Announcement

ILS F-01 (2024)

Interlaboratory Study on Pesticides in Food of Animal Origin and Commodities with High Fat Content

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Pesticides in Fish

February 2024

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

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QCG: Quality Control Group AG: Advisory Group

Introduction

The European Union Reference Laboratory for Pesticides in Food of Animal Origin and Commodities with High Fat Content in Freiburg, Germany, announces its 1st interlaboratory study (ILS) on pesticides in **fish**. Until now, the EURL-AO has never organised an EUPT in fish meat (muscle). However, feedback given by NRLs has shown that laboratories are seeking support from the EURL as regards an interlaboratory comparative testing in fish. Thus, the EURL organises a voluntary interlaboratory study.

The PT item will be spiked with selected analytes of interest. Most of them are in the list of compounds to be analysed in the 2024-26 Multiannual Coordinated Control Programme (MACP), Commission Implementing Regulation (EU) 2023/731 and the Working Document SANCO/12745/2013 rev.14.

The pesticide target list in the annex of this document is - with some exceptions – consists with the pesticide target list of EUPT AO 19. 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 PT item will be dispatched on Monday, 06 May 2024 (together with EUPT AO 19). 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 interlaboratory study are

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

Participants

Participation is voluntary for all laboratories.

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 **15 December to 01 April 2024**. 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 **26 April 2024.**

After the end of the registration deadline, the participants will receive their username and password for DTU EUPT-Webtool (ILS F-01) as well as the latest webtool guideline via e-mail.

IMPORTANT: Before the shipment of the samples, participating laboratories have to select **CAREFULLY** the analytes from the target list being part of their analytical scope via DTU EUPT-Webtool (EUPT AO 19). Deadline for any changes in scope will be one working day before the shipment of samples (**03 May 2024**). After this deadline neither participants nor the EURL is able to change the scope. **The EURL will not accept any changes sent by email.** If scope selection is not performed, all analytes will be automatically selected.

Proficiency test item (PT item)

The PT item consists of one unit of fish with spiked analytes of interest. The EURL AO produces the PT material at a local school for butchers and spikes the analytes of interest. The PT item will be preserved and the containers will be stored chilled until shipment. PT items will be shipped under ambient conditions. Approx. **100 g** of the PT item will be supplied. Please note that for this PT no blank (non-spiked) sample will be provided!

Analytical parameters and reporting of results

The PT 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 fish 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.

Further instructions for analysis and reporting

Laboratories should

- store the PT item cooled (at 4 - 7°C) until analysis,
- mix the whole test item carefully to make sure that the PT item is homogeneous,
- suggestion of EURL: portion the content of the PT 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 (ILS F-01) when the PT item is dispatched. Reporting forms will be accessible after the receipt of the PT item has been confirmed. There will be **no extension of the deadline**. Results should be submitted by using the DTU EUPT-Webtool (ILS F-01) 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

ILS F-01 is an interlaboratory study organised by EURL-AO as part of its work programme for 2024. The performance and the evaluation of ILS F-01 will be similar to 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 cooperation with the Scientific Committee for EUPTs. First, pre-assigned values will be calculated taking into account the results of all participants. 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 at 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	**15 December 2023 – 01 April 2024**
Participant	**Scope selection** via EUPT webtool	**22 April – 03 May 2024**
Participant	Proof of shipment address in EURL-Datapool	Ending 26 April 2024
EURL	**Dispatch of test material**	**06 May 2024**
Participant	Confirmation of test material receipt	07 – 13 May 2024
Participant	**Deadline for reporting of test results**	**05 July 2024***
Participant	Deadline for reporting of additional method information (no changes of reported results possible)	16 July 2024
EURL	Deadline for preliminary report	06 September 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 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 together with the samples.

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 for all 3 studies will be 300 Euro for labs from EU and EFTA countries and 500 Euro for labs from other countries participating in all three studies.

Confidentiality

In each EUPT, the participating laboratories are given a unique code, initially only known to themselves and the organisers. In the final EUPT report, the list of participating laboratories will not be linked to their laboratory codes. The organisers are allowed to provide NRLs with the ILS F-01 codes of all OfLs in their respective networks. The

organisers further reserve the right to share EUPT results and codes with other EURLs of the pesticide network.

Contact information EURL AO

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Annex 1

ILS F-01 Pesticide target list of mandatory analytes

Table A1: List of **mandatory** analytes and minimum required reporting levels (MRRL) in ILS F-01 (PT item fish). 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	0.010	Heptachlor	0.005
Azinphos-ethyl	0.010	Heptachlorepoxid, Cis-	0.010
Azinphos-methyl	0.010	Heptachlorepoxid, trans-	0.010
Bifenthrin (sum of isomers)	0.010	Hexachlorcylcohexane (HCH), alpha-isomer	0.010
Chlordane, cis-	0.010	Hexachlorcylcohexane (HCH), beta-isomer	0.010
Chlordane, trans-	0.010	Hexachlorcylcohexane (HCH), gamma-isomer (Lindane)	0.010
Chlorfenvinphos	0.010	Hexachlorobenzene (HCB)	0.010
Chlorpyrifos(-ethyl)	0.010	Indoxacarb (sum of isomers)	0.010
Chlorpyrifos-methyl	0.010	Malathion (parent only)	0.010
Cyfluthrin (sum of isomers)	0.010	Methidathion	0.010
Cyhalothrin, Lambda- (sum of isomers)	0.010	Methoxychlor, 4,4´-	0.010
Cypermethrin (sum of isomers)	0.010	Nitrofen	0.010
DDD, p,p´- (TDE)	0.010	Oxychlordane	0.010
DDE, p,p´-	0.010	Parathion(-ethyl)	0.010
DDT, o,p´-	0.010	Parathion-methyl (parent only)	0.010
DDT, p,p´-	0.010	Pendimethalin	0.010
Deltamethrin (cis-isomer)	0.010	Permethrin (sum of isomers)	0.010
Diazinon	0.010	Phosmet (parent only)	0.010
Dieldrin	0.010	Phoxim	0.010
Endosulfan sulfate	0.010	Pirimiphos-methyl	0.010
Endosulfan, alpha-	0.010	Profenofos	0.010
Endosulfan, beta-	0.010	Pyrazophos	0.010
Endrin	0.010	Quintozene (parent only)	0.010
Famoxadone (MACP mandatory)	0.010	Resmethrin (sum of isomers)	0.010
Fenvalerate/Esfenvalerate (sum of RR, SS, RS & SR isomers)	0.010	Tecnazene	0.010
Fipronil	0.010	Vinclozolin (parent only)	0.010
Fipronil sulfone	0.005		

Annex 2

ILS F-01 Pesticide target list of voluntary analytes

Table A2: List of **voluntary** analytes and minimum required reporting levels (MRRL) in ILS F-01 (PT item fish). 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)
Benzalkonium Chloride (C8,C10,C12,C14,C16,C18)	0.01	Hexaflumoron	0.01
Benzovindiflupyr	0.01	Imidacloprid	0.01
Bixafen (parent only)	0.01	Lufenuron	0.01
Bixafen-desmethyl	0.01	Mefentrifluconazole	0.01
Boscalid (parent only)	0.01	Metaflumizone (sum of isomers)	0.01
Carbendazim (Carbendazim only)	0.01	Penflufen (sum of isomers)	0.01
Chlorate	0.01	Penthiopyrad	0.01
Chlorpropham (parent only)	0.01	Prochloraz (parent only)	0.01
Cyproconazole	0.01	BTS 44595 (Prochloraz metabolite)	0.01
Didecyldimethylammoniumchlorid (DDAC-C8,C10,C12)	0.01	BTS 44596 (Prochloraz metabolite)	0.01
Diflubenzuron (parent only)	0.01	Prothioconazole-desthio	0.01
Emamectin	0.01	Spinosad (1)	0.01
Epoxiconazole	0.01	Spinosyn A (2)	0.01
Ethoxyquin	0.01	Spinosyn D (2)	0.01
Ethoxyquin dimer, metabolite of ethoxyquin	0.01	Spiroxamine (parent only, sum of isomers)	0.01
Ethoxyquin quinone imine (EQI)	0.01	Sulfoxaflor (sum of isomers)	0.01
Etofenprox	0.01	tau-Fluvalinate (sum of isomers)	0.01
Fenpropidin (parent only)	0.01	Tebuconazole	0.01
Fenpropimorph (parent only)	0.01	Tebuconazole, Hydroxy- (free phenol only)	0.01
Fenpyrazamine	0.01	Teflubenzuron	0.01
Fluopyram	0.01	Tetraconazole	0.01
Fluopyram-benzamide	0.01	Thiacloprid	0.01
Fluquinconazole	0.01	Thiophanate methyl	
Flusilazole (parent only)			

(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.