

SPECIFIC PROTOCOL

3th Interlaboratory Study on the Determination of alpha-Cypermethrin in Animal Fat and Liquid Cream

"alpha-cypermethrin (ILS aC-03)"

(09.04.2025)

Version 1.1: Registration link has been changed (11.04.2025)

> 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. 12¹. The current interlaboratory study covers only alphacypermethrin in two different matrices. This interlaboratory study (ILS) is voluntary for all laboratories participating in this year's EUPT AO 20. This means that there are no additional costs for the participants due to the joint shipping of the PT items. The commodity chosen to prepare the test material is animal fat (porcine) and liquid cream. They are considered as a representative commodity for "milk and milk products, Group 8" and "fat from food of animal origin, Group 10" (see Annex A of document SANTE 11312/2021v2)².

PT item (test material)

This interlaboratory study concerns the analysis of alpha-cypermethrin in liquid cream and animal fat. The matrices contains only spiked alpha-cypermethrin (CAS-Number 67375-30-8).

Since 2020, the EURLs for pesticide residues do not provide blank PT items. Each laboratory is asked to use "pesticide free" representative animal fat and milk (product) for recovery experiments as well as for the preparation of matrix-matched or procedural calibration standards.

The organisers will check the PT items for sufficient homogeneity and for stability using conditions that reproduce sample shipment and storage for the duration of the proficiency test.

All these tests will be conducted by the EURL-AO which is accredited according to ISO 17043 for organising proficiency tests.

https://www.eurl-pesticides.eu/userfiles/file/EurlALL/General Protocol V12 2025.pdf

² https://food.ec.europa.eu/system/files/2023-11/pesticides mrl quidelines wrkdoc 2021-11312.pdf



The participants will receive ~ 50 g of animal fat PT item (one bottle) and ~ 80 g of liquid cream PT item (one bottle) containing spiked alpha-cypermethrin.

Analytical parameters

The PT items contain alpha-cypermethrin. Laboratories should adjust the concentration to their own calibration levels.

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

Shipment of PT item

Dispatch of the PT item was on 07 April 2025 together with the EUPT AO 20 PT items.

PT items will be shipped in bottles in a thermal isolated box with 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 and DHL. 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 frozen (-18°C) to avoid any deterioration/spoilage and to minimise possible pesticide losses.

Bring the content of the PT item slowly to room temperature without applying heat and homogenise (without shaking vigorously) the whole sample thoroughly before analysis, including any liquid that may be present due to the technical processing. Otherwise phase separation could occur for the liquid cream PT item!

It is recommended to divide the whole sample amount into analytical test portions and weigh them into the tubes used for extraction. All analytical test portions not used for analysis should be stored chilled. This procedure helps to avoid possible losses caused by several thawing steps of the test material.

All participants should use their own routine standard operating procedures for extraction, clean-up and analytical measurement and their own reference standards for identification and quantification purposes. Considering the available amount of PT item, laboratories employing methods requiring large analytical portions are advised to scale them down.



The general safety requirements and operating instructions for handling hazardous substances and their disposal must be observed.

The homogeneity tests were conducted using 10 g of the liquid cream PT item and 0.5 g of the porcine fat PT item using EN1528 method.

As sub-sampling variability increases with decreasing analytical portion size, sufficient homogeneity can only be guaranteed where participants employ analytical portions that are equal or larger than those stipulated in the previous sentence.

> 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 (eurl-pesticides@cvuafr.bwl.de) by the **14 April 2025 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 14 April 2025 onwards, it is possible to submit the results by logging into the EU Survey. The deadline for result submission is 30 May 2025 at 23:00 CEST.

The results report is provided via the following survey:

https://ec.europa.eu/eusurvey/runner/ILS_aC_03_Results

You will receive the password in advance together with your personal lab code for this study (not the EUPT AO20 lab code!)

<u>Important:</u> 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.

3. Reporting of quantitative results

Significant figures:



Residue levels shall be expressed to three significant figures, e.g. 0.0581, 0.251 or 1.35 mg/kg.

The expanded measurement uncertainty is queried purely on a voluntary basis for information purposes during result submission. The expanded measurement uncertainty is not intended to be published in reports, but is only used for internal plausibility checks.

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 information. The laboratories are requested to fill-in this information in order to minimise the administrative burden of collecting this information at a later date.

Follow-up actions

By sending the preliminary report to the participants, all participants are requested for a proof reading of the preliminary report. In particular the NRLs and OFLs with an individual z-score ≥ 3.0 are requested to give feedback to EURL-AO on any actions undertaken to find out the reasons for poor performance.

Documents

In the EURL-document repository (CIRCA BC) all documents relating to ILS aC-03 can be found. Links to most of the documents are also available on the ILS aC-03 website.

External supplier

No task was conducted by the assistance of external suppliers.



> Time schedule

Actor	Activity	Date
EURL	Announcement and first information for PT participants via e-mail	24 February 2025
Participant	Registration via EU survey	24 February – 21 March 2025
Participant	Proof of shipment address in EURL-Datapool	Ending 24 March 2025
EURL	Dispatch of test material	07 April 2025
Participant	Information of EURL AO, if no sample was received	08 - 14 April 2025
Participant	Reporting of results and method information	14 April – 21-May 2025
Participant	Deadline for reporting of test results	30 May 2025
EURL	Preliminary report	16 July 2025
Participant	Proof Reading of preliminary report	5 October 2025
EURL	Dispatch of the final report as pdf-file	Approx. end of 2025

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

> Participation fee

There will be <u>no</u> additional fee for participating in this study.

Only the fee for the EUPT AO 20 applies:

There is a fee of EUR 200.00 for shipping and handling to participants within the European Union and EFTA countries (including NRLs). Fees for participants from other countries are EUR 400.00. An invoice will be sent as pdf-file via e-mail.



EUPT-Coordinator:

Björn Hardebusch

Head of EURL-AO (Food of Animal Origin and Commodities with High Fat Content) CVUA Freiburg Department of Residues and Contaminants Bissierstrasse 5 D-79114 Freiburg

Phone: +49-761-8855-137

E-Mail: <u>Bjoern.Hardebusch@cvuafr.bwl.de</u> <u>eurl-pesticides@cvuafr.bwl.de</u>

Organisation Team at the EURL AO Freiburg:

Björn Hardebusch Head of EURL AO CVUA Freiburg Sarah Obermeier Food Chemist CVUA Freiburg

Quality Control Group:

Antonio Valverde University of Almería, Almería, Spain

Paula Medina Pastor EFSA, European Food Safety Authority, Parma, Italy

Advisory Group:

Amadeo R. Fernández-Alba Head of EURL-FV, University of Almería, Spain

André de Kok Wageningen Food Safety Research, Wageningen, The

Netherlands

Carmen Ferrer Amate EURL-FV, University of Almería, Spain

Finbarr O'Regan Pesticide Control Laboratory, Department of Agriculture,

Fisheries and Food, Kildare, Ireland

Hans Mol Wageningen Food Safety Research, Wageningen, The

Netherlands

Hermann Unterluggauer Austrian Agency for Health and Food Safety (AGES), Innsbruck,

Austria

Magnus Jezussek Bavarian Health and Food Safety Authority, Erlangen, Germany Marine Lambert ANSES, Agence nationale de sécurité sanitaire de l'alimentation,

de l'environement et du travail, Maisons-Alfort Cedex, France Head of EURL CF, DTU National Food Institute, Lyngby, Denmark

Mette Erecius Poulsen Head of EURL CF, DTU National Food Institute, Lyngby, D Michelangelo Anastassiades Head of EURL SRM, CVUA Stuttgart, Fellbach, Germany

Patrizia Pelosi Istituto Superiore di Sanità, Rome, Italy

Ralf Lippold CVUA Freiburg, Freiburg, Germany

Stephan Radim Czech Agriculture and Food Inspection Authority, Prague, Czech

Republic

Tuija Pihlström National Food Agency, Uppsala, Sweden