Uncertainty of measurement and pesticide residues in vegetable products: application of alternative approaches based on quality control data for multi/single residue methods

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Abstract

It is a requirement under ISO/IEC 17025 that laboratories determine the measurement uncertainty (MU): in the analysis of pesticide residues in food a value of 50 % of expanded MU is associated with analytical result. A prerequisite for the use of the 50% default expanded MU is that the laboratory demonstrates that its own expanded MU is less than this maximum value (1).

Since 2009, a European Standard has been issued for vegetable products describing a method for the analysis of pesticide residues in foods of plant origin such as fruits, vegetables and cereals (2). This quick and easy method, detecting multi analytes in a single extraction, has been collaboratively studied on a large number of commodity/pesticide combinations. Whereas some specific pesticide residues require Single Residue Methods because are not amenable to the Standard method EN 15662:2018. Consequently, we defined a workflow to estimate measurement uncertainty depending on the type of method employed: multi or single residue.

For the first kind of method, the calculation of individual measurement uncertainty (MU) may not always be possible; so an alternative approach was applied estimating a generic MU using data from three different proficiency tests selected for the three main product groups (fruit, vegetable and cereal) defined in the scope of the Standard method in combination of intra – laboratory precision. This approach presents a limitation due to the minimum number of results (31 results) to take into account. This limitation did not permit to apply the same approach in case of a Single Residue Method owing to the limited data of proficiency tests (often less than 31 results) due to the limited pesticide residues / matrix combinations. Therefore, to estimate the MU we applied a specific approach based on internal laboratory quality control for individual pesticides in a specific family group of commodities obtained from routine analysis on real samples. The Flonicamid residue in vegetable products was reported as a case study for Single Residue Method.

References

1. European Commission. Guidance document on analytical quality control and method validation procedures for pesticides residues in food and feed. Brussels: European Commission; 2017. (SANTE/11813/2017).
2. European Committee for Standardization. Foods of plant origin – Multimethod for the determination of pesticide residues using GC- and LC- based analysis following acetonitrile extraction/partitioning and clean-up by dispersive SPE – Modular QuEChERS method. EN 15662:2018.