SPECIFIC PROTOCOL

for the 12th EU Proficiency Test
on Pesticides requiring Single Residue Methods
EUPT – SRM12 (2017)

(update on 10 March, 2017)

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-SRM12 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-SRM12 deals with the analysis of SRM-pesticides in strawberry purée 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-SRM12. This includes laboratories involved in import control within the frame of Reg. 669/2009/EC. A special **EUPT-SRM12-Website** containing links to the most important documents of relevance was constructed.

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 is shown on the registration page. As far as the EUPT-SRM12 is concerned, all OfLs analysing pesticides in fruits and vegetables were considered as obliged. OfLs listed as "obliged to participate in the EUPT-SRM12" but not intending to participate had to state their reasons for non-participation during the online registration of the EUPT-SRM12, which lasted from 17 January till 20 February, 2017.

Test Item and Blank Material

This EUPT deals with the analysis of pesticide residues in **Strawberry Purée**.

Participants will receive two bottles containing:

1) ca. 400 g **Test Item (with incurred or spiked analytes)**, containing pesticides from the **Target Pesticides List.**
2) ca. 400 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-SRM12 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-SRM12 Target Pesticides List before starting with analysis and result reporting**.
It should not be assumed that only pesticides registered for use in strawberry are present in the Test Item.

Shipment of Test Item

Test item and Blank Material are planned to be shipped on 13 March, 2017.
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 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 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.

Instructions on Handling the Test Item

Both Test Item and Blank material were cryogenically milled and filled into the vessels in a snow-like state.

In case of a **delivery in < 2 days** the material should normally arrive in **snow like state**. If it is put in the freezer immediately the material will retain this state for several days. Prior to withdrawing analytical portions you may loosen the material at the top (e.g. 5-10 cm) with a spatula or knife and mix it a bit. There is no need for mixing the entire material. If the material is very cold (e.g. < -25 °C) you may initially experience difficulties to loosen it up. Wait a few minutes and try again.

In case the **material has melted to a considerable degree** it is advisable to **mix it well with a rod** and to withdraw the required analytical portions directly. If you put the material into the freezer in this state you will end up with an ice-block that is difficult to handle. During the freezing process ice crystals may be formed onto the surface of lid and walls. In this case it is advisable to re-mix the material in its entirety before withdrawing analytical portions.

Please also take note of the following advices:

- **Please note that in stability tests by the organizer the material is maintained in frozen state until analysis.**
- **While mixing and during storage, try to keep the temperature of the material as low as possible to avoid losses of unstable pesticides.**
- **If you still decide to defrost the homogenate prior to analysis keep temperatures low and try to minimize as much as possible the time the material is in a defrosted state. Defrosting in the refrigerator over many hours is also very critical and is not recommended.**
- **Analytical portions which are not analysed immediately should be stored in the freezer until analysis to minimize pesticide degradation.**

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 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 except for dithiocarbamates, where expectedly 20 g will be used. Please note: Sub-sampling 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 website: **EUPT-SRM12 result submission website.**

- **Sub-Page 0 (Sample receipt acknowledgement), accessible from 14 March, 2017.**
- **Sub-Pages 1-3 (analytical results and method information) accessible from 20 March, till 10 April, 2017.**
- **The deadline for result submission is 10 April, 2017 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-SRM12 lab-codes the 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 Test Items it must report to the organiser via the **EUPT-SRM12 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 17 March, 2017. 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 17 March in the afternoon, they must inform the Organiser via e-mail (EURL-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**.
Selected participants might be asked to provide information on the condition of the Test Item upon receipt (e.g. 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-SRM12 Result Submission Website**.
All results must be reported on this website by 10 April, 2017 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-SRM12 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 is future EUPT-SRMs.

For detailed information on the columns on sub-page 3 please refer to the detailed Guide on Result Submission (http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM12_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-SRM12 result submission website

Follow-up actions

After the distribution of the EUPT-SRM12 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-SRM12-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–SRM12 can be found in the **EURL-Document Repository (CIRCA-BC).** Links to the documents can also be found in the **EUPT-SRM12 Website.**
For further information please contact the organizers **EURL-SRM@cvuas.bwl.de**
Please check the **EUPT-SRM12 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: 250 €

- Labs based in third countries: 350 €

An invoice issued to the "invoice address" stated in the registration form will be sent to the e-mail address of the invoice recipient 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.
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 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**

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 will be given in the invoices.

Calendar of EUPT-SRM12

(please see: http://www.eurl-pesticides.eu/userfiles/file//EUPT-SRM12_Calendar.pdf)

Target Pesticides List of EUPT-SRM12

(please see: http://www.eurl-pesticides.eu/userfiles/file//EUPT-SRM12_TargetPesticideList.pdf)

Contact information

EU Reference Laboratory for Single Residue Methods (EURL-SRM)
Chemisches und Veterinäruntersuchunsamt 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
Anne Benkenstein	phone: +49 3426 1151
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, ES
Miguel Gamón	EURL-FV, Laboratorio Agroalimentario Generalitat Valenciana, 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, Innsbruck, AT
Finbarr O'Regan	Pesticide Control Laboratory (PCL), Dept. of Agriculture, Food and the Marine (DAFM) IR
Tuija Pihlström	Swedish National Food Agency (Livsmedelsverket), Uppsala, SE
Carmelo Rodríguez	University of Almería, Spain

Quality Control Group

Antonio Valverde	University of Almería, ES
Paula Medina	EFSA, Parma, EU