SPECIFIC PROTOCOL

for the 9th EU Proficiency Test
on Pesticides requiring Single Residue Methods
EUPT – SRM9 (2014)

(last update 24 April, 2014)

Introduction

This protocol is complementary to the "**General Protocol for EU Proficiency Tests for Pesticide Residues in Food and Feed**" covering all EUPTs.

The EUPT-SRM9 is organised by the EU Reference Laboratory for pesticides requiring Single Residue Methods (EURL–SRM) that is ISO 17043 accredited as a provider of proficiency tests. The EUPT-SRM9 deals with the analysis of SRM-pesticides in cow's milk with 3.5% - 4% fat and is to be performed by all National Reference Laboratories for Single Residue Methods (NRL-SRMs) as well as by all official EU laboratories (OfLs) involved in official pesticide residue controls, including imports control within the frame of Reg. 669/2009/EC, as far as their scope overlaps with that of the EUPT-SRM9. A special **EUPT-SRM9-Website** containing links to the most important documents of relevance was constructed.

Considering only the commodity scope (not the pesticide scope) of OfLs a **Tentative List of obliged labs for EUPTs in 2014** has been prepared by the EURLs and published on the CIRCA-Platform. As far as the EUPT-SRM9 is concerned all laboratories analysing for commodities of animal origin were considered as obliged. The comprehensiveness and correctness of this list was checked by the OfLs and reviewed by the NRL-SRM and any change requests were considered in a new version. OfLs listed as "obliged to participate in the EUPT-SRM9" that have decided not to participate had to state their reasons of non-participation in a special online form within the EUPT-SRM9 registration website which was accessible from 25 Feb. till 12 March, 2014.

Test Item and Blank Material

This EUPT deals with the analysis of pesticide residues in **cow's milk (3.5% – 4% fat)**.

Participants will receive two bottles containing:

1) ca. 250 ml **Test Item (spiked)**, containing pesticides from the **Target Pesticides List.**
2) ca. 250 ml **Blank Material**, that can be used for recovery experiments as well as for the preparation of matrix-matched calibration standards

Using randomly chosen bottles, the Organizers will check the Test Item for sufficient homogeneity and for the stability of the pesticides contained in the Test Item over the period of the exercise. The Blank Material will also be checked to prove that none of the pesticides on the Target pesticides List is contained at relevant levels.

Analytical parameters

The Test Item will contain several pesticides from the **Target Pesticides List.** Laboratories should read this list carefully, as it shows how the residues are expected to be reported as well as the **Minimum Required Reporting Levels (MRRLs)**. 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 in milk are present in the Test Item.

Shipment of Test Item

Test item and Blank Material are planned to be shipped on 28 April, 2014.
Frozen Test Item and Blank Material will be packed in thermo-boxes together with dry ice and shipped to the participants. 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 (except 1 May) 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.**
Should any complications during shipment, delivery or the customs be expected, the participating laboratories should provide the Organizers with contact information of possible contact persons of the lab (e.g. mobile phone numbers) as well as instructions in local language explaining the need to keep the package in freezer during delay in transit. This information will be attached to the package.

Instructions on handling the Test Item

Once received, the Test Item should be stored deep frozen (at -18°C or lower) until analysis in order to avoid any possible deterioration/spoilage and to minimize pesticide degradation.

Before analytical portions are taken for analysis the Test Item should be mixed thoroughly in its entirety. During mixing, try to keep temperatures low (< 4°C or frozen) to avoid degradation of susceptible pesticides.
To avoid frequent thawing of the Test Item it is further recommended preparing all analytical portions that you intend to use for all EUPT-related experiments in the sample preparation tubes and store them at -18°C till use.

All participants should use their own routine standard operating procedures for extraction, clean-up and analytical measurement as well as their own reference standards for identification and quantification purposes. Where the procedure employed has not yet been implemented routinely, any other method can be used. The limited experience and the non-inclusion of the analyte in the routine scope should be indicated in the result submission website.

The homogeneity tests will be conducted using 10 g analytical portions of Test Item for all analytes. Please note: Subsampling variability increases with decreasing analytical portion size, and sufficient homogeneity can only be guaranteed for sample portions ≥ 10 g.

Results submission website

Sample receipt acknowledgement, analytical results and method information are to be submitted via the following web-site: **EUPT-SRM9 Result Submission Website** (http://pesticides.food.dtu.dk/srm).

- **Sub-Page 0 (Sample receipt acknowledgement), accessible from 29 April, 2014**
- **Sub-Pages 1-3 (analytical results and method information) accessible from 6 May till 26 May, 2014.**

To access the data-submission forms participants must use their unique login data (username and password) that will be provided to them, together with their unique EUPT-SRM9 lab-code, in a separate e-mail before sample shipment.

The deadline for result submission is 26 May 2014 at 16 h (CEST).

- Sample Receipt and Acceptance (Sub-Page 0)

Once the laboratory has received the Test Items it must report to the organiser, via the **EUPT-SRM9 Result Submission Website** (sub-page 0) the date of receipt, the condition of the Test Item, and its acceptance. The deadline for acceptance is the 2 May 2014. If a laboratory does not respond by this deadline, the Organisers will assume that Test Item and Blank Material have been received and accepted.
If any participants have not received the Test Items by the 30 April in the afternoon, they must inform the Organiser by e-mail (EURL-SRM@cvuas.bwl.de**) to localize the package and decide on further action including new shipment if necessary**.
Selected participants might be asked to provide information on the condition of the Test Item upon receipt (e.g. existence of residual dry ice, core temperature of Test Item etc.).

- Reporting qualitative and quantitative Results (Sub-Page 1 and 2)

To report their results, laboratories must access the **EUPT-SRM9 Result Submission Website**.
All results must be reported on the above website by 26 May 2014 at 16 h (CEST). The website will not be accessible after this deadline, and all results submitted afterwards will not be included in the statistical treatment or in the final report.

Before entering the results, please study the **Target Pesticide List** carefully, in particular the residue definitions, which are not given on the Result Submission Website.

The following fields will be available for reporting the quantitative results:

- "**Concentration in mg/kg":** the pesticide concentrations that would be reported in routine work. Recovery-corrected results should be reported only where this reflects the routine lab's procedure; otherwise the non-recovery-corrected result should be reported. Results should not be reported where a pesticide was not detected, or was detected below the RL (Reporting Limit) of the laboratory or the MRRL. Results reported as "< RL" will be considered as „Not Detected".

  The residue levels of the pesticides must be reported in mg/kg using the following **significant figures**:
  - Levels <0.010 mg/kg to be expressed to 2 significant figures, e.g. 0.0058 mg/kg;
  - Levels ≥ 0.010 mg/kg to be expressed to 3 significant figures, e.g. 0.156, 1.64, 10.3 mg/kg.

- **"Conc. in blank in mg/kg":** concentration values of any pesticides from the Target Pesticides List determined in the Blank Material (even at levels below the MRRL).

- **"Experience with this compound":** Use the dropdown-menu to indicate for how many years you have been analysing for each compound using the method applied in this EUPT.

- **"Is your result recovery-corrected?":** Please specify via the dropdown-menu whether the reported result was corrected for recovery or not. Please note that in some cases recovery correction is performed automatically, e.g. when employing the standard additions approach with the additions being done to analytical portions prior to extraction or when an isotopically labelled analogue of the target analyte is used as internal standard. If an isotopically labelled compound not corresponding to the target analyte is used as internal standard (e.g. ethephon D4 for glyphosate), this question should be answered with "No". Where recovery correction was carried out using a recovery figure, this recovery figure should be stated (see below).

- **"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 for recovery, the recovery figure (in %) used for the calculation MUST be reported.

- **Recovery Details:** Please indicate here concisely how the recovery experiment(s) was/were conducted, e.g. spiking level, spiked compound.

  Additional information will be asked in separate fields.

- Reporting Information on Analytical Methodology (Sub-Page 3)

In **sub-page 3 of** the "**EUPT-SRM9 Result Submission Website**" the participating laboratories must provide complete information on the analytical method(s) applied to **all pesticides which were analysed, irrespective if they were detected or not**.

The participating laboratories are urged to thoroughly fill-in all requested information in order to minimize the administrative burden of collecting it information a posteriori.

If no sufficient information on the methodology used is provided, the Organisers reserve the right not to accept the analytical results reported by the participant.

Subcontracting

The following tasks will be subcontracted to the EURL-CF, Soeborg, Denmark:

a) The administration of **EUPT-SRM9 Registration and Result Submission Website**

Follow-up actions

After the distribution of the Preliminary EUPT-Report laboratories with poor performance (high absolute z-scores, false negatives or false positives) will be asked to provide information concerning the reasons for poor performance and possible corrective actions. This information will be forwarded to the corresponding NRL-SRMs upon request. All EUPT-SRM9-participants are welcome to ask the EURL-SRM for technical assistance.
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 Community reference laboratories (CRLs) activities**" will be followed for NRLs.

Documents

All documents related to the EUPT–SRM9 can be found in the **EURL-Document Repository (CIRCA/FIS-VL).** Links to the documents can also be found in the **EUPT-SRM9 Website.**
For further information please contact the organizers **EURL-SRM@cvuas.bwl.de**
Please check the **EUPT-SRM9 Website** before starting with the analysis to **make sure that you have the latest version of all documents available**. In case of major changes the participants will be informed via e-mail.

Participation fees and payment details

To cover the costs of production, handling and shipment of the PT-Materials the following fees will be charged for one unit of the PT-Material to the participating laboratories:

- OfLs (including NRLs) from EU countries, EU-candidate countries and EFTA countries: 175 €
- Labs based in third countries: 350 €

Invoices, issued to the "invoice address" stated in the registration form, will be enclosed inside the package containing the PT-Materials.

Payment is expected to be made within 30 days upon the date of shipment. **If for any reason payment cannot be carried out before this date, please contact the Organizer to give explanations. If no payment or no proof of payment is received and no explanation is given to the Organizers, the Organizers reserve the right not to accept the results from those labs or not to include them in the Final EUPT-Report.**
Bank details for remittance will be given in the invoices.

To facilitate tracking of money transfer the special payee identification text (= invoice number) as shown in the invoice must be indicated in the remittance.

Please note:

The bank account of EURL-SRM has been changed since the end of October 2013!

Please inform your financial department!

NEW Bank Details:

Bank account holder:	**Landesoberkasse Baden Wuerttemberg**
Bank Name :	**Baden Wuerttembergische Bank**
IBAN:	**DE 02 6005 0101 7495 5301 02**
BIC/SWIFT:	**SOLADESTXXX**
Payee identification text:	***See invoice (important and must be indicated!)***
VAT of CVUA Stuttgart	**DE 811 600 510**

Calendar of EUPT-SRM9

(see under **http://www.eurl-pesticides.eu/library/docs/srm/EUPT_SRM9_Calendar.pdf**)

Target Pesticides List of EUPT-SRM9

(see under **http://www.eurl-pesticides.eu/library/docs/srm/EUPT_SRM9_TargetPesticideList.pdf**)

Contact information

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