

SPECIFIC PROTOCOL

2nd Interlaboratory study on the quantification of pyrethroids in a standard solution

„Pyrethroids (ILS Pyr-02)“

Version 1.0

(22.04.2024)

➤ 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. 11¹. This interlaboratory study (ILS) is voluntary for all laboratories. The test solution will be a standard solution without matrix.

➤ PT item (test material)

This interlaboratory study concerns the analysis of pyrethroids in a standard solution. The test material contains only the spiked pyrethroids dissolved in solvent (acetone).

All these tests will be conducted by the EURL-AO which is accredited according to ISO 17043 for organising proficiency tests. The participants will receive 1 mL of a standard solution (one ampoule) containing spiked pesticides. The test material is manufactured by an external company. The label of the company is removed and replaced by a label of the EURL AO.

➤ Analytical parameters

The test solution contains 10 different pyrethroids dissolved in acetone given in the annex of this document. Please analyze all analytes. Laboratories should be attentive to the given concentration range of 50-250 µg/ml and adjust the concentration to their own calibration levels. Use at least 4 calibration levels for the quantification. The ILS Pyr-02 standard solution should be independently diluted twice (content 1.x and 2.x in Excel Report Form) with the same dilution factor. Each dilution is injected and measured twice (content x.1 and x.2 in Excel Report Form). The dilution factor used is asked in the EU survey.

¹ <http://www.eurl-pesticides.eu/docs/public/tmpl/article.asp?CntID=821&LabID=100&Lang=EN>

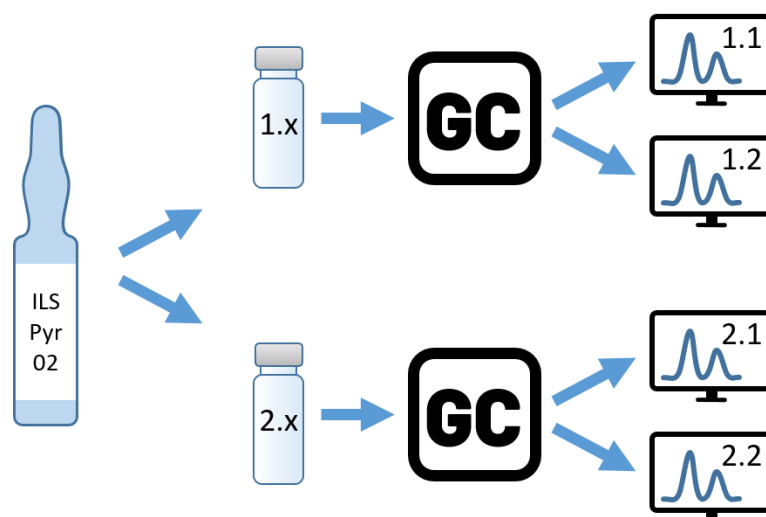


Figure: Procedure for measuring and labelling the various measurements

Please, report important observations during analysis in EU Survey in the special field for comments.

➤ **Shipment of PT item**

Dispatch of the PT item is planned on **06 May 2024**.

PT items will be shipped in tubes in a normal box with no further cooling. The organisers will aim to ensure that all participating laboratories will receive their PT items as soon as possible. PT items will be shipped with TNT/FedEx. Prior to shipment an e-mail will be sent to the participating laboratories from the shipper.

Laboratories must make their own arrangements for the receipt of the package. They should inform the organiser of any public holidays in their country/city during the week of the shipment and must make all necessary arrangements to receive the shipment, even if the laboratory is closed.

➤ **Instructions on PT item handling**

Once received, the PT item shall be stored cooled (4 - 7 °C) to avoid any deterioration/spoilage and to minimise possible pesticide losses.

Bring the content of the PT item to room temperature before taking out any analytical test portion. All participants should use their own analytical measurement and their own reference standards for identification and quantification purposes.

➤ EU Survey and Deadlines

Each participating laboratory is assigned its own lab code. This code is important for reporting. Analytical results and other information (e.g. name of the manufacturer of the own standards used, the batch number, internal standard etc.) are to be submitted via the EU Survey (open in incognito or private window). The laboratory name provided by the participants will not be changed and will be stated on the certificate.

1. PT item receipt and acceptance

Receipt of the sample does not need to be confirmed by the laboratory. However, if participants have not received the PT items or there are any discrepancies, they must inform the organiser immediately by e-mail (eur1-pesticides@cvuafr.bwl.de) by the **13 May 2023 at noon**. Otherwise the EURL AO will assume that the laboratory has received the material in perfect condition.

2. Results and method submission

After shipment of the samples, from 06 May 2024 onwards, it is possible to submit the results by logging into the EU Survey. **The deadline for result submission is 02 August 2024 at 23.00 CET.**

To report the results, fill out the EU survey and download the Report Form Excel file. You have to name the Report Form file with your own lab code at the end. Fill out the table completely (each row) and upload the renamed, completed file again. Please do not complete the survey until you have entered all answers and completely filled out the Excel file.

Important: After the final submission it will NOT be possible to edit the results. The website will not be accessible after this date and any results reported after the deadline will not be included in the statistical treatment, or in the final report. Participants will receive an email confirming the submission of their results.

3. Reporting of qualitative and quantitative results

The concentration of the solution provided (not from self-made dilutions) as well as the dilution stage (dilution factor) must be stated.

Significant figures:

Residue levels shall be expressed to **four** significant figures, e.g. 74.22, 91.15 or 243.8 µg/ml.

Attention: *The number of significant figures has been changed compared to announcement.*

4. Reporting information on analytical methodology

All laboratories are requested to provide information on the analytical method(s) they have used via EU Survey. It is mandatory to state the requested informations. The laboratories are requested to fill-

in this information in order to minimise the administrative burden of collecting this information at a later date.

➤ **Documents**

In the EURL-document repository (CIRCA BC) all documents relating to ILS Pyr-02 can be found. Links to most of the documents are also available on the ILS Pyr-02 website (https://www.eurl-pesticides.eu/docs/public/tmpl_article.asp?LabID=300&CntID=1267&Theme_ID=1&Pdf=False&Lang=EN).

➤ **Subcontracting**

The following tasks were subcontracted: Production of the standard solution

➤ **Time schedule**

Actor	Activity	Date
EURL	Preliminary announcement at Joint Workshop in Stuttgart	20 October 2023
EURL	First information supplied to laboratories and call for participation	Beginning of February 2024
Participant	Registration via EUPT website (EURL-Datapool)	15 December 2023 – 01 April 2024
Participant	Proof of shipment address in EURL-Datapool	Ending 26 April 2024
EURL	Dispatch of test material	06 May 2024
Participant	Information of EURL AO, if no sample was received	07 – 13 May 2024
Participant	Deadline for reporting of test results via EU Survey	02 August 2024*
EURL	Dispatch of the final report as pdf-file	Approx. end of 2024

* Please make sure to report your results on time as there will be **no extension of the deadline**.

➤ **Participation fee**

There is a **fee of EUR 100.00** for shipping and handling to participants within the European Union and EFTA countries (**including NRLs**). Fees for participants from **other countries** are **EUR 200.00**. An invoice will be sent as pdf-file via e-mail.

The EURL AO in conducting three studies in 2024. Participation in all three studies will result in one shipment (also if you take part in two studies). You always have to pay the full fee for EUPT AO 19. If participating in other studies you will be charged with extra costs (50 Euro for labs from EU and EFTA countries and 100 Euro for other countries per study). The maximum costs will be 300 Euro for labs from EU and EFTA countries and 500 Euro for labs from other countries participating in all three studies.

➤ **Delays in Payment**

The participants will receive an **invoice as pdf-file via e-mail** to the corresponding e-mail address given during registration. Laboratories wishing to additionally receive an invoice in paper form should write the request to eurl-pesticides@cvuafr.bwl.de before **29 April 2024**. Please make sure that the payment is made before the stipulated deadline stated on the invoice (**28 June 2024**). If the invoice is not paid within the stipulated time, reminders will be sent within a four week period.

From the second reminder onwards an administration fee of 25 € will be charged per reminder. Based on Reg. (EC) 625/2017, OfLs not paying the EUPT or ILS sample delivery fee will be initially warned that their participation in subsequent EUPTs could be denied. In case of a repetitive non-

payment, the EUPT organisers will inform the corresponding NRL or the competent authority to take action.

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Annex

EUPT-AO19 Pesticide target list of mandatory analytes

Table A1: List of **pyrethroids** analytes in ILS Pyr-02. Results shall be rounded to four significant figures (e.g. 74.22, 91.15 or 243.8 µg/ml)

Analyte	CAS-Number
Resmethrin	10453-86-8
Fenvalerate	51630-58-1
Cypermethrin	52315-07-8
Permethrin (cis & trans)	52645-53-1
Deltamethrin	52918-63-5
Cyfluthrin	68359-37-5
Tetramethrin	7696-12-0
lambda-Cyhalothrin	91465-08-6
Bifenthrin	82657-04-3
tau-Fluvalinate	102851-06-9

Attention: The CAS-number of Resmethrin has been changed compared to announcement.