

SPECIFIC PROTOCOL

for the 19th EU Proficiency Test on Pesticides requiring Single Residue Methods EUPT – SRM19 (2024)

(released on 19 January 2024)

Introduction

This protocol is complementary to the valid version of the "General Protocol for EU Proficiency Testings for Pesticide Residues in Food and Feed, 11th Ed." for all EUPTs in 2024.

The EUPT-SRM19 is organized by the EU Reference Laboratory for pesticides requiring Single Residue Methods (EURL-SRM), named "organizers" in the following. The EURL-SRM is an accredited provider of proficiency tests according to ISO 17043 (see EURL-SRM accreditation).

The EUPT-SRM19 deals with the analysis of SRM-pesticides in grape homogenate. 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 by each laboratory, as stated within the EURL DataPool. Prior to the classification, the laboratories were asked to update this information within the DataPool and the responsible NRLs were asked to verify this information.

The registration of the labs to the PT was run within the DataPool website. 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 received from obliged laboratories during registration, especially details considering the scope, will be considered in the final list of obliged laboratories.

Communication

On matters concerning the EUPT-SRM19, the organizers will communicate with the participating laboratories via emails to the respective "Main Contact Persons" and "Alternative Contact Person(s)" stated in the EUPT-SRM19 registration form. These persons are in the following referred to as "participants" or "PT-participants".

Additional emails will be automatically issues by the Webtool.

The most important documents related to this PT can be accessed via the EUPT-SRM19-Website.

PT Item

The PT Item of this EUPT is "Grape Homogenate".

Participants will receive one bottle of PT Item containing approximately 400 g deeply frozen grape homogenate with incurred and spiked analytes from the Target Pesticides List. Deeply frozen grape was cryogenically milled, spiked with selected compounds and homogenized at low temperature. Additional cryogenic milling using dry ice was carried out. The final material was filled into the bottles in a snow-like state, and will be sent with the addition of dry ice into the parcels, so that the analytical portions can be easily withdrawn. NO Blank material will be sent to the participants for this PT.

Using randomly chosen bottles, the Organisers will check the PT Item for sufficient homogeneity and for the stability of the analytes contained over the period of the exercise.

Target Analytes and MRRLs

The PT Item will contain several analytes from the mandatory, optional and extra section of the EUPT-SRM19 Target Pesticides List (TPL). Laboratories should read the TPL 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. Make sure to download and carefully study the latest version of the EUPT-SRM19 Target Pesticides List before starting with analysis and reporting results.

Shipment of PT Item

Dispatch of the PT Item is planned on 5 February 2024.

PT 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 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.

IMPORTANT:

The PT participants are responsible for facilitating quick customs clearance.

In case of delays at the customs or any other unusual delays within the recipient's country, the participants will be informed by the DHL and are strongly encouraged to contact the local DHL Express office and/or the customs in order to accelerate the clearance and delivery procedures and/or to ensure that the parcel is stored in a freezer during the delay period.

The PT participants are responsible for facilitating quick customs clearance. Where complications during customs clearance or shipment are expected, the participants should provide the Organisers in advance (by 25 January) with all necessary documents to be stuck/attached on the package or to be uploaded, in order to ensure a smooth customs procedure. Such documents may include a permission for importing organic material for scientific purposes (analysis) or an instruction in local language indicating the need to keep the package in a freezer in case of delay during shipment or custom's clearance. The participants should also inform the organizers if a phytosanitary certificate of the PT-material is required by their countries. Where the organizers will not be able to obtain the required phytosanitary certificate, the shipment (and the PT-participation) may need to be cancelled.

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 must make any necessary arrangements to receive the parcel upon delivery.

Instructions on Handling the PT Item

Once arrived, the PT Item should be kept deeply frozen (at -18°C or lower) <u>until analysis</u> in order to avoid any possible deterioration/spoilage of the sample material and to minimize analyte losses.

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-SRM19 result submission Webtool.

The homogeneity tests will be conducted using 10 g portions in the case of pesticides and a 1 g portion in the case of copper. 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-SRM19 Result Submission Webtool:

- Sample receipt acknowledgement: From 6 February, 2024 onwards.
- Reporting of analytical results and method information: 12 February 12 March 11:30 pm (23 h) CET.
- Deadline for result submission is <u>12 March</u>, <u>11 pm</u> (<u>23 h</u>) (<u>CET</u>), <u>2024</u>.
- Reporting of additional information on methods used for tentatively false negative results: 13 21 March, 2024.

A guideline for the new EUPT-SRM19 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-SRM19 Result Submission Webtool, participants must use their <u>PERSONAL</u> LOGIN CREDENTIALS (username and password).

Only persons listed as Main or Alternative Contact Persons for the EUPT-SRM19 will have access to the EUPT-SRM19 section within the Webtool. Prior to opening the EUPT-SRM19 section within the Webtool, the DTU will send a personalized <u>username</u> to each participant via email.

The participants can retrieve their personalized passwords via the following link using either their received usenames or the email address stated during registration: https://guest.dtu.dk/Sites/GuestLogin/RetrievePassword.aspx

PT participants can change their password using the following link: https://guest.dtu.dk/Sites/GuestLogin/Default.aspx For security reasons you are encouraged to update your password once a year.

The lab's <u>unique lab code for the EUPT-SRM19</u> will be provided to the participants following the first access to EUPT-SRM19 Result Submission Webtool upon submitting the sample acknowledgement at the first time. Since the lab code is absolutely necessary for the whole PT organisation, please access the EUPT-SRM19 Webtool and submit sample acknowledgement as soon as possible, once this page becomes accessible and the package with the PT Item arrives your laboratory.

Acknowledgement of Package Receipt and Acceptance of PT Item

Once the laboratory has received the package with the PT Item, it must report to the organiser via the EUPT-SRM19 Result Submission Webtool the date of receipt, the condition of the package, the condition of the PT Item at arrival and any other comments concerning the test material. In case of problems with the sample receipt, sample condition or complaints, the sample receipt form should be completed as soon as possible and not later than 9 February 11:00 am CET to ensure that corrective actions can be taken as early as possible. If a laboratory does not respond by this deadline, the Organisers will assume that the PT Item has been received and accepted.

Participants are encouraged to <u>follow the whereabouts of their parcels using the tracking number of the shipping company</u>, which they will receive via e-mail, and to intervene at the shipping company, the customs or the organisers if they notice any delays. Any participants not having received the PT Item by the afternoon of Thu. 8 February must inform the Organiser via e-mail (EURL-SRM@cvuas.bwl.de) as soon as possible and <u>not later than 9 February 11:00 am CET</u>. The Organiser will consult the shipping company to localize the package and decide on further actions, e.g. arrange a new shipment if necessary.

Please note that <u>saving and closing the sample receipt form is a pre-requisite for accessing the results submission areas</u> <u>and generating participants' lab codes for this PT</u>. However, you can still access sample receipt form and edit it later.

Reporting of Results

To report their results, participants must access the EUPT-SRM19 Result Submission Webtool.

All results must be reported on this website by <u>12 March</u>, <u>11:00 pm (CET)</u>, <u>2024</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-SRM19 Target Pesticides List carefully. Please note that the compound names within the Webtool may appear in a shorter form than in the TPL. Please refer to the EUPT-SRM19 Target Pesticides List for the actual residue definitions applying to the present PT.

IMPORTANT NOTE CONCERNING OUTSOURCING OF ANALYSES

If <u>routine</u> procedures foresee the analysis of certain analytes (e.g. **copper**) by another laboratory*, this practice should be reflected in the PT.

When reporting PT-results participants are obliged to inform the organisers of any outsourced analyses and to provide the details of the laboratories having conducted these analyses.

The above information serves the transparency and allows identifying cases, where multiple results originate from a single source, which may need to be considered when establishing the assigned value.

Please note, that the information concerning the outsourcing of analyses may be highlighted in the PT-certificates.

* This also applies to cases where the analysing laboratory 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, no result should be reported.

The residue levels of the pesticides should be reported in mg/kg preferably using **three significant figures**,: e.g. 0.0585; 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 targeted but not encountered at a quantifiable level (<RL), its concentration should be considered equal to zero when calculating the summed result.

Bias-corrected results should be reported only if this practice reflects the lab's actual or envisaged routine procedure. Where a **result was corrected for bias**, 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 the "Comments" field of the analyte with the complex residue definition. 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 of Information on the Analytical Methodologies Applied

On the page of "Edit methods" of EUPT-SRM19 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 PT Item.

The participating laboratories are urged to thoroughly fill-in all requested information as this information may serve in localizing methodology-related systematic bias.

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 info-box on the Webtool.

For quick information please 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 or 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, typically only results submitted by OfLs from EU and EFTA countries are taken into account.

Subcontracting/External Services

The following tasks are conducted by the EURL-CF, Lyngby, Denmark:

- a) Generation of login credentials
- b) Generation of Lab Codes in the present PT
- c) Programming and administration of EUPT-SRM19 result submission website

Follow-up Actions

After the distribution of the EUPT-SRM19 Preliminary Report, laboratories having submitted poor results (absolute z-scores > 2, 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 organiser. This information will be forwarded to the corresponding NRL-SRMs upon request. All EUPT-SRM19 participants are welcome to ask the EURL-SRM for technical assistance.

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

NRLs should take into account the "Protocol for management of underperformance in comparative testing and/or lack of collaboration of NRLS" by DG-SANTE.

Documents

All documents related to the EUPT–SRM19 can be downloaded from the EUPT-SRM19 Website or the EURL-Document Repository (CIRCA-BC).

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

IMPORTANT:

Please check the **EUPT-SRM19 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 €

After the shipment, the EURL-SRM will issue an invoice in pdf format directed to the "invoice address" stated in the registration form. The invoice will be sent to the invoice contact person as well as to the PT-contact persons stated in the registration form. Should the payment being taken care of by another department/institution, the recipient of the invoice is requested to timely forward the invoice accordingly. **Details on payment will be given in the invoices**. The participants should get informed about the basic requirements of their payment system and are responsible for the correctness of the invoice data stated during registration.

As stated in the **General EUPT Protocol**:

- 1) every lab that has registered for participation in the EUPT-SRM19 and received the test material in good condition has to pay the total fee, irrespective of whether results are submitted or not. This also includes cases where a lab realizes with-in the course of the PT that none of the compounds it has targeted is present at a quantifiable level in the PT-material, or if it realizes that for whatever reasons it cannot conduct any analyses or it cannot submit any results.
- 2) the EURLs 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. certificated or signed e-invoice in XML or using a special billing platform to generate and submit an e-invoice, 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.
- 3) Additional cost may occur if extra services are requested in relation to 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.
- 4) The EURLs will not complete any special forms required by the participating laboratories for their financial department or payment office. If completion of such forms is prerequisite for payment in the institution of the participating laboratory, this laboratory or its payment office is requested to fill-in the forms based on the data in the financial identification note (https://www.eurl-pesticides.eu/library/docs/srm/SRM-Bank_m_Financial_Identification.pdf) and to send the pre-filled form to the EUPT Organisers. The EURLs

are willing to provide any information required in the form as long as it is readily available, but they do not agree to provide any personalized (private) data for this purpose. After verification, and if necessary correction, of the information, the EUPT Organisers will return the form 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 Organisers.

If, for any reason, payment cannot be carried out before this date, please contact the Organisers to give explanations.

If no payment or no proof of payment is received and no explanation is given to the Organisers, the Organisers 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 MUST be indicated in the payment!)

VAT of CVUA Stuttgart DE 811 600 510

Please note:

- Do not make any remittance before you receive the invoice with the <u>Payee Identification Text</u>.
- EURL-AO (@ CVUA Freiburg) and EURL-SRM (@ CVUA Stuttgart) belong to the same Ministry and have thus the same bank account.

If your laboratory is participating in both PTs (EUPT-SRM19 and EUPT-AO19), please ask your financial department to transfer the fee for each of the PTs separately using the respective <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-SRM19

(please see https://www.eurl-pesticides.eu/userfiles/file/EurlSRM/EUPT-SRM19_Calendar.pdf)

Target Pesticides List of EUPT-SRM19

(please see https://www.eurl-pesticides.eu/userfiles/file/EurlSRM/EUPT-SRM19_TargetPesticideList.xlsx)

Contact Information

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