



# EUPF-FV13 SPECIFIC PROTOCOL

## For European Union Proficiency Test for Pesticide Residues in Fruits and Vegetables (2011)

### Introduction

This protocol is complementary to the General protocol for EU Proficiency Tests (EUPF) for Pesticide Residues in Food and Feed. This Proficiency Test is organised by the EU-RL<sup>1</sup> for Pesticide Residues in Fruits and Vegetables and covers Multiresidue Methods (MRM) of analysis.

### Test material

This proficiency test is based on the pesticide residues analysis of mandarins. The mandarins were grown in Málaga, Spain.

The pesticide treatments will be carried out post-harvest using either commercial formulations in micro-spray solutions or using standard 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, and then a few days after the deadline for receipt of participants' results).

There will be an extra analysis during this period reproducing the sample shipment; this is, 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.

---

<sup>1</sup> By the Treaty of Lisbon approved on the 1<sup>st</sup> of December 2009, the Community Union becomes the European Union.



**Fruits and Vegetables**

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.

All the analytical determinations concerning the test material treatment analysis will be performed in a laboratory accredited to ISO 17025.

**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 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. Labs should take note that the pesticide residue definitions within this PT do not always follow Regulation 396/2005. Also, the MRRLs do not always correspond with the EU MRLs set for mandarin.
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 **150 Euros**. The payment procedure must have started before the 24<sup>th</sup> January. An e-mail showing the bank transfer confirmation, or similar, must have been sent beforehand. **Payments without a Laboratory Code or Invoice Number to identify them will not be considered as paid.**
4. This year, a new **Form 0** will be placed in the restricted area named **Laboratory Scope**. It will be open to participants from the 10<sup>th</sup> – 19<sup>th</sup> January 2011, prior to test material shipment. The aim is that laboratories provide information regarding their scope of analysis before receipt of the test material.

**Fruits and Vegetables**

5. When the participant laboratories receive the test material (and not before), they must enter the restricted area and submit the **Test Material Receipt Form 1** on-line to inform the Organiser that they have accepted the test material. This Form has a deadline: 28<sup>th</sup> January 2011, that must be met. If no test material has been received by this deadline, please contact the Organiser by e-mail ([pmedina@ual.es](mailto:pmedina@ual.es) or [omalato@ual.es](mailto:omalato@ual.es))

6. The participant laboratories must respect the deadline for submitting the results - 18<sup>th</sup> February 2011 - using the '**Detected for**' (**Form 2**), '**Results**' (**Form 3**) and '**Methods Form**' (**Form 4**) on-line.

7. One last **Form 5 'Additional Information'** must be filled in after the deadline is over. This Form will be available from 21<sup>st</sup> – 25<sup>th</sup> February 2011. Not all the laboratories may need to fill this in. It will depend upon the 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 send a hard copy of the Final Report to each participant laboratory, and before this, an electronic version will be uploaded on the EURL-FV web site. 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. Any other relevant information considered of value may also be included.

**Laboratory Scope – Form 0**

Before the participant laboratories receive the sample, the restricted area will be open so their laboratory scopes can be recorded. A Form 0 will need to be filled in, to ascertain which of the pesticides in the Target Pesticide List were actually sought. It could happen that the laboratory, after receipt 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.

**Analytical parameters**

The test material will contain several pesticides from the Target Pesticide List. Laboratories should carefully read through the Target Pesticide List, where important information about the reporting of the results, as well as the Minimum Required



## Fruits and Vegetables

Reporting Levels (MRRLs), is given. Where the residue definition in the Target Pesticide List, includes more than one component, the results for the individual components, as well as the respective sum of components, calculated as stated in the residue definition, are to be reported.

For each pesticide included in the residue definitions, MRRL values have been set. The MRRL and the participants own Reporting Levels will be used to help to identify false negative results.

### Amount of Test Material

Participants will receive:

- Approximately 300 g of mandarin test material treated with pesticides
- Approximately 300 g of 'blank' mandarin 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 24<sup>th</sup> January 2011. The Organiser will try to ensure that all the packages arrive on the same day in 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 to receive the shipment, even if the laboratory is closed.

### Advice on Test Material Handling

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

### Test Material Receipt – Form 1

Once the laboratory has received the test materials it must be reported to the organiser using Form 1 in the restricted area by filling in the date of receipt, the condition of the test material, and its acceptance. The deadline for acceptance, or not, is the 28<sup>th</sup>

**Fruits and Vegetables**

January 2011. If the laboratory does not respond by this deadline the organiser will assume that the test material has been received and accepted.

If any laboratory has not received the test material by 28<sup>th</sup> January, they must inform the Organiser **immediately** by e-mail ([pmedina@ual.es](mailto:pmedina@ual.es) and [omalato@ual.es](mailto: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 in 3 different steps on 3 forms by accessing the restricted area in the EURL –FV web site: <http://www.crl-pesticides.eu>

**Detected Pesticides – Form 2**

In this step, the laboratory should indicate the pesticides that have been detected. The information entered in Form 0 – Laboratory Scope, will be made available in this Form. Those new pesticides sought should be indicated in this step, together with the reporting level in mg/kg. This field will not be obligatory for those pesticides that where the MRL definition is the sum of two or more components. The laboratory must save every page of results that are entered. This form can be filled in at different stages, so once entered the data will be safe, and you can add further data at a later date.

**Results – Form 3**

In this step, the laboratory should report the concentrations measured for each determination. Results are requested to be reported in three different ways, one without correction for recovery, a second after correction for recovery and the third one, as you would normally report in routine analyses, in other words in the way you would routinely report to your customer(s). All concentrations must be expressed in mg/kg together with the percentage recovery.

The numbers of significant figures should be based on the guidelines provided in SANCO/10684/2009. Additional significant figures may be recorded for the purpose of statistical analysis. So bare this in mind when reporting data:

- Residue levels <0.010 mg/kg - to be expressed to two significant figures (e.g. 0.0058 mg/kg)
- Residue levels  $\geq$  0.010 mg/kg - to be expressed to three significant figures (e.g. 0.0792, 0.156, 1.64, 10.3 mg/kg)

Results should not be reported where a pesticide was not detected or was detected below the RL (Reporting Limit) of the laboratory. In both cases, it should be recorded as



**Fruits and Vegetables**

'ND' or <RL. 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 with all the pesticides detected in the sample will show-up 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.

On the Reference Method field a published reference is needed for the method used. Use either one from the list or provide details of your own. It is not necessary to give internal country references as this will be of little value in the final report.

Again in this form, information must always be saved so that you can come back to it and continue at any time before the final reporting deadline, which for all the forms is the 18<sup>th</sup> February 2011. Any results reported after the deadline will not be included in the statistical treatment, or in the final report.

Before entering the results please read carefully the Target Pesticide List to be aware of the residue definitions requested. For pesticides where the residue definition is the sum of a parent pesticide and other components, results for both the sum and the individual components must be reported.

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

**False Negatives or Further Information – Form 5**

This Form will be available only for laboratories that have reported that they sought a pesticide present in the test material but no method has been reported in Form 4. If the laboratory access 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 the 21<sup>st</sup> – 25<sup>th</sup> February 2011.

**Calendar**



ACTIVITY	DATE
- Publication of the Target Pesticide List, Calendar and Matrix on the Web page	October 2010
- Receiving Application Form from invited laboratories.	1 <sup>st</sup> – 30 <sup>th</sup> November 2010
- Specific Protocol published on the Web site	3 <sup>rd</sup> January 2011 at the latest
- Receiving Form 0 – Laboratory Scope from participants	10 <sup>th</sup> – 19 <sup>th</sup> January 2011
- Test Material distribution.	24 <sup>th</sup> January 2011
- Deadline for Test Material acceptance: Form 1	28 <sup>th</sup> January 2011
- Deadline for receipt of results: Form 2, Form 3 and Form 4	18 <sup>th</sup> February 2011
- Filling in Form 5: additional information	21 <sup>st</sup> – 25 <sup>th</sup> February 2011
- Preliminary Report: only results, no statistical treatment.	March 2011

#### Cost for shipment of the test material

All laboratories will be charged **150€** for the cost of shipment. For the payment procedures, each laboratory can specify their details and requests for invoices 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:

Universidad de Almería. Edificio Químicas CITE I  
Ctra. Sacramento s/n



04120 Almería - Spain

Fax No.: +34 950015483

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

Amadeo R. Fernández-Alba EURL-FV amadeo@ual.es +34 950015034

Paula Medina Pastor EURL-FV pmedina@ual.es +34 950014102

Octavio Malato Rodríguez EURL-FV omalato@ual.es +34 950015531

**Quality Control Group**

Dr. Antonio Valverde, University of Almería, Spain

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

**Statistical Group**

Dr. Carmelo Rodriguez, senior Mathematician, University of Almeria, Spain

**Advisory Group**

Dr. André de Kok, senior Chemist, VWA, Amsterdam, The Netherlands

Dr. Metter Erecius Poulsen, senior Chemist, NFI, Copenhagen, Denmark

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

Dr. Tuija Pihlström, senior Chemist NFA, Uppsala, Sweden

Dr. Sonja Masselter, senior Chemist, AGES, Innsbruck, Austria

Dr. Michelangelo Anastassiades, senior Chemist, CVUA, Stuttgart, Germany

Dr. Ralf Lippold, senior Chemist, CVUA, Freiburg, Germany