

# **EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS** in Powdered Infant Milk / Formula 2024 EURL-ILS-BCF 2403-PIM

# FOOD

## Announcement

26 June 2024





EURL for halogenated POPs in Feed and Food c/o State Institute for Chemical and Veterinary Analysis Freiburg









## Summary

Test sample	FOOD: Powdered Infant Milk [2403-PIM]		
Analytes of interest Brominated	<ul> <li>PBDEs (Nine PBDE congeners)</li> <li>HBCDDs (Three stereoisomers and/or total HBCDD)</li> <li>TBBPA</li> <li>eBFRs (PBB, HBBz, PBT, PBEB, BTBPE, DBDPE, TBX, DBE-DBCH, BEH-TEBP, EH-TBB)</li> <li>PBDD/Fs (Thirteen 2,3,7,8-substituted PBDD/Fs)</li> <li>PXDD/Fs (Six 2,3,7,8-substituted PXDD/Fs)</li> <li>PBB (PBB-153)</li> </ul>		
Chlorinated	PCNs (26 PCN congeners) PCAs (main constituent of CPs)		
Fluorinated	<b>PFAS</b> (PFOS, PFOA, PFNA, PFHxS, Sum parameters) <b>other PFAS</b> (PFCAs, PFSAs etc.)		
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 13528:2022, IUPAC Protocol		
Participation fee	Participation fee for OFLs, other official and commercial laboratories, research institutes and universities		
Registration	until <b>17 July 2024</b>		
Shipment of samples	23 July 2024		
Reporting of results	Deadline (all analytes): 04 October 2024		



## 1. Introduction

This interlaboratory study (ILS) on the determination of several **brominated contaminants** (BCons), polychlorinated naphthalenes (PCNs), polychlorinated alkanes (PCAs – main constituent of CPs) and per- and polyfluorinated substances (PFAS) in Powdered Infant Milk / Formula is organized by the EURL for halogenated POPs in Feed and Food to be performed between July and October 2024. The objective is to assess analytical performance of laboratories and interlaboratory comparability of results from analyses of PBDEs, HBCDDs, TBBPA, eBFRs, PBDD/Fs, PXDD/Fs, PBB, PCNs, PCAs and PFAS in a Powdered Infant Milk test material.

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. First results will be discussed by representatives of European Commission, NRLs and the EURL at the meetings of the CWGs "BCons and PCNs" and "CPs" in October 2024 and the EURL/NRL workshop in November 2024.

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 Powdered Infant Milk and fortified with analytes of interest. Test sample B is prepared from the same commercially available Powdered Infant Milk but not fortified with analytes of interest.

Each participant will receive about **100 g** of each test sample A in a HDPE bottle. The test sample B can also be ordered if needed. Laboratories that report results on PCAs are strongly encouraged to analyse samples A and B in order to account for a potential background contamination of the matrix.

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

- $\alpha$ -HBCDD,  $\beta$ -HBCDD,  $\gamma$ -HBCDD stereoisomers
- Sum of  $\alpha$ -,  $\beta$ -,  $\gamma$ -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)
- PBEB (1,2,3,4,5-Pentabromo-6-ethylbenzene; CAS 85-22-3)
- BTBPE (1,1'-[1,2-Ethanediyl-bis(oxy)]bis[2,4,6-tribromo]benzene; CAS 37853-59-1)
- DBDPE (1,1'-(1,2-Ethanediyl)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)

#### eBFR group II

- DBE-DBCH (1,2-Dibromo-4-(1,2-dibromoethyl)cyclohexane CAS 3322-93-8)
- BEH-TEBP (Bis(2-ethylhexyl)-tetrabromophthalate CAS 26040-51-7)
- EH-TBB (2-Ethylhexyl-2,3,4,5-tetrabromobenzoate CAS 183658-27-7)



#### 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
- PBB 153

#### **Chlorinated Contaminants:**

- PCN group
  - Priority List: Individual PCNs 42, 48, 53, 59, 63, 69, 70, 73, 74, 75
  - Priority List: Pairs of PCNs 28/36, 52/60, 66/67, 64/68, 71/72 (difficult to separate chromatographically)
  - Additional congeners: 27, 31, 46, 49, 50, 65
  - Sum of all congeners determined by the lab

#### Polychlorinated alkanes (PCAs)

- ΣPCAs-C<sub>10-13</sub> Short chained PCAs (SC-PCAs)
- ΣPCAs-C<sub>14-17</sub> Medium chained PCAs (MC-PCAs)
- ΣPCAs-C<sub>18-20</sub> Long chained PCAs (LC-PCAs)
- ΣPCAs-C<sub>18-x</sub> (x>20) very long chained PCAs (vLCP-CAs)
- Sum of PCAs / total PCAs, Sum of S- and MC-PCAs

#### **Fluorinated Contaminants:**

- Main PFAS
  - Total perfluorooctane sulfonic acid (total PFOS<sup>1</sup>), perfluorooctanoic acid (PFOA), perfluorononanoic acid (PFNA), perfluorohexane sulfonic acid (PFHxS)
  - Sum of total PFOS<sup>1</sup>, PFOA, PFNA, PFHxS
- Additional PFSAs and PFCAs
  - **PerfluoroalkyIsulfonic acids (PFSAs):** perfluorobutanesulfonic acid (PFBS), perfluoropentanesulfonic acid (PFPeS), perfluoroheptanesulfonic acid (PFHpS), linear perfluoroctanesulfonic acid (L-PFOS), branched perfluoroctanesulfonic acids (br-PFOS),

<sup>&</sup>lt;sup>1</sup> sum of linear and branched stereoisomers, whether they are chromatographically separated or not

perfluorononanesulfonic acid (PFNS), perfluorodecanesulfonic acid (PFDS), perfluoroundecane sulfonic acid (PFUnDS), perfluorododecane sulfonic acid (PFDoDS), perfluorotridecane sulfonic acid (PFTrDS)

- **Perfluoroalkylcarboxylic acids (PFCAs):** perfluorobutanoic acid (PFBA), perfluoropentanoic acid (PFPeA), perfluorohexanoic acid (PFHxA), perfluoroheptanoic acid (PFHpA), perfluorodecanoic acid (PFDA), perfluorodecanoic acid (PFDoDA), perfluorotridecanoic acid (PFTrDA), perfluorotetradecanoic acid (PFTeDA)
- Other PFAS
  - 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

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 13528:2022, 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).

## 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 **150 €**.

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  $100 \in$  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-BCF\_2403-PIM\_Registration

Please register using the online registration form linked above until 17 July 2024.

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	Announcement	26 June 2024
Participants	Registration	until 17 July 2024
EURL POPs	Shipment of test samples, sending of instructions and laboratory code	23 July 2024
Participants	Confirmation of receipt of test sample	within 7 days
Participants	Reporting of results	04 October 2024
EURL POPs	Evaluation and preparation of preliminary reports	October 2024
EURL/NRLs	Discussion at EURL/NRL CWG meetings and COM/EURL/NRL workshop with NRLs	October 2024 and November 2024
EURL POPs	Sending of final report to all participants	July 2025

EURL for halogenated POPs in Feed and Food c/o State Institute for Chemical and Veterinary Analysis of Food Freiburg

Theresa Zwickel, Jakob Hauns Phone: +49 761 8855 500 E-Mail: <u>eurl-pops@cvuafr.bwl.de</u>