EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUIT AND VEGETABLES.

SCREENING METHODS 08
(EUPT-FV-SM08)

Pesticide Residues in Spinach Homogenate

Final Report
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CONTENT

EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUIT AND VEGETABLES. SCREENING METHODS 08

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 (EC) No 882/2004[2] lays down the general tasks, duties and requirements of European Union Reference Laboratories (EURLs) for Food, Feed and Animal Health. Among these tasks is the provision for independently-organised comparative tests. This is the eighth time that the EURL for pesticides in fruit and vegetables[3] at the University of Almería, Spain, has organised a proficiency test on qualitative screening methods for pesticides in fruit and vegetable commodities.

The aim of these tests is to evaluate laboratory capability when using wide-scope qualitative and/or semi-quantitative screening methods during routine analysis, for detecting and identifying unexpected pesticides at levels at, or above 0.01 mg/kg – included in and/or in addition to the laboratories' quantitative methods used for frequently-detected pesticides. A second aim is to encourage official laboratories (OfLs) to extend the scope of their methods in a cost-effective way, by using the different mass spectrometry (MS) instruments/software and methods available (whether they are old or new).

Participation in this PT remains on a voluntary basis. Besides this one, official laboratories have a significant number of mandatory PTs annually, given that the EURL-FV already organises the PT for quantitative multi-residue pesticide analysis (EUPT-FV18) over the same time period. Nevertheless, all FV-National Reference Laboratories (FV-NRLs) and FV-Official Laboratories (FV-OfLs) involved in the determination of pesticide residues in fruit and vegetables for the EU-coordinated monitoring programme, or for their own national programmes, were invited to take part.

DG-SANTE will have full access to all EUPT data including the individual lab-codes/lab-name keys. This report may be presented to the European Union Standing Committee on Plants, Animals, Food and Feed.

[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 (EC) No 882/2004 of the European Parliament and of the Council on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules. Published in the OJ of the EU L191 of 28.05.2004
[3]Commission Regulation (EC) No 776/2006 of 23 May 2006 - amending Annex VII of Regulation (EC) No 882/2004 of the European Parliament and of the Council as regards Community Reference Laboratories.

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.

MS plays an essential role in the everyday work carried out by laboratories. Technological improvements in modern MS systems offer new possibilities for greatly increasing the scope of MRM (multiresidue methods) analysis. Whereas full-scan measurements are theoretically the best approach for MS screening, developments in targeted measurements also offer the potential for a substantially increased scope of analysis. Another reason for conducting this proficiency test on screening methods is to gather information from laboratories as to the type of software they use for processing data: whether laboratories are using commercial software and databases or whether they are internally constructed and search manually. This type of test provides an overview of such information as well as valuable insight into the possible need for further software development in the near future.

The aim of the EURL-FV is for laboratories to be able to use mass-spectrometry-based screening methods routinely, following validation. This is in line with Document N° SANTE/11945/2015 (which supersedes Document No. SANCO/12571/2013) - Guidance document on analytical quality control and method validation procedures for pesticides residue analysis in food and feed.

This EUPT-FV-SM08 is aimed at all NRLs and all OfLs for fruit and vegetables in EU Member States. Laboratories outside this EURL/NRL/OfL-Network were also able to participate on a case-by-case basis, following consultation with DG-SANTE.

The evaluation of this PT was based on qualitative information, although an estimated concentration was requested for those pesticides that were detected, only for informative purposes. It was decided, as in previous PTs, not to provide the laboratories with a Target Pesticide List so that their capability in detecting whatever pesticides were present was also evaluated.

2. TEST ITEMS

2.1 Preparation of the treated test item.

This proficiency test is based on the pesticide-residue analysis of spinach. The spinach plants were organically grown in Almería by the organisers in a greenhouse with passive ventilation - typically used for horticulture in the Almeria region ('raspa y amagado' type), in south-eastern Spain.

The soil used for the crops has an artificial layer of sand mulch on top of the soil surface; these kinds of mulched soils are known locally as 'enarenado'. They consist of two layers: a top layer of sand around 10-15 cm thick, and a bottom organic layer 2-3 cm thick, placed directly over the indigenous soil.

Drip fertigation was used to supply both water and fertilizers to the crop.

The pesticides used to spike the spinach heads were decided upon by the Quality Control Group. It was decided that a target pesticide list would not be provided to participants. The pesticides selected for treating the test item for this EUPT-FV-SM08 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 2016, 2017 and 2018 (Regulation (EU) 2015/595).
- That they had particularly acute toxicity and/or had low ARfD values.

Table 2.1 shows the 15 pesticides present in the spinach sample. The pesticide treatments were carried out post-harvest using standard solutions. The test item was frozen (using liquid nitrogen) and chopped. The frozen minced spinach was mixed in a constantly-spinning container until a homogeneous item was obtained. Finally, 200 g portions of the well-mixed homogeneate were weighed out into screw-capped polyethylene plastic bottles, sealed and stored in a freezer at about -20 °C prior to distribution to participants.

Table 2.1 Pesticides present in the sample.

Pesticides			
Benalaxyl	Chlozolinate	Clomazone	Cyazofamid
Fenpyrazamine	Heptachlor	Isopyrazam	Phenthoate
Prosulfocarb	Prothiofos	Pyrethrins	Quintozene
Rotenone	Tetramethrin	Triticonazole	

Pyrethrin was used to spike the test item, but the analytical standard contained a mixture of its six components (cinerin I and II, jasmolin I and II, pyrethrin I and II). The decision of the Advisory Group was to consider the reporting of any of the components as "Pyrethrin".

2.2 Preparation of "blank" test item.

The spinach used for the production of the blank item was organically grown in the same field as the test item. A homogenate was prepared in the same way as the treated test item described previously.

During the blank analysis by the Organiser, spiromesifen was detected above 0.01 mg/kg, and due to the impossibility to grow more blank material, the Quality Control Group (QCG) decided to remove that pesticide from evaluation.

2.3 Homogeneity and stability tests.

The Organiser's homogeneity and stability tests associated with 'quantitative' PTs were conducted with a further acceptance criterion to those in the classical EUPT-FVs, the PT test item

was analysed in order to identify the present pesticides, which were consistently confirmed to be above the Organiser's LODs.

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[4]. 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 2.3a shows the results of these tests, together with the average concentration values for each of the pesticides used to treat the sample along with the RSDs.

Table 2.3a Homogeneity tests

Test item No.	019 A	019 B	027 A	027 B	035 A	035 B	043 A	043 B	053 A	053 B	062 A	062 B	079 A	079 B	095 A	095 B	105 A	105 B	120 A	120 B	A. Cc (mg/kg)	$Ss^2 < c$ Pass/Fail
Benalaxyl	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.053	Pass
Chlozolinate	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.042	Pass
Clomazone	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.085	Pass
Cyazofamid	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.068	Pass
Fenpyrazamine	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.060	Pass
Heptachlor	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.058	Pass
Isopyrazam	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.085	Pass
Phenthoate	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.065	Pass
Prosulfocarb	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.057	Pass
Prothiofos	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.086	Pass
Pyrethrin	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.111	Pass
Quintozene	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.058	Pass
Rotenone	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.085	Pass
Tetramethrin	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.071	Pass
Triticonazole	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	I	0.065	Pass

I: Identified A. Cc: Average Concentration

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

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

Table 2.3b Stability tests performed.

Date	Shipment Day (9th February)			48h later Shipment Day (11th February)			Few days after deadline (17th February)		
Test item No.	014	026	052	016	042	002	024	040	058
Benalaxyl	I	I	I	I	I	I	I	I	I
Chlozolinate	I	I	I	I	I	I	I	I	I
Clomazone	I	I	I	I	I	I	I	I	I
Cyazofamid	I	I	I	I	I	I	I	I	I
Fenpyrazamine	I	I	I	I	I	I	I	I	I
Heptachlor	I	I	I	I	I	I	I	I	I
Isopyrazam	I	I	I	I	I	I	I	I	I
Phenthoate	I	I	I	I	I	I	I	I	I
Prosulfocarb	I	I	I	I	I	I	I	I	I
Prothiofos	I	I	I	I	I	I	I	I	I
Pyrethrin	I	I	I	I	I	I	I	I	I
Quintozene	I	I	I	I	I	I	I	I	I
Rotenone	I	I	I	I	I	I	I	I	I
Tetramethrin	I	I	I	I	I	I	I	I	I
Triticonazole	I	I	I	I	I	I	I	I	I

I: Identified NI: Not identified

2.4 Distribution of test items and protocol to participants

Approximately 200 g of treated spinach homogenate together with another 200 g of 'blank' spinach homogenate were shipped to participants on 8th February 2016. The deadline for results submission to the Organiser was 72 hours after receipt of the test item. Participants were asked to report all the pesticides that they detected.

Laboratories were asked to screen the test items using the wide-scope screening methods they would normally apply, or anticipate applying, for official monitoring purposes. This typically involves full-scan techniques like GC-MS (single quadrupole, ion trap, ToF) and/or LC-MS (ToF and Orbitrap). However, extended targeted methods using LC-MS/MS (triple quadrupole, Q-trap, Q-ToF) or GC-MS/MS (triple quadrupole and ion trap) could also be used.

Before shipment, the laboratories received full instructions for the receipt and analysis of the test item although 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-SM08 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 Form 0 (Sample Receipt) and Form 1 (Results). These allowed the evaluation of the mass-spectrometric screening methods that each of the participants used.

3. STATISTICAL METHODS

3.1 Type of results reported

The evaluation of this PT was based on qualitative information, although an estimated concentration was requested for those pesticides that were detected, only for informative purposes.

In order to minimise the influence of out-lying results on the statistical evaluation, the robust mean of the estimated concentrations reported was calculated using robust statistics as described in ISO 13528:2015, taking into account the results reported by EU and EFTA countries laboratories only.

3.1.1 Other Reported Pesticides

These were considered as those results showing the apparent presence of pesticides which were: (i) not used in the test item treatment, or (ii) not identified by the Organiser, even after repeated analyses. However, if a number of participants detect the same additional pesticide(s), then a decision as to whether, or not, this should be considered an 'Other Reported Pesticide' result was made on a case-by-case basis.

> *Organiser's Note: Not all screening methods immediately provide sufficient information to allow full identification. In such cases, when they detect a pesticide in real life, laboratories normally do a follow-up confirmatory analysis: using, for example, LC-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

Ninety-one laboratories agreed to participate in this eighth proficiency test on screening methods. Eighty-three laboratories submitted results on time (eigth laboratories cancelled their participation). All results reported by the participants are given in Appendix 1. Graphical representations of the results reported are shown in Appendix 2. Details of the screening methods used are provided in Appendix 3 (available on the EUPT-FV-SM08 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 and by laboratory can be seen in Table 4.1a.

Table 4.1a Summary of Reported Results.

Pesticide	Reported		Not Reported	
	No. of laboratories	% of laboratories *	No. of laboratories	% of laboratories *
Benalaxyl	78	94	5	6
Chlozolinate	53	64	30	36
Clomazone	71	86	12	14
Cyazofamid	62	75	21	25
Fenpyrazamine	28	34	55	66
Heptachlor	71	86	12	14
Isopyrazam	33	40	50	60
Phenthoate	76	92	7	8
Prosulfocarb	71	86	12	14
Prothiofos	71	86	12	14
Pyrethrin**	53	64	30	36
Quintozene	60	72	23	28
Rotenone	64	77	19	23
Tetramethrin	71	86	12	14
Triticonazole	69	83	14	17

* The % of laboratories is calculated based on the total number of laboratories submitting results (83 laboratories).
**Pyrethrin was used to spike the test item, but the analytical standard contained a mixture of its six components (cinerin I and II, jasmolin I and II, pyrethrin I and II). The decision of the Advisory Group was to consider the reporting of any of the components as "Pyrethrin".

In this EUPT-FV-SM08 the estimated concentration was requested for those pesticides that were detected, only for informative purposes. However, not all the laboratories reported concentration results (Appendix 1 – Estimated Concentrations Reported). Table 4.1b shows the robust mean of the estimated concentrations reported, the average concentrarion from the homogeneity test and the dispersion of the concentration results reported.

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

Pesticide	Robust mean of estimated concentrations reported (mg/kg)	Average concentration Homogeneity test (mg/kg)	CV (%)
Benalaxyl	0.052	0.053	20.3
Chlozolinate	0.047	0.042	42.4
Clomazone	0.075	0.085	25.0
Cyazofamid	0.070	0.068	33.3
Fenpyrazamine	0.049	0.060	17.7
Heptachlor	0.061	0.058	38.1
Isopyrazam	0.074	0.085	23.2
Phenthoate	0.068	0.065	24.0
Prosulfocarb	0.051	0.057	22.4
Prothiofos	0.076	0.086	24.7
Pyrethrin	As different laboratories reported different pyrethrins, the robust mean has not been calculated		
Quintozene	0.063	0.058	34.0
Rotenone	0.073	0.085	22.5
Tetramethrin	0.068	0.071	30.4
Triticonazole	0.055	0.065	20.7

Other compounds were identified and quantified by the organizer at concentrations below 0.010 mg/kg:

- Azoxystrobin
- Difenoconazole
- Fenamiphos sulfone
- Fenamiphos sulfoxide
- Indoxacarb
- Tecnazene
- Tetraconazole
- Thiabendazole

Spiromesifen was also detected above 0.01 mg/kg, but as it was also present in the blank test item, the Advisory Group decided to remove that pesticide from evaluation.

4.1.1 Other Reported Pesticides

Some laboratories reported additional pesticides to those present in the test item. These reported pesticides are presented in Table 4.1.1.

Table 4.1.1. 'Other reported pesticides' in the test item given by laboratories.

Laboratory Code	Other Reported Pesticides
Lab001*	8-hydroxyquinoline, endosulfan sulfate, flutolanil, imazalil, metconazole
Lab005	cycloxydim, dinoseb, isoxaben, ivermectin A, ivermectin B, metconazole, metolcarb, pyroquilon
Lab008	biphenyl
Lab016	8-hydroxyquinoline
Lab023*	anthraquinone, endosulfan sulfate
Lab025	anthraquinone, diflubenzuron
Lab029*	dimepiperate, sulfometuron methyl, thiometon
Lab032	endosulfan sulfate, hexachlorobenzene
Lab035	hexachlorobenzene, spinosad

Laboratory Code	Other Reported Pesticides
Lab036*	spirodiclofen
Lab037*	2,4 DMF, cyprodinil, endosulfan sulfate, hexachlorobenzene, isoprotiolane, pencycuron
Lab042	spirotetramat
Lab045	fipronil sulfone, pebulate, quinalphos
Lab051	diphenylamine
Lab060	fluazifop-p
Lab065*	acetamiprid, chlorantraniliprole, imidacloprid, metoxyfenozide
Lab070*	metolcarb
Lab073	propiconazole
Lab074	endosulfan sulfate, hexachlorobenzene
Lab075	carbendazim, endosulfan sulfate, pirimicarb, pirimicarb desmethyl
Lab077	methacrifos
Lab078	chlorthiamid, diamidafos, metconazole, metominostrobin
Lab080	chlorpyrifos, endosulfan sulfate
Lab083	endosulfan sulfate, hexachlorobenzene, trifloxystrobin
Lab084*	buprofezin, cyprodinil, desmethyl pirimicarb, flubendiamide, fludioxonil, methoxyfenozide, pirimicarb, trifloxystrobin
Lab089*	coumafuryl, endosulfan sulfate, pencycuron, phenisopham

* National Reference Laboratories for Fruit and Vegetables from the EU participating in this test.

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

4.1.2 Non-Reported Pesticides

In Table 4.1 the number and percentage of laboratories not reporting each of the pesticides present in the sample can be seen. The individual results for each laboratory are given in Appendix 1. Graphical representations can be seen in Appendix 2.

4.2 Concentration levels.

Fifteen pesticides were used to spike the spinach test item at different levels, in the range between 0.040 mg/kg and 0.120 mg/kg. According to the homogeneity table 2.3a, most of them in concentrations lower than 0.100 mg/kg.

4.3 Assessment of laboratory performance.

No z score values were calculated. Classification was based on the number of results reported by each laboratory. Table 4.3.1a classifies the laboratories according to the number of present pesticides reported.

Table 4.3.1a Classification of laboratories
according to the number of present pesticides reported.

Laboratory Code	No of Reported Pesticides	% of Reported Pesticides	Other Reported Pesticides Not Confirmed by the Organiser
Lab004	15	100	0
Lab012*	15	100	0
Lab021	15	100	0
Lab033	15	100	0
Lab041	15	100	0
Lab057	15	100	0
Lab067	15	100	0
Lab079*	15	100	0
Lab025	15	100	2
Lab032	15	100	2
Lab001*	15	100	5
Lab084*	15	100	8
Lab007	14	93	0
Lab019	14	93	0
Lab020	14	93	0
Lab026	14	93	0
Lab034	14	93	0
Lab054	14	93	0
Lab059	14	93	0
Lab061	14	93	0
Lab062	14	93	0
Lab063*	14	93	0
Lab068	14	93	0
Lab082	14	93	0
Lab087	14	93	0
Lab070*	14	93	1
Lab074	14	93	2
Lab029*	14	93	3
Lab075	14	93	4
Lab078	14	93	4
Lab013	13	87	0
Lab018	13	87	0
Lab024	13	87	0
Lab027	13	87	0
Lab031	13	87	0
Lab038	13	87	0
Lab039	13	87	0
Lab040	13	87	0
Lab053	13	87	0
Lab069	13	87	0
Lab072	13	87	0
Lab081*	13	87	0
Lab023*	13	87	2

Laboratory Code	No of Reported Pesticides	% of Reported Pesticides	Other Reported Pesticides Not Confirmed by the Organiser
Lab035	13	87	2
Lab030*	12	80	0
Lab046	12	80	0
Lab071*	12	80	0
Lab090	12	80	0
Lab065*	12	80	4
Lab089*	12	80	4
Lab005	12	80	7
Lab002	11	73	0
Lab056*	11	73	0
Lab064	11	73	0
Lab076*	11	73	0
Lab042	11	73	1
Lab003	10	67	0
Lab015	10	67	0
Lab022	10	67	0
Lab048	10	67	0
Lab016	10	67	1
Lab073	10	67	1
Lab044	9	60	0
Lab037*	9	60	6
Lab049	8	53	0
Lab066	8	53	0
Lab008	8	53	1
Lab051	8	53	1
Lab077	8	53	1
Lab080	8	53	2
Lab014*	7	47	0
Lab060	7	47	1
Lab045	7	47	3
Lab006	6	40	0
Lab085	6	40	0
Lab043	5	33	0
Lab086	4	27	0
Lab011	3	20	0
Lab052	3	20	0
Lab083	2	13	3
Lab047	1	7	0
Lab050	1	7	0
Lab036*	0	0	1

* National Reference Laboratories for Fruit and Vegetables from the EU participating in this test.

The extraction methods used by the laboratories, the chromatographic techniques, detectors, instrumentation, etc… are detailed in Appendix 3 (available only on the EUPT-FV-SM08 webpage, not in the printed version).

In Table 4.3.1b there is a summary of the chromatographic techniques used for each pesticide, and a graphical representation is shown in Appendix 2.

Table 4.3.1b Chromatographic techniques used to determine each pesticide in the test item

Pesticide	Total Number of Laboratories Reporting Data	*Total Number of Reported Detections	GC	Full Scan GC	LC	Full Scan LC
Benalaxyl	78	86	40	11	46	19
Chlozolinate	53	54	50	14	4	2
Clomazone	71	78	30	13	48	18
Cyazofamid	62	64	1	0	63	21
Fenpyrazamine	28	28	3	2	25	13
Heptachlor	71	72	71	22	1	0
Isopyrazam	33	35	6	4	29	14
Phenthoate	76	85	47	13	38	17
Prosulfocarb	71	77	21	10	56	20
Prothiofos	71	72	57	15	15	6
Pyrethrin	53	133	29	17	104	43
Quintozene	60	79	79	24	0	0
Rotenone	64	68	2	1	66	22
Tetramethrin	71	78	48	14	30	15
Triticonazole	69	76	13	6	63	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.

Table 4.3.1c shows the number and percentage of the pesticides present in the sample which were reported by each laboratory. National Reference Laboratories are marked with an asterisk.

Table 4.3.1c. Number and Percentage of Present Pesticides Reported by Laboratory

Laboratory Code	Number of Present Pesticides Reported (15 Evaluated Pesticides)	% of Present Pesticides Reported (15 Evaluated Pesticides)
Lab001*	15	100
Lab002	11	73
Lab003	10	67
Lab004	15	100

Laboratory Code	Number of Present Pesticides Reported (15 Evaluated Pesticides)	% of Present Pesticides Reported (15 Evaluated Pesticides)
Lab005	12	80
Lab006	6	40
Lab007	14	93
Lab008	8	53
Lab011	3	20
Lab012*	15	100
Lab013	13	87
Lab014*	7	47
Lab015	10	67
Lab016	10	67
Lab018	13	87
Lab019	14	93
Lab020	14	93
Lab021	15	100
Lab022	10	67
Lab023*	13	87
Lab024	13	87
Lab025	15	100
Lab026	14	93
Lab027	13	87
Lab029*	14	93
Lab030*	12	80
Lab031	13	87
Lab032	15	100
Lab033	15	100
Lab034	14	93
Lab035	13	87
Lab036*	0	0
Lab037*	9	60
Lab038	13	87
Lab039	13	87
Lab040	13	87
Lab041	15	100
Lab042	11	73
Lab043	5	33
Lab044	9	60
Lab045	7	47
Lab046	12	80
Lab047	1	7
Lab048	10	67
Lab049	8	53
Lab050	1	7
Lab051	8	53
Lab052	3	20

Laboratory Code	Number of Present Pesticides Reported (15 Evaluated Pesticides)	% of Present Pesticides Reported (15 Evaluated Pesticides)
Lab053	13	87
Lab054	14	93
Lab056*	11	73
Lab057	15	100
Lab059	14	93
Lab060	7	47
Lab061	14	93
Lab062	14	93
Lab063*	14	93
Lab064	11	73
Lab065*	12	80
Lab066	8	53
Lab067	15	100
Lab068	14	93
Lab069	13	87
Lab070*	14	93
Lab071*	12	80
Lab072	13	87
Lab073	10	67
Lab074	14	93
Lab075	14	93
Lab076*	11	73
Lab077	8	53
Lab078	14	93
Lab079*	15	100
Lab080	8	53
Lab081*	13	87
Lab082	14	93
Lab083	2	13
Lab084*	15	100
Lab085	6	40
Lab086	4	27
Lab087	14	93
Lab089*	12	80
Lab090	12	80

* National Reference Laboratories for Fruit and Vegetables from the EU participating in this test.

5. CONCLUSIONS

Ninety-one laboratories agreed to participate in this eighth proficiency test on screening methods. Eighty-three laboratories submitted results. Eighteen of the laboratories were National Reference Laboratories for Fruit and Vegetables (marked with an asterisk on the graphs and tables). Twenty two EU Member States and in addition to these, 1 EFTA country (Switzerland) and four non-EU/EFTA countries (China, India, Kenya and Serbia) participated in this European Union Proficiency Test.

Most laboratories analysed the test item using methods based on both gas and liquid chromatography, combined with mass spectrometric detection. Of 1085 detections, 497 were made by GC and 588 by LC, 394 were made using full-scan, meaning 36% of detections (228 by full scan LC techniques and 166 by full scan GC techniques); 35% of the laboratories reported their results using HRAM (high resolution accurate mass spectrometry); 75.2 % of the results were reported indicating a concentration value.

Twelve of the 83 laboratories were able to detect all 15 present pesticides in the spinach test item. Thirteen laboratories detected less than 50 % of the pesticides present.

Sixty seven percent of the laboratories (56 laboratories) that reported results were able to find more than 70 % of the evaluated pesticides.

Twenty-six participants reported 50 different pesticides which were not present in the spinach test items. Whether this should be judged as poor performance, or not, depends on how each participant would act on these positive findings in routine analysis. If the reported pesticide was reported as positive with no further identifying confirmation, then the result would be a false positive and hence erroneous monitoring data would be reported. If the reported pesticide is regarded simply as 'suspect' or 'indicatively present', leading to additional analysis to confirm identity before reporting the result, then those pesticides indicated as 'other reported pesticides' in this report are not really an issue.

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

6. SUGGESTIONS FOR FUTURE WORK

The Organiser and the Scientific Committee consider that screening methods have provided additional value to the current quantitative multiresidue methods routinely used for monitoring purposes. The results of this test are most encouraging, but also indicate the need for continued evaluation of screening methods. Therefore, further proficiency tests will be organised to provide support to those laboratories using screening methods in order to extend their use and improve their reliability. These methods will be used more and more as screens/filters, to make routine laboratory work easier and faster. The need for screening method validation has been recognised and guidelines for such validation have been prepared and included in the Document SANTE/11945/2015.

Next year, the matrix of the test item will be lemon homogenate. Once again participants will be invited to report the estimated concentration of the pesticides identified. The concentration value will be used for informative purposes only, and not for the evaluation of the laboratories.

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8. ACKNOWLEDGEMENTS

The Organiser is grateful to the European Commission for funding this 8th European Proficiency Test for Screening Methods in Fruit and Vegetables.

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

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

APPENDIX 1. Results

Table AP1a Reported pesticides

Laboratory Code Total No of Reporting Laboratories = 83	Evaluated Pesticides (15)															Reported Pesticides by Laboratory	% of Reported Pesticides by Laboratory
	Benalaxyl	Chlozolinate	Clomazone	Cyazofamid	Fenpyrazamine	Heptachlor	Isopyrazam	Phenthoate	Prosulfocarb	Prothiofos	Pyrethrin	Quintozene	Rotenone	Tetramethrin	Triticonazole		
Lab001*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**15**	**100**
Lab002	R	R	R	R		R		R	R	R		R		R	R	**11**	**73**
Lab003	R		R			R		R	R	R		R	R	R	R	**10**	**67**
Lab004	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**15**	**100**
Lab005	R		R	R		R		R	R	R	R	R	R	R	R	**12**	**80**
Lab006	R	R	R			R				R		R				**6**	**40**
Lab007	R		R	R	R	R	R	R	R	R	R	R	R	R	R	**14**	**93**
Lab008	R	R	R			R		R	R	R				R		**8**	**53**
Lab011								R		R					R	**3**	**20**
Lab012*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**15**	**100**
Lab013	R	R	R	R		R		R	R	R	R	R	R	R	R	**13**	**87**
Lab014*	R			R					R	R	R		R		R	**7**	**47**
Lab015	R	R	R			R		R	R	R	R			R	R	**10**	**67**
Lab016	R		R	R		R		R	R		R		R	R	R	**10**	**67**
Lab018	R	R	R	R		R		R	R	R	R	R	R	R	R	**13**	**87**
Lab019	R		R	R	R	R	R	R	R	R	R	R	R	R	R	**14**	**93**
Lab020	R	R	R	R	R	R		R	R	R	R	R	R	R	R	**14**	**93**
Lab021	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**15**	**100**
Lab022	R		R	R		R		R	R	R	R		R	R		**10**	**67**
Lab023*	R	R	R	R		R		R	R	R	R	R	R	R	R	**13**	**87**
Lab024	R	R	R	R		R		R	R	R	R	R	R	R	R	**13**	**87**
Lab025	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**15**	**100**
Lab026	R	R	R	R	R	R		R	R	R	R	R	R	R	R	**14**	**93**
Lab027	R	R	R	R		R		R	R	R	R	R	R	R	R	**13**	**87**
Lab029*	R	R	R	R		R	R	R	R	R	R	R	R	R	R	**14**	**93**
Lab030*	R	R	R	R		R		R	R	R		R	R	R	R	**12**	**80**
Lab031	R	R	R	R		R		R	R	R	R	R	R	R	R	**13**	**87**
Lab032	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**15**	**100**
Lab033	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**15**	**100**
Lab034	R		R	R	R	R	R	R	R	R	R	R	R	R	R	**14**	**93**
Lab035	R	R	R	R		R		R	R	R	R	R	R	R	R	**13**	**87**
Lab036*																**0**	**0**
Lab037*	R	R	R			R		R		R		R		R	R	**9**	**60**
Lab038	R		R	R	R	R	R	R	R	R	R		R	R	R	**13**	**87**
Lab039	R		R	R	R	R	R	R	R	R		R	R	R	R	**13**	**87**
Lab040	R	R	R	R	R	R		R	R	R	R	R	R	R		**13**	**87**
Lab041	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**15**	**100**
Lab042	R		R	R		R		R	R	R	R		R	R	R	**11**	**73**
Lab043	R	R						R				R		R		**5**	**33**
Lab044	R	R				R		R		R	R		R	R	R	**9**	**60**
Lab045	R	R					R	R	R			R			R	**7**	**47**
Lab046	R	R	R	R		R		R	R	R		R	R	R	R	**12**	**80**
Lab047						R										**1**	**7**
Lab048	R		R	R				R	R	R		R	R	R	R	**10**	**67**
Lab049	R	R	R					R	R	R			R		R	**8**	**53**
Lab050						R										**1**	**7**
Lab051	R	R	R					R	R	R		R		R		**8**	**53**
Lab052	R			R										R		**3**	**20**

APPENDIX 1

Laboratory Code Total No of Reporting Laboratories = 83	Evaluated Pesticides (15)															Reported Pesticides by Laboratory	% of Reported Pesticides by Laboratory
	Benalaxyl	Chlozolinate	Clomazone	Cyazofamid	Fenpyrazamine	Heptachlor	Isopyrazam	Phenthoate	Prosulfocarb	Prothiofos	Pyrethrin	Quintozene	Rotenone	Tetramethrin	Triticonazole		
Lab053	R		R	R	R	R	R	R	R	R	R		R	R	R	**13**	**87**
Lab054	R	R	R	R		R	R	R	R	R	R	R	R	R	R	**14**	**93**
Lab056*	R	R	R	R		R		R	R	R		R		R	R	**11**	**73**
Lab057	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**15**	**100**
Lab059	R	R	R	R		R	R	R	R	R	R	R	R	R	R	**14**	**93**
Lab060	R		R			R		R	R			R		R		**7**	**47**
Lab061	R	R	R	R	R	R	R	R	R	R		R	R	R	R	**14**	**93**
Lab062	R	R	R	R	R	R	R	R	R	R	R	R	R	R		**14**	**93**
Lab063*	R	R	R	R		R	R	R	R	R	R	R	R	R	R	**14**	**93**
Lab064	R		R	R		R		R	R	R		R	R	R	R	**11**	**73**
Lab065*	R	R	R	R		R		R	R		R	R	R	R	R	**12**	**80**
Lab066	R		R		R		R	R			R		R		R	**8**	**53**
Lab067	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**15**	**100**
Lab068	R	R	R	R		R	R	R	R	R	R	R	R	R	R	**14**	**93**
Lab069	R	R	R	R		R		R	R	R	R	R	R	R	R	**13**	**87**
Lab070*	R	R	R	R		R	R	R	R	R	R	R	R	R	R	**14**	**93**
Lab071*	R		R	R		R		R	R	R	R	R	R	R	R	**12**	**80**
Lab072	R	R	R	R		R		R	R	R	R	R	R	R	R	**13**	**87**
Lab073	R	R	R			R		R	R	R		R		R	R	**10**	**67**
Lab074	R	R	R	R		R	R	R	R	R	R	R	R	R	R	**14**	**93**
Lab075	R	R	R	R		R	R	R	R	R	R	R	R	R	R	**14**	**93**
Lab076*	R		R	R		R		R	R	R		R	R	R	R	**11**	**73**
Lab077	R		R	R		R		R	R	R					R	**8**	**53**
Lab078	R	R	R	R	R	R		R	R	R	R	R	R	R	R	**14**	**93**
Lab079*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**15**	**100**
Lab080	R					R		R	R	R			R	R	R	**8**	**53**
Lab081*	R	R	R	R		R		R	R	R	R	R	R	R	R	**13**	**87**
Lab082	R	R	R	R	R	R		R	R	R	R	R	R	R	R	**14**	**93**
Lab083						R				R						**2**	**13**
Lab084*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	**15**	**100**
Lab085	R		R					R	R				R		R	**6**	**40**
Lab086	R					R		R						R		**4**	**27**
Lab087	R	R	R	R		R	R	R	R	R	R	R	R	R	R	**14**	**93**
Lab089*	R		R	R	R	R	R	R	R	R			R	R	R	**12**	**80**
Lab090	R		R	R	R		R	R	R	R	R		R	R	R	**12**	**80**
Reported Pesticides	**78**	**53**	**71**	**62**	**28**	**71**	**33**	**76**	**71**	**71**	**53**	**60**	**64**	**71**	**69**		
% of Reported Pesticides	**94**	**64**	**86**	**75**	**34**	**86**	**40**	**92**	**86**	**86**	**64**	**72**	**77**	**86**	**83**		

R: Reported pesticide

*NRLs from EU

Estimated Concentrations Reported
Not all the laboratories reporting results have reported estimated concentration values
Results reported without concentration values are expressed as R.
*NRLs from EU

Laboratory Code Total No of Reporting Laboratories = 83	Evaluated Pesticides (15)													
	Benalaxyl	Chlozolinate	Clomazone	Cyazofamid	Fenpyrazamine	Heptachlor	Isopyrazam	Phenthoate	Prosulfocarb	Prothiofos	Quintozene	Rotenone	Tetramethrin	Triticonazole
Homogeneity (mg/kg)	**0.053**	**0.042**	**0.085**	**0.068**	**0.060**	**0.058**	**0.085**	**0.065**	**0.057**	**0.086**	**0.058**	**0.085**	**0.071**	**0.065**
Robust Mean (mg/kg)	**0.052**	**0.047**	**0.075**	**0.070**	**0.049**	**0.061**	**0.074**	**0.068**	**0.051**	**0.076**	**0.063**	**0.073**	**0.068**	**0.055**
CV (%)	**20.3**	**42.4**	**25.0**	**33.3**	**17.7**	**38.1**	**23.2**	**24.0**	**22.4**	**24.7**	**34.0**	**22.5**	**30.4**	**20.7**
Lab001*	R	0.03	0.11	R	0.05	0.07	0.07	0.04	R	0.05	0.03	R	0.05	0.03
Lab002	0.066	R	R	0.077		R		R	R	R	R		0.114	0.06
Lab003	0.049		0.067			0.064		0.06	0.048	0.079	0.069	0.088	0.05	0.066
Lab004	0.056	0.042	0.09	0.057	0.044	0.1	0.068	0.082	0.063	0.09	0.066	0.16	0.073	0.065
Lab005	R		R	R		R		R	R	R	R	R	R	R
Lab006	0.05	0.08	0.08			0.08				0.08	0.07			
Lab007	0.06		0.075	0.07	0.06	0.075	R	0.09	R	0.085	0.075	0.08	0.085	0.07
Lab008	R	R	R			R		R	R	R			R	
Lab011								0.06		0.09				0.05
Lab012*	0.05	0.05	0.08	0.04	R	0.08	R	0.07	0.03	0.08	0.07	0.03	0.06	0.03
Lab013	R	R	R	R		R		R	R	R	R	R	R	R
Lab014*	0.07			0.15					0.14	0.1		0.11		0.06
Lab015	0.22	0.03	0.43			0.02		0.05	0.05	0.04			0.06	0.04
Lab016	0.063		0.087	0.059		0.007		0.075	0.059			R	0.067	0.069
Lab018	0.049	0.036	0.074	0.062		0.037		0.05	0.045	0.064	0.042	0.076	0.061	0.055
Lab019	R		R	R	R	R	R	R	R	R	R	R	R	R
Lab020	0.056	0.038	0.063	0.051	0.04	0.049		0.093	0.047	0.085	0.049	0.065	0.062	0.057
Lab021	0.07	0.006	0.12	0.065	0.055	0.067	0.1	0.082	0.074	0.093	0.093	0.088	0.13	0.06
Lab022	0.066		0.055	0.24		0.031		0.073	0.032	0.192		0.075	0.083	
Lab023*	R	R	R	R		0.09		0.07	R	0.09	0.09	0.1	0.065	0.07
Lab024	0.04	R	0.12	0.12		0.2		0.06	0.04	0.06	0.06	0.06	0.06	0.06
Lab025	0.047	0.035	0.068	0.065	0.045	0.075	0.077	0.068	0.053	0.067	0.071	0.091	0.068	0.058
Lab026	0.071	0.059	0.08	0.085	0.049	0.047		0.064	0.064	0.079	0.06	0.066	0.077	0.066
Lab027	R	R	R	R		R		R	R	R	R	R	R	R
Lab029*	0.05	0.04	0.09	0.08		0.09	0.08	0.08	0.05	0.08	0.06	0.08	0.06	0.04
Lab030*	R	R	R	R		R		R	R	R	R	R	R	R
Lab031	0.045	0.045	0.066	0.046		0.057		0.056	0.042	0.061	0.062	0.069	0.052	0.048
Lab032	0.052	0.034	0.103	0.053	0.042	0.052	0.084	0.080	0.049	0.108	0.135	0.046	0.099	0.062
Lab033	0.045	0.064	0.069	0.055	0.056	0.065	0.077	0.068	0.061	0.078	0.085	0.073	0.066	0.045
Lab034	R		R	R	R	R	R	R	R	R	R	R	R	R

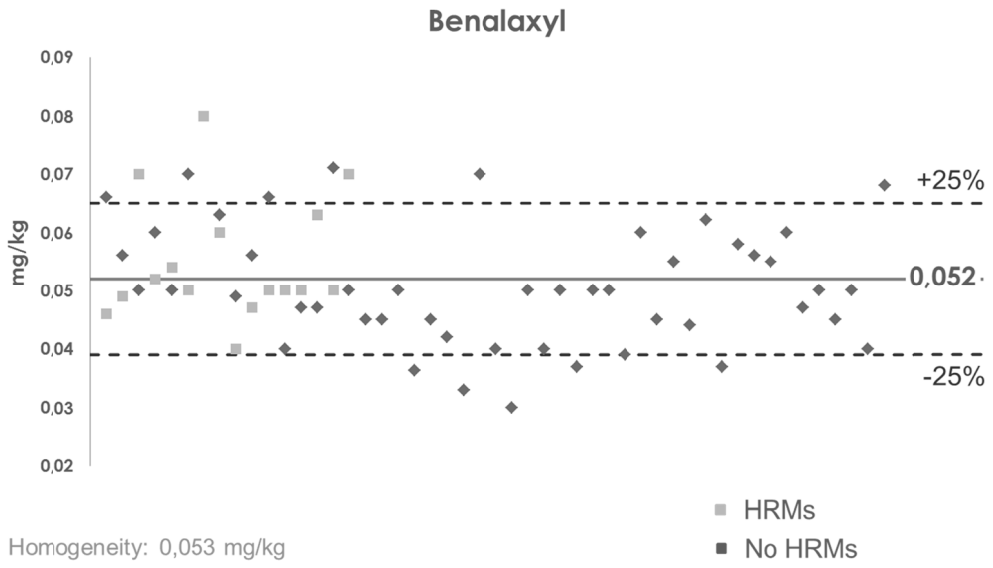
APPENDIX 1

Laboratory Code Total No of Reporting Laboratories = 83	Evaluated Pesticides (15)													
	Benalaxyl	Chlozolinate	Clomazone	Cyazofamid	Fenpyrazamine	Heptachlor	Isopyrazam	Phenthoate	Prosulfocarb	Prothiofos	Quintozene	Rotenone	Tetramethrin	Triticonazole
Homogeneity (mg/kg)	**0.053**	**0.042**	**0.085**	**0.068**	**0.060**	**0.058**	**0.085**	**0.065**	**0.057**	**0.086**	**0.058**	**0.085**	**0.071**	**0.065**
Robust Mean (mg/kg)	**0.052**	**0.047**	**0.075**	**0.070**	**0.049**	**0.061**	**0.074**	**0.068**	**0.051**	**0.076**	**0.063**	**0.073**	**0.068**	**0.055**
CV (%)	**20.3**	**42.4**	**25.0**	**33.3**	**17.7**	**38.1**	**23.2**	**24.0**	**22.4**	**24.7**	**34.0**	**22.5**	**30.4**	**20.7**
Lab035	0.05	0.01	0.06	0.15		0.06		0.06	0.05	0.09	0.07	0.07	0.06	0.06
Lab036*														
Lab037*	R	R	R			R		R		R	R		R	R
Lab038	0.054		0.07	0.06	0.05	0.073	0.093	0.082	0.063	0.079		0.059	0.038	0.05
Lab039	0.0364		0.0531	0.0629	0.0356	0.0375	0.0555	0.0554	0.0352	0.044	0.035	0.0598	0.0405	0.0399
Lab040	0.045	0.037	0.043	0.041	0.041	0.041		0.055	0.043	0.075	0.07	0.033	0.062	
Lab041	R	R	R	R	R	R	R	R	R	R	R	R	R	R
Lab042	R		R	R		R		R	R	R		R	R	R
Lab043	0.042	R						0.075			R		0.101	
Lab044	0.033	0.04				0.099		0.054		0.057		0.075	0.062	0.072
Lab045	R	R					R	R	R		R			R
Lab046	0.07	0.069	0.06	0.078		0.084		0.097	0.069	0.11	0.09	0.074	0.089	0.19
Lab047						0.062								
Lab048	0.04		0.08	0.06				0.05	0.03	0.06	0.05	R	0.05	0.06
Lab049	0.03	0.08	0.11					0.04	0.04	0.08		0.05		0.05
Lab050						0.038								
Lab051	R	R	R					R	R	R	R		R	
Lab052	0.05			0.14									0.05	
Lab053	0.04		0.055	0.046	0.04	0.025	0.058	0.045	0.043	0.047		0.05	0.041	0.046
Lab054	0.05	0.073	0.08	0.1		0.076	0.09	0.075	0.06	0.091	0.088	0.081	0.106	0.055
Lab056*	R	R	R	R		R		R	R	R	R		R	R
Lab057	0.05	0.046	0.082	0.074	0.048	0.067	0.07	0.079	0.057	0.08	0.076	0.069	0.074	0.067
Lab059	0.037	0.037	0.048	0.093		0.054	R	0.061	0.046	0.068	0.053	0.051	0.069	0.045
Lab060	0.08		0.09			0.08		0.07	0.06		0.11		0.16	
Lab061	0.05	0.075	0.055	0.06	0.045	0.08	0.075	0.09	0.045	0.06	0.07	0.08	0.07	0.045
Lab062	0.05	0.04	0.06	0.05	0.05	0.05	0.05	0.08	0.05	0.07	0.05	0.08	0.05	
Lab063*	0.039	0.024	0.063	0.078		0.077	0.06	0.053	0.04	0.058	0.05	0.12	0.052	0.082
Lab064	0.06		0.07	0.07		0.03		0.06	0.05	0.06	0.05	0.08	0.05	0.05
Lab065*	0.045	0.036	0.055	0.034		R		0.051	0.04		0.053	0.045	0.046	0.034
Lab066	R		R		R		R	R				R		R
Lab067	0.06	0.05	0.09	0.06	0.06	0.07	R	0.09	0.06	0.1	0.07	0.09	0.07	0.06
Lab068	0.055	R	0.077	0.067		0.047	0.061	0.064	0.053	0.07	0.074	0.085	0.065	0.056
Lab069	0.044	0.03	0.072	0.047		0.048		0.056	0.052	0.064	0.06	0.07	0.061	0.054

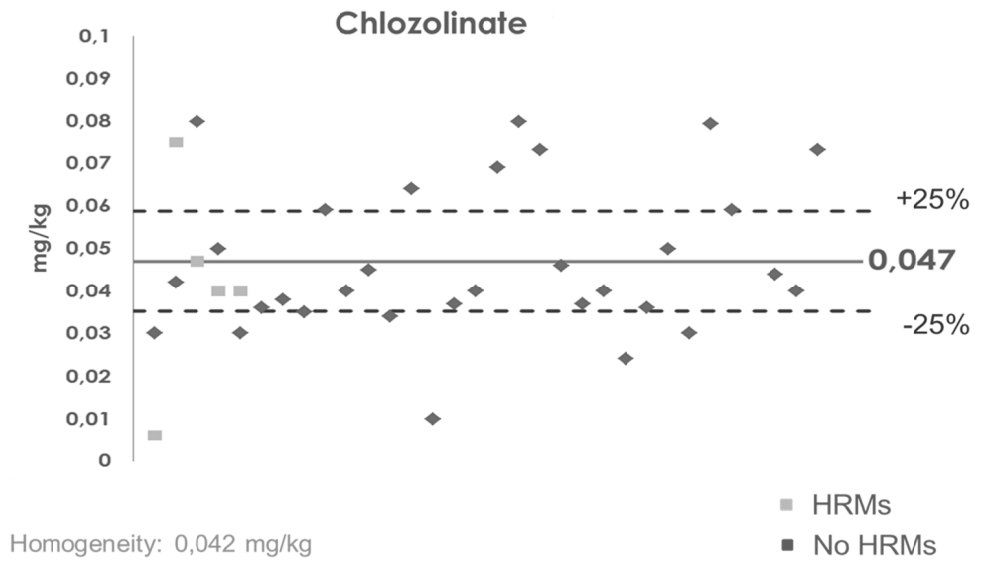
Laboratory Code Total No of Reporting Laboratories = 83	Evaluated Pesticides (15)													
	Benalaxyl	Chlozolinate	Clomazone	Cyazofamid	Fenpyrazamine	Heptachlor	Isopyrazam	Phenthoate	Prosulfocarb	Prothiofos	Quintozene	Rotenone	Tetramethrin	Triticonazole
Homogeneity (mg/kg)	**0.053**	**0.042**	**0.085**	**0.068**	**0.060**	**0.058**	**0.085**	**0.065**	**0.057**	**0.086**	**0.058**	**0.085**	**0.071**	**0.065**
Robust Mean (mg/kg)	**0.052**	**0.047**	**0.075**	**0.070**	**0.049**	**0.061**	**0.074**	**0.068**	**0.051**	**0.076**	**0.063**	**0.073**	**0.068**	**0.055**
CV (%)	**20.3**	**42.4**	**25.0**	**33.3**	**17.7**	**38.1**	**23.2**	**24.0**	**22.4**	**24.7**	**34.0**	**22.5**	**30.4**	**20.7**
Lab070*	0.0622	0.0794	0.0822	0.144		0.0739	0.15	0.072	0.0635	0.106	0.0752	0.0753	0.0835	0.055
Lab071*	0.037		R	0.044		0.055		0.056	R	0.054	0.065	0.048	0.047	0.039
Lab072	0.058	0.059	0.086	0.061		0.065		0.094	0.065	0.108	0.085	0.081	0.078	0.062
Lab073	0.047	0.047	R			0.053		R	R	0.081	R		R	0.066
Lab074	0.05	0.04	0.075	0.05		0.07	0.05	0.07	0.05	0.08	0.07	0.08	0.08	R
Lab075	0.056	R	0.08	0.042		0.073	0.08	0.074	0.056	0.099	0.077	0.063	0.067	0.06
Lab076*	0.055		0.074	0.069		0.06		0.059	0.043	0.074	0.051	0.068	0.072	0.045
Lab077	0.06		0.05	0.06		0.03		0.02	0.03	0.05				0.05
Lab078	R	R	R	R	R	R		R	R	R	R	R	R	R
Lab079*	0.047	0.114	0.073	0.238	0.043	0.06	R	0.07	0.038	0.088	0.062	0.084	0.066	0.071
Lab080	0.05					0.03		0.099	R	R		0.093	0.073	0.047
Lab081*	0.045	0.044	0.078	0.12		0.059		0.06	0.05	0.071	0.052	0.086	0.063	0.057
Lab082	0.05	0.04	0.08	0.06	0.041	0.045		0.06	0.055	0.07	0.07	0.07	4	0.052
Lab083						0.073				0.087				
Lab084*	0.063	R	R	R	R	R	R	R	0.061	R	R	R	0.077	R
Lab085	R		R					R	R			R		R
Lab086	0.04					0.02		0.04					0.04	
Lab087	0.068	0.073	0.099	0.053		0.105	0.067	0.087	0.076	0.076	0.103	0.059	0.091	0.051
Lab089*	0.05		0.07	2.5	0.07	0.052	0.05	0.054	0.051	0.055		0.06	0.04	0.05
Lab090	0.07		0.09	0.067	0.057		0.080	0.094	0.056	0.065		0.065	0.140	0.050

APPENDIX 2. Graphical Representations

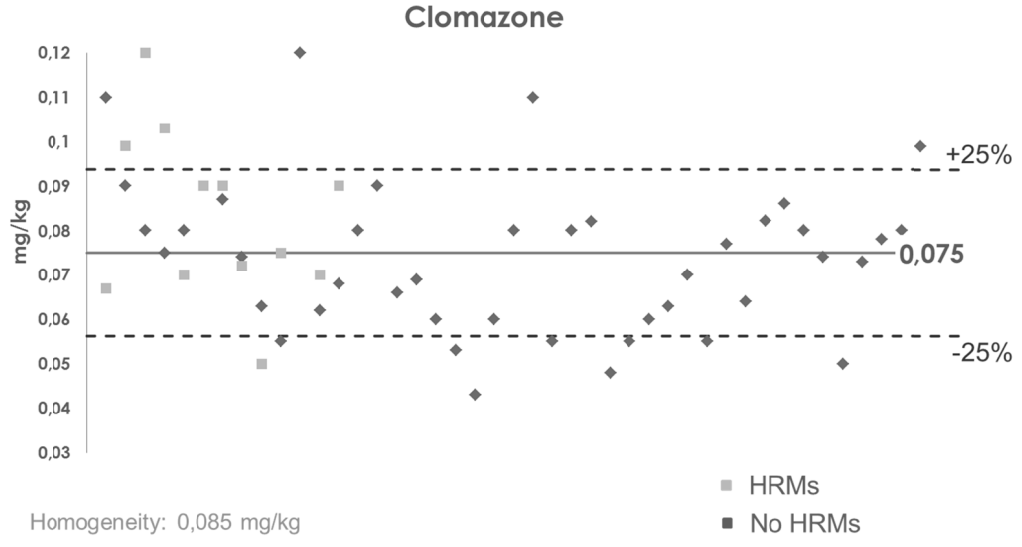
The bold line represents the robust mean



Homogeneity: 0,053 mg/kg



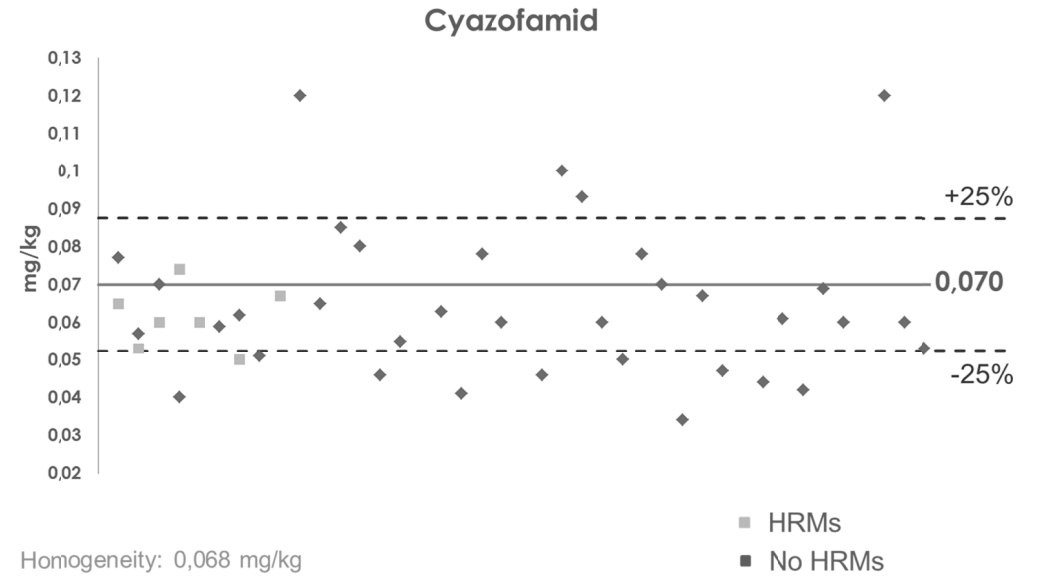
Homogeneity: 0,042 mg/kg



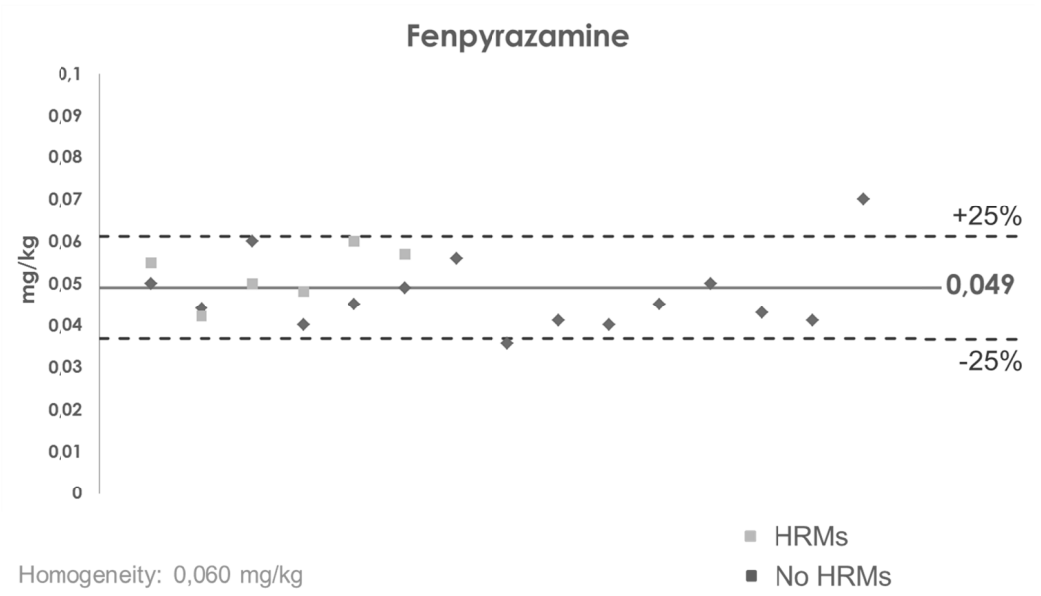
Homogeneity: 0,085 mg/kg

APPENDIX 2. Graphical Representations

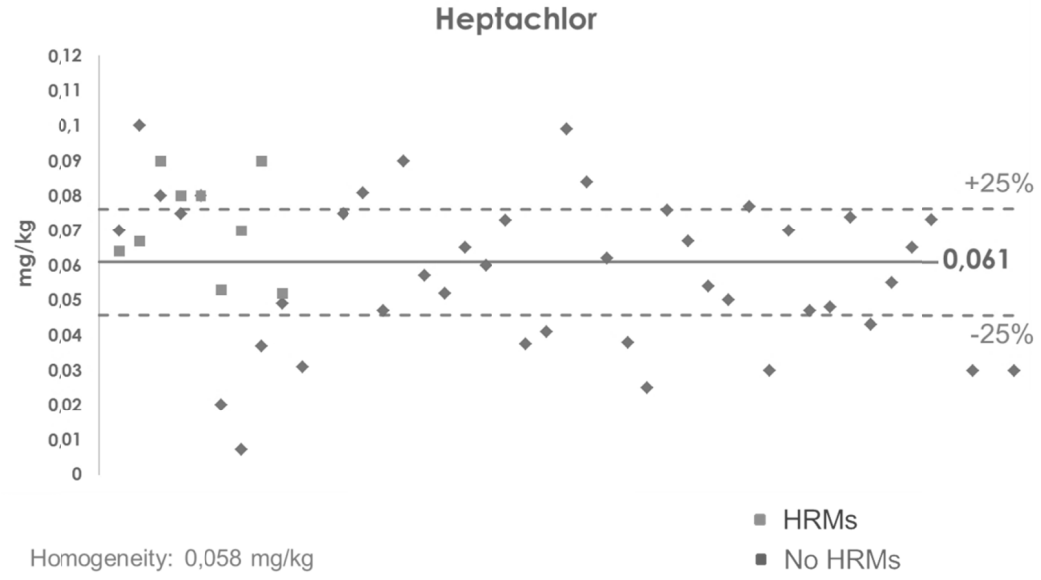
The bold line represents the robust mean



Homogeneity: 0,068 mg/kg

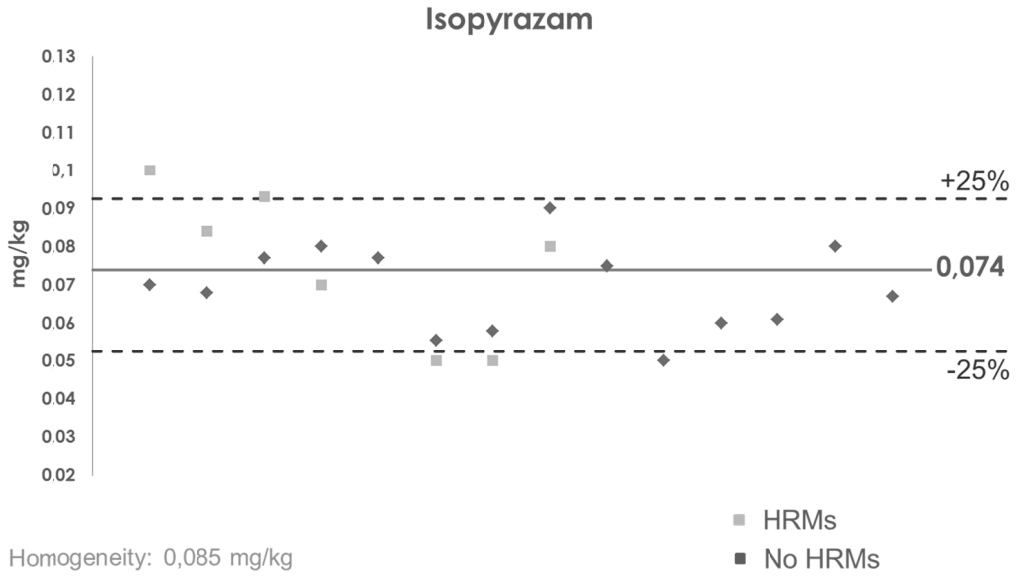


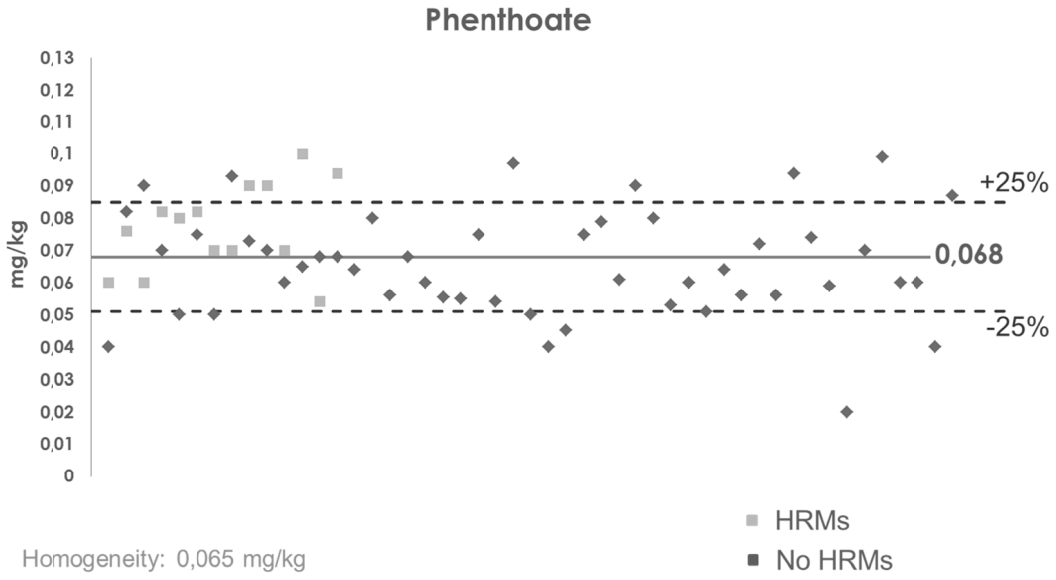
Homogeneity: 0,060 mg/kg

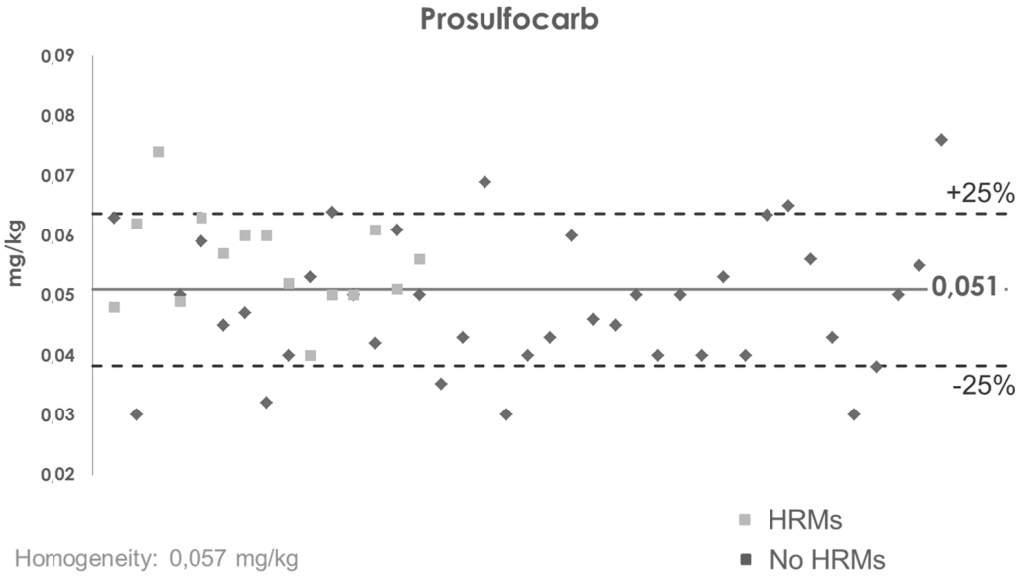


Homogeneity: 0,058 mg/kg

The bold line represents the robust mean

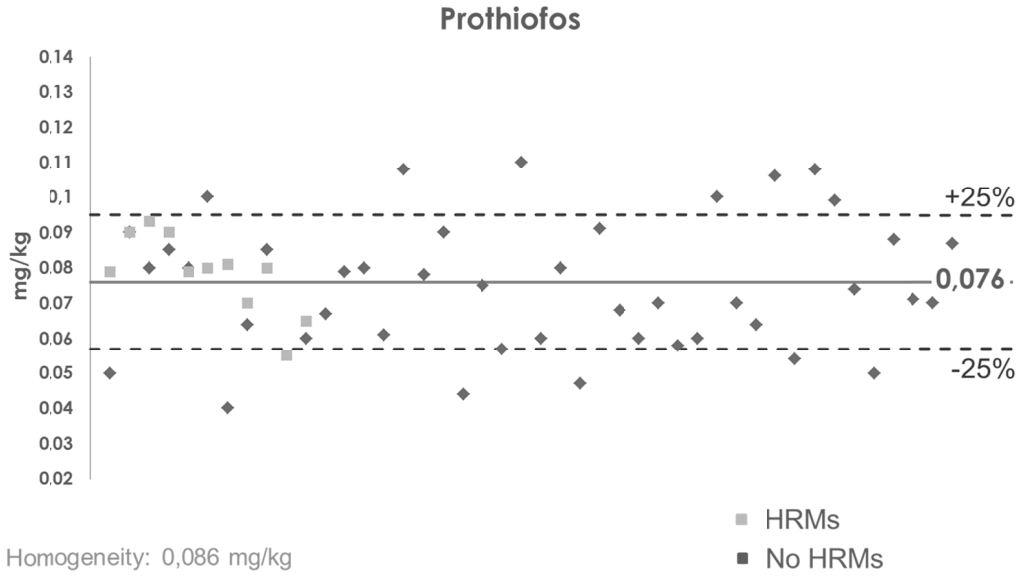




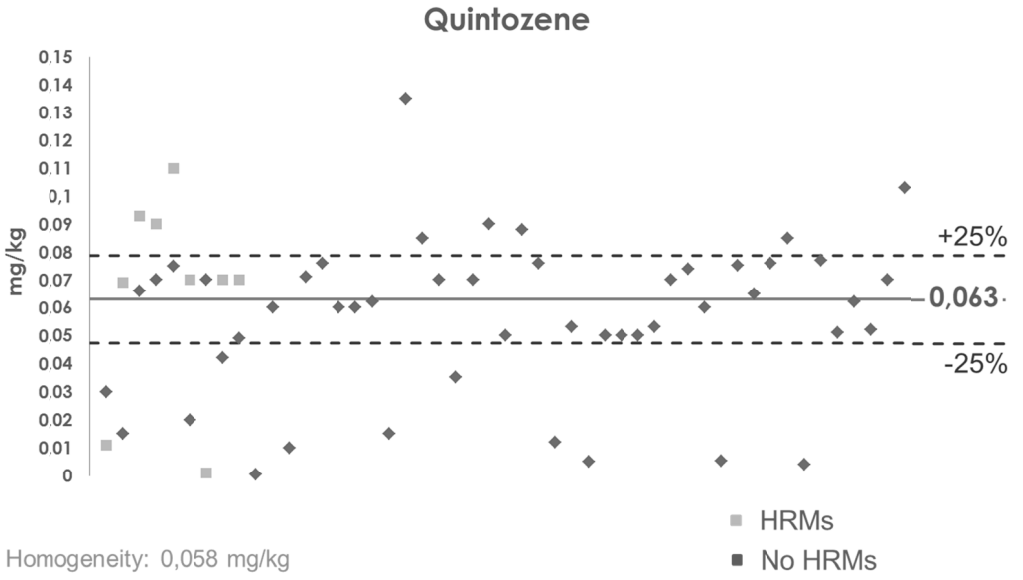


APPENDIX 2. Graphical Representations

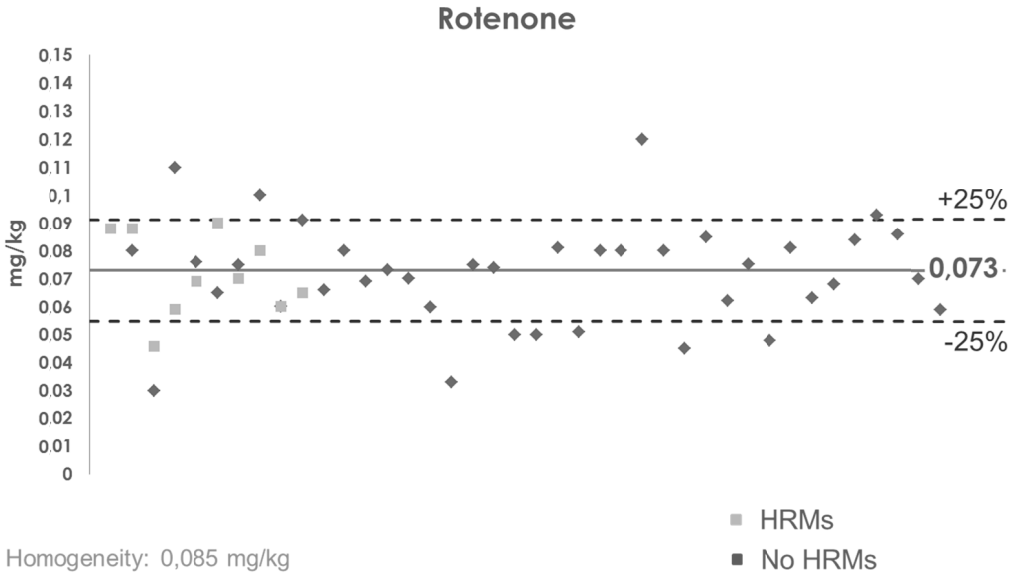
The bold line represents the robust mean



Homogeneity: 0,086 mg/kg

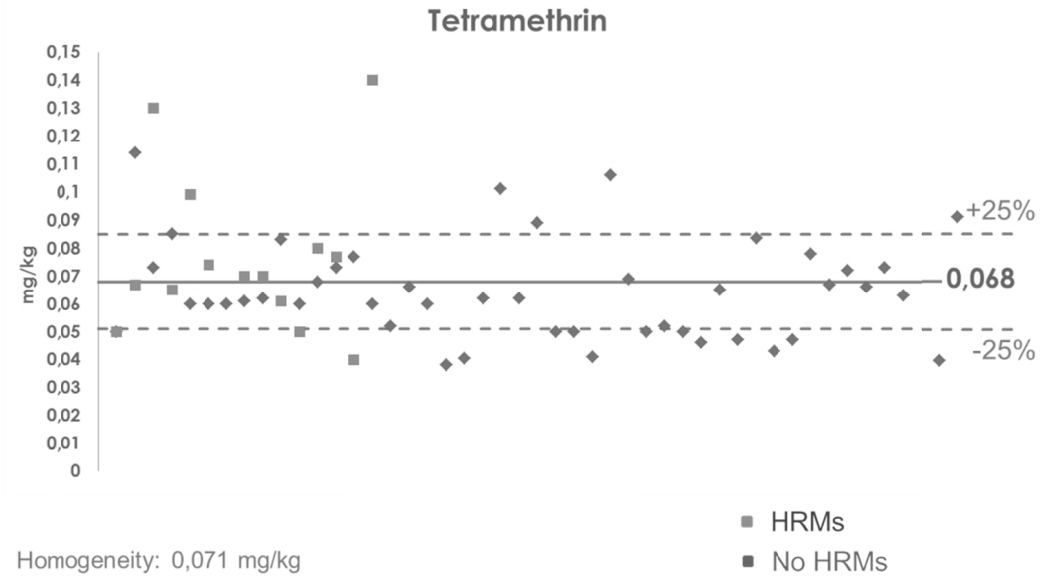


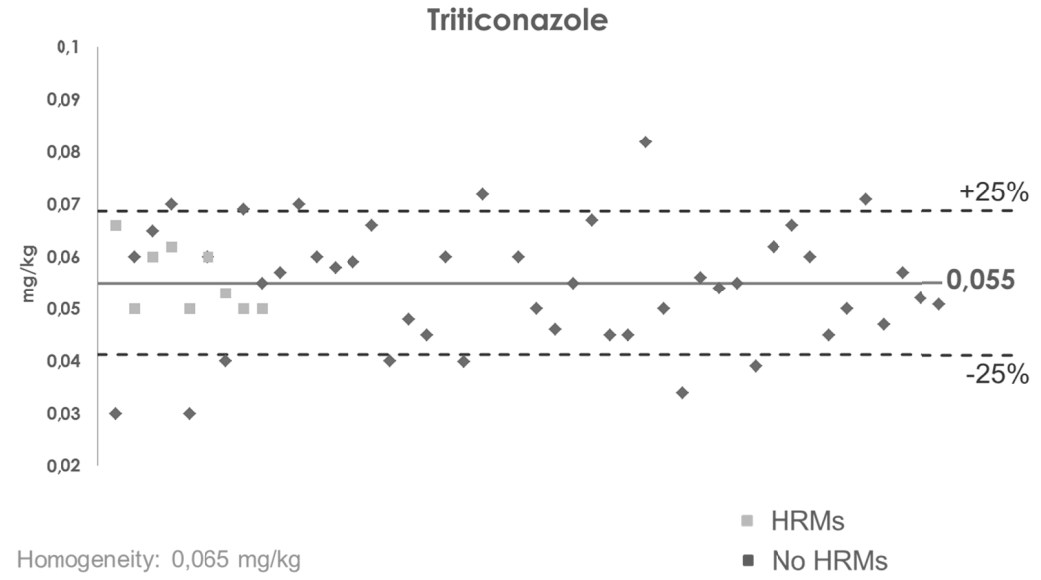
Homogeneity: 0,058 mg/kg



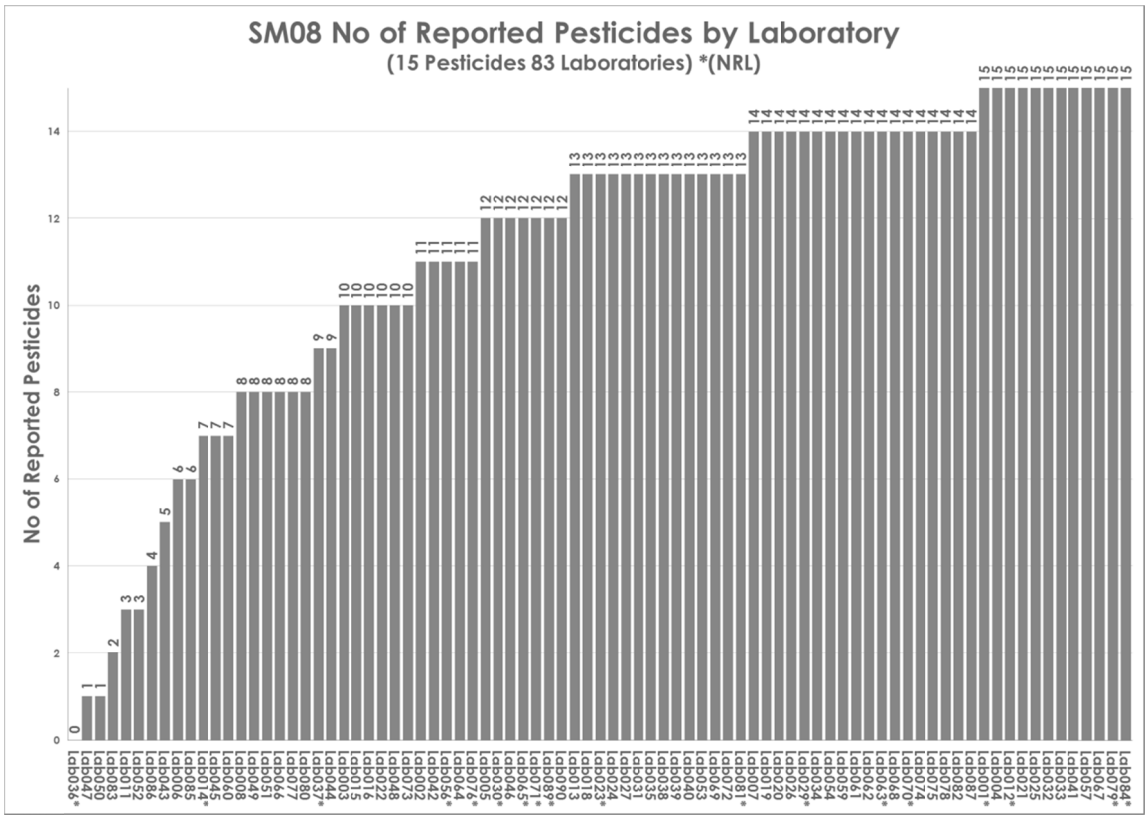
Homogeneity: 0,085 mg/kg

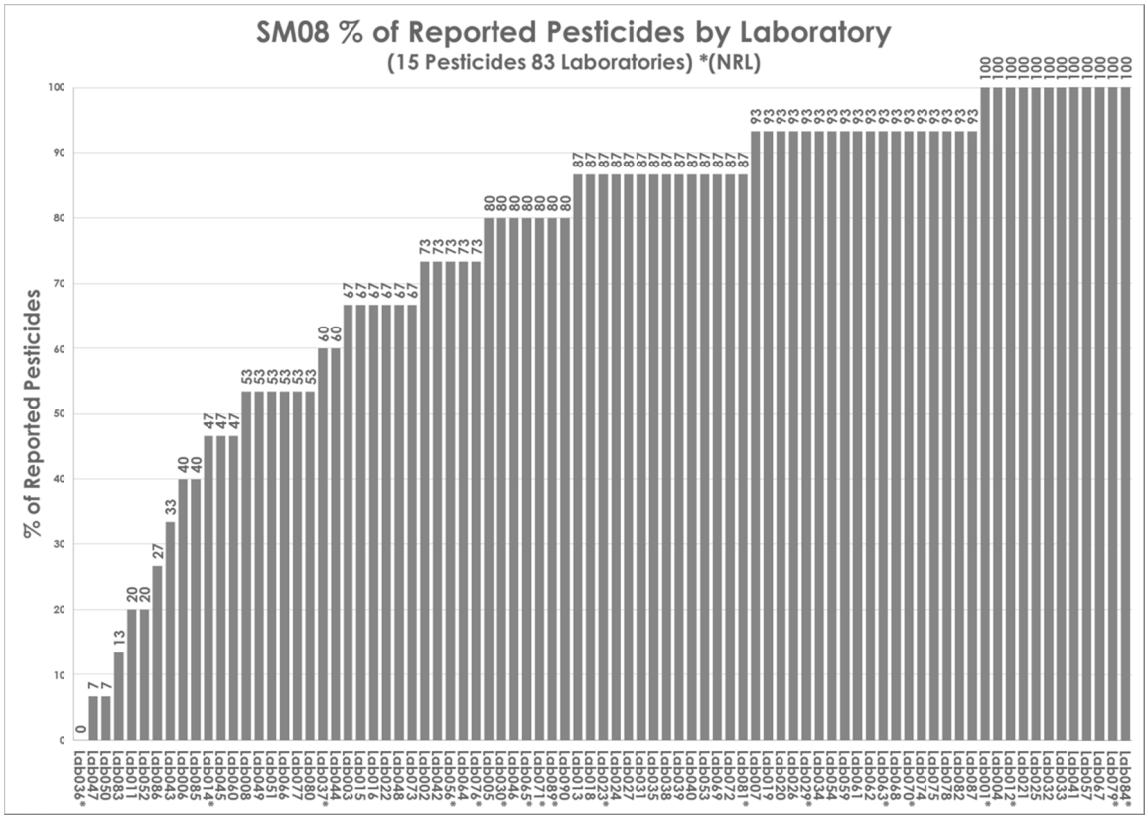
The bold line represents the robust mean

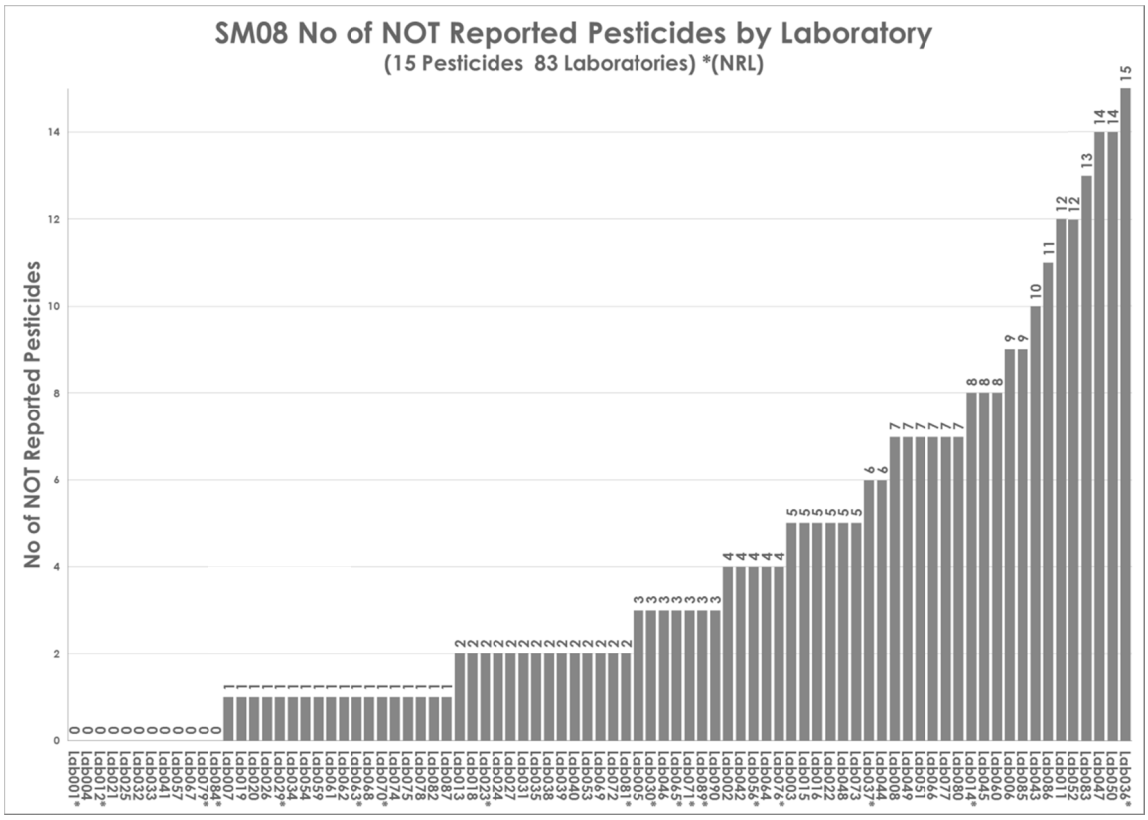


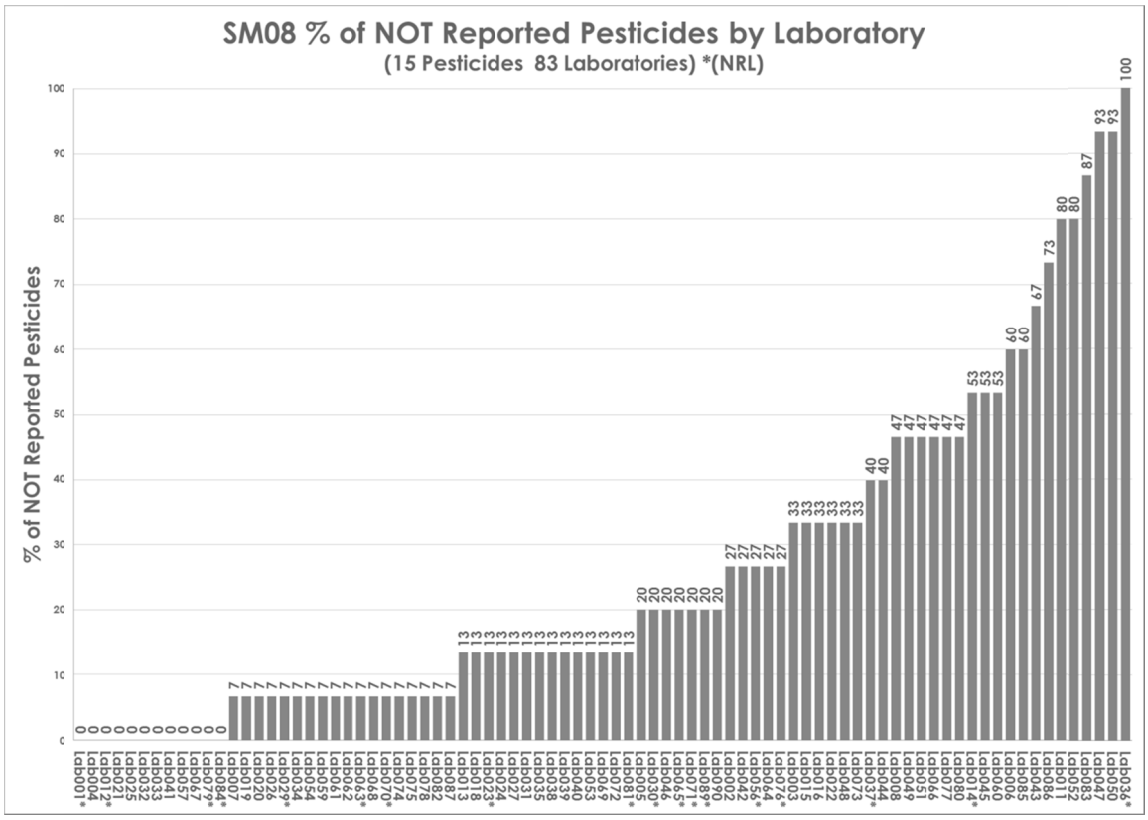


APPENDIX 2. Graphical Representations

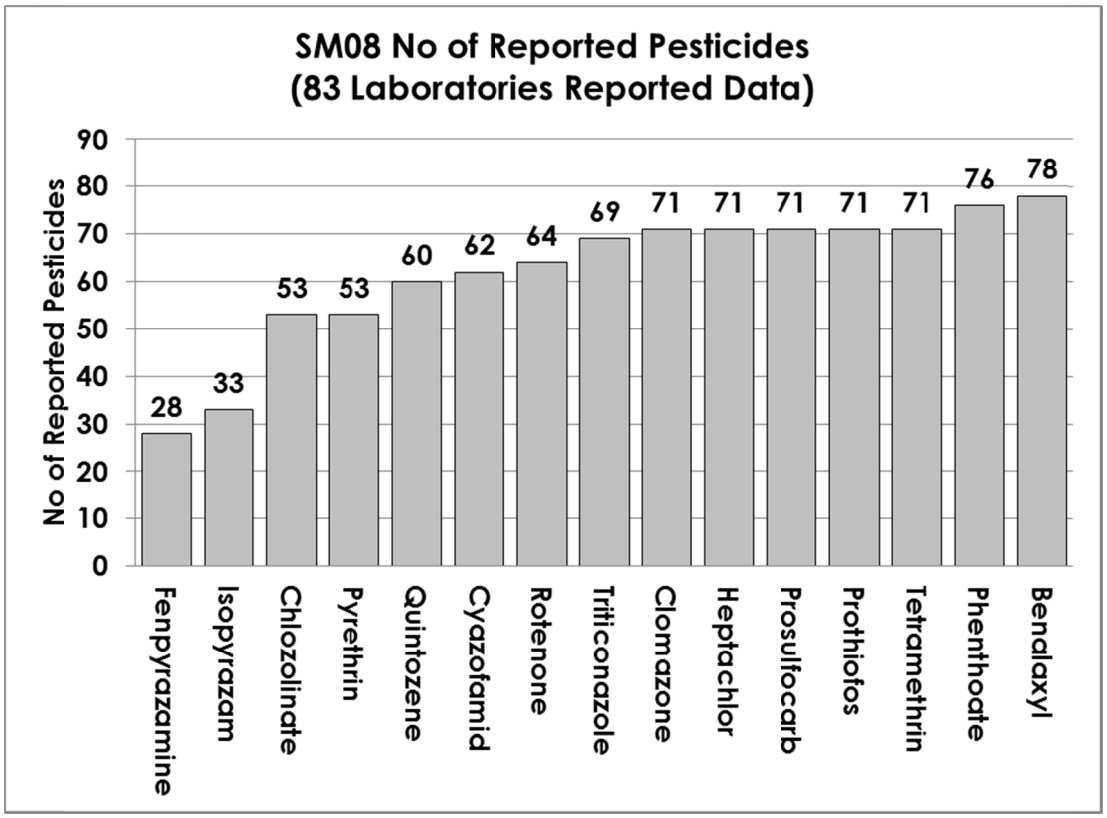


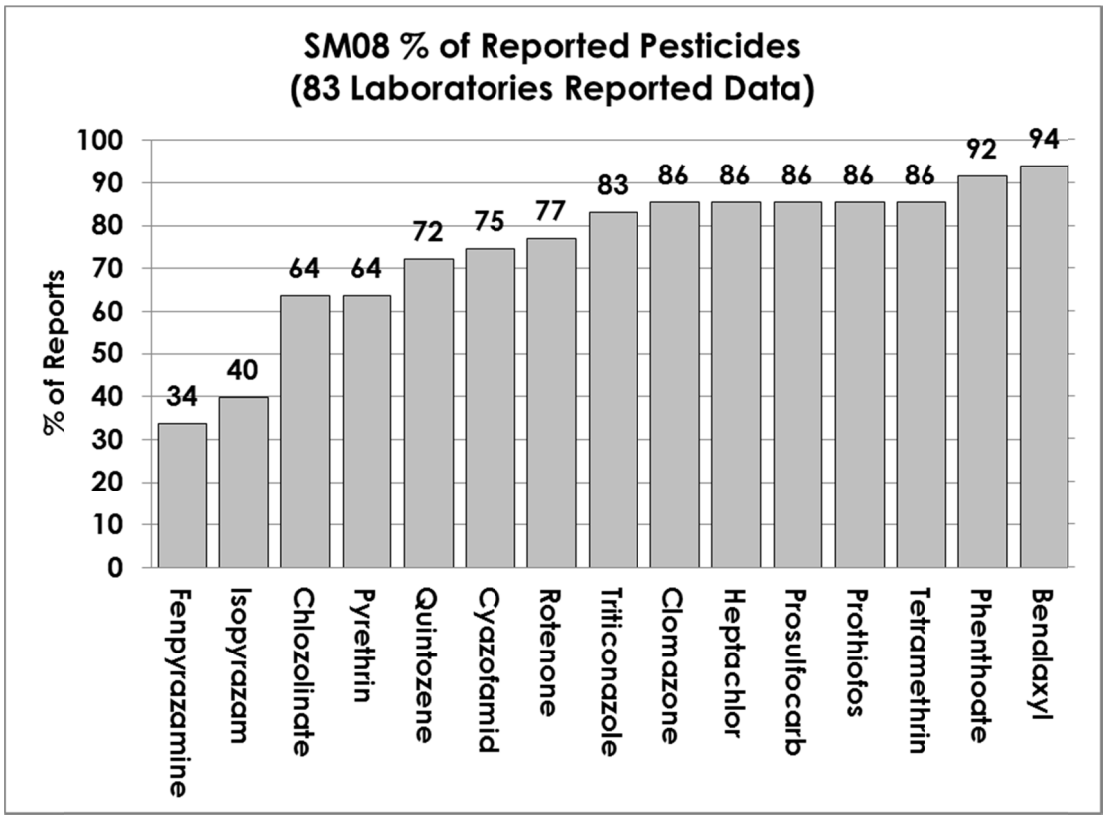



SM08 No of NOT Reported Pesticides by Laboratory
(15 Pesticides 83 Laboratories) *(NRL)
No of NOT Reported Pesticides

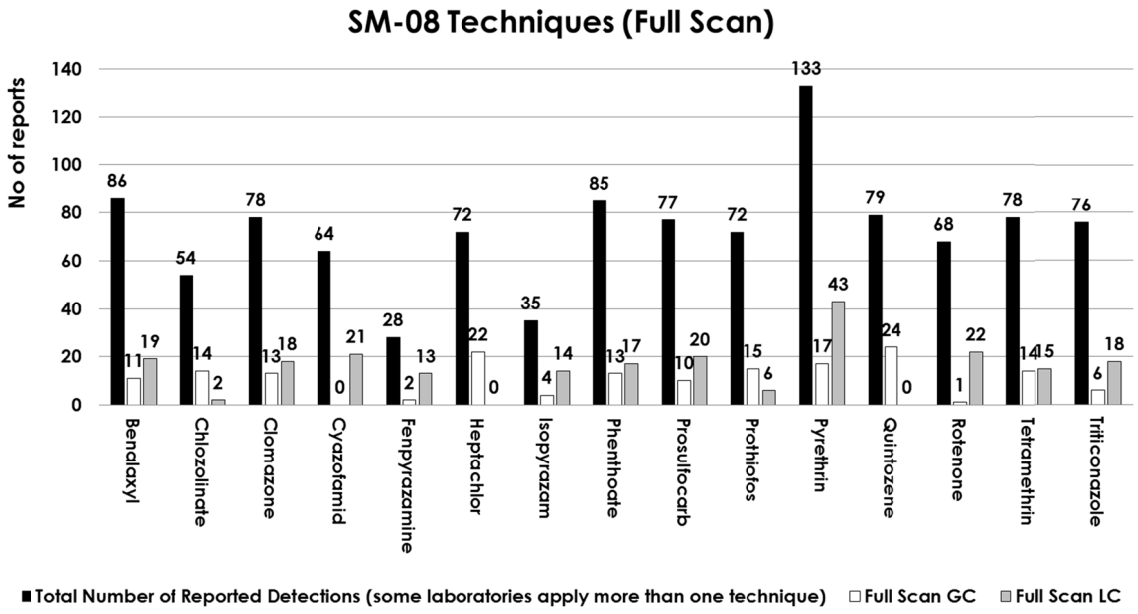

SM08 % of NOT Reported Pesticides by Laboratory
(15 Pesticides 83 Laboratories) *(NRL)
% of NOT Reported Pesticides

APPENDIX 2. Graphical Representations





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



ANNEX 1. List of Laboratories that participate in EUPT-FV-SM08.

COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
AUSTRIA	AUSTRIAN AGENCY FOR HEALTH AND FOOD SAFETY, INSTITUTE FOR FOOD SAFETY, DEPARTMENT FOR PESTICIDE AND FOOD ANALYSIS (PLMA)	INNSBRUCK	YES
BELGIUM	LOVAP NV	GEEL	YES
BELGIUM	PRIMORIS BELGIUM	ZWIJNAARDE	YES
BELGIUM	SCIENTIFIC INSTITUTE OF PUBLIC HEALTH	BRUSSELS	YES
BULGARIA	FYTOLAB BULGARIA LTD.	PLOVDIV	YES
CHINA	AGRO-PRODUCT SAFETY RESEARCH CENTER, CHINESE ACADEMY INSPECTION AND QUARANTINE	BEIJING	YES
CHINA	INSPECTION AND QUARANTINE TECHNIQUE CENTER OF QINHUANGDAO ENTRY-EXIT INSPECTION AND QUARANTINE BUREAU OF P. R. CHINA	QINHUANGDAO	YES
CHINA	SHANGHAI MUNICIPAL CENTER FOR DISEASE CONTROL AND PREVENTION	SHANGHAI	YES
CROATIA	TEACHING INSTITUTE OF PUBLIC HEALTH DR ANDRIJA ŠTAMPAR	ZAGREB	YES
CYPRUS	PESTICIDE RESIDUES LABORATORY OF THE STATE GENERAL LABORATORY	NICOSIA	YES
CZECH REPUBLIC	CENTRAL INSTITUTE FOR SUPERVISING AND TESTING IN AGRICULTURE (UKZUZ)	BRNO	YES
CZECH REPUBLIC	CZECH AGRICULTURE AND FOOD INSPECTION AUTHORITY	PRAHA	YES
CZECH REPUBLIC	DEPARTMENT OF FOOD ANALYSIS AND NUTRITION, UNIVERSITY OF CHEMISTRY AND TECHNOLOGY	PRAGUE	YES
DENMARK	NATIONAL FOOD INSTITUTE, TECHNICAL UNIVERSITY OF DENMARK	LYNGBY	YES
ESTONIA	LABORATORY FOR RESIDUES AND CONTAMINANTS, AGRICULTURAL RESEARCH CENTRE	SAKU	YES
FINLAND	FINNISH CUSTOMS LABORATORY	ESPOO	YES
FRANCE	ANSES-LSAL	MAISONS-ALFORT CEDEX	YES
FRANCE	CENTRE D'ANALYSES MÉDITERRANÉE PYRÉNÉES	PERPIGNAN	NO
FRANCE	CERECO SUD	GARONS	YES
FRANCE	INOVALYS LEMANS	LEMANS	YES
FRANCE	LABORATOIRE DU SCL-IDF MASSY	MASSY CEDEX	YES
FRANCE	LABORATOIRE DU SCL DE MONTPELLIER	MONTPELLIER	YES
FRANCE	LABORATORY PHYTOCONTROL	NIMES	YES
GERMANY	BVL-NRL FOR PESTICIDES	BERLIN	YES
GERMANY	BAYERISCHES LANDESAMT FÜR GESUNDHEIT UND LEBENSMITTELSICHERHEIT (LGL)	ERLANGEN	YES
GERMANY	CHEMICAL AND VETERINARY ANALYTICAL INSTITUT RHINE-RUHR-WUPPER	KREFELD	YES
GERMANY	CHEMISCHES LABOR DR. MANG	FRANKFURT AM MAIN	YES
GERMANY	CHEMISCHES UND VETERINÄRUNTERSUCHUNGSAMT STUTTGART	FELLBACH	YES
GERMANY	EUROFINS DR. SPECHT LABORATORIEN GMBH	HAMBURG	YES
GERMANY	EUROFINS SOFIA GMBH	BERLIN	NO
GERMANY	GALAB LABORATORIES GMBH	HAMBURG	YES

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COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
GERMANY	LABOR DR. LIPPERT	SINZIG	YES
GERMANY	LABOR FRIEDLE GMBH	TEGERNHEIM	YES
GERMANY	LANDESAMT FÜR LANDWIRTSCHAFT, LEBENSMITTELSICHERHEIT UND FISCHEREI	ROSTOCK	YES
GERMANY	LANDESAMT FÜR UMWELT UND ARBEITSSCHUTZ	SAARBRÜCKEN	NO
GERMANY	LTZ AUGUSTENBERG	KARLSRUHE	YES
GERMANY	LUA SACHSEN	DRESDEN	YES
GERMANY	LUFA-ITL GMBH	KIEL	YES
GERMANY	NDS. LANDESAMT FUER VERBRAUCHERSCHUTZ UND LEBENSMITTELSICHERHEIT	OLDENBURG	YES
GERMANY	ILAU GMBH	ANZING	YES
GREECE	BENAKI PHYTOPATHOLOGICAL INSTITUTE, PESTICIDE RESIDUE LAB	KIFISSIA	YES
HUNGARY	NATIONAL FOOD CHAIN OFFICE, DPPSCA, PESTICIDE RESIDUE ANALYTICAL LABORATORY, MISKOLC	MISKOLC	YES
HUNGARY	NATIONAL FOOD CHAIN SAFETY OFFICE, HODMEZOVASARHELY LABORATORY	HODMEZOVASARHELY	YES
HUNGARY	NFCSO PESTICIDE ANALYTICAL LABORATORY, VELENCE	VELENCE	YES
ICELAND	MATISOHF.	REYKJAVÍK	NO
INDIA	NATIONAL REFERRAL LABORATORY, ICAR-NRC FOR GRAPES	PUNE	YES
IRELAND	THE PESTICIDE CONTROL LABORATORY	CELBRIDGE	YES
ITALY	ARPA LAZIO SEZIONE DI LATINA	RIETI	NO
ITALY	ASL MILANO – LABORATORIO DI PREVENZIONE	MILANO	YES
ITALY	LABORATORIO DI SANITA' PUBBLICA AZIEND AUSL TOSCANA CENTRO	FIRENZE	YES
ITALY	A.R.P.A. VENETO DIP. LABORATORI S.O. VERONA	VERONA	YES
ITALY	APPA-SETTORE LABORATORIO – PROVINCIA AUTONOMA DI TRENTO	TRENTO	YES
ITALY	APPA BOLZANO – LABORATORIO ANALISI ACQUE E CROMATOGRAFIA	BOLZANO	YES
ITALY	ARPA FVG LABORATORIO ALIMENTI	PORDENONE	YES
ITALY	ARPA PUGLIA – POLO SPECIALIZZAZIONE ALIMENTI	BARI	NO
ITALY	ISTITUTO SUPERIORE DI SANITÀ	ROMA	YES
ITALY	ISTITUTO ZOOPROFILATTICO DELL'ABRUZZO E DEL MOLISE G.CAPORALE TERAMO ITALY	TERAMO	YES
ITALY	IZS SICILIA AREA CHIMICA E TECNOLOGIE ALIMENTARI	PALERMO	YES
KENYA	SGS KENYA LIMITED MOMBASA LABORATORY	MOMBASA	YES
LATVIA	INSTITUTE OF FOOD SAFETY ANIMAL HEALTH AND ENVIRONMENT BIOR	RIGA	YES
NORWAY	NORWEGIAN INSTITUTE OF BIOECONOMY RESEARCH, BIOTECHNOLOGY AND PLANT HEALTH DIVISION	AAS	YES
ROMANIA	LABORATORY FOR PESTICIDE RESIDUES CONTROL IN PLANTS AND VEGETABLES PRODUCTS	BUCURESTI	NO
ROMANIA	REGIONAL LABORATORY FOR PESTICIDE RESIDUES CONTROL IN PLANT AND PLANT PRODUCTS MURES	TARGUMURES	YES
SERBIA	CENTER FOR FOOD ANALYSIS	BELGRADE	YES

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COUNTRY	LABORATORY NAME	CITY	REPORTED RESULTS
SLOVENIA	NATIONAL LABORATORY FOR HEALTH, ENVIRONMENT AND FOOD	MARIBOR	YES
SPAIN	LABORATORIO AGROALIMENTARIO Y AMBIENTAL DE CASTILLA LA MANCHA	TOLEDO	YES
SPAIN	LABORATORIO AGROALIMENTARIO Y DE SANIDAD ANIMAL DE MURCIA	EL PALMAR (MURCIA)	YES
SPAIN	LABORATORIO DE LA DIRECCION PROVINCIAL DE LA CONSEJERIA DE SANIDAD	CUENCA	YES
SPAIN	LABORATORIO DE SALUD PÚBLICA DE BADAJOZ	BADAJOZ	YES
SPAIN	ANALYTICA ALIMENTARIA GMBH, SUCURSAL EN ESPAÑA	ALMERIA	YES
SPAIN	CNTA	SAN ADRIAN (NAVARRA)	YES
SPAIN	LABORATORIO AGROALIMENTARIO DE EXTREMADURA	CÁCERES	YES
SPAIN	LABORATORIO AGROALIMENTARIO DE VALENCIA	BURJASSOT (VALENCIA)	YES
SPAIN	LABORATORIO DE PRODUCCIÓN Y SANIDAD VEGETAL DE ALMERÍA	LA MOJONERA (ALMERÍA)	YES
SPAIN	LABORATORIO DE PRODUCCION Y SANIDAD VEGETAL DE JAEN	MENGIBAR (JAEN)	YES
SPAIN	LABORATORIO DEL SERVICIO DE INSPECCIÓN SOIVRE DE VALENCIA	VALENCIA	YES
SPAIN	LABORATORIO QUÍMICO MICROBIOLÓGICO, S.A	MURCIA	YES
SPAIN	LABORATORIO AGROALIMENTARIO DE GRANADA	ATARFE (GRANADA)	YES
SPAIN	LABORATORIO SECOSUR, S.A.	LORQUI-MURCIA	YES
SPAIN	LABORATORIO DE SALUD PUBLICA-MADRID SALUD	MADRID	YES
SPAIN	EUROFINS SICA AGRI Q	ALMERIA	YES
SWEDEN	EUROFINS FOOD & FEED TESTING SWEDEN AB	LIDKÖPING	YES
SWEDEN	SWEDISH NATIONAL FOOD AGENCY, DIVISION OF SCIENCE, DEPARTMENT OF CHEMISTRY	UPPSALA	YES
SWITZERLAND	KANTONALES LABORZÜRICH	ZÜRICH	YES
THE NETHERLANDS	GROEN AGROCONTROL	DELFGAUW	YES
THE NETHERLANDS	RIKILT-WAGENINGEN UR	WAGENINGEN	YES
THE NETHERLANDS	NVWA-NETHERLANDS FOOD AND CONSUMER PRODUCT SAFETY AUTHORITY	WAGENINGEN	YES
UNITED KINGDOM	FERA SCIENCE LTD	YORK	YES
UNITED KINGDOM	LGC TEDDINGTON	TEDDINGTON	NO
UNITED KINGDOM	EUROFINS FOOD TESTING UK LTD	WOLVERHAMPTON	YES
UNITED KINGDOM	SCIENTIFIC ANALYSIS LABORATORIES LTD	BARNHILL	YES