

# SPECIFIC PROTOCOL

## for the EU Proficiency Test for Pesticide Residues in Cereals/Feeding stuff using Multi-Residue Methods, EUPT-CF9 (2015)

(last updated: 23 April 2015)

### Introduction

This protocol is complementary to the [General Protocol for EU Proficiency Tests for Pesticide Residues in Food and Feed](#). The current proficiency test covers pesticides that are determined by Multi-Residue Methods. This EUPT is to be performed by all National Reference Laboratories for Cereals and/or Feeding stuffs (NRL-CFs) as well as by all official EU laboratories (OfLs) responsible for official pesticide residue controls on cereals and/or feeding stuff, as far as their scope overlaps with that of the EUPT-CF9.

### Test Item (Test Material)

This proficiency test concerns the analysis of pesticide residues in maize flour. The maize was grown in Denmark in 2014 and pesticides were applied in the field. Following harvest, the maize kernels were spiked with additional pesticides.

The blank Test Item provided, can be used for recovery experiments as well as for the preparation of matrix-matched calibration standards. However, the blank Test Item must also be analysed and possible detected pesticides reported.

The Organizers will check the Test Items for sufficient homogeneity and for stability at conditions reproducing sample shipment and storage during the duration of the test. The blank Test Item will also be checked to prove that the target analytes are not contained at any relevant levels. All these tests will be conducted by the EURL-CF, which is ISO 17025 accredited.

### Analytical Parameters

The Test Item contains several pesticides from the Target Pesticides List.

Laboratories should carefully read the Target Pesticides List, where important information about reporting of results, as well as the Minimum Required Reporting Levels (MRRLs), is given. The Target Pesticides List contains only individual compounds, and results should only be reported for individual compounds, no matter how the residue definitions have been set.

The MRRL values will be used to help identify false positive and false negative results and for the calculation of z-scores for false negatives.

### **Amount of Test Item**

The participants will receive:

- approximately 125 g of maize flour Test Item with incurred and spiked pesticides and
- approximately 125 g of blank maize flour Test Item.

### **Shipment of Test Items**

The Test Items are planned to be shipped on 20 April, 2015.

Test Items will be shipped frozen and packed in thermo-boxes together with a freezer block. The organisers will aim to ensure that all participating laboratories will receive their shipments on the same day. Prior to shipment a reminder will be sent to the participating laboratories by e-mail.

Laboratories must make their own arrangements for the receipt of the package. They should inform the Organiser of any public holidays in their country/city during the week of the shipment, and must make the necessary arrangements to receive the shipment, even if the laboratory is closed.

### **Instructions on Test Item Handling**

Once received, the Test Items should be stored deep-frozen (at  $-18^{\circ}\text{C}$  or less) before analysis to avoid any possible deterioration/spoilage and to minimize pesticide losses. **The Test Items 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 purposes. Considering the available amount of Test Items, laboratories employing methods requiring large analytical portions are advised to scale them down.

The homogeneity tests will be conducted using 5 g of Test Item in all cases. As sub-sampling variability increases with decreasing analytical portion size, sufficient homogeneity can only be guaranteed where participants employ sample portions that are equal or larger than the ones stated above.

### **Results Submission Website and Deadlines**

Sample receipt acknowledgement, analytical results and method information are to be submitted via the [EUPT-CF9 Result Submission Website](http://www.crl-pesticides.eu/docs/public/tmpl/article.asp?LabID=400&CntID=961&Theme_ID=1&Pdf=False&Lang=EN). Links to this can be found on the EURL-CF webpage: [http://www.crl-pesticides.eu/docs/public/tmpl/article.asp?LabID=400&CntID=961&Theme\\_ID=1&Pdf=False&Lang=EN](http://www.crl-pesticides.eu/docs/public/tmpl/article.asp?LabID=400&CntID=961&Theme_ID=1&Pdf=False&Lang=EN).

The website will be accessible from 21 April 2015. The webpage contains a link to specific instructions on how to enter the data in the result submission website.

To access the data submission forms, participants must use their unique login data (username and password). **Username and password will be email to the participants on 17 April.**

To access the data submission forms participants must use their unique login data (username and password), which will be sent by e-mail on 17 April 2015.

The labs can fill in the sub-pages at different stages/sessions. Remember to save the data of each page before leaving it.

**The deadline for result submission is 19 May 2015 at 13.00 CEST.**

#### Test Item Receipt and Acceptance - Subpage 0

Once the laboratory has received the Test Items it must report to the organiser, via the [EUPT-CF9 Result Submission Website](#), the date of receipt, the condition of the Test Item, and its acceptance. The deadline for acceptance is the 24 April 2015. If the laboratory does not respond by this deadline, the Organisers will assume that the Test Items have been received and accepted.

**If participants have not received the Test Items by the 24 April 2015 at noon, they must inform the Organiser immediately by e-mail ([eur1-cf@food.dtu.dk](mailto:eur1-cf@food.dtu.dk)).**

#### Reporting Qualitative and Quantitative Results - Subpages 1 and 2

To report their results, laboratories must access the [EUPT-CF9 Result Submission Website](#).

Deadline: All results must be reported on the online result submission website by 19 May 2015 at 13.00 CEST. The website will NOT be accessible for result submission after this date and time, and any results reported after the deadline will not be included in the statistical treatment, or in the final report.

Results should **not** be reported where a pesticide

- a) was not detected,
- b) was detected below the RL (Reporting Limit) of the laboratory, or
- c) below the MRRL.

Results reported as <RL will be considered as „Not Detected“.

Significant Figures:

Residue levels <0.010 mg/kg;

- to be expressed by two significant figures (e.g. 0.0058 mg/kg).

Residue levels  $\geq$  0.010 mg/kg;

- to be expressed by three significant figures, e.g. 0.156, 1.64, 10.3 mg/kg.

The following fields will be available for reporting the quantitative results:

- **“Concentration in mg/kg”**: here you should fill in the results that you would report in your routine work. That means, the recovery-corrected result should be reported, if it reflects the normal procedure in your lab, otherwise the non-recovery-corrected result should be reported.
- **“Conc. in blank in mg/kg”**: any concentration values of pesticides from the Target Pesticides List you will determine in the blank (even at levels below the MRRL), you can enter here.
- **“Experience with this compound”**. Use the dropdown-menu to indicate how many years you have analysed for this compound using the method applied in this EUPT.
- **“Is your result recovery-corrected?”**: Please specify whether the result was recovery-corrected and what kind of recovery-correction via the dropdown-menu.
- **“Recovery figure (in %)”**: Here, labs can report any recovery figures (in %) obtained for the analyte in question. If a recovery factor was used to correct the result, the recovery figure (in %) used for the calculation **MUST** be reported.

Additional information on how each recovery figure was derived will be asked in separate fields.

#### Reporting Information on Analytical Methodology - Subpage 3

All laboratories are requested to provide information on the analytical method(s) they have used via the [EUPT-CF9 Result Submission Website](#). The laboratories are asked to thoroughly fill in this important information in order to minimize the administrative burden of collecting this information at a later stage.

#### Reporting missing information after result submission deadline – Subpage 4

In case of false negative results the affected laboratories will be asked to provide details on the methodology used after the deadline for result submission. This can be done by accessing subpage 4 within the [EUPT-CF9 Result Submission Website](#). This subpage will be accessible from 25-27 May 2015.

**If no sufficient information on the methodology used is provided, the Organiser reserves the right not to accept the analytical results reported by the participant.**

#### **Follow-up actions**

According to Art. 32 1b of Regulation (EC) No 882/2004, underperformance of any NRL-CF in comparative testing will be followed by EURL-CF.

## Documents

All documents relating to EUPT-CF9 can be found in the EURL-Document Repository ([CIRCA/FIS-VL](#)).  
Links to the documents can be found in the [EUPT-CF9 Website](#).

## Calendar

Activity	Dates
Announcement Calendar Target Pesticide List	January 2015
EUPT-Registration Website	9 February 2015
Deadline for registration	20 March 2015
Release of Specific Protocol	7 April 2015
Distribution of Test items	20 April 2015
Deadline for Receipt and Acceptance of Test Materials	within 24 hr on receipt
Deadline for Result Submission	19 May 2015 at 13.00 CET
Deadline for submission of additional method information for false negative results	26 May 2015
Preliminary Report (only compilation of results)	26 June 2015
EUPT Evaluation Meeting	July 2015
Final Report	December 2015

## Participation Fees

For participating laboratories from the EU, EU-candidate states and EFTA states the participation fee will be

- 175 €

The participation fees for laboratories from third countries:

- 350 €

For further information, visit this website [www.eurl-pesticides.eu](http://www.eurl-pesticides.eu)

### **Delays in Payment**

The participants will receive an invoice from DTU. The invoice will be sent by ordinary mail. The terms of payment are 30 days net. After this deadline reminders will be sent. From the second reminder onwards an administration fee of DKK 100.00 excluding VAT (ca. 13 €) will be charged per reminder.

Any questions concerning invoices must be directed to Peter Dahm-Jappe at the financial department [pdahm@adm.dtu.dk](mailto:pdahm@adm.dtu.dk).

Contact information:

DTU Food  
National Food Institute



## Mette Erecius Poulsen

Head of EURL Cereals and Feeding stuff

National Food Institute  
Technical University of Denmark  
Moerkhoej Bygade 19  
DK-2860 Soeborg  
Phone: +45-3588-7463  
Fax: +45-3588-7448  
E-Mail: eurl-cf@food.dtu.dk  
<http://www.eurl-pesticides.eu>

### Organising Team:

Merete B. Ludwigsen, Technician	EURL for Cereals and Feeding stuff
Susan Strange Herrmann, Chemist	EURL for Cereals and Feeding stuff
Lisbet Pilhøj, Technician	EURL for Cereals and Feeding stuff
Gitte Andersen, Chemist	EURL for Cereals and Feeding stuff
Jens-Ole Frimann, System Developer	EURL for Cereals and Feeding stuff

### Quality Control Group:

Prof. Antonio Valverde	University of Almería, Spain
Stewart Reynolds, Senior Chemist	Food and Environmental Research Agency, York, United Kingdom

### Advisory Group

Prof. Amadeo R. Fernández-Alba	University of Almeria, Spain
Dr. Miguel Gamón, Senior Chemist	Pesticide Residue Laboratory of the Generalitat Valenciana, Valencia, Spain
Dr. André de Kok, Senior Chemist	Food and Consumer Product Safety Authority (VWA), Amsterdam, The Netherlands
Ralf Lippold, Senior Chemist	Chemisches und Veterinäruntersuchungsamt (CVUA) Freiburg, Germany
Dr. Michelangelo Anastassiades	Chemisches und Veterinäruntersuchungsamt (CVUA) Stuttgart, Germany
Dr. Sonja Masselter, Senior Chemist	AGES Competence Center for Residues of Plant Protection Products, Innsbruck, Austria
Dr. Tuija Pihlström, Senior Chemist	National Food Administration, Uppsala, Sweden
Dr. Magnus Jezussek	Bavarian Authority of Health and Food Safety, Erlangen, Germany
Dr. Darinka Štajnbaher	National Laboratory for Health Environment and food (NLZOH), Slovenia
Philippe Gros	SCL, Montpellier
Finbarr o'Regan	Pesticide Control Laboratory, Department of Agriculture, Fisheries and Food, Kildare