	13	76	1
Lab060	13	76	2
Lab008	12	71	0
Lab026	12	71	0
Lab041	12	71	0
Lab061	12	71	0
Lab024	11	65	0
Lab063	11	65	0
Lab064	11	65	1
Lab006	11	65	2
Lab048	11	65	2
Lab019	6	35	0
Lab054	6	35	0
Lab031	6	35	4
Lab036	4	24	0
Lab007	3	18	0
Lab027	1	6	0
Lab043	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-SM12 webpage, not in the printed version).

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

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

Laboratory Code	Number of Present Pesticides Reported (17 Evaluated Pesticides)	% of Present Pesticides Reported (17 Evaluated Pesticides)
Lab001	14	82
Lab002	16	94
Lab003	16	94
Lab004	14	82
Lab005	15	88
Lab006	11	65
Lab007	3	18
Lab008	12	71
Lab009	17	100
Lab010	15	88
Lab011	13	76
Lab013	16	94
Lab014	14	82
Lab015	16	94
Lab016	16	94
Lab017	15	88
Lab018	17	100
Lab019	6	35
Lab020	17	100
Lab021	16	94
Lab022	13	76
Lab023	17	100
Lab024	11	65
Lab025	17	100
Lab026	12	71
Lab027	1	6
Lab028	13	76
Lab029	14	82
Lab030	16	94
Lab031	6	35
Lab032	17	100
Lab033	13	76
Lab034	16	94
Lab035	17	100
Lab036	4	24
Lab037	13	76
Lab039	13	76
Lab040	14	82
Lab041	12	71
Lab042	17	100
Lab043	0	0
Lab044	16	94

Laboratory Code	Number of Present Pesticides Reported (17 Evaluated Pesticides)	% of Present Pesticides Reported (17 Evaluated Pesticides)
Lab045	16	94
Lab046	16	94
Lab047	17	100
Lab048	11	65
Lab049	17	100
Lab050	16	94
Lab051	16	94
Lab052	15	88
Lab053	13	76
Lab054	6	35
Lab055	13	76
Lab056	16	94
Lab057	15	88
Lab058	16	94
Lab059	16	94
Lab060	13	76
Lab061	12	71
Lab062	17	100
Lab063	11	65
Lab064	11	65

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

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

Pesticide	Total Number of Laboratories Reporting Data	*Total Number of Reported Detections	GC	Full Scan/AIF GC	LC	Full Scan/AIF LC
Alachlor	44	48	31	9	17	11
Cyanofenphos	37	41	33	12	8	6
Diuron	53	55	5	1	50	21
Dodemorph	39	45	19	10	26	14
Endrin	53	66	66	21	0	0
Fluacrypyrim	20	22	10	3	12	10
Fonofos	57	62	40	11	22	11
Isoprocarb	50	55	12	2	43	18
Metamitron	47	57	4	0	53	25
Metazachlor	56	61	27	8	34	14
Metobromuron	55	60	9	1	51	19
Monolinuron	47	51	10	2	41	15
Prometryn	55	62	29	7	33	17
Propazine	50	55	22	7	33	20
Propoxur	60	66	20	8	46	15
Simazine	53	58	24	6	34	16
Tetrachlorvinphos	58	63	37	10	26	18

*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-four laboratories agreed to participate in this proficiency test on screening methods. Sixty-two laboratories submitted results on time (two laboratories did not send results).

Seventeen EU Member States, 2 EFTA countries (Norway and Switzerland) and three non-EU/EFTA countries (China, Kenya and Turkey) 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-SM12 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 was 860; 331 were made by GC and 529 by LC. 43% of the detections were made using full-scan or all ion fragmentation (AIF)(118 by GC-full scan/AIF techniques and 250 by LC-full scan/AIF techniques). 41% of the laboratories reported their results using HRMS and 723 of the results were reported indicating a concentration value (84% of the total results).

Eleven of the 64 laboratories were able to detect all 17 present pesticides in the test item. Seven laboratories detected less than 50% of the pesticides present. Seventy-eight percent of the laboratories (50 laboratories) that reported results were able to detect more than 70 % of the evaluated pesticides.

Three laboratories reported Chlorbufam and four laboratories reported 3,4-Dichloroaniline, but none of them where detected by the Organiser.

Twenty-one participants reported 34 different compounds which were not present in the test items. Whether this should be judged as poor performance, or not, depends on how each participant would act on these positive findings in routine analysis. If the reported pesticide was reported as positive with no further identifying confirmation, then the result would be a false positive and hence erroneous monitoring data would be reported. If the reported pesticide is regarded simply as 'suspect' or 'indicatively present', leading to additional analysis to confirm identity before reporting the result, then those pesticides indicated as 'other reported pesticides' in this report are not really an issue.

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

6. SUGGESTIONS FOR FUTURE WORK

The Organiser and the Scientific Committee consider that screening methods have provided additional value to the current quantitative multiresidue methods routinely used for monitoring purposes. The results of this test are most encouraging, but also indicate the need for continued evaluation of screening methods. Therefore, further proficiency tests will be organised to provide support to those laboratories using screening methods in order to extend their use and improve their reliability. These methods will be used more and more as screens/filters, to make routine

laboratory work easier and faster. The need for screening method validation has been recognised and guidelines for such validation have been prepared and included in Document SANTE/12682/2019

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/12682/2019).

8. ACKNOWLEDGEMENTS

The Organiser is grateful to the European Commission for funding this 12th European Proficiency Test for Screening Methods in Fruit and Vegetables.

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

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

APPENDIX 1. Results

Table AP1.1. Reported pesticides

Laboratory Code Total No of Reporting Laboratories = 62	Evaluated Pesticides (17) R: Reported Pesticide																	Reported Pesticides by Laboratory	% Reported Pesticides by Laboratory
	Alachlor	Cyanofenphos	Diuron	Dodemorph	Endrin	Fluacryptrim	Fonofos	Isoprocarb	Metamitron	Metazachlor	Metobromuron	Monolinuron	Prometryn	Propazine	Propoxur	Simazine	Tetrachlorvinphos		
Lab001			R		R	R	R	R	R	R	R	R	R	R	R	R	R	14	82
Lab002	R	R	R	R	R	R	R	R		R	R	R	R	R	R	R	R	16	94
Lab003	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	16	94
Lab004			R	R	R		R	R	R	R	R	R	R	R	R	R	R	14	82
Lab005	R	R	R	R	R		R	R		R	R	R	R	R	R	R	R	15	88
Lab006	R		R		R		R	R		R	R	R	R		R		R	11	65
Lab007	R						R								R			3	18
Lab008		R	R	R	R	R	R	R	R	R	R				R		R	12	71
Lab009	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	17	100
Lab010	R		R	R	R		R	R	R	R	R	R	R	R	R	R	R	15	88
Lab011			R		R		R	R	R	R	R	R	R	R	R	R	R	13	76
Lab012	no results reported																		
Lab013	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	16	94
Lab014		R	R		R	R	R	R	R	R	R	R	R		R	R	R	14	82
Lab015	R	R	R	R	R	R	R		R	R	R	R	R	R	R	R	R	16	94
Lab016	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	16	94
Lab017	R	R	R	R	R		R		R	R	R	R	R	R	R	R	R	15	88
Lab018	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	17	100
Lab019				R	R		R								R	R	R	6	35
Lab020	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	17	100
Lab021	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	16	94
Lab022			R		R		R	R	R	R	R	R	R	R	R	R	R	13	76
Lab023	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	17	100
Lab024	R		R		R		R			R	R	R	R		R	R	R	11	65
Lab025	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	17	100
Lab026			R		R		R	R	R	R	R		R	R	R	R	R	12	71
Lab027															R			1	6
Lab028			R		R		R	R	R	R	R	R	R	R	R	R	R	13	76
Lab029	R		R		R		R	R	R	R	R	R	R	R	R	R	R	14	82
Lab030	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	16	94
Lab031				R		R				R				R	R		R	6	35
Lab032	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	17	100
Lab033	R		R				R	R	R	R	R	R	R	R	R	R	R	13	76
Lab034	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	16	94
Lab035	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	17	100
Lab036	R						R									R	R	4	24
Lab037	R		R		R		R	R	R	R	R		R	R	R	R	R	13	76
Lab038	no results reported																		
Lab039		R	R	R	R		R		R	R	R	R	R		R	R	R	13	76
Lab040	R	R	R		R	R	R	R		R	R		R	R	R	R	R	14	82
Lab041	R		R		R		R			R	R	R	R	R	R	R	R	12	71
Lab042	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	17	100
Lab043	no detections																	0	0
Lab044	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	16	94
Lab045	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	16	94
Lab046	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	16	94
Lab047	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	17	100
Lab048		R	R	R	R			R	R	R			R	R	R		R	11	65
Lab049	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	17	100
Lab050	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	16	94
Lab051	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	16	94
Lab052		R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	15	88
Lab053			R	R			R	R	R	R	R	R	R	R	R	R	R	13	76

APPENDIX 1. Results

Laboratory Code Total No of Reporting Laboratories = 62	Evaluated Pesticides (17) R: Reported Pesticide																	Reported Pesticides by Laboratory	% Reported Pesticides by Laboratory
	Alachlor	Cyanofenphos	Diuron	Dodemorph	Endrin	Fluacryprim	Fonofos	Isoprocarb	Metamitron	Metazachlor	Metobromuron	Monolinuron	Prometryn	Propazine	Propoxur	Simazine	Tetrachlorvinphos		
Lab054									R		R	R	R		R	R		6	35
Lab055	R	R		R	R		R	R		R	R		R	R	R	R	R	13	76
Lab056	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	16	94
Lab057	R	R	R		R		R	R	R	R	R	R	R	R	R	R	R	15	88
Lab058	R		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	16	94
Lab059	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	16	94
Lab060	R		R		R		R	R	R	R	R		R	R	R	R	R	13	76
Lab061			R	R			R	R	R	R	R		R	R	R	R	R	12	71
Lab062	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	17	100
Lab063	R		R		R		R	R		R	R	R	R		R		R	11	65
Lab064	R	R			R		R	R		R	R		R	R	R		R	11	65
Lab001			R		R	R	R	R	R	R	R	R	R	R	R	R	R	14	82
Lab002	R	R	R	R	R	R	R	R		R	R	R	R	R	R	R	R	16	94
Lab003	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	16	94
Reported Pesticides	44	37	53	39	53	20	57	50	47	56	55	47	55	50	60	53	58		
% of Reported Pesticides	71	60	85	63	85	32	92	81	76	90	89	76	89	81	97	85	94		

Table AP1.2. Estimated Concentrations Reported on a voluntary basis (only informative purposes)
Not all the laboratories reporting results have reported estimated concentration values
Results reported without concentration values are expressed as R.

Evaluated Pesticides (17)																	
LABORATORY CODE Total No of Reporting Laboratories = 62	Alachlor	Cyanofenphos	Diuron	Dodemorph	Endrin	Fluacryprim	Fonofos	Isoprocarb	Metamitron	Metazachlor	Metobromuron	Monolinuron	Prometryn	Propazine	Propoxur	Simazine	Tetrachlorvinphos
Average concentration Homogeneity test (mg/kg)	**0.039**	**0.060**	**0.079**	**0.037**	**0.052**	**0.032**	**0.059**	**0.031**	**0.042**	**0.044**	**0.057**	**0.059**	**0.038**	**0.044**	**0.065**	**0.037**	**0.054**
Robust mean of estimated concentrations reported (mg/kg)	**0.041**	**0.057**	**0.078**	**0.037**	**0.054**	**0.03**	**0.062**	**0.033**	**0.038**	**0.042**	**0.057**	**0.054**	**0.038**	**0.043**	**0.059**	**0.033**	**0.051**
CV (%)	**20.5**	**25.2**	**35.3**	**16**	**22.1**	**22.6**	**23.2**	**35.9**	**32**	**27.5**	**34.5**	**36.1**	**19.5**	**25**	**37.2**	**40.2**	**36.3**
Lab001			R		0.049	R	R	0.036	0.029	R	0.056	R	0.038	R	0.053	0.024	R
Lab002	0.035	0.025	0.079	0.025	0.051	0.025	0.061	0.03		0.041	0.054	0.051	0.034	0.025	0.047	0.025	0.055
Lab003	0.043	0.057	0.076	0.03	0.044		0.06	0.056	0.063	0.047	0.064	0.059	0.035	0.038	0.092	0.046	0.0807
Lab004			0.0417	0.0338	0.077		0.0459	0.0228	0.034	0.0376	0.0323	0.0345	0.0282	0.0404	0.0488	0.0191	0.0424
Lab005	0.04325	0.06275	0.1	0.035	0.051		0.063	0.041		0.049	0.08	0.062	0.037	0.05	0.068	0.033	0.058
Lab006	0.015		0.08		0.05		0.04	0.03		0.012	0.06	0.06	0.03		0.05		0.02
Lab007	0.0461						0.0751								0.0651		
Lab008		0.043	0.068	0.037	0.046	0.029	0.043	0.028	0.045	0.034	0.045				0.06		0.041
Lab009	0.0434	0.0593	0.1046	0.0408	0.0547	0.0268	0.06	0.041	0.0744	0.0432	0.0676	0.0421	0.0357	0.0501	0.1316	0.0217	0.0629
Lab010	0.035		0.086	0.037	0.053		0.066	0.03	0.036	0.044	0.062	0.06	0.032	0.043	0.061	0.025	0.069
Lab011			0.031		0.023		0.034	0.021	0.019	0.021	0.04	0.039	0.031	0.031	0.029	0.021	0.033
Lab013	0.043	0.055	0.1	0.041	0.07		0.082	0.032	0.048	0.05	0.05	0.065	0.05	0.055	0.067	0.028	0.1
Lab014		0.062	0.0857		0.057	0.028	0.0634	0.0368	0.0447	0.059	0.0657	0.0649	0.0403		0.0694	0.0319	0.0573
Lab015	R	R	0.063	R	0.042	R	0.077		R	R	R	R	R	R	0.063	R	R
Lab016	R	R	R	R	0.128		R	R	R	R	R	R	R	R	R	R	R
Lab017	0.04	R	0.04	R	0.065		0.05		0.035	0.04	0.04	R	0.045	0.03	0.045	0.02	0.04
Lab018	0.0449	0.0707	0.1008	0.0437	0.0665	0.0348	0.0592	0.0457	0.0523	0.0476	0.0859	0.0747	0.0423	0.0509	0.0998	0.0194	0.0546
Lab019				R	0.09		0.17								0.09	0.03	0.04
Lab020	0.044	0.066	0.3	0.051	0.064	0.031	0.045	0.036	0.066	0.063	0.19	0.14	0.045	0.059	0.081	0.12	0.063

APPENDIX 1. Results

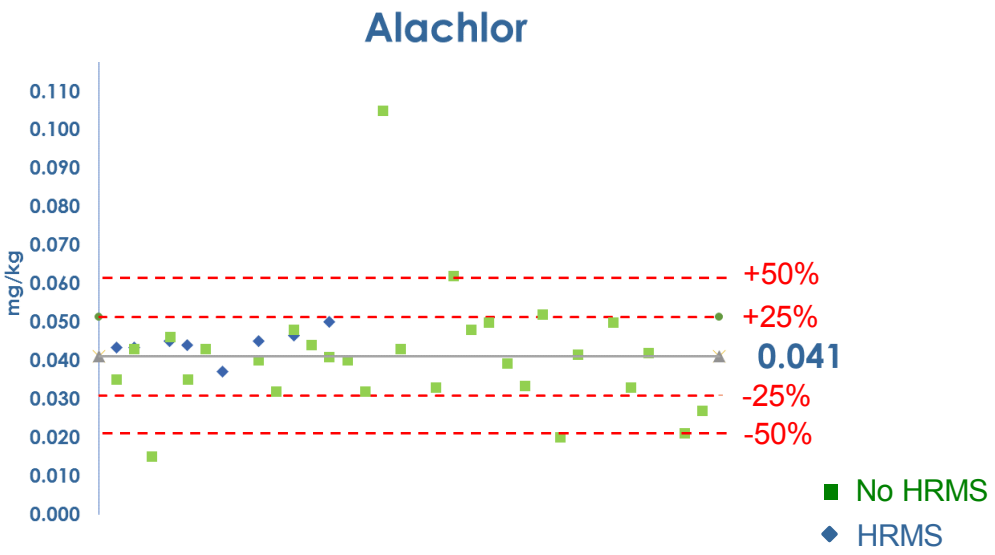
Evaluated Pesticides (17)																	
LABORATORY CODE Total No of Reporting Laboratories = 62	Alachlor	Cyanofenphos	Diuron	Dodemorph	Endrin	Fluacrypirim	Fonofos	Isoprocarb	Metamitron	Metazachlor	Metobromuron	Monolinuron	Prometryn	Propazine	Propoxur	Simazine	Tetrachlorvinphos
Average concentration Homogeneity test (mg/kg)	**0.039**	**0.060**	**0.079**	**0.037**	**0.052**	**0.032**	**0.059**	**0.031**	**0.042**	**0.044**	**0.057**	**0.059**	**0.038**	**0.044**	**0.065**	**0.037**	**0.054**
Robust mean of estimated concentrations reported (mg/kg)	**0.041**	**0.057**	**0.078**	**0.037**	**0.054**	**0.03**	**0.062**	**0.033**	**0.038**	**0.042**	**0.057**	**0.054**	**0.038**	**0.043**	**0.059**	**0.033**	**0.051**
CV (%)	**20.5**	**25.2**	**35.3**	**16**	**22.1**	**22.6**	**23.2**	**35.9**	**32**	**27.5**	**34.5**	**36.1**	**19.5**	**25**	**37.2**	**40.2**	**36.3**
Lab021	0.032	0.081	0.05	0.029	0.05		0.053	0.029	0.032	0.036	0.049	0.057	0.031	0.035	0.049	0.046	0.055
Lab022			0.055		0.056		0.045	0.028	0.028	0.026	0.059	0.04	0.033	0.044	0.051	0.044	0.059
Lab023	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R
Lab024	0.048		R		0.063		0.07			R	R	R	0.043		R	0.038	0.063
Lab025	0.037	0.07	0.089	0.035	0.05	0.044	0.07	0.037	0.037	0.051	0.066	0.066	0.04	0.047	0.07	0.032	0.045
Lab026			0.079		0.062		0.058	0.035	0.03	0.078	0.057		0.041	0.044	0.096	0.018	0.07
Lab027															0.053		
Lab028			0.041		0.044		0.057	0.02	0.034	0.018	0.041	0.044	0.035	0.033	0.018	0.022	0.04
Lab029	0.044		0.158		0.046		0.049	0.067	0.035	0.039	0.155	0.028	0.034	0.05	0.043	0.025	0.031
Lab030	0.041	0.069	0.078	0.033	0.051		0.079	0.039	0.05	0.042	0.059	0.063	0.056	0.056	0.059	0.1	0.036
Lab031				0.04		R				0.04				R	0.06		R
Lab032	0.04	0.06	0.03	0.04	0.05	0.03	0.06	0.015	0.025	0.04	0.025	0.03	0.035	0.04	0.035	0.025	0.03
Lab033	0.045		0.096				R	0.018	R	0.046	0.04	R	0.066	0.04	0.064	0.168	R
Lab034	0.032	0.049	0.076	0.034	0.064		0.048	0.038	0.05	0.059	0.098	0.059	0.04	0.065	0.042	0.051	0.051
Lab035	R	0.09	0.095	0.035	0.05	R	0.95	0.04	0.035	0.055	0.065	0.07	0.05	R	0.08	0.045	R
Lab036	0.105						0.067									0.125	0.125
Lab037	0.043		0.09		0.054		0.063	0.03425	0.036	0.041	0.08375		0.036	0.034	0.026	0.023	0.049
Lab039		0.042	0.087	0.043	0.052		0.065		0.036	0.046	0.06	0.068	0.033		0.065	0.038	0.053
Lab040	R	R	R		R	R	R	R		R	2		R	R	R	R	R
Lab041	0.033		0.069		0.042		0.057			0.051	0.079	0.102	0.036	0.04	0.039	0.052	0.027
Lab042	R	0.057	0.064	R	0.032	R	0.052	0.03	1.8	0.031	0.053	0.052	0.033	0.046	0.049	0.041	0.06
Lab043																	
Lab044	0.062	0.046	0.197		0.041	0.025	0.082	0.062	0.055	0.042	0.125	0.087	0.068	R	0.134	0.053	0.108
Lab045	0.048	0.051	0.077	0.034	0.077		0.055	0.031	0.044	0.042	0.051	0.058	0.045	0.041	0.055	0.038	0.069
Lab046	0.05	0.055	0.108	0.04	0.065		0.09	0.038	0.048	0.053	0.06	0.064	0.046	0.058	0.075	0.029	0.06
Lab047	0.05	0.09	R	0.04	0.047	0.02	0.062	0.34	R	0.056	0.064	0.072	0.05	0.03	0.067	0.033	0.055

Evaluated Pesticides (17)																	
LABORATORY CODE Total No of Reporting Laboratories = 62	Alachlor	Cyanofenphos	Diuron	Dodemorph	Endrin	Fluacrypirim	Fonofos	Isoprocarb	Metamitron	Metazachlor	Metobromuron	Monolinuron	Prometryn	Propazine	Propoxur	Simazine	Tetrachlorvinphos
Average concentration Homogeneity test (mg/kg)	**0.039**	**0.060**	**0.079**	**0.037**	**0.052**	**0.032**	**0.059**	**0.031**	**0.042**	**0.044**	**0.057**	**0.059**	**0.038**	**0.044**	**0.065**	**0.037**	**0.054**
Robust mean of estimated concentrations reported (mg/kg)	**0.041**	**0.057**	**0.078**	**0.037**	**0.054**	**0.03**	**0.062**	**0.033**	**0.038**	**0.042**	**0.057**	**0.054**	**0.038**	**0.043**	**0.059**	**0.033**	**0.051**
CV (%)	**20.5**	**25.2**	**35.3**	**16**	**22.1**	**22.6**	**23.2**	**35.9**	**32**	**27.5**	**34.5**	**36.1**	**19.5**	**25**	**37.2**	**40.2**	**36.3**
Lab048		0.06	R	0.04	0.04			0.06	R	0.04			0.04	0.04	0.16		R
Lab049	0.039	0.047	0.091	0.034	0.043	0.049	0.058	0.033	0.024	0.056	0.051	0.045	0.036	0.051	0.041	0.045	0.050
Lab050	R	R	R	R	0.062		0.042	0.018	0.029	R	0.024	0.025	R	R	0.1	R	R
Lab051	0.0335	0.044	0.107	0.034	0.049		0.06	0.04525	0.05575	0.04475	0.06475	0.0735	0.0275	0.034	0.05075	0.015	0.02
Lab052		0.04	0.095	0.062	0.06		0.13	0.038	0.036	0.035	0.05	0.061	0.042	0.083	0.068	0.19	0.069
Lab053			R	R			R	R	R	R	R	R	R	R	R	R	R
Lab054									0.03		0.02	0.02	0.02		0.03	0.02	
Lab055	0.052	R		R	0.067		0.075	R		0.055	R		0.048	0.052	0.086	0.036	0.071
Lab056	0.02	0.02	0.02	0.02	0.07		0.07	0.01	0.03	0.02	0.02	0.02	0.03	0.02	0.04	0.02	0.06
Lab057	0.0415	0.0595	0.091		0.052		0.065	0.033	0.039	0.048	0.0615	0.0725	0.0415	0.0415	0.0565	0.031	
Lab058	R		R	R	0.03	R	R	0.029	R	0.042	0.052	R	0.02	0.03	0.04	R	R
Lab059	0.05	0.07	0.054	0.037	0.063		0.072	0.05	0.031	0.051	0.037	0.032	0.036	0.052	0.064	0.027	0.06
Lab060	0.033		0.044		0.056		0.061	0.0215	0.0212	0.037	0.0346		0.034	0.045	0.0409	0.029	0.064
Lab061			0.069	0.122			0.084	0.047	0.037	0.048	0.084		0.043	0.05	0.087	0.028	0.041
Lab062	0.042	R	0.054	R	R	0.028	0.06	0.021	0.033	0.031	0.043	0.045	0.034	0.038	0.041	0.033	R
Lab063	0.021		0.097		0.025		0.032	0.028		0.017	0.042	0.034	0.034		0.04		0.022
Lab064	0.027	0.046					0.06	0.019		0.023	0.057		0.03	0.049	0.047		0.005

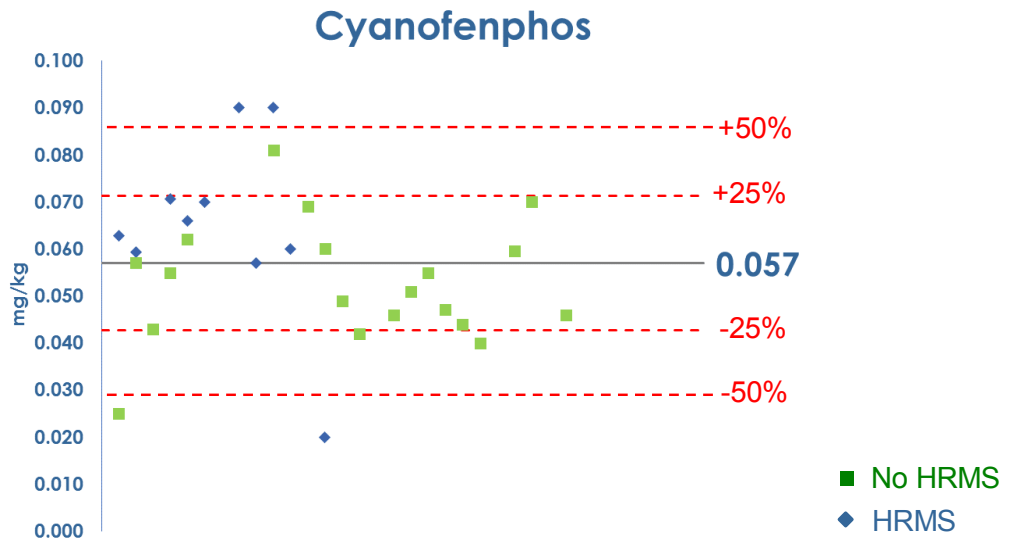
APPENDIX 2. Graphical Representations

The bold line represents the robust mean

Pesticide	Conc. Homogeneity Test (mg/kg)	Robust Mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Alachlor	0.039	0.041	20.5	48	44	71	37

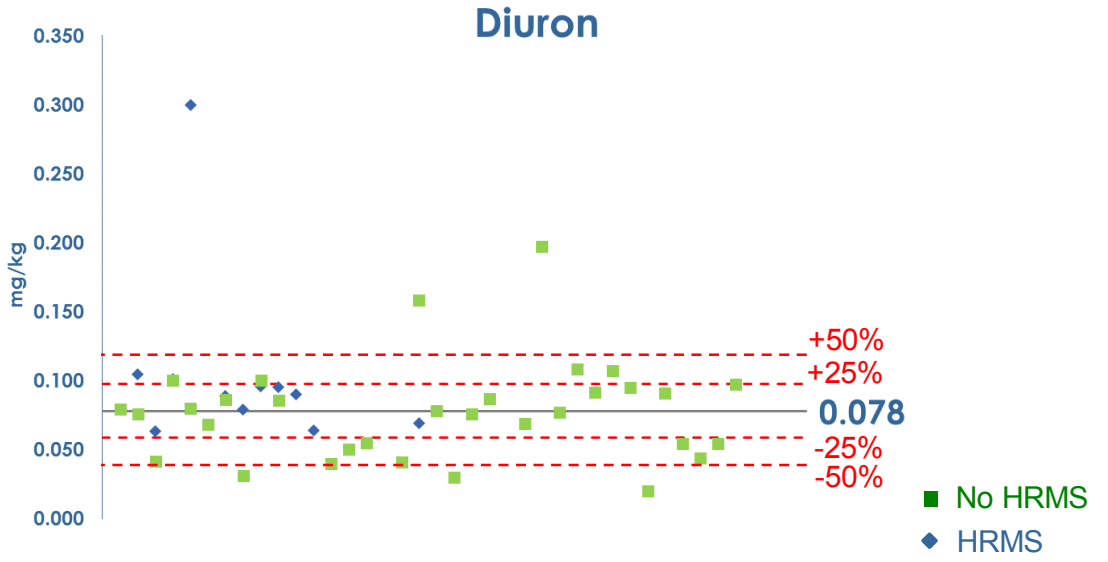


Pesticide	Conc. Homogeneity Test (mg/kg)	Robust Mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Cyanofenphos	0.060	0.057	25.2	41	37	60	29

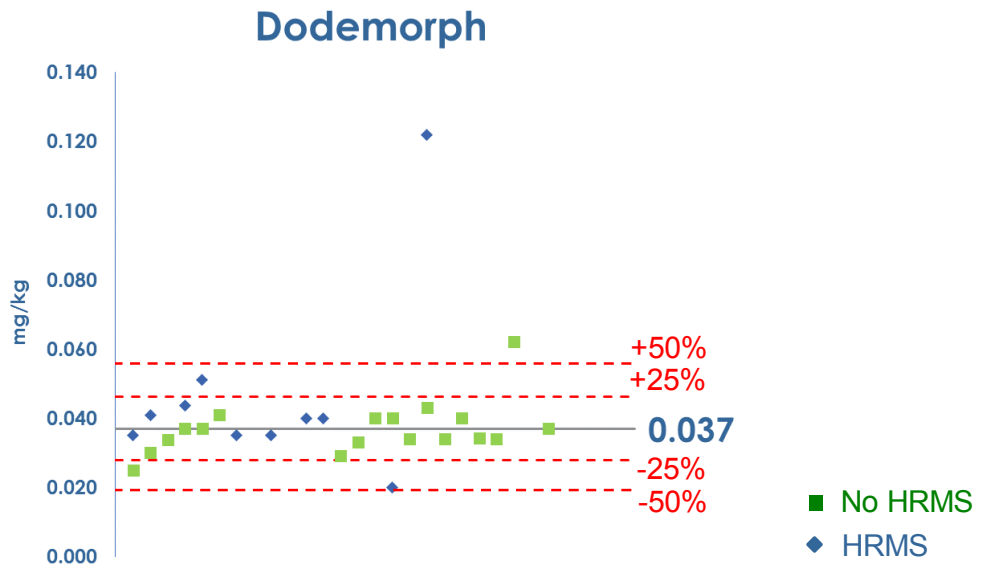


The bold line represents the robust mean

Pesticide	Conc. Homogeneity Test (mg/kg)	Robust Mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Diuron	0.079	0.078	35.3	55	53	85	43

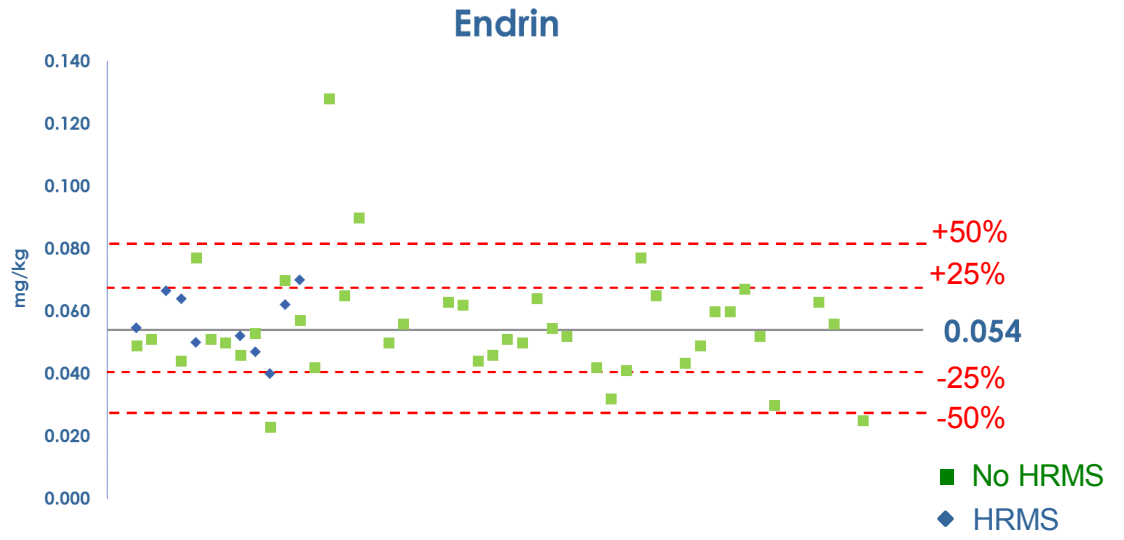


Pesticide	Conc. Homogeneity Test (mg/kg)	Robust Mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Dodemorph	0.037	0.037	16.0	45	39	63	28

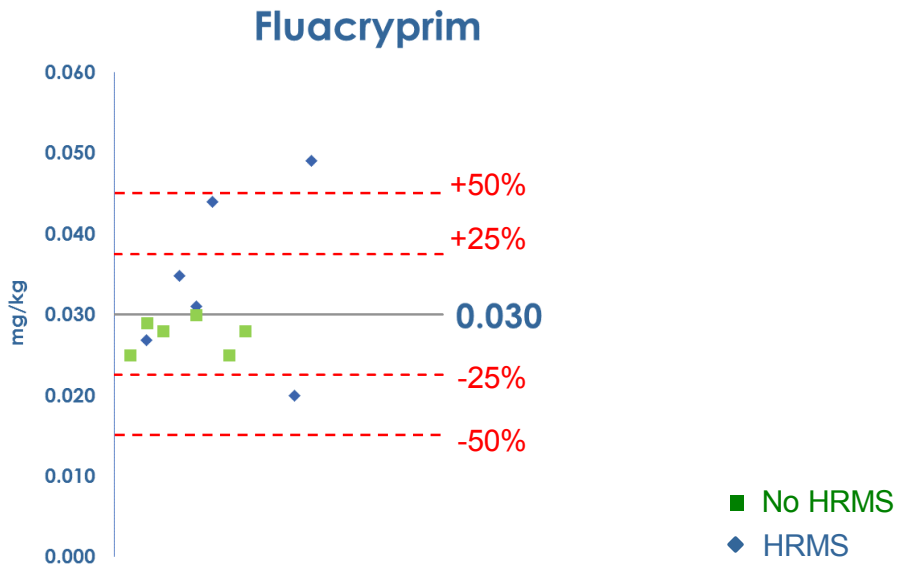


The bold line represents the robust mean

Pesticide	Conc. Homogeneity Test (mg/kg)	Robust Mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Endrin	0.052	0.054	22.1	66	53	85	51



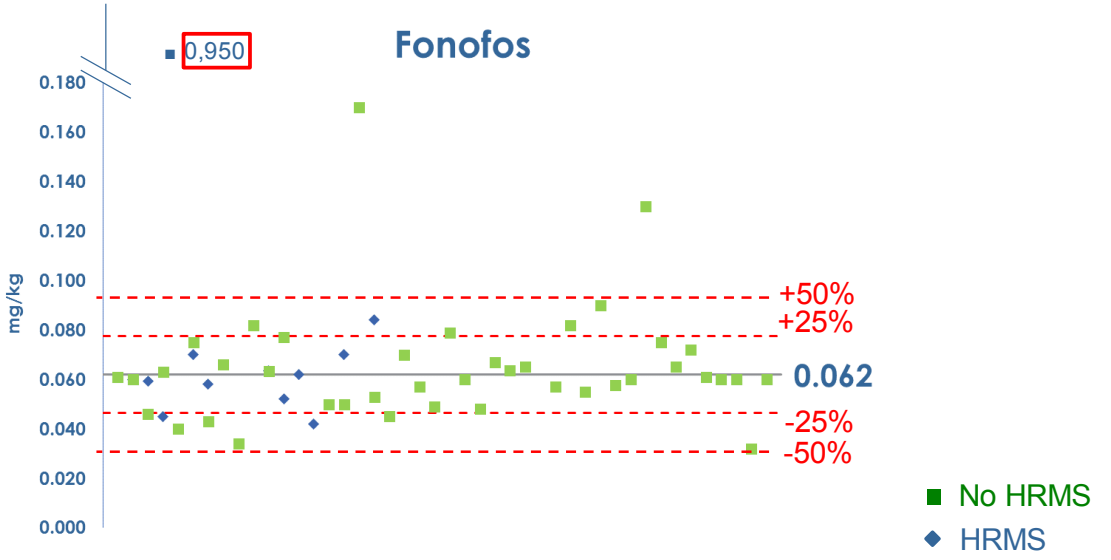
Pesticide	Conc. Homogeneity Test (mg/kg)	Robust Mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Fluacrypyrim	0.032	0.030	22.6	22	20	32	12



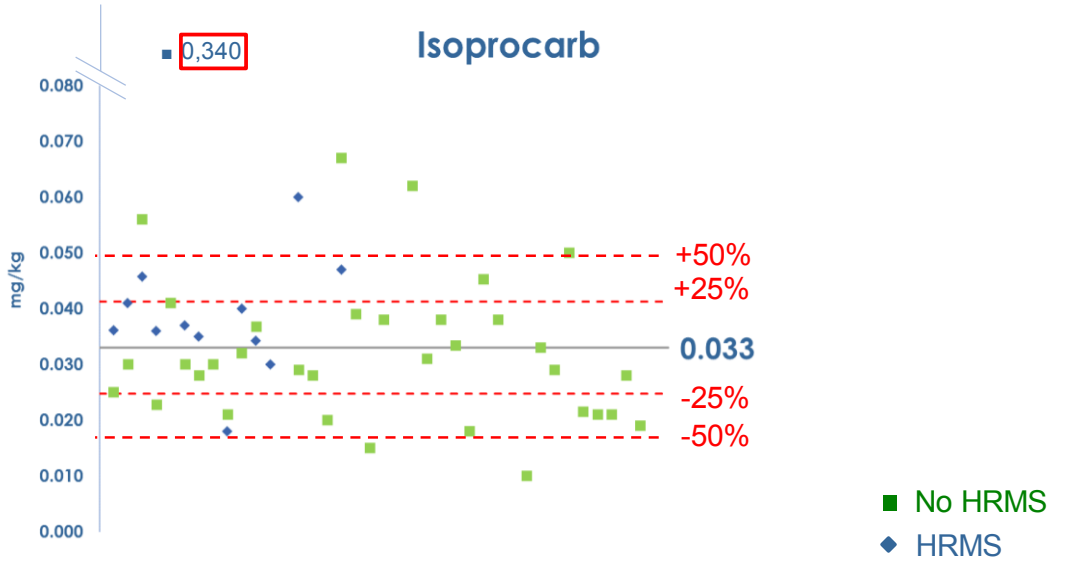
APPENDIX 2. Graphical Representations

The bold line represents the robust mean

Pesticide	Conc. Homogeneity Test (mg/kg)	Robust Mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Fonofos	0.059	0.062	23.2	62	57	92	53

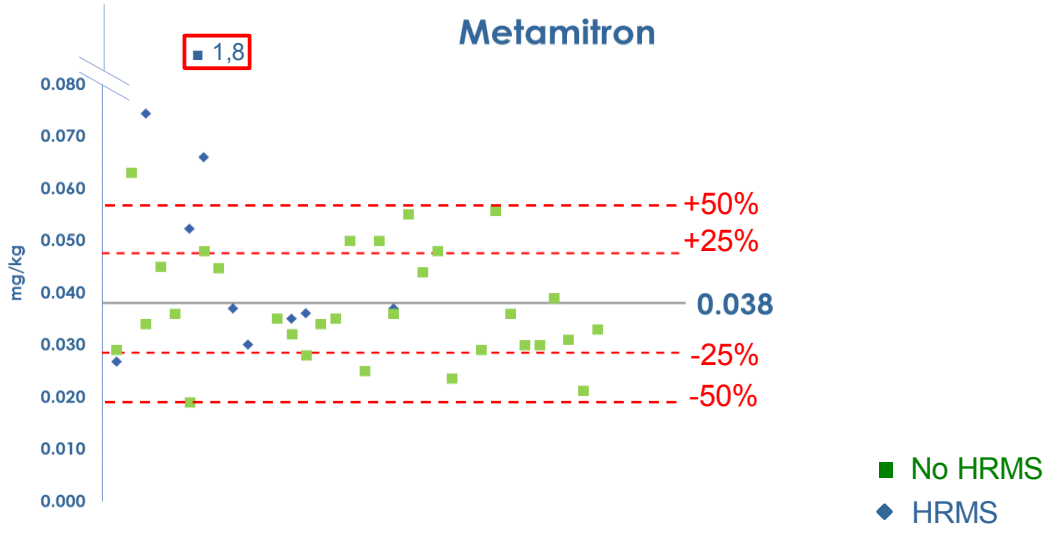


Pesticide	Conc. Homogeneity Test (mg/kg)	Robust Mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Isoprocarb	0.031	0.033	35.9	55	50	81	47

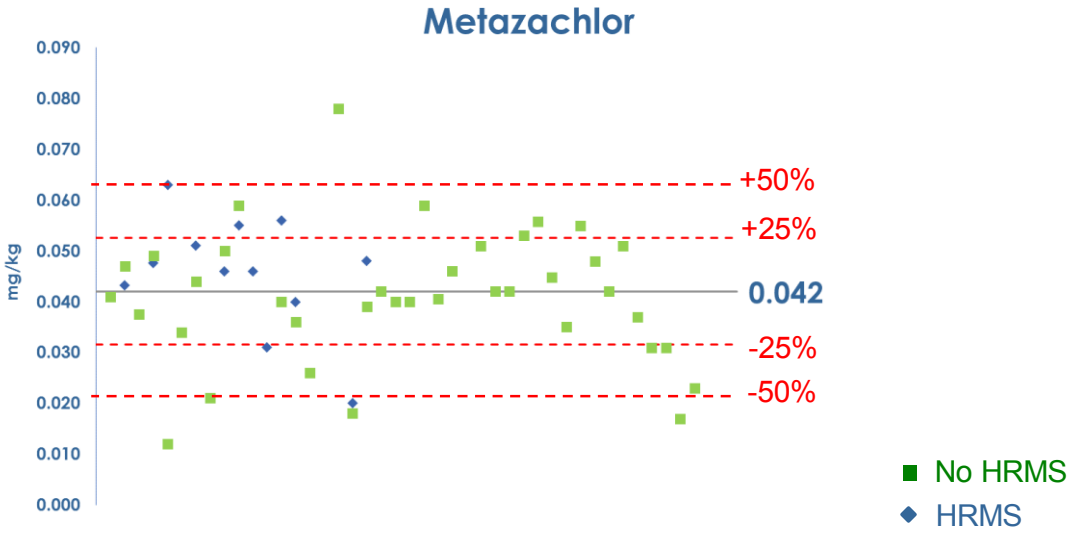


The bold line represents the robust mean

Pesticide	Conc. Homogeneity Test (mg/kg)	Robust Mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Metamitron	0.042	0.038	32.0	57	47	76	40

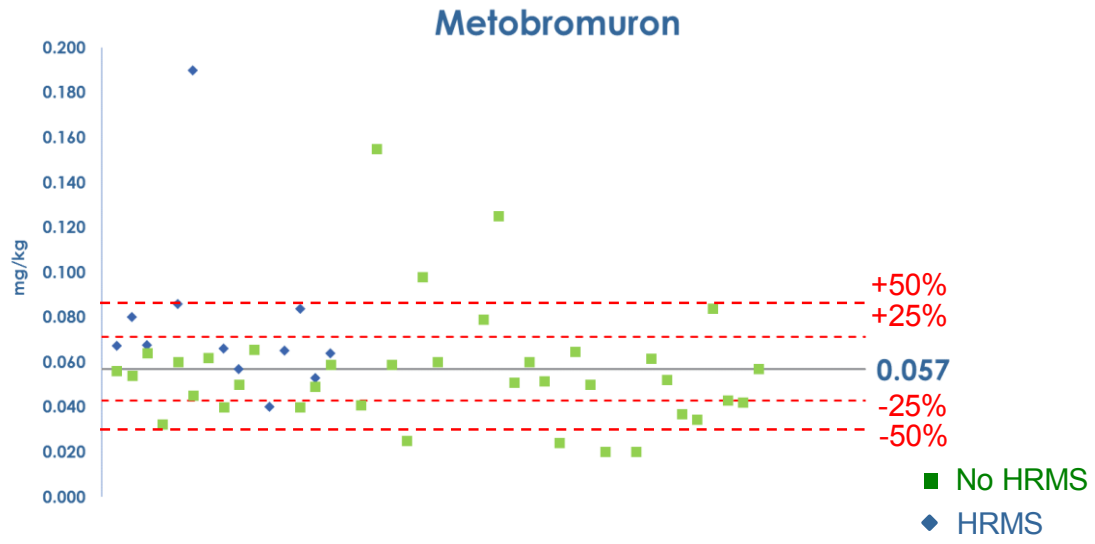


Pesticide	Conc. Homogeneity Test (mg/kg)	Robust Mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Metazachlor	0.044	0.042	27.5	61	56	90	50

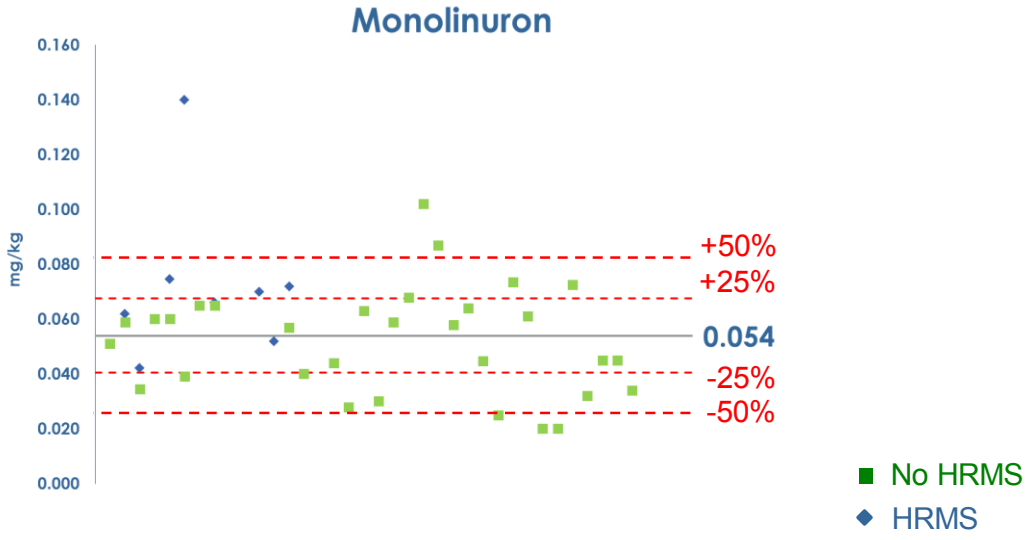


The bold line represents the robust mean

Pesticide	Conc. Homogeneity Test (mg/kg)	Robust Mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Metobromuron	0.057	0.057	34.5	60	55	89	49

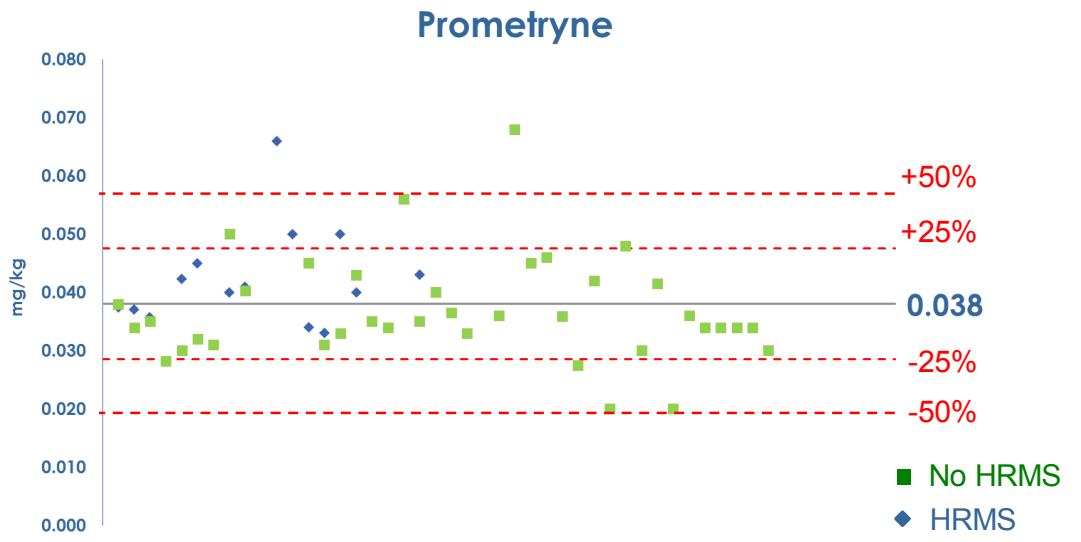


Pesticide	Conc. Homogeneity Test (mg/kg)	Robust Mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Monolinuron	0.059	0.054	36.1	51	47	76	39

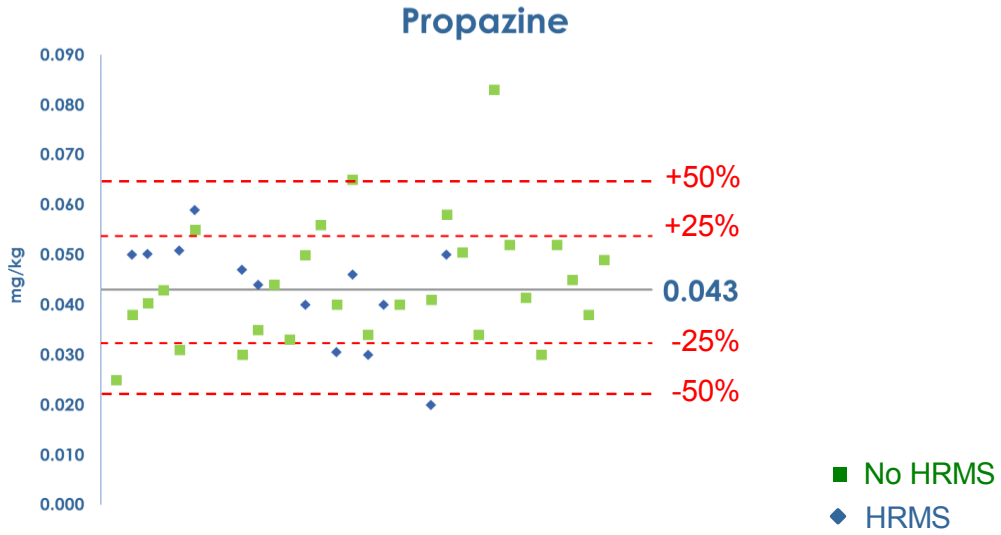


The bold line represents the robust mean

Pesticide	Conc. Homogeneity Test (mg/kg)	Robust Mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Prometryne	0.038	0.038	19.5	62	55	89	52

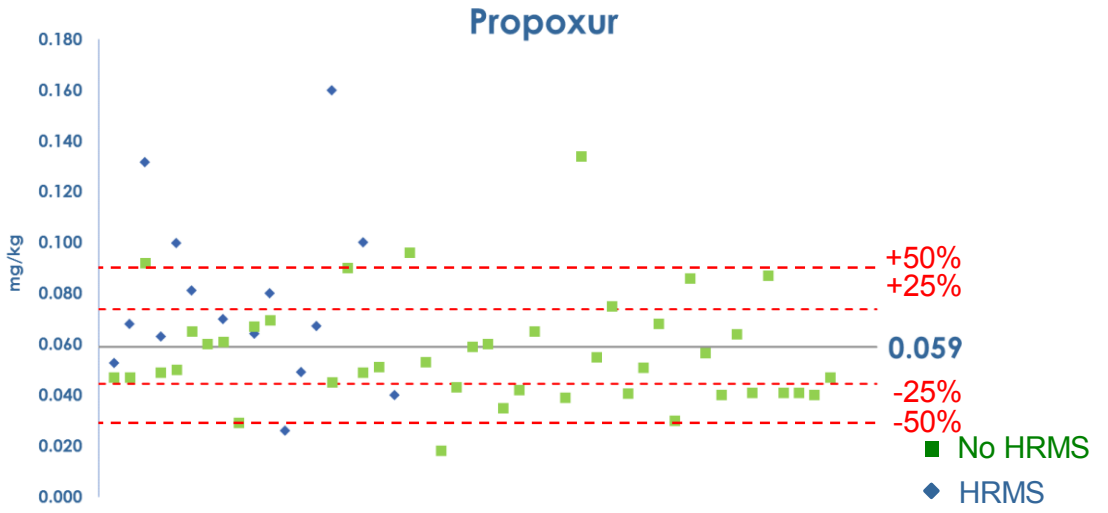


Pesticide	Conc. Homogeneity Test (mg/kg)	Robust Mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Propazine	0.044	0.043	25.0	55	50	81	41

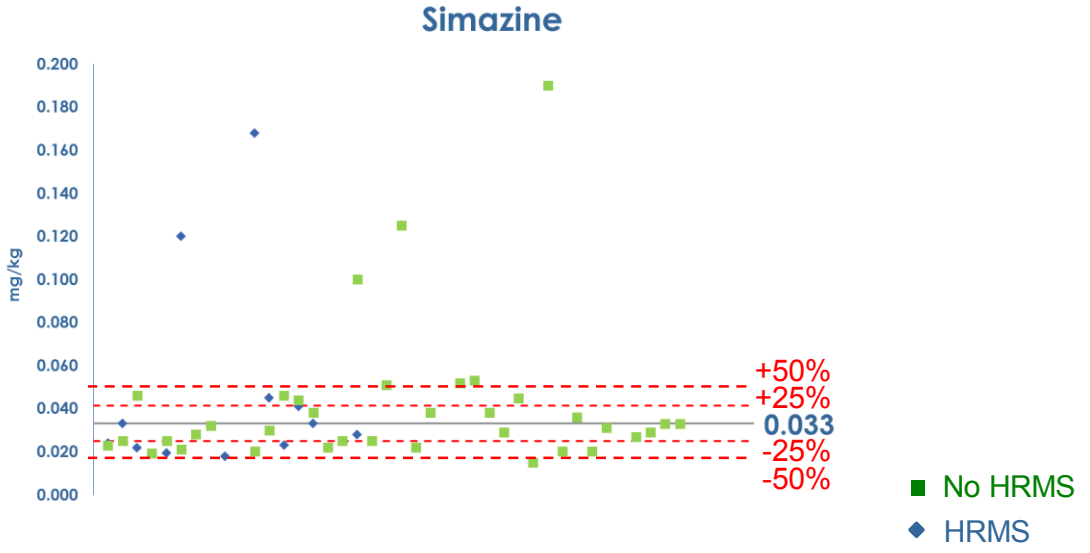


The bold line represents the robust mean

Pesticide	Conc. Homogeneity Test (mg/kg)	Robust Mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Propoxur	0.065	0.059	37.2	66	60	97	57

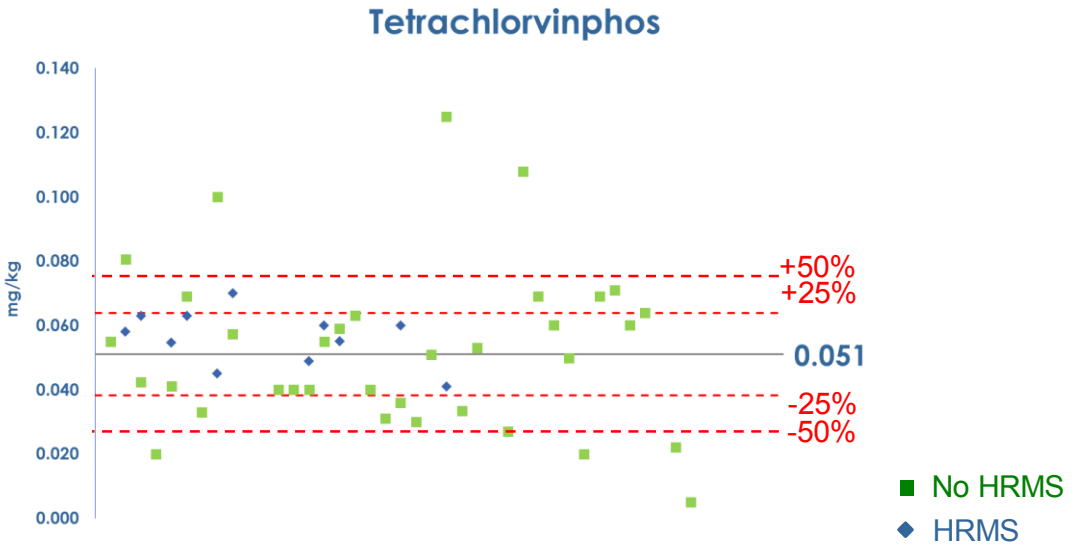


Pesticide	Conc. Homogeneity Test (mg/kg)	Robust Mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Simazine	0.037	0.033	40.2	58	53	85	49

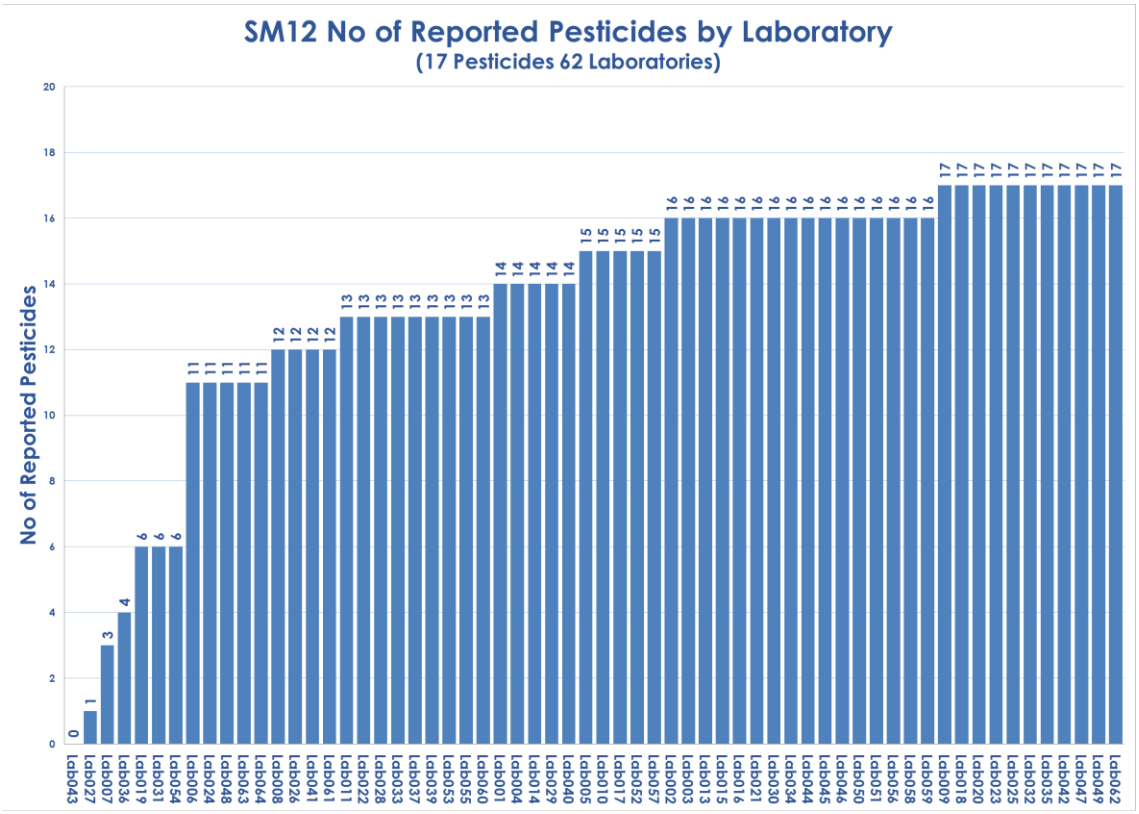


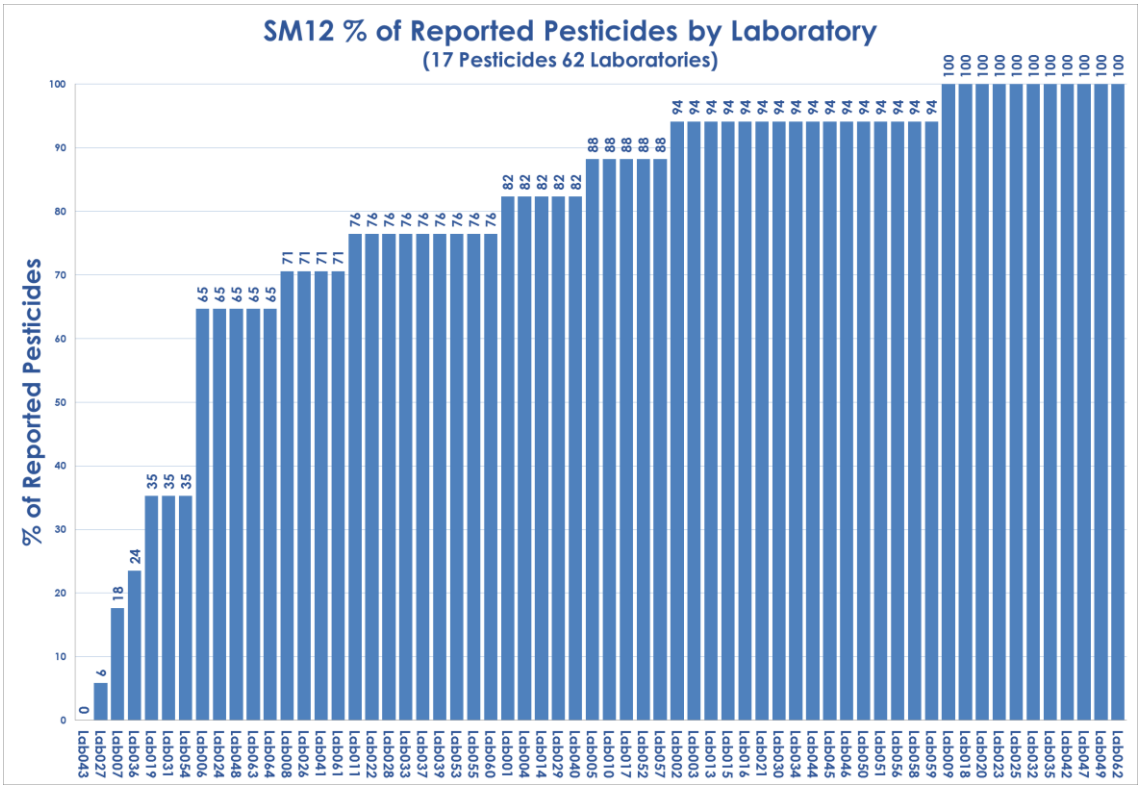
The bold line represents the robust mean

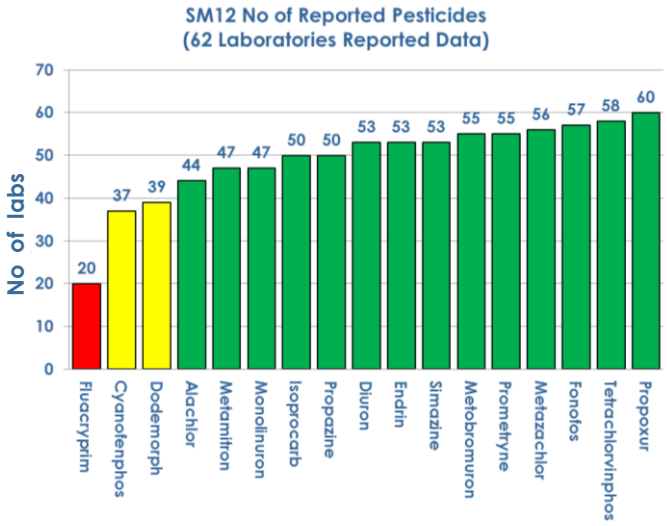
Pesticide	Conc. Homogeneity Test (mg/kg)	Robust Mean (mg/kg)	CV (%)	No of Detections	Nº of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Tetrachlorvinphos	0.054	0.051	36.3	63	58	94	46

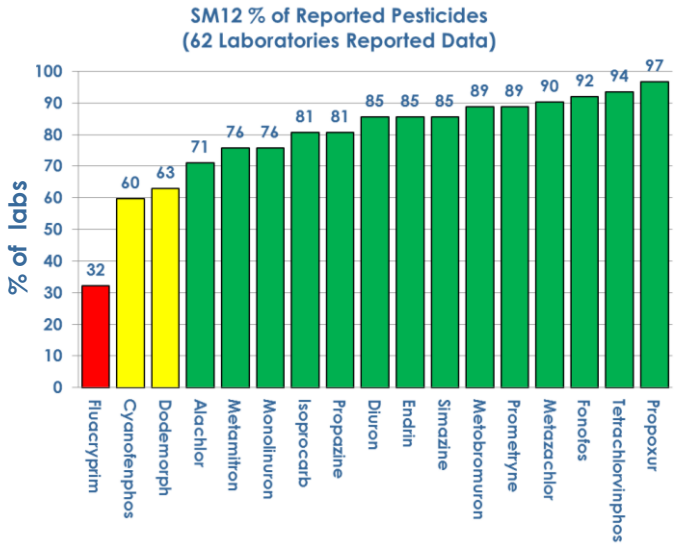


APPENDIX 2. Graphical Representations

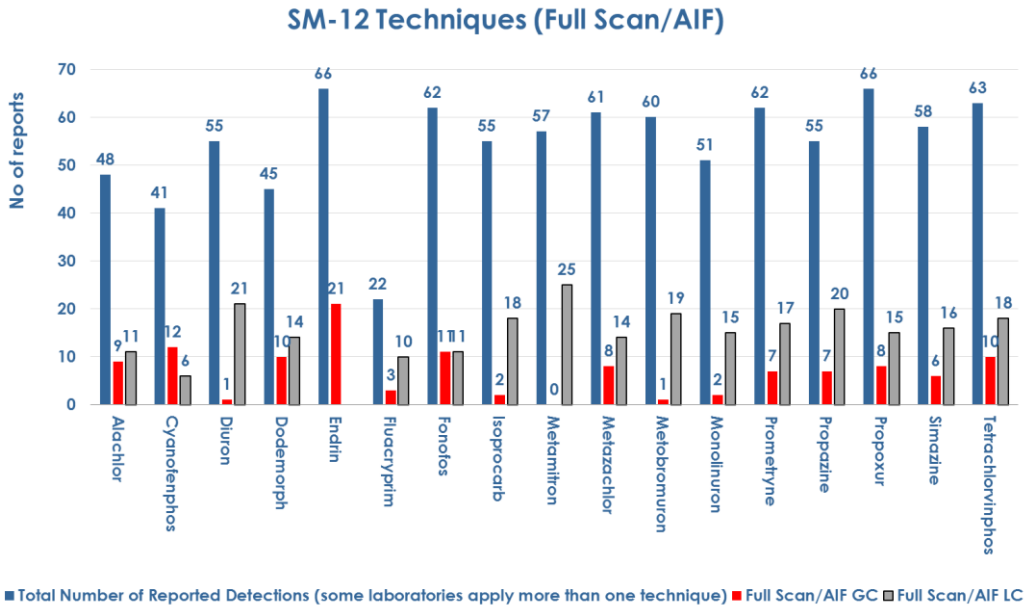








Chromatographic Techniques used in Full Scan/AIF to determine each pesticide in the test item



ANNEX 1. List of Laboratories that reported results in EUPT-FV-SM12.

COUNTRY	LABORATORY NAME	CITY
AUSTRIA	DEPARTMENT FOR PESTICIDE AND FOOD ANALYTICS (PLMA)	INNSBRUCK
BELGIUM	PRIMORIS (PHYTOLAB)	GENT - ZWIJNAARDE
BELGIUM	LOVAP NV	GEEL
CHINA	SCDC - PESTICIDE LAB	SHANGHAI
CHINA	BEIJING UNI-STAR INSPECTION - PESTICIDE LAB	BEIJING
CHINA	AGRO-PRODUCT SAFETY RESEARCH CENTER	BEIJING
CROATIA	SAMPLE CONTROL - PESTICIDE LAB	LUČKO
CZECH REPUBLIC	CAFIA - PESTICIDE LAB	PRAHA
CZECH REPUBLIC	VSCHT / UCT PRAGUE - FOOD ANALYSIS (323)	PRAHA
DENMARK	DTU, NATIONAL FOOD INSTITUTE	LYNGBY
FINLAND	FINNISH CUSTOMS LABORATORY	ESPOO
FRANCE	INOVALYS LE MANS - PESTICIDE LAB	LE MANS
FRANCE	SCL	MONTPELLIER
FRANCE	SCL	MASSY CEDEX
FRANCE	ANSES - LSAL - UNITÉ PBM	MAISONS-ALFORT CEDEX
GERMANY	LAVES - PESTICIDE LAB	OLDENBURG
GERMANY	LUFA KIEL - PESTICIDE LAB	KIEL
GERMANY	LUA SACHSEN - PESTICIDE LAB	DRESDEN
GERMANY	LGL ERLANGEN - PESTICIDE LAB	ERLANGEN
GERMANY	CVUA RRW - PESTICIDE LAB	KREFELD
GERMANY	BVL UNIT 504 NRL FOR PESTICIDE RESIDUES	BERLIN
GERMANY	GALAB LABORATORIES GMBH	HAMBURG
GERMANY	LABOR FRIEDLE	TEGERNHEIM
GERMANY	LALLF - PESTICIDE LAB	ROSTOCK
GERMANY	LTZ AUGUSTENBERG - ORGANIC ANALYSIS	KARLSRUHE
GERMANY	BUNDESWEHR - PESTICIDE LAB	GARCHING-HOCHBRÜCK
GERMANY	CVUA STUTTGART - PESTIS	STUTTGART
GERMANY	ILAU GMBH	ANZING
HUNGARY	NFCSO - PESTICIDE LAB	VELENCE
HUNGARY	NFCSO PESTICIDE LAB	SZOLNOK
HUNGARY	NFCSO PESTICIDE LAB	HÓDMEZOVÁSÁRHELY
IRELAND	PESTICIDE CONTROL LAB	CO. KILDARE
ITALY	IZS SARDEGNA - PESTICIDE LAB	SASSARI
ITALY	IZS LT (SEZIONE FIRENZE) - PESTICIDE LAB	SAN MARTINO ALLA PALMA SCANDICCI (FI)
ITALY	ASF - PESTICIDE LAB	FIRENZE
ITALY	APPA BOLZANO - PESTICIDE LAB	BOLZANO
ITALY	IZS SICILIA - PESTICIDE LAB	PALERMO
ITALY	IZSAM - PESTICIDE LAB	TERAMO

ANNEX 1. List of Laboratories that participate in EUPT-FV-SM12

COUNTRY	LABORATORY NAME	CITY
KENYA	SGS - PESTICIDE LAB	MOMBASA
LATVIA	BIOR - PESTICIDE LAB	RIGA
LITHUANIA	NMVRVI - PESTICIDE LAB	VILNIUS
NORWAY	NIBIO - DEPARTMENT OF PESTICIDE CHEMISTRY	ÅS
SPAIN	LABORATORIO SOIVRE	ALMERÍA
SPAIN	LABORATORIO AGROAMBIENTAL DE ZARAGOZA	ZARAGOZA
SPAIN	LABORATORIO AGROALIMENTARIO DE EXTREMADURA	CÁCERES
SPAIN	EUROFINS ECOSUR - PESTICIDE LAB	LORQUÍ
SPAIN	NATIONAL CENTER FOR TECHNOLOGY AND FOOD SAFETY	SAN ADRIÁN (NAVARRA)
SPAIN	SALUD PUBLICA	MADRID
SPAIN	ANALYTICA ALIMENTARIA GMBH	ALMERIA
SPAIN	LABORATORIO AGROALIMENTARIO Y DE SANIDAD ANIMAL	MURCIA
SPAIN	LARAGA - PESTICIDE LAB	TOLEDO
SPAIN	AGRICULTURAL AND PHYTOPATHOLOGICAL LAB. OF GALICIA	ABEGONDO. A CORUÑA
SPAIN	EUROFINS SICA AGRIQ	ALMERIA
SPAIN	LABORATORIO ANALÍTICO BIOCLÍNICO	ALMERIA
SPAIN	LABORATORIO KUDAM, S.L.	PILAR DE LA HORADADA (ALICANTE)
SWEDEN	EUROFINS FOOD & FEED - PESTICIDE LAB	LIDKÖPING
SWEDEN	NATIONAL FOOD AGENCY	UPPSALA
SWITZERLAND	SCAV - PESTICIDE LAB	GENEVE
SWITZERLAND	KANTONALES LABOR ZURICH	ZURICH
THE NETHERLANDS	GROEN AGRO CONTROL	DELFGAUW
THE NETHERLANDS	WFSR - NRL FOR PESTICIDES	WAGENINGEN
THE NETHERLANDS	NOFALAB B.V.	SCHIEDAM
TURKEY	SGS - FOOD CONTROL LABORATORY	MERSIN
UNITED KINGDOM	FERA - PESTICIDE LAB	YORK