

EUPT-FV26 SPECIFIC PROTOCOL

European Union Proficiency Test for Pesticide Residues in Fruits and Vegetables (2024)

Introduction

This protocol is complementary to the General Protocol of EU Proficiency Tests (EUPT) for Pesticide Residues in Food and Feed (11th 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 (0.001 - 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.

Proficiency testing Item

This proficiency test is based on the analysis of pesticide residues in **banana**. Organic bananas were purchased from a specialised organic market in Almería. The pesticide treatments carried out were all post-harvest using analytical standards. Bananas were milled with 1 % of ascorbic acid in order to prevent oxidation. They were spiked with the pesticides analytical standards, homogenised and packed in plastic bags. Once frozen, the material was milled again, 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.

A minimum of 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 PT item. If the sample shipment of EU/EFTA labs takes more than 48 hours, three extra bottles will be analysed each day of delay, studying this way the stability of the samples that took longer to arrive to an EU/EFTA laboratory.

All analytical determinations concerning the PT item treatment analysis will be performed in a laboratory which is ISO 17025 accredited, in this case, the EURL-FV laboratory.

Blank material will not be distributed to the participants.

Amount of Test Item

Participants will receive:

- Approximately 200 g of banana test item spiked with pesticides.

Shipment of PT Item

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

The shipment of the PT items will be carried out over a one-week period from the 26th February 2024. 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 PT Item Handling

Once received, the PT 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.

Target List

Participants will be provided with two target pesticide lists, one with pesticides that have to be analysed on a compulsory basis, and a second one with pesticides to be analysed voluntarily. 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, 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 evaluated 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, which can be found on the EURL-FV Web page, before the deadline stipulated on the Calendar. It is recommended that laboratories download the Target Pesticide Lists from this web site. Laboratories should carefully read the Target Pesticide Lists, where the Minimum Required Reporting Limits (MRRLs) are given. The MRRLs do not always correspond with the EU MRLs set for banana.
2. The participation fee will be **350 euros** for EU/EFTA participants and **450 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 the invoice number identifying them will not be considered as paid.**



3. Any communication with the Organisation should be made using a **Contact Form** placed in the restricted area, or by e-mail (cferrer@ual.es).
4. Laboratories will be assigned a user name and password for the restricted area of submission of results.
5. **Scope Form** will be placed in the restricted area and will be open to participants from the 13th – 26th February 2024, prior to PT item shipment. The aim is that laboratories provide information regarding their scope of analysis before receipt of the test item. As default, all compounds of the mandatory target list are selected and all compounds of the voluntary target list are deselected, and the MRRL is listed in the scope. Laboratories will be asked to indicate the compounds they have in their PT scope and insert their Reporting Limits for each pesticide. If a laboratory does not select their scope, the default values will be considered for its evaluation.
6. When the participant laboratories receive the PT item (and not before), they must enter the restricted area again and submit the **PT Item Receipt Form** to inform the Organiser that they have accepted the test item. If no PT item has been received by 1st March 2024, the laboratories should contact the Organiser. If the test item receipt form is not filled in, the Organiser will consider that the participant has accepted the PT 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 EUPT webtool. The participant laboratories must respect the deadline for submitting their results – 21st March 2024 (23:30 pm at the latest) - using the tabs **Detected, Edit results and Edit Methods** on-line.

For each pesticide included in the laboratory scope, the Reporting Limit (RL) will be requested. 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.

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:

- Two, for residue levels <0.010 mg/kg (e.g. 0.0086 mg/kg).
- Three, for residue levels ≥ 0.010 mg/kg (e.g. 0.0673, 0.245, 1.32, 10.1 mg/kg).



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, their results will be: "No results reported"**.

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.

When all fields are filled out, laboratories must accept and submit their final results by clicking the check box and then click on Final submission, before 21st March 2024 (23:45 pm at the latest).

IMPORTANT: After the final submission it will NOT be possible to edit the results.

Participants will receive an email confirming the submission of their results, and with an attached excel file with their submitted data.

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

8. One final tab, **Additional Info**, will be accessible after the deadline for submission of results has passed. In this Form it will be possible to submit the method information of false negative results. The deadline for this form will be 12th April 2024. 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. When necessary, the Organiser will ask the participants by e-mail specific details about the methods of analysis used. 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 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

Registration period	15 th December 2023 - 30 th January 2024
Specific Protocol published on the Web site.	12 th February 2024 at the latest
Selection of the scope	13 th – 26 th February 2024
Sample distribution.	26 th February 2024
Deadline for receiving sample acceptance	1 st March 2024
Deadline for receiving results	21 st March 2024 23:30 pm
Filling in additional information, if necessary.	22 nd March– 1 st April 2024
Preliminary Report: (containing preliminary assigned values and z scores)	April 2024
Final Report distributed to the Laboratories.	September 2024

Cost of PT item shipment.

EU/EFTA laboratories will be charged **350 €** for the shipment cost, for **non-EU/EFTA** laboratories the amount will be **450 €**. 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 you receive the invoice.

Remember to include your Invoice number 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:

Universidad de Almería. Edificio Químicas CITE I
 Ctra. Sacramento s/n
 04120, La Cañada de San Urbano
 Almería - Spain

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

Carmen Ferrer Amate	EURL-FV cferrer@ual.es	+34 950214102
Octavio Malato Rodríguez	EURL-FV omalato@ual.es	+34 950214423
María Murcia Morales	EURL-FV mmm371@ual.es@ual.es	+34 950215645
Amadeo R. Fernández-Alba	EURL-FV amadeo@ual.es	+34 950015034

Quality Control Group

- Antonio Valverde, University of Almería, Spain
- Paula Medina, European Food Safety Authority, Italy.

Advisory Group

- Michelangelo Anastassiades, EURL-SRM, CVUA Stuttgart, Fellbach, Germany.
- Björn Hardebusch, EURL-AO, CVUA Freiburg, Germany.
- Magnus Jezussek, LGL, Erlangen, Germany.
- André de Kok, Formerly Wageningen Food Safety Research, Wageningen, The Netherlands.
- Marine Lambert, French Agency for Food, Environmental and Occupational Health & Safety (ANSES), France.
- Ralf Lippold, EURL-AO, CVUA Freiburg, Germany.
- Hans Mol, Wageningen Food Safety Research, Wageningen, The Netherlands.
- Finbarr O'Regan, Pesticide Registration Division, DAFM, Kildare, Ireland.
- Patrizia Pelosi, Istituto Superiore di Sanità, Rome, Italy.
- Tuija Pihlström, National Food Agency, Uppsala, Sweden.
- Mette Erecius Poulsen, EURL-CF, National Food Institute (DTU), Lyngby, Denmark.
- Radim Štěpán, Czech Agriculture and Food Inspection Authority Inspectorate, Prague, Czech Republic.
- Hermann Unterluggauer, AGES, Institute for Food Safety, Innsbruck, Austria.