

SPECIFIC PROTOCOL

for the 17th EU Proficiency Test on Pesticides requiring Single Residue Methods EUPT – SRM17 (2022)

(Update on 23 February 2022)

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. 9" covering all EUPTs in 2020, 2021 and 2022.

This PT is organized by the EU Reference Laboratory for pesticides requiring Single Residue Methods (EURL-SRM). The EU Reference Laboratory for pesticides residues in Fruits and Vegetables (EURL-FV)) has collaborated in the production of PT Test Item. Both EURLs are accredited as providers of proficiency tests according to ISO 17043 (please see EURL-SRM accreditation see EURL-FV accreditation).

The EUPT-SRM17 deals with the analysis of SRM-pesticides in tomatoes. Participation is obligatory for all National Reference Laboratories for Single Residue Methods (NRL-SRMs), as well as for all official EU laboratories (OfLs) involved in the official analyses of pesticide residues in fruits and vegetables. The tentative classification of labs into "obliged" and "not obliged" to participate in this PT was based on information on the scope of commodities covered, as stated within the EURL Data-Pool. Prior to the classification, the laboratories were asked to update this information within the Data-Pool and the responsible NRLs were asked to verify this information.

The registration of the labs to the PT was run through the EUPT-Registration website, which is connected to the EURL-DataPool. All laboratories classified as obliged were notified that they should enter the online registration platform irrespective if they intend to participate or not. In the latter case, the labs had to state their reasons for non-participation. The reasons for non-participation from obliged laboratories received during registration, especially details considering the scope, will be considered in the final list of obliged laboratories. The registration period started on 6 December and ended on 31 December 2021.

The most important documents related to this PT can be accessed via the EUPT-SRM17-Website.

Test Item

The Test Item of this EUPT is "Tomatoes Homogenate".

Participants will receive one Test Item bottle containing ~350 g deeply frozen tomatoes homogenate with incurred and spiked analytes from the Target Pesticides List. The field-treated tomatoes were milled at ambient temperature, then spiked with additional pesticides and homogenized further first at ambient temperature and then in addition cryogenically

using dry ice. The final material was filled into the bottles in a snow-like state, so that the analytical portions can be easily withdrawn. Shipment will be done in frozen condition with the addition of dry ice into the parcels. <u>NO</u> Blank material will be sent to the participants for this PT.

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.

Target Analytes and MRRLs

The Test Item will contain several analytes from the mandatory and optional section of the EUPT-SRM17 Target Pesticides List. Laboratories should read the Target List carefully as it shows how the residues should 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-SRM17 Target Pesticides List before starting with analysis and reporting results.

Shipment of Test Item

Dispatch of the Test Item is planned on 31 January 2022.

Test Item will be packed into thermo-boxes together with dry ice and will be shipped from Germany via DHL Express 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 Organizers 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.

Where complications during customs clearance or shipment are expected, the participants should provide the Organizers in advance (by 21 January) with detailed contact Information (e.g. mobile phone numbers of laboratory personnel) and all necessary documents to be stuck/attached on the package to ensure smooth customs clearance. Such documents may be e.g. a permission for importing organic material for scientific purposes (lab analysis) or an instruction in local language to explain the need to keep the package in a freezer in case of delay during shipment or clearance at customs.

After the shipment is tracked within the DHL delivery system and the waybill is printed out, the <u>main contact person</u> for the PT will be informed by DHL on the <u>tracking number of his package</u>. The participants can track their own packages online and <u>must make any necessary arrangements to receive the delivery</u>.

IMPORTANT:

In case of delays at the customs or any other unusual delays within the recipient's country, the participants themselves are strongly encouraged to contact the 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, the Test Item should be stored deeply frozen (at -18°C or lower) until analysis in order to avoid any possible deterioration/spoilage of the sample material and to minimize analyte losses.

The Test Item was prepared and shipped in way allowing analytical portions to be taken directly without additional milling or thawing. Should you notice the formation of ice-crystals or signs that the material was thawn during transport,

it is recommended mixing the material thoroughly in its entirety before analytical portions are taken. While mixing, keep temperatures as low as possible to avoid thawing and the potential loss of unstable pesticides.

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-SRM17 result submission webtool.

The homogeneity tests will be conducted using 10 g or 20 g portions. As sub-sampling variability increases with decreasing analytical portion size, sufficient homogeneity can be guaranteed only for sample portions roughly 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-SRM17 Result Submission Webtool:**

- Sample receipt acknowledgement: From 1 Feb. 2022 onwards.
- Reporting of Analytical results and method information: 1 Feb. 8 Mar. 23:30 h CET.
- Deadline for result submission is 8 March, 11.30 pm (CET), 2022.
- Reporting of <u>additional information on methods</u> used <u>for tentatively false negative results</u>: 9 16 March, 2022.

A guideline for the new **EUPT-SRM17 Result Submission Webtool** will be provided to the participants in due time and a link to it can also be found in the info-box on the Webtool. The **participants are urged to read it carefully before submitting their results**.

- Login credentials and lab code

To access the EUPT-SRM17 Result Submission Webtool, participants must use their <u>PERSONAL</u> LOGIN CREDENTIALS (username and password). <u>The personal login credentials will be provided to the PT-contact persons by DTU prior to opening of the webtool for the EUPT-SRM17.</u>

The lab's <u>unique lab code for the EUPT-SRM17</u> will be provided to the participants following the first access to EUPT-SRM17 Result Submission Webtool.

- Acknowledgement of package receipt and acceptance of PT-materials

Once the laboratory has received the package with the PT material, it must report to the organizer via the EUPT-SRM17 Result Submission Webtool the date of receipt, the condition of the package, the condition of the test material at arrival, whether the material is accepted or not, and any other comments concerning the test material. This task should be finalized by 4 February 11:00 am CET. If a laboratory does not respond by this deadline, the Organizers will assume that the Test Item has been received and accepted. In case of problems with the sample receipt or condition, please contact the organizers additionally via e-mail ASAP (eurl-srm@cvuas.bwl.de) to ensure that corrective actions are taken as early as possible. Please note that completing the sample receipt form is a pre-requisite for accessing the results submission areas. However, you can still access sample receipt form and edit it later.

Participants are encouraged to <u>follow the whereabouts of their parcels using the tracking code of the shipping company</u> that they will receive via e-mail and intervene at the shipping company, the customs or the organizers if they notice any delays. Any participants not having received the Test Item by the Fri. 4 February, at 10:00 am CET, must inform the Organizer via e-mail (EURL-SRM@cvuas.bwl.de) immediately but not later than <u>Fri. 4 February 11:00 am CET</u>. The Organizer 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-SRM17 Result Submission Webtool.

All results must be reported on this website by <u>8 March, 11.30 pm (CET), 2022.</u> The pages for the "scope, detected and results" will not be accessible after this deadline, and no results submitted afterwards will be accepted.

Before entering the results, please study the **EUPT-SRM17 Target Pesticides List** carefully. Please note, that the residue definitions applying to the EUPT may appear in a shortened form on the result submission website.

If a lab routinely subcontracts analyses of one or more compounds to another lab, this subcontracted lab may (and is even encouraged to) also take part in the EUPT exercise.

IMPORTANT:

The participants are obliged to inform the organizers of all cases where PT results were generated by subcontracted labs, and to provide details on the subcontracting laboratory. This also applies where the subcontracted lab belongs to the same institution/company but runs its own quality control system.

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.

If a pesticide was not detected or if it was detected but the quantitative result is below the RL (Reporting Limit) of the laboratory or the MRRL, no result should be reported. In accordance with the General Protocol, results reported as "< RL" or "< #.# mg/kg" will be judged as "false negatives" if the concerned analyte is present in the test material at levels ≥ 3 times the MRRL.

The residue levels of the pesticides must be reported in mg/kg using **three significant figures**: e.g. 0.0582; 0.156, 1.64, 20.3 mg/kg.

Where a target analyte on the target pesticide list is defined as the sum of two or more components, a result for this "summed target analyte" should only be reported if

- a) the method used covers the entire residue definition of this "summed target analyte", e.g. if the method involves a chemical conversion to one component, or
- b) if all individual components entailed in this residue definition were targeted.

In the latter case, the concentrations of the individual components of the "summed target analyte" should be added-up and expressed as stated in the residue definition on the target pesticide list. If at least one of the components within the "summed target analyte" was not analysed, this "summed target analyte" should be marked as "not analysed". In case one of the components within the complex residue definition was not encountered at a quantifiable level (<RL), its concentration should be considered equal to zero when calculating the summed result.

Recovery-corrected results should be reported only if this reflects the lab's actual or envisaged routine procedure. Where a **result was corrected for recovery**, the approach(es) applied 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.

- **Reporting Limit (RL) in mg/kg:** the lab's reporting limit for an analyte.

Where two or more components of a complex residue definition are analyzed individually, the RL of the sum is also formally required. It should be calculated by summing up the individual RLs of the constituent components expressed as prescribed by the residue definition (applying conversion factors based on the molecular weight of the components). The individual RLs of each component (without conversion) can be reported in the respective fields of the individual components or, if these are not available, in "Comments" field of the sum analyte. Where the analytical method for the analysis of a complex residue definition involves a chemical transformation, thus generating a single analytical result, the RL of the method is to be reported, but again expressed as prescribed by the residue definition.

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

- Reporting Information on Analytical Methodology

On the page of "Edit methods" of EUPT-SRM17 Result Submission Webtool the participating laboratories have to provide information on the analytical method(s) applied for the analysis of the target analytes detected in the Test Item.

The participating laboratories are urged to thoroughly fill-in all requested information.

IMPORTANT:

If entries in required fields within the Result Submission Webtool are missing, you will not be able to proceed with the final submission. Therefore, please fill-in your method information in due time to be on the safe side.

For detailed information on how to fill-in the columns on the "Edit methods" page, please refer to the <u>Guideline for Results Submission</u> that will be distributed to all participants in due time. A link to this guideline can also be found in the <u>info-box on the Result Submission webtool</u>. Please also read the <u>mouse-over messages</u> popping-up when your mouse cursor meets a field name in the table header for a few seconds.

- Submission of results

Once you have entered all your results and checked their correctness, you have to submit them by clicking "Final Submission" button before the submission deadline. The "Final submission" button can be found at the bottom of each page. To avoid accidents, a confirmation is requested after clicking the "Final Submission" button.

IMPORTANT:

Following "Final Submission", 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

After the results submission deadline, **if a laboratory has obtained a tentatively false negative result**, it will be asked to enter the method information for this analyte, within 7 working days.

Establishment of assigned values

In addition to OfLs from EU Member States, EFTA countries, a limited number of laboratories from EU candidate countries and third countries are allowed to take part in this exercise. For the establishment of the assigned values only results submitted by OfLs from EU and EFTA countries are taken into account.

Subcontracting

The following tasks were subcontracted to the EURL-CF, Lyngby, Denmark:

- a) Generation of login credentials
- b) Programming and administration of EUPT-SRM17 result submission website

Follow-up actions

After the distribution of the EUPT-SRM17 Preliminary Report, laboratories having submitted poor results (high absolute z-scores, false negatives or false positives) will be asked to investigate the reason behind the poor performance, and to report their insights and possible corrective actions to the Organizer. This information will be forwarded to the corresponding NRL-SRMs upon request. All EUPT-SRM17-participants are welcome to ask the EURL-SRM for technical assistance.

In the course of results evaluation, the organizer may ask laboratories to provide additional methodology information relevant to the evaluation and interpretation of the PT.

According to instructions from DG-SANTE, the "Protocol for management of underperformance in comparative testing and/or lack of collaboration of NRLS" is to be followed by NRLs.

Documents

All documents related to the EUPT–SRM17 can be downloaded from EUPT-SRM17 Website or EURL-Document Repository (CIRCA-BC).

For any questions, please contact the organizers EURL-SRM@cvuas.bwl.de

IMPORTANT:

Please check the **EUPT-SRM17 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 in PDF format issued to the "invoice address" stated in the registration form will be sent <u>after the results</u> <u>submission deadline</u> to the "invoice e-mail address" stated in the registration form and to the PT main contact person. If the payment is being taken care of by another department/institution, the recipient of the invoice is requested to timely forward the invoice to the responsible persons. Details on payment are given in the invoices.

<u>Additional cost may occur</u> if extra services are requested in relation with the payment, such as the completion of additional paperwork and the generation of a new modified invoice in order to include information that was missing or incorrectly provided during registration.

IMPORTANT:

The EURL-SRM will issue digital invoices in PDF format only and without any electronic signature.

If, due to locally applying legal requirements, a participating laboratory needs an electronic invoice (e.g. certificed or signed e-invoice in XML), it has to provide the PT-Organizers a suitable and free tool to generate the necessary e-invoice and provide full assistance in case this tool requires the use of a language other than English. Otherwise, the PT-Organizer will not issue an e-invoice. Depending on the incurring extra workload, the participating laboratory may be charged for this extra service.

The EURL-SRM will not complete any special form required by the participating laboratories for their financial department or payment office and does not agree to give any personal or private data for this purpose. If completion of such forms is prerequisite for payment in your laboratory, please fill in the forms yourself based on the data in our financial identification note (https://www.eurl-pesticides.eu/library/docs/srm/SRM-Bank_m_Financial_Identification.pdf) and send us the filled form. After verification, and if necessary correction, we will return it with signature and stamp.

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

If, for any reason, payment cannot be carried out before this date, please contact the Organizers 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 issue the participation certificate for the concerned laboratory, to exclude its results and its name from the Final EUPT-Report, and 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 (This number <u>MUST</u> be indicated in the payment!)

VAT of CVUA Stuttgart DE 811 600 510

Please note:

- Please do not make any remittance before you receive the invoice with the Payee Identification Text.
- 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 is participating in both PTs (EUPT-SRM17 and EUPT-AO17), please ask your financial department to transfer the fee for each of the PTs separately using the corresponding <u>payee identification text</u> (= invoice number) given in each invoice. Without this text, your payment will not be able to reach the correct EURL.

Calendar of EUPT-SRM17

(please see https://www.eurl-pesticides.eu/userfiles/file/EurlSRM/EUPT-SRM17_Calendar.pdf)

Target Pesticides List of EUPT-SRM17

(please see http://www.eurl-pesticides.eu/userfiles/file/EurlSRM/EUPT-SRM17_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

Organizing Group at the EURL-SRM (Stuttgart)

Michelangelo Anastassiades phone: +49 3426 1124
Pat Schreiter phone: +49 3426 1029
Ann-Kathrin Wachtler phone: +49 3426 1151
Hubert Zipper phone: +49 3426 1141
Anja Barth phone: +49 3426 1935
Giovanna Cerchia phone: +49 3426 1120

Quality Control Group

Paula Medina European Food Safety Authority (EFSA)
Antonio Valverde Professor at University of Almería, Spain

Advisory Group

Amadeo R. Fernández-Alba Co-Head of EURL FV, University of Almería, Spain Carmen Ferrer Amate Co-Head of EURL FV, University of Almería, Spain

Magnus Jezussek LGL; Erlangen, Germany

André de Kok Formerly working at NVWA, Wageningen, The Netherlands

Björn Hardebusch Head of EURL AO, CVUA Freiburg, Freiburg, Germany

Ralf Lippold Head of CVUA Freiburg, Germany

Hermann Unterluggauer AGES; Institute for Food Safety Innsbruck, Austria.

Hans Mol Wageningen Food Safety Research, Wageningen, The Netherlands

Finbarr O'Regan Pesticide Control Laboratory, Celbridge Co. Kildare Ireland

Patrizia Pelosi Istituto Superiore di Sanità, Rome, Italy
Tuija Pihlström National Food Agency, Uppsala, Sweden

Mette Erecius Poulsen Head of EURL-CF, DTU National Food Institute, Lyngby, Denmark

Radim Štěpán Czech Agriculture and Food Inspection Authority, Prague, Czech Republic