

SPECIFIC PROTOCOL

for the 13th EU Proficiency Test on Pesticides requiring Single Residue Methods

EUPT – SRM13 (2018)

(released on 09.April, 2018)

Introduction

This protocol is complementary to the valid version of the "[General Protocol for EU Proficiency Tests for Pesticide Residues in Food and Feed](#)" covering all EUPTs.

The EUPT-SRM13 is organised by the EU Reference Laboratory for pesticides requiring Single Residue Methods (EURL-SRM) which is [accredited according to ISO 17043](#) as providers of proficiency tests.

The EUPT-SRM13 deals with the analysis of SRM-pesticides in dry soybeans 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-SRM13. This includes laboratories involved in import control within the frame of Reg. 669/2009/EC. A special [EUPT-SRM13-Website](#) containing links to the most important documents of relevance was constructed.

A preliminary selection of the laboratories considered to be obliged to participate in the present was done based on the information in the official Lab-Network-database hosted in the EURL DataPool and considering the commodity scope only (not the pesticide scope). The status of the OfLs concerning their participation in the current PT was shown on the registration page. As far as the EUPT-SRM13 is concerned, all OfLs analysing pesticides of food and feed in the commodity group "high oil content and very low water content" were considered as obliged to participate in this PT. OfLs listed as "obliged to participate in the EUPT-SRM13" but not intending to participate had to state their reasons for non-participation during the online registration of the EUPT-SRM13, which lasted from 26 February till 16 March, 2018. The feedback received during registration is considered in the final list of obliged laboratories.

Test Item and Blank Material

This EUPT deals with the analysis of pesticide residues in [Soybean Flour](#).

Participants will receive two bottles containing:

- 1) ca. 200 g **Test Item with a selection of incurred and spiked analytes** from the [Target Pesticides List](#).
- 2) ca. 200 g **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 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-SRM13 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-SRM13 Target Pesticides List](#) before starting with analysis and result reporting.**

Please note, the definitions of the following target pesticides was modified:

- “Chlormequat cation” and “mepiquat cation” were changed to “chlormequat [chloride](#)” and “mepiquat [chloride](#)” respectively, to reflect the new wording of the MRL-regulation
- “Phosphane” was changed to “Phophane (phosphine, incl. phosphane released from phosphide salts)”, to reflect the new wording of the MRL-regulation

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

Shipment of Test Item

Test item and Blank Material are planned to be shipped on 23 April, 2018.

Cooled Test Item and Blank Material will be packed in thermo-boxes together with cool gel packs 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.

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 were 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.

Once a package has arrived the recipient country and an unexpected delay is notices, the participant himself is strongly encouraged to contact his local DHL Express or the customs in order to accelerate the clearance and delivery procedures.

Instructions on handling the Test Item

Both Test Item and Blank material are provided as milled flour. Once arrived, 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 to avoid losses of susceptible pesticides.

Participating laboratories should use 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-SRM13 Result Submission Website**.

The homogeneity tests will be conducted using 5 g for 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 used portion size in the homogeneity test.

Results submission website

Sample receipt acknowledgement, analytical results and method information are to be submitted via the following website: **EUPT-SRM13 result submission website**.

- **Sub-Page 0 (Sample receipt acknowledgement), accessible from 25 April, 2018.**
- **Sub-Pages 1-3 (analytical results and method information) accessible from 30 April, till 22 May, 2018.**
- **The deadline for result submission is 22 May, 2018 at 16 h (CEST).**

- Login Credential and Lab-Code

To access the data-submission forms participants must use their unique login credentials (username and password). The login credential together with the EUPT-SRM13 lab-codes will be provided to each of the participating laboratories on the shipment day.

- Sample Receipt and Acceptance (Sub-Page 0)

Once the laboratory has received the package it must report to the organiser via the **EUPT-SRM13 Result Submission Website** (sub-page 0) the date of receipt, the condition of the Test Item, and its acceptance. For laboratories in the EU- and EFTA countries and EU candidate countries, the deadline for acceptance is 27 April, 2018. 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 that have not received the Test Items by the 27 April at noon, they must inform the Organiser via e-mail (EUURL-SRM@cvuas.bwl.de). 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 (Sub-Page 1 and 2)

To report their results, laboratories must access the **EUPT-SRM13 Result Submission Website**.

All results must be reported on this website by 22 May, 2018 at 16 h (CEST). 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 [Target Pesticide List](#) carefully, in particular the residue definitions that apply to the EUPT, which are not necessarily given in full 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. 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 must be reported in the respective fields in **Subpage 3** factor, use of ILIS).

- **“Conc. in blank in mg/kg”**: concentration values of any pesticides from the [Target Pesticide 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 (Sub-Page 3)

On **sub-page 3** of the **“EUPT-SRM13 Result Submission Website”** the participating laboratories must provide **COMPLETE** information on the analytical method(s) applied to **all pesticides which were analysed, irrespective of whether they were detected or not.**

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 in future EUPT-SRMs.

For detailed information on the columns on sub-page 3 please refer to the [Guide on Result Submission \(http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM13_Short_Guide_SubPages.pdf\)](#). **Once your results are completely submitted, please export them via the function on the submission Main Page as a csv-format. This file can be viewed via Excel, and you can easily check your entries stored in the database.**

Subcontracting

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

- a) Generation of the login credentials
- b) Administration of EUPT-SRM13 result submission website

Follow-up actions

After the distribution of the EUPT-SRM13 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-SRM13-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–SRM13 can be found in the [EURL-Document Repository \(CIRCA-BC\)](#). Links to the documents can also be found in the [EUPT-SRM13 Website](#).

For further information please contact the organizers EURL-SRM@cvas.bwl.de

Please check the [EUPT-SRM13 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: 200 €
- Labs based in third countries: 350 €

An invoice issued to the "invoice address" stated in the registration form will be sent to the invoice e-mail address stated during registration. 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 date of shipment 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:	<i>See invoice (important and <u>MUST</u> be indicated!)</i>
VAT of CVUA Stuttgart	DE 811 600 510

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

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

Calendar of EUPT-SRM13

(please see http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM13_Calendar.pdf)

Target Pesticides List of EUPT-SRM13

(please see http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM13_TargetPesticideList.pdf)

Contact information

EU Reference Laboratory for Single Residue Methods (EURL-SRM)

Chemisches und Veterinäruntersuchungsamt Stuttgart
Schaflandstr. 3/2,
D-70736 Fellbach
Germany

e-mail: EURL-SRM@cvuas.bwl.de

Fax: +49 3426 1124

Organising Group at the EURL-SRM (Stuttgart)

Michelangelo Anastassiades	phone: +49 3426 1124
Pat Schreiter	phone: +49 3426 1029
Carmen Wauschkuhn	phone: +49 3426 1159
Hubert Zipper	phone: +49 3426 1141
Anja Barth	phone: +49 3426 1935
Giovanna Cerchia	phone: +49 3426 1114

Advisory Group

Amadeo Fernández-Alba	EURL-FV, University of Almería (UAL), ES
Miguel Gamón	EURL-FV, Laboratorio Agroalimentario Generalitat Valenciana (LAGV), ES
Mette Erecius Poulsen	EURL-CF, National Food Institute (DTU), Søborg, DK
Ralf Lippold	EURL-AO, CVUA Freiburg, DE
Philippe Gros	Service Commun des Laboratoires (SCL) / Laboratoire de Montpellier, FR
Magnus Jezussek	Bavarian Health and Food Safety Authority (LGL), Erlangen, DE
André de Kok	Netherlands Food and Consumer Product Safety Authority (NVWA), Amsterdam, NL
Sonja Masselter	Austrian Agency for Health and Food Safety (AGES), Innsbruck, AT
Finbarr O'Regan	Pesticide Control Laboratory (PCL), Dept. of Agriculture, Food and the Marine (DAFM) IR
Tuija Pihlström	Swedish National Food Agency (SNFA-Livsmedelsverket), Uppsala, SE
Carmelo Rodríguez	University of Almería (UAL), Spain

Quality Control Group

Antonio Valverde	University of Almería (UAL), ES
Paula Medina	European Food Safety Authority (EFSA)