

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder

2023

EURL-PT-POP_2301-MP

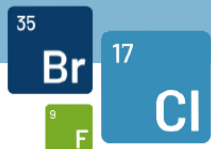
Announcement

12 December 2022



Summary

Test sample	FOOD: Milk Powder [2301-MP]
Analytes of interest	<p>PCDD/Fs (17 2,3,7,8-substituted PCDD/Fs)</p> <p>PCBs (12 DL-PCBs, 6 NDL-PCBs)</p> <p>Mandatory for NRLs: PBDEs (BDE-28, -47, -49, -99, -100, -153, -154, -183, -209) HBCDDs (α-HBCDD, β-HBCDD, γ-HBCDD or total HBCDD)</p> <p>Optional for NRLs: PFASs (PFOS, PFOA, PFNA, PFHxS, Sum of PFOS, PFOA, PFNA, PFHxS) Other PFASs (perfluoroalkylcarboxylic acids, perfluoroalkylsulfonic acids, perfluoroalkane sulphonamides)</p>
Participants	NRLs, OFLs, other official laboratories, commercial laboratories performing the analysis of samples taken by food business operators
Statistical evaluation	ISO 13528:2022, IUPAC Protocol
Participation fee	Participation fee for OFLs, other official and commercial laboratories
Registration	Online registration until 20 January 2023
Shipment of samples	07 February 2023
Deadline for reporting of results	<p>PCDD/Fs and PCBs: 21 April 2023</p> <p>PBDEs and HBCDDs: 28 April 2023</p> <p>PFASs: 28 April 2023</p>



1. Introduction

This proficiency test (PT) on the determination of **PCDD/Fs, PCBs, PBDEs, HBCDDs** and **PFASs in milk powder** is organized by the EURL for halogenated POPs in Feed and Food to be performed between February and April 2023. The objective is to assess analytical performance of laboratories and inter-laboratory comparability of results from analyses of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in one sample of **milk powder**.

National Reference Laboratories (NRLs) for halogenated POPs in Feed and Food from EU member states are requested to participate as part of their work programme for 2023. NRLs are invited to encourage the participation of Official Laboratories (OFLs) from their member states as part of their duties following Article 101 of regulation (EU) 2017/625 of the European Parliament and of the Council of 15 March 2017. Furthermore, participation of OFLs will allow the extension of the data basis for calculation of assigned values and evaluation of results.

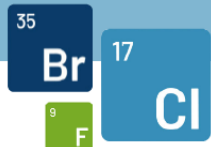
Official laboratories and **commercial laboratories** performing the analysis of samples taken by food business operators are invited to participate in this interlaboratory study. First results will be discussed by representatives of European Commission, NRLs and the EURL at the EURL/NRL workshop in May 2023 in Berlin, Germany.

Participating laboratories will receive the evaluation of the PT 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

The test sample is prepared from commercially available food and fortified with some analytes of interest using analytical standards or technical mixtures of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs. Each participant will receive about **90 g** of the test sample.



3. Analytes of interest

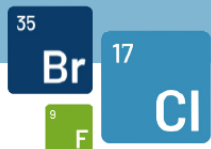
NRLs for halogenated POPs in feed and food are requested to determine the following parameters:

- **PCDD/Fs and PCBs:**
 - 17 2,3,7,8-substituted PCDD/Fs
 - WHO-PCDD/F-TEQ (using WHO2005-TEF)
 - 12 dioxin-like PCBs
 - WHO-PCB-TEQ (using WHO2005-TEF)
 - WHO-PCDD/F-PCB-TEQ (using WHO2005-TEF)
 - Six non-dioxin-like PCBs (indicator PCBs): PCB 28, 52, 101, 138, 153, 180
 - Sum of six non-dioxin-like PCBs (indicator PCBs): Sum of PCB 28, 52, 101, 138, 153, 180
 - PCDD/F-PCB-BEQ, PCDD/F-BEQ and/or PCB-BEQ, if applicable (using bioanalytical screening methods)
- **PBDEs and HBCDDs:**
 - PBDEs: BDE-28, BDE-47, BDE-49, BDE-99, BDE-100, BDE-153, BDE-154, BDE-183, BDE-209
 - Sum of 8 PBDEs (without BDE-209), sum of 9 PBDEs (with BDE-209)
 - HBCDDs: α -HBCDD, β -HBCDD, γ -HBCDD
 - Sum of α -, β -, γ -HBCDD (using HPLC methods) or total HBCDD (using GC methods)
- **PFASs**
 - Total perfluorooctane sulfonic acid (total PFOS¹), perfluorooctanoic acid (PFOA), perfluorononanoic acid (PFNA), perfluorohexane sulfonic acid (PFHxS)
 - Sum of total PFOS¹, PFOA, PFNA, PFHxS

NRLs for halogenated POPs in feed and food are encouraged to determine the following additional parameters for PFASs:

- **Optional PFASs**
 - **Perfluoroalkylsulfonic acids (PFASs):** perfluorobutanesulfonic acid (PFBS), perfluoropentanesulfonic acid (PFPeS), perfluoroheptanesulfonic acid (PFHpS), linear perfluorooctanesulfonic acid (L-PFOS), branched perfluorooctanesulfonic acids (br-PFOS), perfluorononanesulfonic acid (PFNS), perfluorodecanesulfonic acid (PFDS), perfluoroundecane sulfonic acid (PFUnDS), perfluorododecane sulfonic acid (PFDoDS), perfluorotridecane sulfonic acid (PFTrDS)

¹ sum of linear and branched stereoisomers, whether they are chromatographically separated or not



- **Perfluoroalkylcarboxylic acids (PFCAs):** perfluorobutanoic acid (PFBA), perfluoropentanoic acid (PFPeA), perfluorohexanoic acid (PFHxA), perfluoroheptanoic acid (PFHpA), perfluorodecanoic acid (PFDA), perfluoroundecanoic acid (PFUnDA), perfluorododecanoic acid (PFDoDA), perfluorotridecanoic acid (PFTTrDA), perfluorotetradecanoic acid (PFTeDA)
- Perfluorooctane sulphonamide (**FOSA**)
- 2,2,3-Trifluoro-3-[1,1,2,2,3,3-hexafluor-3-(trifluoromethoxy)propoxy]-propionic acid (**DONA**)
- 2,3,3,3-tetrafluoro-2-(heptafluoropropoxy)-propanoic acid (**GenX**)
- Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonate (major component of **F-53B**)
Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonate (minor component of **F-53B**)
- 1-Propanaminium, N,N-dimethyl-N-oxide-3-[[[(3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluorooctyl)sulfonyl]amino]-, hydroxide (**Capstone A**)
- 1-Propanaminium, N-(carboxymethyl)-N,N-dimethyl-3-[[[(3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluorooctyl)sulfonyl]amino]-, hydroxide (**Capstone B**)

4. Methods

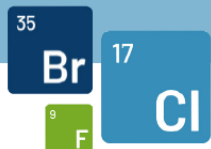
One or more of the following **detection methods** can be applied:

- GC-HRMS-, GC-MS/MS-methods or other alternative methods for PCDD/Fs and dioxin-like PCBs
- Bioanalytical screening methods for PCDD/Fs and dioxin-like PCBs
- Any kind of method for indicator PCBs, PBDEs, HBCDDs and PFASs

All analyses must be performed in the participant's own laboratory with its own personnel and equipment.

5. Participation

NRLs for halogenated POPs in feed and food shall participate in this EURL PT and report the mandatory analytes of interest. From NRLs not participating in this PT or not reporting the mandatory analytes an explanation justifying the non-participation will be requested. In case of lack of collaboration the suggested "protocol for management of underperformance in comparative testing or lack of collaboration of NRLs" provided by DG SANTE will be followed. A coordination of the participation of OFLs through NRLs is required. The EURL will send the samples only to the NRLs, including the samples for the OFLs in the respective member state, if applicable. For the shipment of the sample by these NRLs to the OFLs a written agreement between EURL and respective NRLs concerning responsibility for forwarding of samples will be provided.



6. Statistical evaluation of results

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

- ISO 13528:2022, 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).

7. 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).

8. Confidentiality

The identity of participating laboratories will be kept confidential.

For NRLs of EU member states, the suggested “protocol for management of underperformance in comparative testing or lack of collaboration of National Reference Laboratories (NRLs)” will be followed. The confidentiality of NRLs will be kept according to this protocol.

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.

9. Participation fee

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

For **OFLs of EU member states (in cooperation with NRLs)** the following participation fees have to be paid:

- 250 € for determination of PCDD/Fs and/or DL-PCBs, NDL-PCBs
- 150 € for determination of PCDD/Fs, DL-PCBs using bioanalytical screening methods only
- 150 € for determination of NDL-PCBs only
- 150 € for determination of PBDEs, HBCDDs only
- 150 € for determination of PFASs only



In case of analysis of different groups of analytes (PBDEs, HBCDDs, PFASs) in the same sample the additional costs per group are 50 € (e.g. for additional analysis of PBDEs and/or HBCDDs and/or PFASs in addition to PCDD/Fs or PCBs).

The participation fees for **other official laboratories and commercial laboratories** are:

- 350 € for determination of PCDD/Fs and/or DL-PCBs, NDL-PCBs
- 250 € for determination of PCDD/Fs, DL-PCBs using bioanalytical screening methods only
- 250 € for determination of NDL-PCBs only
- 250 € for determination of PBDEs, HBCDDs only
- 250 € for determination of PFASs only

In case of analysis of different groups of analytes (PBDEs, HBCDDs, PFASs) in the same sample the additional costs per group are 75 €.

Invoices for participation of **OFLs and other official and commercial laboratories** will be sent before sending of the final report and the certificate of participation. In case of registration for the PT and not reporting of any results a fee of 150 € will be charged.

10. Registration

For registration for this interlaboratory study, 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-PT-POP_2301-MP_Registration)

Please register using the online registration form linked above until 20 January 2023.

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

11. Time schedule

Who	What	When
EURL POPs	Announcement	09 December 2022
Participants	Registration	until 20 January 2023
EURL POPs	Shipment of test samples, sending of instructions and laboratory code	07 February 2023
Participants	Confirmation of receipt of test sample	within 7 days
Participants	Reporting of results	
	PCDD/Fs and PCBs	21 April 2023
	PBDEs and HBCDDs	28 April 2023
	PFASs	28 April 2023
EURL POPs	Evaluation and preparation of a preliminary report	May 2023
EURL/NRLs	Discussion at COM/EURL/NRL workshop	16/17 May 2023
EURL POPs	Sending of final report to all participants	September 2023

EURL for halogenated POPs in Feed and Food
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