



**EURL Interlaboratory Study on the Determination of
Brominated Contaminants, PCNs and CPs
in Fish Oil
2022/23**

EURL-ILS-BC_2202-FO

FOOD

Second Announcement

16 December 2022



Summary

Test sample	FOOD: Fish Oil [2202-FO]
Analytes of interest	<p>PBDEs (Nine PBDE congeners)</p> <p>HBCDDs (Three stereoisomers and/or total HBCDD)</p> <p>TBBPA</p> <p>eBFRs (PBB, HBBz, PBT, PBEB, BTBPE, DBDPE, TBX)</p> <p>PBDD/Fs (Thirteen 2,3,7,8-substituted PBDD/Fs)</p> <p>PXDD/Fs (Six 2,3,7,8-substituted PXDD/Fs)</p> <p>PCNs (26 PCN congeners)</p> <p>CPs (short, medium and long chained chlorinated paraffins)</p>
Methods	Any kind of method
Participants	NRLs, OFLs, other official laboratories, commercial laboratories performing the analysis of samples taken by food business operators, research institutes and universities
Statistical evaluation	ISO 13528:2020, IUPAC Protocol
Participation fee	Participation fee for OFLs, other official and commercial laboratories, research institutes and universities
Registration	Ongoing registration is possible until 15 October 2023
Shipment of samples	From 16 January 2023 (second round – ongoing)
Deadline for reporting of results*	<p>First deadline (all analytes): 15 October 2022</p> <p>Second deadline (all analytes): 31 March 2023</p> <p>Third deadline (all analytes): 31 October 2023</p>

**Up to five sets of results can be reported until each deadline for all analytes to compare the intra-laboratory (repeatability) precision between the laboratories*



1. Introduction

This interlaboratory study (ILS) on the determination of several **brominated contaminants (BCons)**, **polychlorinated naphthalenes (PCNs)** and **chlorinated paraffins (CPs)** in fish oil is organized by the EURL for halogenated POPs in Feed and Food to be performed between May and September 2022 (first deadline, closed), **April 2023 (second deadline)** and until October 2023 (third round). The objective is to characterize a quality control material for method development purpose and quality control during routine analysis. Therefore up to five sets of results for all analyte groups can be reported until each deadline using the same or different methods. In addition, the focus is on the assessment of intra- and interlaboratory comparability of results from **PBDEs, HBCDDs, TBBPA, eBFRs, PBDD/Fs, PXDD/Fs, PCNs** and **CPs** in two fish oil samples.

As a **second exercise** solutions of **CP technical mixtures**, to be analysed as samples without further preparation, are provided to assess intra- and interlaboratory comparability of CP results. Laboratories performing CP analysis, or even just starting to analyse CPs, are encouraged to submit at least total CP results for fish oil and technical mixture samples.

Official laboratories, research laboratories, universities and commercial laboratories performing the analysis of samples taken by food business operators are invited to participate in this interlaboratory study. Second and third results will be discussed by representatives of European Commission, NRLs and the EURL at the EURL/NRL workshops in May and November 2023.

Participating laboratories will receive the evaluation of the ILS results in preliminary and final reports.

EURL reserves all rights to publish and present the results of the interlaboratory study in scientific journals and/or conferences.

2. Test samples

Test sample A is prepared from commercially available fish oil and is naturally contaminated with some analytes of interest. **Test sample B** is fish oil fortified with analytes of interest. Each participant will receive about 20 g of each test sample in a PTFE bottle.

Test samples C and **D** are solutions of CP technical mixtures in cyclohexane, to be analysed without further preparation. Solvent exchange may be required for LC analysis and has to be performed in the own laboratory. Detailed information will follow in the ILS instructions.

Additionally small amounts of standard mixture of some of the analyte groups of interest may be provided on request.



3. Analytes of interest

Participants are asked to determine at least one of the following parameters (as many analytes as possible from each group):

Brominated Contaminants:

■ PBDE group

- Individual congeners: BDE-28, -47, -49, -99, -100, -153, -154, -183, -209
- Sum of 8 PBDEs (without BDE-209)
- Sum of 9 PBDEs (with BDE-209)

■ HBCDD and TBBPA group

- α -HBCDD, β -HBCDD, γ -HBCDD stereoisomers
- Sum of α -, β -, γ -HBCDD (using HPLC methods)
- Total HBCDD (using GC methods)
- TBBPA

■ eBFR group

- PBBz (1,2,3,4,5-Pentabromobenzene; CAS 608-90-2)
- HBB (1,2,3,4,5,6-Hexabromobenzene; CAS 87-82-1)
- PBT (1,2,3,4,5-Pentabromo-6-methylbenzene; CAS 87-83-2)
- PEBB (1,2,3,4,5-Pentabromo-6-ethylbenzene; CAS 85-22-3)
- BTBPE (1,1'-[1,2-Ethanediy-bis(oxy)]bis[2,4,6-tribromo]benzene; CAS 37853-59-1)
- DBDPE (1,1'-(1,2-Ethanediy)bis[2,3,4,5,6-pentabromo-benzene]; CAS 84852-53-9)
- TBX (1,2,4,5-Tetrabromo-3,6-dimethylbenzene; CAS 23488-38-2)
- BEH-TEBP (Bis(2-ethylhexyl)-tetrabromophthalate)
- EH-TBB (2-Ethylhexyl-2,3,4,5-tetrabromobenzoate)
- DBE-DBCH (1,2-Dibromo-4-(1,2-dibromoethyl)cyclohexane)

■ PBDD/F and PXDD/F group

- 2,3,7,8-TBDD; 1,2,3,7,8-PeBDD; 1,2,3,4,7,8-HxBDD; 1,2,3,6,7,8-HxBDD;
- 1,2,3,7,8,9-HxBDD; 1,2,3,4,6,7,8-HpBDD; OBDD;
- 2,3,7,8-TBDF; 1,2,3,7,8-PeBDF; 2,3,4,7,8-PeBDF; 1,2,3,4,7,8-HxBDF;
- 1,2,3,4,6,7,8-HpBDF; OBDF



- 2,3-DiBr-7,8-DiCDD; 2-Br-3,7,8-TrCDD; 1-Br-2,3,7,8-TCDD; 2-Br-1,3,7,8-TCDD;
3-Br-2,7,8-TrCDF; 1-Br-2,3,7,8-TCDF

Chlorinated Contaminants:

■ PCN group

- Individual PCNs 14, 21, 24, 27, 31, **42**, 46, **48**, 49, 50, **53**, **59**, **63**, 65, **69**, **70**, **73**, **74**, **75**
- Pairs of PCNs **28/36**, **52/60**, **66/67**, **64/68**, **71/72** (difficult to separate chromatographically)
*[It is recommended to focus at least on the 20 main congeners (in **bold**) when starting method development]*
- Sum of **main** congeners
- Sum of laboratories individual measured congeners for comparison

■ Chlorinated paraffins (CPs)

- **Total CPs (please report at least results for total CPs)**
- Sum of SCCPs, MCCPs, LCCPs
- Short chained chlorinated paraffins (SCCPs)
- Medium chained chlorinated paraffins (MCCPs)
- Long chained chlorinated (LCCPs)

4. Methods

All kinds of detection and quantification methods may be applied.

5. Statistical evaluation of results

Statistical evaluation of the ILS results is performed by the EURL POPs according to:

- ISO 13528:2020, Statistical methods for use in interlaboratory studying by interlaboratory comparisons, International Organization for Standardization
- International Harmonized Protocol for the Interlaboratory studying of Analytical Chemistry Laboratories (IUPAC Technical Report, Pure Appl. Chem., Vol. 78, No. 1, pp-145-196, 2006).



6. Quality control

The Deutsche Akkreditierungsstelle GmbH attests that the provider of proficiency testing Chemisches und Veterinäruntersuchungsamt Freiburg, EU Reference Laboratory (EURL) for halogenated persistent organic pollutants (POPs) in Feed and Food is competent under the terms of DIN EN ISO/IEC 17043:2010 to carry out proficiency testing in the testing field of determination of halogenated persistent organic pollutants (POPs) in food and feed (Accreditation number: D-EP-18625-01-00).

7. Confidentiality

The identity of participating laboratories will be kept confidential.

For OFLs of EU member states cooperating with NRL, the respective NRLs will inform the EURL for halogenated POPs about the participating OFLs and will receive the respective laboratory codes, invoices for participation fee and certificates of participation of the OFLs.

8. Participation fee

The participation of **NRLs** of EU member states is free of charge.

For **OFLs** of EU member states (in cooperation with NRLs) a participation fee of **150 €** applies.

The participation fee for other official laboratories and commercial laboratories is **250 €**.

The participation fee for universities/research institutes is **100 €**.

Invoices for participation will be sent before sending of the final report and the certificate of participation. In case of registration for the ILS and not reporting of any results a fee of **100 €** will be charged.

9. Registration

For registration for this ILS, participants are asked to fill out the respective online registration form using the following link.

Registration form

(direct link: https://ec.europa.eu/eusurvey/runner/EURL-ILS-BC_2022-FO_Registration)

Please register using the online registration form linked above. This is an ongoing registration in 2023, until 15 October 2023.

Registration for this ILS and reporting of results/method information is **only** possible using the supplemented online forms. Please contact us directly in case of technical difficulties.

10. Time schedule

Who	What	When
EURL POPs	Second Announcement	16 December 2022
Participants	Registration	Ongoing until 15 October 2023
EURL POPs	Shipment of test samples, sending of instructions and laboratory code	From 16 January 2023 (ongoing)
Participants	Confirmation of receipt of test sample	within 7 days
Participants	Reporting of results <i>First deadline</i> Second deadline Third D deadline	<i>Until 15 October 2022</i> Until 31 April 2023 Until 31 October 2023
EURL POPs	Evaluation and preparation of preliminary reports	May 2023 and November 2023
EURL/NRLs	Discussion at COM/EURL/NRL workshop with NRLs	May 2023 and November 2023
EURL POPs	Sending of final report to all participants	Not before evaluation of third round of results

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