

# SPECIFIC PROTOCOL

# for the EU Proficiency Test for Pesticide Residues in Cereals/Feeding stuff using Multi Residue Methods, EUPT-CF12 (2018)

(last updated: 22 January 2018)

#### Introduction

This protocol is complementary to the <u>General Protocol for EU Proficiency Tests for Pesticide Residues in Food and Feed</u>. 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-CF12.

#### **Test Item (Test Material)**

This proficiency test concerns the analysis of pesticide residues in hay flour. The hay has been grown in Denmark and pesticides were applied in the field.

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 Organiser 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 20 g of hay flour Test Item with incurred pesticides and
- approximately 50 g of blank hay flour Test Item.

# **Shipment of Test Items**

The Test Items are planned to be shipped on 29 January, 2018.

Test Items will be shipped frozen and packed in thermo-boxes together with a freezer block. The organiser 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 below) 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. Because the hay matrix will swell up when water is added, only 1g samples should be used for extraction. The MRRL are adjusted to this.

The homogeneity test is conducted using 1 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**

The analytical scope should be selected prior to the shipment of the samples. This can be done via the **EUPT-CF12 Result Submission Website**. The scope selection subpage will be open from 16-26 January 2018

Sample receipt acknowledgement, analytical results and method information are to be submitted via the **EUPT-CF12 Result Submission Website**.

Relevant links and documents can be found on the EURL-CF webpage: **EUPT-CF12 Website**.

The Result Submission Website will be accessible from 30 January 2018. 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) send to the laboratory via email.

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 1 March 2018 at 24.00 CET.

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

Once the laboratory has received the Test Items it must report to the organiser, via the <u>EUPT-CF12</u> <u>Result Submission Website</u>, the date of receipt, the condition of the Test Item, and its acceptance. The deadline for acceptance is the 2 February 2018. If the laboratory does not respond by this deadline, the Organiser will assume that the Test Items have been received and accepted.

If participants have not received the Test Items by the 2 February 2018 at noon, they must inform the Organiser immediately by e-mail to <a href="mailto-eurl-cf@food.dtu.dk">eurl-cf@food.dtu.dk</a>.

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

To report their results, laboratories must access the EUPT-CF12 Result Submission Website.

<u>Deadline</u>: All results must be reported on the online result submission website by 1 March 2018 at 24.00 CET. 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  $\geq$  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-CF12 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-CF12 Result Submission Website**. This subpage will be accessible from 5 March 2018.

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-CF12 can be found on EUPT-CF12 Website.

#### Calendar

Activity	Dates
Announcement Calendar Target Pesticide List	November 2017
EUPT-Registration Website open	5 December 2017
Deadline for registration	12 January 2018
Specific Protocol published	15 January 2018
Website for selecting pesticide scope open	15 January 2018
Website for selecting pesticide scope closed	26 January 2018
Distribution of Test items	29 January 2018
Deadline for receipt and acceptance of Test Materials	within 24 hr on receipt
Deadline for Result Submission	1 March 2018
	at 24.00 CET
Deadline for submission of additional method information for	5 March 2018
false negative results	at 24.00 CET
Preliminary Report (only compilation of results) published	30 April 2018
Final Report published	December 2018

# **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 <u>www.eurl-pesticides.eu</u>.

# **Delays in Payment**

The participants will receive an invoice from DTU. 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 Miche Micereme Esati, <a href="mmes@adm.dtu.dk">mmes@adm.dtu.dk</a> at the financial department of DTU.

#### Contact information:







# **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:

Susan Strange Herrmann, Chemist	EURL for Cereals and Feeding stuff
Merete B. Ludwigsen, Technician	EURL for Cereals and Feeding stuff
Lisbet Pilhkjær, Technician	EURL for Cereals and Feeding stuff
Ban M. Kadhum, 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**

raviou, c. cup	
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), Wargeningen, The Netherlands
Mr. Ralf Lippold	Chemisches und Veterinäruntersuchungsamt (CVUA) Freiburg, Germany
Dr. Michelangelo Anastassiades	Chemisches und Veterinäruntersuchungsamt (CVUA) Stuttgart, Germany
Dr. Sonja Masselter	AGES Competence Center for Residues of Plant Protection Products, Innsbruck, Austria
Dr. Tuija Pihlström	National Food Administration, Uppsala, Sweden
Dr. Magnus Jezussek	Bavarian Authority of Health an Food Safety, Erlangen, Germany
Mr. Finbarr o´Regan	Pesticide Control Laboratory, Celbridge, Ireland
Dr. Philippe Gros	Laboratoire du SCL, Montpellier, France