Interlaboratory Proficiency Testing: a key requirement of ISO 17025 for analytical quality

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OVERVIEW

Pesticide residue analysis in agricultural products is essential to ensure food safety and regulatory compliance. Residues are routinely determined using advanced chromatographic techniques such as Liquid Chromatography-Tandem Mass spectrometry (LC-MS/MS) and Gas Chromatography-Mass Spectrometry (GC-MS). In the European Union, maximum residue limits (MRLs) are established by Regulation (EC) n°396/2005 (**see Fig1**). To demonstrate compliance with these requirements, laboratories must generate reliable, valid, and traceable results through validated analytical methods and robust quality assurance systems, in accordance with the requirements of ISO/IEC 17025.

Pesticide	Matrix / Type	Typical EU MRL (mg/kg)
Glyphosate	Wheat / Cereal	10
Cypermethrin	Wheat / Cereal	2
Imidacloprid	Tomato / Vegetable	0,5
Acetamiprid	Honey / Apiculture product	0,05
Chlorpyriphos	Carrot / Vegetable	0,01*

Fig. 1 – Examples of MRLs values, extracted from Regulation (EC) n°396/2005 (may be sujected to amendments)

** Defaut limit of quantification (LOQ) generally applied to non-approved active substances in the European Union*

INTRODUCTION

In this context, participation in interlaboratory Proficiency Testing (PT) is an essential tool for evaluating the analytical performance of laboratories and thus ensuring the quality and validity of analytical results.

These proficiency tests (**see Fig2**) are carried out according to the NF EN ISO 17043 standard.

Participation in these trials is a requirement of the NF EN ISO/IEC 17025 standard for all accredited laboratories, thereby contributing to the improvement of the analytical capabilities of participating laboratories.

BIPEA proficiency testing program	Matrices
19A	Pesticides: Fruits and citrus fruits
19B	Pesticides: Cereals
19C	Pesticides: Vegetables
19D	Pesticides: Fats and oils
19E	Bromides / nitrates : Vegetables
19F	Pesticides: wines
19G	Pesticides: Honey
19H	Dithiocarbamates: Fruits / Vegetables
19J	Pesticides: Dairy food
19K	Pesticides – Medicinal and aromatic plants
19L	Pesticides – Dried fruits and nuts
19M	Pesticides – Egg products
19N	Pesticides – Sea products
19P	Pesticides – Feed

Fig. 2 – Examples of proficiency testing programs

PARTICIPATION

Long-term participation in proficiency testing scheme Is a key component of analytical quality assurance for pesticide residue laboratories. In accordance with ISO/IEC 17025 requirements, proficiency testing supports the continuous monitoring of analytical methods and their performance. Furthermore, a high level of participation (**see Fig3**) strengthens the statistical robustness of the assigned values and performance evaluations, thereby increasing the confidence that participants can place in the results obtained. The growing numbers and diversity of PT dedicated to pesticide residue analysis (**see Fig4**) reflect the increasing need among laboratories to assess their measurement capabilities across a wide range of matrices, analytes, and concentration levels. Consequently, PT plays a central role in harmonizing analytical practices.

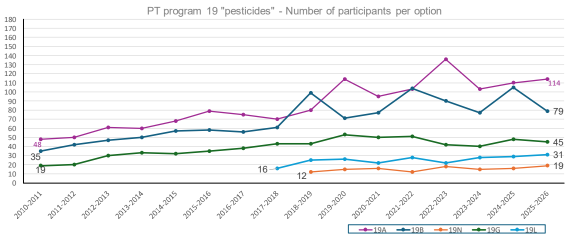

Fig. 3 – Evolution of the number of participants

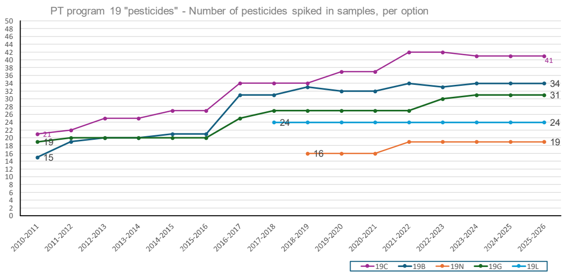

Fig. 4 – Evolution of the number of pesticides spiked in samples

METHODOLOGY

The setting up of a proficiency test can be schematized by 4 main steps: test design, preparation of homogenous samples, analyses by the laboratories, statistical treatment of the data.

For these PT 'pesticides', laboratories are asked to analyze samples to which pesticide solutions have been added. As an example, a test was conducted in November 2024 using a vegetable matrix - tomato - characterized first to be free from any pesticide's residues, and then spiked with about 40 pesticides, mainly at levels between 20 and 200 µg/kg.

63 laboratories took part in this test. The techniques used by the laboratories were LC-MS-MS, GC-MS-MS, GC-MS and GC-ECD, according to the molecules. Participating laboratories were required to return their results on a dedicated website

SAMPLES PRODUCTION

The raw material was an organic product, first analyzed to detect the possible presence of pesticides before the production (**see Fig5**).

The whole batch of tomato was ground to obtain a pasta and then spiked with a pesticides mix of all the molecules. After mixing, the product was sampled using an automatic piston system, which distributes the products in successive layers into flasks positioned on a conveyor belt, involving thus the quasi simultaneous filling of the samples and allowing to ensure the homogeneity between the samples.

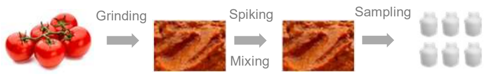

Fig. 5 – Process for the samples preparation

ANALYSIS BY LABORATORIES AND STATISTICAL TREATMENT

Samples were shipped frozen to all participants who are invited to analyze the samples as soon as possible after reception.

A statistical treatment was conducted according to ISO 13528 standard. An assigned value (x_{pt}) was estimated for each parameter.

Performances of each laboratory were evaluated using a tolerance value, set by the Technical Committee, by calculating z-scores (z).

The z-score (z_i) for a result x_i is calculated as :

$$z_i = \frac{(x_i - x_{pt})}{\sigma_{pt}}$$

Where σ_{pt} is the standard deviation for proficiency assessment (VT/2).

The results can then be classified as follows:

- $|z| \leq 2$: satisfactory
- $2 < |z| < 3$: questionable
- $|z| \geq 3$: unsatisfactory.

RESULTS, DISCUSSION and CONCLUSION

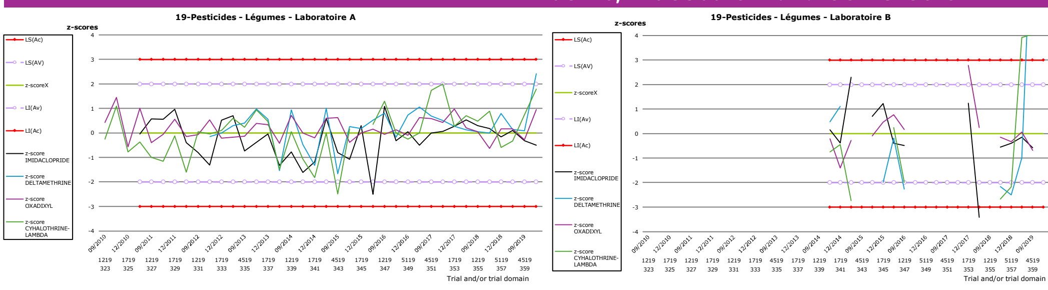

Fig. 6 – Trend chart: evolution of the z-score over the period 2010–2024

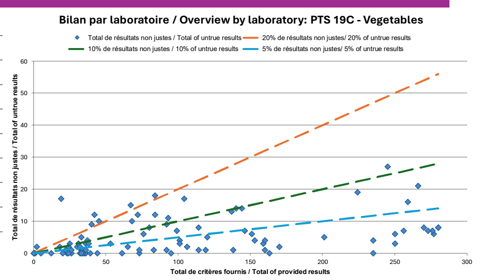

Fig. 7 – Laboratory review for the period 2023–2025

(**see Fig6**)The comparison is made between Laboratory A, which has been participating in the long-term 'pesticides–vegetables' trials (on a regular basis since 2010), and Laboratory B, which has only been participating in these trials more recently and on a much more intermittent basis. A long-term involvement enables laboratories to detect bias or non-compliant results; these serve as a warning signal for the implementation of corrective and/or curative actions in the laboratory.

(**see Fig7**) The review is also carried out by laboratory, listing for each one (indicated by a blue diamond) the number of non-compliant results relative to the number of criteria analysed, over the specified period. Once again, it is evident that laboratories which carry out analyses on a regular basis generally achieve better results than those which participate in the tests on a more occasional basis.

Interlaboratory proficiency testing is therefore an essential tool enabling participants to demonstrate their competence and guarantee the quality of their results, in accordance with the requirements of the NF/EN ISO 17025 standard. In the field of pesticide analysis, the tightening of regulations in recent years highlights the importance of using such quality tools.