



# SPECIFIC PROTOCOL

# for the EU Proficiency Test for Pesticide Residues in Cereals/Feeding stuff using Multi Residue Methods, EUPT-CF13 (2019)

(last updated: 18 January 2019)

#### 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-CF13.

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

This proficiency test concerns the analysis of pesticide residues rye kernels. The rye 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 100 g of rye kernel Test Item with incurred and spiked pesticides and
- approximately 100 g of blank rye kernel Test Item.

# **Shipment of Test Items**

The Test Items are planned to be shipped on 28 January 2019.

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. Contrary to previous EUPT-CF this Test Item is not flour and the participant must mill before analyses. After milling mix the flour thoroughly, before taking the analytical portion(s).

All participants should use their own routine standard operating procedures for milling, extraction, clean-up and analytical measurement and their own reference standards for identification and quantification purposes.

The homogeneity test is conducted using 5 g of milled 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.

#### **DTU EUPT Webtool and Deadlines**

To select pesticide scope and report results and method information, the participants should log in to the <u>DTU EUPT Webtool</u> using the username and password send by email. For this current EUPT it will not be possible to change the password or ask for a new one. So it is important to save the information.

The analytical scope must be selected prior to the shipment of the samples. This can be done via the **DTU EUPT Webtool**. The scope selection subpage will be open from 21-28 January 2019.

The <u>DTU EUPT Webtool</u> will be accessible from 29 January 2019 for sample receipt acknowledgement and submission results and method information.

The deadline for submission is 25 February 2019 at 24.00 CET.

#### <u>Test Item Receipt and Acceptance</u>

Once the laboratory has received the Test Items it must report to the organiser, via the <u>DTU EUPT</u> <u>Webtool</u>, the date of receipt, and its acceptance. If the laboratory does not respond by 8 February 2019, the Organiser will assume that the Test Items have been received and accepted.

If participants have not received the Test Items by the 1 February 2019 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 Quantitative Results and method information

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.

Method information on the analytical method(s) used must also be reported

<u>Deadline</u>: All results and method information must be reported and submitted via <u>DTU EUPT Webtool</u> by **25 February 2019 at 24.00 CET.** The website will NOT be accessible for result submission after this date and time. The results and method information must be submitted before deadline by using the submit button

# Reporting of supplementary information in case of false negative results

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 also be done by accessing <u>DTU</u> **EUPT Webtool**. Deadline for this is 6 March 2019.

#### **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-CF13 can be found on <u>EUPT-CF13 Website</u>.

#### Calendar

Activity	Dates
Announcement Calendar Target Pesticide List	Oktober 2018
EUPT-Registration Website open	November 2018
Deadline for registration	10 January 2019
Specific Protocol published	21 January 2019
Website for selecting pesticide scope open	21 January 2019
Website for selecting pesticide scope closed	28 January 2019
Distribution of Test items	28 January 2019
Deadline for receipt and acceptance of Test Materials	within 24 hr on receipt
Deadline for Result Submission	25 February 2019
	at 24.00 CET
Deadline for submission of additional method information for false negative results	6 March 2019
	at 24.00 CET
Preliminary Report (only compilation of results) published	26 April 2019
Final Report published	December 2019

# **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 Tom Schmidt Christensen, <a href="mailto:tomsc@adm.dtu.dk">tomsc@adm.dtu.dk</a> at the financial department of DTU.

#### Contact information:

**DTU Food** National Food Institute





# **Mette Erecius Poulsen**

Head of EURL Cereals and Feeding stuff

National Food Institute Technical University of Denmark Kemitorvet, Building 202 DK-2800 Lyngby

Phone: +45-3588-7463 E-Mail: eurl-cf@food.dtu.dk http://www.eurl-pesticides.eu

# **Organising Team:**

Susan Strange Herrmann, Chemist	EURL for Cereals and Feeding stuff
Elena Hakme, 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

# **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), 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. Patrizia Pelosi	Istituto Superiore di Sanità, Roma, Italy