

Chemisches und Veterinäruntersuchungsamt Freiburg European Union Reference Laboratory for Pesticides in Food of Animal Origin and Commodities with High Fat Content





EUROPEAN UNION REFERENCE LABORATORY

PESTICIDE RESIDUES IN FOOD OF ANIMAL ORIGIN & COMMODITIES WITH HIGH FAT CONTENT

January 2022

17th European Union Proficiency Test on Pesticides in Food of Animal Origin and Commodities with High Fat Content (EUPT AO 17) Native Rape Seed Oil Test Item

Introduction

The European Union Reference Laboratory for Pesticides in Food of Animal Origin and Commodities with High Fat Content in Freiburg, Germany, announces its 17th proficiency test (PT), thus enabling again each participating laboratory to assess its analytical capability by comparing its results with the assigned values.

The matrix will be **native rape seed oil**. The test item will be spiked with selected analytes of interest and contains additionally incurred pesticides. They are included in the list of maximum residue levels in Commission Regulation 396/2005 and most of them also in the list of compounds to be analysed in the 2022-24 Multiannual Coordinated Control Programme (MACP, COMMISSION IMPLEMENT-ING REGULATION (EU) 2021/601 and the Working Document SANCO/12745/2013 V.12).

The pesticide target list in the annex consists of pesticides from former EUPT lists and in addition few pesticides considered to be relevant for the matrix rape seed oil. Some pesticides are marked to be analysed on voluntary basis. For sufficient scope it is necessary

- to analyse at least 90 % of the mandatory analytes from the list in the annex and
- to detect at least 90 % of the analytes present in the test item.

The voluntary pesticides will be statistical treated as the mandatory pesticides but their results will not influence the categorising in A and B.

The test item will be dispatched on 25 April 2022. Participating laboratories may use any analytical method of their choice. Results are to be reported to the EURL AO within the stipulated deadline. After receipt of the results, the EURL AO will carry out a statistical evaluation of the submitted data and all quantitative laboratory results will be assessed by means of z-scores. Thereafter, a report will be sent to the participating laboratories together with a certificate of participation.

Objectives

The objectives of this survey are

- to assess the inter laboratory consistency of results from the analyses of pesticides in samples of animal origin and
- to provide a quality assurance assessment of the NRLs and the official laboratories within the EU.



Participants

According to Art. 101 of Reg. (EU) 625/207 and Art. 28 (3) of Reg. (EC) 396/2005, participation is mandatory for all laboratories selected as NRL for Pesticides in Food of Animal Origin and Commodities with High Fat Content and for all official laboratories undertaking the analysis of these commodities for the official control on pesticide residues. If your laboratory is obliged to participate and you do not participate in this PT, the Commission expects an explanation for non-participation. Based on the data stored in the Lab-Network Database about the commodity scope and the status of each lab, each laboratory is classified as obliged or not obliged to take part in this PT. This information can also be found on the **EUPT-Registration** page. Errors should be reported to the corresponding NRL and to eurl-pesticides@cvuafr.bwl.de.

Laboratories are requested to enrol for participation within the EUPT-Registration Website (www.eupt-registration.eu) which is going to be used for all EUPTs performed by the EURLs for Pesticide Residues. The registration period will last from **07 February to 04 March 2022**. Participants will be able to re-enter the registration website and change/update the entries (e.g. addresses for shipment and invoice, contact data). Deadline for these changes is **15 April 2022**.

After the end of the registration deadline the participants will receive their username and password for DTU EUPT-Webtool (EUPT AO 17) as well as the latest EUPT-AO-Webtool guideline via e-mail.

Before the shipment of the samples, participating laboratories have to select the analytes from the target list being part of their analytical scope via DTU EUPT-Webtool (EUPT AO 17). Deadline for any changes in scope will be one working day before the shipment of samples (22 April 2022).

Test Item

The test item consists of a unit of rape seed oil with spiked and incurred selected analytes of interest. The test item will be shipped under ambient conditions. Approx. 50 g of the test item will be supplied. Please take note that for this PT no blank (non-spiked) sample will be provided!

Analytical parameters and reporting of results

The test item may contain any analyte from the lists given in the annex. For each of the analytes a specific minimum required reporting level (MRRL) is given. Selected analytes were added to the rape seed oil at relevant concentrations. Single results of each analyte detected shall be reported in **mg/kg**, rounded to three significant figures (e.g. 0.0581, 0.251 or 1.35). Analyte concentrations below the individual reporting levels (RL) shall be considered as "not detected" and **no figures shall be typed into the database**. The results for all pesticides must be reported on product base.

Further instructions for analysis and reporting

The tube contains native rape seed oil obtained from pressed, spiked rapeseeds. The oil was additionally spiked with pesticides of interest after pressing. Laboratories should

- store the test item cooled (at 4 7°C) until analysis,
- stir/shake carefully the content to make sure that the test item is homogeneous,
- suggestion of EURL: portion the content of the test item into subsamples and store unused portions cooled (at 4 - 7°C) for later analysis,
- use their own standard operating procedures for extraction, clean-up and analytical measurement,
- use their own reference standards for identification and quantification,
- provide a detailed method description and any additional information.

Sample receipt forms will be made available in the DTU EUPT-Webtool (EUPT AO 17) when the test item is dispatched. Reporting forms will be accessible after the receipt of the test item has been confirmed. There will be **no extension of the deadline**. Results should be submitted by using the



DTU EUPT-Webtool (EUPT AO 17) before the deadline. As laid down in Regulation 2017/625, NRLs are responsible for evaluating and improving their OfL network. For this reason, the EURL AO will confide the laboratory codes of OfLs to their NRLs together with the final report. On request of NRLs the organisers will confide the laboratory codes one month after dispatch of the preliminary report.

Statistical evaluation of results

EUPT AO 17 is one of five proficiency tests organised by the EURLs for pesticide residues as part of their work programmes for 2022. Thus, the performance and the evaluation of EUPT AO 17 will be similar to those that will be used in the other EUPTs.

The performance of each laboratory will be evaluated and presented in an anonymous format in a report written after the final evaluation. The organisers will calculate the mean, robust mean, median and standard deviation for each spiked analyte. The procedure will follow the General Protocol for EU proficiency Tests for Pesticide Residues in Food and Feed and the IUPAC/ISO/AOAC International Harmonised Protocol for the Proficiency Testing of Chemical Analytical Laboratories (see also ISO 13528). The evaluation will be performed in close coordination with the Scientific Committee for EUPTs. First, pre-assigned values will be calculated taking into account the results of all participants. At the meeting of the Scientific Committee for EUPTs (June 2022) the pre-assigned values will be discussed. The pre-assigned values will be confirmed or recalculated after omitting results of laboratories according to the suggestions of the Scientific Committee for EUPTs.

Time schedule

Actor	Activity	Date
EURL	Preliminary Announcement matrix rape seed oil at EURL-NRL workshop	22.October 2021
EURL	First Information supplied to laboratories and call for participation	Mid of January 2022
Participant	Registration via EUPT website	07 February 2022; ending 04 March 2022
Participant	Scope selection via EUPT webtool	28 March 2022;
		Ending 22 April 2022
Participant	Proof of shipment address in EURL-Datapool	Ending 15 April 2022
EURL	Shipment of test material	25 April 2022
Participant	Confirmation of test material receipt	Within 7 days
Participant	Reporting of test results and method information	By 23 May 2022*
Participant	Reporting of additional method information (no changes of reported results possible)	Within 7 working days after end of result reporting
EURL	Dispatch of a preliminary report to all participants	By 22 July 2022
EURL	Dispatch of the final report as pdf-file	Approx. end of 2022

^{*)} 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 190.00** for shipping and handling to participants within the European Union and EFTA countries (**including NRLs**). Fees for participants from **other countries** are **EUR 380.00**. An invoice will be sent together with the samples.



Contact information

Contact address of EURL Freiburg:

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QCG: Quality Control Group AG: Advisory Group

For any further clarification, please, don't hesitate to contact the organisers.

Björn Hardebusch Head of EURL AO



EUPT AO 17 Pesticide Target List of Mandatory Analytes

PLANT OIL (native rape seed oil) Test Item

Annex 1: List of analytes and MRRL-values (all in mg/kg product)
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]
Acetamiprid	0.01	Dimethoate	0.01
Aldrin	0.01	Diphenylamine	0.01
Azinphos-ethyl	0.01	Endosulfan, alpha-	0.01
Azinphos-methyl	0.01	Endosulfan, beta-	0.01
Azoxystrobin	0.01	Endosulfansulfate	0.01
Bifenthrin	0.01	Endrin	0.01
Bixafen (parent only)	0.01	Epoxiconazole	0.01
Boscalid (parent only)	0.01	Etofenprox	0.01
Buprofezin	0.01	Famoxadone	0.01
Carbendazim (parent only)	0.01	Fenitrothion	0.01
Chlorobenzilate	0.01	Fenoxycarb	0.01
Chlordane, alpha(cis)-	0.01	Fenpropidin (parent only)	0.01
Chlordane, gamma(trans)-	0.01	Fenpropimorph (parent only)	0.01
Chlorfenvinphos	0.01	Fenpyrazamine	0.01
Chlorpropham (parent only)	0.01	Fenthion (parent)	0.01
Chlorpyrifos-ethyl	0.01	Fenthion-oxon	0.01
Chlorpyrifos-methyl	0.01	Fenthion-oxon-sulfone	0.01
Cyfluthrin (sum of isomers)	0.01	Fenthion-oxon-sulfoxide	0.01
Cyhalothrin, lambda- (sum of isomers)	0.01	Fenthion-sulfone	0.01
Cypermethrin (sum of isomers)	0.01	Fenthion-sulfoxide	0.01
Cyproconazole	0.01	Fenvalerate/Esfenvalerate (sum of RR, SS, RS & SR isomers)	0.01
DDD, p,p-	0.01	Fipronil	0.005
DDE, p,p-	0.01	Fipronil-sulfone	0.005
DDT, o,p-	0.01	Fluopyram (parent only)	0.01
DDT, p,p-	0.01	Fluquinconazole	0.01
Deltamethrin	0.01	Flusilazole (parent only)	0.01
Diazinon	0.01	HCH, alpha-	0.01
Dichlorvos	0.01	HCH, beta-	0.01
Dieldrin	0.01	HCH, gamma- (Lindane)	0.01
Difenoconazole	0.01	Heptachlor	0.01



Analyte	MRRL Analyte		MRRL
	[mg/kg]		[mg/kg]
Heptachlorepoxid, cis-	0.01	Prosulfocarb	0.01
Heptachlorepoxid, trans-	0.01	Prothioconazol-desthio	0.01
Hexachlorobenzene (HCB)	0.01	Pyraclostrobin	0.01
Imidacloprid	0.01	Pyrazophos	0.01
Indoxacarb (sum of isomers)	0.01	Pyridalyl	0.01
Kresoxim-methyl	0.01	Pyrimethanil	0.01
Malathion (parent only)	0.01	Quinalphos	0.01
Metaflumizone (sum of isomers)	0.01	Quintozene (parent only)	0.01
Methidathion	0.01	Resmethrin (sum of isomers)	0.01
Methoxychlor, 4,4-	0.01	Spinosad*	0.01
Nitrofen	0.01	Spinosyn A**	0.01
Omethoate	0.01	Spinosyn D**	0.01
Oxychlordane	0.01	Spiroxamine	0.01
Oxyfluorfen	0.01	tau-Fluvalinate (sum of isomers)	0.01
Parathion-ethyl	0.01	Tebuconazole	0.01
Parathion-methyl (parent only)	0.01	Tebufenozid	0.01
Pendimethalin	0.01	Tefluthrine	0.01
Penflufen	0.01	Terbutylazine	0.01
Penthiopyrad	0.01	Tecnazene (Tetrachlornitrobenzol, 2,3,5,6-)	0.01
Permethrin (sum of isomers)	0.01	Tetraconazole	0.01
Phosalon	0.01	Thiacloprid	0.01
Phosmet (parent only)	0.01	Tolclofos-methyl	0.01
Phoxim	0.01	Triazophos	0.01
Piperonylbutoxide	0.01	Trifloxystrobin	0.01
Pirimicarb	0.01	Trifluralin	0.01
Pirimiphos-methyl	0.01	Vinclozolin (parent only)	0.01
Prochloraz (parent only)	0.01		
Procymidon	0.01		
Profenofos	0.01		
Propyzamid	0.01		

^{*} Results for Spinosad should be reported either if individual standards for Spinosyn A and D are used for calibration

New in pesticides target list

of Spinosyn A and D are used for calibration.

** Results for Spinosyn A or D should only be reported, if the individual standards were used for quantification!



EUPT AO 17 Pesticide Target List of Voluntary Analytes

PLANT OIL (native rape seed oil) Test Item

Annex 2: List of analytes and MRRL-values (all in mg/kg product)
Results shall be rounded to three significant figures (e.g. 0.0581, 0.251 or 1.35).

Analyte	MRRL [mg/kg]
Benzovindiflupyr	0.01
BTS 44595 (Prochloraz metabolite)	0.01
BTS 44596 (Prochloraz metabolite)	0.01
Clomazone	0.01
Cyantraniliprole	0.01
Fenbuconazole	0.01
Fluoxastrobin	0.01
Flupyradifurone	0.01
Fludioxonil	0.01
Flutriafol	0.01
Fluxapyroxad	0.01
Hydroxy-Tebuconazole	0.01
Isopyrazam	0.01
Mefentrifluconazole	0.01
Metconazol	0.01
Molinate	0.01
Oxadiargyl	0.01
Oxasulfuron	0.01
Oxyfluorfen	0.01
Picolinafen	0.01
Propaquizafop	0.01
Quinoclamine	0.01
Sulfoxaflor	0.01
Thiophanate methyl	0.01

^{*} 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 calibration.

New in pesticides target list

Updated version 1.1 editorial update, highlighting additional 7 new pesticides in voluntary pesticide target list

^{**} Results for Spinosyn A or D should only be reported, if the individual standards were used for quantifica-