SPECIFIC PROTOCOL

for the 14th EU Proficiency Test
on Pesticides requiring Single Residue Methods
EUPT – SRM14 (2019)

(released on 4 March 2019)

Introduction

This protocol is complementary to the valid version of the "**General Protocol for EU Proficiency Tests for Pesticide Residues in Food and Feed, Ed. 8**" covering all EUPTs in 2019.
The EUPT-SRM14 is organised by the EU Reference Laboratory for pesticides requiring Single Residue Methods (EURL–SRM) in cooperation with EU Reference Laboratory for residues of pesticides in food of animal origin and commodities with high fat content (EURL-AO). Both EURLs are accredited according to ISO 17043 as providers of proficiency tests (Please see **EURL-SRM accreditation** and **EURL-AO accreditation**).
The EUPT-SRM14 deals with the analysis of SRM-pesticides in bovine liver 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, as far as their scope overlaps with that of the EUPT-SRM14. A special **EUPT-SRM14-Website** containing links to the most important documents of relevance was constructed.

A preliminary classification of laboratories into obliged and non-obliged to participate in the present PT was prepared based on information within the EURL DataPool. NRL-SRMs and OfLs performing pesticide residue analyses in food of animal origin within the frame of National and EU official controls were considered as tentatively obliged to participate in this PT. The laboratories were asked to update this information prior to the EUPT-registration period. This tentative classification was only based on the commodity scope (not the pesticide scope) of the laboratories and was also visible to the participants within the registration page. OfLs listed as “obliged to participate in the EUPT-SRM14” but not intending to participate had to state their reasons for non-participation during the online registration of the EUPT-SRM14, which lasted from 18 January till 8 February, 2019. The feedback received during registration, especially details considering the scope, will be considered in the final list of obliged laboratories.

Test Item and Blank Material

The Test Item of this EUPT is **Bovine Liver**.

Participants will receive two bottles containing:

1) ~ 200 g **Test Item containing spiked analytes** from the **Target Pesticides List.** This **Test Item** will be prepared by cryogenic milling and will be shipped in a snow-like condition within a 500ml plastic jar [1].

2) ~ 200 g **Blank Material**, that can be used for recovery experiments as well as for the preparation of matrix-matched calibration standards. This **Blank Material** will be shipped as an ice block within a 300ml plastic jar [1].

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

Target Analytes and MRRLs

The Test Item will contain several pesticides from the **EUPT-SRM14 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. **Make sure to download the latest version of the EUPT-SRM14 Target Pesticides List before starting with analysis and result reporting**.

Shipment of Test Item

Test item and Blank Material are planned to be shipped on 18 March, 2019.
Frozen Test Item and Blank Material will be packed in thermo-boxes together with dry ice[1] and shipped from Germany via DHL express parcel to the participants. Prior to shipment a reminder will be sent to the participating laboratories by e-mail.
The participating laboratories must make their own arrangements for the receipt of the package. They should inform the Organisers 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.

Should any complications during shipment, delivery or the customs be expected, the participating laboratories should provide the Organizers in advance 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 and delivery. This information will be attached to the package.

After the packages will be picked up and stored in the DHL delivery system, each participant will be informed by DHL about the tracking number of his package. The participants can follow the delivery state of their own packages online and must make any necessary arrangements to receive the delivery.

Once unexpected delay is noticed in the customs or within the recipient's country, the participant himself is strongly encouraged to contact his local DHL Express office and/or the customs in order to accelerate the clearance and delivery procedures.

Instructions on handling the Test Item

Once arrived, both the Test Item and the Blank Material should be stored deeply frozen (at -18°C or lower) until analysis in order to avoid any possible deterioration/spoilage of the sample and to minimize pesticide degradation.

The **Test Item** was homogenized via cryogenic milling. It should normally arrive in a snow-like condition so that it can be easily loosened up with a spatula and the analytical portions can be conveniently withdrawn. Any coagulations of the

[1] PT-Material for participating laboratories in countries or locations where the shipment with dry ice is not allowed will be packed in thermo-isolated jars, pre-cooled at -70°C and shipped in thermo-boxes additionally containing freeze elements.

homogenate are not expected to have any impact on the homogeneity, so in principle there is no need of re-mixing the Test Item in its entirety.

The **Blank Material** is provided as an ice block. Prior to taking analytical portions, it is recommended leaving the Blank Material at ambient temperature for approx. 1 hour to partly defrost followed by a mixing with a knife mill or leaving the material to almost entirely defrost (e.g. 2.5 – 3 hours at ambient temperature) followed by stirring with a spatula while still cold.

Participating laboratories are recommended using their routine standard operating procedures for extraction, clean-up and analytical measurement as well as their own reference standards for identification and quantification purposes. Laboratories may also employ methods not yet implemented routinely, for example, if they are in the test-phase of implementing them. In this case the limited experience and the non-inclusion of the analytes in the routine scope should be indicated in the **EUPT-SRM14 result submission webtool**.

The homogeneity tests will be conducted using 10 g for both QuEChERS and QuPPe amenable pesticides. As sub-sampling variability increases with decreasing analytical portion size, sufficient homogeneity can be guaranteed only for sample portions equal to or bigger than the portion size used in the homogeneity test.

Results submission webtool

Sample receipt acknowledgement, analytical results and method information are to be submitted via the **EUPT-SRM14 result submission webtool**:

- **Sample receipt acknowledgement: accessible from 19 March till 28 March, 2019.**
- **Analytical results and method information: accessible from 19 March till 15 April midnight (CEST), 2019.**
- **The deadline for result submission is 15 April midnight (CEST), 2019.**
- **Additional information on the methods used for tentatively false negative results: accessible from 16 April till 29 April midnight, 2019.**

A guideline for the new **EUPT-SRM14 result submission webtool** will be provided to the participants in due time. The **participants are urged to read it carefully before submitting their results**.

- Login Credentials and Lab Code

To access the EUPT-SRM14 result submission webtool, participants must use their personal login credentials (username and password). The link to the EUPT-SRM14 result submission webtool and the personal login credentials will be provided to the PT-contact persons approx. two weeks prior to sample shipment.

The lab's unique lab code for the EUPT-SRM14 will be provided to the participants after accessing to EUPT-SRM14 result submission webtool for the first time.

- Acknowledgement of Package Receipt and Acceptance of PT-Materials

Once the laboratory has received the package, it must report to the organiser via the **EUPT-SRM14 result submission webtool** the date of receipt, the condition of the Test Item, and its acceptance. The page Sample Acknowledgement will remain open till 28 March. If a laboratory does not respond by this deadline, the Organisers will assume that Test Item and Blank Material have been received and accepted.

Any participants having not received the Test Items by the Fri. 22 March at noon must inform the Organiser via e-mail (EURL-SRM@cvuas.bwl.de) by Fri. 22 March 2:30 pm. The Organiser will consult the shipping company to localize the package and decide on further actions including new shipment, if necessary.

- Reporting qualitative and quantitative Results

To report their results, laboratories must access the **EUPT-SRM14 result submission webtool**.
All results must be reported on this website by 15 April midnight (CEST), 2019. The website will not be accessible after this deadline, and all results submitted afterwards will not be accepted.

Before entering the results, please study the **EUPT-SRM14 Target Pesticides List** carefully, in particular the residue definitions that apply to the EUPT, which may not be given in full on the result submission website.

Among others, the following fields will be available for reporting the quantitative results:

- "**Concentration in mg/kg":** the numerical pesticide concentrations that would be reported in routine work. 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" or "< #,# mg/kg" 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

  Recovery-corrected results should be reported only where this reflects the routine lab's procedure; otherwise the non-recovery-corrected result should be reported. Where a **result was corrected for recovery,** the approach(es) followed to achieve this correction (e.g. standard additions to sample portions, procedural calibration, recovery factor, use of ILIS) must be reported in the respective fields.

- **"Conc. in blank in mg/kg":** concentration values of any pesticides from the **EUPT-SRM14 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.

- Reporting Information on Analytical Methodology

On the page of "**Edit methods"** of **EUPT-SRM14 result submission webtool** the participating laboratories must provide information on the analytical method(s) applied to pesticides which were analysed and detected either in the Test Item or in the Blank Material.

The participating laboratories are urged to thoroughly fill-in all requested information and control it carefully in order to minimize the administrative burden of collecting and correcting it 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 or to refuse participation is future EUPT-SRMs.

For detailed information on the columns on the page of "**Edit methods" please refer to the guideline for results submission that will be issued and provided to you in due time**.

- Submission of results

Once you have entered all your results and checked their correctness, you have to submit them by clicking the bottom "Final submission" before the submission deadline. Afterwards, you will NOT be able to change your data anymore. Without "Final submission" your results and method information will not be included in the evaluation!

- Additional Information

If the laboratory has obtained tentatively false negative result(s), it will be asked for entering the method information for the pesticide(s) in question after the results submission period is closed.

Subcontracting

The following task was subcontracted to the EURL-CF, Søborg, Denmark:

a) Generation of the login credentials
b) Administration of EUPT-SRM14 result submission website

Follow-up actions

After the distribution of the EUPT-SRM14 Preliminary Report, laboratories with poor results (high absolute z-scores, false negatives or false positives) will be asked to provide information concerning the reasons for this and possible corrective actions. This information will be forwarded to the corresponding NRL-SRMs upon request. All EUPT-SRM14-participants are welcome to ask the EURL-SRM for technical assistance.

The Organiser might ask laboratories to provide missing methodology information that is important for the evaluation and interpretation of the PT.

According to instructions by DG-SANTE, 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 by NRLs.

Documents

All documents related to the EUPT–SRM14 can be found in the **EURL-Document Repository (CIRCA-BC).** Links to the documents can also be found in the **EUPT-SRM14 Website.**
For further information please contact the organizers **EURL-SRM@cvuas.bwl.de**
Please check the **EUPT-SRM14 Website** before starting with the analysis in order 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: 250 €
- Labs based in third countries: 400 €

An invoice issued to the "invoice address" stated in the registration form will be sent two weeks after sample shipment to the invoice e-mail address stated in the registration form. Should the payment being taken care of by another department/institution, the recipient of the invoice is requested to forward the invoice accordingly. Details of payment will be given in the invoices.

Payment is expected to be made within 30 days upon the invoice issue date unless special information was provided by the participant during registration and/or otherwise agreed between participant and organiser.

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 to exclude the results of the concerned laboratories from the Final EUPT-Report or to refuse its participation in future EUPT-SRMs.

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**

Please note:
EURL-AO based in CVUA Freiburg and EURL-SRM based in CVUA Stuttgart belong to the same ministry and have thus the same bank account.
If your laboratory participated in both the EUPT-SRM14 and the EUPT-AO14, please ask your financial department to transfer the fee for each of the PTs separately using the corresponding payee identification text (= invoice number) given in the invoice. Without this text, your payment will not be able to reach the correct EURL.

More details for bank-remittance are given in the invoices.

Calendar of EUPT-SRM14

(please see http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM14_Calendar.pdf)

Target Pesticides List of EUPT-SRM14

(please see http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM14_TargetPesticideList.pdf)

Supplementary Information on EUPT-SRM14 Analytes

(please see http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM14_Suppl_Info.xls)

Contact information

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