



EUPT-FV15 SPECIFIC PROTOCOL

European Union Proficiency Test for Pesticide Residues in Fruit and Vegetables (2013)

Introduction

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

Test material

This proficiency test is based on the pesticide residue analysis of potato. The potatoes were grown in Almería, Spain.

The pesticide treatments will be carried out post-harvest using either commercial formulations or using standard solutions in micro-spray solutions. The test material will be frozen (using liquid nitrogen), chopped, homogenised and sub-sampled into polyethylene bottles that have previously been coded.

Ten of these bottles containing the test material will be chosen randomly, and analysed to check for homogeneity.

The test material will be stored frozen (-20°C) 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 material (firstly, when the test materials 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 i.e. maintaining the sample at room temperature for a few days to see if there is any degradation of any of the pesticides present in the test material.

These results will not be included in the proficiency test's statistical analysis. The aim is solely to check pesticide stability during the shipping process and over the duration of the proficiency test.



All analytical determinations concerning the test material 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 8 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 website. 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 potatoes.

2. Laboratories will then 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 replying 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 **200 Euros** for any other participants. The payment procedure must have started before 21st January. 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. **Form 0 - Laboratory Scope** will be placed in the restricted area and will be open to participants from the 10th – 18th January 2013, prior to test material shipment. The aim is that laboratories provide information regarding their scope of analysis before receipt of the test material and detailed information regarding which pesticide is within the accredited scope of the lab and which is not.

5. When the participant laboratories receive the test material (and not before), they must enter the restricted area again and submit **Form 1 - Test Material Receipt** to inform the Organiser that they have accepted the test material. This Form has a deadline: 25th January 2013, which must be met. If no test material has been received



by this deadline, please contact the Organiser via e-mail (cferrer@ual.es, or omalato@ual.es)

6. The participant laboratories must respect the deadline for submitting their results - 13th February 2013 - using **Form 2 – Detected pesticides**; **Form 3 - Results** and **Form 4 - Methods** on-line.

7. One final form, **Form 5 - Additional Information Requested** can be filled in after the deadline has passed. This Form will be available from 20th – 27th February 2013. Not all laboratories will need to fill this in. It will depend upon information reported on previous Forms.

8. 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 onto the EURL-FV website 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 material, performs further validations for some of the pesticides and then reports results for these pesticides. Therefore, the information on this Form will be made available again for possible modification in Form 2. 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 Material

Participants will receive:

- Approximately 300 g of potato test material treated with pesticides.
- Approximately 300 g of 'blank' potato test material.

Shipment of Test Materials

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

The shipment of the test materials will be carried out over a one-week period from the 21st January 2013. 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 Material Handling

Once received, the test material should be stored deeply frozen (-18°C or less) prior to analysis thus avoiding any possible deterioration/spoilage. The test material 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 Material Receipt

Once the laboratory has received the test materials, their 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 material, and its acceptance. The deadline for acceptance (or non-acceptance) is 25th January 2013. If the laboratory does not respond by this date, the Organiser will assume that the test material has been received and accepted.

If any laboratory has not received the test material by 24th January, they must inform the Organiser **immediately** by e-mail (cferrer@ual.es and omalato@ual.es)

Submission of results:

Once the laboratory has analysed the test material and is ready to submit their data, they must enter their results at various steps on the 3 forms by accessing the restricted area in the EURL –FV website: <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. Those new pesticides sought should be indicated in this step.



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

The laboratory should mark the pesticides which have been detected twice given that these have been sought and then detected.

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

This form can be filled in at various stages - so once entered, the data will be saved, and you 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 guidelines provided in SANCO/12495/2011. Additional significant figures may be recorded for the purpose of statistical analysis. Please bear this in mind when reporting data:

- Residue levels < 0.010 mg/kg should be rounded to one significant figure
- Residue levels \geq 0.010 mg/kg and < 10 mg/kg should be rounded to two significant figures
- Residue levels \geq 10 mg/kg may be rounded to three significant figures or to a whole number.

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 you can go back to it and continue at any time before the final reporting deadline - which for all forms is 13th



February 2013. 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 potatoes are present in the test material.

False Negatives or Further Information – Form 5

This Form will be available only for those laboratories reporting that they sought a pesticide present in the test material 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 20th – 27th February 2013.

Calendar

ACTIVITY	DATE
Publishing the Target Pesticide List, Calendar and Matrix on the Webpage.	22nd October 2012
Receiving Application Form from invited laboratories.	3rd Dec 2012 - 7th Jan 2013
Specific Protocol published on the Website.	7th Jan 2013 at the latest
Deadline for receiving Laboratory scope: Form 0	10th-18th January 2013
Sample distribution.	21st January 2013
Deadline for receiving sample acceptance: Form 1	25th January 2013
Deadline for receiving results: Forms 2, 3 and 4	13th February 2013
Filling in Form 5	20th-27th February 2013
Preliminary Report: only results, no statistical treatment.	April 2013
Final Report distributed to the Laboratories.	December 2013

Cost of test material shipment.

EU and EFTA laboratories will be charged **175€** for the shipment cost. Other laboratories will be charged **200 €**. 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 HOLDER: Universidad de Almeria

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

ACCOUNT NUMBER: 30580130172731005000

IBAN: ES0730580130172731005000

SWIFT: CCRIES2A

REFERENCE GIVEN: Invoice No. or Lab Code

Contact information

The official organising group details are as follows:

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