SPECIFIC PROTOCOL

for the EU Proficiency Test for Pesticide Residues in Cereals using Multi- and Single-Residue Methods, EUPT-C5/SRM6 (2011)

(last updated: 09.03.2011)

Introduction

This protocol is complementary to the General Protocol for EU Proficiency Tests for Pesticide Residues in Food and Feed[1]. The current proficiency test is collaboratively organized by the EURL-CF[2] and the EURL-SRM[3] and covers pesticides that are determined by both Multi-Residue Methods (MRMs) and Single-Residue Methods (SRMs). This EUPT is to be performed by all National Reference Laboratories for Cereals and Feeding stuffs (NRL-CFs), all NRLs for Single Residue Methods (NRL-SRMs) 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-C5/SRM6. The commodity rice is to be considered as representative for commodities with "high starch and/or protein content and low water and fat content" **(see** SANCO document 10684/2009**)**[4]**.**

Test Items (Test Materials)

This proficiency test concerns the analysis of pesticide residues in rice. The rice was grown in Brazil in 2010.

Two different spiked Test Items are available:

1) **MRM-Test Item**, with the contained pesticides having been partly applied in the field and partly spiked in the laboratory
2) **SRM-Test Item**, with all pesticides having been spiked in the laboratory

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[1] http://www.crl-pesticides.eu/library/docs/allcrl/General%20Protocol%20for%20EUPTs-2ndEd-Nov2010.pdf

[2] EURL-CF= European Union Reference Laboratory for pesticides in Cereals and Feed

[3] EURL-SRM= European Union Reference Laboratory for pesticides requiring Single Residue Methods

[4] http://www.crl-pesticides.eu/library/docs/allcrl/AqcGuidance_Sanco_2009_10684.pdf

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.

The Organizers will check the spiked 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 test will be conducted by an ISO 17025 accredited laboratory.

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 summed residue definitions, that should be calculated and reported as stated in the list, as well as individual components where these should be analyzed and reported separately. For practical reasons, the residue definitions listed in the Target Pesticides List, in some cases, do not fully match with the legal ones.

The MRRL values will be used to help to identify false positive and false negative results and for the calculation of z-scores for false negatives.

Amount of Test Item

For the analysis of the MRM-compounds the participants will receive:

- approximately 50 g of rice Test Item with incurred and spiked pesticides and
- approximately 50 g of blank rice Test Item.

For the analysis of the SRM-compounds the participants will receive:

- approximately 250 g of rice Test Item with spiked pesticides and
- approximately 250 g of blank rice Test Item.

All samples will be frozen and packed in thermo boxes together with a freeze gel pack.

As the amount of available material is limited, additional material can only be delivered to the laboratories in exceptional cases and only if sufficient explanations are given by the requesting laboratory. No additional material will be provided to be used for purposes not relevant to the current EUPT.

Shipment of Test Items

The Test Items are planned to be shipped on 14 March, 2011.

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 Item 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 Item 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 except for dithiocarbamates, where 20 g will be used. 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 Deadline

Sample receipt acknowledgement, analytical results and method information are to be submitted via the "EUPT-C5/SRM6 Result Submission Website" (http://thor.dfvf.dk/eupt-c5/srm6).

This website will be **accessible from 15 March 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.

Test Item Receipt and Acceptance - Subpage 0

Once the laboratory has received the Test Items it must report to the organiser, via the EUPT-C5/SRM6 Result Submission Website (subpage 0), the date of receipt, the condition of the Test Item, and its acceptance. The deadline for acceptance is the 18 March 2011. 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 16th of March 2011 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-C5/SRM6 Result Submission Website (subpages 1 and 2). **Before entering the results please carefully read the Target Pesticide List, since the residue definitions are not given on the Result Submission Website.**

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

Deadline: **All results must be reported on the online result submission website by 11 April 2011 at 14:00 p.m., at the latest.** The website will not be accessible after this date, and any results reported after the deadline will not be included in the statistical treatment, or in the final report.

Summed residue definitions: For pesticides where the residue definition is a sum of components, results for both the sum and the listed individual components must be reported.
- If all listed[5] components of a summed residue definition were targeted by a laboratory but only part of them were detected and quantified, the laboratory should calculate and report the result of the respective summed residue definition, considering only the quantified components.
- If none or only part of the listed components within a summed residue definition were targeted by a laboratory, the Organizers will consider the summed residue definition as non-analyzed and ignore any numerical results reported.

[5] separately listed in the Pesticide Target List

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. In these cases, it should be recorded as 'ND' (Not Detected). 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-C5/SRM6 Result Submission Website (subpage 3). The laboratories are asked to thoroughly fill-in this important information in order to minimize the administrative burden of collecting this information a posteriori.

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-C5/SRM6 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–C5/SRM6 can be found in the EURL-Document Repository (CIRCA/FIS-VL). Links to the documents can be found in the EUPT–C5/SRM6 Website.

Calendar (see also http://www.crl-pesticides.eu/library/docs/cf/EUPT_C5_SRM6_Calendar.pdf)

Who?	**Activity**	**Date / Period**
Organizers	Release of "Specific PT-Protocol"	February 2011
Participants	Deadline for registration for the EUPT-C5/SRM6 (http://thor.dfvf.dk/eupt-signup)	Extended to 28 February 2011
Organizers	Test Item distribution and information to the laboratories regarding upcoming shipment	Starting 14 March 2011
Participants	Confirmation of Test Item receipt - **Subpage 0**	18 March 2011
Participants	Reporting of test results and method information **Subpages 1, 2 and 3**	11 April 2011 at 14:00 p.m.
Participants	Reporting missing information - **Subpage 4**	To be announced in due time
Organizers	Dispatch of a preliminary report to all participants (only results, no statistical treatment)	June 2011
Organizers	Dispatch of the final report as pdf-file	November 2011

Participation Fees

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

- 150 € for all labs participating only in the MRM-part of this EUPT
- 150 € for the labs participating only in the SRM part
- 200 € for the labs participating in both parts.

The participation fees for laboratories from third countries:

- 300 € for all labs participating only in the MRM-part of this EUPT
- 300 € for the labs participating only in the SRM part
- 450 € for the labs participating in both parts.

For further information visit the website www.eurl-pesticides.eu

Delays in Payment

The participants will receive an invoice from DTU-Dianova. 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 DTU-Dianova at proficiencytest@food.dtu.dk

Contact information

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***EURL-SRM (SRM part of EUPT)**:*

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