



SPECIFIC PROTOCOL

for the 11th EU Proficiency Test on Pesticides requiring Single Residue Methods

EUPT – SRM11 (2016)

(update on 27 April, 2016)

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-SRM11 is organised by the EU Reference Laboratory for pesticides requiring Single Residue Methods (EURL-SRM) in cooperation with the EU Reference Laboratory for pesticide residues in Fruits and Vegetables (EURL-FV). Both EURLs are [accredited according to ISO 17043](#) as providers of proficiency tests.

The EUPT-SRM11 deals with the analysis of SRM-pesticides in spinach homogenate 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-SRM11. This includes laboratories involved in import control within the frame of Reg. 669/2009/EC. A special [EUPT-SRM11-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 2016](#) has been prepared by the EURLs and published on the [CIRCA BC-Platform](#). As far as the EUPT-SRM11 is concerned, all OfLs analysing pesticides in fruits and vegetables were considered as obliged. The initial list has been updated considering all input received by the NRL-SRMs and the OfLs. OfLs listed as "obliged to participate in the EUPT-SRM11" but not intending to participate had to state their reasons for non-participation during the online registration the EUPT-SRM11 from 8 February till 11 March, 2016.

Test Item and Blank Material

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

Participants will receive two bottles containing:

- 1) ca. 300 g **Test Item (with incurred or spiked analytes)**, containing pesticides from the [Target Pesticides List](#).
- 2) ca. 300 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-SRM11 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-SRM11 Target Pesticides List before starting with analysis and reporting of results.**

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

Shipment of Test Item

Test item and Blank Material are planned to be shipped on 4 April, 2016 to laboratories in Spain and Portugal and on 5 April, 2016 to laboratories in all other countries.

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. 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, it is recommended to mix the material thoroughly in its entirety. While mixing, try to keep temperatures as low as possible to avoid the loss of unstable 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 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-SRM11 result submission website**.

- **Sub-Page 0 (Sample receipt acknowledgement), accessible from 5 April, 2016**
- **Sub-Pages 1-3 (analytical results and method information) accessible from 7 April till 20 May, 2016.**

To access the data-submission forms participants must use their unique login data (username and password) that will be provided to them before sample shipment. The unique EUPT-SRM11 lab-codes will be provided in a separate e-mail.

The deadline for result submission is 20 May, 2016 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-SRM11 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 the 8 April, 2016. 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 8 April 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-SRM11 Result Submission Website**.

All results must be reported on this website by 20 May, 2016 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, which are not necessarily 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” 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.
- **“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.
- **“Did you use an Internal standard (IS)”**: Please only choose **one of the three “Yes” options if the IS was used for calculation of the result of the target analyte (not merely for quality control purposes)**. Please choose **“No”** if the IS was only used for quality control purposes and not for the calculation of the target analyte result.
 - **Yes, Isotope labelled analogue of target analyte (ILIS)** if you have employed a real ILIS as defined in the SANTE document (e.g. Ethephon D₄ for Ethephon)
 - **Yes, other IS** if you have employed a compound other than ILIS as internal standard (e.g. MCPA or MCPA D₃ for 2,4-D or TPP, Chlorpyrifos D₁₀, Nicarbazin)
- **“At what stage of the procedure did you employ the IS”**:
 - **at the beginning** if the IS was added to the sample portion directly or shortly following extraction solvent addition
 - **to an aliquot of the final extract** if the IS was added to an isolated aliquot of the final extract
 - **at an intermediate stage** if the IS was added at any stage in-between the above two
- **“Other means of recovery or matrix-effect correction used”**
 - ***None***
 - ***Procedural calibration***
 - ***Standard addition to sample portions***
 - ***Standard addition to extract aliquots***
 - ***Matrix-matched calibration***
 - ***Use of a recovery factor (please indicate recovery figure)***
 - ***Other (please specify the details)***
- **“Recovery figure (in %)”**: Here laboratories 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-SRM11 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.

Subcontracting

The following task was subcontracted to the EURL-FV, Almería, Spain:

- a) Production, preparation and shipment of Test Items and Blank Materials for the EUPT-SRM11

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

- a) Administration of EUPT-SRM11 registration and result submission website

Follow-up actions

After the distribution of the EUPT-SRM11 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-SRM11-participants are welcome to ask the EURL-SRM for technical assistance.

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–SRM11 can be found in the [EURL-Documents Repository \(CIRCA/FIS-VL\)](#). Links to the documents can also be found in the [EUPT-SRM11 Website](#).

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

Please check the [EUPT-SRM11 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: 230 €
- Labs based in third countries: 350 €

An invoice issued for the "invoice address" stated in the registration form will be sent to the e-mail address(es) responsible for the PT. Should the payment being take care of by another department/institution the participating laboratories are 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 not to exclude the results of the concerned laboratories from the Final EUPT-Report or to refuse participation in future EUPT-SRMs.

Please note:

The bank account of EURL-SRM has been changed since the end of October 2013! Please ask your financial department to update the bank account of EURL-SRM!

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:	<i>See invoice (important and MUST 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 will be given in the invoices.

Calendar of EUPT-SRM11

(please see: http://www.eurl-pesticides.eu/userfiles/file//EUPT-SRM11_Calendar.pdf)

Target Pesticides List of EUPT-SRM11

(please see: http://www.eurl-pesticides.eu/userfiles/file//EUPT-SRM11_TargetPesticideList.pdf)

Contact information

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