

# SPECIFIC PROTOCOL

## for the EU Proficiency Test for Pesticide Residues in Cereals/Feeding stuff using Multi Residue Methods, EUPT-CF11 (2017)

(last updated: 10 March 2017)

### 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-CF11.

### Test Item (Test Material)

This proficiency test concerns the analysis of pesticide residues in oat flour. The oat has been grown in Denmark and pesticides were applied in the field. Following harvest, the oat 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 (MRRs), 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 100 g of oat flour Test Item with incurred and spiked pesticides and
- approximately 100 g of blank oat flour Test Item.

### **Shipment of Test Items**

The Test Items are planned to be shipped on 8 May, 2017.

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°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 to 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-CF11 Result Submission Website](#) .

Relevant links and documents can be found on the EURL-CF webpage: [EUPT-CF11 Website](#) .

The Result Submission Website will be accessible from 9 May 2017. 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 are included in the registration confirmation mail.**

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 6 June 2017 at 24.00 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-CF11 Result Submission Website](#), the date of receipt, the condition of the Test Item, and its acceptance. The deadline for acceptance is the 11 May 2017. 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 11 May 2017 at noon, they must inform the Organiser immediately by e-mail to [eurl-cf@food.dtu.dk](mailto:eurl-cf@food.dtu.dk).**

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

To report their results, laboratories must access the [EUPT-CF11 Result Submission Website](#).

Deadline: All results must be reported on the online result submission website by 06 June 2017 at 24.00 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

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 ≥ 0.010 mg/kg;

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

### 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-CF11 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 of supplementary information in case of false negative results – 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 on the [EUPT-CF11 Result Submission Website](#). This subpage will be accessible from 7-9 June 2017.

**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**

In accordance with 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 related to EUPT-CF11 can be found on [EUPT-CF11 Website](#).

## Calendar

Activity	Dates
Announcement Calendar Target Pesticide List	February 2017
EUPT-Registration Website open	13 March 2017
Deadline for registration	23 April 2017
Specific Protocol published	24 April 2017
Distribution of Test items	8 May 2017
Deadline for receipt and acceptance of Test Materials	within 24 hr on receipt
Deadline for Result Submission	6 June 2017 at 24.00 CEST
Deadline for submission of additional method information for false negative results	9 June 2017 at 13.00 CEST
Preliminary Report (only compilation of results) published	3 August 2017
Final Report published	December 2017

## Participation Fees

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

- 200 €

The participation fees for laboratories from third countries will be:

- 350 €

For further information, visit [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 Skovgaard Christensen, [pschr@adm.dtu.dk](mailto:pschr@adm.dtu.dk) at the financial department of DTU.

Contact information:

DTU Food  
National Food Institute



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### Organising Team:

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

### Quality Control Group:

Dr. Antonio Valverde	University of Almería, Spain
Dr. Paula Medina	European Food Safety Agency, Italy

### Advisory Group

Prof. Amadeo R. Fernández-Alba	University of Almeria, Spain
Dr. Miguel Gamón	Pesticide Residue Laboratory of the Generalitat Valenciana, Valencia, Spain
Dr. André de Kok	Food and Consumer Product Safety Authority (NVWA), Wageningen, The Netherlands
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