SPECIFIC PROTOCOL

for the EU Proficiency Test for Pesticide Residues in Cereals/Feeding stuff using Multi-Residue Methods, EUPT-CF7 (2013)

(last updated: 1 May 2013)

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 Feeding stuffs (NRL-CFs) as well as by all official EU laboratories (OfLs) responsible for official pesticide residue controls on feeding stuff, as far as their scope overlaps with that of the EUPT-CF7.

Test Item (Test Material)

This proficiency test concerns the analysis of pesticide residues in feed for laying hens. The feed has been produced by IFF-Braunschweig[1] of raw ingredients provided by EURL-CF. Some of the cereals used have been grown in Denmark and contain incurred pesticides. Furthermore, some of the ingredients were spiked with additional pesticides before all ingredients were mixed into the feeds. Ingredients in the two Test Items, marked "sample" and "blank" are listed in Table 1 below.

Table 1. Test item composition:

Ingredients	Test item with pesticides (sample)	Test item (blank sample)
Cereals (including maiz):	62%	62%
Soya meal	25%	
Soya cake		27%
Soya oil	3%	1%
Lime	9%	9%
Mineral and vitamin premix	1%	1%

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

[1] Forschungsinstitut Futtermitteltechnik der IFF, Research Institute of Feed Technology. Frickenmühle 1A, 38110 Braunschweig-Thune, Germany

detected pesticides reported. It should be noted that the composition of the blank Test Item is slightly different from the Sample Test Item, because soya cake has been used instead of soya meal. It is not possible to obtain organically produced soya meal. To equalize the fat content to the same level as in the sample, less soya oil has been added. Consequently, the fat content in both Test Items are the same.

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 that 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 feed Test Item with incurred and spiked pesticides and
- approximately 100 g of blank feed Test Item.

Shipment of Test Items

The Test Items are planned to be shipped on 13 May, 2013.

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. As the test material is already milled and sufficiently homogeneous, method sensitivity is the only major factor to consider when deciding the size of the analytical portion.

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-CF7 Result Submission Website** (http://thor.dfvf.dk/EUPT-CF7).

Subpage 0 (Test Item recipe) will be accessible from 14 May 2013 and subpage 1-3 from 20 May 2013 and onwards. 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) given in the confirmation e-mails sent to the laboratories upon registration.

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 7 June 2013

Test Item Receipt and Acceptance - Subpage 0

Once the laboratory has received the Test Items it must report to the organiser, via the **EUPT-CF7 Result Submission Website** the date of receipt, the condition of the Test Item, and its acceptance. The deadline for acceptance is the 17 May 2013. 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 16 May 2013 at noon, they must inform the Organiser immediately by e-mail (eurl-cf@food.dtu.dk).

Reporting Qualitative and Quantitative Results - Subpages 1 and 2

To report their results, laboratories must access the **EUPT-CF7 Result Submission Website**.

Deadline: All results must be reported on the online result submission website by 7 June 2013. 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.

The results should be reported in mg/kg test item with no re-calculation regarding water or fat content.

Results should not be reported where a pesticide was not detected, or was detected below the RL (Reporting Limit) of the laboratory, or below the MRRL. Results reported as <RL will be considered as „Not Detected".

The results (residue levels of the pesticides detected) must be expressed in mg/kg.
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.

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-CF7 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 a posterior.

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-CF7 Result Submission Website**. This subpage will be accessible from 11-16 June 2013.

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 instructions by DG-SANCO, the "Protocol for management of underperformance in comparative testing and/or lack of collaboration of National Reference Laboratories (NRLs) with EU Reference Laboratories (EURLs) activities" will be followed for NRLs.

Documents

All documents relating to EUPT-CF7 can be found in the EURL-Document Repository (CIRCA/FIS-VL). Links to the documents can be found in the EUPT–CF7 Website.

Calendar (see also http://www.eurl-pesticides.eu/library/docs/cf/EUPT_CF7_Calendar130304.pdf)

Activity	Dates
Announcement Calendar Target Pesticide List	January 2013
EUPT-Registration Website	18 March 2013
Deadline for registration	15 April 2013
Release of Specific Protocol	08 April 2013
Distribution of Test items	13 May 2013
Deadline for Receipt and Acceptance of Test Materials	within 24 hr on reciept
Deadline for Result Submission	7 June 2013
EUPT Evaluation Meeting	Ultimo June 2013
Preliminary Report (only compilation of results)	July 2013
Final Report	December 2013

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

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 Elena Soerensen at the financial department elso@adm.dtu.dk

Contact information:

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