SPECIFIC PROTOCOL

21st European Proficiency Test on Pesticides in Food of Animal Origin and Commodities with High Fat Content

„Pesticides in Whole Milk (EUPT-AO21)“

(24.03.2026)

➢ 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. 12[1]. The current proficiency test covers pesticides that can be determined by multi-residue methods. This EUPT is to be performed by all National Reference Laboratories for Pesticides in Food of Animal Origin and Commodities with High Fat Content (NRL-AO) as well as by all official EU laboratories (OfLs) responsible for official pesticide residue controls on food of animal origin, as far as their scope overlaps with that of the EUPT-AO21. The commodity chosen to prepare the test material is whole milk and it is considered as a representative commodity for “Milk and milk products, Group 8” (see Annex A of document SANTE 11312/2021 v2026)[2].

➢ PT item (test material)

This proficiency test concerns the analysis of pesticide residues in whole milk. The matrix contains only spiked pesticides.

Since 2020, the EURLs for pesticide residues do not provide blank PT items. Each laboratory is asked to use “pesticide free” representative whole milk 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.

1 https://www.eurl-pesticides.eu/userfiles/file/EurlALL/General_Protocol_V12_2025.pdf

2 https://www.eurl-pesticides.eu/userfiles/file/EurlALL/SANTE-11312_2021-V2026.pdf

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 ~ 125 g of whole milk (one bottle) containing spiked pesticides.

- **Analytical parameters**

The PT item contains several pesticides from the target pesticide lists given in Annex 1 and 2 of this document.

It is mandatory to analyse all pesticides included in Annex 1. Pesticides included in Annex 2 can be analysed on voluntary basis.

Laboratories should carefully read the target pesticide lists, where important information about reporting of results, as well as the Minimum Required Reporting Levels (MRRLs), are given. The target pesticide lists contain only individual compounds and results should only be reported for individual compounds, no matter how the residue definitions are set.

During EUPT AO 12 - 16 a quite number of laboratories had problems to report spinosyn A and D individually. Therefore, a possibility for reporting the result of spinosad (sum of spinosyn A and D) was added. Please, report results for spinosyn A and D only, if you use the individual standards for calibration.

The MRRL values will be used to identify false positive and false negative results and for the calculation of z-scores for false negatives.

Please, report important observations during analysis in the EUPT Webtool in the special field for comments (e.g. damaged can or losses, additional pesticides not listed in the target lists). Please consider therefore the EUPT Webtool Guideline.

- **Shipment of PT item**

Dispatch of the PT item is planned on **13 April 2026**.

PT items will be shipped in bottles in an isolated box with cooling elements. 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 or 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 cooled (-18°C) to avoid any deterioration/spoilage and to minimise possible pesticide losses.

Bring the content of the PT item to room temperature and homogenise the whole sample thoroughly before analysis.

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 5 g of the PT item and automated EN1528 method (for GC-amenable pesticides) and 5 g of the PT item for QuEChERS-AO method (for LC-amenable pesticides). 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.

- **EUPT Webtool and Deadlines**

Sample receipt acknowledgement, scope selection, analytical results and method information are to be submitted via the EUPT Webtool (open in incognito or private window). Please consider the guideline on how to use the EUPT Webtool: EUPT Webtool Guideline. To access the EUPT Webtool participants must use their unique login data (username and password), which was sent to you from the colleagues from DTU Denmark. The password can be retrieved via https://quest.dtu.dk/Sites/GuestLogin/RetrievePassword.aspx using your email address or your username.

1. PT item receipt and acceptance

Once the laboratory has received the PT item, it must report to the organiser, via the EUPT Webtool (Result Submission Website EUPT-AO21), the date of receipt, the condition of the PT item, and its acceptance. The deadline for acceptance is **17 April 2026**. If the laboratory does not respond by this deadline the organiser will assume that the PT item has been received and accepted. If participants have not received the PT items by the **17 April 2026 at noon**, they must inform the organiser immediately by e-mail (eurl-pesticides@cvuafr.bwl.de).

2. Scope selection

The analytical scope must be selected prior to the shipment of the samples. This is done via the EUPT Webtool. The scope selection subpage will be opened from **30 March to 10 April 2026**. As default all mandatory pesticides are preselected. Results can only be reported for analytes that have been selected during the scope selection procedure.

Important: If you did not select your scope in time, all analytes of the mandatory pesticide list (see Annex 1) will be selected for your scope!

3. Results and method submission

After shipment of the samples, from 14 April 2026 onwards, it is possible to submit the results by logging into the EUPT Webtool. **The deadline for result submission is 08 May 2026 at 23:00 CEST.** Method information can be added within the next 7 days (until 15 May 2026).

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. Attached to the email will be an excel file with all their submitted data and a pdf of the pesticide and concentration submitted.

4. Reporting of qualitative and quantitative results

Results shall NOT be reported where a pesticide

a) was not detected,

b) was detected below the RL (Reporting Limit) of the laboratory or

c) was detected below the MRRL.

Results reported as "< RL" will be considered as „not detected".

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.

5. Reporting information on analytical methodology

All laboratories are requested to provide information on the analytical method(s) they have used via EUPT Webtool (Result Submission Website EUPT-AO21). The laboratories are requested to fill-in this important information in order to minimise the administrative burden of collecting this information at a later date. Submission of method information is only possible until 7 working days after result submission deadline (until **15 May 2026**).

6. Reporting of supplementary information in case of false negative results

In case of false negative results, the affected laboratories will be asked via e-mail to provide details of the methodology used after the deadline for results submission. This can be done by accessing the EUPT Webtool (Result Submission Website EUPT-AO21) until 7 working days after result submission deadline (until **15 May 2026**).

Important: If no sufficient information on the methodology used is provided, the organiser reserves the right not to accept the analytical results reported by the participant.

- **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** or **false positive/negative results** are requested to **give feedback to EURL-AO** on any actions undertaken to find out the reasons for poor performance. For reporting necessary corrections, please use the excel-sheet that will be attached to the email with the final report.

According to article 94 2c of Regulation (EU) No 2017/625, underperformance of any NRL-AO in comparative testing will be followed by EURL-AO.

- **Documents**

In the EURL-document repository (CIRCA BC) all documents relating to EUPT AO21 can be found. Links to most of the documents are also available on the EUPT-AO21 website.

- **External supplier**

The following tasks were conducted by the assistance of external suppliers:

1. Generation of login credentials for EUPT webtool (EURL-CF, Lyngby, Denmark)

- Programming and administration of EUPT-AO21 result submission website (EURL-CF, Lyngby, Denmark)

Time schedule

Actor	Activity	Date
EURL	Preliminary announcement whole milk at the Joint Workshop in Freiburg	September 2025
EURL	First information supplied to laboratories and call for participation	13 January 2026
Participant	**Registration** via EUPT website	**13 January 2026 – 06 March 2026**
Participant	**Scope selection** via EUPT webtool	**30 March – 10 April 2026**
Participant	Proof of shipment address in EURL-Datapool	30 March – 10 April 2026
EURL	**Dispatch of test material**	**13 April 2026**
Participant	Confirmation of test material receipt	14 – 17 April 2026
Participant	**Deadline for reporting of test results**	**08 May 2026***
Participant	Deadline for reporting of additional method information (no changes of reported results possible)	15 May 2026
EURL	Deadline for preliminary report	Approx. 31 July 2026
Participant	Proof Reading of preliminary report	Approx. September 2026
EURL	Dispatch of the final report as pdf-file	Approx. End of 2026

*) 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 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.

- **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 **08 April 2026**. Please make sure that the payment is made before the stipulated deadline stated on the invoice (**30 May 2026**). 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 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. Additionally, certificates will not be provided, if the invoice is not paid.

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Annex 1

EUPT-AO21 Pesticide target list of mandatory analytes

Table A1: List of 80 **mandatory** analytes and minimum required reporting levels (MRRL) in EUPT-AO21. Results shall be rounded to three significant figures (e.g. 0.0581, 0.251 or 1.35).

Analyte	MRRL (mg/kg)	Analyte	MRRL (mg/kg)
Aldrin	0.003	Fenpropidin	0.005
Azinphos-methyl	0.005	Fenpyrazamine	0.005
Bifenthrin	0.005	Fenthion	0.005
Bixafen	0.005	Fenthion-Oxon	0.005
Boscalid	0.005	Fenthion-Oxonsulfone	0.005
Carbendazim	0.005	Fenthion-Oxonsulfoxide	0.005
Chlordane, cis-	0.001	Fenthion-Sulfone	0.005
Chlordane, trans-	0.001	Fenthion-Sulfoxide	0.005
Chlorfevinphos	0.005	Fenvalerate/Esfenvalerate (RR/SS/RS/SR)	0.005
Chlorpropham	0.005	Fipronil	0.002
Chlorpyrifos	0.005	Fipronil-Sulfone	0.002
Chlorpyrifos-Methyl	0.005	Fluopyram (new)	0.005
cis-Heptachlor epoxide	0.002	Fluopyram-Benzamide (M25) (new)	0.005
Cyfluthrin (sum)	0.005	Fluquinconazole	0.005
Cypermethrin (sum)	0.005	Flusilazole	0.005
Cyproconazole	0.005	HCH, alpha-	0.005
DDD, p,p-	0.005	HCH, beta-	0.005
DDE, p,p-	0.005	HCH, gamma-	0.005
DDT, o,p-	0.005	Heptachlor	0.002
DDT, p,p-	0.005	Hexachlorobenzene	0.005
Deltamethrin	0.005	Indoxacarb	0.005
Diazinon	0.005	Lambda-Cyhalothrin	0.005
Dieldrin	0.002	Malathion	0.005
Endosulfan, alpha-	0.005	Metaflumizone	0.005
Endosulfan, beta-	0.005	Methoxychlor	0.005
Endosulfan sulfate	0.005	Nitrofen	0.005
Endrin	0.001	Oxychlordane	0.002
Epoxiconazole	0.002	Parathion	0.005

Etofenprox	0.005	Parathion-methyl	0.005
Famoxadone	0.005	Pendimethalin	0.005
Penflufen	0.005	Quintozene	0.005
Penthiopyrad	0.005	Resmethrin, (cis; trans)	0.005
Permethrin (sum)	0.005	Sulfoxaflor	0.005
Phosmet	0.005	Fluvalinate (sum)	0.005
Phoxim	0.005	Tebuconazole	0.005
Pirimiphos-Methyl	0.005	Tecnazene	0.005
Prochloraz	0.005	Tetraconazole	0.005
Profenofos	0.005	Thiacloprid	0.005
Prothioconazole-Desthio	0.005	trans-Heptachlor epoxide	0.002
Pyrazophos	0.005	Vinclozolin	0.005

Legend:

New as mandatory analytes!

PFAS Pesticides (overall 64)

Annex 2

EUPT-AO21 Pesticide target list of voluntary analytes

Table A2: List of 78 **voluntary** analytes and minimum required reporting levels (MRRL) in EUPT-AO21. Results shall be rounded to three significant figures (e.g. 0.0581, 0.251 or 1.35).

Analyte	MRRL (mg/kg)	Analyte	MRRL (mg/kg)
Acrinathrin	0.005	Fluometuron	0.005
Alpha-Cypermethrin (aka alphamethrin)	0.005	Fluopicolide	0.005
Beflubutamid	0.005	Flupyradifurone	0.005
Benfluralin	0.005	Flurochloridone	0.005
Benzalkonium chloride n-C12	0.005	Flutianil	0.005
Benzalkonium chloride n-C14	0.005	Flutolanil	0.005
Benzovindiflupyr	0.005	Fluxapyroxad	0.005
Bixafen, Desmethyl-	0.005	Hydroxy-Tebuconazole	0.005
Boscalid-5-hydroxy (M510F01)	0.005	Isopyrazam	0.005
BTS 44595	0.005	Isoxaflutole	0.005
BTS 44596	0.005	Isoxaflutole diketonitrile-metabolite	0.005
Chlorfenapyr	0.005	Lufenuron (sum of isomers)	0.005
Chlorfluazuron	0.005	Malaoxon	0.005
Cyclobutrifluram	0.005	Mefentrifluconazole	0.005
Cyflufenamid (sum of isomers)	0.005	Metconazole	0.005
Cyflumetofen	0.005	Molinate	0.005
Cyflumetofen (sum of isomers)	0.005	Novaluron	0.005
Didecyldimethylammonium chloride n-C10	0.005	Oxadiargyl	0.005
Diflufenacin	0.005	Oxasulfuron	0.005
Flazasulfuron	0.005	Oxathiapiprolin	0.005
Flonicamid	0.005	Oxyfluorfen	0.005
Flonicamid Metabolite TFNA-AM	0.005	Paraoxon-methyl	0.005
Fluazinam	0.005	Penoxulam	0.005
Flubendiamide	0.005	Pentachloroaniline	0.005
Fludioxonil	0.005	Picolinafen	0.005
Fluensulfone	0.005	Picoxystrobin	0.005
Flufenacet	0.005	Propaquizafop	0.005

Flufenoxuron	0.005	Prosulfuron	0.005
Flumetralin	0.005	Pydiflumetofen	0.005
Pyraclostrobin	0.005	Tetraniliprole	0.005
Pyridalyl	0.005	Thiophanate-Methyl	0.005
Pyroxsulam	0.005	Trifloxystrobin	0.005
Quinoclamine	0.005	Triflumizole	0.005
Spinosad (sum)[1]	0.005	Triflumizole metab. FM-6-1	0.005
Spinosyn A[2]	0.005	Triflumuron	0.005
Spinosyn D[2]	0.005	Trifluralin	0.005
Spirodiclofen	0.002	Triflusulfuron metab. IN-M7222	0.005
Spiroxamine	0.005	Tritosulfuron	0.005
Tembotrione metabolite M5 (4,6-dihydroxy tembotrione)	0.005	Tritosulfuron metab. AMTT	0.001

(1) Results for Spinosad should be reported either if individual standards for Spinosyn A and D or a mixture of Spinosyn A and D are used for quantification.

(2) Results for Spinosyn A or D should be reported, if the individual standards were used for quantification.

Legend:

New as mandatory analytes!

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