General protocol
for EU proficiency Tests for Pesticide Residues in Food and Feed

Introduction

This protocol contains general procedures valid for all European Union proficiency tests (EUPTs) organised on behalf of the European Commission, Health & Consumer Protection Directorate-General (DG-SANCO) by the four Community Reference Laboratories (CRLs) for pesticide residues in food and feed. These EUPTs are directed at all National Reference Laboratories (NRLs) and Official Laboratories (OfLs) in the EU Member States. Laboratories outside this CRL/NRL/OfL-Network[1] may be permitted to participate on a case-by-case basis after consultation with DG SANCO.

The following four CRLs for pesticides were appointed by DG-SANCO based on regulation 882/2004/EC[2]:

- CRL for Fruits and Vegetables (CRL-FV),
- CRL for Cereals and Feedingstuff (CRL-CF),
- CRL for Food of Animal Origin and Commodities with high Fat Content (CRL-AO) and
- CRL for Single Residue Methods (CRL-SRM)

NRLs are appointed by the National Food or Feed Authorities based on the provisions of Regulation 882/2004/EC, whereas OfLs are laboratories that are actively involved in providing residue data for the national control programme and/or the co-ordinated multiannual Community control programme.

According to Regulation 396/2005/EC[3] all laboratories analysing samples for the official controls on pesticide residues shall participate in the Community proficiency test(s)

[1] For more information about the CRL/NRL/OfL-Network please refer to the CRL-Web-portal under: http://www.crl-pesticides.eu

[2] Regulation (EC) No 882/2004 of the European Parliament and of the Council on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules. Published at OJ of the EU L191 of 28.05.2004

organised by the Commission. The aim of these EUPTs is to obtain information regarding the quality, accuracy and comparability of the pesticide residue data in food and feed sent to the European Commission within the framework of the national control programmes and the co-ordinated multiannual community control programme. Participating laboratories will be provided with an assessment of their analytical performance and the reliability of their data - compared to the other participating laboratories.

EUPT-organisation

EUPTs are organised by individual CRLs or by more than one CRL in cooperation with one another.

For each EUPT an Organising Team is appointed by the CRL(s) that is responsible for the EUPT. This team is then responsible for all administrative and technical matters concerning the organisation of the PT, e.g. PT-announcement, production of the test material, undertaking the homogeneity and stability tests, packing and shipment of test material, and the handling and first assessment of participant's results.

A common Scientific Committee entailing the following two subgroups:

a) An Advisory Group (AG) and

b) An independent Quality Control Group (QCG)

consisting of expert scientists with long experience in pesticide residue analysis that have been appointed by the CRLs and approved by the DG-SANCO.

The role of the AG is to help the organisers in making decisions concerning the design of the EUPT: selection of pesticides to be included in the Target Pesticide List (see below), the establishment of the minimum required reporting levels (MRRLs), the evaluation and statistical treatment of the results and the drafting of the protocol anf final report. The QCG has the additional function of supervising the quality of the EUPT and to assisting the CRLs with confidential aspects such as the choice of the pesticides, and levels to be present in the test material.

The EUPT-Organising Team, AG and QCG together form the **EUPT-Panel**.

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

Confidentiality:

In each EUPT the laboratories are given a unique code only known to themselves, the Organisers, and DG-SANCO. In the final EUPT-Report the list of participating laboratories will not be linked to their laboratory codes. It should be noted that the organisers, at the request of the Commission may present the results to the Standing Committee on the Food Chain and Animal Health on a country-to-country basis. It is therefore possible that a link between codes and National Reference Laboratories could be made, especially for those Member States where only one laboratory has participated. The owner of all EUPT data is DG SANCO.

Communication

The official language used in all EUPTs is English.

Communication between participating laboratories during the test on matters concerning this PT exercise is not permitted.

Announcement

The announcement of the individual EUPT will be issued at least 3 months before the test material is distributed to the laboratories. The announcement will be published on the CRL portal and distributed via mail to the NRL/OfL mailing list available to the CRLs. The announcement will contain an invitation letter, details on how to register and where to locate additional related documents, and some preliminary information on the specific protocol such as the tentative calendar, the name of the commodity expected to be used, and the tentative Target Pesticide List.

Specific Protocol

For each PT a Specific Protocol will be published at least 2 weeks before the test material is distributed to the laboratories. This protocol will contain all information the included in the invitation in its final version, information on payment for delivery service and/or participation. Furthermore, it will also include instructions on how to handle the test material upon receipt, on how to submit results, and other relevant information.

General procedures for reporting results

Laboratories are responsible for reporting their results to the Organiser within the stipulated deadlines. Each laboratory must only report one result for each of the pesticides present in the test material, using the analytical procedure(s) that they would routinely use for each compound for monitoring purposes. More than one method may be used to cover all the compounds to be sought. The results (residue levels of the pesticides detected) must be, expressed in mg/kg.

Correction of results for recovery

According to the Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed, (Document SANCO in force each year) residues data should not normally be adjusted for recovery, when the mean recovery is within the range of 70-120%. If residues data are adjusted for recovery, then this must be clearly stated. Therefore laboratories are required to report whether their results were adjusted for recovery and if this was the case, the recovery factor used. No recovery factors are required where recovery adjustments resulted from using the 'standard addition(s)' approach, or from the use of isotopically labelled internal standards (with spiking of the test material at the beginning of the extraction procedures). In this case, the laboratories should report the technique used for calculation of the results instead of the recovery factor.

Evaluation of the Results

The procedures used for the treatment and assessment of results are described below.

- ***False Positives***

These are the results that show the apparent presence of pesticides that were listed in the Target Pesticide List, but which were (i) not used in the sample treatment, (ii) and not detected by the organiser, even after a repeat analysis. However, if a number of participants do detect the same additional pesticide, or if the concentration is above the MRRL, then a decision as to whether, or not, this should be considered to be a false

positive result will be made on a case-by-case basis. Any results reported that are lower than the MRRL will not be considered as false positives, even though these results should not have been reported.

- ***False Negatives***

These are results for pesticides reported by the laboratories as “analysed” but that no numerical values were given, although they were used by the Organiser to treat the test material and were detected by the majority of participants at or above the MRRL.

- ***Estimation of the true concentration (μ)***

The “true” concentration will be typically estimated using the median of all the results. Therefore a **median value** for every compound present will be calculated and used as the assigned value. In special justifiable cases, the EUPT Panel may decide to use only part of the population of results to establish the median (e.g. using only results with z-scores ≤ 5.0).

- ***Establishing the standard deviation of the assigned value (target standard deviation)***

The target standard deviation (δ) of the median will be calculated using a Fit-For-Purpose Relative Standard Deviation (FFP-RSD) approach, as follows:

$$\delta = b_i * \mu_i \qquad \text{with } b_i \text{ i} = \text{FFP-RSD (= 0.25)}$$

The percentage FFP-RSD is typically set at 25% based on experience from previous EUPTs. The EUPT-Panel reserves the right to also employ other approaches on a case-by-case basis considering analytical difficulties, and experience gained from previous proficiency tests.

– ***z-scores***

This parameter is calculated using the following formula:

$$z_i = (x_i - \mu_i) / \delta_i$$

Where x_i is the value reported by the laboratory, μ_i the assigned value, and δ_i the standard deviation at that level for each pesticide (i).

Any z-scores of > 5 will be reported as "+5" particularly where summed z-scores of many pesticides are calculated (see SWZ below).

z-scores will be interpreted in the following way:

$|z| \leq 2$ Acceptable

$2 < |z| \leq 3$ Questionable

$|z| > 3$ Unacceptable

For results that are considered to be false negatives, z-scores will be calculated using the MRRL or RL (the laboratory's Reporting Limit), if the RL < MRRL.

The EUPT-Panel will consider whether, or not, these values should appear in the z-score histograms.

However, a z-score will not be calculated for any false positive result.

– ***Category A and B classification***

The EUPT-Panel will decide whether to classify the laboratories in two groups, A and B. Laboratories that detected a sufficiently high percentage of the pesticides present in the test material (e.g. at least 90%), reported no false positives, and sought all the pesticides on the Target Pesticide List marked with an asterisk that were present in the test material, will have demonstrated 'sufficient scope' and will therefore be classified in Category A.

– ***Combined z-scores***

For evaluation of the overall performance of the laboratories within Category A, a ranking according to the sum of weighted z-scores (SWZ) will be calculated.

The sum of weighted z-scores formula uses the z-scores with a fixed maximum value of 5 for individual z-scores, using the following formula:

$$\text{'Sum of weighted z-scores'}\ (Z) = \frac{\sum_{i=0}^{i\leq 2}|z|\cdot 1 + \sum_{i>2}^{i\leq 3}|z|\cdot 3 + \sum_{i>3}^{\infty}|z|\cdot 5}{n}$$

n = number of reported results

So for each laboratory:

- The first summation is the sum of all their /z-scores/ between zero to two, multiplied by 1.
- The second summation is the sum of all their /z-scores/ greater than two but less than or equal to, three, multiplied by 3.
- The third summation is the sum of all their z-scores greater than three, multiplied by 5.

This SWZ has the following classification similar to the z-score:

$Z \leq 2$ Good

$2 < Z \leq 3$ Satisfactory

$Z > 3$ Unsatisfactory

The sum of weighted z-scores is considered to be of lesser importance than the individual z-scores. Therefore the organiser, in agreement with the EUPT-Panel, retains the right not to use them if they are considered to be unhelpful.

Publication of results

The preliminary results from the EUPTs will be published within 2 months from the deadline for result submission.

The final report will be published shortly after the organiser and the EUPT-Panel have discussed the results. Taking into account that the EUPT-Panel normally only meets once a year, the final report may be published up to 8 months after the deadline for results submission.

Disclaimer

The EUPT-Panel retains the right to change any parts of this EUPT - General Protocol based on new scientific or technical information. Any changes will be communicated in due course.