EUPT-FV18 SPECIFIC PROTOCOL

European Union Proficiency Test for Pesticide Residues in Fruits and Vegetables (2016)

Introduction

This protocol is complementary to the General Protocol of EU Proficiency Tests (EUPT) for Pesticide Residues in Food and Feed (6th Edition). This Proficiency Test is organised by the EURL for Pesticide Residues in Fruits and Vegetables covering Multiresidue Methods (MRM) of analysis.

According to Article 28 of Regulation 396/2005/EC (23rd February, 2005) of the European Parliament and of the Council, all laboratories analysing samples for the official control of pesticide residues shall participate in the European Union Proficiency Tests (EUPTs) for pesticide residues organised by the European Union.

These proficiency tests are carried out in order to improve the quality, accuracy and comparability of the residue data and to evaluate the laboratory capacity to report results that covers the entire range of maximum residue limits (0.005 - 15 mg/kg) in all groups of fruit and vegetable matrices (high water, acid and fat content). Bearing that, a wide concentration range should be covered with the different analytes present in the test item.

Test Item

This proficiency test is based on the analysis of incurred pesticide residues in **spinach**. The spinaches were grown in a greenhouse. The pesticide treatments carried out were pre-harvest using commercial formulations. The test item was frozen (using liquid nitrogen), chopped, homogenised and sub-sampled into polyethylene bottles that had previously been coded.

Ten of these bottles containing the test item were chosen randomly, and analysed to check for homogeneity.

The test item was stored frozen (–20°C) prior to shipment to participants.
Six bottles, again chosen randomly, will be analysed over a period of time to confirm the stability of the pesticides in the test item (three when the test items are shipped, then other three bottles a few days after the deadline for submitting results). There will be one further analysis during this period reproducing the sample shipment to see if there is any degradation of any of the pesticides present in the test item.

Subcontracting

All analytical determinations concerning the test item treatment analysis will be performed in a laboratory which is ISO 17025 accredited.

Target List

In addition to the pesticide target list of mandatory compounds, a "voluntary target list" containing pesticides which might be present in the test item will be published. Those voluntary pesticides will not be used for the evaluation of the laboratories into Category A or B, and a separate statistical evaluation will be made for them.

Assigned value and robust relative standard deviation

In order to minimise the influence of out-lying results on the statistical evaluation, the assigned value will be estimated using the robust statistics as described In ANNEX C of ISO 13258:2015, where the robust average (x*) according algorithm A is defined.
Also, the robust relative standard deviation (CVs*) will be calculated for each analyte.

Laboratory assessment

For the assessment of the overall laboratory performance, the Average of the Squared z-Score (AZ^2) will be used as in the last Proficiency Test, but only for those laboratories in Category A, which will be those laboratories that are able to analyse at least 90% of the pesticides in the target list, that are able to detect at least 90% of the pesticides present in the test material and that report no false negatives neither false positives. Within Category A, the laboratories will be sub-classified as "good", "satisfactory" or "unsatisfactory". All the other laboratories will be classified in Category B. This information will be available in the General Protocol.

Steps to follow

This Proficiency Test will be made up of the following 9 essential steps:

1. To participate, each laboratory must complete the Application Form on-line, available on the EURL-FV Web page, before the deadline stipulated on the Calendar. It is recommended that laboratories download the Target Pesticide List from this web site. Laboratories should carefully read the Target Pesticide List, where important information about the reporting of the results, as well as the Minimum Required Reporting Limits (MRRLs), is given. The MRRLs do not always correspond with the EU MRLs set for spinach.

2. When the registration period is closed, laboratories will receive an e-mail confirming their participation in this exercise, and assigning them each a Laboratory Code. Laboratories with this code will be able to access the restricted area containing the forms using their login information - consisting of their **USER NAME**, which is the Laboratory Code expressed as **LabXXX** (three digits with no spaces between them) and their **PASSWORD**, as chosen on the application form.

3. The sample delivery will be **200 euros** for EU-NRLs, **230 euros** for EU/EFTA official laboratories and **250 euros** for official laboratories from other countries. The laboratories will receive an invoice and after that they can start the payment procedure. An e-mail showing the bank transfer confirmation, or similar, may be requested at any time by the Organiser. **Payments without a Laboratory Code identifying them will not be considered as paid.**

4. Any communication with the Organisation should be made using a **Contact Form** placed in the restricted area.

5. **Form 0 - Laboratory Scope** will be placed in the restricted area and will be open to participants from the 25th January - 5th February 2016, prior to test item shipment. The aim is that laboratories provide information regarding their scope of analysis before receipt of the test item and detailed information regarding which pesticide is within the accredited scope of the lab and which is not. After the deadline it will not be possible to change the scope.

6. When the participant laboratories receive the test item (and not before), they must enter the restricted area again and submit **Form 1 - Test Item Receipt** to inform the Organiser that they have accepted the test item. This Form has a deadline: 12th

February 2016, which must be met. If no test item has been received by this deadline, the laboratories should contact the Organiser using the Contact Form of the restricted area. If form 1 is not filled in, the Organiser will consider that the participant has accepted the test item.

7. The participant laboratories must respect the deadline for submitting their results – 1st March 2016- using **Form 2** – **Identified pesticides**; **Form 3** - **Results** and **Form 4** - **Methods** on-line.

8. One final form, **Form 5 - Additional Information Requested** can be filled in after the deadline has passed. This Form will be available from 7th-11th March 2016. Not all laboratories may need to fill this in. It will depend upon information reported on previous Forms.

9. The Organiser will evaluate the results at the end of the proficiency test, once the deadline for receipt of results has passed. The Organiser will upload an electronic version on the EURL-FV web site and afterwards send a hard copy of the Final Report to each participant laboratory. This report will include information regarding the design of the test, the homogeneity and stability results, a statistical evaluation of the participant's results as well as graphical displays of the results and any conclusions. Further relevant information considered to be of value may also be included.

Form 0 - Laboratory Scope

Before the participant laboratories receive the sample, the restricted area will be open so that their laboratory scopes can be recorded. Form 0 will need to be filled in to ascertain which of the pesticides in the Target Pesticide List were actually sought. After the deadline it will not be possible to change the scope.

This form will also request information on which of the pesticides sought by the laboratory is within the laboratory's accredited scope.

Amount of Test Item

Participants will receive:

- Approximately 200 g of spinach test item treated with pesticides.
- Approximately 200 g of 'blank' spinach test item.

Shipment of Test Item

All Test Items will be frozen and packed in polystyrene boxes surrounded in dry ice and packed into cardboard boxes.

The shipment of the test items will be carried out over a one-week period from the 8th February 2016. The Organiser will try to ensure that all the packages arrive on the same day to each laboratory. An information message will be sent out by e-mail before shipment. Laboratories must make their own arrangements for the receipt of the package. They must inform the Organiser of any public holidays in their country/city during the delivery period given in the calendar, as well as making the necessary arrangements for receiving the shipment, even if the laboratory is closed.

Advice on Test Item Handling

Once received, the test item should be stored deeply frozen (-18°C or less) prior to analysis thus avoiding any possible deterioration/spoilage. The test item should be mixed thoroughly before taking the analytical portion(s).

All participants should use their own routine standard operating procedures for extraction, clean-up and analytical measurement and their own reference standards for identification and quantification.

Form 1 - Test Item Receipt

Once the laboratory has received the test item, its arrival must be reported to the Organiser using Form 1 in the restricted area; filling in the date of receipt, the condition of the test item, and its acceptance. The deadline for acceptance (or non-acceptance) is 12th February 2016. If the laboratory does not respond by this date, the Organiser will assume that the test item has been received and accepted.

If any laboratory has not received the test item by 12th February, they must inform the Organiser **immediately** using the Contact Form of the restricted area.

Submission of results:

Once the laboratory has analysed the test item and is ready to submit their data, they must enter their results at various steps on 3 forms by accessing the restricted area in the EURL –FV web site: http://www.eurl-pesticides.eu

Identified Pesticides – Form 2

In Form 2, the information entered in Form 0 – Laboratory Scope, will be made available again.

For each pesticide included in the laboratory scope, the Limit of Quantification (LOQ) will be requested. The MRRL and the participant's own LOQ will be used to help identify false negative results.

Before this, a question will be requested as to which approach was used for the relative expanded uncertainty estimation in multiresidue methods for fruits and vegetables.

The laboratories will be also asked to report any pesticide that may have been detected in the blank test item

This form can be filled in at various stages - so once entered, the data will be saved, and the laboratories can add further data at a later date.

Results – Form 3

In this step, the laboratory should report the measured concentrations for each determination. All concentrations must be expressed in mg/kg together with the recovery as a percentage.

The number of significant figures should be based on the procedures provided in SANTE/11945/2015. Additional significant figures may be recorded for the purpose of statistical analysis.

Results should not be reported where a pesticide was not detected or was detected below the laboratory LOQ. In both cases, this should be recorded as 'ND'. If a pesticide was not sought, it should be recorded as 'NA' (Not Analysed). The actual results/residue levels measured must be reported as numbers. Symbols (>, <, ±, ≥, ≤, …) will not be accepted. IMPORTANT: If your result is not correctly expressed it will be considered as 'NA' (Not Analysed).

Methods – Form 4

In this step, the laboratory must report the details of the analytical methods they used. A list including all the pesticides detected in the sample will be shown along with a pesticide reference number. Laboratories may describe a method for the first pesticide and use this pesticide reference number to refer to other pesticides determined using the same method.

Again in this form, information must always be saved so that laboratories can go back to it and continue at any time before the final reporting deadline - which for all forms is 1st March 2016. Any results reported after this deadline will not be included in the statistical treatment, nor in the final report.

It should **not** be assumed that only pesticides registered for use on spinach are present in the test item.

False Negatives or Additional Information – Form 5

This Form will be available only for those laboratories reporting that they sought a pesticide present in the test item but for which no method was reported in Form 4. If a laboratory accesses this Form and it is empty, this will mean that there is no need to enter further information. This Form will be available after the deadline is over - from 7th-11th March 2016.

Calendar

ACTIVITY	DATE
Publishing the Calendar and Matrix on the Web page.	9th November 2015
Receiving Application Form from invited laboratories.	1st December-11th January 2016
Specific Protocol published on the Web site.	25th January 2016 at the latest
Deadline for receiving Laboratory scope: Form 0	25th January - 5th February 2016
Sample distribution.	8th February 2016
Deadline for receiving sample acceptance: Form 1	12th February 2016
Deadline for receiving results: Forms 2, 3 and 4	1st March 2016
Filling in Form 5	7th-11th March 2016
Preliminary Report: only results, no statistical treatment.	Last week of March 2016
Final Report distributed to the Laboratories.	December 2016

Cost of test item shipment.

EU-NRLs laboratories will be charged **200 €** for the shipment cost, for **EU/EFTA official laboratories** the amount will be **230 €**, and **250 €** for official laboratories from other countries. Regarding payment procedures - each laboratory can specify their details and invoice requests when applying for the test.

Please, do not pay for this EUPT until we send you the invoice.

Remember to include your Laboratory Code in the subject of the bank transfer.

Payment details are as follows:

BANK NAME: CAJAMAR - Caja Rural Sociedad Corporativa de Crédito

BANK ACCOUNT HOLDER: Universidad de Almeria

BANK ADDRESS: Office Number 990. Universidad de Almeria. Spain

ACCOUNT NUMBER: ES0730580130172731005000

SWIFT: CCRIES2A

REFERENCE GIVEN: Lab Code

Contact information

The official organising group details are as follows:

Universidad de Almería. Edificio Químicas CITE I
Ctra. Sacramento s/n
04120, La Cañada de San Urbano
Almería - Spain
Fax No.: +34 950015008

Organising team (e-mail and phone no.):

Dr. Amadeo R. Fernández-Alba	EURL-FV amadeo@ual.es	+34 950015034
Ms. Carmen Ferrer Amate	EURL-FV cferrer@ual.es	+34 950014102
Mr. Octavio Malato Rodríguez	EURL-FV omalato@ual.es	+34 950214423
Ms. Ana Lozano Fernández	EURL-FV analozano@ual.es	+34 950015645
Ms. Ana Uclés Moreno	EURL-FV aum085@ual.es	+34 950014102

Quality Control Group

Dr. Antonio Valverde, Senior Chemist, University of Almería, Spain

Mr. Stewart Reynolds, Senior Chemist, FERA, York, United Kingdom

Statistical Group

Dr. Carmelo Rodríguez, Senior Mathematician, University of Almeria, Spain

Advisory Group

Dr. Michelangelo Anastassiades, Senior Chemist, CVUA, Stuttgart, Germany.

Dr. Miguel Gamón, Senior Chemist, Laboratorio Agroalimentario, Valencia, Spain.

Dr. Philippe Gros, Senior Chemist, Laboratoire du SCL, Montpellier, France.

Dr. Magnus Jezussek, Senior Chemist, Erlangen, Germany.

Dr. André de Kok, Senior Chemist, NVWA, Wageningen, The Netherlands.

Mr. Ralf Lippold, Senior Chemist, CVUA, Freiburg, Germany.

Dr. Sonja Masselter, Senior Chemist, AGES, Innsbruck, Austria.

Mr. Finbarr O'Regan, Senior Chemist, Pesticide Control Laboratory, Celbridge, Ireland.

Dr. Tuija Pihlström, Senior Chemist NFA, Uppsala, Sweden.

Dr. Mette Erecius Poulsen, Senior Chemist, DTU, Copenhagen, Denmark.