European Union proficiency tests for pesticide residues in fruit and vegetables from 2009 to 2016: Overview of the results and main achievements

Carmen Ferrer [a, b, *], Ana Lozano [a, b], Samanta Uclés [a, b], Antonio Valverde [a], Amadeo R. Fernández-Alba [a, b]

[a] Agrifood Campus of International Excellence (CeiA3), Department of Chemistry and Physics, Pesticide Residue Research Group, University of Almeria, 04120 Almería, Spain
[b] European Union Reference Laboratory for Pesticide Residues in Fruits and Vegetables, University of Almeria, 04120 Almería, Spain

ARTICLE INFO

ABSTRACT

The European Union Reference Laboratory for pesticide residues in Fruits and Vegetables (EURL-FV) is organising, every year since 2004, the European Commission Proficiency Test for multiresidue analysis of pesticides in fruits and vegetables (EUPT-FV), which is directed to all National Reference Laboratories (NRLs) and all Official Laboratories (OfLs) responsible for pesticide residue control in the European Union. This work provides an updated overview of the last eight editions of EUPT-FV (2009—2016), highlighting the main difficulties encountered by the participant laboratories and the key achievements in the field of sample extraction and data dispersion and statistical evaluation. The number of participant laboratories was every year increasing from 151 in 2009 (EUPT-FV11) to 191 in 2016 (EUPT-FV18), and the total number of pesticide residue results reported by the laboratories in this period was close to 18,000. The percentage of acceptable, questionable and unacceptable z scores values assigned in each one of these eight EUPT-FVs has remained very similar with average values of 91.8, 3.5, and 4.7 percent, respectively. The large amount of data results evaluated has led to a strengthening in the use of the 25 percent fit-for-purpose relative standard deviation (FFP-RSD) as well as the use of an internationally accepted 50 percent target expanded measurement uncertainty default value when reporting results for multiresidue analysis of pesticides.

© 2017 Elsevier Ltd. All rights reserved.

1. Introduction

The monitoring of pesticide residues in food is relevant for the implementation of Regulations (EC) No 396/2005 on maximum residue levels of pesticides (MRLs) and (EC) No 882/2004 on official controls in the framework of an integrated and uniform approach to official controls along the agrifood chain, allowing competent authorities in the Member States to verify compliance with food and feed law. Article 53 of Regulation (EC) No 882/2004 on official controls empowers the Commission to recommend coordinated plans organised on an ad hoc basis. The EU multiannual control programme establishes the products to be sampled by the EU Member States and a list of priority substances to be analysed, in order to ensure compliance with MRLs of pesticides and to assess the consumer exposure to pesticide residues in and on food of plant and animal origin. Regulation (EC) N° 882/2004 also establishes EU Reference Laboratories (EURLs) with the aim to ensure high-quality, uniform testing in the EU and support Commission activities on risk management and risk assessment in the area of laboratory analysis.

Proficiency tests by interlaboratory comparison are an effective tool to assess and monitor the quality performance of analytical laboratories (Fernández-Alba, 2004). They are designed principally to enable participant laboratories to detect and remedy short-comings in their procedures (International Organization for Standardization-ISO, 2010; Thompson, Ellison, & Wood, 2006).

* Corresponding author. Agrifood Campus of International Excellence (CeiA3), Department of Chemistry and Physics, Pesticide Residue Research Group, European Union Reference Laboratory for Pesticide Residues in Fruits and Vegetables, University of Almeria, 04120 Almería, Spain.
E-mail addresses: cferrer@ual.es (C. Ferrer), analozano@ual.es (A. Lozano), samantaucles@ual.es (S. Uclés), avalverd@ual.es (A. Valverde), amadeo@ual.es (A.R. Fernández-Alba).

Participation in proficiency tests is part of the external quality assessment of the laboratory, and it is a requirement in order to be accredited under ISO/IEC 17025:2005 (International Organization for Standardization-ISO, 2005).

Within this context, the EURL for pesticide residues in fruits and vegetables (EURL-FV) annually organises EU-proficiency tests (EUPTs) on behalf of the European Commission to evaluate the competency of the EU National Reference Laboratories (NRLs) and EU Official Laboratories (OfLs) for analysing pesticide residues in fruits and vegetables. For those laboratories undertaking pesticide residue analyses within the frame of national and EU official controls, participation in EUPTs is mandatory. Laboratories from the European Free Trade Association (EFTA), as well as laboratories from non-EU/EFTA countries can also participate after approval by DG SANTE (Directorate General for Health and Food Safety-European Commission). In 1996, the National Food Administration in Uppsala (Sweden) in cooperation with the University of Almería (Spain) started organising the EUPTs-FV. Since 2004, the EURL-FV has organised them on behalf of the European Commission, DG SANTE. In that 20-year period (1996−2016), 18 editions have been performed in 17 different fruit and vegetable commodities, and using a total of 113 different pesticides, which has resulted in the generation of more than 30600 pesticide residue results using multiresidue methods (European Union Reference Laboratory for Residues of Pesticides in Fruits and Vegetables-EURL-FV) (the network of EU OfLs is comprised of around 170 routine laboratories analysing fruits and vegetables using multiresidue methods). Detailed information on the history, organization and protocols of the EUPTs-FV was published by Medina-Pastor, Fernández-Alba, Andersson, and Rodríguez-Torreblanca (2010), together with the evaluation of the results from EUPT-FV01 to EUPT-FV10. A summary of the matrices, number of participants, number of possible pesticides and number of pesticides evaluated in the test item of the last eight EUPTs-FV is presented in Table 1. As can be observed in the table, participation in these EUPTs has increased over the years, being the number of participants 25 percent higher in EUPT-FV18 than in EUPT-FV11. This can be explained by the growing increase of OfLs due to the incorporation of new Member States into the EU, to the growth of the laboratory networks, and the increment of non-EU/EFTA participants as a result of the big impact of the international trade between EU and other external countries. In the last eight years, the percentage of non-EU/EFTA participants has ranged between 5 and 10 percent.

The number of pesticides in the Pesticide Target List (list of possible pesticides in the test item) has also increased in the last eight-year period, from 128 compounds in EUPT-FV11 to 190 in EUPT-FV18, in parallel with the growth of the number of pesticides included in the EU multiannual control programme.

From 2014 onwards, the EUPTs-FV have been organised under ISO/IEC 17043 accreditation (International Organization for Standardization-ISO, 2010), which is the International Standard that specifies general requirements for the competence of providers of proficiency testing schemes and for the development and operation of proficiency testing schemes. Before that, all EUPTs-FV were performed according to the specifications of the International Laboratory Accreditation Cooperation Guideline (International Laboratory Accreditation Cooperation, 2000), of ISO/IEC 17043 (International Organization for Standardization-ISO, 2010) and ISO Guide 43 (International Organization for Standardization-ISO, 1999). Based on the aforementioned standards, the organization of the EUPTs-FV follows the procedures established in both the EUPT-General Protocol and the corresponding EUPT-Specific Protocol, drafted and updated by the EUPT-Scientific Committee. That committee also sets out the Pesticide Target List for each round, together with the Minimum Required Reporting Levels (MRRLs) for each pesticide (the level that laboratories are expected to attain using their analytical methods).

This work aims to provide an updated overview of the last eight years of EUPTs in fruits and vegetables, highlighting the main difficulties encountered by the participant laboratories and the key achievements in the field of sample extraction and data dispersion and statistical evaluation.

2. Overview of the results

2.1. Estimation of the assigned value and data variability

Different approaches can be followed to calculate the assigned value (International Laboratory Accreditation Cooperation, 2000; International Organization for Standardization-ISO, 2010; Thompson, Mertens, Kessler, & Fearn, 1993). In the case of EUPTs-FV, it is the consensus value (taking into account the results reported by EU and EFTA countries' laboratories only) that derives directly from the reported results. From EUPT-FV17 onwards, and in order to minimise the influence of outlying results on the statistical evaluation, the assigned value has been estimated using robust estimate of the participant's mean, as described in Annex C of ISO 13528:2015 (International Organization for Standardization-ISO, 2015), where the robust mean according to algorithm A is defined. The uncertainty of the assigned values $u(x_{pt})$ is also calculated according to ISO 13528:2015 as

$$u(x_{pt}) = 1.25 \times \frac{s^*}{\sqrt{p}}$$

where s^* is the robust standard deviation and p is the number of results. If $u(x_{pt}) < 0.3\ \sigma_{pt}$ then the uncertainty of the assigned value may be considered to be negligible and wouldn't need to be included in the interpretation of the results of the round of proficiency testing (being σ_{pt} the standard deviation for proficiency assessment).

Data variability has been calculated in various ways in the different EUPTs-FV. Since EUPT-FV04, the FFP-σ_{pt} has been

Table 1
General information of the EUPTs-FV organised during the period 2009−2016.

EUPT Number	Year	Matrices	Number of Participating Labs.	Number of possible pesticides	Number of pesticides evaluated in the test item
FV-11	2009	Cauliflower	151	128	21
FV-12	2010	Leek	153	144	17
FV-13	2011	Mandarin	154	144	19
FV-14	2012	Pear	167	175	17
FV-15	2013	Potato	175	175	18
FV-16	2014	Pepper	183	175	22
FV-17	2015	Broccoli	185	183	11
FV-18	2016	Spinach	191	190	12