



EUPT-FV-SM08 SPECIFIC PROTOCOL

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

Introduction

This protocol is complementary to the General Protocol for EU Proficiency Tests (EUPT) dealing with Pesticide Residues in Food and Feed. This Proficiency Test is organised by the EURL for Pesticide Residues in Fruits and Vegetables and covers the screening of pesticides using multiresidue methods of analysis.

The aim of this test is to evaluate laboratory capability when using large-scope quantitative and/or screening methods during routine analysis, for detecting and identifying unexpected pesticides at levels at, or above 0.01 mg/kg – included in and/or in addition to the laboratories' quantitative methods used for frequently detected pesticides. A second aim is to encourage official laboratories to extend the scope of their methods in a cost-effective way, by using the different MS instruments/software and methods available (whether old or new).

The evaluation of this PT will be based on qualitative information, although an **estimated concentration** will be requested for those pesticides that are detected, **only for informative purposes.**

Test item

This proficiency test is based on the pesticide-residue analysis of spinach.

The pesticide treatments will be carried out post-harvest using either commercial formulation in micro-spray solutions or using standard solutions. The test item 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 item will be chosen randomly and analysed to check for homogeneity.

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

Steps to follow

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

- To participate, each laboratory must complete the Application Form online, available on the EURL-FV Web page, before the deadline stipulated on the Calendar.
- 2. Laboratories will then receive an e-mail confirming their participation in this exercise, and assigning them a unique Laboratory Code. With this code, laboratories will be able to access the restricted area containing the reply 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 free of charge to those laboratories already participating in EUPT-FV18. For those who are not EUPT-FV18 participants, please see Cost for shipment of the test item for further details. Payments without a <u>Laboratory Code</u> or <u>Invoice Number to identify them will not be considered paid.</u>
- 4. When the participant laboratories receive the test item (and not before), they must enter the restricted area and submit **Form 0 Test Item Receipt** to inform the Organiser that they have accepted the test item. If no test item has been received by 12nd February, please contact the Organiser by e-mail (<u>cferrer@ual.es</u> and omalato@ual.es).



- The participating laboratories must respect the deadline for submitting the results.
 Results must be reported using Form 1 Results within 72 hours after the arrival of the test item.
- 6. The Organiser will evaluate the results at the end of the proficiency test, once the deadline for the receipt of results has passed. The Organiser will prepare a Preliminary Report that will be sent to the participants and uploaded to the website to show the pesticides reported, after the revision of all the data by the Scientific Panel a Final Report will be done and the organiser will upload an electronic version on the EURL-FV website and, afterwards, send a hard copy to each participant laboratory. This report will include information regarding the design of the test, the homogeneity and stability test results, an evaluation of the participant's results as well as graphical displays of the results and any conclusions. Any other relevant information considered of value may also be included.

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 (the same as for EUPT-FV18).

Shipment of Test Items

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

The shipment of the test items will start on 8th February 2016. 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 to receive the shipment, even if the laboratory is closed.

If your laboratory participates in the EUPT-FV-18 too, only one bottle of "blank" SPINACH homogenate will be shipped for both Proficiency Tests.

Advice on Test Item Handling

Once received, the test item should be stored deeply frozen (-18°C or less) prior to analysis to avoid 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.

Form 0 - Test Item Receipt

Once the laboratory has received the test item, the Organiser must be notified using Form 0 in the restricted area; filling in the date of receipt, the condition of the test item, and acknowledging its acceptance. If the laboratory does not inform the Organiser by 12^{nd} February 2016 (at the latest) via email (to <u>cferrer@ual.es</u> and <u>omalato@ual.es</u>), stating that no sample has been received, the Organiser will assume that the test item has been received and accepted.

Form 1 - Results

The evaluation of this PT will be based on qualitative information, although an estimated concentration will be requested for those pesticides that are detected, only for informative purposes.

It has been decided by the Quality Control Group, and based on the received questionnaires, that a target pesticide list will not be provided.

Laboratories must enter their results in Form 1 by accessing the restricted area on the EURL-FV web site: http://www.eurl-pesticides.eu

On this form, the laboratory should report the name of each of the pesticides detected. Each pesticide may be reported more than once if it has been detected by more than one method or identification criteria, as long as details of each method used are also provided.

Information on the parameters and/or criteria used for detecting and reporting the pesticides found will be requested, such as deviation from expected retention time, and MS identification details.

The idea is to ascertain if the methods are used in routinely or just specifically for this test and if the identification is undertaken manually or automatically. Moreover, the range over which your method operates will be required. This is the concentration range - the minimum and maximum level of your screening method that is used to detect pesticides.

Information in this form will be saved and may be changed or updated up to 72 hours after sample arrival in the laboratory. After the deadline, results submission and/or changes to the results form will no longer be possible.





Calendar

ACTIVITY	DATE	
Publishing the Calendar and Matrix on the Web page.	9 th December 2015	
Receiving Application Form from invited laboratories.	1st Dec-11th Jan 2016	
Specific Protocol published on the Web site.	25 th Jan 2016 at the latest	
Sample distribution.	8 th February 2016	
Deadline for receiving results: Fill in "Results Page"	72 hours after receiving the sample	
Preliminary Report	Last week of March 2016	
Final Report distributed to the Laboratories.	December 2016	

Cost for shipment of the test item

Only those laboratories not participating in EUPT-FV18 will have to pay the following fee for sample shipment: **all laboratories** will be charged **250 €**. For the payment procedures, each laboratory can specify their details and requests for invoices 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 OWNER: Universidad de Almeria

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

ACCOUNT NUMBER: 30580130172731005000

IBAN: ES0730580130172731005000

SWIFT: CCRIES2A

CONCEPT: INVOICE NUMBER

Contact information

The official organising group details are as follows:

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

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Quality Control Group

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

Statistical Group

Dr. Carmelo Rodriguez, 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.