EUPT-FV16 SPECIFIC PROTOCOL

European Union Proficiency Test for Pesticide Residues in Fruits and Vegetables (2014)

Introduction

This protocol is complementary to the General Protocol of EU Proficiency Tests (EUPT) for Pesticide Residues in Food and Feed (4th Edition, Approved: May 2013). This Proficiency Test is organised by the EURL for Pesticide Residues in Fruits and Vegetables covering Multiresidue Methods (MRM) of analysis.

Test Item

This proficiency test is based on the analysis of incurred pesticide residues in green pepper. The peppers were grown in a greenhouse located in the University of Almería facilities. 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 (–20ºC) prior to shipment to participants.

Two bottles, again chosen randomly, will be analysed over a period of time to confirm the stability of the pesticides in the test item (firstly, when the test items are shipped, then a few days after the receipt deadline for participants´ 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.

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

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 peppers.

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 cost **175 Euros** for EU and EFTA laboratories and **250 Euros** for any other participants. The payment procedure must have started before 24th February 2014. An e-mail showing the bank transfer confirmation, or similar, must have been sent beforehand; or may be requested at any time by the Organiser. **Payments without a Laboratory Code or Invoice Number 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 3rd – 24th February 2014, 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.

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: 28th February 2014, 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.

7. The participant laboratories must respect the deadline for submitting their results - 17th March 2014 - using **Form 2** – **Detected 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 24th – 28th March 2014. 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. It is possible that the laboratory, after receipt of the test item, performs further validations for some of the pesticides and then wants to report results for these pesticides. In that case, the laboratory will have to inform the organisers so they can include the new pesticides in their scope. This year again no residue definition needs to be followed so only individual contributions will be requested.

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 300 g of pepper test item treated with pesticides.
- Approximately 300 g of 'blank' pepper 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 24th February 2014. 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 28th February 2014. 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 28h 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

Detected 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 SANCO/12571/2013. 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' or <LOQ. 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.

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 17th March 2014. 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 peppers 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 24th – 28th March 2014.

Calendar

ACTIVITY	DATE
Publishing the Target Pesticide List, Calendar and Matrix on the Web page.	15th November 2013
Receiving Application Form from invited laboratories.	23rd Dec 2013 – 31st Jan 2014
Specific Protocol published on the Web site.	3rd Feb 2014 at the latest
Deadline for receiving Laboratory scope: Form 0	3rd-14th February 2014
Sample distribution.	24th February 2014
Deadline for receiving sample acceptance: Form 1	28th February 2014
Deadline for receiving results: Forms 2, 3 and 4	17th March 2014
Filling in Form 5	24th-28th March 2014
Preliminary Report: only results, no statistical treatment.	End of April 2014
Final Report distributed to the Laboratories.	November 2014

Cost of test item shipment.

EU and EFTA laboratories will be charged **175€** for the shipment cost. Other laboratories will be charged **250 €**. Regarding payment procedures - each laboratory can specify their details and invoice requests when applying for the test. Payment details are as follows:

BANK NAME: CAJAMAR - Caja Rural Sociedad Corporativa de Crédito

BANK ACCOUNT OWNER: Universidad de Almeria

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

ACCOUNT NUMBER: 30580130172731005000

IBAN: ES0730580130172731005000

SWIFT: CCRIES2A

CONCEPT: Invoice No. or Lab Code

Contact information

The official organising group details are as follows:

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