SPECIFIC PROTOCOL

for the EU Proficiency Test for Pesticide Residues in Cereals using Multi-Residue Methods, EUPT-C6 (2012)

(last updated: 18.01.2012 – new email address for contact about invoice)

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 cereals and feeding stuff, as far as their scope overlaps with that of the EUPT-C6. The commodity barley is to be considered as representative for commodities with "high starch and/or protein content and low water and fat content" (see SANCO document 12495/2011).

Test Item (Test Material)

This proficiency test concerns the analysis of pesticide residues in barley. The barley was grown in Denmark in 2011 and pesticides were applied in the field. Following harvest, the rye was also spiked with some additional pesticides.

In addition, a blank Test Item is also provided, that can be used for recovery experiments as well as for the preparation of matrix-matched calibration standards for both MRM and SRM-pesticides. 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 be also 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 are 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.

It should not be assumed that only pesticides registered for use on barley are present in the Test Item.

Amount of Test Item

The participants will receive:

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

Shipment of Test Items

The Test Items are planned to be shipped on 30 January, 2012.

Test Items will be shipped frozen and packed in thermo-boxes together with a freeze gel pack. 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 Item, 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 about 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-C6 Result Submission Website** (http://thor.dfvf.dk/eupt-c6).

This website will be accessible from 30 January 2012 onwards and also 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 27 February at 14.00 CET

Test Item Receipt and Acceptance - Subpage 0

Once the laboratory has received the Test Items it must report to the organiser, via the **EUPT-C6 Result Submission Website** the date of receipt, the condition of the Test Item, and its acceptance. The deadline for acceptance is the 3 February 2012. If the laboratory does not respond by this deadline the Organisers will assume that the Test Items have been received and accepted. **If any participants have not received the Test Items by the 2 February at noon, they must inform the Organiser immediately by e-mail** (crlcereal@food.dtu.dk).

Reporting Qualitative and Quantitative Results - Subpages 1 and 2

To report their results, laboratories must access the **EUPT-C6 Result Submission Website**.

Deadline: **All results must be reported on the online result submission website by 27 February at 14:00 p.m., at the latest.** The website will not be accessible for result submission after this date, 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 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 to two significant figures (e.g. 0.0058 mg/kg).

Residue levels ≥ 0.010 mg/kg;

- to be expressed to 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 the results should be filled-in, 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 the 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 for recovery, 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-C6 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-C6 Result Submission Website**. The dates this subpage will be accessible will be announced in due time. If the page is empty when you access subpage 4, no further information is needed from you and you can leave the page without any further actions.

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–C6 can be found in the EURL-Document Repository (CIRCA/FIS-VL). Links to the documents can be found in the EUPT–C6 Website.

Calendar (see also http://www.crl-pesticides.eu/library/docs/cf/EUPT_C5_SRM6_Target.pdf)

Activity	*Who ?*	*Dates*
Access to "**EUPT-Registration Website**"	EURL-CF	15 December 2011
Deadline for registration	Invited Labs	15 January 2012
Release of Specific Protocol	EURL-CF	January 2012
Preparation of Test Material	EURL-CF	January 2012 (final preparation)
Homogeneity tests	EURL-CF	January 2012
Stability tests	EURL-CF	February 2012
▪ **Distribution of Test materials** ▪ Information to the laboratories regarding shipment	EURL-CF	**30 January 2012**
Activation of "**EUPT-C6 Result Submission Website**"	EURL-CF	30 January 2012
Deadline for Receipt and Acceptance of Test Materials: Online Submission of Form 0 (sub-page 0)	Participating Labs	within 24 hr of receipt and not later than 3 February 2012
Deadline for Result Submission **Pesticide scope, Results, Method Information** Submission of Form 1 – 3 (sub-pages 1 – 3)	Participating Labs	**27 February 2012 at 14.00 CET**
EUPT Evaluation Meeting	EURL-FV, EURL-SRM, Commission, EUPT-Scientific Committee	September 2012
Preliminary Report (only compilation of results)	EURL-CF	May 2012
Final Report	EURL-CF	December 2012

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 the 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 question concerning invoices must be directed to Carina Hillingsoe Groelsted at the financial department cah@adm.dtu.dk

Contact information

EURL-CF

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