EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUIT AND VEGETABLES.

# SCREENING METHODS 16 (EUPT-FV-SM16)

## Pesticide Residues in banana homogenate

## Final Report

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# EUROPEAN UNION PROFICIENCY TEST FOR PESTICIDES IN FRUIT AND VEGETABLES. SCREENING METHODS 16

## BACKGROUND

According to Article 28 of Regulation 396/2005/EC of the European Parliament and European Council regarding maximum residue levels for pesticides in, or on, food and feed of plant and animal origin[1]: all laboratories analysing samples for the official control of pesticide residues shall participate in the European Union Proficiency Tests (EUPTs) for pesticide residues, facilitated by the Commission. These proficiency tests are carried out on an annual basis in order to ensure the quality, accuracy and comparability of the residue data reported by EU Member States to the European Commission, as well as by other Member States within the framework of coordinated national monitoring and surveillance programmes.

Regulation (EU) No 625/2017[2] lays down the responsibilities and tasks of European Union Reference Laboratories (EURLs) for Food, Feed and Animal Health. Among these tasks is the provision for regular inter-laboratory comparative testing or proficiency tests. This is a proficiency test on qualitative screening methods for pesticides in fruits and vegetable commodities.

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

Participation in this PT remains on a voluntary basis, given that the EURL-FV already organises the Proficiency Tests for quantitative multi-residue pesticide analysis (EUPT-FVs) over the same time period. Nevertheless, all FV-National Reference Laboratories (FV-NRLs) and FV-Official Laboratories (FV-OfLs) involved in the determination of pesticide residues in fruit and vegetables for the EU-coordinated monitoring programme, or for their own national programmes, are invited to take part.

DG-SANTE has full access to all EUPT data including the individual lab-codes/lab-name keys. This report may be presented to the Phytopharmaceuticals – Pesticides Residues section of the Plants, Animals, Food and Feed Committee.

[1]Regulation (EC) No 396/2005, published in the OJ of the EU L70 of 16.03.2005, as last amended by Regulation 839/2008 published in the OJ of the EU L234 of 30.08.2008.

[2]Regulation (EU) No 625/2017 of of the European Parliament and of the Council on official controls and other official activities performed to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection products. Published in the OJ of the EU L95/1 of 07/04/2017

## 1. INTRODUCTION

The EURL-FV has decided to continue its operation in these screening proficiency tests because of the good acceptance in the EURL-FV laboratory network.

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

The objective of the EURL-FV screening proficiency tests is for laboratories to be able to use mass-spectrometry-based screening methods routinely, following validation. This is in line with Document No SANTE/11312/2021v2 "Analytical quality control and method validation procedures for pesticide residues and analysis in food and feed".

This EUPT-FV-SM16 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 banana homogenate. Bananas were purchased in the local market in Almería, Spain.

The pesticides used to spike the banana 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-SM16 were mainly chosen taking into account the following considerations:

- That they were not included in the EU-Coordinated Multiannual Control Programme.
- That they had particularly acute toxicity and/or had low ARfD values.

Approximately 40 kg of bananas were ground and homogenised in a large stainless steel container. Before grinding, ascorbic acid was added to the bananas to prevent oxidation (1 % ascorbic acid). Subsequently, they were spiked with the analytical standards dissolved in acetonitrile. Once homogenized, the material was packed in zip bags and frozen at -18° C. Eight days later, the resulting ice blocks were crushed with ice crushers, and 200 g portions of the material were weighed out into screw-capped polyethylene plastic bottles, sealed and stored in a freezer at - 20 °C prior to distribution to participants.

The bananas purchased contained incurred pesticides: acetamiprid, azoxystrobin and pentachloroaniline. However, acetamiprid and pentachloroaniline were present at concentrations below 0.01 mg/kg, so they will not be evaluated in this Proficiency Test.

Hexachlorobenzene and tecnazene were present in the PT material because they are impurities of quintozene, one of the spiked pesticides.

Table 2.1.1 shows the pesticide residues detected in the EUPT-SM16 test item, but ONLY those compounds above 0.01 mg/kg have been considered for the evaluation of this proficiency test (table 2.1.2). All of them are above this concentration as can be seen in section 2.2.

Table 2.1.1 Pesticides present in the test item.

| **Spiked Pesticides** | | |
|---|---|---|
| Benzovindiflupyr | Isopyrazam | Quinalphos |
| Chloridazon | Metamitron | Quintozene |
| Clopyralid | Metazachlor | Rotenone |
| Fluazinam | Novaluron | Tecnazene* |
| Heptachlor | Oxadiargyl | Tetramethrin |
| Hexachlorobencene* | Penflufen | Tolfenpyrad |
| | Penthiopyrad | |
| **Pesticides in bananas** | | |
| Acetamiprid* | Azoxystrobin | Pentachloroaniline* |

*** below 0.01 mg/kg, not evaluated**

Table 2.1.2 Pesticides evaluated in EUPT-SM16

| **Evaluated Pesticides** | | |
|---|---|---|
| Azoxystrobin | Isopyrazam | Penthiopyrad |
| Benzovindiflupyr | Metamitron | Quinalphos |
| Chloridazon | Metazachlor | Quintozene |
| Clopyralid | Novaluron | Rotenone |
| Fluazinam | Oxadiargyl | Tetramethrin |
| Heptachlor | Penflufen | Tolfenpyrad |

### 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 to check for the presence of the pesticides.

The injection sequence of the 20 analyses by GC and LC was determined from a table of randomly-generated numbers. The statistical evaluation was performed according to the International Harmonized Protocol published by IUPAC, ISO and AOAC[3]. The results of the homogeneity tests are given in Table 2.2.1 The acceptance criteria for the test item to be sufficiently homogenous for the proficiency test were that: $Ss^2 < c$, where Ss is the between-bottle sampling standard deviation and $c = F_1\sigma^2_{all} + F_2S^2_{an}$; $F_1$ and $F_2$ being constant values of 1.88 and 1.01, respectively, from the ten samples taken, and $\sigma^2_{all}$ = 0.3 x FFP RSD(25 %) x the analytical sampling mean for all the pesticides. This was used to demonstrate that the between-bottle variance was not higher than the within-bottle variance. All the compounds passed the homogeneity test.

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

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

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

| Pesticide | Mean Conc. (mg/Kg) | $Ss^2$ | c | $Ss^2 < c$ Pass/Fail |
|---|---|---|---|---|
| Azoxystrobin | 0.037 | 0 | 0.00002 | Pass |
| Benzovindiflupyr | 0.037 | 0 | 0.00001 | Pass |
| Chloridazon | 0.162 | 0 | 0.00031 | Pass |
| Clopyralid | 0.080 | 0.00007 | 0.00010 | Pass |
| Fluazinam | 0.064 | 0 | 0.00005 | Pass |
| Heptachlor | 0.045 | 0.00003 | 0.00005 | Pass |
| Isopyrazam | 0.078 | 0 | 0.00007 | Pass |
| Metamitron | 0.098 | 0.00007 | 0.00017 | Pass |
| Metazachlor | 0.090 | 0.00003 | 0.00011 | Pass |
| Novaluron | 0.036 | 0.00001 | 0.00002 | Pass |
| Oxadiargyl | 0.036 | 0 | 0.00002 | Pass |
| Penflufen | 0.050 | 0 | 0.00003 | Pass |
| Penthiopyrad | 0.030 | 0.00001 | 0.00002 | Pass |
| Quinalphos | 0.116 | 0 | 0.00019 | Pass |
| Quintozene | 0.038 | 0 | 0.00002 | Pass |
| Rotenone | 0.072 | 0.00001 | 0.00007 | Pass |
| Tetramethrin | 0.108 | 0 | 0.00041 | Pass |
| Tolfenpyrad | 0.046 | 0 | 0.00003 | Pass |

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

A pesticide was considered to be adequately stable if $|x_1 - y_i| \leq 0.3 \times \sigma$, where x1 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).

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.

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.

### 2.3 Distribution of test item and protocol to participants

Approximately 200 g of banana homogenate were shipped to participants on 26th February 2024. 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-SM16 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

Fifty-five laboratories agreed to participate in this proficiency test on screening methods. Fifty-two laboratories submitted results on time. All results reported by the participants are given in Appendix 1. Graphical representations of the results reported are shown in Appendix 2. Details of the methods used are provided in Appendix 3 (available on the EUPT-FV-SM16 webpage, not in the printed version).

A summary of the results reported by pesticide can be seen in Table 4.1.1.

Table 4.1.1 Summary of Reported Results.

| Pesticide | Reported | | Not Reported | |
|---|---|---|---|---|
| | No. of laboratories | % of Laboratories# | No. of laboratories | % of laboratories * |
| Azoxystrobin | 51 | 98 | 1 | 2 |
| Benzovindiflupyr | 40 | 77 | 12 | 23 |
| Chloridazon | 43 | 83 | 9 | 17 |
| Clopyralid | 21 | 40 | 31 | 60 |
| Fluazinam | 39 | 75 | 13 | 25 |
| Heptachlor | 48 | 92 | 4 | 8 |
| Isopyrazam | 45 | 87 | 7 | 13 |
| Metamitron | 45 | 87 | 7 | 13 |
| Metazachlor | 46 | 88 | 6 | 12 |
| Novaluron | 43 | 83 | 9 | 17 |
| Oxadiargyl | 38 | 73 | 14 | 27 |
| Penflufen | 37 | 71 | 15 | 29 |
| Penthiopyrad | 46 | 88 | 6 | 12 |
| Quinalphos | 51 | 98 | 1 | 2 |
| Quintozene | 45 | 87 | 7 | 13 |
| Rotenone | 46 | 88 | 6 | 12 |
| Tetramethrin | 51 | 98 | 1 | 2 |
| Tolfenpyrad | 41 | 79 | 11 | 21 |

#The % of laboratories is calculated based on the total number of laboratories submitting results (52 laboratories).

In this EUPT-FV-SM16, the estimated concentration was requested for those pesticides that were detected, only for informative purposes. However, not all the laboratories reported concentration results (Appendix 1 – Estimated Concentrations Reported). Table 4.1.2 shows the average concentration from the homogeneity test, the robust mean of the estimated concentrations reported by EU/EFTA laboratories, the number of concentration results reported and the dispersion of the concentration results reported.

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

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

| Pesticides | No of Conc. Reported | No of Conc. Reported by EU/EFTA labs | Conc. Homogeneity Test (mg/kg) | Robust mean (mg/kg) | CV (%) |
|---|---|---|---|---|---|
| Azoxystrobin | 51 | 49 | 0.037 | 0.038 | 21.4 |
| Benzovindiflupyr | 41 | 39 | 0.037 | 0.037 | 22.5 |
| Chloridazon | 41 | 40 | 0.162 | 0.156 | 18.4 |
| Clopyralid | 22 | 21 | 0.080 | 0.067 | 49.7 |
| Fluazinam | 38 | 38 | 0.064 | 0.062 | 22.9 |
| Heptachlor | 50 | 48 | 0.045 | 0.045 | 30.0 |
| Isopyrazam | 46 | 44 | 0.078 | 0.084 | 21.3 |
| Metamitron | 45 | 44 | 0.098 | 0.100 | 24.0 |
| Metazachlor | 46 | 44 | 0.090 | 0.088 | 16.6 |
| Novaluron | 43 | 41 | 0.036 | 0.034 | 30.8 |
| Oxadiargyl | 36 | 34 | 0.036 | 0.037 | 23.7 |
| Penflufen | 37 | 36 | 0.050 | 0.050 | 15.7 |
| Penthiopyrad | 48 | 46 | 0.030 | 0.031 | 23.5 |
| Quinalphos | 54 | 52 | 0.116 | 0.106 | 18.8 |
| Quintozene | 46 | 45 | 0.038 | 0.038 | 28.0 |

| Pesticides | No of Conc. Reported | No of Conc. Reported by EU/EFTA labs | Conc. Homogeneity Test (mg/kg) | Robust mean (mg/kg) | CV (%) |
|---|---|---|---|---|---|
| Rotenone | 45 | 43 | 0.072 | 0.071 | 21.9 |
| Tetramethrin | 53 | 51 | 0.108 | 0.110 | 23.3 |
| Tolfenpyrad | 40 | 38 | 0.046 | 0.051 | 19.0 |

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

Some pesticides were reported by more than 3 laboratories:

- Acetamiprid (34 laboratories)
- Hexachlorobenzene (5 laboratories)
- Pentachloroaniline (18 laboratories)
- Tecnazene (4 laboratories)

and they were present in the sample but below 0.01 mg/kg; therefore they were not included in the evaluation of the labs. Acetamiprid and pentachloroaniline were present in the bananas, and hexachlorobenzene and tecnazene were impurities of quintozene, one of the spiked pesticides.

4.1.1 Other Reported Compounds

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

Table 4.1.1.1 Other reported compounds.

| Laboratory Code | Other reported compounds | Concentration Reported (mg/kg) |
|---|---|---|
| LAB009 | 2-Phenylphenol | |
| LAB009 | Biphenyl | |
| LAB007 | Bromide | 2.100 |
| LAB014 | Chlordane | |
| LAB052 | Diflubenzuron | 0.074 |
| LAB015 | Dikegulac | |
| LAB013 | Ditalimfos | 0.088 |
| LAB007 | Dithiocarbamates | 0.016 |
| LAB013 | Dodemorph | |
| LAB052 | Flufenacet | 0.004 |
| LAB010 | Fluxapyroxad | 0.001 |
| LAB051 | Mercaptobenzothiazole | |
| LAB014 | Metaldehyde | |
| LAB014 | Phthalimide | |
| LAB012 | S-421 | 0.024 |
| LAB009 | Thiabendazole | |

| Laboratory Code | Other reported compounds | Concentration Reported (mg/kg) |
|---|---|---|
| LAB046 | Trichlorfon | 0.020 |
| LAB010 | Triclosan | 0.086 |

### 4.2 Assessment of laboratory performance.

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

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

| Laboratory Code | Number of Present Pesticides Reported (18 Evaluated Pesticides) | % of Present Pesticides Reported (18 Evaluated Pesticides) | Other Reported Pesticides Not Confirmed by the Organiser |
|---|---|---|---|
| LAB003 | 18 | 100 | |
| LAB004 | 18 | 100 | |
| LAB006 | 18 | 100 | |
| LAB017 | 18 | 100 | |
| LAB018 | 18 | 100 | |
| LAB019 | 18 | 100 | |
| LAB025 | 18 | 100 | |
| LAB026 | 18 | 100 | |
| LAB029 | 18 | 100 | |
| LAB030 | 18 | 100 | |
| LAB039 | 18 | 100 | |
| LAB042 | 18 | 100 | |
| LAB007 | 18 | 100 | 2 |
| LAB010 | 18 | 100 | 2 |
| LAB013 | 18 | 100 | 2 |
| LAB016 | 17 | 94 | |
| LAB020 | 17 | 94 | |
| LAB027 | 17 | 94 | |
| LAB031 | 17 | 94 | |
| LAB033 | 17 | 94 | |
| LAB037 | 17 | 94 | |
| LAB038 | 17 | 94 | |
| LAB040 | 17 | 94 | |
| LAB055 | 17 | 94 | |
| LAB015 | 17 | 94 | 1 |
| LAB051 | 17 | 94 | 1 |
| LAB014 | 17 | 94 | 3 |
| LAB008 | 16 | 89 | |
| LAB022 | 16 | 89 | |
| LAB024 | 16 | 89 | |
| LAB032 | 16 | 89 | |
| LAB043 | 16 | 89 | |
| LAB045 | 16 | 89 | |
| LAB002 | 15 | 83 | |
| LAB011 | 15 | 83 | |
| LAB041 | 15 | 83 | |

| Laboratory Code | Number of Present Pesticides Reported (18 Evaluated Pesticides) | % of Present Pesticides Reported (18 Evaluated Pesticides) | Other Reported Pesticides Not Confirmed by the Organiser |
|---|---|---|---|
| LAB012 | 15 | 83 | 1 |
| LAB001 | 14 | 78 | |
| LAB034 | 14 | 78 | |
| LAB048 | 14 | 78 | |
| LAB035 | 13 | 72 | |
| LAB046 | 13 | 72 | 1 |
| LAB023 | 11 | 61 | |
| LAB052 | 11 | 61 | 2 |
| LAB005 | 10 | 56 | |
| LAB036 | 10 | 56 | |
| LAB050 | 10 | 56 | |
| LAB053 | 8 | 44 | |
| LAB021 | 7 | 39 | |
| LAB028 | 4 | 22 | |
| LAB009 | 4 | 22 | 3 |
| LAB054 | 3 | 17 | |

The extraction methods used by the laboratories, the chromatographic techniques, detectors, instrumentation, etc… are detailed in Appendix 3 (available only on the EUPT-FV-SM16 webpage).

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

Table 4.2.2 Results classified by laboratory code

| Laboratory Code | Number of Present Pesticides Reported (18 Evaluated Pesticides) | % of Present Pesticides Reported (18 Evaluated Pesticides) | Other Reported Pesticides Not Confirmed by the Organiser |
|---|---|---|---|
| LAB001 | 14 | 78 | |
| LAB002 | 15 | 83 | |
| LAB003 | 18 | 100 | |
| LAB004 | 18 | 100 | |
| LAB005 | 10 | 56 | |
| LAB006 | 18 | 100 | |
| LAB007 | 18 | 100 | 2 |
| LAB008 | 16 | 89 | |
| LAB009 | 4 | 22 | 3 |
| LAB010 | 18 | 100 | 2 |
| LAB011 | 15 | 83 | |
| LAB012 | 15 | 83 | 1 |
| LAB013 | 18 | 100 | 2 |
| LAB014 | 17 | 94 | 3 |
| LAB015 | 17 | 94 | 1 |
| LAB016 | 17 | 94 | |
| LAB017 | 18 | 100 | |
| LAB018 | 18 | 100 | |
| LAB019 | 18 | 100 | |
| LAB020 | 17 | 94 | |
| LAB021 | 7 | 39 | |

| Laboratory Code | Number of Present Pesticides Reported (18 Evaluated Pesticides) | % of Present Pesticides Reported (18 Evaluated Pesticides) | Other Reported Pesticides Not Confirmed by the Organiser |
|---|---|---|---|
| LAB022 | 16 | 89 | |
| LAB023 | 11 | 61 | |
| LAB024 | 16 | 89 | |
| LAB025 | 18 | 100 | |
| LAB026 | 18 | 100 | |
| LAB027 | 17 | 94 | |
| LAB028 | 4 | 22 | |
| LAB029 | 18 | 100 | |
| LAB030 | 18 | 100 | |
| LAB031 | 17 | 94 | |
| LAB032 | 16 | 89 | |
| LAB033 | 17 | 94 | |
| LAB034 | 14 | 78 | |
| LAB035 | 13 | 72 | |
| LAB036 | 10 | 56 | |
| LAB037 | 17 | 94 | |
| LAB038 | 17 | 94 | |
| LAB039 | 18 | 100 | |
| LAB040 | 17 | 94 | |
| LAB041 | 15 | 83 | |
| LAB042 | 18 | 100 | |
| LAB043 | 16 | 89 | |
| LAB045 | 16 | 89 | |
| LAB046 | 13 | 72 | 1 |
| LAB048 | 14 | 78 | |
| LAB050 | 10 | 56 | |
| LAB051 | 17 | 94 | 1 |
| LAB052 | 11 | 61 | 2 |
| LAB053 | 8 | 44 | |
| LAB054 | 3 | 17 | |
| LAB055 | 17 | 94 | |

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

Table 4.2.3 Chromatographic techniques used to determine each pesticide in the test item

| Pesticide | Total Number of Laboratories Reporting Data | *Total Number of Reported Detections | GC | Full Scan/AIF GC | LC | Full Scan/AIF LC |
|---|---|---|---|---|---|---|
| Azoxystrobin | 51 | 55 | 7 | 4 | 48 | 14 |
| Benzovindiflupyr | 40 | 42 | 5 | 2 | 37 | 9 |
| Chloridazon | 43 | 47 | 7 | 4 | 40 | 13 |
| Clopyralid | 21 | 22 | 1 | 1 | 21 | 2 |
| Fluazinam | 39 | 40 | 4 | 1 | 36 | 6 |
| Heptachlor | 48 | 51 | 49 | 13 | 2 | 1 |
| Isopyrazam | 45 | 47 | 6 | 2 | 41 | 11 |
| Metamitron | 45 | 48 | 2 | 2 | 46 | 13 |

| Pesticide | Total Number of Laboratories Reporting Data | *Total Number of Reported Detections | GC | Full Scan/AIF GC | LC | Full Scan/AIF LC |
|---|---|---|---|---|---|---|
| Metazachlor | 46 | 49 | 21 | 8 | 28 | 10 |
| Novaluron | 43 | 45 | 3 | 1 | 42 | 11 |
| Oxadiargyl | 38 | 40 | 3 | 1 | 37 | 9 |
| Penflufen | 37 | 39 | 6 | 3 | 33 | 7 |
| Penthiopyrad | 46 | 49 | 9 | 5 | 40 | 9 |
| Quinalphos | 51 | 58 | 32 | 8 | 26 | 10 |
| Quintozene | 45 | 47 | 45 | 12 | 2 | 1 |
| Rotenone | 46 | 47 | 0 | 0 | 47 | 14 |
| Tetramethrin | 51 | 55 | 29 | 9 | 26 | 9 |
| Tolfenpyrad | 41 | 42 | 12 | 3 | 30 | 6 |

*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

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

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

All results reported by the participants are given in Appendix 1. Graphical representations of the results reported are shown in Appendix 2. Details of the methods used are provided in Appendix 3 (available on the EUPT-FV-SM16 webpage, not in the printed version).

Most laboratories analysed the test item using methods based on both gas and liquid chromatography combined with mass spectrometric detection. The number of detections (without the other reported compounds) were 823; 241 were made by GC and 582 by LC. 28% of the detections were made using full-scan or all ion fragmentation (AIF)(79 by GC-full scan/AIF techniques and 155 by LC-full scan/AIF techniques). 33% of the laboratories reported their results using HRMS and 782 of the results were reported indicating a concentration value (95% of the total results).

Fifteen laboratories were able to detect all 18 pesticides present in the test item. Only five laboratories detected less than 50% of the pesticides present. Seventy-nine percent of the laboratories (42 laboratories) that reported results were able to detect more than 70% of the evaluated pesticides.

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

Some pesticides were reported by more than 3 laboratories, acetamiprid (34 laboratories), hexachlorobenzene (5 laboratories), pentachloroaniline (18 laboratories) and tecnazene (4 laboratories). They were present in the sample but below 0.01 mg/kg, therefore they have not been evaluated. Aacetamiprid and pentachloroaniline were present in the bananas, and hexachlorobenzene and tecnazene were impurities of quintozene, one of the spiked pesticides.

Ten participants reported 18 different compounds not evaluated in this proficiency test.

Whether this should be judged as poor performance, or not, depends on how each participant would act on these positive findings in routine analysis. If the reported pesticide was reported as positive with no further identifying confirmation, then the result would be a false positive and hence erroneous monitoring data would be reported. If the reported pesticide is regarded simply as

'suspect' or 'indicatively present', leading to additional analysis to confirm identity before reporting the result, then those pesticides indicated as 'other reported pesticides' in this report are not really an issue.

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

## 6. SUGGESTIONS FOR FUTURE WORK

The Organiser and the Scientific Committee consider that screening methods have provided additional value to the current quantitative multiresidue methods routinely used for monitoring purposes. The results of this test are most encouraging, but also indicate the need for continued evaluation of screening methods. Therefore, further proficiency tests will be organised to provide support to those laboratories using screening methods in order to extend their use and improve their reliability. These methods will be used more and more as screens/filters, to make routine laboratory work easier and faster. The need for screening method validation has been recognised and guidelines for such validation have been prepared and included in Document SANTE/11312/2021v2.

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

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## 8. ACKNOWLEDGEMENTS

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

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

## APPENDIX 1. Results

Table AP1.1. Reported pesticides

| **Laboratory Code** Total No of Reporting Laboratories=52 | **Evaluated Pesticides (14)** **R: Reported Pesticide** | | | | | | | | | | | | | | | | | | **Reported Pesticides by Laboratory** | **% Reported Pesticides by Laboratory** |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Azoxystrobin | Benzovindiflupyr | Chloridazon | Clopyralid | Fluazinam | Heptachlor | Isopyrazam | Metamitron | Metazachlor | Novaluron | Oxadiargyl | Penflufen | Penthiopyrad | Quinalphos | Quintozene | Rotenone | Tetramethrin | Tolfenpyrad | | |
| LAB001 | R | R | R | | R | R | R | R | | R | | | R | R | R | R | R | R | 14 | 78 |
| LAB002 | R | | R | | R | R | R | R | R | R | R | | R | R | R | R | R | R | 15 | 83 |
| LAB003 | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | 18 | 100 |
| LAB004 | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | 18 | 100 |
| 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 | R | R | R | 18 | 100 |
| LAB007 | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | 18 | 100 |
| LAB008 | R | R | R | | | R | R | R | R | R | R | R | R | R | R | R | R | R | 16 | 89 |
| LAB009 | R | | | | R | | | R | | | | | | R | | | | | 4 | 22 |
| 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 | R | 15 | 83 |
| LAB012 | R | R | | | R | R | R | R | R | R | | R | R | R | R | R | R | R | 15 | 83 |
| 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 | R | R | 17 | 94 |
| LAB015 | R | R | R | | R | R | R | R | R | R | R | R | R | R | R | R | R | R | 17 | 94 |
| LAB016 | R | R | R | | R | R | R | R | R | R | R | R | R | R | R | R | R | R | 17 | 94 |
| LAB017 | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | 18 | 100 |
| 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 | R | 17 | 94 |
| LAB021 | R | | R | | | R | | | R | | | | | R | R | | R | | 7 | 39 |
| LAB022 | R | R | R | | | R | R | R | R | R | R | R | R | R | R | R | R | R | 16 | 89 |
| LAB023 | R | | R | | | R | | R | R | R | R | | | R | R | R | R | | 11 | 61 |
| LAB024 | R | R | | | R | R | R | R | R | R | R | R | R | R | R | R | R | R | 16 | 89 |
| LAB025 | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | 18 | 100 |
| LAB026 | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | 18 | 100 |
| LAB027 | R | R | R | | R | R | R | R | R | R | R | R | R | R | R | R | R | R | 17 | 94 |
| LAB028 | | | | | | R | | | | | | | | R | R | | R | | 4 | 22 |
| 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 | R | R | 18 | 100 |
| LAB031 | R | R | R | R | R | R | R | R | R | R | | R | R | R | R | R | R | R | 17 | 94 |
| 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 | R | 17 | 94 |
| LAB034 | R | | R | R | R | R | R | R | R | R | | | R | R | | R | R | R | 14 | 78 |
| LAB035 | R | | R | R | R | R | R | R | R | R | R | | | R | R | | R | | 13 | 72 |
| LAB036 | R | R | | | | R | R | | R | | | R | R | R | | R | R | | 10 | 56 |
| LAB037 | R | R | R | | R | R | R | R | R | R | R | R | R | R | R | R | R | R | 17 | 94 |
| LAB038 | R | R | R | | R | R | R | R | R | R | R | R | R | R | R | R | R | R | 17 | 94 |
| LAB039 | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | 18 | 100 |
| LAB040 | R | R | R | R | R | R | R | R | R | R | | R | R | R | R | R | R | R | 17 | 94 |
| LAB041 | R | R | R | | R | R | R | R | | R | R | | R | R | R | R | R | R | 15 | 83 |
| LAB042 | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | 18 | 100 |
| LAB043 | R | | R | | R | R | R | R | R | R | R | R | R | R | R | R | R | R | 16 | 89 |
| LAB045 | R | R | R | | R | R | R | R | R | R | R | R | R | R | R | R | R | | 16 | 89 |
| LAB046 | R | R | | R | | R | R | | R | R | R | | R | R | | R | R | R | 13 | 72 |
| LAB048 | R | R | | | | R | R | R | R | | R | R | R | R | R | R | R | R | 14 | 78 |
| LAB050 | R | | R | | | R | | R | R | | | | R | R | R | R | R | | 10 | 56 |
| 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 | | 11 | 61 |
| LAB053 | R | | | | R | | R | R | | R | | | R | | | R | R | | 8 | 44 |
| LAB054 | R | | | | | | | | | | | | | R | | | R | | 3 | 17 |
| LAB055 | R | R | R | R | R | R | R | R | R | R | | R | R | R | R | R | R | R | 17 | 94 |
| **Reported Pesticides** | 51 | 40 | 43 | 21 | 39 | 48 | 45 | 45 | 46 | 43 | 38 | 37 | 46 | 51 | 45 | 46 | 51 | 41 | | |
| **% of Reported Pesticides** | 98 | 77 | 83 | 40 | 75 | 92 | 87 | 87 | 88 | 83 | 73 | 71 | 88 | 98 | 87 | 88 | 98 | 79 | | |

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

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

| LABORATORY CODE | Reported Pesticide Concentratons | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Azoxystrobin | Benzovindiflupyr | Chloridazon | Clopyralid | Fluazinam | Heptachlor | Isopyrazam | Metamitron | Metazachlor | Novaluron | Oxadiargyl | Penflufen | Penthiopyrad | Quinalphos | Quintozene | Rotenone | Tetramethrin | Tolfenpyrad |
| **Concentration Homogeneity test (mg/kg)** | 0.037 | 0.037 | 0.162 | 0.08 | 0.064 | 0.045 | 0.078 | 0.098 | 0.09 | 0.036 | 0.036 | 0.05 | 0.03 | 0.116 | 0.038 | 0.072 | 0.108 | 0.046 |
| **Robust mean of estimated concentrations reported (mg/kg)** | 0.038 | 0.037 | 0.156 | 0.067 | 0.062 | 0.045 | 0.084 | 0.1 | 0.088 | 0.034 | 0.037 | 0.05 | 0.031 | 0.106 | 0.038 | 0.071 | 0.11 | 0.051 |
| **CV (%)** | 21.4 | 22.5 | 18.4 | 49.7 | 22.9 | 30 | 21.3 | 24 | 16.6 | 30.8 | 23.7 | 15.7 | 23.5 | 18.8 | 28 | 21.9 | 23.3 | 19 |
| LAB001 | 0.031 | 0.027 | 0.141 | | 0.050 | 0.043 | 0.053 | 0.080 | | 0.025 | | | 0.030 | 0.107 | 0.045 | 0.054 | 0.080 | 0.043 |
| LAB002 | 0.035 | | 0.130 | | 0.068 | 0.051 | 0.080 | 0.070 | 0.083 | 0.022 | 0.049 | | 0.030 | 0.110 | 0.042 | 0.063 | 0.120 | 0.051 |
| LAB003 | 0.046 | 0.050 | | 0.070 | 0.075 | 0.072 | 0.066 | 0.087 | 0.109 | 0.040 | 0.059 | 0.053 | 0.045 | 0.126 | 0.063 | 0.107 | 0.118 | 0.060 |
| LAB004 | 0.032 | 0.038 | 0.146 | 0.076 | 0.050 | 0.047 | 0.084 | 0.110 | 0.088 | 0.026 | 0.037 | 0.054 | 0.031 | 0.300 | 0.058 | 0.080 | 0.120 | 0.051 |
| LAB005 | 0.034 | | 0.106 | | 0.063 | | | | 0.075 | | 0.025 | | 0.026 | 0.097 | | 0.059 | 0.102 | 0.064 |
| LAB006 | 0.033 | 0.040 | 0.133 | 0.071 | 0.062 | 0.037 | 0.079 | 0.097 | 0.080 | 0.034 | 0.036 | 0.043 | 0.026 | 0.098 | 0.035 | 0.057 | 0.100 | 0.051 |
| LAB007 | 0.035 | 0.028 | 0.160 | 0.053 | 0.048 | 0.068 | 0.072 | 0.096 | 0.082 | 0.025 | 0.028 | 0.044 | 0.025 | 0.110 | 0.025 | 0.053 | 0.075 | 0.043 |
| LAB008 | 0.042 | 0.036 | 0.177 | | | 0.027 | 0.103 | 0.107 | 0.121 | 0.041 | 0.038 | 0.050 | 0.032 | 0.093 | 0.030 | 0.077 | 0.101 | 0.049 |
| | | | | | | 0.044 | | | | | | | | 0.096 | 0.032 | | | |
| | | | | | | | | | | | | | | 0.097 | | | | |
| | | | | | | | | | | | | | | 0.163 | | | | |
| LAB009 | | | | | | | | | | | | | | | | | | |
| LAB010 | 0.034 | 0.042 | 0.170 | 0.189 | 0.077 | 0.045 | 0.080 | 0.104 | 0.090 | 0.090 | 0.029 | 0.050 | 0.031 | 0.083 | 0.036 | 0.065 | 0.126 | 0.051 |
| | | | 0.171 | | | | 0.106 | 0.116 | 0.093 | 0.093 | 0.034 | 0.059 | 0.031 | 0.101 | | | 0.135 | |
| LAB011 | 0.037 | 0.039 | | | | 0.036 | 0.093 | | 0.082 | 0.035 | 0.042 | 0.054 | 0.034 | 0.110 | 0.024 | 0.066 | 0.110 | 0.055 |
| LAB012 | 0.021 | 0.021 | | | 0.043 | 0.028 | 0.050 | 0.078 | 0.060 | 0.021 | | 0.034 | 0.018 | 0.084 | 0.029 | 0.051 | 0.082 | 0.034 |
| LAB013 | 0.029 | 0.032 | 0.135 | 0.054 | 0.055 | 0.037 | 0.070 | 0.085 | 0.094 | 0.025 | 0.031 | 0.040 | 0.024 | 0.094 | 0.033 | 0.056 | 0.092 | 0.039 |
| LAB014 | | | | | | | | | | | | | | | | | | |
| LAB015 | 0.043 | 0.042 | 0.147 | | 0.069 | 0.037 | 0.087 | 0.079 | 0.088 | 0.034 | 0.037 | 0.052 | 0.028 | 0.123 | 0.027 | 0.083 | 0.107 | 0.025 |
| | | | | | | 0.043 | | | | | | | 0.028 | | 0.030 | | | |
| LAB016 | 0.033 | 0.036 | 0.158 | | 0.058 | 0.039 | 0.076 | 0.109 | 0.086 | 0.028 | | 0.046 | 0.028 | 0.097 | 0.035 | 0.059 | 0.118 | 0.035 |
| LAB017 | 0.040 | 0.037 | 0.138 | 0.053 | 0.066 | 0.053 | 0.092 | 0.085 | 0.087 | 0.038 | 0.038 | 0.051 | 0.035 | 0.123 | 0.047 | 0.078 | 0.109 | 0.057 |

## APPENDIX 1. Results

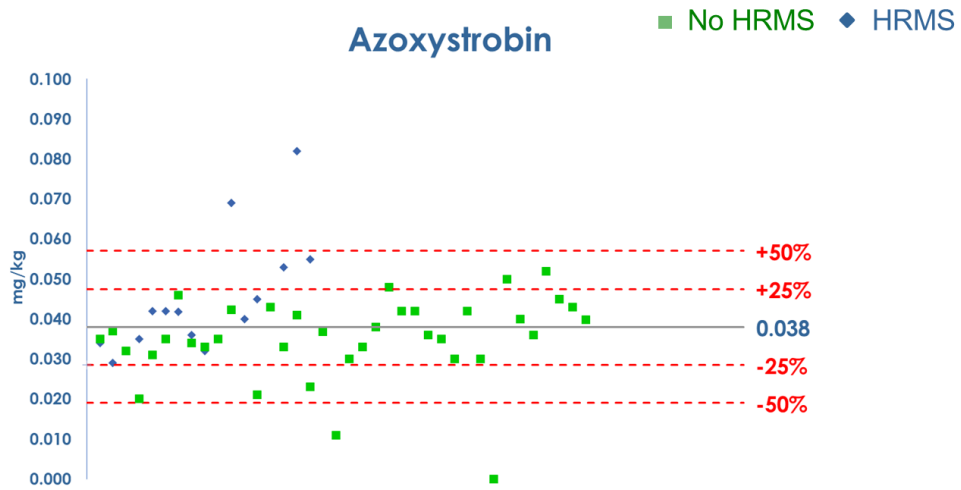
| LABORATORY CODE | Reported Pesticide Concentratons | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Azoxystrobin | Benzovindiflupyr | Chloridazon | Clopyralid | Fluazinam | Heptachlor | Isopyrazam | Metamitron | Metazachlor | Novaluron | Oxadiargyl | Penflufen | Penthiopyrad | Quinalphos | Quintozene | Rotenone | Tetramethrin | Tolfenpyrad |
| **Concentration Homogeneity test (mg/kg)** | 0.037 | 0.037 | 0.162 | 0.08 | 0.064 | 0.045 | 0.078 | 0.098 | 0.09 | 0.036 | 0.036 | 0.05 | 0.03 | 0.116 | 0.038 | 0.072 | 0.108 | 0.046 |
| **Robust mean of estimated concentrations reported (mg/kg)** | 0.038 | 0.037 | 0.156 | 0.067 | 0.062 | 0.045 | 0.084 | 0.1 | 0.088 | 0.034 | 0.037 | 0.05 | 0.031 | 0.106 | 0.038 | 0.071 | 0.11 | 0.051 |
| **CV (%)** | 21.4 | 22.5 | 18.4 | 49.7 | 22.9 | 30 | 21.3 | 24 | 16.6 | 30.8 | 23.7 | 15.7 | 23.5 | 18.8 | 28 | 21.9 | 23.3 | 19 |
| LAB018 | 0.041 | 0.043 | 0.183 | 0.133 | 0.061 | 0.046 | 0.093 | 0.145 | 0.094 | 0.050 | 0.041 | 0.060 | 0.036 | 0.096 | 0.033 | 0.067 | 0.080 | 0.056 |
| LAB019 | 0.023 | 0.018 | 0.143 | 0.033 | 0.042 | 0.027 | 0.065 | 0.091 | 0.083 | 0.024 | 0.031 | 0.044 | 0.021 | 0.078 | 0.020 | 0.052 | 0.046 | 0.030 |
| LAB020 | 0.037 | 0.027 | 0.169 | | 0.058 | 0.040 | 0.069 | 0.094 | 0.105 | 0.034 | 0.040 | 0.054 | 0.009 | 0.090 | 0.035 | 0.073 | 0.083 | 0.056 |
| LAB021 | 0.011 | | 0.075 | | | 0.003 | | | 0.064 | | | | | 0.027 | 0.007 | | 0.013 | |
| LAB022 | 0.069 | 0.061 | 0.198 | | | 0.080 | 0.120 | 0.120 | 0.017 | 0.042 | 0.062 | 0.075 | 0.056 | 0.165 | 0.080 | 0.097 | 0.146 | 0.076 |
| LAB023 | 0.040 | | | | | 0.054 | | | | | | | | | 0.039 | | | |
| LAB024 | 0.032 | 0.033 | | | 0.091 | 0.036 | 0.135 | 0.163 | 0.092 | 0.054 | 0.052 | 0.047 | 0.033 | 0.079 | 0.046 | 0.085 | 0.120 | 0.208 |
| LAB025 | 0.030 | 0.040 | 0.181 | 0.067 | 0.067 | 0.049 | 0.081 | 0.107 | 0.083 | 0.035 | 0.033 | 0.049 | 0.027 | 0.112 | 0.036 | 0.068 | 0.102 | 0.048 |
| LAB026 | 0.033 | 0.036 | 0.154 | 0.096 | 0.063 | 0.061 | 0.050 | 0.085 | 0.091 | 0.027 | 0.028 | 0.053 | 0.027 | 0.113 | 0.049 | 0.071 | 0.108 | 0.038 |
| LAB027 | 0.038 | 0.042 | 0.130 | | 0.050 | 0.050 | 0.095 | 0.084 | 0.087 | 0.036 | 0.030 | 0.048 | 0.028 | 0.120 | 0.040 | 0.065 | 0.100 | 0.040 |
| | | | | | 0.050 | | | | | | | | | | | | | |
| LAB028 | | | | | | 0.192 | | | | | | | | 0.173 | 0.034 | | 0.152 | |
| LAB029 | 0.048 | 0.029 | 0.196 | 0.101 | 0.079 | 0.038 | 0.090 | 0.102 | 0.103 | 0.042 | 0.041 | 0.049 | 0.034 | 0.096 | 0.033 | 0.068 | 0.095 | 0.045 |
| LAB030 | 0.035 | 0.037 | 0.12 | 0.024 | 0.077 | 0.052 | 0.084 | 0.098 | 0.082 | 0.037 | 0.041 | 0.040 | 0.025 | 0.105 | 0.043 | 0.079 | 0.108 | 0.048 |
| | 0.045 | 0.045 | 0.26 | 0.061 | | | | | | | | | | | | | | |
| LAB031 | 0.042 | 0.057 | 0.180 | 0.044 | 0.073 | 0.054 | 0.092 | 0.079 | 0.090 | 0.037 | | 0.054 | 0.036 | 0.129 | 0.037 | 0.073 | 0.118 | 0.065 |
| LAB032 | 0.042 | 0.042 | 0.150 | | | 0.044 | 0.090 | 0.086 | 0.089 | 0.028 | 0.041 | 0.049 | 0.035 | 0.122 | 0.042 | 0.073 | 0.120 | 0.060 |
| LAB033 | 0.036 | 0.038 | 0.160 | | 0.057 | 0.030 | 0.078 | 0.096 | 0.091 | 0.023 | 0.028 | 0.057 | 0.035 | 0.100 | 0.029 | 0.057 | 0.120 | 0.030 |
| LAB034 | 0.035 | | 0.100 | 0.031 | 0.075 | 0.055 | 0.096 | 0.069 | 0.060 | 0.033 | | | 0.022 | 0.090 | | 0.048 | 0.079 | 0.055 |
| LAB035 | 0.035 | | 0.157 | 0.038 | 0.062 | 0.051 | 0.084 | 0.083 | 0.093 | 0.033 | 0.033 | | | 0.105 | 0.053 | | 0.112 | |
| LAB036 | 0.030 | 0.028 | | | | 0.058 | 0.058 | | 0.088 | | | 0.037 | 0.024 | 0.108 | | 0.044 | 0.115 | |
| LAB037 | 0.042 | 0.040 | | | 0.094 | 0.044 | 0.095 | 0.079 | | 0.037 | 0.045 | 0.053 | 0.032 | 0.130 | 0.041 | 0.082 | 0.110 | 0.061 |
| | 0.042 | 0.042 | | | | | 0.110 | 0.082 | | 0.037 | 0.046 | 0.054 | 0.034 | 0.130 | | 0.083 | 0.120 | 0.064 |
| LAB038 | 0.030 | 0.037 | 0.139 | | 0.051 | 0.035 | 0.056 | 0.130 | 0.094 | 0.017 | 0.016 | 0.040 | 0.027 | 0.095 | 0.033 | 0.048 | 0.037 | 0.057 |
| LAB039 | 0.042 | 0.041 | 0.160 | 0.080 | 0.073 | 0.054 | 0.091 | 0.100 | 0.114 | 0.040 | | 0.051 | 0.033 | 0.124 | 0.056 | 0.072 | 0.205 | |
| LAB040 | 0.029 | 0.027 | 0.137 | 0.114 | 0.045 | 0.046 | 0.059 | 0.070 | 0.068 | 0.014 | | 0.060 | 0.028 | 0.103 | 0.037 | 0.082 | 0.104 | 0.046 |

| LABORATORY CODE | Reported Pesticide Concentratons | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Azoxystrobin | Benzovindiflupyr | Chloridazon | Clopyralid | Fluazinam | Heptachlor | Isopyrazam | Metamitron | Metazachlor | Novaluron | Oxadiargyl | Penflufen | Penthiopyrad | Quinalphos | Quintozene | Rotenone | Tetramethrin | Tolfenpyrad |
| **Concentration Homogeneity test (mg/kg)** | 0.037 | 0.037 | 0.162 | 0.08 | 0.064 | 0.045 | 0.078 | 0.098 | 0.09 | 0.036 | 0.036 | 0.05 | 0.03 | 0.116 | 0.038 | 0.072 | 0.108 | 0.046 |
| **Robust mean of estimated concentrations reported (mg/kg)** | 0.038 | 0.037 | 0.156 | 0.067 | 0.062 | 0.045 | 0.084 | 0.1 | 0.088 | 0.034 | 0.037 | 0.05 | 0.031 | 0.106 | 0.038 | 0.071 | 0.11 | 0.051 |
| **CV (%)** | 21.4 | 22.5 | 18.4 | 49.7 | 22.9 | 30 | 21.3 | 24 | 16.6 | 30.8 | 23.7 | 15.7 | 23.5 | 18.8 | 28 | 21.9 | 23.3 | 19 |
| | | | 0.156 | | | | | | 0.085 | | | | | 0.11 | | | 0.108 | |
| LAB041 | 0.050 | 0.038 | 0.170 | | 0.056 | 0.037 | 0.090 | 0.120 | | 0.033 | 0.038 | | 0.023 | 0.077 | 0.032 | 0.065 | 0.119 | 0.042 |
| LAB042 | 0.040 | 0.045 | 0.134 | 0.092 | 0.059 | 0.046 | 0.086 | 0.104 | 0.083 | 0.036 | 0.039 | 0.051 | 0.028 | 0.114 | 0.038 | 0.130 | 0.115 | 0.059 |
| LAB043 | 0.020 | | 0.230 | | 0.077 | 0.023 | 0.130 | 0.150 | 0.140 | 0.050 | 0.047 | 0.069 | 0.047 | 0.098 | 0.026 | 0.097 | 0.110 | 0.073 |
| LAB045 | 0.036 | 0.049 | 0.136 | | 0.071 | 0.046 | 0.094 | 0.129 | 0.083 | 0.034 | 0.036 | 0.068 | 0.041 | 0.127 | 0.042 | 0.084 | 0.136 | |
| LAB046 | 0.052 | 0.033 | | 0.025 | | 0.019 | 0.079 | | 0.051 | 0.052 | 0.028 | | 0.052 | 0.080 | | 0.070 | 0.075 | 0.050 |
| LAB048 | | 0.025 | | | | 0.075 | 0.075 | 0.005 | 0.050 | | 0.015 | 0.015 | 0.070 | 0.110 | 0.040 | 0.070 | 0.015 | 0.050 |
| LAB050 | 0.036 | | 0.462 | | | 0.092 | | 0.321 | 0.098 | | | | 0.030 | 0.126 | 0.064 | 0.080 | 0.173 | |
| LAB051 | 0.053 | 0.039 | 0.160 | | 0.016 | 0.043 | 0.078 | 0.110 | 0.100 | 0.049 | 0.043 | | 0.036 | 0.110 | 0.050 | 0.070 | 0.200 | 0.050 |
| LAB052 | 0.055 | 0.054 | 0.989 | | | 0.140 | 0.084 | 0.271 | 0.108 | | | | 0.046 | 0.073 | 0.119 | | 0.121 | |
| | 0.082 | | | | | | | 0.313 | 0.206 | | | | | 0.080 | | | 0.163 | |
| LAB053 | 0.042 | | | | 0.027 | | 0.145 | 0.115 | | 0.017 | | | 0.036 | | | 0.125 | 0.193 | |
| LAB054 | 0.045 | | | | | | | | | | | | | | | | 0.111 | |
| LAB055 | 0.043 | 0.036 | 0.160 | 0.062 | 0.073 | 0.058 | 0.078 | 0.090 | 0.090 | 0.045 | | 0.053 | 0.046 | 0.114 | 0.039 | 0.085 | 0.120 | 0.065 |

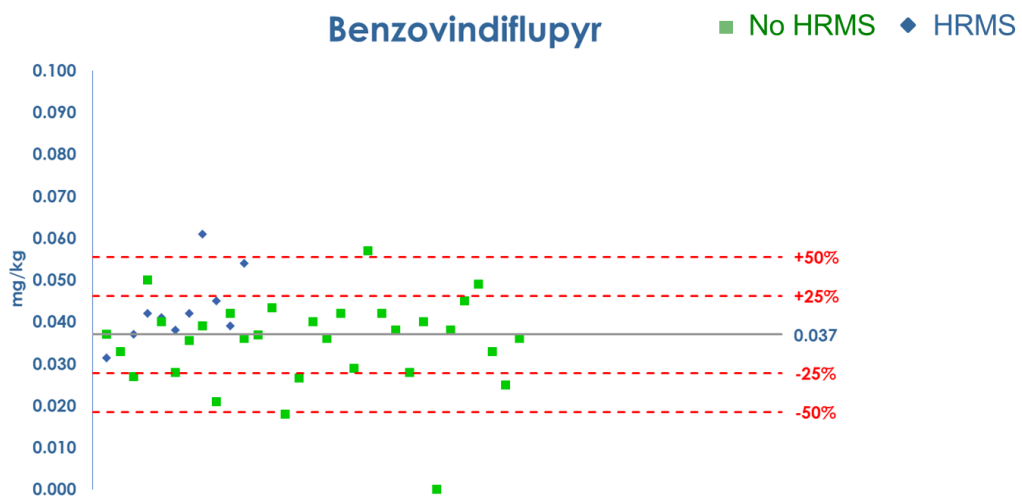
## APPENDIX 2. Graphical Representations

The grey line represents the robust mean

| Pesticides | No of Detections | N° of Labs detected the pesticide | % of Labs detected the pesticide | No of Conc. Reported | No of Conc. Reported by EU/EFTA labs | Conc. Homogeneity test (mg/kg) | Robust mean (mg/kg) | CV (%) |
|---|---|---|---|---|---|---|---|---|
| Azoxystrobin | 22 | 21 | 40 | 51 | 49 | 0.037 | 0.038 | 21.4 |

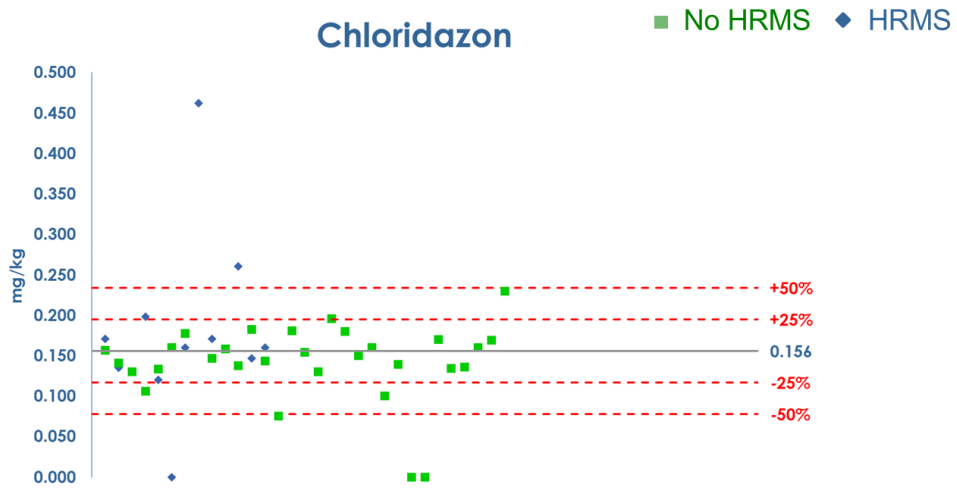



| Pesticides | No of Detections | N° of Labs detected the pesticide | % of Labs detected the pesticide | No of Conc. Reported | No of Conc. Reported by EU/EFTA labs | Conc. Homogeneity test (mg/kg) | Robust mean (mg/kg) | CV (%) |
|---|---|---|---|---|---|---|---|---|
| Benzovindiflupyr | 35 | 34 | 65 | 41 | 39 | 0.037 | 0.037 | 22.5 |

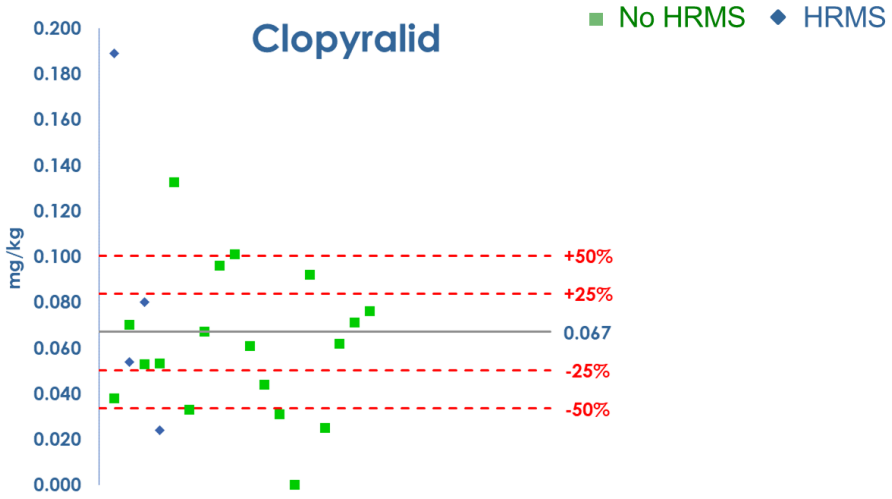


The grey line represents the robust mean

| Pesticides | No of Detections | N° of Labs detected the pesticide | % of Labs detected the pesticide | No of Conc. Reported | No of Conc. Reported by EU/EFTA labs | Conc. Homogeneity test (mg/kg) | Robust mean (mg/kg) | CV (%) |
|---|---|---|---|---|---|---|---|---|
| Chloridazon | 39 | 37 | 71 | 41 | 40 | 0.162 | 0.156 | 18.4 |

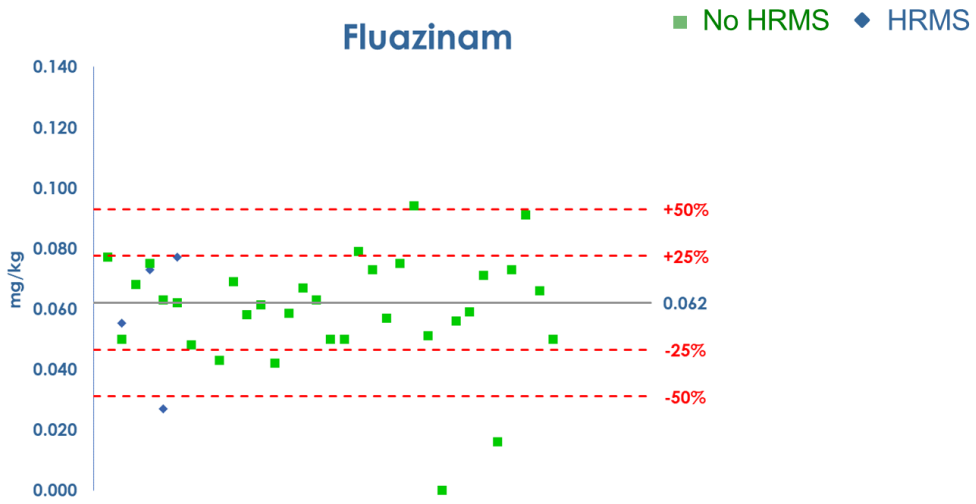



| Pesticides | No of Detections | N° of Labs detected the pesticide | % of Labs detected the pesticide | No of Conc. Reported | No of Conc. Reported by EU/EFTA labs | Conc. Homogeneity test (mg/kg) | Robust mean (mg/kg) | CV (%) |
|---|---|---|---|---|---|---|---|---|
| Clopyralid | 40 | 39 | 75 | 22 | 21 | 0.080 | 0.067 | 49.7 |

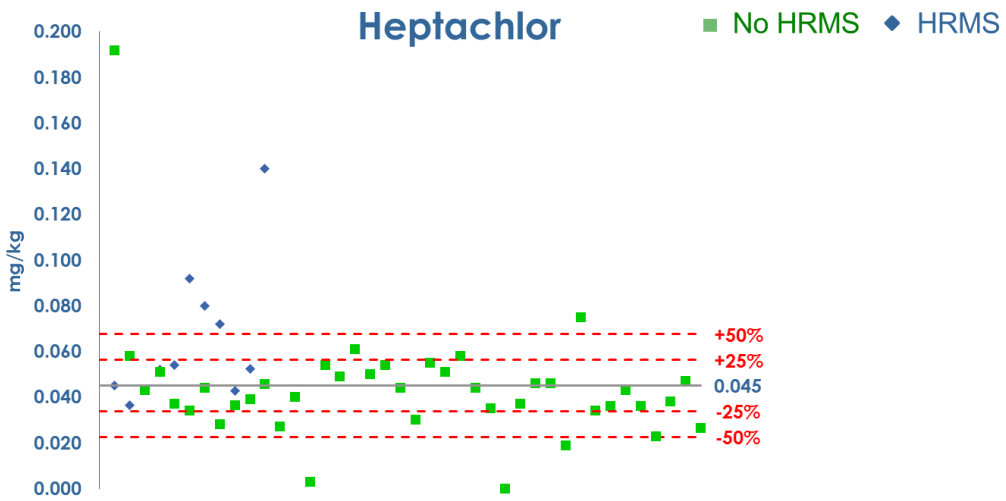


The grey line represents the robust mean

| Pesticides | No of Detections | N° of Labs detected the pesticide | % of Labs detected the pesticide | No of Conc. Reported | No of Conc. Reported by EU/EFTA labs | Conc. Homogeneity test (mg/kg) | Robust mean (mg/kg) | CV (%) |
|---|---|---|---|---|---|---|---|---|
| Fluazinam | 40 | 38 | 73 | 38 | 38 | 0.064 | 0.062 | 22.9 |



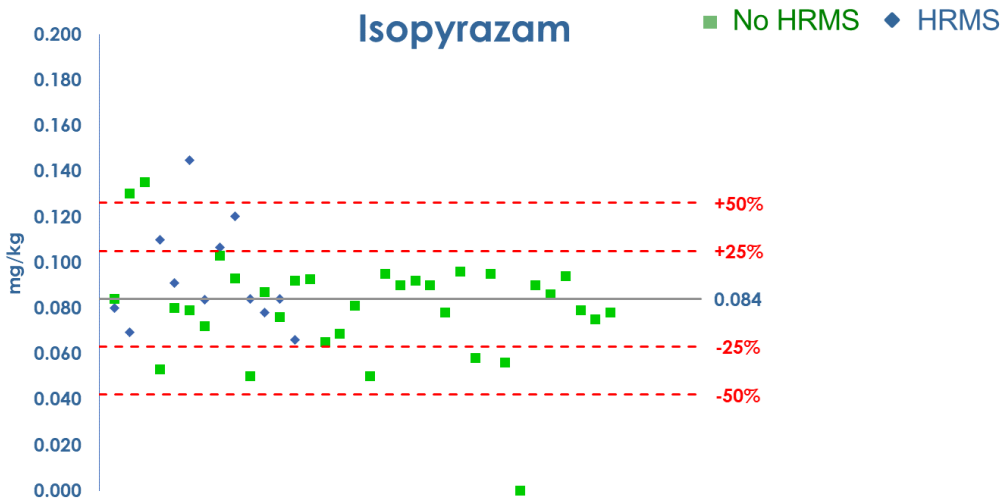

| Pesticides | No of Detections | N° of Labs detected the pesticide | % of Labs detected the pesticide | No of Conc. Reported | No of Conc. Reported by EU/EFTA labs | Conc. Homogeneity test (mg/kg) | Robust mean (mg/kg) | CV (%) |
|---|---|---|---|---|---|---|---|---|
| Heptachlor | 42 | 40 | 77 | 50 | 48 | 0.045 | 0.045 | 30.0 |



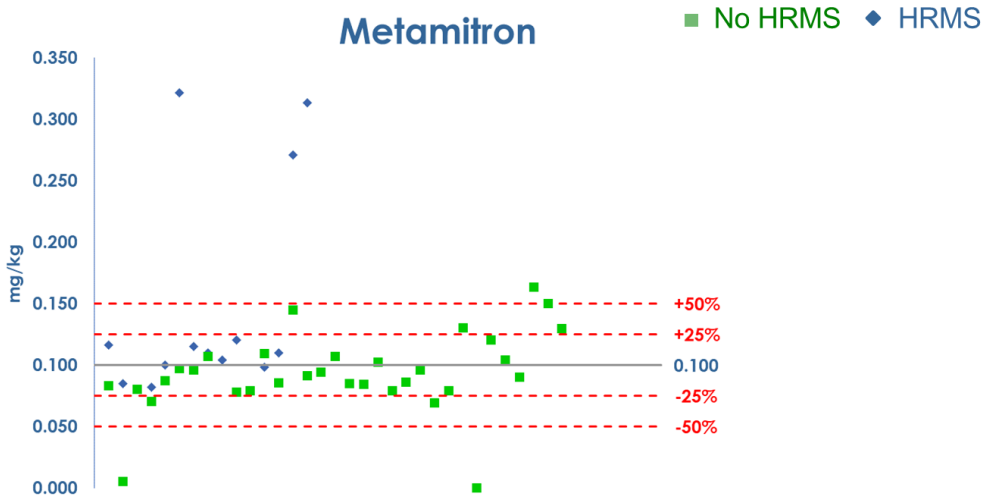
## APPENDIX 2. Graphical Representations

The grey line represents the robust mean

| Pesticides | No of Detections | N° of Labs detected the pesticide | % of Labs detected the pesticide | No of Conc. Reported | No of Conc. Reported by EU/EFTA labs | Conc. Homogeneity test (mg/kg) | Robust mean (mg/kg) | CV (%) |
|---|---|---|---|---|---|---|---|---|
| Isopyrazam | 42 | 41 | 79 | 46 | 44 | 0.078 | 0.084 | 21.3 |



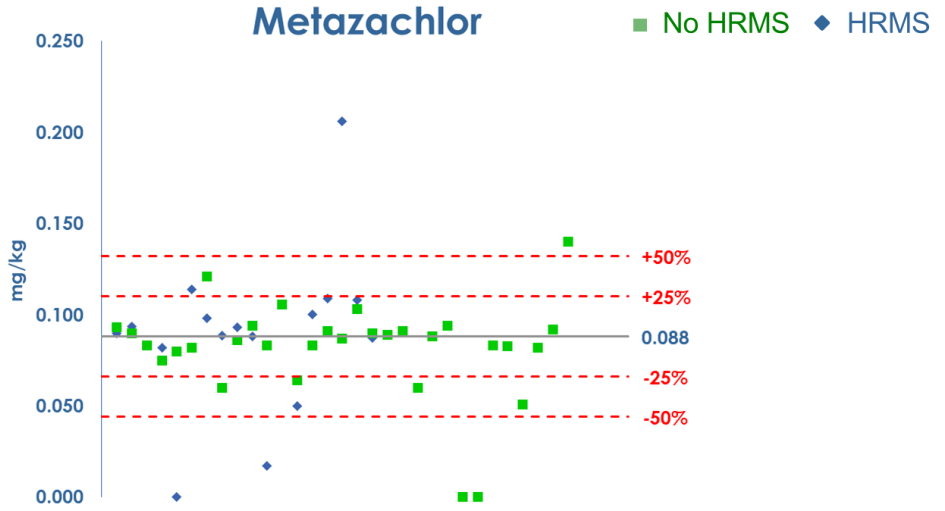

| Pesticides | No of Detections | N° of Labs detected the pesticide | % of Labs detected the pesticide | No of Conc. Reported | No of Conc. Reported by EU/EFTA labs | Conc. Homogeneity test (mg/kg) | Robust mean (mg/kg) | CV (%) |
|---|---|---|---|---|---|---|---|---|
| Metamitron | 45 | 43 | 83 | 45 | 44 | 0.098 | 0.100 | 24.0 |



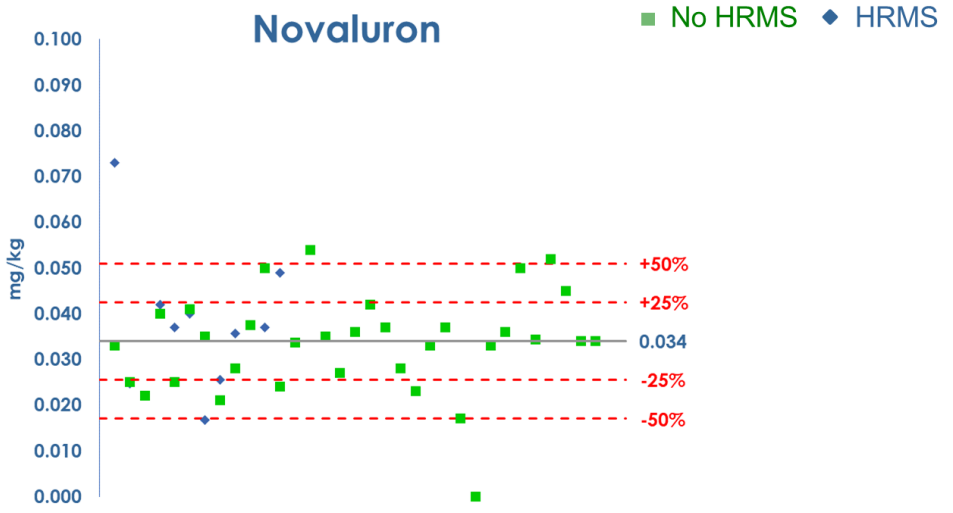

11

The grey line represents the robust mean

| Pesticides | No of Detections | N° of Labs detected the pesticide | % of Labs detected the pesticide | No of Conc. Reported | No of Conc. Reported by EU/EFTA labs | Conc. Homogeneity test (mg/kg) | Robust mean (mg/kg) | CV (%) |
|---|---|---|---|---|---|---|---|---|
| Metazachlor | 47 | 43 | 83 | 46 | 44 | 0.090 | 0.088 | 16.6 |



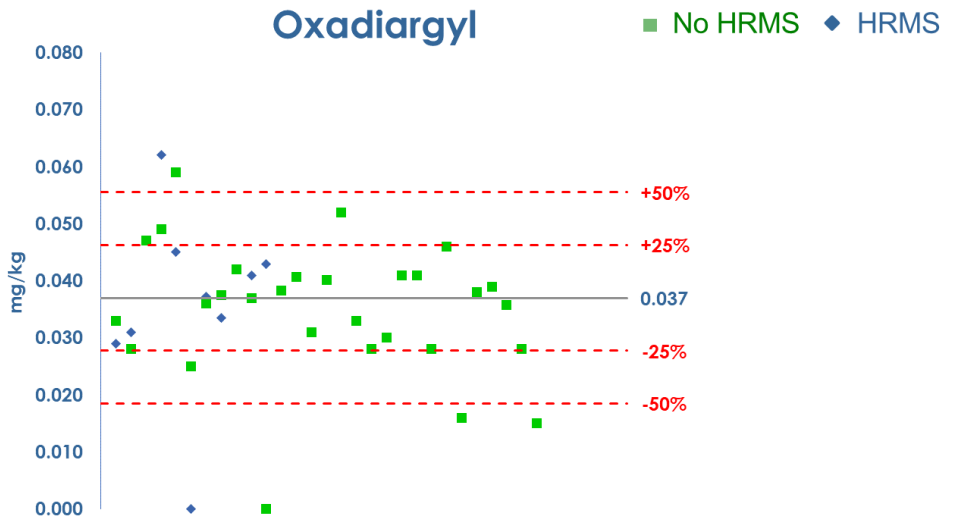

| Pesticides | No of Detections | N° of Labs detected the pesticide | % of Labs detected the pesticide | No of Conc. Reported | No of Conc. Reported by EU/EFTA labs | Conc. Homogeneity test (mg/kg) | Robust mean (mg/kg) | CV (%) |
|---|---|---|---|---|---|---|---|---|
| Novaluron | 47 | 45 | 87 | 43 | 41 | 0.036 | 0.034 | 30.8 |



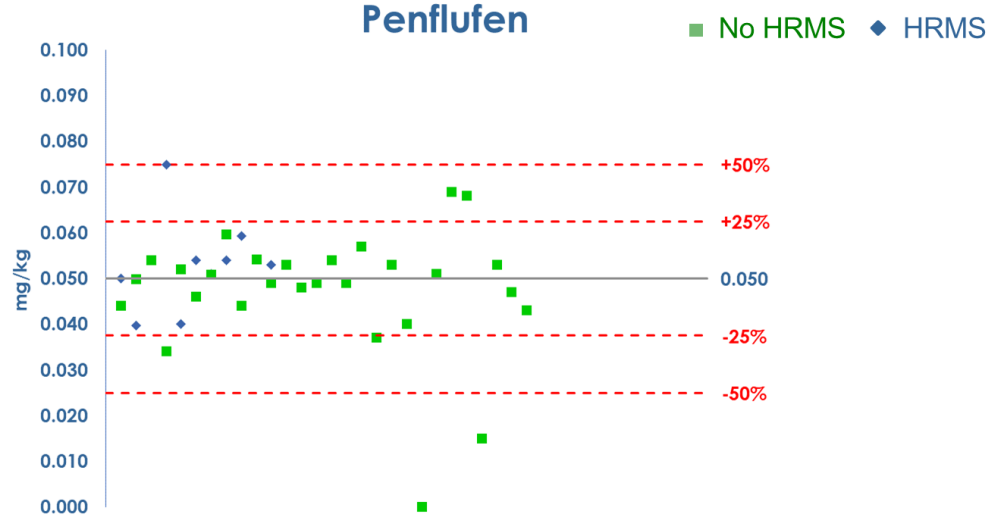
## APPENDIX 2. Graphical Representations

The grey line represents the robust mean

| Pesticides | No of Detections | N° of Labs detected the pesticide | % of Labs detected the pesticide | No of Conc. Reported | No of Conc. Reported by EU/EFTA labs | Conc. Homogeneity test (mg/kg) | Robust mean (mg/kg) | CV (%) |
|---|---|---|---|---|---|---|---|---|
| Oxadiargyl | 47 | 45 | 87 | 36 | 34 | 0.036 | 0.037 | 23.7 |

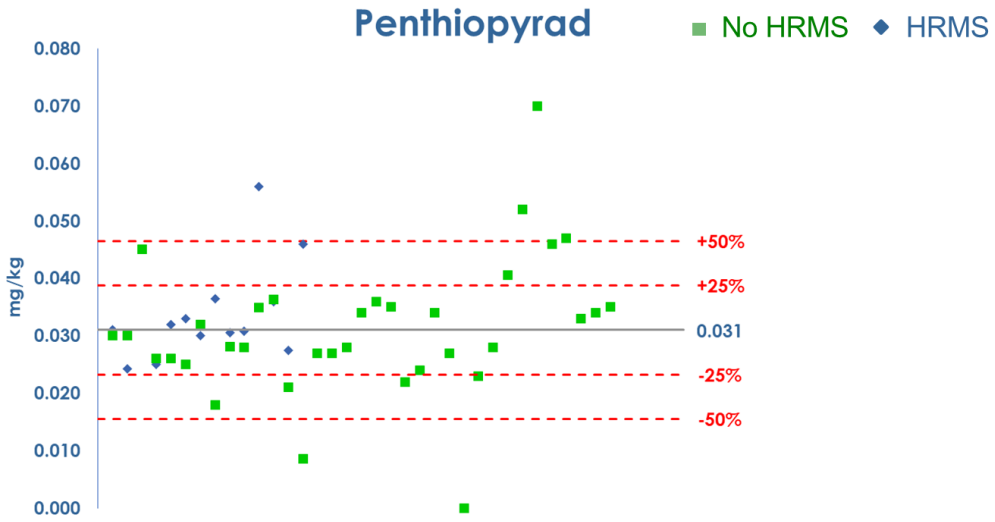



| Pesticides | No of Detections | N° of Labs detected the pesticide | % of Labs detected the pesticide | No of Conc. Reported | No of Conc. Reported by EU/EFTA labs | Conc. Homogeneity test (mg/kg) | Robust mean (mg/kg) | CV (%) |
|---|---|---|---|---|---|---|---|---|
| Penflufen | 47 | 46 | 88 | 37 | 36 | 0.050 | 0.050 | 15.7 |

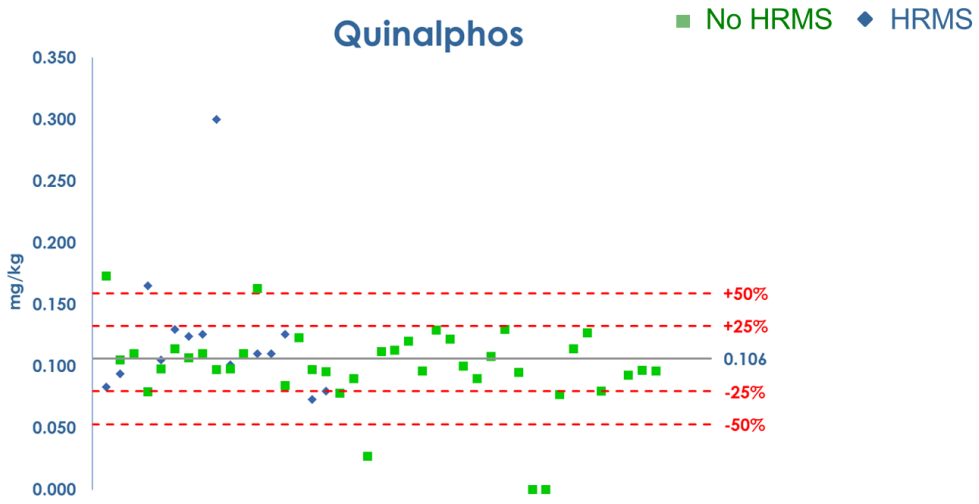


The grey line represents the robust mean

| Pesticides | No of Detections | N° of Labs detected the pesticide | % of Labs detected the pesticide | No of Conc. Reported | No of Conc. Reported by EU/EFTA labs | Conc. Homogeneity test (mg/kg) | Robust mean (mg/kg) | CV (%) |
|---|---|---|---|---|---|---|---|---|
| Penthiopyrad | 49 | 46 | 88 | 48 | 46 | 0.030 | 0.031 | 23.5 |

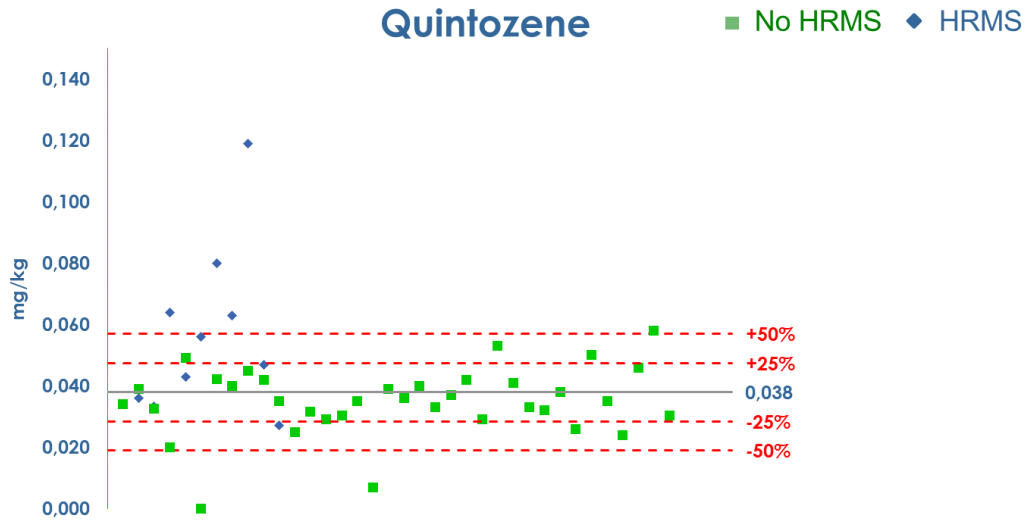



| Pesticides | No of Detections | N° of Labs detected the pesticide | % of Labs detected the pesticide | No of Conc. Reported | No of Conc. Reported by EU/EFTA labs | Conc. Homogeneity test (mg/kg) | Robust mean (mg/kg) | CV (%) |
|---|---|---|---|---|---|---|---|---|
| Quinalphos | 49 | 46 | 88 | 54 | 52 | 0.116 | 0.106 | 18.8 |

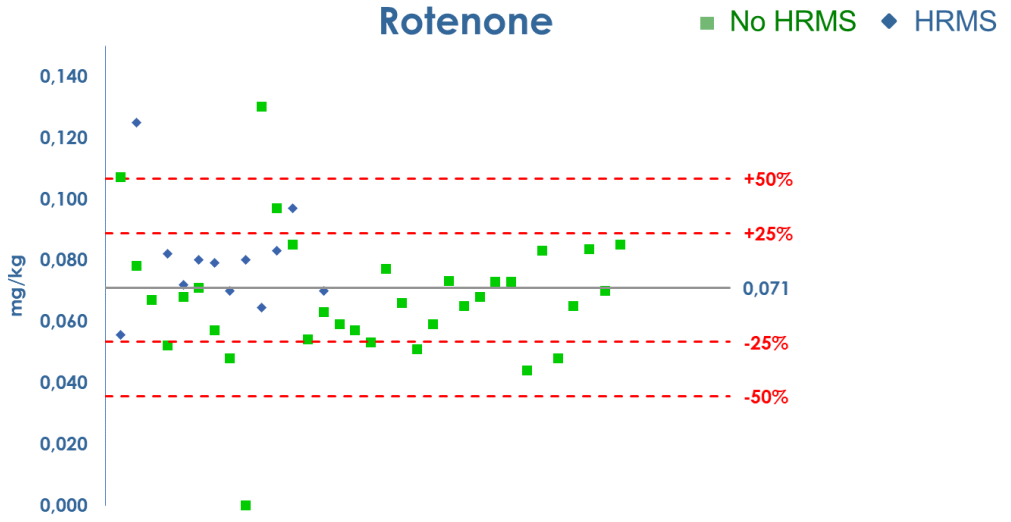


The grey line represents the robust mean

| Pesticides | No of Detections | N° of Labs detected the pesticide | % of Labs detected the pesticide | No of Conc. Reported | No of Conc. Reported by EU/EFTA labs | Conc. Homogeneity test (mg/kg) | Robust mean (mg/kg) | CV (%) |
|---|---|---|---|---|---|---|---|---|
| Quintozene | 51 | 48 | 92 | 46 | 45 | 0.038 | 0.038 | 28.0 |

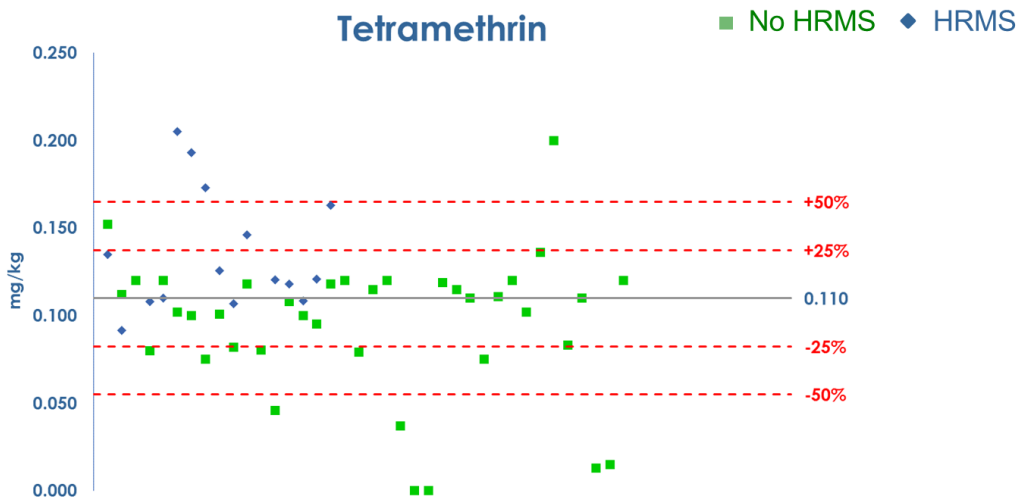



| Pesticides | No of Detections | N° of Labs detected the pesticide | % of Labs detected the pesticide | No of Conc. Reported | No of Conc. Reported by EU/EFTA labs | Conc. Homogeneity test (mg/kg) | Robust mean (mg/kg) | CV (%) |
|---|---|---|---|---|---|---|---|---|
| Rotenone | 55 | 51 | 98 | 45 | 43 | 0.072 | 0.071 | 21.9 |

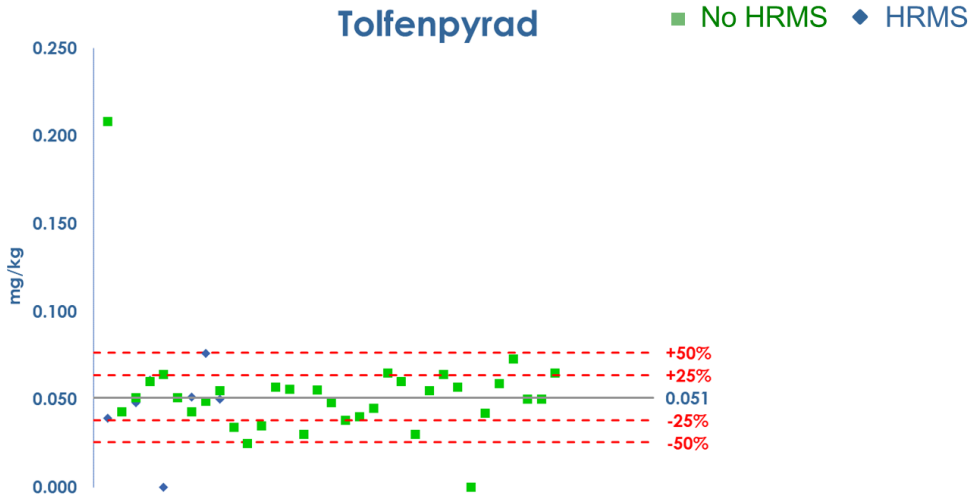


The grey line represents the robust mean

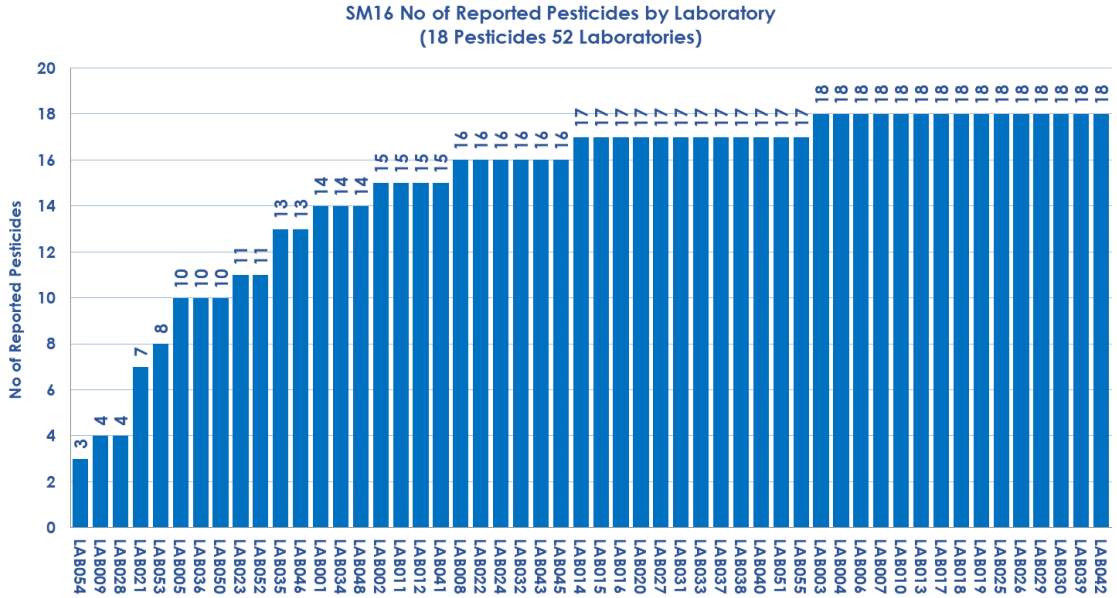
| Pesticides | No of Detections | N° of Labs detected the pesticide | % of Labs detected the pesticide | No of Conc. Reported | No of Conc. Reported by EU/EFTA labs | Conc. Homogeneity test (mg/kg) | Robust mean (mg/kg) | CV (%) |
|---|---|---|---|---|---|---|---|---|
| Tetramethrin | 55 | 51 | 98 | 53 | 51 | 0.108 | 0.110 | 23.3 |

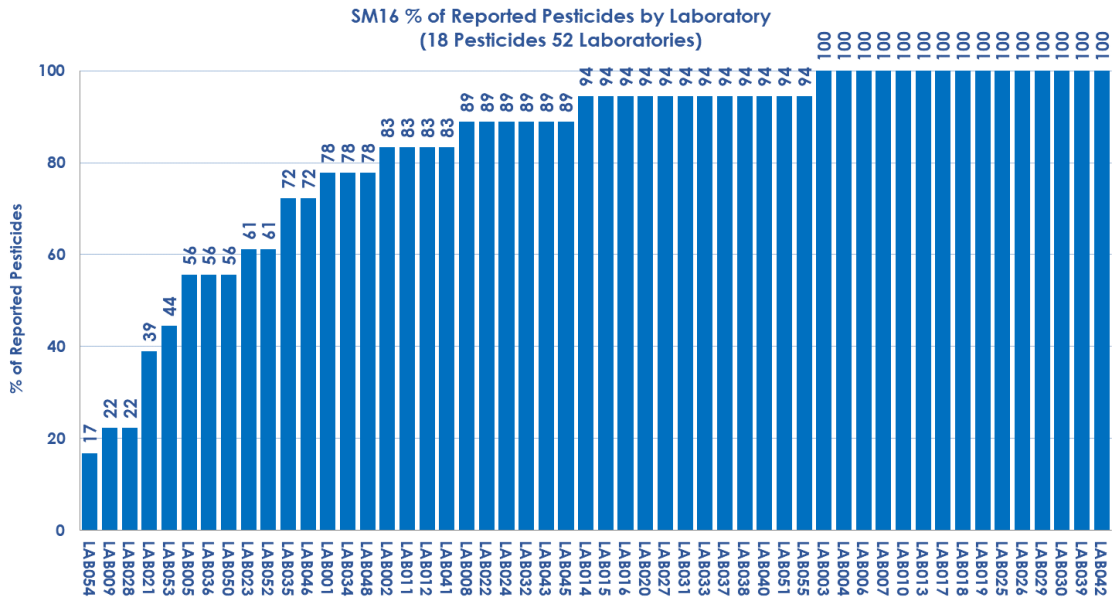


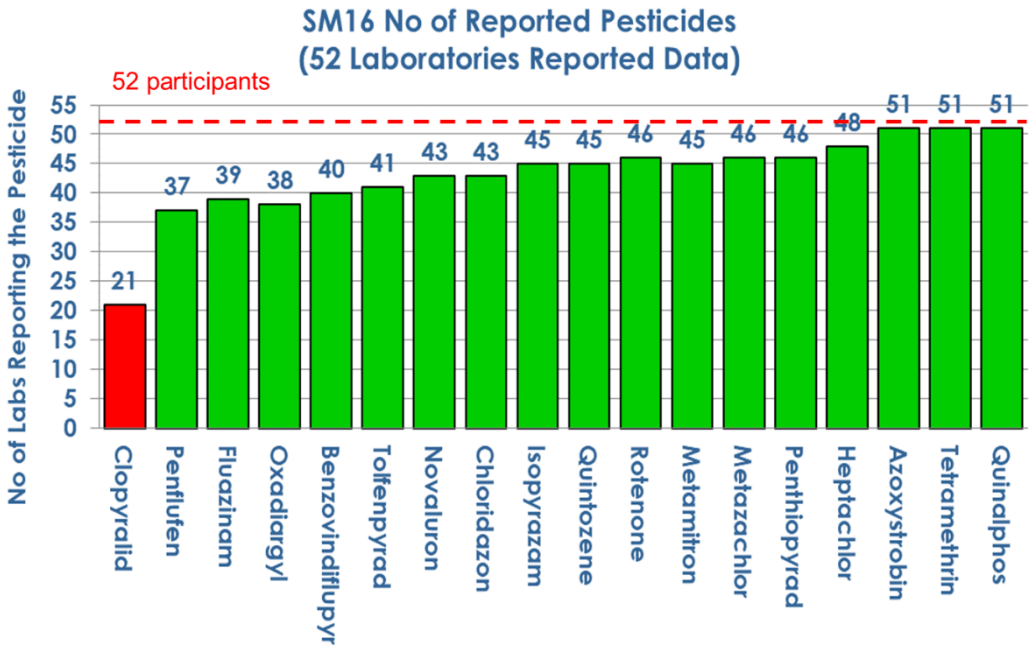

| Pesticides | No of Detections | N° of Labs detected the pesticide | % of Labs detected the pesticide | No of Conc. Reported | No of Conc. Reported by EU/EFTA labs | Conc. Homogeneity test (mg/kg) | Robust mean (mg/kg) | CV (%) |
|---|---|---|---|---|---|---|---|---|
| Tolfenpyrad | 58 | 51 | 98 | 40 | 38 | 0.046 | 0.051 | 19.0 |

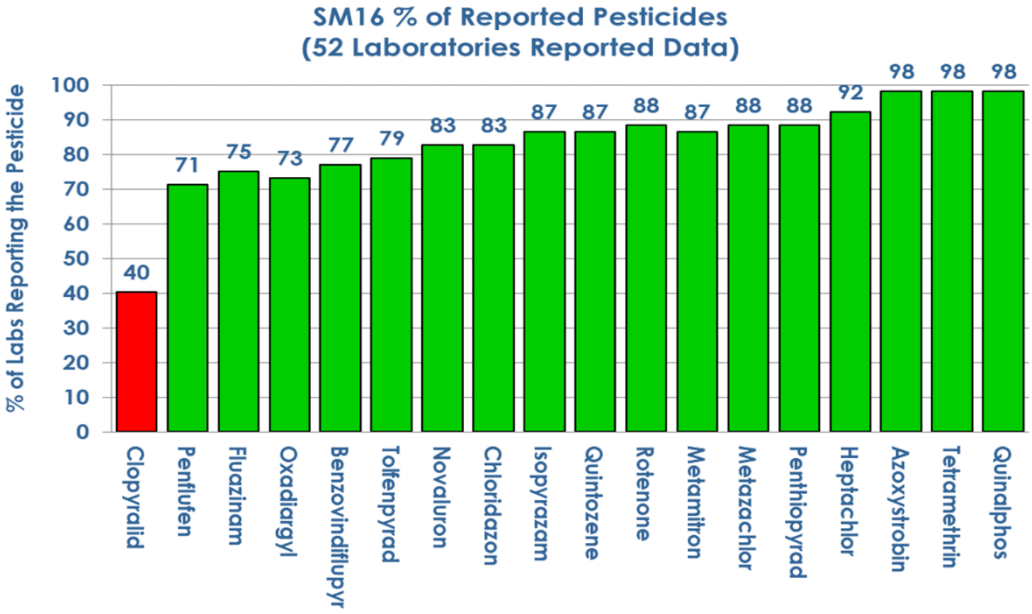


## APPENDIX 2. Graphical Representations



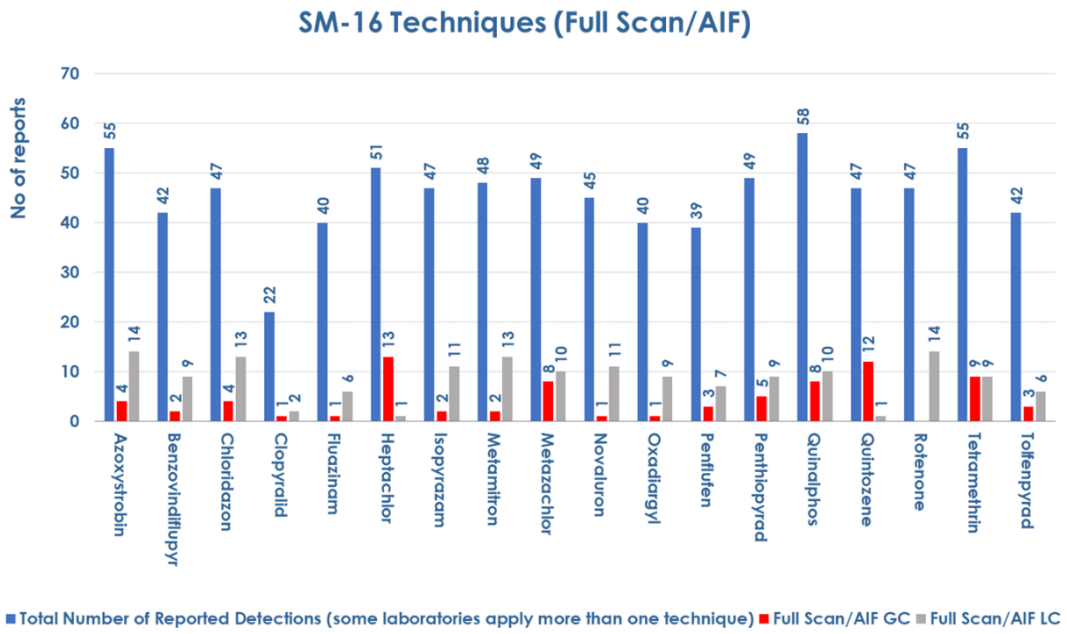




SM16 No of Reported Pesticides
(52 Laboratories Reported Data)
52 participants
No of Labs Reporting the Pesticide
0
5
10
15
20
25
30
35
40
45
50
55
21
37
39
38
40
41
43
43
45
45
46
45
46
46
48
51
51
51
Clopyralid
Penflufen
Fluazinam
Oxadiargyl
Benzovindiflupyr
Tolfenpyrad
Novaluron
Chloridazon
Isopyrazam
Quintozene
Rotenone
Metamitron
Metazachlor
Penthiopyrad
Heptachlor
Azoxystrobin
Tetramethrin
Quinalphos


SM16 % of Reported Pesticides
(52 Laboratories Reported Data)
% of Labs Reporting the Pesticide
0
10
20
30
40
50
60
70
80
90
100
40
71
75
73
77
79
83
83
87
87
88
87
88
88
92
98
98
98
Clopyralid
Penflufen
Fluazinam
Oxadiargyl
Benzovindiflupyr
Tolfenpyrad
Novaluron
Chloridazon
Isopyrazam
Quintozene
Rotenone
Metamitron
Metazachlor
Penthiopyrad
Heptachlor
Azoxystrobin
Tetramethrin
Quinalphos

## APPENDIX 2. Graphical Representations

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




Number of laboratories analysing the test items by HRMs