EUPT-FV20 SPECIFIC PROTOCOL

European Union Proficiency Test for Pesticide Residues in Fruits and Vegetables (2018)

Introduction

This protocol is complementary to the General Protocol of EU Proficiency Tests (EUPT) for Pesticide Residues in Food and Feed (8th 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 in mind, 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 pesticide residues in **green beans with pods**. The green beans were grown in a greenhouse in Almería. The pesticide treatments carried out were pre-harvest using commercial formulations and post-harvest using analytical standards. 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 is 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 using three bottles more and reproducing the sample shipment to see if there is any degradation of any of the pesticides present in the test item.

Together with the test item, a blank sample of green beans homogenate will be sent to the participants. The blank material should not contain any of the pesticides in the Target Pesticides List, at or above, the specified MRRLs. If a pesticide is identified by the Organisers in the blank material and there is no possibility to find a different blank, that pesticide could be removed from the Target Pesticides List and it will be communicated to all the participants by e-mail.

Amount of Test Item

Participants will receive:

- Approximately 200 g of green beans test item treated with pesticides.
- Approximately 200 g of 'blank' green beans 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 5th March 2018. 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.

The Organisers will not take the responsibility for a parcel if it is retained at customs.

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). **IMPORTANT: Due to the weather conditions, the green beans used for the test item contained less water amount than the green beans used as blank material as they were harvested three weeks later and the temperature was high. For that reason, it is necessary to add 0.5 ml of water per**

gram of test item before the extraction (e.g. if 10 g are used for the extraction, 5 ml of water should be added to the test item) Water should only be added to the test item material, NOT TO THE BLANK MATERIAL.

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.

Subcontracting

All analytical determinations concerning the test item treatment analysis will be performed in a laboratory which is ISO 17025 accredited, and which has been previously evaluated by the Organisers.

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 mean (x*) according algorithm A is defined. For the calculation of the assigned value only results reported by EU and EFTA countries laboratories will be taken into account.

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 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 nine essential steps:

1. To participate, each laboratory must complete the Application Form on-line, whose link is 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 the Minimum Required Reporting Limits (MRRLs) are given. The MRRLs do not always correspond with the EU MRLs set for green beans.

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**.

3. The sample delivery will be **250 euros** for EU/EFTA participants and **350 euros** for participants 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. **Scope Form** will be placed in the restricted area and will be open to participants from the 22nd February - 5th March 2018, prior to test item shipment. The aim is that laboratories provide information regarding their scope of analysis before receipt of the test item. 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 the **Test Item Receipt Form** to inform the Organiser that they have accepted the test item. This Form has a deadline: 9th March 2018, 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 the test item receipt form is not filled in, the Organiser will consider that the participant has accepted the test item.

7. Once the laboratory has analysed the test item and is ready to submit their data, they must enter their results at various steps by accessing the restricted area in the EURL–FV web site. The participant laboratories must respect the deadline for submitting their results – 26th March 2018- using the **Identified Pesticides Form**, **Results Form** and the **Methods Form** on-line.

For each pesticide included in the laboratory scope, the Reporting Limit (RL) will be requested. The MRRL and the participant's own RL will be used to help identify and calculate z scores for false negative results. This form will also request information on which of the pesticides sought by the laboratory is within the laboratory's routine scope and whether it is accredited.

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

All concentrations must be expressed in mg/kg together with the recovery as a percentage. 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 'ND' (Not Detected).

The number of significant figures should be based on the procedures provided in SANTE/11813/2017. 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 will be recorded as 'ND'. If a pesticide was not sought, it will be recorded as 'NA' (Not Analysed). **If a laboratory fills in the scope form, but it does not report results neither fills in the methods form, all the pesticides will be considered as NA.**

The laboratory will also be asked to 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.

These forms 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 stage, always considering the deadline to submit results, which is 26th March 2018. 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 green beans are present in the test item.

8. One final form, **Additional Information Requested,** will be accessible after the deadline has passed. This Form will be available only for those laboratories that reported that they analysed a pesticide present in the test item but they did not detect it. 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 from 2nd-6th April 2018. 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 send all the participants the preliminary results, containing all the reported concentrations, in order to make sure that there was not any mistake from the Organisation side. When necessary, the Organiser will ask the participants by e-mail specific details about the methods of analysis used. After a stipulated deadline, the results will be evaluated and a preliminary report containing the preliminary assigned values and z scores will be sent to the participants. Finally, after evaluation by the Scientific Committee, the Final Report will be published online and a hard copy will be sent 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. Results submitted by non-EU/EFTA laboratories might not always be used in the tables or figures in the final report. Further relevant information considered to be of value may also be included.

Calendar

EUPT-FV20 CALENDAR	
Activity	**Date**
Registration period in EURL DataPool	20th December-5th February
Specific Protocol published on the Web site.	19th February 2018 at the latest
Sample distribution.	5th March 2018
Deadline for receiving sample acceptance	9th March 2018
Deadline for receiving results	26th March 2018
Filling in additional information, if necessary.	2nd -6th April 2018
Preliminary Results: only results, no statistical treatment.	April 2018
Preliminary Report (containing preliminary assigned values and z scores)	May 2018
Final Report distributed to the Laboratories.	August 2018

Cost of test item shipment.

EU/EFTA laboratories will be charged **250 €** for the shipment cost, for **non-EU/EFTA** laboratories the amount will be **350 €**. 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

Contact information

The official organising group details are as follows:

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Quality Control Group

Dr. Antonio Valverde, University of Almería, Spain

Dr. Paula Medina, European Food Safety Authority, Italy.

Advisory Group

Dr. Michelangelo Anastassiades, CVUA, Stuttgart, Germany.

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

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

Dr. Magnus Jezussek, Bavarian Health and Food Safety Authority, Erlangen, Germany.

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

Mr. Ralf Lippold, CVUA, Freiburg, Germany.

Dr. Sonja Masselter, AGES GmbH Institute for Food Safety, Innsbruck, Austria.

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

Dr. Tuija Pihlström, National Food Agency, Uppsala, Sweden.

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