



January 2018

13th European Union Proficiency Test on Pesticides in Food of Animal Origin and Commodities with High Fat Content (EUPT AO-13) Milk Powder 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 13th proficiency test (PT), thus enabling again each participating laboratory to assess its analytical capability by comparing its results with the assigned values.

As announced the EURL/NRL/OfL Joint Workshop for Pesticide Residues in Food in Freiburg in September 2017, a test item of milk powder will be supplied, spiked with selected analytes of interest. 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 2018 Multiannual Coordinated Control Programme (MACP, COMMISSION IMPLEMENTING REGULATION (EU) 2016/662 and the Working Document SANCO/12745/2013 V.9).

As in previous EUPTs AO there are two annexes with pesticides. **Annex 1** contains the pesticides to be analysed for in routine analysis according to the MACP plus a few other compounds of interest in the commodity group milk. This list is comparable to the list from previous EUPT AOs. Thus, all laboratories involved in the MACP 2018 are obliged to analyse the pesticides in this list. For sufficient scope it is necessary

- to analyse at least 90 % of the analytes from the list in annex 1 and
- to detect at least 90 % of the analytes present in the test item.

Annex 2 contains pesticides, which can be analysed on voluntary basis (SANCO/12745/2013 V.9, requirements on pesticides (infant formula and follow-on formula)). **The voluntary pesticides in annex 2 will be statistical treated as the pesticides in annex 1 but their results will not influence the categorising in A and B.**

The test item will be dispatched on 09 April 2018. Participating laboratories may use any analytical method of their choice. Results are to be reported to the EURL four weeks after receipt of the test item. After receipt of the results, the EURL will carry out a statistical evaluation of the submitted data and all quantitative laboratory results will be assessed by means of z-scores. Then, 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. 28 of EC Reg. 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. The list of obliged laboratories is published in CIRCA BC. Please, follow the link on the EURL website:

<http://www.eurl-pesticides.eu/docs/public/tmpl/article.asp?CntID=962&LabID=100&Lang=EN>

Laboratories are requested to enrol for participation within a **web based database** which is going to be used for all EUPTs performed by the EURLs for Pesticide Residues. For registration a link to the database will be sent on **26 February 2018**. Registration is possible until **15 March 2018**.

After registration the participants will receive their username and password for EUPT AO 13. Therefore, participants will be able to re-enter the database and change/update the entries (e.g. addresses for shipment and invoice, contact data). Deadline for these changes is **29 March 2018**.

Before the shipment of the samples participating laboratories have to select the analytes from the target list being part of their analytical scope. Deadline for any changes in scope will be one day before the shipment of samples (**06 April 2018**).

Test item

The test item consists of a unit of milk powder spiked with selected analytes of interest. The test item will be shipped at ambient temperature. Approx. 75 g of the test item will be supplied. In addition, a blank (unspiked) portion of the test item will be provided.

Analytical parameters and reporting of results

The test item may contain any analyte from the lists given in the annexes. For each of the analytes a specific minimum required reporting level (MRRL) is given. Selected analytes were added to the homogenised milk powder at relevant concentrations. Both, the spiked and the blank test item should be analysed. 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**. In accordance to the European legislation the results of all pesticides must be reported on product base.

Further instructions for analysis and reporting

The tube contains milk powder as available on the market. Laboratories should

- store the item in the refrigerator (at least 7°C) until analysis,
- stir the content to make sure that the test item is homogeneous,
- suggestion of EURL: portion the content of the content into subsamples and store unused portions in the freezer (-18°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 database 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 database on the website before the deadline. As laid down in Regulation 882/2004, 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-13 is one of five proficiency tests organised by the EURLs for pesticide residues as part of their work programmes for 2018. Thus, the performance and the evaluation of EUPT AO-13 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 EUPT Scientific Committee. First, pre-assigned values will be calculated taking into account the results of all participants. At the meeting of the Scientific Committee for EUPTs (begin of July 2018) 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

EURL	First Information supplied to laboratories and call for participation	Mid of January 2018
Participant	Registration via EUPT website	26 February 2018; ending 16 March 2018
Participant	Proof of data entered into the EUPT website	By 28 March 2018
Participant	Entering of the analytical scope	Ending 06 April 2018
EURL	Shipment of test material	09 April 2018
Participant	Confirmation of test material receipt	Within 7 days
Participant	Reporting of test results and method information	By 11 May 2018*
Participant	Reporting of additional method information (no changes of reported results possible)	Starting 14 May 2018; ending 28 May 2018
EURL	Dispatch of a preliminary report to all participants	By 29 June 2018
EURL	Dispatch of the final report as pdf-file	End of 2018

*) 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 175.00** for shipping and handling to participants within the European Union and EFTA countries (**including NRLs**). Fees for participants from **other countries** are **EUR 350.00**. The organizers will not accept participants from Third Countries when they cannot guarantee that the test items are still cooled arriving in the laboratories (duration of shipping > 3 days). An invoice will be sent together with the samples.

Contact information

Contact address of EURL Freiburg:

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EUPT Scientific Committee:

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QCG: Quality Control Group AG: Advisory Group

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

Ralf Lippold
Head of EURL AO

EUPT AO 13 Pesticide Target List of Mandatory Analytes

Milk Powder 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]
Abamectin B1a	0.01	Fenthion oxon	0.01
Aldrin	0.01	Fenthion oxon sulfone	0.01
Azinphos-ethyl	0.01	Fenthion oxon sulfoxide	0.01
Azinphos-methyl	0.01	Fenvalerate/Esfenvalerate (sum of RR, SS, RS & SR isomers)	0.01
Bifenthrin	0.01	Hexachlorobenzene (HCB)	0.01
cis-Chlordane	0.01	alpha-HCH	0.01
trans-Chlordane	0.01	beta-HCH	0.01
Oxychlordane	0.01	gamma-HCH (Lindane)	0.01
Chlorfenvinphos	0.01	Heptachlor	0.01
Chlorobenzilate	0.01	cis-Heptachlorepoxyd	0.01
Chlorpyrifos(-ethyl)	0.01	trans-Heptachlorepoxyd	0.01
Chlorpyrifos-methyl	0.01	Indoxacarb (sum of isomers)*	0.01
Cyfluthrin (sum of isomers)	0.01	Malathion (parent only)	0.01
Cypermethrin (sum of isomers)	0.01	Methidathion	0.01
lambda-Cyhalothrin (sum of isomers)	0.01	4,4'-Methoxychlor	0.01
p,p'-DDE	0.01	Nitrofen	0.01
p,p'-DDD (TDE)	0.01	Parathion(-ethyl)	0.01
p,p'-DDT	0.01	Parathion-methyl (parent only)	0.01
o,p'-DDT	0.01	Pendimethalin	0.01
Deltamethrin	0.01	Permethrin (sum of isomers)	0.01
Diazinon	0.01	Phosmet (parent only)	0.01
Dieldrin	0.01	Phoxim	0.01
alpha-Endosulfan	0.01	Pirimiphos-methyl	0.01
beta-Endosulfan	0.01	Profenofos	0.01
Endosulfan sulfate	0.01	Pyrazophos	0.01
Endrin	0.005	Quintozene (parent only)	0.01
Famoxadone	0.01	Resmethrin (sum of isomers)	0.01
Fenthion (parent)	0.01	Tecnazene	0.01
Fenthion sulfoxide	0.01	Triazophos	0.01
Fenthion sulfone	0.01	Vinclozolin (parent only)	0.01

*new in list of mandatory pesticides

EUPT AO 13 Pesticide Target List of Voluntary Analytes Milk Powder Test Item

Annex 2: List of analytes and MRRL values (all analytes 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]
BAC-C8 (expressed as chloride salt)	0.01	Fluopyram	0.01
BAC-C10 (expressed as chloride salt)	0.01	Fluopyram-Benzamide	0.01
BAC-C12 (expressed as chloride salt)	0.01	Fluquinconazole	0.01
BAC-C14 (expressed as chloride salt)	0.01	Flusilazole (parent only)	0.01
BAC-C16 (expressed as chloride salt)	0.01	Metaflumizone (sum of isomers)	0.01
BAC-C18 (expressed as chloride salt)	0.01	Penflufen	0.01
Benzovindiflupyr	0.01	Penthiopyrad	0.01
Bixafen (parent only)	0.01	Prochloraz (parent only)	0.01
Boscalid (parent only)	0.01	Prothioconazole-desthio	0.01
Carbendazim (Carbendazim only)	0.01	Spinosad	0.01
Chlorpropham (parent only)	0.01	Spinosyn A	0.01
Cyproconazole	0.01	Spinosyn D	0.01
DDAC-C10 (expressed as chloride salt)	0.01	Spiroxamine	0.01
Epoxiconazole	0.01	Sulfoxaflor	0.01
Etofenprox	0.01	tau-Fluvalinate	0.01
Fenpropidin (parent only)	0.01	Tebuconazole	0.01
Fenpyrazamine	0.01	Tetraconazole	0.01
Fenpropimorph (parent only)	0.01	Thiacloprid	0.01

New in the voluntary list

Requirements on pesticides (infant formula and follow-on formula)

Cadusafos	0.01	Ethoprophos	0.01
Demeton-S-methyl	0.01	Fipronil	0.01
Demeton-S-methyl-sulfon	0.01	Fipronil sulfone (AO metabolite)	0.01
Oxydemeton-methyl	0.01	Fipronil desulfinyl	0.01
Disulfoton	0.01	Fensulfothion	0.01
Disulfoton-sulfoxid	0.01	Omethoate	0.01
Disulfoton-sulfon	0.01	Terbufos	0.01