PESTICIDE RESIDUE RESEARCH GROUP
European Union Reference Laboratory for Pesticide Residues in Fruits and Vegetables
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Ten years of European Union Proficiency Tests for Pesticide Residues in Fruits and Vegetables

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ABSTRACT

The European Union (EU) Reference Laboratory for pesticide residues in Fruits and Vegetables (EURL-FV) has been organizing a proficiency testing scheme annually since 2004. This scheme is specifically designed for the National Reference Laboratories (NRLs) and Official Control Laboratories (OCLs) responsible for pesticide residue control in the EU. The scheme gives additional value to the multiresidue qualitative and quantitative techniques used in routine analysis and facilitate the extension of the scope of the laboratories. The presentation is an overview of the EUPT-FV during 2014-2023, highlighting the challenges faced by participating laboratories and the significant achievements in sample extraction, data dispersion, and statistical evaluation.

Over the last ten years, the number of participating laboratories varied between 174 and 191 and more than 26000 results were evaluated (including false negatives). Furthermore, the distribution of z scores remained relatively consistent, with an average of 92 % of satisfactory, 3 % of questionable, and 5 % of unsatisfactory results.

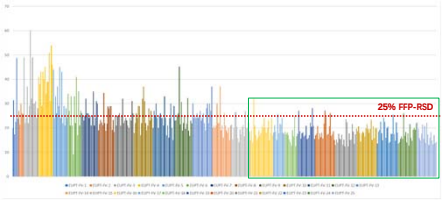
The huge sets of results has reinforced the adoption of the following two major practices: (i) the standard deviation for performance assessment (SDPA) set to 25 % was proven to be fit-for-purpose; and (ii) a target expanded measurement uncertainty of 50 % (=2*SDPA) is internationally accepted and widely used when reporting results for pesticide residue in fruits and vegetables analyses.

The PTs organised by the EURL-FV serve as a crucial platform for enhancing the proficiency and quality of pesticide residue analysis in fruits and vegetables, fostering a continuous improvement in analytical methodologies. The dedication and participation of NRLs and OCLs in these PTs contributes significantly to ensuring the safety and compliance of fruits and vegetables consumed in the EU.

Overview of EUPT-FV Results

EUPT No.	Matrices	Year	No. of participants	No. of possible pesticides	No. of pesticides evaluated in test item
16	Pepper	2014	183	175	22
17	Broccoli	2015	185	183	11
18	Spinach	2016	191	190	14 (+ 2 voluntary)
19	Lemon	2017	174	192	17 (+ 2 voluntary)
20	Green Bean	2018	184	195	19 (+ 2 voluntary)
21	Red Cabbage	2019	190	205	17 (+ 3 voluntary)
22	Onion	2020	176	208	17 (+ 2 voluntary)
23	Aubergine	2021	182	215	18 (+ 2 voluntary)
24	Tomato	2022	179	211	16 (+ 2 voluntary)
25	Melon	2023	175	212	17 (+ 1 voluntary)

Data dispersion (CV, %)



The representation of the dispersion of the EU proficiency tests results reveals the improvement of the participant laboratories, as it has decreased with the years independently of the concentration levels. In the figure, there is a clear trend towards improvement among European laboratories, being 18 % the average CV of the last ten EUPTs-FV (green rectangle). This figure reinforces the use of the 25 % FFP-RSD as well as of the internationally accepted 50 % target expanded measurement uncertainty for multiresidue analysis of pesticides.

Preparation of the test item

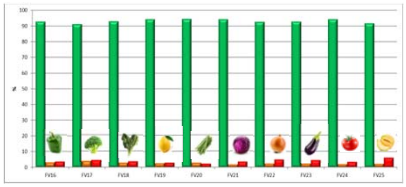
One of the peculiarities of EUPTs-FV is that the fruits and vegetables used to produce the test items are grown by the EURL-FV (the producer) in a greenhouse, whenever possible. In the last ten rounds, all the products except lemons were cultivated in experimental greenhouses of the University of Almería.

Plants are treated before harvest with some of the pesticides as commercial formulations.

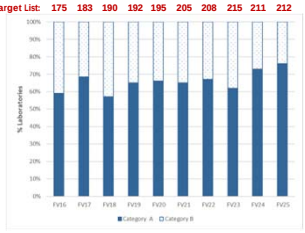
After harvest, plant products are spiked with pesticides as analytical standards. Depending on the commodity, spiking is carried out directly on the product or on the puree. After spiking, the material is homogeneised and packed.

z scores

In the last ten years, the percentage of acceptable, questionable and unacceptable z scores has remained very similar, with an average of 92 % of satisfactory, 3 % of questionable, and 5 % of unsatisfactory results.



Classification of the laboratories-Category A and B

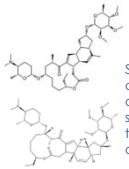


Laboratories in Category A were those that were able to analyse for at least 90 % of the pesticides of the target list, that reported at least 90 % of the pesticides present in the test item and did not report any false positive result. The rest of participants were classified in Category B. To better understand the real evolution of those laboratories it is important to consider the number of pesticides included in the Pesticide Target List, as the more pesticides on the list, the more difficult it is for laboratories to be in Category A. Over the same period of time, the number of pesticides in the Target List has increased from 175 to 212 pesticides, which emphasizes the progress made by laboratories in Category A.

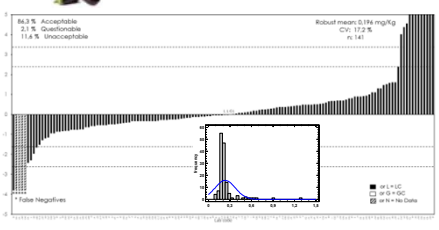
Analytical difficulties encountered by the participants

Spinosad

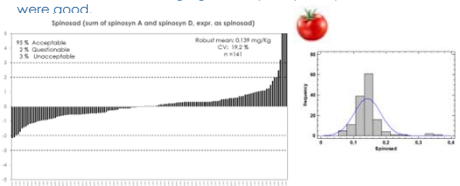
(EUPT-FV23 and EUPT-FV24)



Spinosad is a pesticide consisting of two components: spinosyn A and D, both present at different proportions in the spinosad analytical standard. Spinosad was added to the EUPT-FV23 test item, where the results showed bimodality due to the different quantification strategies.



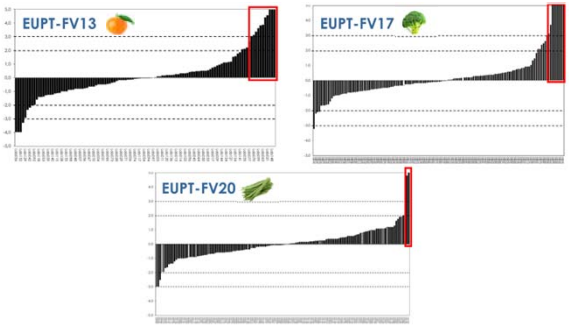
A year later, spinosad was also present in EUPT-FV24 test item, and due to the knowledge gained by the participants, the results were good.



Carbendazim

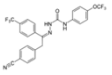
(EUPT-FV13, EUPT-FV17 and EUPT-FV20)

In some cases, questionable and unacceptable results in proficiency tests are closely related to an inadequate quantification as a result of a problem with the standard solutions.
The poor performance of carbendazim in all rounds where it was present could be attributed to the low solubility of carbendazim in organic solvents, causing overestimation by the use of incorrectly dissolved analytical standard solutions. EUPT-FV20 was the last round in which carbendazim was present. The figure below shows how the number of unacceptable z scores due to overestimation of the concentration has decreased over time, highlighting that participation in PTs is an effective quality control tool.

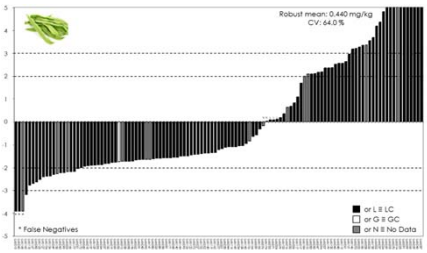


Metaflumizone

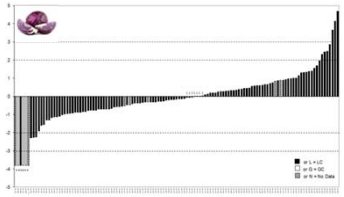
EUPT-FV20 and EUPT-FV21



Metaflumizone is a pesticide composed of two isomers: E- and Z-. However, in EUPT-FV20, some labs were not aware of this, resulting in a very high number of questionable and unacceptable z scores, as shown in the figure below.



In the subsequent round, EUPT-FV21, metaflumizone was again present in the test item, and the experience gained by most of the participants became evident, as z scores were good for most of them.



Acknowledgements: The authors wish to acknowledge funding support from the European Commission, DG SANTE (Grant decision SI2.870831). They would also like to thank the members of the Advisory Group for their valuable advice: André de Kok, Antonio Valverde, Björn Hardebusch, Finbarr O´Regan, Hans Mol, Hermann Unterluggauer, Magnus Jezussek, Marine Lambert, Mette E. Poulsen, Michelangelo Anastassiades, Patrizia Pelosi, Paula Medina, Radim Štěpán, Ralf Lippold, and Tuija Pihlström.