

SCREENING METHODS 13 (EUPT-FV-SM13)

Pesticide Residues in Aubergine Homogenate

Final Report

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EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUIT AND VEGETABLES. SCREENING METHODS 13

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 origin¹: 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 625/2017² 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.

¹Regulation (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.

²Regulation (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-SM13 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 aubergine homogenate. The aubergines were cultivated in a greenhouse in Almería, Spain.

The pesticides used to spike the aubergine 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-SM13 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 pesticides used to spike the aubergine 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 aubergine 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		
3-Hydroxy Carbofuran	Fenobucarb	Omethoate
Acephate	Fenpicoxamid	Penthiopyrad
Aclonifen	Isoprothiolane	Picolinafen
Acrinathrin	Mefentrifluconazole	Profenofos
Cyantraniliprole	Molinate	Sulfoxaflor
Dinotefuran	Novaluron	Triflumizole

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 AOAC³. 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 $c = F_1\sigma_{all}^2 + F_2S_{an}^2$; F_1 and F_2 being constant values of 1.88 and 1.01, respectively, from the ten samples taken, and $\sigma_{all}^2 = 0.3 \times \text{FFP RSD}(25\%) \times \text{the analytical sampling mean for all the pesticides}$. This was used to demonstrate that the between-bottle variance was not higher than the within-bottle variance.

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
3-Hydroxy Carbofuran	0.057	0	0.00020	Pass
Acephate	0.094	0	0.00185	Pass
Aclonifen	0.068	0.00010	0.00022	Pass
Acrinathrin	0.098	0	0.00281	Pass
Cyantraniliprole	0.055	0.00001	0.00020	Pass
Dinotefuran	0.047	0	0.00036	Pass
Fenobucarb	0.095	0.00007	0.00035	Pass
Fenpicoxamid	0.061	0	0.00284	Pass
Isoprothiolane	0.070	0	0.00013	Pass
Mefentrifluconazole	0.029	0.00001	0.00005	Pass
Molinate	0.083	0	0.00031	Pass
Novaluron	0.075	0.00016	0.00049	Pass
Omethoate	0.068	0	0.00051	Pass
Penthiopyrad	0.071	0.00003	0.00018	Pass
Picolinafen	0.099	0.00016	0.00037	Pass
Profenofos	0.067	0	0.00019	Pass
Sulfoxaflor	0.048	0	0.00017	Pass
Triflumizole	0.010	0.00001	0.00001	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 Almeria. 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 $|x_1 - y_i| \leq 0.3 \times \sigma$, where x_1 is the mean value of the Day 1 stability test, y_i 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							(M2 - M1)	M2-M1 ≤ 0.3*σ
	Sample 02_A	Sample 02_B	Sample 75_A	Sample 75_B	Sample 09_A	Sample 09_B	Mean 1	Sample 05_A	Sample 05_B	Sample 59_A	Sample 59_B	Sample 33_A	Sample 33_B	Mean2		
3-Hydroxy Carbofuran	0.052	0.064	0.054	0.051	0.055	0.067	0.057	0.056	0.076	0.042	0.047	0.067	0.055	0.057	0.000	Pass
Acephate	0.091	0.095	0.085	0.083	0.097	0.097	0.091	0.074	0.095	0.099	0.069	0.100	0.099	0.090	-0.002	Pass
Aclonifen	0.072	0.073	0.063	0.072	0.082	0.062	0.071	0.085	0.098	0.046	0.055	0.062	0.056	0.067	-0.004	Pass
Acrinathrin	0.102	0.099	0.095	0.086	0.088	0.090	0.093	0.089	0.095	0.090	0.085	0.082	0.084	0.088	-0.006	Pass
Cyantraniliprole	0.057	0.054	0.050	0.052	0.055	0.060	0.055	0.055	0.042	0.050	0.055	0.063	0.041	0.051	-0.004	Pass
Dinotefuran	0.057	0.041	0.045	0.040	0.045	0.055	0.047	0.043	0.051	0.031	0.035	0.060	0.055	0.046	-0.001	Pass
Fenobucarb	0.098	0.095	0.094	0.092	0.100	0.098	0.097	0.088	0.095	0.080	0.099	0.095	0.085	0.090	-0.006	Pass
Fenpicoxamid	0.065	0.060	0.050	0.068	0.058	0.050	0.058	0.064	0.055	0.050	0.040	0.065	0.063	0.056	-0.002	Pass
Isoprothiolane	0.075	0.066	0.063	0.076	0.061	0.081	0.070	0.067	0.066	0.065	0.065	0.063	0.070	0.066	-0.004	Pass
Mefentrifluconazole	0.046	0.024	0.026	0.030	0.020	0.029	0.029	0.029	0.022	0.027	0.029	0.026	0.029	0.027	-0.002	Pass
Molinate	0.080	0.087	0.081	0.084	0.082	0.087	0.084	0.088	0.079	0.064	0.085	0.088	0.076	0.080	-0.004	Pass
Novaluron	0.088	0.067	0.073	0.077	0.059	0.079	0.074	0.110	0.074	0.054	0.063	0.070	0.044	0.069	-0.005	Pass
Omethoate	0.064	0.065	0.060	0.062	0.068	0.065	0.064	0.064	0.068	0.070	0.069	0.076	0.065	0.069	0.005	Pass
Penthiopyrad	0.067	0.075	0.065	0.068	0.065	0.069	0.068	0.061	0.057	0.066	0.071	0.072	0.055	0.064	-0.005	Pass
Picolinafen	0.096	0.110	0.100	0.110	0.097	0.100	0.100	0.095	0.091	0.096	0.100	0.085	0.099	0.094	-0.005	Pass
Profenofos	0.065	0.072	0.063	0.068	0.071	0.069	0.068	0.069	0.062	0.064	0.060	0.065	0.070	0.065	-0.003	Pass
Sulfoxaflor	0.050	0.052	0.049	0.030	0.044	0.061	0.048	0.040	0.073	0.038	0.033	0.067	0.041	0.049	0.001	Pass
Triflumizole	0.008	0.010	0.010	0.012	0.009	0.008	0.009	0.017	0.010	0.005	0.007	0.010	0.010	0.010	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 02_A	Sample 02_B	Sample 75_A	Sample 75_B	Sample 09_A	Sample 09_B	Mean 1	Sample 120_A	Sample 120_B	Sample 78_A	Sample 78_B	Sample 103_A			Sample 103_B	Mean2
3-Hydroxy Carbofuran	0.052	0.064	0.054	0.051	0.055	0.067	0.057	0.069	0.055	0.055	0.055	0.047	0.070	0.059	0.001	Pass
Acephate	0.091	0.095	0.085	0.083	0.097	0.097	0.091	0.092	0.100	0.095	0.100	0.090	0.097	0.097	0.005	Pass
Aclonifen	0.072	0.073	0.063	0.072	0.082	0.062	0.071	0.110	0.068	0.066	0.043	0.061	0.060	0.068	-0.003	Pass
Acrinathrin	0.102	0.099	0.095	0.086	0.088	0.090	0.093	0.101	0.092	0.088	0.100	0.089	0.085	0.093	-0.002	Pass
Cyantraniliprole	0.057	0.054	0.050	0.052	0.055	0.060	0.055	0.055	0.060	0.051	0.050	0.052	0.067	0.056	0.001	Pass
Dinotefuran	0.057	0.041	0.045	0.040	0.045	0.055	0.047	0.046	0.050	0.052	0.055	0.048	0.049	0.050	0.003	Pass
Fenobucarb	0.098	0.095	0.094	0.092	0.100	0.098	0.097	0.110	0.110	0.085	0.095	0.099	0.098	0.100	0.001	Pass
Fenpicoxamid	0.065	0.060	0.050	0.068	0.058	0.050	0.058	0.070	0.065	0.058	0.062	0.060	0.056	0.062	0.003	Pass
Isoprothiolane	0.075	0.066	0.063	0.076	0.061	0.081	0.070	0.086	0.065	0.066	0.065	0.075	0.076	0.072	0.000	Pass
Mefentrifluconazole	0.046	0.024	0.026	0.030	0.020	0.029	0.029	0.044	0.035	0.020	0.025	0.030	0.033	0.031	0.002	Pass
Molinate	0.080	0.087	0.081	0.084	0.082	0.087	0.084	0.089	0.100	0.053	0.085	0.074	0.100	0.084	-0.002	Pass
Novaluron	0.088	0.067	0.073	0.077	0.059	0.079	0.074	0.150	0.083	0.047	0.060	0.049	0.077	0.078	0.004	Pass
Omethoate	0.064	0.065	0.060	0.062	0.068	0.065	0.064	0.072	0.075	0.065	0.060	0.062	0.069	0.067	0.005	Pass
Penthiopyrad	0.067	0.075	0.065	0.068	0.065	0.069	0.068	0.098	0.075	0.066	0.065	0.078	0.070	0.075	0.004	Pass
Picolinafen	0.096	0.110	0.100	0.110	0.097	0.100	0.100	0.140	0.110	0.092	0.085	0.100	0.110	0.108	0.007	Pass
Profenofos	0.065	0.072	0.063	0.068	0.071	0.069	0.068	0.097	0.055	0.057	0.065	0.060	0.075	0.068	-0.002	Pass
Sulfoxaflor	0.050	0.052	0.049	0.030	0.044	0.061	0.048	0.057	0.053	0.041	0.050	0.035	0.054	0.048	0.001	Pass
Triflumizole	0.008	0.010	0.010	0.012	0.009	0.008	0.009	0.011	0.012	0.010	0.010	0.010	0.005	0.010	0.000	Pass

2.3 Distribution of test item and protocol to participants

Approximately 200 g of treated aubergine homogenate were shipped to participants on 1st February 2021. 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-SM13 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-SM13 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 *
3-Hydroxy Carbofuran	59	98	1	2
Acephate	59	98	1	2
Aclonifen	51	85	9	15
Acrinathrin	58	97	2	3
Cyantraniliprole	47	78	13	22
Dinotefuran	47	78	13	22
Fenobucarb	55	92	5	8
Fenpicoxamid	32	53	28	47
Isoprothiolane	58	97	2	3
Mefentrifluconazole	26	43	34	57
Molinate	52	87	8	13
Novaluron	54	90	6	10
Omethoate	59	98	1	2
Penthiopyrad	55	92	5	8
Picolinafen	55	92	5	8
Profenofos	59	98	1	2
Sulfoxaflor	52	87	8	13
Triflumizole	52	87	8	13

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

In this EUPT-FV-SM13, 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 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.

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 (%)
3-Hydroxy Carbofuran	0.057	0.053	61	21.5
Acephate	0.094	0.084	61	32.6
Aclonifen	0.068	0.066	51	31.4
Acrinathrin	0.098	0.096	58	43.1
Cyantranilprole	0.055	0.054	46	23.0
Dinotefuran	0.047	0.044	48	24.4
Fenobucarb	0.095	0.090	53	15.9
Fenpicoxamid	0.061	0.043	30	25.4
Isoprothiolane	0.070	0.066	61	13.2
Mefentrifluconazole	0.029	0.027	23	19.1
Molinate	0.083	0.081	53	18.2
Novaluron	0.075	0.069	53	28.1
Omethoate	0.068	0.066	61	30.2
Penthiopyrad	0.071	0.069	57	19.5
Picolinafen	0.099	0.096	56	21.9
Profenofos	0.067	0.066	61	18.1
Sulfoxaflor	0.048	0.047	52	23.5
Triflumizole	0.010	0.010	46	34.1

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)
Lab004	Fonofos	0.08
Lab006	Bromide	1.404
	Chlorate	0.016
	Perchlorate	0.008
	Phosphonic acid	0.029
Lab015	Quinoclamine	0.076
Lab021	Cyhalothrin	
	Diamidafos	
	Fenpropimorph	
	Metolachlor CGA 50267	
	Promecarb	
	S-Abscisic Acid	
	Tetramethrin	

Laboratory Code	Other Reported Compounds	Concentration Reported (mg/kg)
Lab024	Dimethoate	0.02
Lab028	Aldimorph	
	Cyhalothrin, lambda-	0.09
	Dodemorph	
	Empenthrin	
	Santonin	
Lab030	Benzisothiazolinone	0.008
Lab038	DMPF	0.0390
Lab044	Allethrin	0.430
Lab049	Fosetyl-Aluminium (sum)	0.280
	Phosphonic acid	0.210
Lab060	Isocarbophos	
	Methidathion	
Lab061	Diflubenzuron	0.022
Lab062	Didecyldimethylammoniumchloride (DDAC)	0.14

None of the pesticides were reported by three or more laboratories.

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
Lab036	18	100	
Lab064	18	100	
Lab029	18	100	
Lab001	18	100	
Lab009	18	100	
Lab010	18	100	
Lab013	18	100	
Lab016	18	100	
lab018	18	100	
Lab019	18	100	
Lab022	18	100	
Lab023	18	100	
Lab027	18	100	
Lab034	18	100	
Lab040	18	100	
Lab041	18	100	
Lab043	18	100	
Lab045	18	100	
Lab047	18	100	
Lab048	18	100	
Lab058	18	100	
Lab062	18	100	1

Laboratory Code	No of Reported Pesticides	% of Reported Pesticides	Other Reported Pesticides Not Confirmed by the Organiser
Lab021	18	100	7
Lab051	17	94	
Lab056	17	94	
Lab044	17	94	1
Lab031	16	89	
Lab002	16	89	
Lab003	16	89	
Lab012	16	89	
Lab020	16	89	
Lab025	16	89	
Lab032	16	89	
Lab033	16	89	
Lab053	16	89	
Lab055	16	89	
Lab059	16	89	
Lab030	16	89	1
Lab014	15	83	
Lab063	15	83	
Lab015	15	83	1
Lab038	15	83	1
Lab049	15	83	2
Lab006	15	83	4
Lab011	14	78	
Lab042	14	78	
Lab054	14	78	
Lab065	14	78	
Lab057	13	72	
Lab026	12	67	
Lab037	12	67	
Lab052	12	67	
Lab028	12	67	5
Lab004	11	61	1
Lab061	11	61	1
Lab060	11	61	2
Lab005	10	56	
Lab046	10	56	
Lab024	7	39	1
Lab017	6	33	

The extraction methods used by the laboratories, the chromatographic techniques, detectors, instrumentation, etc... are detailed in Appendix 3 (available only on the EUPT-FV-SM13 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 (18 Evaluated Pesticides)	% of Present Pesticides Reported (18 Evaluated Pesticides)
Lab001	18	100
Lab002	16	89
Lab003	16	89
Lab004	11	61
Lab005	10	56
Lab006	15	83
Lab009	18	100
Lab010	18	100
Lab011	14	78
Lab012	16	89
Lab013	18	100
Lab014	15	83
Lab015	15	83
Lab016	18	100
Lab017	6	33
lab018	18	100
Lab019	18	100
Lab020	16	89
Lab021	18	100
Lab022	18	100
Lab023	18	100
Lab024	7	39
Lab025	16	89
Lab026	12	67
Lab027	18	100
Lab028	12	67
Lab029	18	100
Lab030	16	89
Lab031	16	89
Lab032	16	89
Lab033	16	89
Lab034	18	100
Lab036	18	100
Lab037	12	67
Lab038	15	83
Lab040	18	100
Lab041	18	100
Lab042	14	78
Lab043	18	100
Lab044	17	94
Lab045	18	100
Lab046	10	56
Lab047	18	100
Lab048	18	100
Lab049	15	83
Lab051	17	94
Lab052	12	67

Laboratory Code	Number of Present Pesticides Reported (18 Evaluated Pesticides)	% of Present Pesticides Reported (18 Evaluated Pesticides)
Lab053	16	89
Lab054	14	78
Lab055	16	89
Lab056	17	94
Lab057	13	72
Lab058	18	100
Lab059	16	89
Lab060	11	61
Lab061	11	61
Lab062	18	100
Lab063	15	83
Lab064	18	100
Lab065	14	78

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
3-Hydroxy Carbofuran	59	65	2	2	63	15
Acephate	59	65	5	2	60	14
Aclonifen	51	55	39	9	16	5
Acrinathrin	58	63	51	13	12	5
Cyantraniliprole	47	52	2	2	50	15
Dinotefuran	47	52	2	2	50	13
Fenobucarb	55	62	20	6	42	13
Fenpicoxamid	32	36	1	1	35	12
Isoprothiolane	58	66	18	5	48	13
Mefentrifluconazole	26	30	6	1	24	9
Molinate	52	60	23	8	37	11
Novaluron	54	59	3	2	56	13
Omethoate	59	65	5	2	60	14
Penthiopyrad	55	61	14	6	47	12
Picolinafen	55	63	23	7	40	14
Profenofos	59	66	32	5	34	13
Sulfoxaflor	52	57	2	2	55	13
Triflumizole	52	58	13	6	45	9

*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.

Sixteen EU Member States, 2 EFTA countries (Norway and Switzerland) and two non-EU/EFTA countries (China and Perú) 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-SM13 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 1035; 261 were made by GC and 774 by LC. 28% of the detections were made using full-scan or all ion fragmentation (AIF)(81 by GC-full scan/AIF techniques and 213 by LC-full scan/AIF techniques). 37% of the laboratories reported their results using HRMS and 931 of the results were reported indicating a concentration value (90% of the total results).

Twenty-three of the 60 laboratories were able to detect all 18 present pesticides in the test item. Two laboratories detected less than 50% of the pesticides present. Eighty-two percent of the laboratories (49 laboratories) that reported results were able to detect more than 70 % of the evaluated pesticides.

No other compound apart from the present pesticides was reported by three or more laboratories.

Thirteen participants reported 26 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.

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- 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

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APPENDIX 1. Results

Table AP1.1. Reported pesticides

Laboratory Code Total No of Reporting Laboratories = 60	Evaluated Pesticides (18)																		Reported Pesticides by Laboratory	% Reported Pesticides by Laboratory
	3-Hydroxy Carbofuran	Acephate	Aclonifen	Acrinathrin	Cyrantranilprole	Dinotefuran	Fenobucarb	Fenpicoxamid	Isoprothiolane	Mefentrifluconazole	Molinate	Novaluron	Omethoate	Penthiopyrad	Picolinafen	Profenofos	Sulfoxaflor	Triflumizole		
Lab001	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100
Lab002	R	R	R	R		R	R	R	R		R	R	R	R	R	R	R	R	16	89
Lab003	R	R	R	R	R	R	R		R		R	R	R	R	R	R	R	R	16	89
Lab004	R	R		R		R	R		R			R	R	R			R	R	11	61
Lab005	R	R	R	R			R		R			R	R		R	R			10	56
Lab006	R	R	R	R	R	R	R		R		R	R	R	R		R	R	R	15	83
Lab009	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100
Lab010	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100
Lab011	R	R		R	R		R		R		R	R	R	R	R	R	R	R	14	78
Lab012	R	R	R	R	R	R	R		R		R	R	R	R	R	R	R	R	16	89
Lab013	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100
Lab014	R	R	R	R	R	R	R		R		R		R	R	R	R	R	R	15	83
Lab015	R	R	R	R	R		R		R		R	R	R	R	R	R	R	R	15	83
Lab016	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100
Lab017				R			R				R		R		R	R			6	33
lab018	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100
Lab019	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100
Lab020	R	R	R	R	R		R	R	R		R	R	R	R	R	R	R	R	16	89
Lab021	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100
Lab022	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100
Lab023	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100
Lab024	R	R				R	R					R				R		R	7	39
Lab025	R	R	R	R	R		R	R	R		R	R	R	R	R	R	R	R	16	89
Lab026	R	R	R	R		R			R		R	R	R	R		R		R	12	67
Lab027	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100
Lab028	R	R		R			R		R			R	R	R	R	R	R	R	12	67
Lab029	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100
Lab030	R	R	R	R	R	R	R		R		R	R	R	R	R	R	R	R	16	89
Lab031	R	R	R	R	R	R	R		R		R	R	R	R	R	R	R	R	16	89
Lab032	R	R	R	R	R	R	R		R		R	R	R	R	R	R	R	R	16	89
Lab033	R	R	R	R		R	R	R	R		R	R	R	R	R	R	R	R	16	89
Lab034	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100
Lab036	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100
Lab037	R	R		R	R	R			R			R	R	R		R	R	R	12	67
Lab038	R	R	R	R	R		R	R	R	R		R	R	R	R	R	R	R	15	83
Lab040	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100
Lab041	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100
Lab042	R	R	R	R	R		R		R		R	R	R	R	R	R	R	R	14	78
Lab043	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100
Lab044	R	R	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	17	94
Lab045	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100
Lab046	R	R	R	R			R		R		R		R		R	R			10	56

APPENDIX 1. Results

Laboratory Code Total No of Reporting Laboratories = 60	Evaluated Pesticides (18)																		R: Reported Pesticide	Reported Pesticides by Laboratory	% Reported Pesticides by Laboratory
	3-Hydroxy Carbofuran	Acephate	Aclonifen	Acrinathrin	Cyantiranilprole	Dinoteturan	Fenobucarb	Fenpicoxamid	Isoprothiolane	Mefenitrufluconazole	Molinate	Novaluron	Omethoate	Penthiopyrad	Picollinafen	Profenofos	Sulfoxaflor	Triflumizole			
Lab047	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100	
Lab048	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100	
Lab049	R	R	R		R	R	R		R		R	R	R	R	R	R	R	R	15	83	
Lab051	R	R	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	17	94	
Lab052	R	R		R		R	R	R	R		R		R	R	R	R			12	67	
Lab053	R	R	R	R	R	R	R	R	R			R	R	R	R	R	R	R	16	89	
Lab054	R	R	R	R			R		R		R	R	R	R	R	R	R	R	14	78	
Lab055	R	R	R	R	R	R	R		R		R	R	R	R	R	R	R	R	16	89	
Lab056	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	R	R	17	94	
Lab057	R	R	R	R	R	R			R		R	R	R	R	R	R			13	72	
Lab058	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100	
Lab059	R	R	R	R	R		R	R	R		R	R	R	R	R	R	R	R	16	89	
Lab060	R	R		R		R	R		R		R	R	R			R		R	11	61	
Lab061	R	R	R	R	R				R				R	R	R	R	R		11	61	
Lab062	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100	
Lab063	R	R	R	R	R	R			R		R	R	R	R	R	R	R	R	15	83	
Lab064	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	18	100	
Lab065	R	R		R		R	R		R		R	R	R	R	R	R	R	R	14	78	
Reported Pesticides	59	59	51	58	47	47	55	32	58	26	52	54	59	55	55	59	52	52			
% of Reported Pesticides	98	98	85	97	78	78	92	53	97	43	87	90	98	92	92	98	87	87			

Table AP1.2. Estimated Concentrations Reported on a voluntary basis (only informative purposes)
 Not all the laboratories reporting results have reported estimated concentration values
 Some Laboratories reported more than one result for the same pesticide.

Evaluated Pesticides (18)																		
LABORATORY CODE Total No of Reporting Laboratories = 60	3-Hydroxy Carbofuran	Acephate	Aclonifen	Acrinathrin	Cyantianilprole	Dinotefuran	Fenobucarb	Fenpicoxamid	Isoprothiolane	Metentrifluconazole	Molinate	Novaluron	Omethoate	Penthiopyrad	Picolinafen	Profenofos	Sulfoxaflor	Triflumizole
Average concentration Homogeneity test (mg/kg)	0.057	0.094	0.068	0.098	0.055	0.047	0.095	0.061	0.07	0.029	0.083	0.075	0.068	0.071	0.099	0.067	0.048	0.01
Robust mean of estimated concentrations reported (mg/kg)	0.053	0.084	0.066	0.096	0.054	0.044	0.09	0.043	0.066	0.027	0.081	0.069	0.066	0.069	0.096	0.066	0.047	0.01
CV (%)	21.5	32.6	31.4	43.1	23	24.4	15.9	25.4	13.2	19.1	18.2	28.1	30.2	19.5	21.9	18.1	23.5	34.1
Lab001	0.0515	0.0912	0.0723	0.1117	0.0574	0.0769	0.1277	0.0649	0.0874	0.0456	0.0991	0.0878	0.0605	0.0666	0.0963	0.0649	0.0496	0.0045
Lab002	0.048 0.049	0.071 0.072	0.056	0.098 0.067		0.043 0.042	0.082 0.091	0.038 0.038	0.063 0.070		0.075 0.081	0.062 0.061	0.057 0.057	0.068 0.071	0.087 0.097	0.066 0.066	0.039 0.042	0.008 0.008
Lab003	0.0536	0.0947	0.0406	0.0479	0.0552	0.0517	0.0804		0.0513		0.068	0.0261	0.0711	0.0929	0.0592	0.0442	0.0406	0.008
Lab004	0.051	0.102		0.13		0.045	0.095		0.07				0.069	0.08	0.11		0.046	0.01
Lab005	0.0477	0.1189	0.099	0.154			0.0765		0.06933333			0.0647	0.0572		0.0938	0.0726		
Lab006	0.048	0.072	0.105	0.138	0.046	0.04	0.101		0.057		0.075	0.065	0.05	0.051		0.115	0.039	0.01
Lab009	0.0503	0.0829	0.0723	0.0306	0.0558	0.0485	0.0924	0.0316	0.071	0.0256	0.0793	0.068	0.0619	0.0706	0.1088	0.0656	0.0427	0.004
Lab010	0.054	0.079	0.063	0.195	0.050	0.039	0.094	0.050	0.063		0.077	0.073	0.060	0.065	0.094	0.063	0.049	0.010
Lab011	0.055	0.06		0.15	0.065		0.055		0.065		0.058	0.053	0.044	0.065	0.066	0.049	0.052	0.01
Lab012	0.059	0.12	0.043	0.096	0.055	0.053	0.08		0.06		0.12	0.068	0.069	0.07	0.076	0.047	0.049	0.009
Lab013	0.0546	0.0777	0.0822	0.0781	0.0552	0.0454	0.1027	0.0379	0.0612	0.0198	0.0823	0.0588	0.0702	0.059	0.0965	0.071	0.0443	0.0066
Lab014	0.055	0.031	0.052	0.07	0.085	0.036	0.083		0.056		0.072		0.046	0.065	0.072	0.054	0.13	
Lab015	0.049	0.070	0.046	0.050	0.041		0.088		0.060		0.071	0.036	0.096	0.060	0.064	0.054	0.043	0.005
Lab016	0.051	0.075	0.081	0.032	0.048	0.041	0.083	0.036	0.065	0.031	0.079	0.068	0.057	0.0793	0.113	0.074	0.041	0.004
Lab017				0.164			0.09						0.14			0.075		
lab018	0.069	0.167	0.11	0.152	0.104	0.046	0.111	0.177	0.086	0.044	0.089	0.15	0.098	0.098	0.148	0.097	0.057	0.02
Lab019	0.055	0.095	0.066	0.056	0.051	0.052	0.038	0.037	0.066	0.02	0.053	0.047	0.065	0.056	0.092	0.057	0.041	0.01
Lab020	0.0633	0.0837	0.0871	0.0857	0.0512		0.125	0.0288	0.0695		0.104	0.0694	0.0556	0.0774	0.11	0.0732	0.0455	0.0126
Lab022	0.047	0.068	0.061	0.089	0.042	0.038	0.099	0.035	0.075	0.03	0.074	0.049	0.068	0.078	0.1	0.06	0.035	0.01

APPENDIX 1. Results

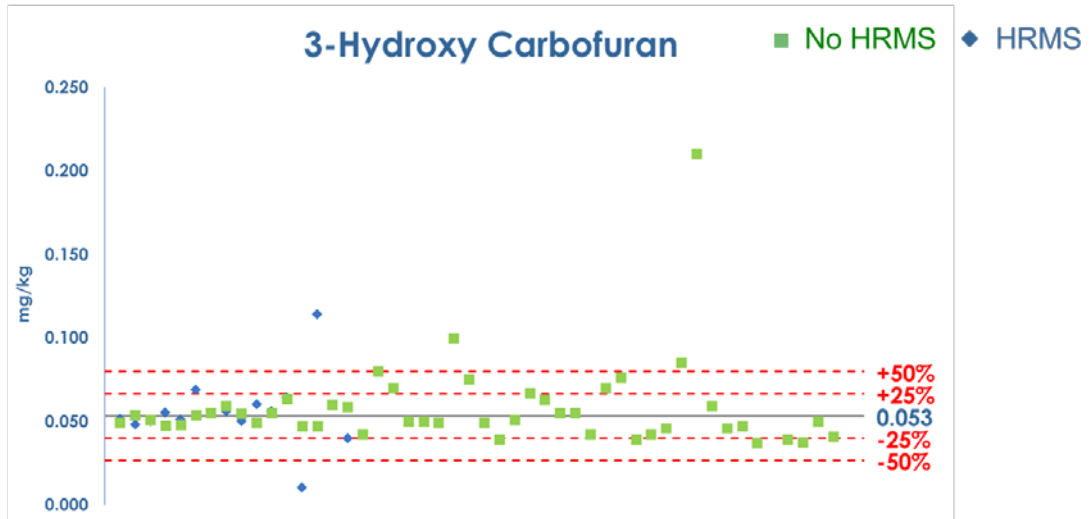
Evaluated Pesticides (18)																		
LABORATORY CODE Total No of Reporting Laboratories = 60	3-Hydroxy Carbofuran	Acephate	Aclonifen	Acrinathrin	Cyrantraniliprole	Dinotefuran	Fenobucarb	Fenpicoxamid	Isoprothiolane	Mefentrifluconazole	Molinate	Novaluron	Omethoate	Penthiopyrad	Picolinafen	Profenofos	Sulfoxaflor	Triflumizole
Average concentration Homogeneity test (mg/kg)	0.057	0.094	0.068	0.098	0.055	0.047	0.095	0.061	0.07	0.029	0.083	0.075	0.068	0.071	0.099	0.067	0.048	0.01
Robust mean of estimated concentrations reported (mg/kg)	0.053	0.084	0.066	0.096	0.054	0.044	0.09	0.043	0.066	0.027	0.081	0.069	0.066	0.069	0.096	0.066	0.047	0.01
CV (%)	21.5	32.6	31.4	43.1	23	24.4	15.9	25.4	13.2	19.1	18.2	28.1	30.2	19.5	21.9	18.1	23.5	34.1
Lab023	0.056	0.074	0.085	0.089	0.055	0.043	0.088	0.044	0.067	0.029	0.094	0.11	0.059	0.061	0.06	0.069	0.04	0.017
Lab024	0.047	0.029				0.01	0.078					0.121				0.074		0.013
Lab025	0.06	0.08	0.059	0.096	0.041		0.098	0.046	0.073		0.087	0.07	0.063	0.086	0.111	0.075	0.06	0.01
Lab026	0.0586	0.0807	0.0791	0.0772		0.0409			0.0627		0.1042	0.085	0.0512	0.0734		0.0714		0.0055
Lab027	0.042	0.099	0.046	0.059	0.05	0.031	0.077	0.036	0.059	0.027	0.064	0.054	0.1	0.058	0.086	0.064	0.038	0.005
Lab028	0.05	0.08		0.09					0.06				0.06	0.06	0.1	0.06	0.04	0.007
Lab029	0.08	0.166	0.062	0.074	0.063	0.06	0.079	0.04	0.063	0.026	0.09	0.07	0.076	0.072	0.085	0.052	0.067	0.01
Lab030	0.060 0.070	0.090 0.085 0.091	0.033	0.057	0.060 0.050	0.050 0.056	0.076 0.096 0.067		0.050 0.062		0.040 0.075 0.045	0.050 0.040	0.050 0.060 0.060	0.060 0.070	0.110 0.120	0.070 0.046	0.05	0.004
Lab031	0.1	0.2	0.168	0.3	0.088	0.114	0.168		0.132		0.18	0.15	0.172	0.152	0.19	0.148	0.09	0.022
Lab032	0.049	0.105	0.075	0.139	0.07	0.043	0.092		0.07		0.077	0.093	0.062	0.088	0.113	0.091	0.042	0.023
Lab033	0.1	0.06	0.09	0.14		0.05	0.115	0.052	0.112		0.086	0.09	0.07	0.057	0.15	0.065	0.13	0.01
Lab034	0.056	0.033	0.073	0.076	0.055	0.011	0.09	0.042	0.078	0.029	0.09	0.09	0.019	0.069	0.137	0.076	0.047	
Lab036	0.064 0.075	0.096 0.112	0.072 0.064	0.099 0.088	0.054 0.054	0.041 0.045	0.095 0.112	0.039 0.036	0.066 0.068 0.070	0.024 0.026	0.087 0.087	0.067 0.058	0.056 0.062	0.078 0.076	0.104 0.091 0.110	0.087 0.055	0.052 0.051	0.014 0.016
Lab037	0.049	0.072		0.13		0.051			0.062				0.068	0.078		0.067		
Lab038	0.039	0.019	0.074	0.104	0.086		0.08	0.034	0.049	0.026		0.073	0.026	0.054	0.073	0.068	0.041	
Lab040	0.051	0.057	0.062	0.086	0.052	0.037	0.088	0.068	0.076	0.03	0.084	0.077	0.043	0.068	0.115	0.068	0.03	0.008
Lab041	0.067	0.097	0.068	0.26	0.06	0.065	0.11	0.05	0.081	0.029	0.087	0.079	0.1	0.087	0.1	0.069	0.061	0.014
Lab042	0.063	0.13	0.065	0.085	0.072		0.088		0.07		0.1	0.079	0.081	0.085	0.15	0.071	0.062	
Lab043	0.055	0.11	0.043	0.085	0.06	0.055	0.11	0.049	0.065	0.035	0.1	0.083	0.075	0.075	0.11	0.055	0.053	0.01
Lab044	0.010	0.091	0.043	0.028	0.038	0.024	0.068		0.038	0.028	0.047	0.037	0.068	0.040	0.053	0.016	0.031	0.009
Lab045	0.055	0.105	0.06	0.1	0.05	0.055	0.095	0.04	0.065	0.025	0.085	0.06	0.075	0.065	0.085	0.065	0.05	0.005

Evaluated Pesticides (18)																		
LABORATORY CODE Total No of Reporting Laboratories = 60	3-Hydroxy Carbofuran	Acephate	Aclonifen	Acrinathrin	Cyrantriliprole	Dinotefuran	Fenobucarb	Fenpicoxamid	Isoprothiolane	Mefentrifluconazole	Molinate	Novaluron	Omethoate	Penthiopyrad	Picolinafen	Profenofos	Sulfoxaflor	Triflumizole
Average concentration Homogeneity test (mg/kg)	0.057	0.094	0.068	0.098	0.055	0.047	0.095	0.061	0.07	0.029	0.083	0.075	0.068	0.071	0.099	0.067	0.048	0.01
Robust mean of estimated concentrations reported (mg/kg)	0.053	0.084	0.066	0.096	0.054	0.044	0.09	0.043	0.066	0.027	0.081	0.069	0.066	0.069	0.096	0.066	0.047	0.01
CV (%)	21.5	32.6	31.4	43.1	23	24.4	15.9	25.4	13.2	19.1	18.2	28.1	30.2	19.5	21.9	18.1	23.5	34.1
Lab046	0.042	0.072	0.063	0.094					0.067		0.09		0.117			0.09		
Lab047	0.07	0.097	0.098	0.085	0.067	0.049	0.098	0.056	0.076	0.033	0.104	0.077	0.069	0.087	0.142	0.075	0.054	0.01
Lab048	0.076	0.14	0.0545	0.0775	0.0415	0.051	0.0945	0.237	0.0655	0.0215	0.051	0.0735	0.0815	0.05675	0.091	0.059	0.073	0.007
Lab049	0.081	0.067	0.083		0.077	0.031	0.076		0.055		0.079	0.067	0.05	0.085	0.12	0.085	0.03	0.0058
Lab051	0.046	0.086	0.056	0.079	0.043	0.047	0.077		0.068	0.024	0.068	0.072	0.063	0.052	0.101	0.064	0.043	0.01
Lab052	0.114	0.0965		0.0845		0.0491	0.0997	0.0608	0.0677		0.0829		0.0649	0.0739	0.093	0.0703		
Lab054	0.0854	0.0897	0.035	0.042					0.056		0.072	0.0641	0.105	0.059	0.089	0.05	0.0603	
Lab055	0.21	0.21	0.043	0.086	0.072	0.055	0.085		0.084		0.081	0.07	0.086	0.049	0.105	0.08	0.143	0.004
Lab056	0.059	0.107	0.069	0.119	0.066	0.046	0.095	0.033	0.072		0.087	0.169	0.059	0.062	0.116	0.075	0.04	
Lab057	0.0459	0.072	0.0357	0.0327	0.0405	0.0292			0.0507		0.0295	0.0358	0.0695	0.0574	0.0851	0.0582		
Lab058	0.047	0.069	0.056	0.067		0.035			0.065			0.063	0.069	0.071		0.06	0.033	0.01
Lab059	0.037	0.077	0.079	0.1	0.052		0.1	0.034	0.085		0.089	0.098	0.014	0.074	0.11	0.073	0.038	0.015
Lab061	0.04	0.04	0.021	0.04	0.033				0.035				0.022	0.032	0.06	0.036	0.048	
Lab062	0.039	0.036	0.056	0.03	0.041	0.029	0.085		0.07		0.076	0.044	0.03	0.055	0.069	0.057	0.041	0.005
Lab063	0.0377	0.0667	0.049	0.11	0.0575	0.0394			0.0627		0.088	0.063	0.0891	0.0737	0.0995	0.061	0.0366	0.0086
Lab064	0.05	0.12	0.1	0.08	0.045	0.072	0.11	0.03	0.068	0.02	0.085	0.08	0.08	0.09	0.1	0.075	0.05	
Lab065	0.041	0.052		0.149		0.039	0.055		0.051		0.054	0.024	0.044	0.054	0.105	0.062	0.086	0.010

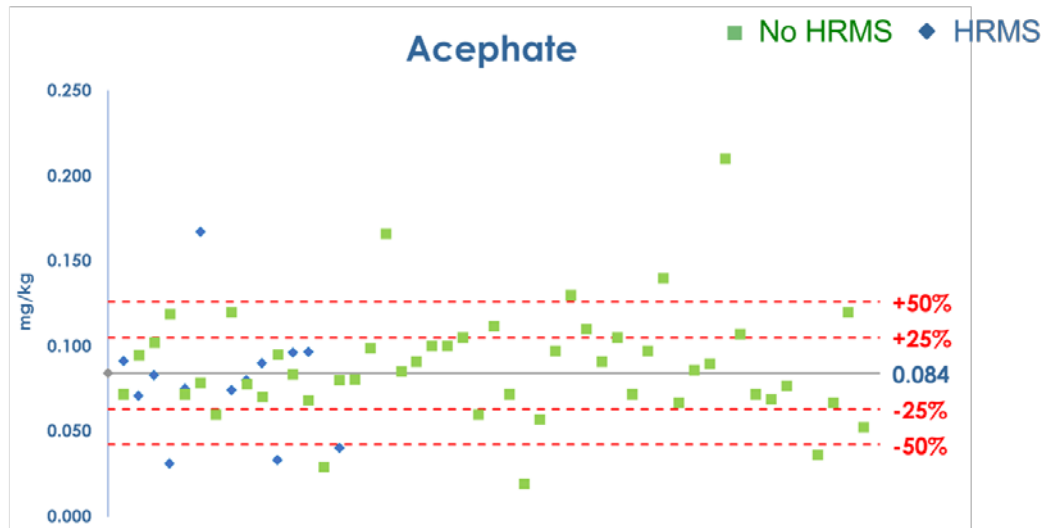
APPENDIX 2. Graphical Representations

The grey line represents the robust mean

Pesticides	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
3-Hydroxy Carbofuran	0.057	0.053	21.5	64	59	98	61



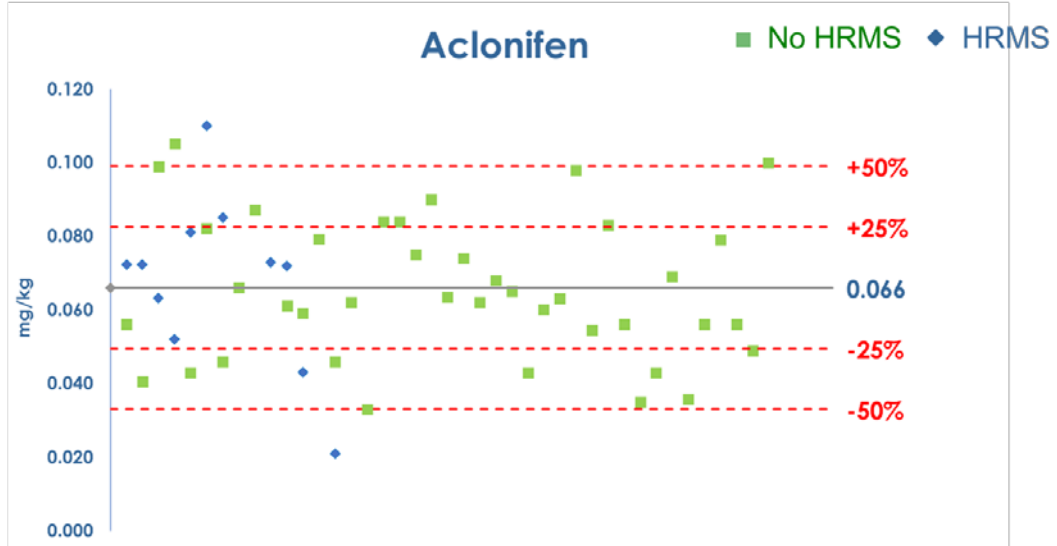
Pesticides	Conc. Homogeneity test (mg/kg)	Robust mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Acephate	0.094	0.084	32.6	65	59	98	61



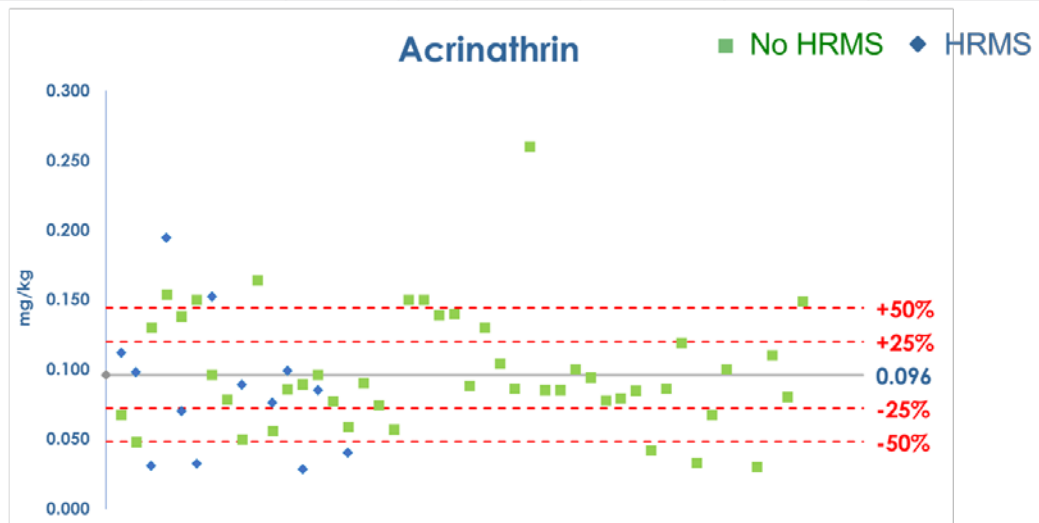
APPENDIX 2. Graphical Representations

The grey line represents the robust mean

Pesticides	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
Aclonifen	0.068	0.066	31.4	55	51	85	51

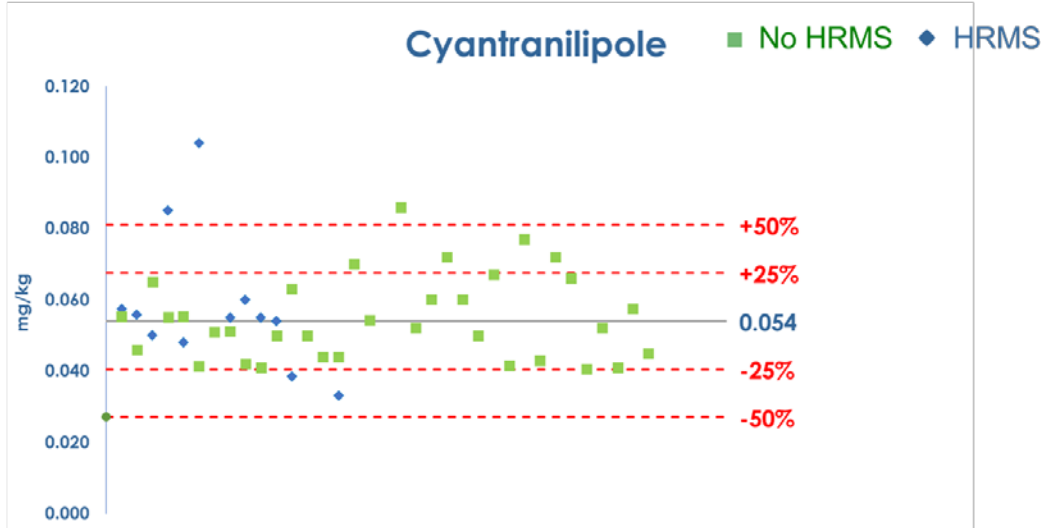


Pesticides	Conc. Homogeneity test (mg/kg)	Robust mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Acrinathrin	0.098	0.096	43.1	63	58	97	58

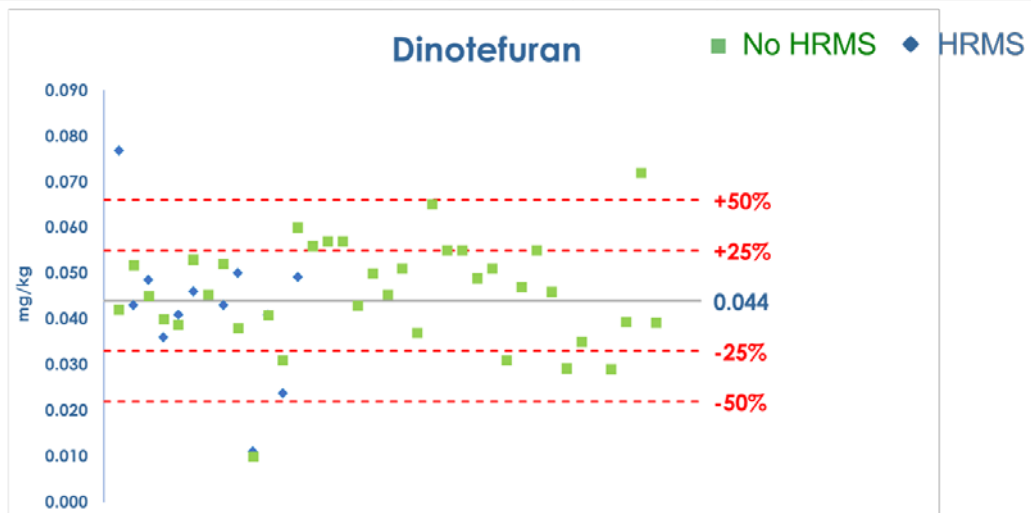


The grey line represents the robust mean

Pesticides	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
Cyantraniliprole	0.055	0.054	23	52	47	78	46



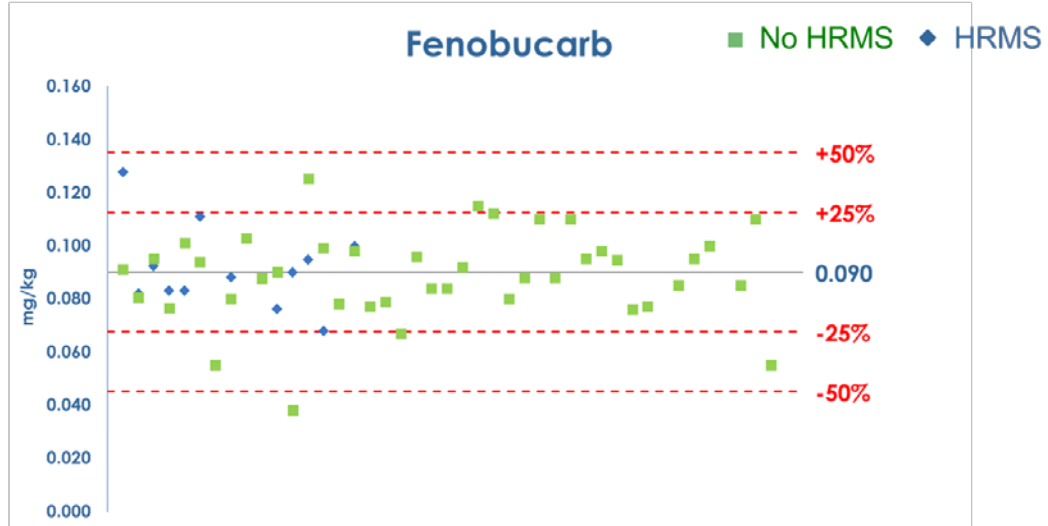
Pesticides	Conc. Homogeneity test (mg/kg)	Robust mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Dinotefuran	0.047	0.044	24.4	52	47	78	48



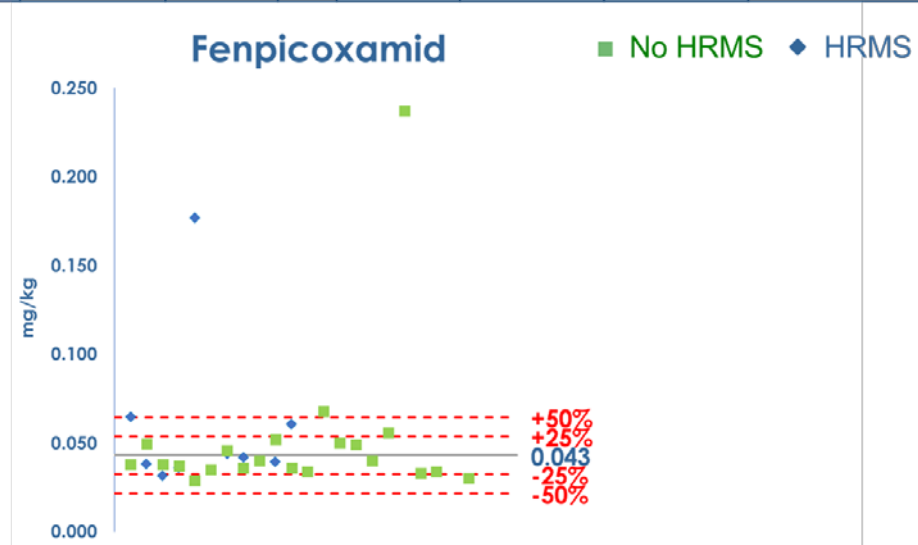
APPENDIX 2. Graphical Representations

The grey line represents the robust mean

Pesticides	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
Fenobucarb	0.095	0.090	15.9	62	55	92	53

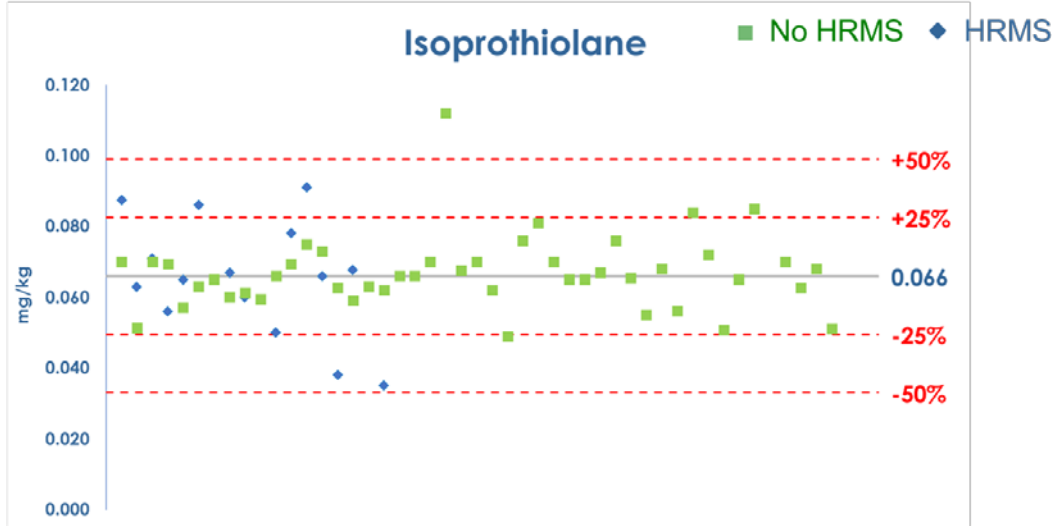


Pesticides	Conc. Homogeneity test (mg/kg)	Robust mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Fenpicoxamid	0.061	0.043	25.4	36	32	53	30

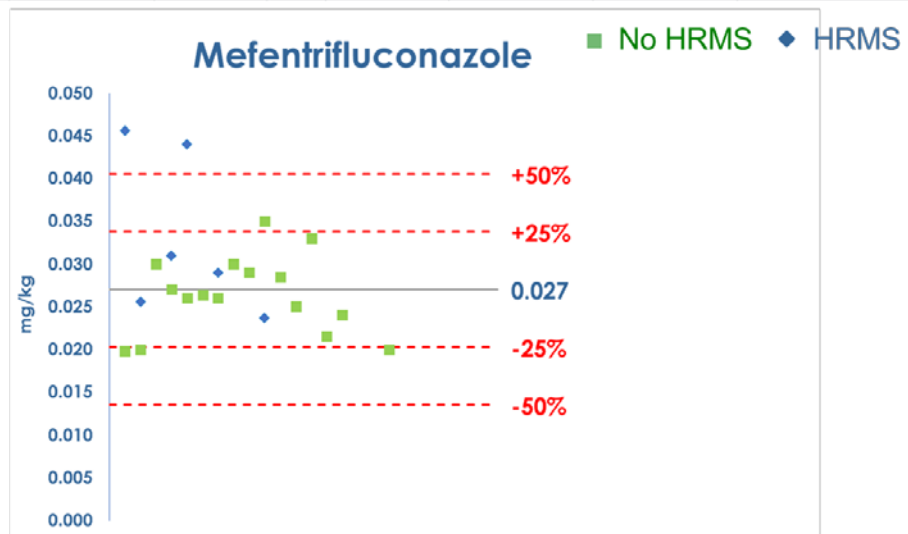


The grey line represents the robust mean

Pesticides	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
Isoprothiolane	0.070	0.066	13.2	66	58	97	61



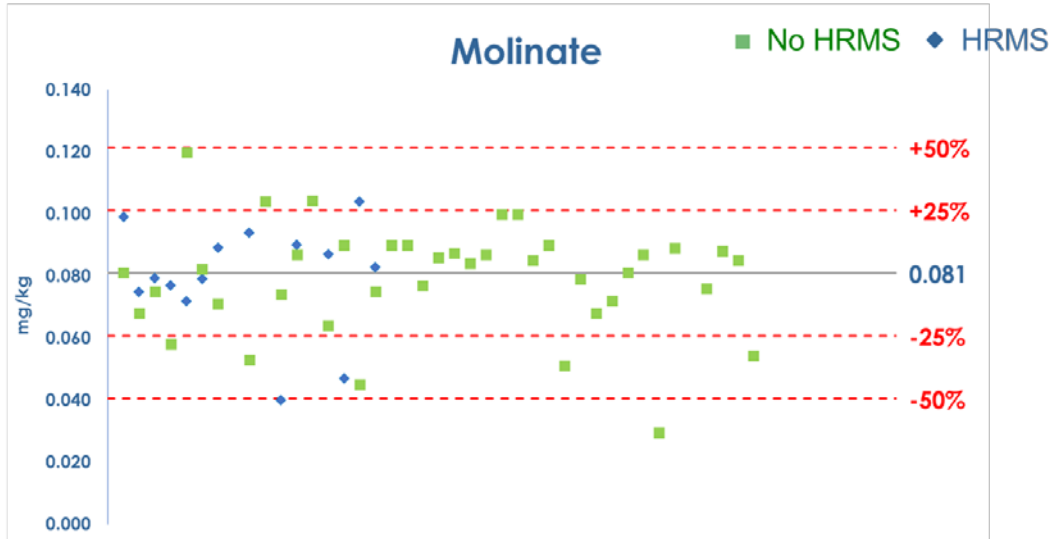
Pesticides	Conc. Homogeneity test (mg/kg)	Robust mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Mefentrifluconazole	0.029	0.027	19.1	30	26	43	23



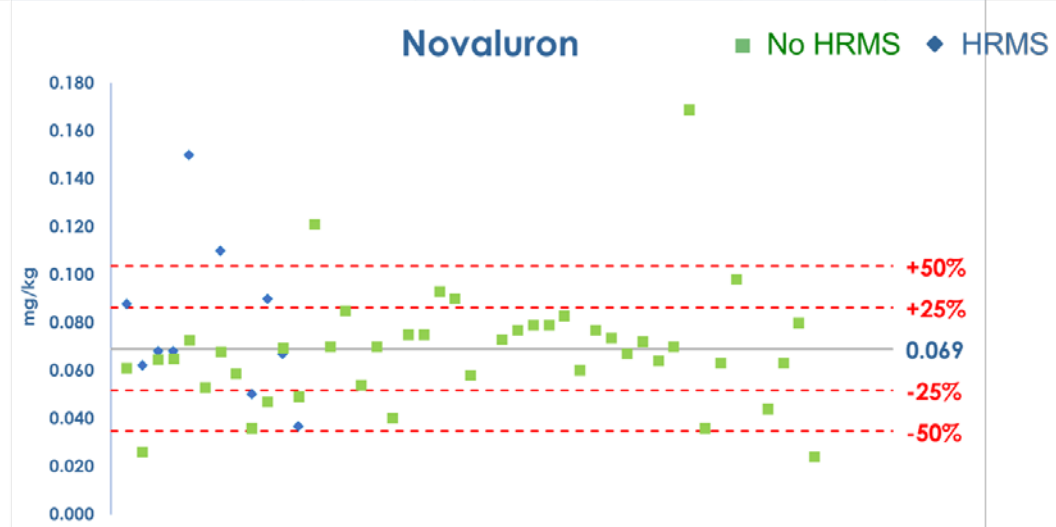
APPENDIX 2. Graphical Representations

The grey line represents the robust mean

Pesticides	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
Molinate	0.083	0.081	18.2	60	52	87	53

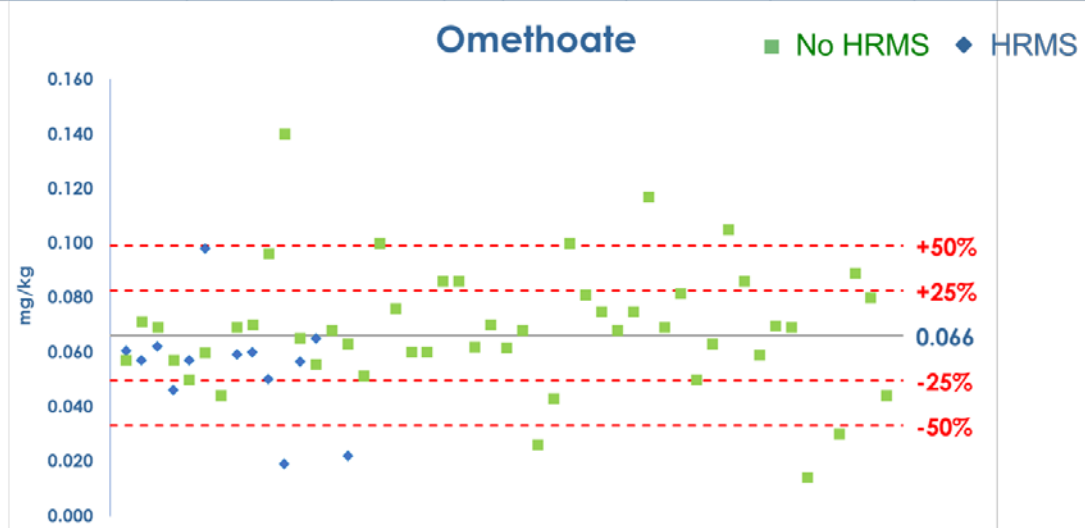


Pesticides	Conc. Homogeneity test (mg/kg)	Robust mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Novaluron	0.075	0.069	28.1	59	54	90	53

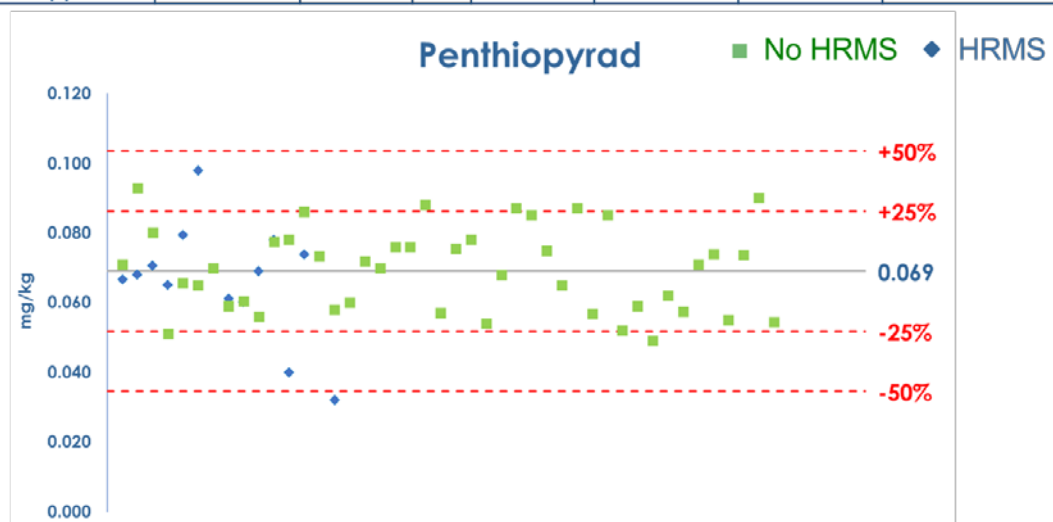


The grey line represents the robust mean

Pesticides	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
Omethoate	0.068	0.066	30.2	65	59	98	61



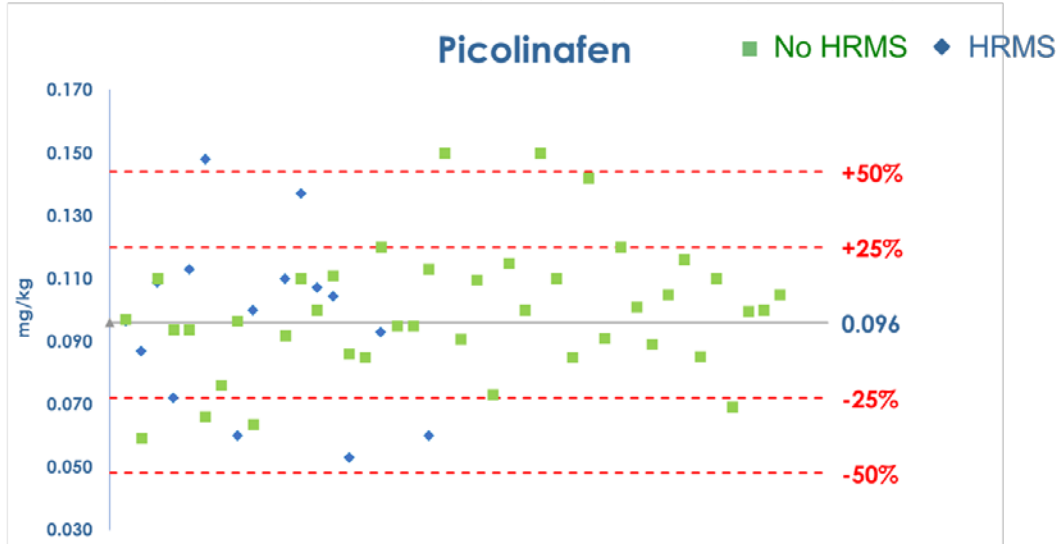
Pesticides	Conc. Homogeneity test (mg/kg)	Robust mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Penthiopyrad	0.071	0.069	19.5	61	55	92	57



APPENDIX 2. Graphical Representations

The grey line represents the robust mean

Pesticides	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
Picolinafen	0.099	0.096	21.9	63	55	92	56

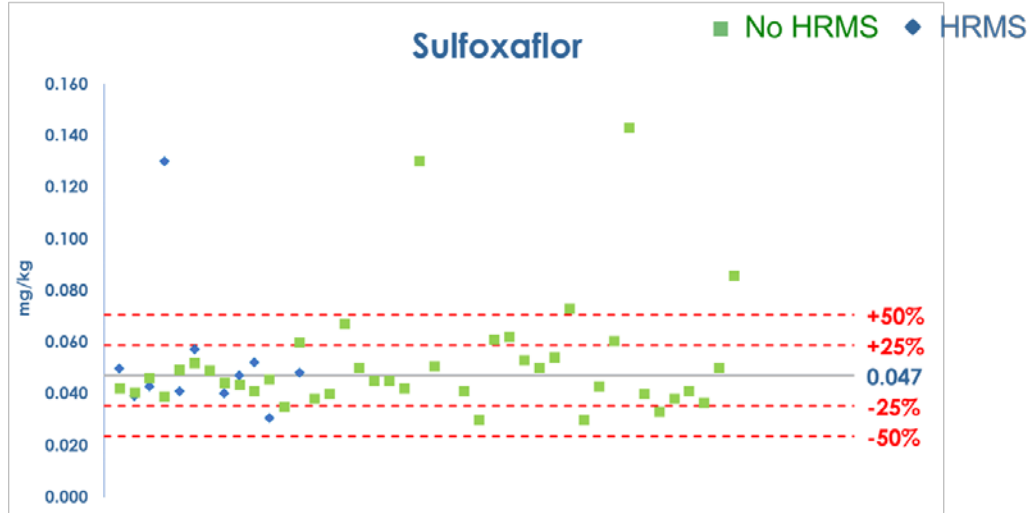


Pesticides	Conc. Homogeneity test (mg/kg)	Robust mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Profenofos	0.067	0.066	18.1	66	59	98	61

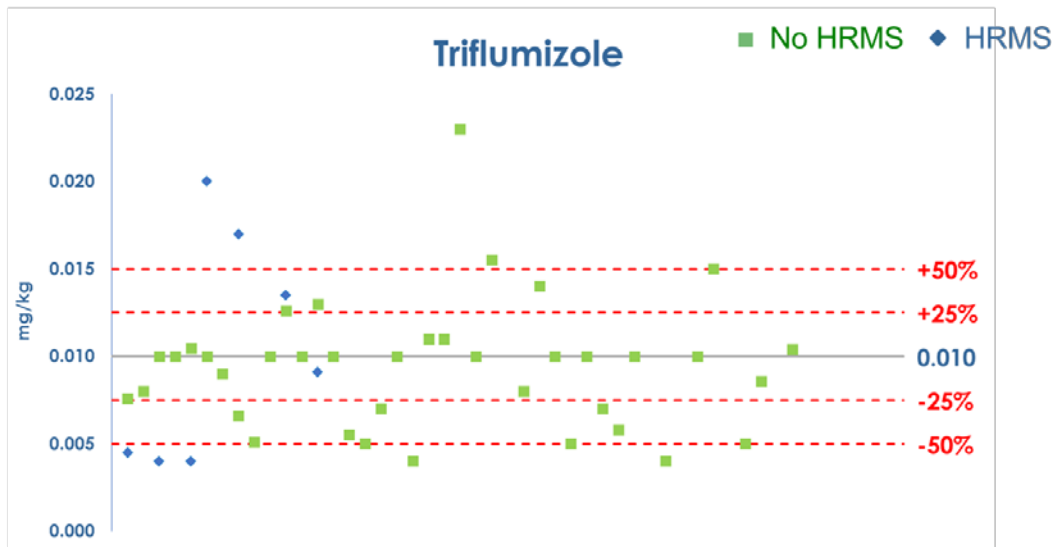


The grey line represents the robust mean

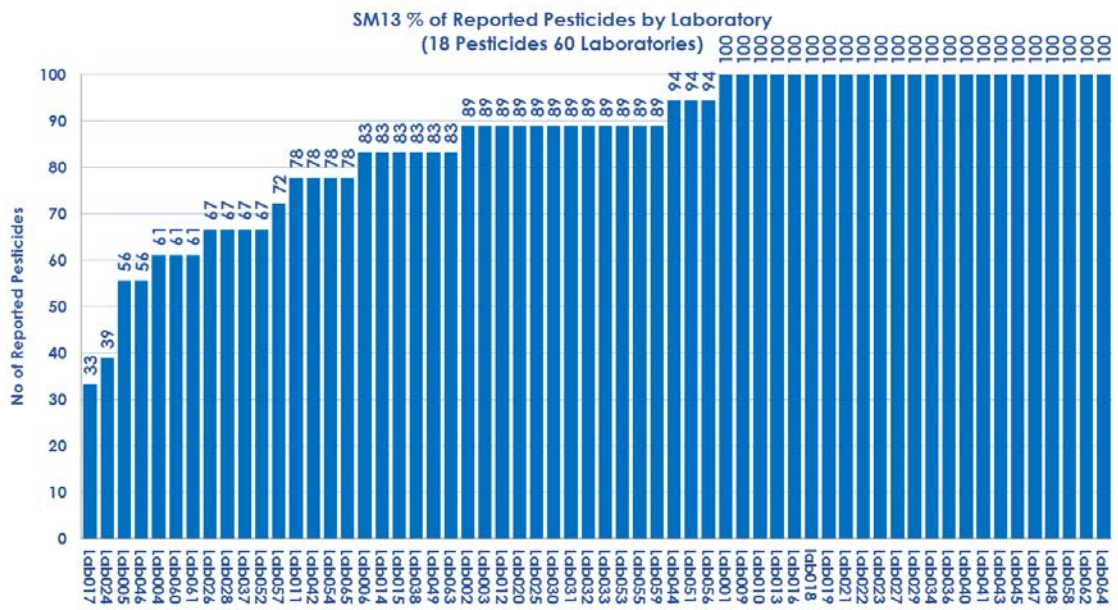
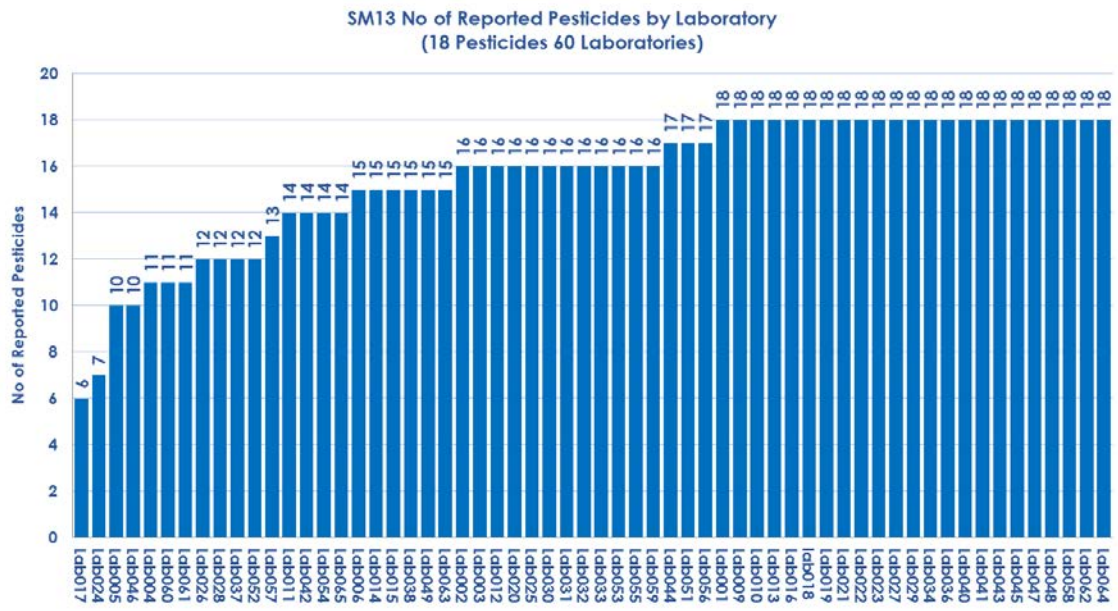
Pesticides	Conc. Homogeneity test (mg/kg)	Robust mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Sulfoxaflor	0.048	0.047	23.5	57	52	87	52

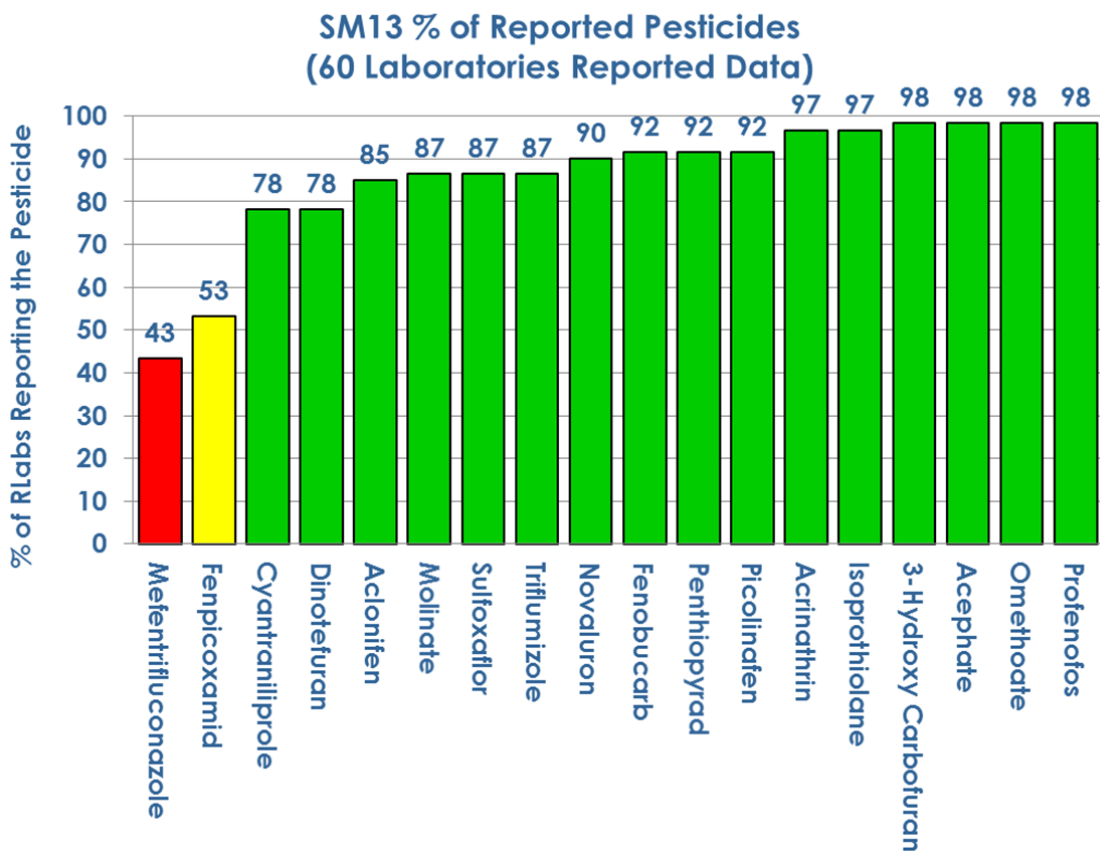
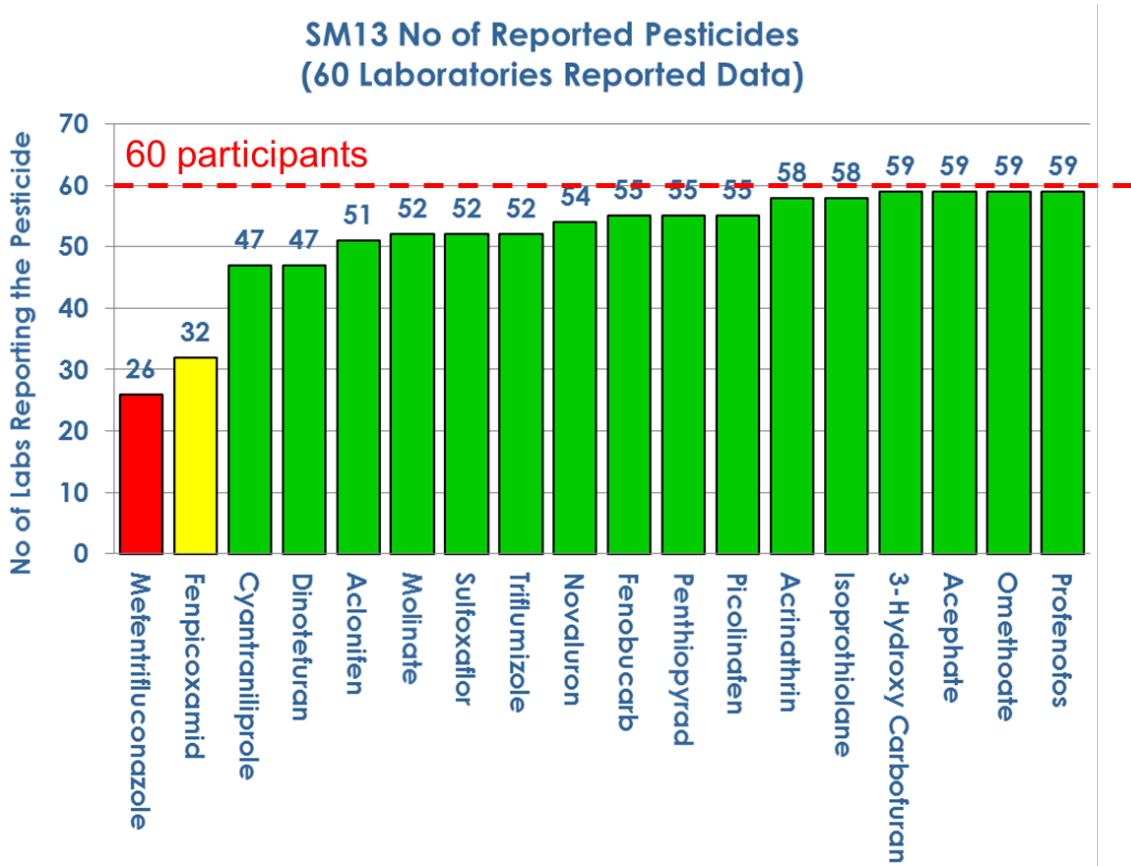


Pesticides	Conc. Homogeneity test (mg/kg)	Robust mean (mg/kg)	CV (%)	No of Detections	N° of Labs detected the pesticide	% of Labs detected the pesticide	No of concentrations Reported
Triflumizole	0.010	0.01	34.1	58	52	87	46



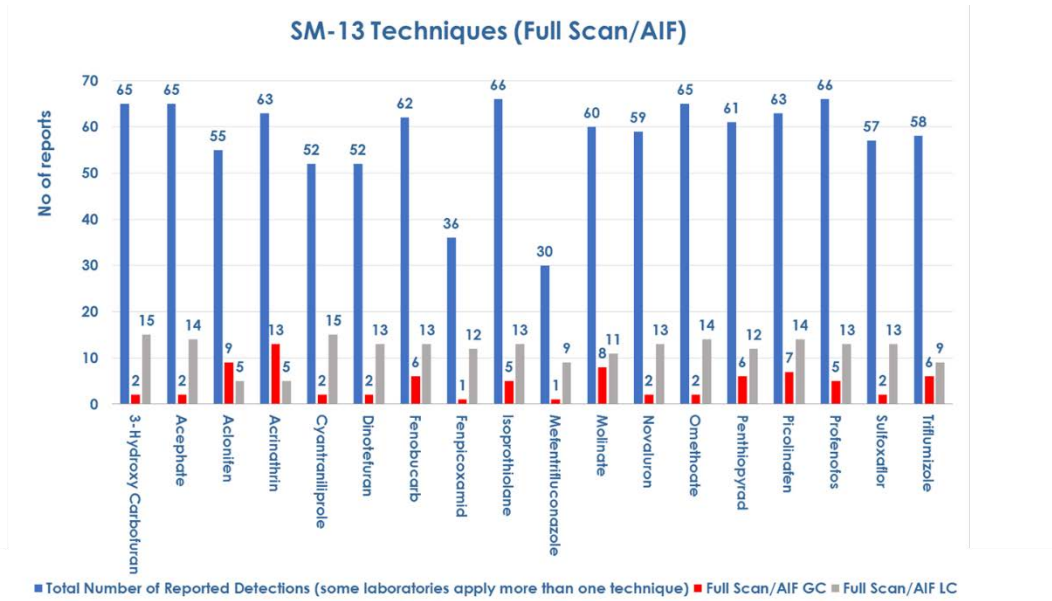
APPENDIX 2. Graphical Representations



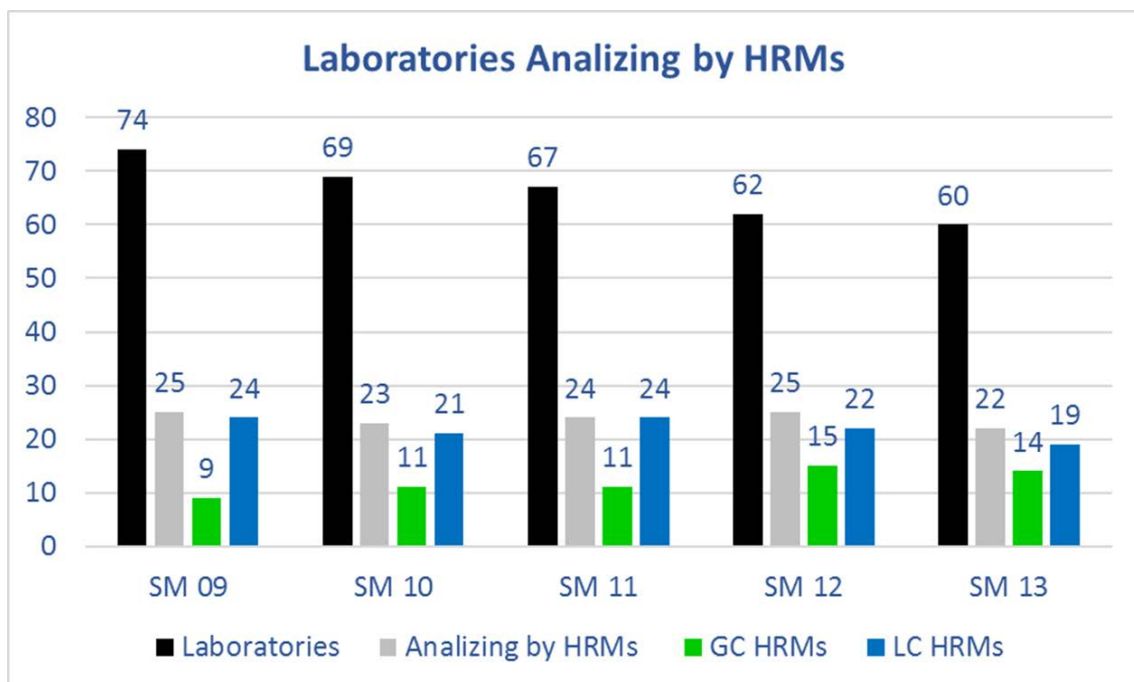


APPENDIX 2. Graphical Representations

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



Laboratories analyzing the test items by HRMs



ANNEX 1. List of Laboratories that participate in EUPT-FV-SM13.

ANNEX 1. List of Laboratories that reported results in EUPT-FV-SM13.

COUNTRY	LABORATORY NAME	CITY
AUSTRIA	AGES (AUSTRIAN AGENCY FOR HEALTH AND FOOD SAFETY) INSTITUTE FOR FOOD SAFETY	INNSBRUCK
BELGIUM	LOVAP	GEEL
BELGIUM	GROEN AGRO CONTROL	DELFGAUW
BELGIUM	NOFALAB B.V.	SCHIEDAM
BELGIUM	PRIMORIS BELGIUM	ZWIJNAARDE
CHINA	LANZHOU INSTITUTE FOR FOOD AND DRUG CONTROL	LANZHOU
CHINA	UNI-STAR INSPECTION TECHNOLOGY CO. LTD.	BEIJING
CHINA	AGRO-PRODUCT SAFETY RESEARCH CENTER, CAIQ	BEIJING
CHINA	SHANGHAI MUNICIPAL CENTER FOR DISEASE CONTROL AND PREVENTION	SHANGHAI
CHINA	AGRO-ENVIRONMENTAL PROTECTION INSTITUTE, MINISTRY OF AGRICULTURE AND RURAL AFFAIRS (AEPI)	TIANJIN
CROATIA	BIOINSTITUTE LTD.	CAKOVEC
CROATIA	SAMPLE CONTROL D.O.O.	PUŠKARIČEVA ULICA 18, ZAGREB-LUČKO
CZECH REPUBLIC	CZECH AGRICULTURE AND FOOD INSPECTION AUTHORITY	PRAHA
CZECH REPUBLIC	UNIVERSITY OF CHEMISTRY AND TECHNOLOGY PRAGUE	PRAGUE
DENMARK	DTU NATIONAL FOOD INSTITUTE	LYNGBY
ESPAÑA	EUROFINS ECOSUR, S.A.	LORQUI - MURCIA
FINLAND	FINNISH CUSTOMS LABORATORY	ESPOO
FRANCE	SCL PARIS	MASSY
FRANCE	INOVALYS - LE MANS	LE MANS
FRANCE	SCL	MONTPELLIER
FRANCE	CERECO LABORATOIRE SUD	GARONS
GERMANY	LANDESUNTERSUCHUNGSANSTALT DRESDEN	DRESDEN
GERMANY	CVUA STUTTGART	FELLBACH
GERMANY	CENTRAL INSTITUTE OF THE BUNDESWEHR MEDICAL SERVICE	GARCHING
GERMANY	ILAU GMBH	ANZING
GERMANY	CHEMISCHES LABOR DR. MANG	FRANKFURT
GERMANY	LAVES, LVI OLDENBURG	OLDENBURG
GERMANY	CVUA RRW	KREFELD
GERMANY	BAVARIAN HEALTH AND FOOD SAFETY AUTHORITY	ERLANGEN
GERMANY	LABOR FRIEDLE GMBH	TEGERNHEIM
GERMANY	LANDWIRTSCHAFTLICHES TECHNOLOGIEZENTRUM AUGUSTENBERG (LTZ)	KARLSRUHE
HUNGARY	NEBIH PESTICIDE LABORATORY	HODMEZOVASARHELY
HUNGARY	NFCSO PESTICIDE LAB, VELENCE	VELENCE
HUNGARY	FCSCN LTD. PESTICIDE RESIDUE ANALYTICAL LABORATORY	SZOLNOK
HUNGARY	FCSCN LTD. PESTICIDE RESIDUE ANALYTICAL LABORATORY	MISKOLC
IRELAND	PESTICIDE RESIDUE LABORATORY	CELBRIDGE
ITALY	IZSLT SEZIONE DI FIRENZE	SAN MARTINO

ANNEX 1. List of Laboratories that participate in EUPT-FV-SM13

COUNTRY	LABORATORY NAME	CITY
ITALY	ISITUTO ZOOPROFILATTICO SPERIMENTALE DELLA SICILIA	PALERMO
ITALY	ATS MILANO LABORATORIO DI PREVENZIONE	MILANO
ITALY	ISTITUTO ZOOPROFILATTICO ABRUZZO E MOLISE IZSAM	TERAMO
ITALY	APPA BOLZANO	BOLZANO
LITHUANIA	GALAB LABORATORIES GMBH	HAMBURG
LITHUANIA	NATIONAL FOOD AND VETERINARY RISK ASSESSMENT INSTITUTE	VILNIUS
NONE	EUROFINS DR. SPECHT EXPRESS GMBH	HAMBURG
NORWAY	NIBIO, PESTICIDES AND NATURAL PRODUCTS CHEMISTRY	AAS
PERÚ	INSPECTORATE SERVICES PERÚ SAC	LIMA
POLSKA	HAMILTON UO-TECNOLOGIA	GRÓJEC
SPAIN	LABORATORIO KUDAM SLU	PILAR DE LA HORADADA
SPAIN	LABORATORIO AGROALIMENTARIO Y SANIDAD ANIMAL DE LA REGIÓN DE MURCIA	EL PALMAR
SPAIN	LABS & TECHNOLOGICAL SERVICES AGO	BURGUILLOS
SPAIN	LABORATORIO SALUD PÚBLICA AYUNTAMIENTO DE MADRID.	MADRID
SPAIN	LABORATORIO ANALITICO BIOCLINICO, SLU	ALMERIA
SPAIN	LABORATORIO AGROAMBIENTAL DE ZARAGOZA (GOBIERNO DE ARAGÓN)	ZARAGOZA
SPAIN	ANALYTICA ALIMENTARIA GMBH	ALMERÍA
SPAIN	NATIONAL CENTER FOR TECHNOLOGY AND FOOD SAFETY (CNTA)	SAN ADRIÁN
SWEDEN	EUROFINS FOOD AND FEED TESTING SWEDEN AB	LIDKOPING
SWEDEN	NATIONAL FOOD AGENCY	UPPSALA
SWITZERLAND	SERVICE DE LA CONSOMMATION ET DES AFFAIRES VÉTÉRINAIRES	GENÈVE
SWITZERLAND	KANTONALES LABOR ZÜRICH	ZÜRICH
THE NETHERLANDS	WFSR - NRL FOR PESTICIDES	WAGENINGEN