EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder 2023

EURL-PT-POP_2301-MP

FOOD

Report

PFASs

(Report Version 1.0)

23 February 2024

EURL for halogenated POPs in Feed and Food
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Summary

Test sample	**FOOD:** Milk Powder [2301-MP]
Analytes of interest	
Mandatory for NRLs:	**PFASs** (PFOS, PFOA, PFNA, PFHxS, Sum of PFOS, PFOA, PFNA, PFHxS)
Optional for NRLs:	**Other PFASs** (perfluoroalkylcarboxylic acids, perfluoroalkylsulfonic acids, perfluoroalkane sulfonamides)
Methods	Any kind of method
Participants	NRLs, OFLs, other official laboratories, commercial laboratories performing the analysis of samples taken by food business operators
Statistical evaluation	ISO 13528:2022 [1], IUPAC Protocol [2]
Report of final results	23 February 2024 (Version 1.0)
Publication	EURL POPs reserves all rights to publish and present the anonymised results of the interlaboratory study in scientific journals and/or during conferences.

1. Structure of the ILS, test material and analytes

This proficiency test (PT) on the determination of **PCDD/Fs, PCBs, PBDEs, HBCDDs** and **PFASs** in **milk powder** was organized by the EURL for halogenated POPs in Feed and Food to be performed between February and April 2023. The objective was to assess analytical performance of laboratories and interlaboratory 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 were requested to participate as part of their work programme for 2023. NRLs were 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 allowed the extension of the data basis for calculation of assigned values and evaluation of results.

Other official laboratories and **commercial laboratories** performing the analysis of samples taken by food business operators were invited to participate in this proficiency test.

First results were discussed by representatives of European Commission, NRLs and the EURL at the EURL/NRL workshop in May 2023 in Berlin, Germany.

1.1. Samples and coding

The test sample was prepared from commercially available food and fortified with analytes of interest using analytical standards or technical mixtures of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs.

Milk powder	**Sample no. 2301-MP-xxx**

Each participant received about **90 g** of the test sample in a HDPE bottle.

1.2. Analytes of interest

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

- **PFASs**
  - Total perfluorooctane sulfonic acid (total PFOS[1]), perfluorooctanoic acid (PFOA), perfluorononanoic acid (PFNA), perfluorohexane sulfonic acid (PFHxS)
  - Sum of total PFOS[1], 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 (PFSAs):** perfluorobutanesulfonic acid (PFBS), perfluoropentanesulfonic acid (PFPeS), perfluoroheptanesulfonic acid (PFHpS), linear perfluoroctanesulfonic acid (L-PFOS), branched perfluoroctanesulfonic acids (br-PFOS), 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), perfluoroundecanoic acid (PFUnDA), perfluorododecanoic acid (PFDoDA), perfluorotridecanoic acid (PFTrDA), 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**)

1.3. Methods

All kinds of detection and quantification methods could be applied.

[1] Total PFOS: sum of linear and branched stereoisomers, whether they are chromatographically separated or not

1.4. Coding of laboratories and 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.

1.5. Results of PFAS

Laboratories should:

- use their own reference standards for identification and quantification,
- report results for each analyte,
- report the limit of quantification (LOQ), at least for each non-quantified analyte,
- give method information and
- give information about the accreditation of the laboratory according to ISO/IEC 17025 *(for metrological traceability of consensus values of participants used as assigned values).*

Results had to be reported in **µg/kg wet weight (w. w.).**

2. Participating laboratories

This proficiency test was open for participation of:

- National Reference Laboratories (NRLs) of EU member states
- National Reference Laboratories of other European countries
- Official laboratories
- Commercial laboratories

129 laboratories registered for this proficiency test. 49 laboratories reported results for PFASs. One laboratory reported two sets of results.

Table 1: Participating laboratories

Participating laboratories	Region	No. of participants
National Reference Laboratories	European Union Other Countries	17 -
Official Laboratories	European Union Other European Countries Africa Americas Asia Oceania	21 - - - - 1
Commercial Laboratories	European Union Other European Countries Africa Americas Asia Oceania	9 - - - - 1
	Total	**49**

2.1. Number of reported results

Table 2: Reported results for individual PFAS substances for milk powder (2301-MP) of all laboratories

Analyte	Abbreviation	2301-MP
Perfluorobutanoic acid	(PFBA)	31
Perfluoropentanoic acid	(PFPeA)	40
Perfluorohexanoic acid	(PFHxA)	43
Perfluoroheptanoic acid	(PFHpA)	42
Perfluorooctanoic acid	(PFOA)	50
Perfluorononanoic acid	(PFNA)	49
Perfluorodecanoic acid	(PFDA)	43
Perfluoroundecanoic acid	(PFUnDA)	41
Perfluorododecanoic acid	(PFDoDA)	38
Perfluorotridecanoic acid	(PFTrDA)	38
Perfluorotetradecanoic acid	(PFTeDA)	38

Analyte	Abbreviation	2301-MP
Perfluorobutanesulfonic acid	(PFBS)	41
Perfluoropentanesulfonic acid	(PFPeS)	30
Perfluorohexanesulfonic acid	(PFHxS)	49
Perfluoroheptanesulfonic acid	(PFHpS)	36
Linear Perfluorooctanesulfonic acid	(L-PFOS)	42
Perfluorononanesulfonic acid	(PFNS)	28
Perfluorodecanesulfonic acid	(PFDS)	35
Perfluoroundecanesulfonic acid	(PFUnDS)	10
Perfluorododecanesulfonic acid	(PFDoDS)	21
Perfluorotridecanesulfonic acid	(PFTrDS)	11
Perfluorooctane sulphonamide	(FOSA)	17
2,2,3-Trifluoro-3-[1,1,2,2,3,3-hexafluor-3-(trifluoromethoxy)propoxy]-propionic acid	(DONA)	21
2,3,3,3-tetrafluoro-2-(heptafluoropropoxy)-propanoic acid	(GenX)	18
Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonate	(major component of F-53B)	14
Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonate	(minor component of F-53B)	14
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)	2
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)	2

Table 3: Reported results for PFASs sum parameters for milk powder (2301-MP) of all laboratories

Analyte	2301-MP
Sum of branched perfluorooctanesulfonic acids (br-PFOS)	36
Sum of branched & linear perfluorooctanesulfonic acids (Total-PFOS)	47
Sum of Total-PFOS, PFOA, PFNA, PFHxS (upper bound)	48
Sum of Total-PFOS, PFOA, PFNA, PFHxS (lower bound)	48

2.2. Accreditation

Table 4: Reported accreditation according to ISO/IEC 17025 by participants for PFASs

Milk powder	PFASs
Accreditation	37
No accreditation	12

2.3. Detection methods

Any kind of chromatographic separation and detection methods could be applied for analysis. Most of the participating laboratories applied ultra- or high-performance liquid chromatography (U/HPLC) as separation method combined with low resolution tandem mass spectrometry (MS/MS) as detection method, except two laboratories, which applied high resolution mass spectrometry (Orbitrap HRMS) as detection method.

3. Test for sufficient homogeneity

The test for sufficient homogeneity was performed according to ISO 13528:2022 [1] and the International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [2]. Therefore, 10 portions of the test samples 2301-MP were analysed in duplicate for PFOA, PFNA, PFDA, PFHxS, br-PFOS, L-PFOS, and total PFOS. The test for sufficient homogeneity was performed for the individual substances. The test materials showed sufficient homogeneity for PFSAs and PFCAs in this proficiency test. The stability check of the analytes of interest applying room temperature storage was performed according to ISO 13528:2022 [1]. The test material showed sufficient stability for PFAS for this proficiency test.

4. Determination of the assigned value

Statistical evaluation of the PT results was performed by the EURL for halogenated POPs in feed and food according to ISO 13528:2022 [1] and the International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [2].

The determination of the assigned value was performed according [1] by estimating of the assigned value as the consensus of participants' results (using only results of physico-chemical methods). The Huber robust mean was taken as assigned value after excluding extreme outliers (outside the range of ± 50% of the median of all reported results) and examination of the distribution of the remaining results using histogram and Kernel density estimation, if necessary.

Assigned values were calculated for WHO-PCDD/F-PCB-TEQ, WHO-PCDD/F-TEQ, WHO-PCB-TEQ, the sum of six non-dioxin-like PCBs and individual PCDD/F and PCB congeners (including limits of quantification (LOQs)), if possible. Additionally the median of all values is calculated.

For individual congeners (including LOQs) assigned values were only calculated according to the above mentioned procedure, if more than 2/3 of all results were above the LOQ and less than 1/3 of all results (including LOQs) were outside the range of ± 50% of the median of all reported results. Levels for individual congeners were only used for evaluation and calculation if these levels were equal to or above the LOQ; otherwise the LOQ was used instead.

Due to high variation of participants' results, no assigned values could be calculated for:

- PFBA, PFHxA, PFHpA, PFUnDA, PFDoDA, PFTrDA, PFTeDA
- PFBS, PFPeS, PFHpS, PFNS, PFDS, PFUnDS, PFDoDS, PFTrDS
- FOSA, GenX, major component of F-53B, minor component of F-53B, Capstone A, Capstone B

Since there are no traceable reference values available, the assigned values in this PT were calculated based on the Huber robust mean of the participants' results. Therefore, the assigned values are only traceable to these submitted results. Additionally the results of all participants reporting results and the results of participants having accreditation according ISO/IEC 17025 were compared for individual and sum parameters. No significant differences between the assigned values calculated for both data sets were observed (Table 5).

Table 5: Comparison of assigned values for 2301-MP for all participants and participants with reported accreditation according to ISO/IEC 17025 for PFAS

Individual compounds and sum parameters	Assigned value All participants	Assigned value ISO/IEC 17025 accreditation	Deviation
	µg/kg w.w.		%
PFPeA	0.727	0.718	1
PFOA	0.501	0.499	<1
PFNA	0.217	0.219	<1
PFDA	0.387	0.385	<1
PFHxS	0.0762	0.0769	<1
L-PFOS	0.161	0.160	<1
Br-PFOS	0.144	0.147	2
Total PFOS	0.300	0.305	<2
Sum of total PFOS, PFOA, PFNA, PFHxS (ub)	1.09	1.12	<3
Sum of total PFOS, PFOA, PFNA, PFHxS (lb)	1.07	1.09	<2

4.1. PFASs – individual substances and sum parameter

The assigned values for the test sample 2301-MP were calculated as consensus of participants' results for individual PFASs and sum parameters, taking into account the calculation criteria described above (Table 6 - 8 ; tabular summary see annex 1; Figure 1-3).

Table 6: Assigned values for Perfluoroalkylcarboxylic acids (rounded to three significant figures)

Milk powder (2301-MP)	Assigned value PFCAs µg/kg w.w.
PFPeA	0.727
PFOA	0.501
PFNA	0.217
PFDA	0.387

Table 7: Assigned values for Perfluoroalkylsulfonic acids (rounded to three significant figures)

Milk powder (2301-MP)	Assigned value PFSAs µg/kg w.w.
PFHxS	0.0762
L-PFOS	0.161
Br-PFOS	0.144
Total PFOS	0.300

Table 8: Assigned values for sum of PFOS, PFOA, PFNA, PFHxS (rounded to three significant figures)

Milk powder (2301-MP)	Assigned value µg/kg w.w.
Sum of total PFOS, PFOA, PFNA, PFHxS (ub)	1.09
Sum of total PFOS, PFOA, PFNA, PFHxS (lb)	1.07
DONA	0.407

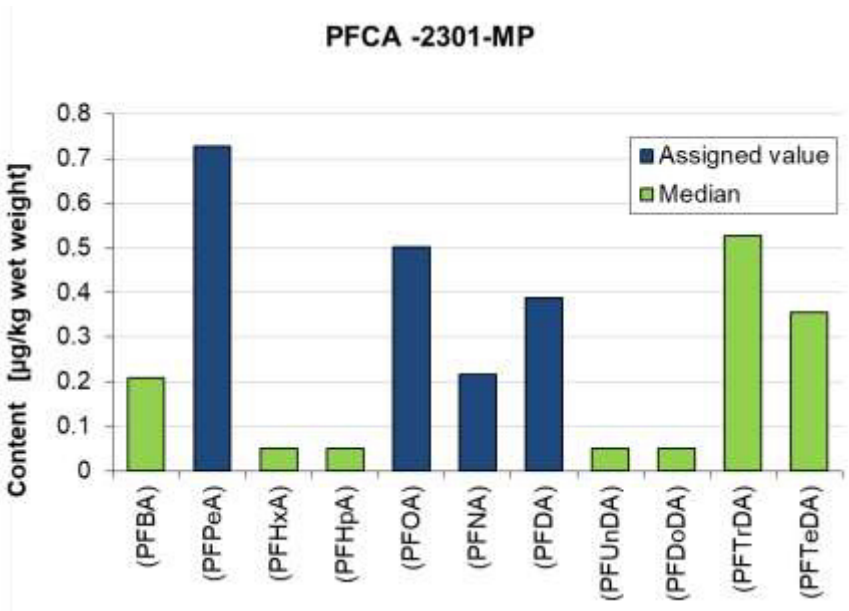

Figure 1: Assigned values (blue) and median values (green) for PFCAs individual substances for milk powder (2301-MP) [µg/kg w.w.]

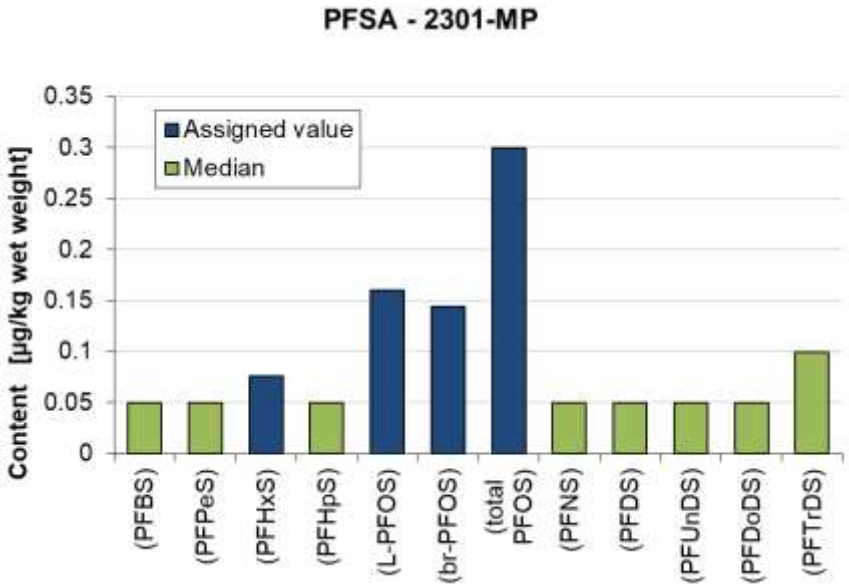

Figure 2: Assigned values (blue) and median values (green) for PFSAs individual substances for milk powder (2301-MP) [µg/kg w.w.]

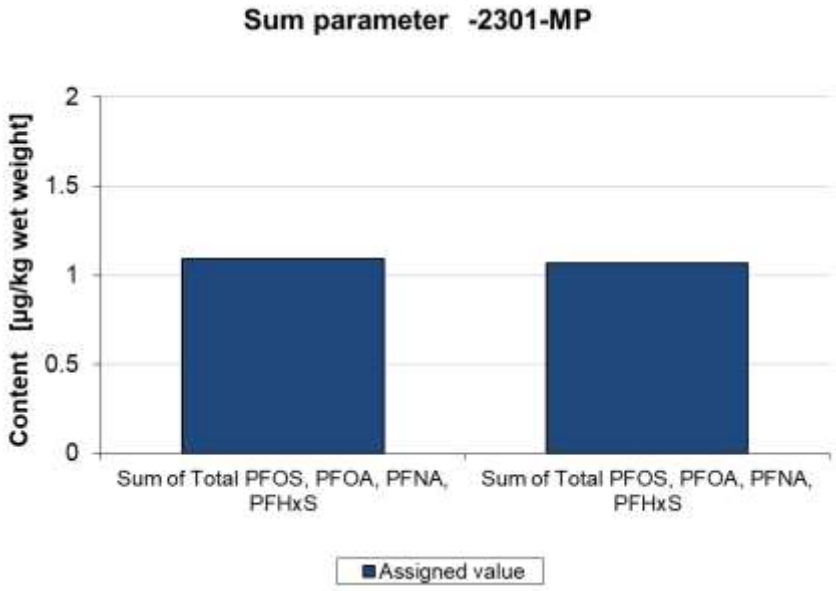

Figure 3: Assigned values (dark blue upper bound and light blue lower bound values) for sum parameters of L-PFOS, PFOA, PFNA and PFHxS and total-PFOS, PFOA, PFNA and PFHxS for milk powder (2301-MP) [µg/kg w.w.]

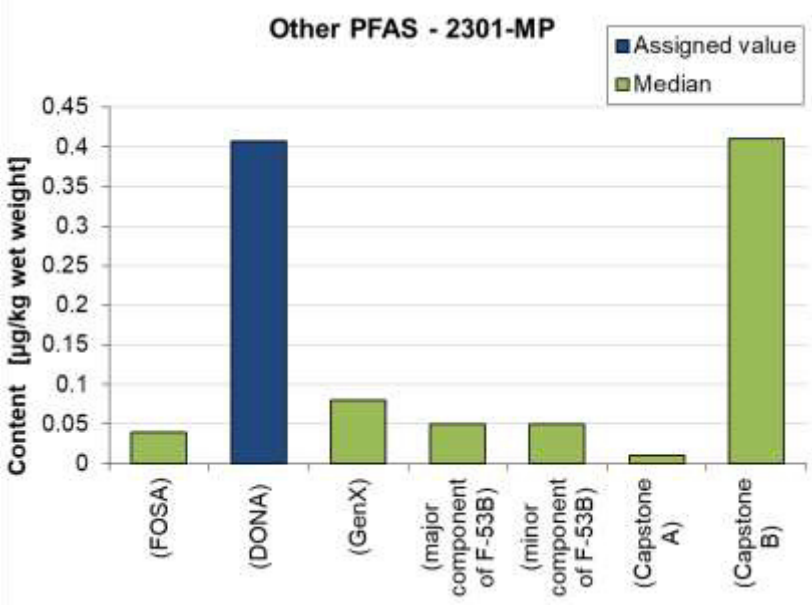

Figure 4: Assigned values (dark blue upper bound and light blue lower bound values) for other PFAS compounds for milk powder (2301-MP) [µg/kg w.w.]

5. Evaluation of results

5.1. Z-scores calculation

For evaluation of results, the z-scores were calculated according to the following formula:

$$z = \frac{(x - x_a)}{\sigma_p}$$

x: *participant's result*
x_a: *assigned value*
σ_p: *fitness-for-purpose-based standard deviation for proficiency assessment*

For individual substances and sum parameters, the standard deviation for proficiency assessment σ_p is defined as 20 %.

Z-scores for individual substances and sum parameters were only calculated and reported if levels for these congeners are equal to or above the LOQ. Otherwise, no z-scores will be given.

Interpretation of z-scores:

$\lvert z\text{-score} \rvert \leq 2$	*satisfactory performance*
$2 < \lvert z\text{-score} \rvert < 3$	*questionable performance (warning signal)*
$\lvert z\text{-score} \rvert \geq 3$	*unsatisfactory performance (action signal)*

5.2. PFASs - Participants' z-scores

Z-scores for individual substances and sum parameters were within the range of ± 2 for 82% of all participants (Table 9 - 11; tabular summary see annex 3; Figure 4-6).

Table 9: Distribution of participants' z-scores for PFCAs for milk powder (2301-MP)

Percentage of participants' results PFCAs	|z-score| ≤ 2	2 < |z-score| < 3	|z-score| ≥ 3
PFPeA	90%	5%	5%
PFOA	88%	2%	10%
PFNA	94%	2%	4%
PFDA	90%	3%	7%

Table 10: Distribution of participants' z-scores for PFSAs for milk powder (2301-MP)

Percentage of participants' results PFSAs	|z-score| ≤ 2	2 < |z-score| < 3	|z-score| ≥ 3
PFHxS	90%	5%	5%
L-PFOS	92%	3%	5%
br-PFOS	84%	13%	3%
total PFOS	82%	7%	11%

Table 11: Distribution of participants' z-scores for sum parameters and DONA for milk powder (2301-MP)

Percentage of participants' results Sum parameters	|z-score| ≤ 2	2 < |z-score| < 3	|z-score| ≥ 3
Sum of t-PFOS, PFOA, PFNA, PFHxS (ub)	86%	8%	6%
Sum of t-PFOS, PFOA, PFNA, PFHxS (lb)	86%	8%	6%
DONA	85%	-	15%

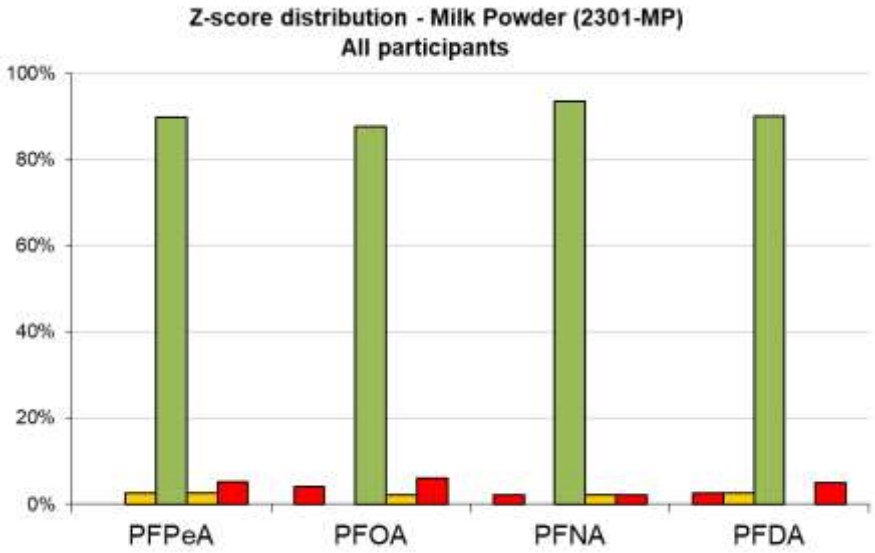

Figure 5: Distribution of participants' z-scores for individual PFCAs for milk powder (2301-MP) [Green bars: -2 ≤ z-score ≤ 2, orange bars: -3 < z-score < -2, 2 < z-score < 3, red bars: z-score ≤ -3, z-score ≥ 3]

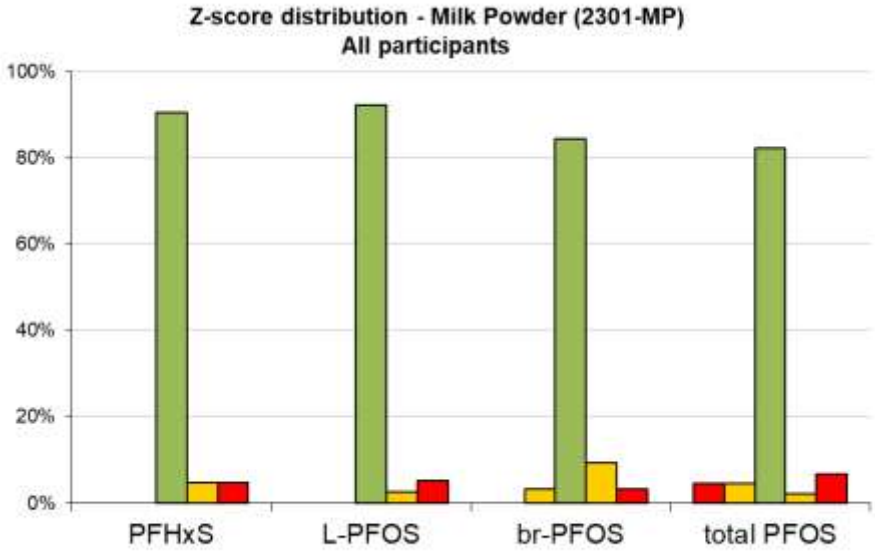

Figure 6: Distribution of participants' z-scores for individual PFSAs for milk powder (2301-MP) [Green bars: -2 ≤ z-score ≤ 2, orange bars: -3 < z-score < -2, 2 < z-score < 3, red bars: z-score ≤ -3, z-score ≥ 3]

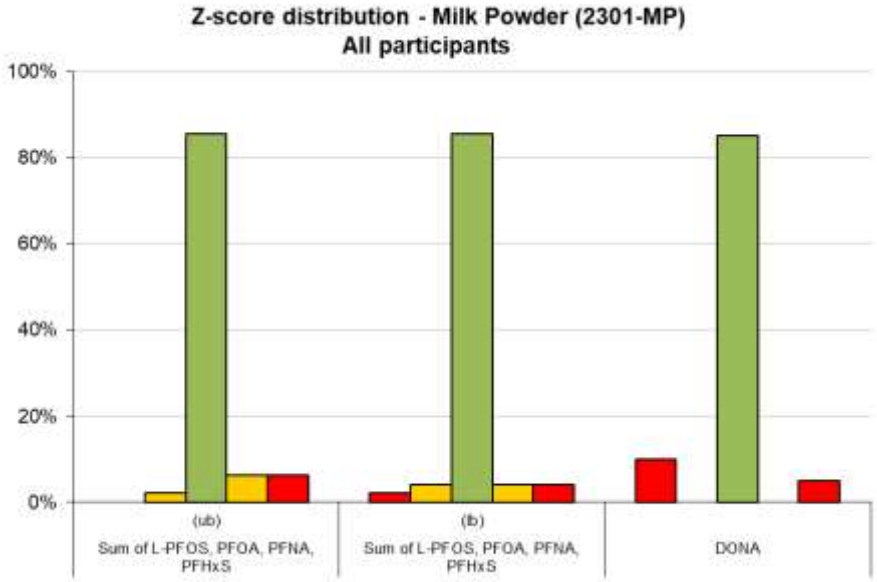

Figure 7: Distribution of participants' z-scores for sum parameters and DONA for milk powder (2301-MP) [Green bars: -2 ≤ z-score ≤ 2, orange bars: -3 < z-score < -2, 2 < z-score < 3, red bars: z-score ≤ -3, z-score ≥ 3]

6. Participants' feedback

A questionnaire for feedback from participants of this EURL proficiency test was available as online survey between 15 May 2023 and 23 June 2023. The survey was anonymous, but participants could also give their laboratory name. The identity of the laboratories is kept confidential. The survey included several questions related to different topics (participants' information, organization of the proficiency test, PT test samples and evaluation of results and summary of data) and a possibility to include comments and further suggestions. In total, 2 laboratories (1.5 % of all PT participants) replied to this survey.

Participants

Type of laboratory	Answers
National Reference Laboratory (NRL)	0
Official Laboratory (OFL)	0
Commercial laboratory	2
Other (e.g. research and development)	0
No Answer	0

General aspects

How satisfied are you with the organization of this proficiency test in general? Please rate the parts below according to your experience, with 0 stars meaning "no opinion" and 5 stars meaning "full satisfaction".

Announcement	
Instructions	
Sample shipment	
Reporting of results	
Preliminary report	

Specific aspects of this proficiency test

We would like to know a bit more about specific aspects of this proficiency test. Please rate the aspects below according to your experience, with 0 stars meaning "no opinion" and 5 stars meaning "full satisfaction".

Was all necessary information for participation and performance of the PT provided in an understandable way?	
Was the time frame acceptable?	
Was the handling of EUSurvey as webtool for reporting and source of instructions manageable?	
Was the evaluation of participant's results and the information in the preliminary report clear and comprehensible?	

Additional comments:

- report was very comprehensive and good; convoluted structure of the document does make it difficult to read
- it is easier with the webtool than sending email with an excel file
- the delay to give the preliminary results was very short

Was the selected sample adequate for the goal to assess analytical performance of laboratories in relevant matrices?

Choice of matrix and Level of contamination	

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. Results of participants

An overview of the PFASs results for the PT test sample milk powder (2301-MP) are given in the following annexes. Laboratories are coded according to the laboratory codes sent after registration.

9. References

[1] ISO 13528:2022, Statistical methods for use in proficiency testing by interlaboratory comparisons, International Organization for Standardization

[2] M. Thompson, S.L.R. Ellison, R. Wood: The International Harmonized Protocol For The Proficiency Testing Of Analytical Chemistry Laboratories, Pure Appl. Chem., Vol. 78, No. 1, pp. 145-196, 2006.

9. Annex

Milk Powder – 2301-MP	
1	Assigned values – PFCAs, PFSAs and sum parameters
2	Participants' results – Tables – PFCAs, PFSAs, sum parameters and other PFAS
3	Participants' z-scores – Tables – PFCAs, PFSAs, sum parameters and DONA
4	Participants' z-scores – Charts – PFCAs, PFSAs, sum parameters and DONA
5	Test for sufficient homogeneity and stability – PFCAs and PFSAs
6	Overview participants' methods – Weighed sample, internal and recovery standards and comments
7	Overview participants' methods – Extractions, clean-up and detection
8	Overview participants' methods – Measurement uncertainty and Limit of Quantification

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EURL Proficiency Test on Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Milk Powder 2023 [EURL-PT-POP_2301-MP]

EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

23 February 2024

Annex 1: Assigned values of PFCAs, PFSAs, sum of PFOS, PFOA, PFNA, PFHxS and DONA

Test sample - Milk Powder (2301-MP)

Assigned values of individual substances and sum parameters

Estimation of the assigned value as the consensus of participants' results
Assigned value = Huber robust mean after exlusion of extreme outliers

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder 2023 [EURL-PT-POP_2301-MP]

EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Powder (2301-MP)

Perfluoroalkylcarboxylic acids (PFCAs) - Assigned values

Analyte		Result µg/kg wet weight	**Assigned value** [outliers removed]	**Robust standard deviation** [outliers removed]	**Standard uncertainty** [outliers removed]	**No. of results contributing to assigned value**	**Median** [all values]
Perfluorobutanoic acid	(PFBA)						0.208
Perfluoropentanoic acid	(PFPeA)		0.727	0.15	0.031	36	0.710
Perfluorohexanoic acid	(PFHxA)						0.0500
Perfluoroheptanoic acid	(PFHpA)						0.0500
Perfluorooctanoic acid	(PFOA)		0.501	0.082	0.015	44	0.489
Perfluorononanoic acid	(PFNA)		0.217	0.036	0.0069	43	0.215
Perfluorodecanoic acid	(PFDA)		0.387	0.066	0.013	39	0.380
Perfluoroundecanoic acid	(PFUnDA)						0.0500
Perfluorododecanoic acid	(PFDoDA)						0.0500
Perfluorotridecanoic acid	(PFTrDA)						0.526
Perfluorotetradecanoic acid	(PFTeDA)						0.355

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder 2023 [EURL-PT-POP_2301-MP]

EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Powder (2301-MP)

Perfluoroalkylsulfonic acids (PFSAs) - Assigned values

Analyte		Result µg/kg wet weight	**Assigned value** [outliers removed]	**Robust standard deviation** [outliers removed]	**Standard uncertainty** [outliers removed]	**No. of results contributing to assigned value**	**Median** [all values]
Perfluorobutanesulfonic acid	(PFBS)						0.0500
Perfluoropentanesulfonic acid	(PFPeS)						0.0500
Perfluorohexanesulfonic acid	(PFHxS)		0.0762	0.018	0.0034	44	0.0746
Perfluoroheptanesulfonic acid	(PFHpS)						0.0500
Linear Perfluorooctanesulfonic acid	(L-PFOS)		0.161	0.03	0.006	38	0.159
Sum of branched Perfluorooctanesulfonic acids	(br-PFOS)		0.144	0.037	0.0086	29	0.145
Sum of branched and linear perfluorooctanesulfonic acids	(total PFOS)		0.300	0.051	0.01	38	0.298
Perfluorononanesulfonic acid	(PFNS)						0.0500
Perfluorodecanesulfonic acid	(PFDS)						0.0500
Perfluoroundecane sulfonic acid	(PFUnDS)						0.0500
Perfluorododecane sulfonic acid	(PFDoDS)						0.0500
Perfluorotridecane sulfonic acid	(PFTrDS)						0.100

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder 2023 [EURL-PT-POP_2301-MP]

EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Powder (2301-MP)

Sum of PFOS, PFOA, PFNA, PFHxS - Assigned values

Analyte		Result µg/kg wet weight	**Assigned value** [outliers removed]	**Robust standard deviation** [outliers removed]	**Standard uncertainty** [outliers removed]	**No. of results contributing to assigned value**	**Median** [all values]
Sum of total PFOS, PFOA, PFNA, PFHxS	(ub)		1.09	0.23	0.044	45	1.11
Sum of total PFOS, PFOA, PFNA, PFHxS	(lb)		1.07	0.22	0.041	45	1.05

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder 2023 [EURL-PT-POP_2301-MP]

EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Powder (2301-MP)

Other PFAS - Assigned values

Analyte		Result µg/kg wet weight	**Assigned value** [outliers removed]	**Robust standard deviation** [outliers removed]	**Standard uncertainty** [outliers removed]	**No. of results contributing to assigned value**	**Median** [all values]
Perfluorooctane sulphonamide	(FOSA)						0.0400
2,2,3-Trifluoro-3-[1,1,2,2,3,3-hexafluor-3-(trifluoromethoxy)propoxy]-propionic acid	(DONA)		0.407	0.093	0.027	18	0.412
2,3,3,3-tetrafluoro-2-(heptafluoropropoxy)-propanoic acid	(GenX)						0.0760
Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonate	(major component of F-53B)						0.0500
Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonate	(minor component of F-53B)						0.0500
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)						0.0125
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)						0.410

EURL Proficiency Test on Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Milk Powder 2023 [EURL-PT-POP_2301-MP]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food
23 February 2024

Annex 2: Participants' results of PFCAs, PFSAs, FOSA, sum of PFOS, PFOA, PFNA, PFHxS and other PFASs

Test sample - Milk Powder (2301-MP)

* Modified/additional results reported after distribution of preliminary results to all participating laboratories

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder 2023 [EURL-PT-POP_2301-MP]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Powder (2301-MP)
Perfluoroalkylcarboxylic acids (PFCAs) - Results

LC	Sample	Result µg/kg wet weight	Perfluorobutanoic acid (PFBA)	Perfluoropentanoic acid (PFPeA)	Perfluorohexanoic acid (PFHxA)	Perfluoroheptanoic acid (PFHpA)	Perfluorooctanoic acid (PFOA)	Perfluorononanoic acid (PFNA)	Perfluorodecanoic acid (PFDA)	Perfluoroundecanoic acid (PFUnDA)	Perfluorododecanoic acid (PFDoDA)	Perfluorotridecanoic acid (PFTrDA)	Perfluorotetradecanoic acid (PFTeDA)
1	2301-MP						0.601	0.296					
2	2301-MP		< 0.5	0.81	< 0.5	< 0.5	0.6	< 0.5	< 0.5	< 0.5	< 0.5	< 1	< 1
3	2301-MP			0.647	< 0.05	< 0.05	0.432	0.192	0.337	< 0.05	< 0.05	0.485	0.293
6	2301-MP						0.46	0.21					
8	2301-MP		< 5	0.76	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 5	< 5	< 5
11	2301-MP			0.623	< 0.05	< 0.05	0.426	0.192	0.356	< 0.05	< 0.05	0.526	0.313
11A	2301-MP			0.623	< 0.05	< 0.05	0.426	0.192	0.356	< 0.05	< 0.05	0.526	0.313
14	2301-MP		< 0.8	0.73	< 0.08	< 0.08	0.47	0.22	0.38	< 0.08	< 0.08	0.44	0.37
15	2301-MP		0.71	0.87	< 0.01	< 0.01	0.48	0.21	0.44	< 0.01	< 0.01	0.85	0.78
17	2301-MP			0.448	< 0.012	< 0.012	0.604	0.222	0.4	0.064	0.028	0.0578	0.74
18	2301-MP		0.387	0.943	< 0.03	< 0.03	0.602	0.254	0.434	< 0.03	< 0.02		
19	2301-MP			0.652	< 0.00954	0.00967	0.471	0.205	0.386	< 0.174	< 0.0163		0.323
20	2301-MP				0.654	0.66	1.87	< 0.015	0.944	< 0.063		< 0.069	< 0.093
24	2301-MP						0.121	0.223	0.228			0.878	
25	2301-MP		0.16	0.518	< 0.01	< 0.01	0.451	0.19	0.302	< 0.01	< 0.01	0.612	0.313
27	2301-MP						0.49	0.22					
28	2301-MP		< 0.5	0.84	< 0.05	< 0.05	0.56	0.26	0.45	< 0.05	< 0.05	< 0.05	< 0.05
31	2301-MP			0.81	< 0.08	< 0.08	0.54	0.24	0.4	< 0.08	< 0.08		0.35
34	2301-MP		< 0.1	0.7	< 0.1	< 0.1	0.61	0.27	0.43	< 0.1	< 0.1	0.59	0.37
45	2301-MP		0.19	1.05	< 0.00035	< 0.0001	0.82	0.23	0.44	< 0.0001	< 0.0001	0.85	0.79
46	2301-MP				< 0.009	< 0.01	0.658	0.288	0.513	< 0.008	< 0.02		
47	2301-MP			1.4	< 0.05	< 0.05	0.58	0.21	0.36	< 0.05	< 0.05	0.32	0.19
49	2301-MP		0.216	0.662	< 0.02	< 0.02	0.515	0.214	0.351	< 0.02	< 0.02	0.432	0.315
51	2301-MP		0.113	0.612	< 0.1	< 0.1	0.421	0.187	0.344	< 0.1	< 0.1	0.409	0.365
53	2301-MP		0.219	1.16	< 0.05	< 0.01	0.636	0.471	0.473	< 0.05	< 0.05	0.539	0.377
55	2301-MP				0.0058	0.0106	0.551	0.267	0.458	< 0.0025	< 0.5	0.582	0.516
57	2301-MP		< 10	0.668	< 0.2	< 0.2	0.456	0.331	0.409	< 0.2	< 0.2	0.602	0.335
58	2301-MP		0.208	0.704	< 0.05	< 0.02	0.441	0.164	0.295	< 0.02	< 0.02	0.475	0.237
59	2301-MP		< 0.3	0.73	< 0.1	< 0.01	0.49	0.23	0.35	< 0.01		0.58	0.41
60	2301-MP		0.094	0.305	0.005	0.191	0.473	0.191	0.362	0.004	0.005	1.21	0.71
63	2301-MP						0.587	0.215					
76	2301-MP			0.59	< 0.1	< 0.1	0.48	0.2	0.36	< 0.1	< 0.1	0.46	0.29
77	2301-MP						0.44	0.23					
78	2301-MP						0.525	0.184					
82	2301-MP		0.17	0.792	< 0.2	< 0.05	0.488	0.228	0.413	< 0.05	< 0.05	0.422	0.371
84	2301-MP		< 0.2	< 0.1	0.74	< 0.05	0.136	0.061	0.125	< 0.15	< 0.2	< 0.2	< 0.2
93	2301-MP		0.186	0.985	< 0.03	< 0.02	0.554	0.242	0.414	< 0.02	< 0.02	0.674	0.434
95	2301-MP		< 1.26	0.71	< 0.12	< 0.07	0.41	0.17	0.32	< 0.04	< 0.05	0.18	< 0.05
96	2301-MP		0.11	0.649	< 0.05	< 0.02	0.479	0.214	0.368	< 0.05	< 0.05	0.568	< 0.05
99	2301-MP		0.13	0.56	< 0.05	< 0.05	0.3	0.16	0.32	< 0.05	< 0.05	0.21	
100	2301-MP		< 0.1	0.91	< 0.1	< 0.1	0.54	0.21	0.41			0.59	0.38
107	2301-MP		< 0.546	0.787	< 0.009	0.007	0.663	0.288	0.461	< 0.017		0.618	0.359
108	2301-MP			0.98	0.73		1.52						
109	2301-MP		0.205	0.736	< 0.05	< 0.05	0.535	0.244	0.437	< 0.05	< 0.05	0.646	0.39
110	2301-MP		0.22	1	< 0.1	< 0.1	0.78	0.3	0.68	< 0.1	< 0.1	< 1.6	< 0.62
115	2301-MP		0.16	0.71	< 0.01	< 0.01	0.48	0.22	0.37	< 0.01	< 0.01	0.44	0.34
119	2301-MP		0.11	0.68	< 0.05	< 0.05	0.44	0.21	0.37	< 0.05	< 0.05		
122	2301-MP		< 0.5	0.53	< 0.1	< 0.05	0.36	0.18	0.29	< 0.05	< 0.1	< 0.5	< 0.5
123	2301-MP		< 0.1	0.77	< 0.1	< 0.1	0.44	0.14	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
128	2301-MP		< 0.5	0.57	< 0.01	< 0.01	0.38	0.17	0.3	< 0.005	< 0.005	0.4	0.27
60*	2301-MP		0.094	0.557	0.005	0.005	0.473	0.191	0.362	0.004	0.005	0.528	0.375

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Milk Powder (2301-MP)
Perfluoroalkylsulfonic acids (PFSAs) - Results

LC	Sample	Result µg/kg wet weight	Perfluorobutanesulfonic acid (PFBS)	Perfluoropentanesulfonic acid (PFPeS)	Perfluorohexanesulfonic acid (PFHxS)	Perfluoroheptanesulfonic acid (PFHpS)	Linear Perfluorooctanesulfonic acid (L-PFOS)	Sum of branched Perfluorooctanesulfonic acid (br-PFOS)	Sum of branched and linear Perfluorooctanesulfonic acid (total PFOS)	Perfluorononane-sulfonic acid (PFNS)	Perfluorodecane-sulfonic acid (PFDS)	Perfluoroundecane-sulfonic acid (PFUnDS)	Perfluorododecane-sulfonic acid (PFDoDS)	Perfluorotridecane-sulfonic acid (PFTrDS)
1	2301-MP				0.089		0.188	0.12	0.308					
2	2301-MP		< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 1		< 1	
3	2301-MP		< 0.05	< 0.05	0.066	< 0.05	0.132	0.108	0.24	< 0.05	< 0.1	< 0.1	< 0.1	< 0.1
6	2301-MP				0.08				0.29					
8	2301-MP		< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5			
11	2301-MP		< 0.05	< 0.05	0.067	< 0.05	0.149	0.128	0.277	< 0.05	< 0.05		< 0.05	
11A	2301-MP		< 0.05	< 0.05	0.067	< 0.05	0.149	0.149	0.298	< 0.05	< 0.05		< 0.05	
14	2301-MP		< 0.072	< 0.076	0.059	< 0.038	0.14	0.13	0.27	< 0.08	< 0.078			
15	2301-MP		< 0.01		0.1	< 0.01	0.22				< 0.01			
17	2301-MP				0.074				0.25					
18	2301-MP		< 0.05	< 0.05	0.0993	< 0.05	0.281	0.203	0.484	< 0.05	< 0.1			
19	2301-MP		< 0.00838	< 0.0135	0.0647	< 0.006	0.142	0.165	0.307	< 0.00486	< 0.00814	< 0.0107	< 0.00702	< 0.0091
20	2301-MP		0.401	0.065	1.1	< 0.14			1.53		< 0.035			
24	2301-MP				0.11		0.207	0.062	0.269					
25	2301-MP		< 0.01	< 0.01	0.06	< 0.01	0.137	0.096	0.233	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
27	2301-MP				0.06		0.39	< 0.01	0.39					
28	2301-MP		< 0.05		0.09	< 0.05	0.2	0.22	0.42					
31	2301-MP				0.08	< 0.08	0.16	0.17	0.33					
34	2301-MP		< 0.1	< 0.1	0.1	< 0.1	0.17	0.14	0.31	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
45	2301-MP		< 0.0001	< 0.025	0.11	< 0.00035	0.18	0.2	0.38		< 0.00035		< 0.00035	
46	2301-MP		0.005	< 0.003	0.086	< 0.007	0.19	0.204	0.394					
47	2301-MP		< 0.05		0.086				0.29		< 0.05			
49	2301-MP		< 0.02	< 0.02	0.069	< 0.02	0.151	0.169	0.32	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02
51	2301-MP		< 0.1	< 0.1	0.07	< 0.1	0.128	0.14	0.271		< 0.1			
53	2301-MP		< 0.01	< 0.01	0.0852	< 0.1	0.195	0.156	0.352	< 0.01	< 0.01		< 0.05	
55	2301-MP		0.0071		0.0746	0.0037	0.201	0.102	0.303		< 0.001			
57	2301-MP		< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	0.297	< 0.2	< 0.2			
58	2301-MP		< 0.01	< 0.01	0.056	< 0.01	0.0953	0.1	0.195	< 0.01	< 0.05		< 0.1	
59	2301-MP		< 0.01		0.076	< 0.01	0.155	0.1	0.255	< 0.1	< 0.1		< 0.1	< 0.1
60	2301-MP		0.005	< 0.001	0.055	0.004	0.127		0.127	< 0.001	< 0.01	< 0.01	< 0.001	< 0.01
63	2301-MP				0.0705				0.331					
76	2301-MP		< 0.1		0.06		0.16	0.14	0.3		< 0.1			
77	2301-MP				< 0.1		0.15	0.13	0.28					
78	2301-MP				0.0644		0.148							
82	2301-MP		< 0.05	< 0.05	0.072	< 0.05	0.16		0.318	< 0.2	< 0.05			
84	2301-MP		0.368		0.217		0.158		0.158					
93	2301-MP		< 0.04		0.076		0.159	0.101	0.26		< 0.05			
95	2301-MP		< 0.04	< 0.03	0.069	< 0.03	0.14	0.15	0.29	< 0.035	< 0.055	< 0.07	< 0.078	< 0.1
96	2301-MP		< 0.05	< 0.1	0.061	< 0.02	0.136	0.16	0.296	< 0.02	< 0.03	< 0.05	< 0.1	< 0.1
99	2301-MP		< 0.05	< 0.05	< 0.05	< 0.05			0.12	< 0.1	< 0.05	< 0.05	< 0.05	< 0.05
100	2301-MP		< 0.1		0.07	< 0.05	0.15	0.24	0.39	< 0.1				
107	2301-MP		< 0.012	< 0.01	0.102	< 0.01	0.141	0.139	0.28	< 0.01	< 0.016			
108	2301-MP		0.87						1.18					
109	2301-MP		< 0.05	< 0.05	0.09	< 0.05	0.187	0.17	0.357	< 0.05	< 0.05		< 0.05	
110	2301-MP		< 0.1	< 0.1	< 0.1	< 0.1	0.18	0.18	0.36	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
115	2301-MP		< 0.01	< 0.01	0.074	< 0.01	0.15	0.14	0.29	< 0.01	< 0.01		< 0.01	
119	2301-MP		< 0.05		0.06		0.15							
122	2301-MP		< 0.05	< 0.1	0.061	< 0.1	0.12	0.11	0.24	< 0.1	< 0.1			
123	2301-MP		< 0.1	< 0.1	< 0.1	< 0.1	0.234	0.223	0.46	< 0.1	< 0.1		< 0.1	
128	2301-MP		< 0.005	< 0.01	0.055	< 0.005			0.12	< 0.005	< 0.005		< 0.005	
60*	2301-MP		0.005	< 0.001	0.055	0.004	0.127		< 0.001	< 0.001	< 0.01	< 0.01	< 0.001	< 0.01
128*	2301-MP						0.12							

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Milk Powder (2301-MP)
Sum of PFOS, PFOA, PFNA, PFHxS - Results

LC	Sample	Result µg/kg wet weight	Sum of L-PFOS, PFOA, PFNA, PFHxS **(ub)**	Sum of L-PFOS, PFOA, PFNA, PFHxS **(lb)**
1	2301-MP		1.29	1.29
2	2301-MP		1.8	0
3	2301-MP		0.93	0.93
6	2301-MP			
8	2301-MP		2	
11	2301-MP		0.962	0.962
11A	2301-MP		0.983	0.983
14	2301-MP		1.02	1.02
15	2301-MP		1.01	1.01
17	2301-MP		1.15	1.15
18	2301-MP		1.44	1.44
19	2301-MP		1.05	1.05
20	2301-MP		4.51	4.5
24	2301-MP		0.723	0.773
25	2301-MP		0.934	0.934
27	2301-MP		1.16	1.16
28	2301-MP		1.33	1.33
31	2301-MP		1.2	1.2
34	2301-MP		1.29	1.29
45	2301-MP		1.54	1.54
46	2301-MP		1.43	1.43
47	2301-MP		1.64	0.85
49	2301-MP		1.12	1.12
51	2301-MP		0.949	0.949
53	2301-MP		1.54	1.54
55	2301-MP		1.19	1.19
57	2301-MP		1.16	1.16
58	2301-MP		0.856	0.856
59	2301-MP		1.05	1.05
60	2301-MP		0.846	0.846
63	2301-MP		1.2	1.2
76	2301-MP		1.04	1.04
77	2301-MP		1.05	0.95
78	2301-MP		0.921	0.921
82	2301-MP		1.1	1.1
84	2301-MP		0.573	0.573
93	2301-MP		1.13	1.13
95	2301-MP		0.94	0.94
96	2301-MP		1.05	1.05
99	2301-MP		0.66	0.61
100	2301-MP		1.21	1.21
107	2301-MP		1.33	1.33
108	2301-MP			2.7
109	2301-MP		1.23	1.23
110	2301-MP		1.5	1.4
115	2301-MP		1.1	1.1
119	2301-MP		0.86	0.86
122	2301-MP		0.84	0.84
123	2301-MP		1.14	1.04
128	2301-MP		0.73	0.73
60*	2301-MP		0.846	0.846

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Milk Powder (2301-MP)
Other PFAS - Results

LC	Sample	Result µg/kg wet weight	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**
1	2301-MP								
2	2301-MP		< 0.5	0.43	< 0.3	< 0.3	< 0.3		
3	2301-MP				< 1	< 0.05	< 0.05		
6	2301-MP								
8	2301-MP		< 0.5	< 0.5	< 0.5	< 5	< 0.5		
11	2301-MP								
11A	2301-MP								
14	2301-MP			0.42		< 0.038	< 0.08		
15	2301-MP		< 0.01						
17	2301-MP								
18	2301-MP		< 0.029	0.368					
19	2301-MP								
20	2301-MP			0.144					
24	2301-MP		< 0.01	0.286	< 0.01	< 0.01	< 0.01		
25	2301-MP								
27	2301-MP				< 0.05				
28	2301-MP								
31	2301-MP		< 0.1	0.28	< 0.1				
34	2301-MP			0.44	< 0.025				
45	2301-MP								
46	2301-MP		< 0.05						
47	2301-MP		< 0.02	0.247	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02
49	2301-MP		< 0.1	0.344	< 0.5				
51	2301-MP		< 0.01	0.402	< 1	< 0.5	< 0.5		
53	2301-MP			0.486					
55	2301-MP								
57	2301-MP			0.499	< 0.05				
58	2301-MP		< 0.01	0.44	< 0.1	< 0.01	< 0.01		
59	2301-MP		< 0.01						
60	2301-MP		< 0.1						
63	2301-MP								
76	2301-MP								
77	2301-MP			0.412	< 0.05	< 0.05	< 0.05		
78	2301-MP								
82	2301-MP		< 0.04						
84	2301-MP		< 0.065	0.32	< 0.025	< 0.03	< 0.05		< 0.8
93	2301-MP			0.389	< 0.2	< 0.05	< 0.05		
95	2301-MP			0.15		< 0.05	< 0.05		
96	2301-MP								
99	2301-MP		< 0.01	0.522	< 0.05	< 0.005	< 0.005	< 0.005	
100	2301-MP			0.8					
107	2301-MP			0.517	0.052	< 0.05	< 0.05		
108	2301-MP								
109	2301-MP		< 0.05						
110	2301-MP				< 0.1				
115	2301-MP								
119	2301-MP								
122	2301-MP								
123	2301-MP								
128	2301-MP								
60*	2301-MP								

EURL Proficiency Test on Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Milk Powder 2023 [EURL-PT-POP_2301-MP]

EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

23 February 2024

Annex 3: Participants' z-scores of PFCAs, PFSAs, sum of PFOS, PFOA, PFNA, PFHxS and DONA

Test sample - Milk Powder (2301-MP)

Z-scores of individual substances and sum parameters

Calculation of z-score on basis of assigned value

$z = (x - x_a) / \sigma_p$

x_a: assigned value

x: participant's result

σ_p: fitness-for-purpose-based standard deviation for proficiency assessment

20%: Evaluated individual substances and sum parameters

* Modified/additional results reported after distribution of preliminary results to all participating laboratories

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder 2023 [EURL-PT-POP_2301-MP]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Powder (2301-MP)
Perfluoroalkylcarboxylic acids (PFCAs) - Z-scores

LC	Sample	Z-score [σ_p = 20 %]	Perfluoropentanoic acid **(PFPeA)**	Perfluorooctanoic acid **(PFOA)**	Perfluorononanoic acid **(PFNA)**	Perfluorodecanoic acid **(PFDA)**
1	2301-MP			1.0	1.8	
2	2301-MP		0.6	1.0		
3	2301-MP		-0.6	-0.7	-0.6	-0.6
6	2301-MP			-0.4	-0.2	
8	2301-MP		0.2			
11	2301-MP		-0.7	-0.7	-0.6	-0.4
11A	2301-MP		-0.7	-0.7	-0.6	-0.4
14	2301-MP		0.0	-0.3	0.1	-0.1
15	2301-MP		1.0	-0.2	-0.2	0.7
17	2301-MP		-1.9	1.0	0.1	0.2
18	2301-MP		1.5	1.0	0.9	0.6
19	2301-MP		-0.5	-0.3	-0.3	0.0
20	2301-MP			13.7		7.2
24	2301-MP			-3.8	0.1	-2.1
25	2301-MP		-1.4	-0.5	-0.6	-1.1
27	2301-MP			-0.1	0.1	
28	2301-MP		0.8	0.6	1.0	0.8
31	2301-MP		0.6	0.4	0.5	0.2
34	2301-MP		-0.2	1.1	1.2	0.6
45	2301-MP		2.2	3.2	0.3	0.7
46	2301-MP			1.6	1.6	1.6
47	2301-MP		4.6	0.8	-0.2	-0.3
49	2301-MP		-0.4	0.1	-0.1	-0.5
51	2301-MP		-0.8	-0.8	-0.7	-0.6
53	2301-MP		3.0	1.3	5.9	1.1
55	2301-MP			0.5	1.2	0.9
57	2301-MP		-0.4	-0.4	2.6	0.3
58	2301-MP		-0.2	-0.6	-1.2	-1.2
59	2301-MP		0.0	-0.1	0.3	-0.5
60	2301-MP		-2.9	-0.3	-0.6	-0.3
63	2301-MP			0.9	0.0	
76	2301-MP		-0.9	-0.2	-0.4	-0.3
77	2301-MP			-0.6	0.3	
78	2301-MP			0.2	-0.8	
82	2301-MP		0.4	-0.1	0.3	0.3
84	2301-MP			-3.6	-3.6	-3.4
93	2301-MP		1.8	0.5	0.6	0.3
95	2301-MP		-0.1	-0.9	-1.1	-0.9
96	2301-MP		-0.5	-0.2	-0.1	-0.2
99	2301-MP		-1.1	-2.0	-1.3	-0.9
100	2301-MP		1.3	0.4	-0.2	0.3
107	2301-MP		0.4	1.6	1.6	1.0
108	2301-MP		1.7	10.2		
109	2301-MP		0.1	0.3	0.6	0.6
110	2301-MP		1.9	2.8	1.9	3.8
115	2301-MP		-0.1	-0.2	0.1	-0.2
119	2301-MP		-0.3	-0.6	-0.2	-0.2
122	2301-MP		-1.4	-1.4	-0.9	-1.3
123	2301-MP		0.3	-0.6	-1.8	
128	2301-MP		-1.1	-1.2	-1.1	-1.1
60*	2301-MP		-1.2	-0.3	-0.6	-0.3

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Milk Powder (2301-MP)
Perfluoroalkylsulfonic acids (PFSAs) - Z-scores

LC	Sample	Z-score [σ_p = 20 %]	Perfluorohexanesulfonic acid **(PFHxS)**	Linear Perfluorooctanesulfonic acid **(L-PFOS)**	Sum of branched Perfluorooctanesulfonic acid **(br-PFOS)**	Sum of branched and linear Perfluorooctanesulfonic acid **(total PFOS)**
1	2301-MP		0.8	0.8	-0.8	0.1
2	2301-MP					
3	2301-MP		-0.7	-0.9	-1.3	-1.0
6	2301-MP		0.2			-0.2
8	2301-MP					
11	2301-MP		-0.6	-0.4	-0.6	-0.4
11A	2301-MP		-0.6	-0.4	0.2	0.0
14	2301-MP		-1.1	-0.7	-0.5	-0.5
15	2301-MP		1.6	1.8		
17	2301-MP		-0.1			-0.8
18	2301-MP		1.5	3.7	2.0	3.1
19	2301-MP		-0.8	-0.6	0.7	0.1
20	2301-MP		67.2			20.5
24	2301-MP		2.2	1.4	-2.8	-0.5
25	2301-MP		-1.1	-0.7	-1.7	-1.1
27	2301-MP		-1.1	7.1		1.5
28	2301-MP		0.9	1.2	2.6	2.0
31	2301-MP		0.2	0.0	0.9	0.5
34	2301-MP		1.6	0.3	-0.1	0.2
45	2301-MP		2.2	0.6	1.9	1.3
46	2301-MP		0.6	0.9	2.1	1.6
47	2301-MP		0.6			-0.2
49	2301-MP		-0.5	-0.3	0.9	0.3
51	2301-MP		-0.4	-1.0	-0.1	-0.5
53	2301-MP		0.6	1.1	0.4	0.9
55	2301-MP		-0.1	1.2	-1.5	0.1
57	2301-MP					-0.1
58	2301-MP		-1.3	-2.0	-1.5	-1.8
59	2301-MP		0.0	-0.2	-1.5	-0.8
60	2301-MP		-1.4	-1.1		-2.9
63	2301-MP		-0.4			0.5
76	2301-MP		-1.1	0.0	-0.1	0.0
77	2301-MP			-0.3	-0.5	-0.3
78	2301-MP		-0.8	-0.4		
82	2301-MP		-0.3	0.0		0.3
84	2301-MP		9.2	-0.1		-2.4
93	2301-MP		0.0	-0.1	-1.5	-0.7
95	2301-MP		-0.5	-0.7	0.2	-0.2
96	2301-MP		-1.0	-0.8	0.6	-0.1
99	2301-MP					-3.0
100	2301-MP		-0.4	-0.3	3.3	1.5
107	2301-MP		1.7	-0.6	-0.2	-0.3
108	2301-MP					14.7
109	2301-MP		0.9	0.8	0.9	1.0
110	2301-MP			0.6	1.3	1.0
115	2301-MP		-0.1	-0.3	-0.1	-0.2
119	2301-MP		-1.1	-0.3		
122	2301-MP		-1.0	-1.3	-1.2	-1.0
123	2301-MP			2.3	2.7	2.7
128	2301-MP		-1.4			-3.0
60*	2301-MP		-1.4	-1.1		
128*	2301-MP			-1.3		

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Milk Powder (2301-MP)
Sum of Total PFOS, PFOA, PFNA, PFHxS - Z-scores

LC	Sample	Z-score [σ_p = 20 %]	Sum of Total PFOS, PFOA, PFNA, PFHxS **(ub)**	Sum of Total PFOS, PFOA, PFNA, PFHxS **(lb)**
1	2301-MP		0.9	1.0
2	2301-MP		3.3	-5.0
3	2301-MP		-0.7	-0.7
6	2301-MP			
8	2301-MP		4.2	
11	2301-MP		-0.6	-0.5
11A	2301-MP		-0.5	-0.4
14	2301-MP		-0.3	-0.2
15	2301-MP		-0.4	-0.3
17	2301-MP		0.3	0.4
18	2301-MP		1.6	1.7
19	2301-MP		-0.2	-0.1
20	2301-MP		15.7	16.0
24	2301-MP		-1.7	-1.4
25	2301-MP		-0.7	-0.6
27	2301-MP		0.3	0.4
28	2301-MP		1.1	1.2
31	2301-MP		0.5	0.6
34	2301-MP		0.9	1.0
45	2301-MP		2.1	2.2
46	2301-MP		1.6	1.7
47	2301-MP		2.5	-1.0
49	2301-MP		0.1	0.2
51	2301-MP		-0.6	-0.6
53	2301-MP		2.1	2.2
55	2301-MP		0.5	0.6
57	2301-MP		0.3	0.4
58	2301-MP		-1.1	-1.0
59	2301-MP		-0.2	-0.1
60	2301-MP		-1.1	-1.0
63	2301-MP		0.5	0.6
76	2301-MP		-0.2	-0.1
77	2301-MP		-0.2	-0.6
78	2301-MP		-0.8	-0.7
82	2301-MP		0.0	0.1
84	2301-MP		-2.4	-2.3
93	2301-MP		0.2	0.3
95	2301-MP		-0.7	-0.6
96	2301-MP		-0.2	-0.1
99	2301-MP		-2.0	-2.1
100	2301-MP		0.6	0.7
107	2301-MP		1.1	1.2
108	2301-MP			7.6
109	2301-MP		0.6	0.7
110	2301-MP		1.9	1.5
115	2301-MP		0.0	0.1
119	2301-MP		-1.1	-1.0
122	2301-MP		-1.1	-1.1
123	2301-MP		0.2	-0.1
128	2301-MP		-1.7	-1.6
60*	2301-MP		-1.1	-1.0

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Milk Powder (2301-MP)
DONA - Z-scores

LC	Sample	Z-score [σ_p = 20 %]	2,2,3-Trifluoro-3-[1,1,2,2,3,3-hexafluor-3-(trifluoromethoxy)propoxy]-propionic acid **DONA**
1	2301-MP		
2	2301-MP		0.3
3	2301-MP		
6	2301-MP		
8	2301-MP		
11	2301-MP		
11A	2301-MP		
14	2301-MP		0.2
15	2301-MP		
17	2301-MP		
18	2301-MP		-0.5
19	2301-MP		
20	2301-MP		-3.2
24	2301-MP		-1.5
25	2301-MP		
27	2301-MP		
28	2301-MP		
31	2301-MP		-1.6
34	2301-MP		0.4
45	2301-MP		
46	2301-MP		
47	2301-MP		-2.0
49	2301-MP		-0.8
51	2301-MP		-0.1
53	2301-MP		1.0
55	2301-MP		
57	2301-MP		1.1
58	2301-MP		0.4
59	2301-MP		
60	2301-MP		
63	2301-MP		
76	2301-MP		
77	2301-MP		0.1
78	2301-MP		
82	2301-MP		
84	2301-MP		-1.1
93	2301-MP		-0.2
95	2301-MP		-3.2
96	2301-MP		
99	2301-MP		1.4
100	2301-MP		4.8
107	2301-MP		1.4
108	2301-MP		
109	2301-MP		
110	2301-MP		
115	2301-MP		
119	2301-MP		
122	2301-MP		
123	2301-MP		
128	2301-MP		
60*	2301-MP		

EURL Proficiency Test on Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Milk Powder 2023 [EURL-PT-POP_2301-MP]

EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

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Annex 4: Participants' z-scores of PFCAs, PFSAs,sum of PFOS, PFOA, PFNA, PFHxS and DONA - charts

Test sample - Milk Powder (2301-MP)

Z-scores of individual substances and sum parameters

Calculation of z-score on basis of assigned value

$z = (x - x_a) / \sigma_p$

x_a: assigned value

x: participant's result

σ_p: fitness-for-purpose-based standard deviation for proficiency assessment

20%: Evaluated individual substances and sum parameters

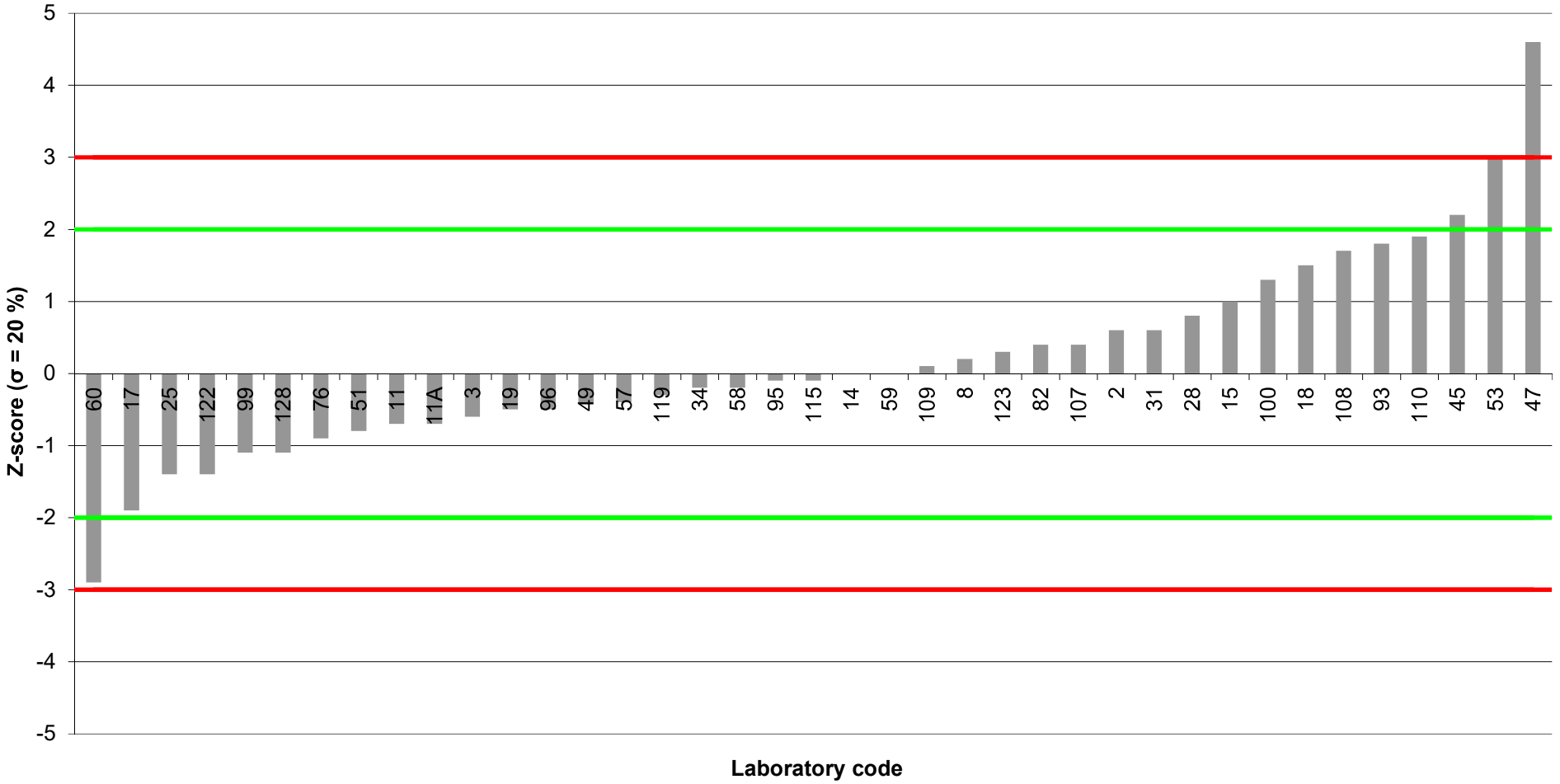
± 2 z-scores:

± 3 z-scores:

Milk Powder (2301-MP)

Perfluoropentanoic acid

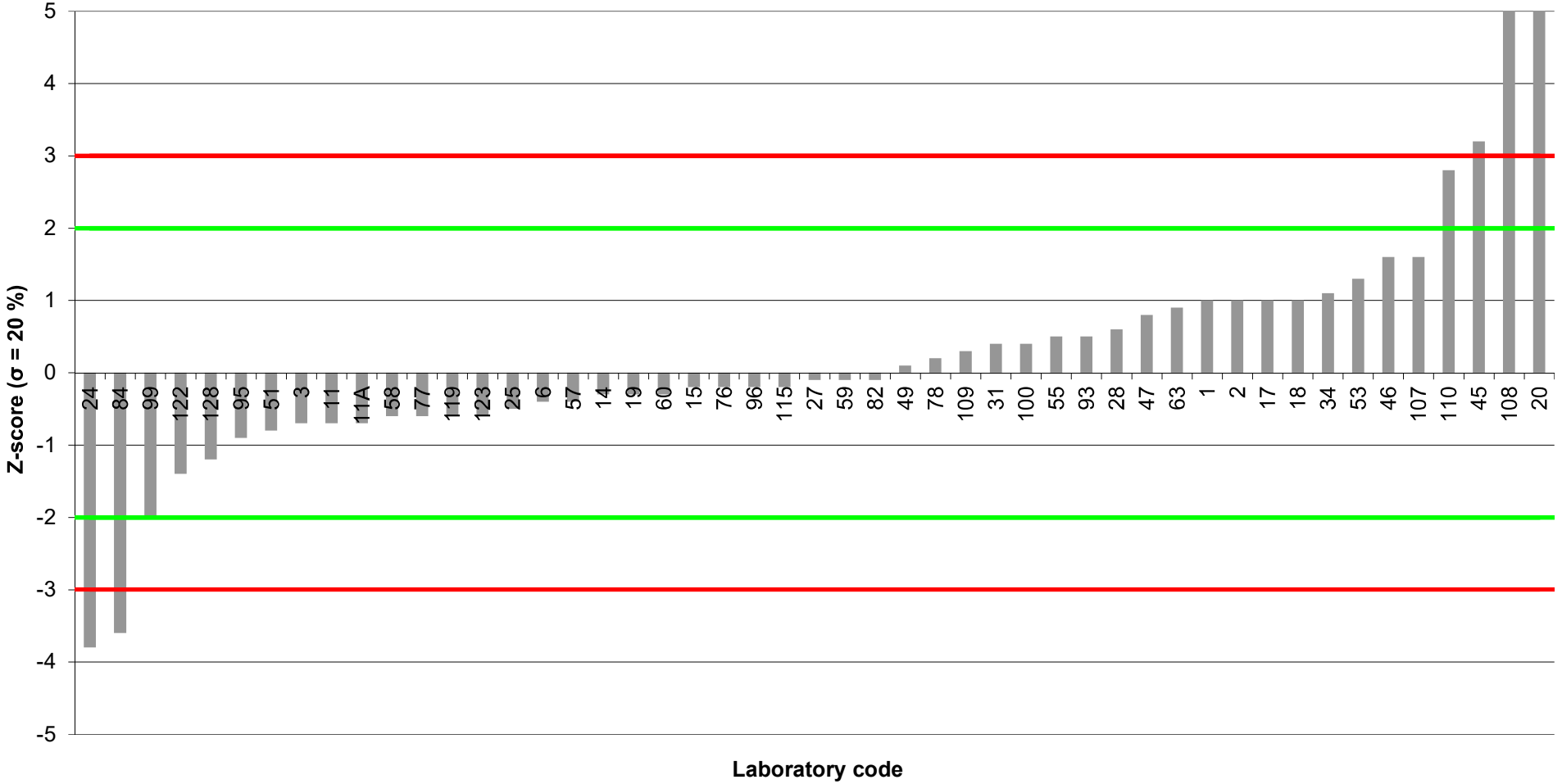
Assigned value: 0.727 µg/kg wet weight



Milk Powder (2301-MP)

Perfluorooctanoic acid

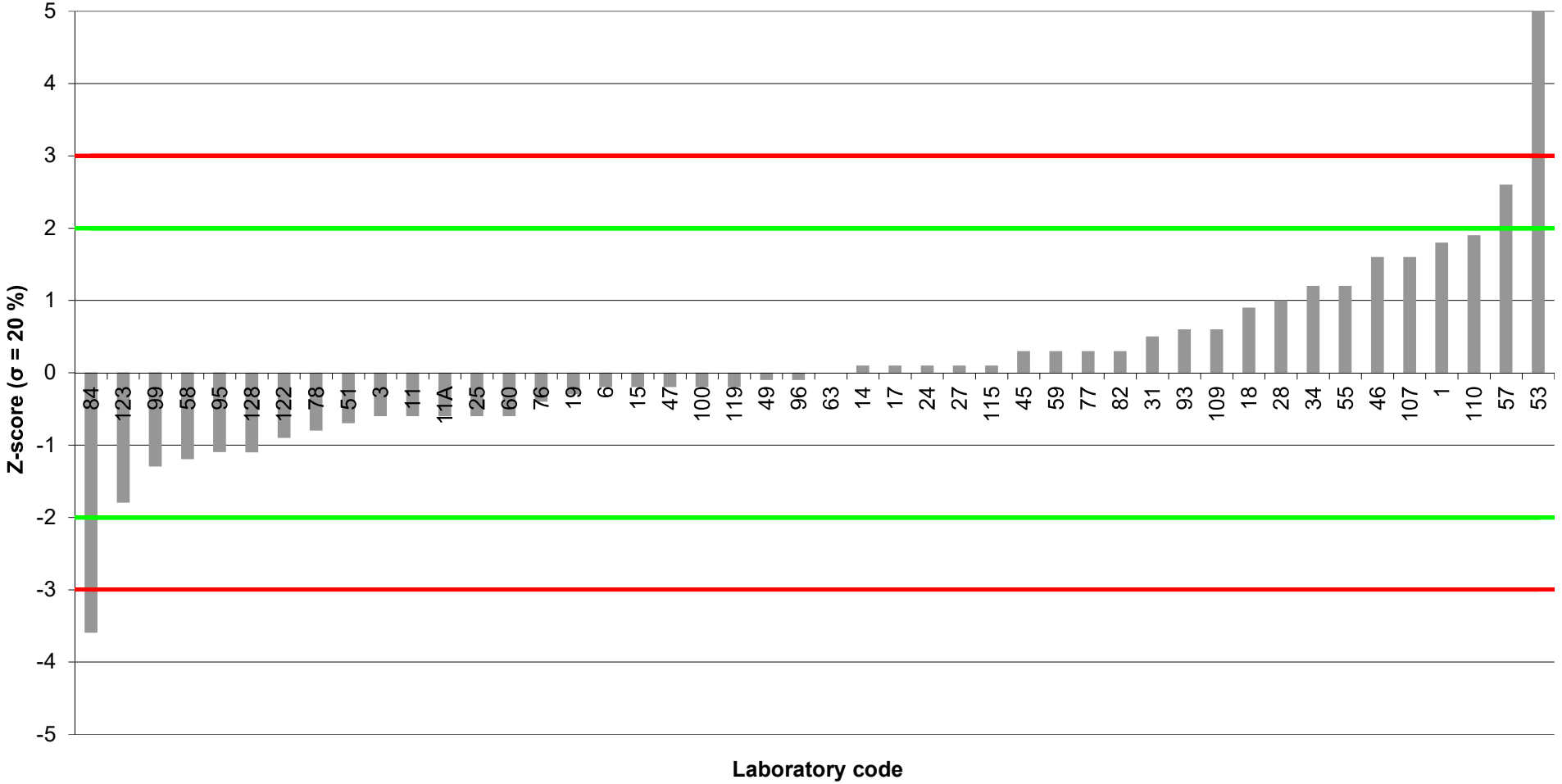
Assigned value: 0.501 µg/kg wet weight



Milk Powder (2301-MP)

Perfluorononanoic acid

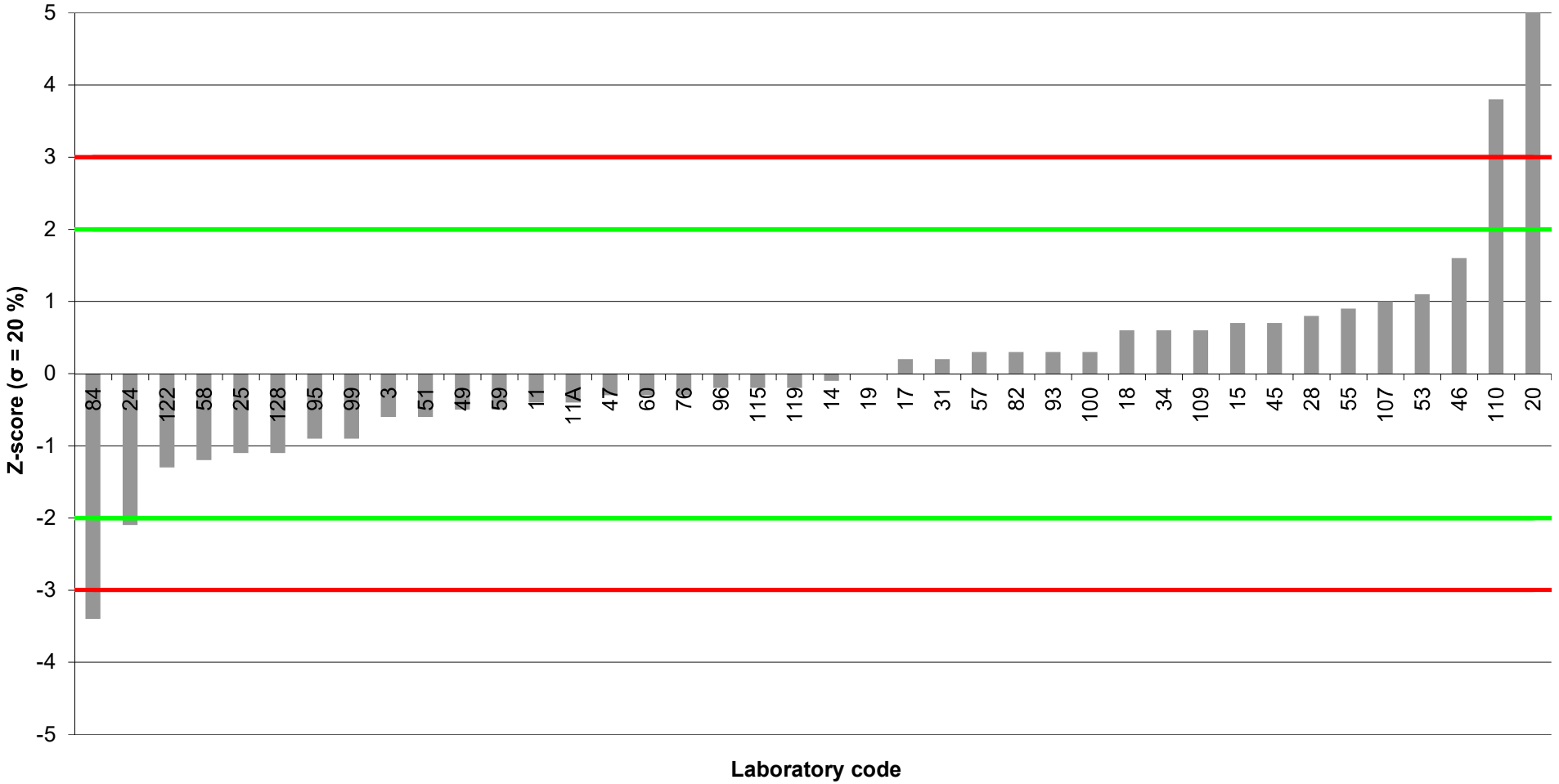
Assigned value: 0.217 µg/kg wet weight



Milk Powder (2301-MP)

Perfluorodecanoic acid

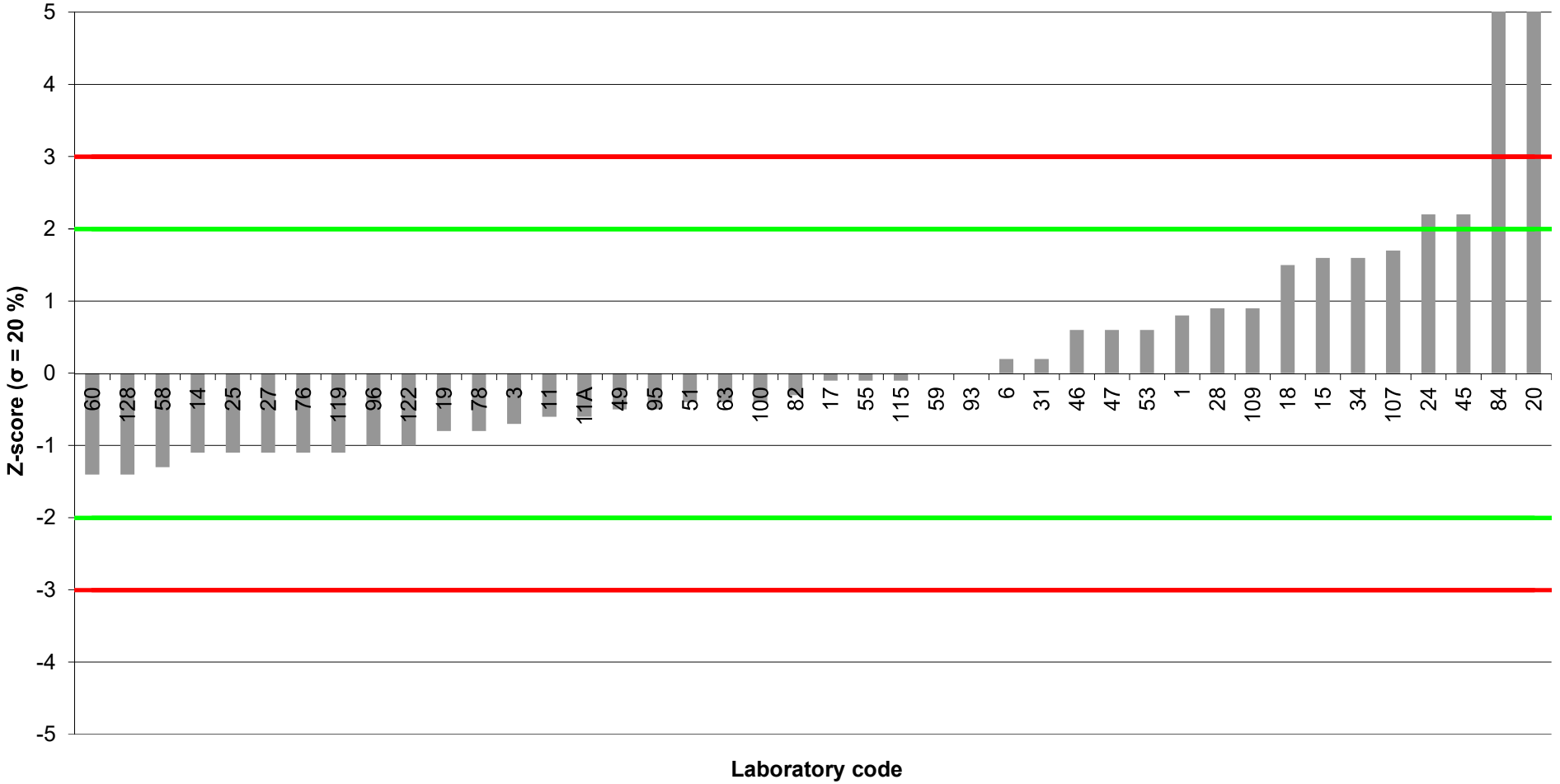
Assigned value: 0.387 µg/kg wet weight



Milk Powder (2301-MP)

Perfluorohexanesulfonic acid

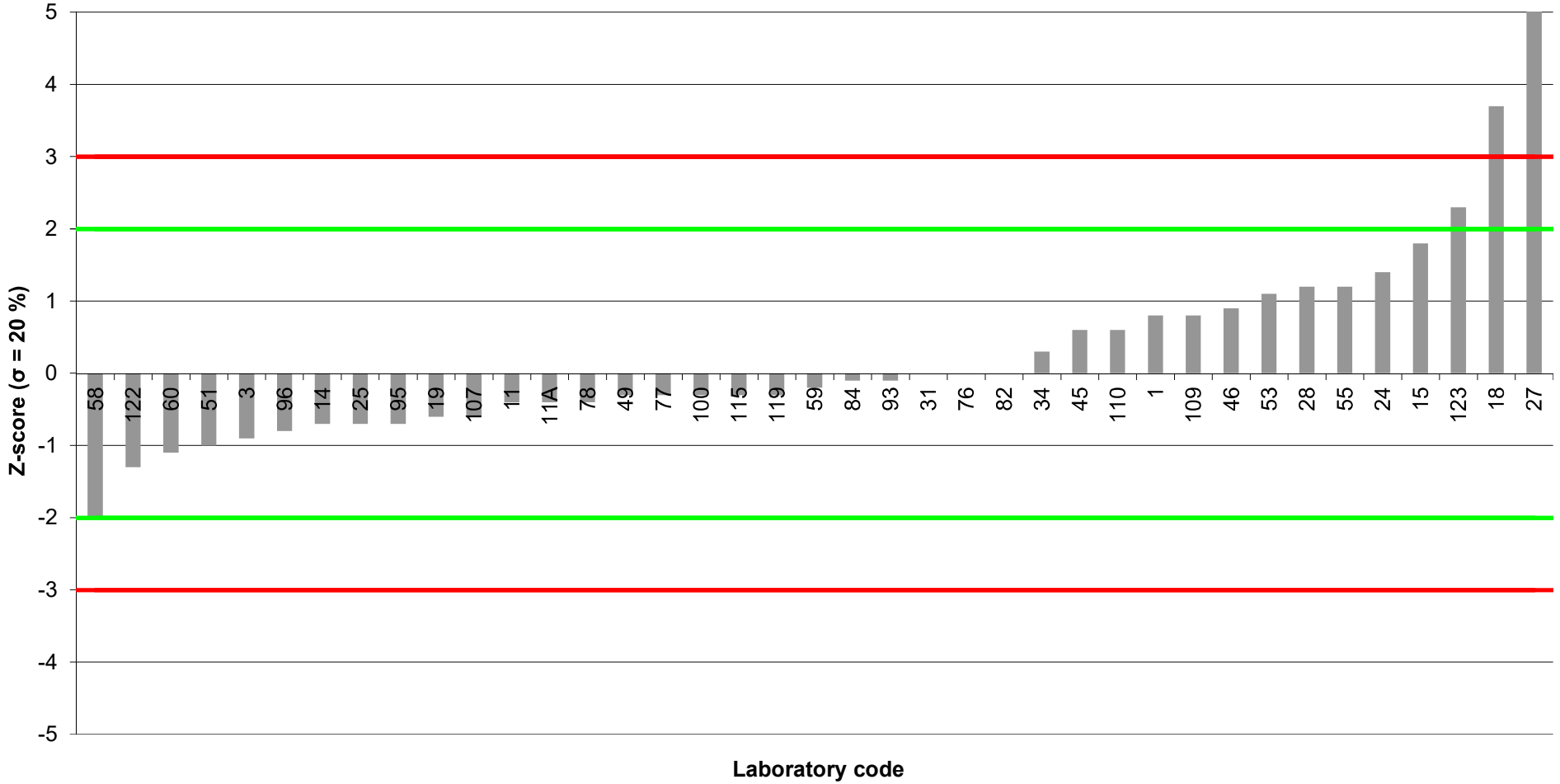
Assigned value: 0.0762 µg/kg wet weight



Milk Powder (2301-MP)

Linear Perfluorooctanesulfonic acid

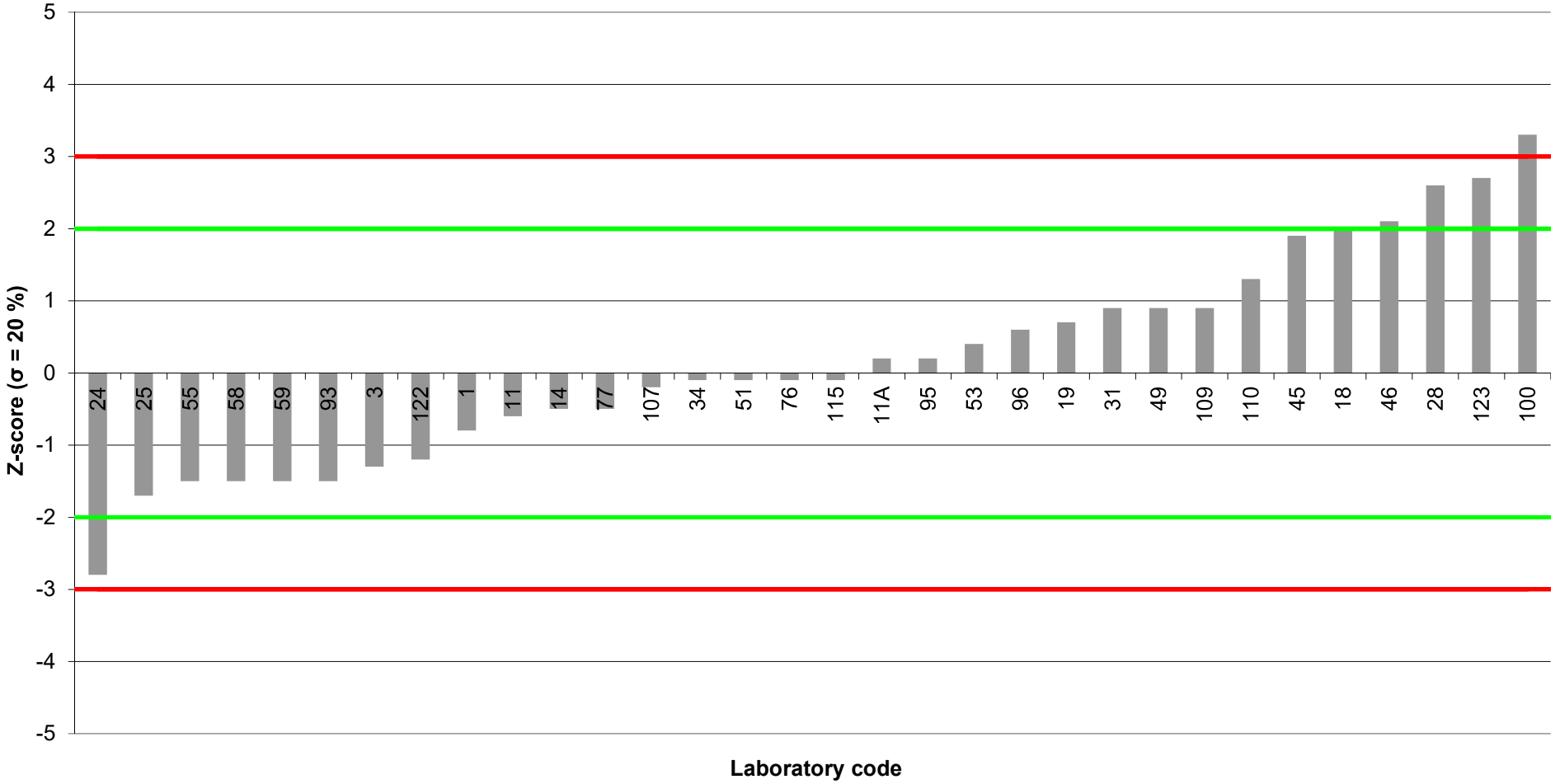
Assigned value: 0.161 µg/kg wet weight



Milk Powder (2301-MP)

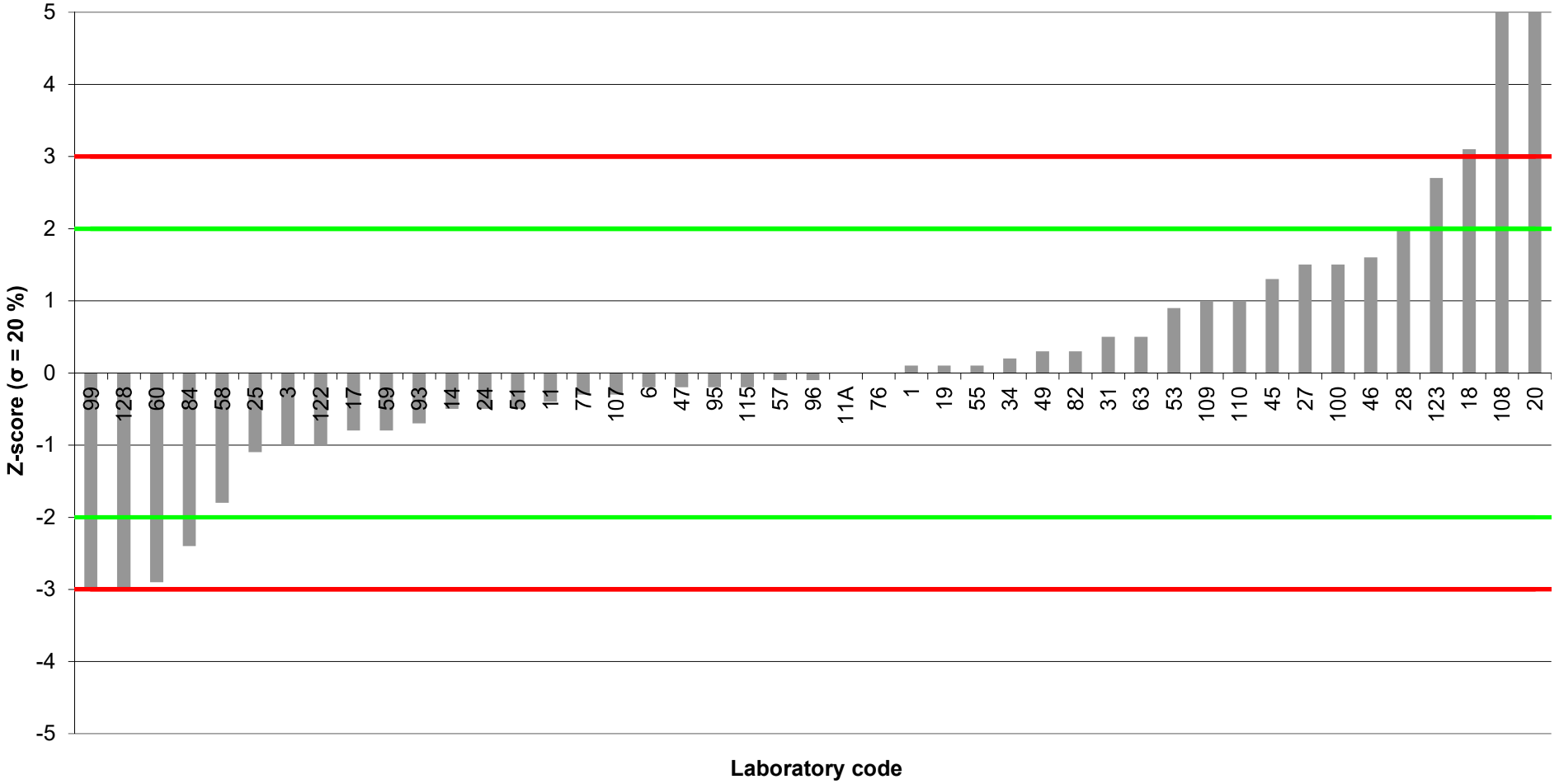
Sum of branched Perfluorooctanesulfonic acid

Assigned value: 0.144 µg/kg wet weight



Milk Powder (2301-MP)
Sum of branched and linear Perfluorooctanesulfonic acid

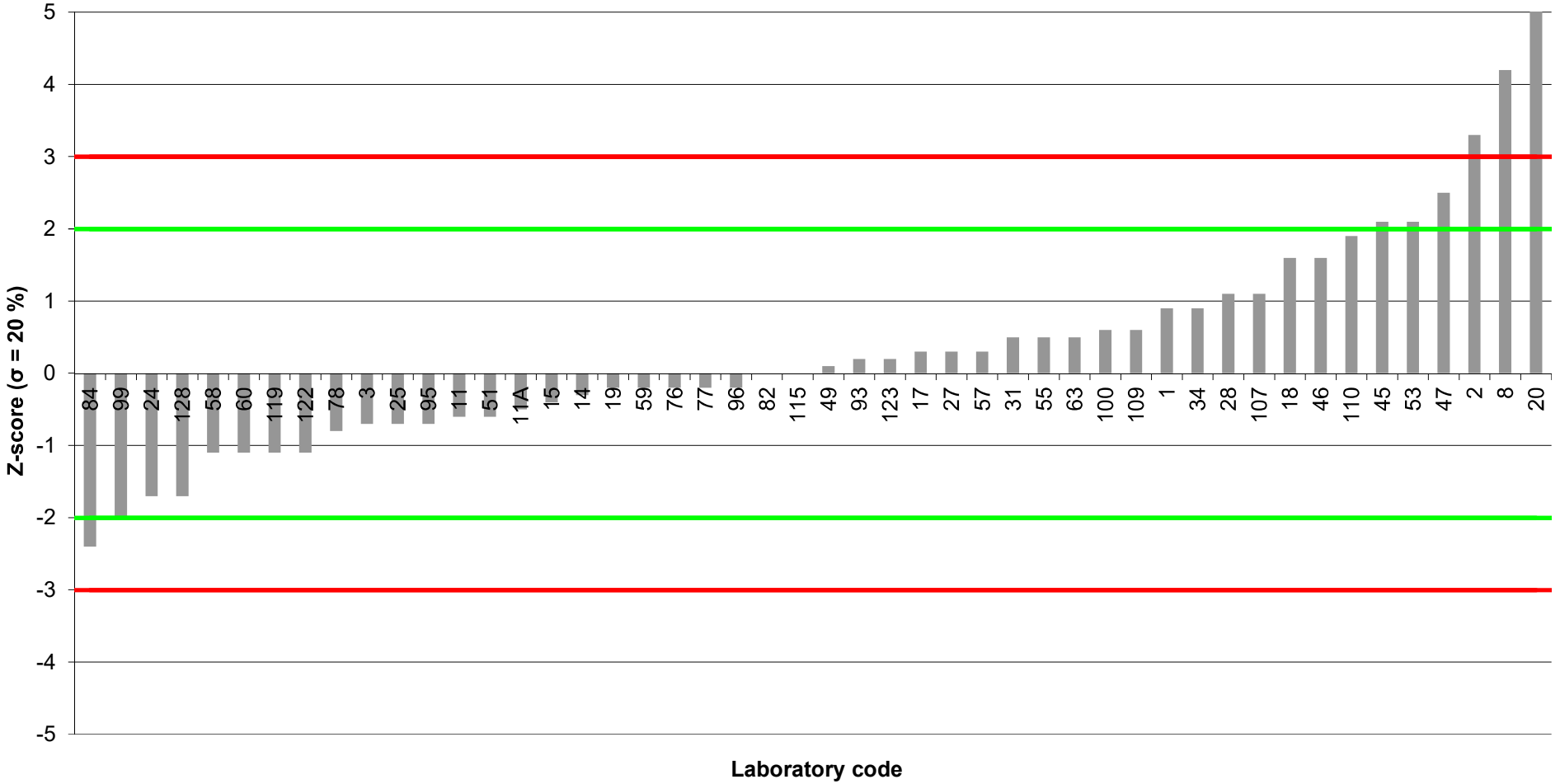
Assigned value: 0.300 µg/kg wet weight



Milk Powder (2301-MP)

Sum of total PFOS, PFOA, PFNA, PFHxS (ub)

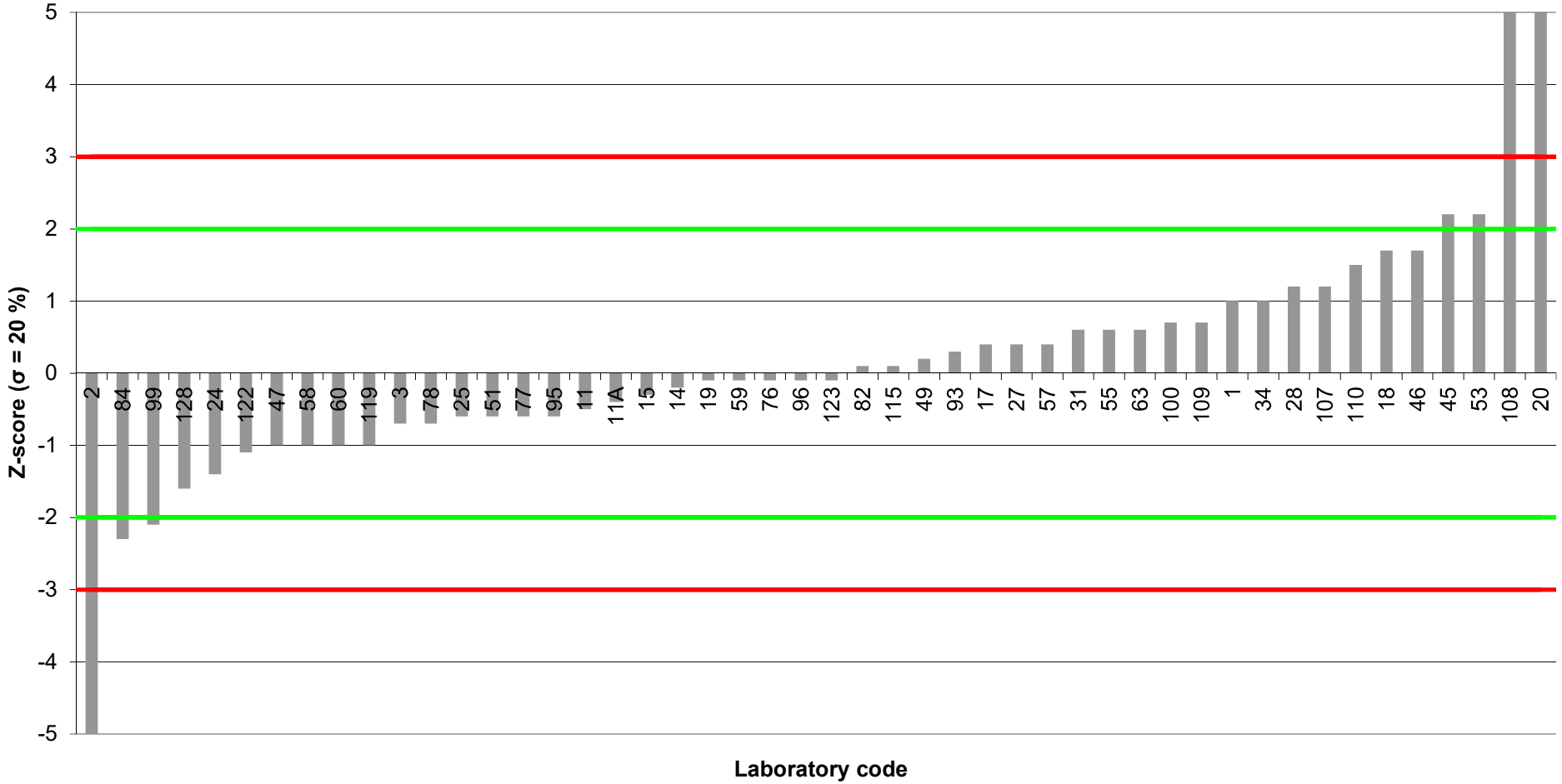
Assigned value: 1.09 µg/kg wet weight



Milk Powder (2301-MP)

Sum of total PFOS, PFOA, PFNA, PFHxS (lb)

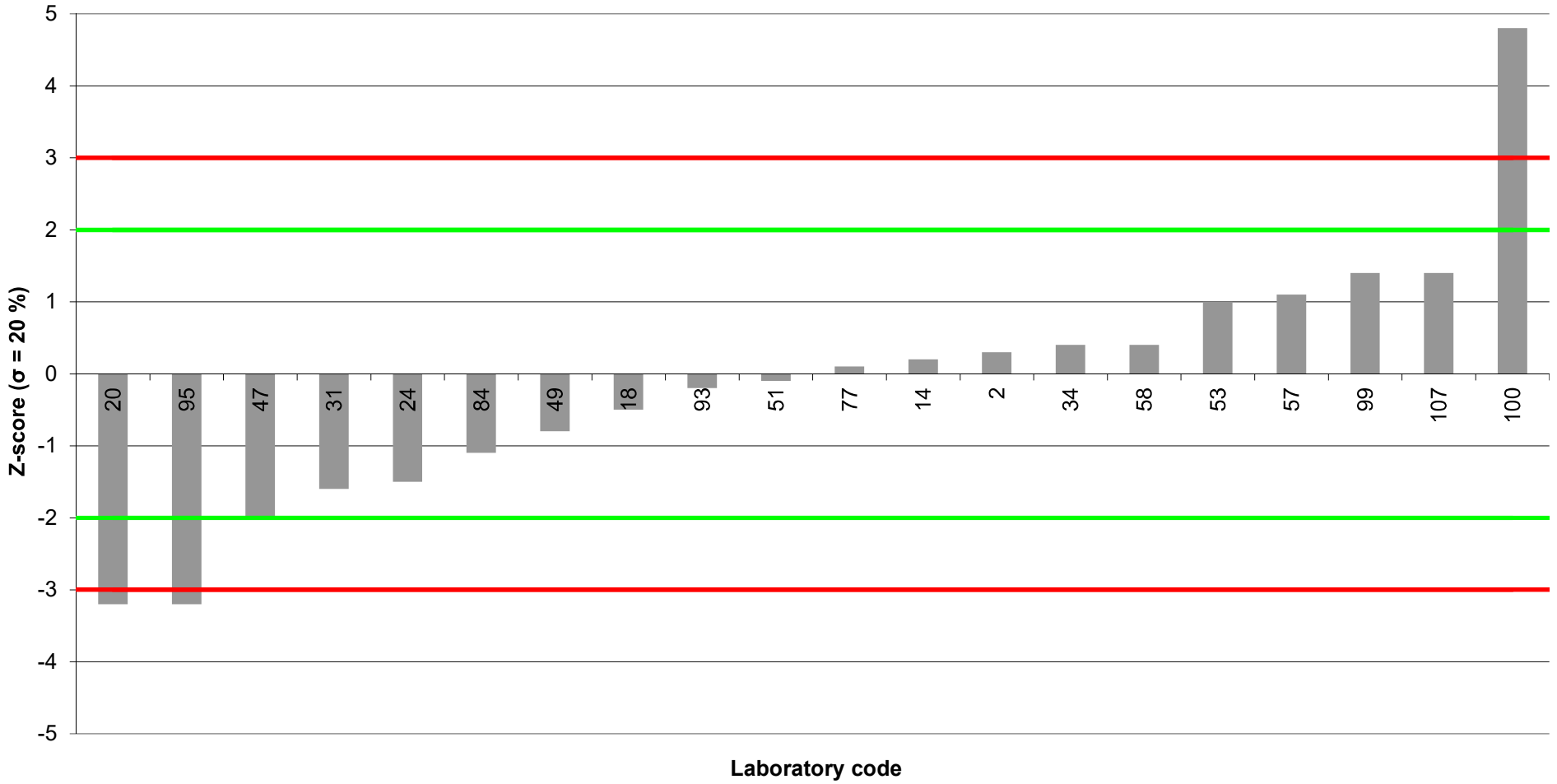
Assigned value: 1.07 µg/kg wet weight



Milk Powder (2301-MP)

DONA

Assigned value: 0.407 µg/kg wet weight



EURL Proficiency Test on Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Milk Powder 2023 [EURL-PT-POP_2301-MP]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food
23 February 2024

Annex 5: Test for sufficient homogeneity and stability for PFCAs, PFSAs

Test sample - Milk Powder (2301-MP)

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder 2023 [EURL-PT-POP_2301-MP]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Powder (2301-MP)
PFCAs, PFSAs - Homogeneity test - Data

Analyte	Result µg/kg wet weight	**Mean** (n = 10, duplicate analysis)	**Median** (n = 10, duplicate analysis)	**Relative standard deviation** [%]
Perfluoropentanoic acid (PFPeA)		0.906	0.887	6%
Perfluorohexanesulfonic acid (PFHxS)		0.0773	0.0773	1%
Perfluorooctanoic acid (PFOA)		0.540	0.545	4%
Sum of branched Perfluorooctanesulfonic acid (br-PFOS)		0.133	0.135	1%
Linear Perfluorooctanesulfonic acid (L-PFOS)		0.153	0.153	2%
Sum of branched and linear Perfluorooctanesulfonic acid (total PFOS)		0.285	0.288	2%
Perfluorononanoic acid (PFNA)		0.231	0.228	2%
Perfluorodecanoic acid (PFDA)		0.422	0.427	3%

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder 2023 [EURL-PT-POP_2301-MP]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Powder (2301-MP)
Selected compounds - Homogeneity test - Data

Sample	Replicate	Result µg/kg wet weight	PFOA	total-PFOS	PFHxS
13	1		0.570	0.186	0.091
	2		0.498	0.136	0.066
22	1		0.530	0.143	0.080
	2		0.494	0.140	0.075
77	1		0.575	0.172	0.085
	2		0.562	0.160	0.086
102	1		0.539	0.142	0.067
	2		0.592	0.179	0.091
128	1		0.551	0.159	0.075
	2		0.595	0.166	0.084
141	1		0.609	0.160	0.083
	2		0.502	0.140	0.068
177	1		0.505	0.130	0.068
	2		0.493	0.127	0.070
179	1		0.486	0.136	0.070
	2		0.559	0.158	0.083
180	1		0.584	0.177	0.087
	2		0.504	0.149	0.066
230	1		0.564	0.157	0.080
	2		0.499	0.136	0.071
Cochran's C-test					
C			0.295	0.409	0.279
$C_{critical}$ (α = 0.05, m = 2, n = 10)			0.602	0.602	0.602
$C_{critical}$ (α = 0.01, m = 2, n = 10)			0.718	0.718	0.718
$C < C_{critical}$			yes	yes	yes
Outliers			no evidence for analytical outliers	no evidence for analytical outliers	no evidence for analytical outliers
Homogeneity test					
General average $\bar{x}$			0.540	0.153	0.0773
Standard deviation of sample averages s_x			0.025	0.012	0.0041
Wthin-sample standard deviation s_w			0.044	0.018	0.0105
Between-sample standard deviation s_s			0.000	0.000	0.000
Standard deviation for proficiency assessment σ_{PT}			0.11	0.03	0.015
s_s / σ_{PT}			0.000	0.000	0.000
Test for homogeneity ($s_s \leq 0.3\ \sigma_{PT}$)			passed	passed	passed

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder 2023 [EURL-PT-POP_2301-MP]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Powder (2301-MP)
Selected compounds - Stability test - Data

Sample	Replicate	Result µg/kg wet weight	**PFHxS**
18	1		0.083
	2		0.076
110	1		0.078
	2		0.080
202	1		0.083
	2		0.062
Stability test			
General average (stability test) $\bar{y}$			0.08
General average (homogeneity test) $\bar{x}$			0.08
Standard deviation for proficiency assessment σPT			0.015
$\lvert \bar{y} - \bar{x} \rvert$			0.0005
Test for stability ($\lvert \bar{y} - \bar{x} \rvert \leq 0.3$ σPT)			passed

EURL Proficiency Test on Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Milk Powder 2023 [EURL-PT-POP_2301-MP]

EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

23 February 2024

Annex 6: Participants' methods for PFASs - Weighed sample, internal and recovery standards and comments

Test sample - Milk Powder (2301-MP)

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder 2023 [EURL-PT-POP_2301-MP]

EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Power (2301-MP)

Methods Perfluoroalkylcarboxylic acids (PFCAs), Perfluoroalkylsulfonic acids (PFSAs) and Other PFAS - Weighed sample and internal standards

LC	Sample	Weighed sample [g]	Use of isotope-labelled internal standards for … PFCAs (yes/no)	PFSAs (yes/no)	FOSA (yes/no)	Other internal standards	Use of recovery / injection standard(s) (yes/no)	Matrix calibration (yes/no)
1	2301-MP	5	yes	yes	no			yes
2	2301-MP	1	Yes	Yes	Yes		Yes	No
3	2301-MP	5	yes : MPFAC-C-ES from Wellington Laboratories	yes : MPFAC-C-ES from Wellington Laboratories	M3HFPO-DA from Wellington Laboratories		yes : MPFAC-C-IS from Wellington Laboratories	no
6	2301-MP	5	yes	yes	yes		yes	no
8	2301-MP	1	Yes	Yes	Yes		No	No
11	2301-MP	1	yes	yes	no	no	yes	no
11A	2301-MP	1	yes	yes	no	no	yes	no
14	2301-MP	0.5	yes	yes	yes		no	no
15	2301-MP	5g	yes	yes	yes	M5PFHxA (Perfluoro-n-(1,2,3,4,6-13C5) Hexanoic acid), M3PFHxS (Sodium perfluor-1-(1,2,3-13C3)Hexane sulfonate) - M8PFOA (perfluoro-n-(13C8) octanoic acid), M8PFOS (Sodium perfluoro-1-(1,2,3,4-13C4) octanesulfonate), MPFDoDA (Perfluoro-n-(1,2-13C2) dodecanoic acid)	no	no
17	2301-MP	2.5	yes	yes	no		yes	no
18	2301-MP	0.5	yes	yes			no	no
19	2301-MP	1.5	yes	yes			yes	no
20	2301-MP	1	no	no	yes		yes	yes
24	2301-MP	5g.	Yes	Yes	Yes		Yes	Yes
25	2301-MP	5	yes	yes	yes		no	no
27	2301-MP		yes (13C labeled PFOA and PFNA)	yes (13C labeled PFOS and PFHxS)	no	no	no	no
28	2301-MP	1	yes	yes	no		yes	no
31	2301-MP	1.3	Y -MPFAC-C-ES - wellington	Y -MPFAC-C-ES wellington		MPFAC-C-ES - wellington	Y- MPFAc-C-IS wellington	no
34	2301-MP	5	Yes	Yes	Yes		No	No
45	2301-MP	10	yes	yes	yes		no	no
46	2301-MP	2	yes	yes			yes	no
47	2301-MP	5	yes	yes	no		no	no
49	2301-MP	1	yes	yes (except PFDoDS)	yes (except Capstone A)		no	no (except PFDoDS and Capstone A)
51	2301-MP	5g	yes	yes	yes		yes	no
53	2301-MP	2	yes	yes	yes		no	no
55	2301-MP	1.34	Yes	Yes			Yes	Yes
57	2301-MP	1	yes	yes	no	-	yes	no
58	2301-MP	2	yes	yes	yes		yes	no
59	2301-MP	2g	Yes	Yes	Yes		No	No
60	2301-MP	2g	yes	yes	no		no	no
63	2301-MP	1 g	yes	yes			yes	no
76	2301-MP	0.5	Yes	Yes	Yes		No	No
77	2301-MP	1	yes	yes			yes	no
78	2301-MP	1g	yes	yes	no		no	no
82	2301-MP	1	yes	yes	yes		no	no
84	2301-MP	2	yes	yes	yes	Fluoerotelomer sulfonic acids (4:2, 6:2 and 8:2)	Yes	No
93	2301-MP	2	yes	yes	yes		no	no
95	2301-MP	1	yes	yes	yes and no		no	no
96	2301-MP	1	YES	YES			YES	NO
99	2301-MP	2	yes	yes	no		no	no
100	2301-MP		yes	yes	no	no	no	no
107	2301-MP	2	yes	yes	yes	M2-6FTS, M2-6FTS, M2-8FTS, d3-N-MeFOSAA, d5-N-EtFOSAA, M8FOSA	yes	yes
108	2301-MP	2.5	Yes	Yes	Yes		No	No
109	2301-MP	1	yes	yes	yes		no	yes
110	2301-MP	2.0	yes	yes	no		yes	no
115	2301-MP	0,5-1	YES	YES	YES		YES	NO
119	2301-MP	5.0	yes	yes	yes		yes	no
122	2301-MP	1	yes	yes	no		no	no
123	2301-MP	2	yes	yes			injection standard	no
128	2301-MP	5	yes	yes	no	none	yes	no
60*	2301-MP	2g	yes	yes	no		no	no

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EU-RL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Power (2301-MP)
Methods Perfluoroalkylcarboxylic acids (PFCAs) - Internal Standards

LC	Sample	Perfluorobutanoic acid **(PFBA)**	Perfluoropentanoic acid **(PFPeA)**	Perfluorohexanoic acid **(PFHxA)**	Perfluoroheptanoic acid **(PFHpA)**	Perfluorooctanoic acid **(PFOA)**	Perfluorononanoic acid **(PFNA)**	Perfluorodecanoic acid **(PFDA)**	Perfluoroundecanoic acid **(PFUnDA)**	Perfluorododecanoic acid **(PFDoDA)**	Perfluorotridecanoic acid **(PFTrDA)**	Perfluorotetradecanoic acid **(PFTeDA)**
1	2301-MP					ILIS PFOA	ILIS PFNA					
2	2301-MP	13C4 PFBA	13C5 PFPeA	13C5 PFHxA	13C4 PFHpA	13C8 PFOA	13C9 PFNA	13C6 PFDA	13C7 PFUnDA	13C2 PFDoDA	13C2 PFTeDA	13C2 PFTeDA
3	2301-MP		MPFPeA	MPFHxA	MPFHpA	MPFOA	MPFNA	MPFDA	MPFUnDA	MPFDoDA	MPFDoDA/MPFTeDA	MPFTeDA
6	2301-MP					MPFOA	MPFNA					
8	2301-MP	M4PFBA	M5PFPeA	M5PFHxA	MPFHpA	M8PFOA	M9PFNA	M6PFDA	M7PFUnDA	MPFDoDA	MPFDoDA	MPFTeDA
11	2301-MP		PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUnDA	PFDoDA	PFDoDA	PFTeDA
11A	2301-MP		PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUnDA	PFDoDA	PFDoDA	PFTeDA
14	2301-MP	PFBA-13C4	PFPeA-13C5	PFHxA-13C5	PFHpA-13C2	PFOA-13C8	PFNA-13C9	PFDA-13C6	PFUnA-13C7	PFDoA-13C2	PFTeDA-13C2+PFDoA-13C2	PFTeDA-13C2
15	2301-MP	M5PFHxA (Perfluoro-n-(1,2,3,4,6-13C5) Hexanoic acid)	M5PFHxA (Perfluoro-n-(1,2,3,4,6-13C5) Hexanoic acid)	M5PFHxA (Perfluoro-n-(1,2,3,4,6-13C5) Hexanoic acid)	M5PFHxA (Perfluoro-n-(1,2,3,4,6-13C5) Hexanoic acid)	M8PFOA (perfluoro-n-(13C8) octanoic acid)	M8PFOA (perfluoro-n-(13C8) octanoic acid)	M8PFOA (perfluoro-n-(13C8) octanoic acid)	M8PFOA (perfluoro-n-(13C8) octanoic acid)	MPFDoDA (Perfluoro-n-(1,2-13C2) dodecanoic acid)	MPFDoDA (Perfluoro-n-(1,2-13C2) dodecanoic acid)	MPFDoDA (Perfluoro-n-(1,2-13C2) dodecanoic acid)
17	2301-MP		PFPeA isotope-labelled	PFHxA isotope-labelled	PFPHpA isotope-labelled	PFOA isotope-labelled	PFNA isotope-labelled	PFDA isotope-labelled	PFUnDA isotope-labelled	PFDoDA isotope-labelled	PFTrDA+PFTeDA isotope-labelled	PFTeDA isotope-labelled
18	2301-MP	PFBA-13C2	PFPeA-13C5	PFHxA-13C2	PFOA-13C4	PFOA-13C4	PFNA-13C5	PFDA-13C2	PFUnDA-13C2	PFDoDA-13C2		
19	2301-MP	13C-PFBA	13C-PFPeA	13C-PFHxA	13C-PFHpA	13C-PFOA	13C-PFNA	13C-PFDA	13C-PFUnDA	13C-PFDoDA		13C-PFTeDA
20	2301-MP											
24	2301-MP					M8PFOA	M8PFOA	M8PFOA			M8PFOA	
25	2301-MP	13C4-PFBA	13C5-PFPeA	13C5-PFHxA	13C4-PFHpA	13C8-PFOA	13C9-PFNA	13C6-PFDA	13C7-PFUnDA	13C2-PFDoDA	13C6-PFDA	13C2-PFTeDA
27	2301-MP					13C-PFOA	13C-PFNA					
28	2301-MP	PFBA13C4	PFPeA13C5	PFHxA13C5	PFHpA13C4	PFOA13C8	PFNA13C9	PFDA13C6	PFUNDA13C7	PFDoDA13C2	PFTeDA13C2	PFTeDA13C2
31	2301-MP		13C-PFPeA	13C-PFHxA	13C-PFHpA	13C-PFOA	13C-PFNA	13C-PFDA	13C-PFUnDA	13C-PFDoDA		13C-PFTeDA
34	2301-MP	13C4-PFBA	13C5-PFPeA	13C2-PFHxA	13C4-PFHpA	13C4-PFOA	13C5-PFNA	13C2-PFDA	13C2-PFUnDA	13C2-PFDoDA	13C2-PFDoDA	13C2-PFTeDA
45	2301-MP	13C4-PFBA	13C5-PFPeA	13C5-PFHxA	13C4-PFHpA	13C8-PFOA	13C9-PFNA	13C6-PFDA	13C7-PFUnDA	13C2-PFDoA	13C2-PFTeA	13C2-PFTeA
46	2301-MP			Perfluoro-n-[1,2,3,4,6-13C12] hexanoic acid	Perfluoro-n-[1,2,3,4-13C12] heptanoic acid	Perfluoro-n-[1,2,3,4-13C12] octanoic acid	Perfluoro-n-[1,2,3,4,5-13C12] nonanoic acid	Perfluoro-n-[1,2-13C12] decanoic acid	Perfluoro-n-[1,2,3,4,5,6,7-113C12]undecanoic acid	Perfluoro-n-[1,2-13C12] dodecanoic acid		
47	2301-MP		Perfluoro-n-(1,2-13C2)hexanoic acid	Perfluoro-n-(1,2-13C2)hexanoic acid	Perfluoro-n-(1,2,3,4-13C4)octanoic acid	Perfluoro-n-(1,2,3,4-13C4)octanoic acid	Perfluoro-n-(1,2,3,4,5-13C5)nonanoic acid	Perfluoro-n-(1,2-13C2)decanoic acid	Perfluoro-n-(1,2-13C2)undecanoic acid	Perfluoro-n-(1,2-13C2)dodecanoic acid	Perfluoro-n-(1,2-13C2)dodecanoic acid	Perfluoro-n-(1,2-13C2)dodecanoic acid
49	2301-MP	MPFBA	MPFPeA	MPFHxA	MPFOS	MPFOA	MPFNA	MPFDA	MPFUnDA	MPFDoDA	MPFDoDA	MPFTeDA
51	2301-MP	13C4-PFBA	13C5-PFPeA	13C2-PFHxA	13C4-PFHpA	13C8-PFOA	13C5-PFNA	13C2-PFDA	13C2-PFUnA	13C2-PFDoA	13C2-PFDoA	13C2-PFTeDA
53	2301-MP	MPFBA	MPFPeA	MPFHxA	MPFHpA	MPFOA	MPFNA	MPFDA	MPFUnDa	MPFDoDA	MPFTeDA	MPFTeDA
55	2301-MP	13C-PFBA	13C-PFPA	13C-PFHxA	13C-PFHpA	13C-PFOA	13C-PFNA	13C-PFDA	13C-PFUnDA	13C-PFDoDA	13C-PFDoDA	13C-PFTeDA
57	2301-MP	PFBA-13C4	PFPeA-13C3	PFHxA-13C2	PFHpA-13C4	PFOA-13C4	PFNA-13C5	PFDA-13C2	PFDoDA-13C2	PFDoDA-13C2	PFDoDA-13C2	PFTeDA-13C2
58	2301-MP	13C4-PFBA	13C5-PFPA	13C5-PFHxA	13C4-PFHpA	13C8-PFOA	13C9-PFNA	13C6-PFDA	13C7-PFUnDA	13C2-PFDoDA	13C2-PFDoDA	13C2-PFTeDA
59	2301-MP	13C4-PFBA	13C5-PFPeA	13C5-PFHxA	13C4-PFHpA	13C4-PFOA	13C5-PFNA	13C6-PFDA	13C7-PFUdA		13C2-PFDoA	13C2-PFTeDA
60	2301-MP	Perfluoro-n-(13C4)butanoic acid	Perfluoro-n-[3,4,5-13C3]pentanoic acid	Perfluoro-n-[1,2-13C2]hexanoic acid	Perfluoro-n-[1,2-13C2]hexanoic acid	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	Perfluoro-n-[1,2,3,4,5-13C5]nonanoic acid	Perfluoro-n-[1,2-13C2]decanoic acid	Perfluoro-n-[1,2-13C2]undecanoic acid	Perfluoro-n-[1,2-13C2]dodecanoic acid	Sodium perfluoro-1-(1,2,3,4-13C4)octanesulfonate	Sodium perfluoro-1-(1,2,3,4-13C4)octanesulfonate
63	2301-MP					13C4 - PFOA	13C9 - PFNA					
76	2301-MP		Perfluoro-n-[13C5]pentanoic acid (M5PFPeA)	Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid (M5PFHxA)	Perfluoro-n-[1,2,3,4-13C4]heptanoic acid (M4PFHpA)	Perfluoro-n-[13C8]octanoic acid (M8PFOA)	Perfluoro-n-[13C9]nonanoic acid (M9PFNA)	Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid (M6PFDA)	Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid (M7PFUdA)	Perfluoro-n-[1,2-13C2]dodecanoic acid (MPFDoA)	Perfluoro-n-[1,2-13C2]dodecanoic acid (MPFDoA)	Perfluoro-n-[1,2-13C2]tetradecanoic acid M2PFTeDA
77	2301-MP					13C8-PFOS	13C9-PFNA					
78	2301-MP					13C PFOA	13 PFNA					
82	2301-MP	13C4-PFBA	13C5-PPeA	13C5-PFHxA	13C4-PFHpA	13C8-PFOA	13C9-PFNA	13C6-PFDA	13C7-PFUnDA	13C2-PFDoDA	13C2-PFDoDA	13C2-PFTeDA
84	2301-MP	MPFBA	M5PFPeA	M5PFHxA	M4PFHpA	M8PFOA	M9PFNA	M6PFDA	M7PFUnDA	MPFDoA	M2PFTeDA	M2PFTeDA
93	2301-MP	MPFBA	MPFPeA	MPFHxA	MPFOA	MPFOA	MPFNA	MPFDA	MPFUnDA	MPFDoDA	MPFDoDA	MPFDoDA
95	2301-MP	Perfluoro-n-(1,2,3,4-13C4)butansäure (MPFBA)	Perfluoro-n-(13C5)pentansäure (M5PFPeA)	Perfluoro-n-(1,2,3,4,6-13C5)hexansäure (M5PFHxA)	Perfluoro-n-[1,2,3,4-13C4]heptansäure (M4PFHpA)	Perfluoro-n-(13C8)octansäure (M8PFOA)	Perfluoro-n-(13C9)nonansäure (M9PFNA)	Perfluoro-n-(1,2,3,4,5,6-13C6)decansäure (M6PFDA)	Perfluoro-n-(1,2,3,4,5,6,7-13C7)undecansäure (M7PFUdA)	Perfluoro-n-(1,2-13C2)dodecansäure (MPFDoA)	external calibration	Perfluoro-n-(13C2)tetradecansäure (M2PFTeDA)
96	2301-MP	13C PFBA	13C PFPA	13C PFHxA	13C PFHpA	13C PFOA	13C PFNA	13C PFDA	13C PFUnDA	13C PFDoDA	13C PFDoDA	13C PFDoDA
99	2301-MP	Perfluoro-n-(13C4)butanoic acid	Perfluoro-n-(13C5)pentanoic acid	Perfluoro-n-(1,2,3,4,6-13C5)hexanoic acid	Perfluoro-n-(1,2,3,4-13C4)heptanoic acid	Perfluoro-n-(13C8)octanoic acid	Perfluoro-n-(13C9)nonanoic acid	Perfluoro-n-(1,2,3,4,5,6-13C6)decanoic acid	Perfluoro-n-(1,2,3,4,5,6,7-13C7)undecanoic acid	Perfluoro-n-(1,2-13C2)dodecanoic acid	Perfluoro-n-(1,2-13C2)tetradecanoic acid	
100	2301-MP	MPFBA	MPFBA	MPFHxA	MPFHxA	MPFOA	MPFNA	MPFDA			MPFDoDA	MPFDoDA
107	2301-MP	M3PFBS.IS	M5PFPeA.IS	M5PFPHxA.IS	M4PFHpA.IS	M8PFOA.IS	M9PFNA.IS	M6PFDA.IS	M7PFUdA.IS	MPFDo .IS	M8PFOS.IS	M2PFTeDA.IS
108	2301-MP	Isotope labeled analogue	Isotope labeled analogue	Isotope labeled analogue	Isotope labeled analogue	Isotope labeled analogue	Isotope labeled analogue	Isotope labeled analogue	Isotope labeled analogue	Isotope labeled analogue	isotope labeled PFDoDA	Isotope labeled analogue
109	2301-MP	13C4-PFBA	13C5-PFPeA	13C5-PFHxA	13C4-PFHpA	13C8-PFOA	13C9-PFNA	13C6-PFDA	13C7-PFUdA	13C2-PFDoA	13C2-PFTeDA	13C2-PFTeDA
110	2301-MP	13C4_PFBA	13C5_PFPeA	13C5_PFHxA	13C4_PFHpA	13C8_PFOA	13C9-PFNA	13C6-PFDA	13C7-PFUdA	13C2_PFDoA	13C2_PFTeDA	13C2_PFTeDA
115	2301-MP	13C4-PFBA	13C5-PFPeA	13C5-PFHxA	13C4-PFHpA	13C8-PFOA	13C9-PFNA	13C6-PFDA	13C7-PFUnDA	13C2-PFDoDA	13C2-PFDoDA	13C2-PFTeDA
119	2301-MP	perfluoro-n-[13C4] perfluoro-n-[13C4] butanoic acid	perfluoro-n-[13C5] pentanoic acid	perfluoro-n-[1,2,3,4,6-13C5] hexanoic acid	perfluoro-n-[1,2,3,4, -13C4] heptanoic acid	perfluoro-n-[13C8]octanoic acid	nonanoic acid (M9PFNA)	perfluoro-n-[1,2,3,4,5,6-13C6] decanoic acid	perfluoro-n-[1,2,3,4,5,6,7-13C7] undecanoic acid	perfluoro-n-[1,2 -13C2] dodecanoic acid (MPFDoA)		
122	2301-MP	M4PFBA	M5PFPeA	MPFHxA	M4PFHpA	M4PFOA	M5PFNA	M6PFDA	M7PFUnDA	M2PFDoDA	M2PFDoDA	M2PFTeDA
123	2301-MP	PFBA-13C3	PFPeA-13C5	PFHxA-13C5	PFHpA--13C4	PFOA-13C8	PFNA-13C9	PFDA-13C6	PFUnDA-13C7	PFDoDA-13C2	PFTrDA-13C2	PFTeDA-13C2
128	2301-MP	13C4-PFBA	13C5-PFPeA	13C5-PFHxA	13C4-PFHpA	13C8-PFOA	13C9-PFNA	13C6-PFDA	13C7-PFUnDA	13C2-PFDoDA	13C2-PFDoDA	13C2-PFTeDA
60*	2301-MP	Perfluoro-n-(13C4)butanoic acid	Perfluoro-n-[3,4,5-13C3]pentanoic acid	Perfluoro-n-[1,2-13C2]hexanoic acid	Perfluoro-n-[1,2-13C2]hexanoic acid	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	Perfluoro-n-[1,2,3,4,5-13C5]nonanoic acid	Perfluoro-n-[1,2-13C2]decanoic acid	Perfluoro-n-[1,2-13C2]undecanoic acid	Perfluoro-n-[1,2-13C2]dodecanoic acid	Sodium perfluoro-1-(1,2,3,4-13C4)octanesulfonate	Sodium perfluoro-1-(1,2,3,4-13C4)octanesulfonate

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder 2023 [EURL-PT-POP_2301-MP]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Power (2301-MP)
Methods Perfluoroalkylcarboxylic acids (PFCAs) - Recovery Standards

LC	Sample		Perfluorobutanoic acid **(PFBA)**	Perfluoropentanoic acid **(PFPeA)**	Perfluorohexanoic acid **(PFHxA)**	Perfluoroheptanoic acid **(PFHpA)**	Perfluorooctanoic acid **(PFOA)**	Perfluorononanoic acid **(PFNA)**	Perfluorodecanoic acid **(PFDA)**	Perfluoroundecanoic acid **(PFUnDA)**	Perfluorododecanoic acid **(PFDoDA)**	Perfluorotridecanoic acid **(PFTrDA)**	Perfluorotetradecanoic acid **(PFTeDA)**
1	2301-MP												
2	2301-MP		13C3 PFBA	13C3 PFBA	13C2 PFHxA	13C2 PFHxA	13C4 PFOA	13C5 PFNA	13C2 PFDA	13C2 PFDA	13C2 PFDA	13C2 PFDA	13C2 PFDA
3	2301-MP			MPFBA injection	MPFBA injection	MPFBA injection	MPFOA injection	MPFOA injection	MPFDA injection	MPFDA injection	MPFDA injection	MPFDA injection	MPFDA injection
6	2301-MP												
8	2301-MP												
11	2301-MP			PFBA	PFBA	PFOA	PFOA	PFOA	PFDA	PFDA	PFDA	PFDA	PFDA
11A	2301-MP			PFBA	PFBA	PFOA	PFOA	PFOA	PFDA	PFDA	PFDA	PFDA	PFDA
14	2301-MP												
15	2301-MP		-	-	-	-	-	-	-	-	-	-	-
17	2301-MP												
18	2301-MP												
19	2301-MP		R-PFBA	R-PFOA	R-PFOS	R-PFOA	R-PFOA	R-PFOA	R-PFOA	R-PFOA	R-PFOA		R-PFOA
20	2301-MP												
24	2301-MP						d5-N-EtFOSA	d5-N-EtFOSA	d5-N-EtFOSA			d5-N-EtFOSA	
25	2301-MP												
27	2301-MP												
28	2301-MP		PFBA13C3	PFBA13C3	PFBA13C3	PFBA13C3	PFOA13C2	PFOA13C2	PFOA13C2	PFDA13C2	PFDA13C2	PFDA13C2	PFDA13C2
31	2301-MP			M2PFOA	M2PFOA	M2PFOA	M2PFOA	M2PFOA	MPFDA	MPFDA	MPFDA		MPFDA
34	2301-MP												
45	2301-MP												
46	2301-MP				Sodium perfluoro-[13C8]octanesulfonate	Sodium perfluoro-[13C8]octanesulfonate	Sodium perfluoro-[13C8]octanesulfonate	Sodium perfluoro-[13C8]octanesulfonate	Sodium perfluoro-[13C8]octanesulfonate	Sodium perfluoro-[13C8]octanesulfonate	Sodium perfluoro-[13C8]octanesulfonate		
47	2301-MP												
49	2301-MP												
51	2301-MP		13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA
53	2301-MP												
55	2301-MP		PFOA - M8	PFOA - M8	PFOA - M8	PFOA - M8	PFOA - M8	PFOA - M8	PFOA - M8	PFOA - M8	PFOA - M8	PFOA - M8	PFOA - M8
57	2301-MP												
58	2301-MP		13C2-PFHxA	13C2-PFHxA	13C2-PFHxA	13C2-PFHxA	13C5-PFNA	13C5-PFNA	13C5-PFNA	13C5-PFNA	13C5-PFNA	13C5-PFNA	13C5-PFNA
59	2301-MP												
60	2301-MP												
63	2301-MP												
76	2301-MP												
77	2301-MP						13C4-PFOS	13C5-PFNA					
78	2301-MP						none	none					
82	2301-MP		no recovery standard	no recovery standard	no recovery standard	no recovery standard	no recovery standard	no recovery standard	no recovery standard	no recovery standard	no recovery standard	no recovery standard	no recovery standard
84	2301-MP		72.23	87.06	63.10	64.37092439	52.10	51.93	50.90	60.59	57.20	47.10	47.10
93	2301-MP		no	no	no	no	no	no	no	no	no	no	no
95	2301-MP												
96	2301-MP		13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS
99	2301-MP												
100	2301-MP		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.			n.a.	n.a.
107	2301-MP												
108	2301-MP		Not used	Not used	Not used	Not used	Not used	Not used	Not used	Not used	Not used	Not used	Not used
109	2301-MP		No	No	No	No	No	No	No	No	No	No	No
110	2301-MP												
115	2301-MP		13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS
119	2301-MP		116.50	103.50			92.00	99.20	120.00				
122	2301-MP		-	-	-	-	-	-	-	-	-	-	-
123	2301-MP		PFOA-13C2	PFOA-13C2	PFOA-13C2	PFOA-13C2	PFOA-13C2	PFOA-13C2	PFDA-13C2	PFDA-13C2	PFDA-13C2	PFDA-13C2	PFDA-13C2
128	2301-MP		13C3-PFBA	13C3-PFBA	13C2-PFOA	13C2-PFOA	13C2-PFOA	13C2-PFOA	13C2-PFDA	13C2-PFDA	13C2-PFDA	13C2-PFDA	13C2-PFDA
60*	2301-MP												

Milk Power (2301-MP)
Methods Perfluoroalkylcarboxylic acids (PFCAs) - Comments

LC	Sample	Perfluorobutanoic acid **(PFBA)**	Perfluoropentanoic acid **(PFPeA)**	Perfluorohexanoic acid **(PFHxA)**	Perfluoroheptanoic acid **(PFHpA)**	Perfluorooctanoic acid **(PFOA)**	Perfluorononanoic acid **(PFNA)**	Perfluorodecanoic acid **(PFDA)**	Perfluoroundecanoic acid **(PFUnDA)**	Perfluorododecanoic acid **(PFDoDA)**	Perfluorotridecanoic acid **(PFTrDA)**	Perfluorotetradecanoic acid **(PFTeDA)**
1	2301-MP											
2	2301-MP	one MRM used	one MRM used HRMS									
3	2301-MP											
6	2301-MP											
8	2301-MP											
11	2301-MP		Identification and quantification were performed by HRMS									
11A	2301-MP		Identification and quantification were performed by HRMS									
14	2301-MP	not confirmed	not confirmed									
15	2301-MP			Below our LOQ	Below our LOQ				Detected below our LOQ	Detected below our LOQ		
17	2301-MP		one MS/MS transition									
18	2301-MP											
19	2301-MP	content could not be confirmed by HILIC chromatography (0,249 µg/kg (C18) vs. 0,136 µg/kg (HILIC)	content was confirmed by HILIC chromatography								analyte is in sample, but accuracy is not in the range of 80 % - 120 %	
20	2301-MP											
24	2301-MP											
25	2301-MP	only one MS/MS transition	only one MS/MS transition	not detected	not detected				not detected	not detected		
27	2301-MP											
28	2301-MP	213->169	262,9->219									
31	2301-MP	not validated	just 1 ms/ms transition used, not confirmed. LOQ in milk = 0.01ug/kg (used for routine samples), LOQ in FD milk = 0.08ug/kg	LOQ in milk = 0.01ug/kg (used for routine samples), LOQ in FD milk = 0.08ug/kg	LOQ in milk = 0.01ug/kg (used for routine samples), LOQ in FD milk = 0.08ug/kg	LOQ in milk = 0.01ug/kg (used for routine samples), LOQ in FD milk = 0.08ug/kg	LOQ in milk = 0.01ug/kg (used for routine samples), LOQ in FD milk = 0.08ug/kg	LOQ in milk = 0.01ug/kg (used for routine samples), LOQ in FD milk = 0.08ug/kg	LOQ in milk = 0.01ug/kg (used for routine samples), LOQ in FD milk = 0.08ug/kg	LOQ in milk = 0.01ug/kg (used for routine samples), LOQ in FD milk = 0.08ug/kg	not validated	LOQ in milk = 0.01ug/kg (used for routine samples), LOQ in FD milk = 0.08ug/kg
34	2301-MP	Only one MS/MS transition was used, no other confirmation	Only one MS/MS transition was used, no other confirmation			Linear part reported						
45	2301-MP	only one MS/MS Transition	only one MS/MS Transition									
46	2301-MP											
47	2301-MP		one transition, no confirmation									
49	2301-MP					only linear form						
51	2301-MP											
53	2301-MP	no confirmation	no confirmation									
55	2301-MP	2 transitions:169 and 213	2 transitions: 219 and 262									
57	2301-MP	MSMS confirmation only	MSMS confirmation only									
58	2301-MP	only one MS/MS transition	only one MS/MS transition									
59	2301-MP											
60	2301-MP	only one MS/MS	only one MS/MS									
63	2301-MP											
76	2301-MP	Not analyzed	Not yet validated. Confirmed by a second chromatographic system	Not yet validated	Not yet validated			Not yet validated	Not yet validated	Not yet validated	Not yet validated	Not yet validated
77	2301-MP											
78	2301-MP											
82	2301-MP	not confirmed	not confirmed									
84	2301-MP	HRMS	HRMS	The uncertainty is the %RSD		The uncertainty is the %RSD	The uncertainty is the %RSD	The uncertainty is the %RSD				
93	2301-MP											
95	2301-MP	a content of 0,075 µg/kg was quantified based on standard-addition									standard-addition was used to quantify the content	
96	2301-MP	1 MS/MS transition / no confirmation	1 MS/MS transition / no confirmation									
99	2301-MP	Confirmed by HRMS	Confirmed by HRMS									
100	2301-MP	< LOQ		< LOQ	< LOQ				not analyzed	not analyzed		
107	2301-MP	0,433µg/kg <LOQ as screeningresult	confirmation with second chromatographic run							not part of used Standardmix		
108	2301-MP	HRMS	HRMS									
109	2301-MP	HRMS	HRMS									
110	2301-MP											
115	2301-MP	Confirmed by a second chromatographic separation	Confirmed by a second chromatographic separation									
119	2301-MP											
122	2301-MP	only one MS/MS transition and no additional confirmation	only one MS/MS transition and no additional confirmation									
123	2301-MP		only one MS/MS transition									
128	2301-MP	only one MS/MS transition was used for identification	only one MS/MS transition was used for identification									
60*	2301-MP	only one MS/MS	only one MS/MS									

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder 2023 [EURL-PT-POP_2301-MP]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Power (2301-MP)
Methods Perfluoroalkylsulfonic acids (PFSAs) - Internal Standards

LC	Sample	Perfluorobutanesulfonic acid (PFBS)	Perfluoropentanesulfonic acid (PFPeS)	Perfluorohexanesulfonic acid (PFHxS)	Perfluoroheptanesulfonic acid (PFHpS)	Linear Perfluorooctanesulfonic acid (L-PFOS)	Sum of branched PFOS acids (br-PFOS)	Total-Perfluorooctanesulfonic acids (total PFOS)	Perfluorononanesulfonic acid (PFNS)	Perfluorodecanesulfonic acid (PFDS)	Perfluoroundecane-sulfonic acid (PFUnDS)	Perfluorododecane-sulfonic acid (PFDoDS)	Perfluorotridecane-sulfonic acid (PFTrDS)
1	2301-MP			ILIS L-PFHxS		ILIS L-PFOS	ILIS L-PFOS						
2	2301-MP	13C3 PFBS	13C3 PFBS	13C3 PFHxS	13C3 PFHxS	13C8 PFOS	13C8 PFOS		13C8 PFOS	13C8 PFOS		13C8 PFOS	
3	2301-MP	MPFBS	MPFHxS	MPFHxS	MPFOS	MPFOS	MPFOS	MPFOS	MPFHxS	MPFHxS	MPFHxS	MPFHxS	MPFHxS
6	2301-MP			MPFHxS				MPFOS					
8	2301-MP	M3PFBS	M5PFHxA	M3PFHxS	M8PFOA	M8PFOS	M8PFOS	M8PFOS	M8PFOS	M8PFOS			
11	2301-MP	PFBS	PFBS	PFHxS	PFHxS	PFOS	PFOS		PFOS	PFOS		PFOS	
11A	2301-MP	PFBS	PFBS	PFHxS	PFHxS	PFOS		PFOS	PFOS	PFOS		PFOS	
14	2301-MP	PFBS-13C3	PFHxS-13C3	PFHxS-13C3	lin-PFOS-13C8	lin-PFOS-13C8	lin-PFOS-13C8		lin-PFOS-13C8	lin-PFOS-13C8			
15	2301-MP	M3PFHxS (Sodium perfluor-1-(1,2,3-13C3)Hexane sulfonate)		M3PFHxS (Sodium perfluor-1-(1,2,3-13C3)Hexane sulfonate)	M3PFHxS (Sodium perfluor-1-(1,2,3-13C3)Hexane sulfonate)	M8PFOS (Sodium perfluoro-1-(1,2,3,4-13C4) octanesulfonate)				M8PFOA (perfluoro-n-(13C8) octanoic acid)			
17	2301-MP			PFHxS isotope-labelled				PFOS isotope-labelled					
18	2301-MP			PFHxS-18O2		PFOS-13C4	PFOS-13C4						
19	2301-MP	13C-PFBS	13C-PFHpA	13C-PFHxS	13C-PFNA	13C-PFOS	13C-PFOS		13C-PFUnDA	13C-PFOS	13C-PFTeDA	13C-PFTeDA	13C-PFOS
20	2301-MP												
24	2301-MP			MPFxS		MPFxS	MPFxS	MPFxS					
25	2301-MP	13C3-PFBS	13C3-PFHxS	13C3-PFHxS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C8-PFOS
27	2301-MP			13C-PFHxS		13C-PFOS	13C-PFOS	13C-PFOS					
28	2301-MP	PFBS13C3		PFHxS13C3	PFHxS13C3	PFOS13C8							
31	2301-MP			13C-PFHxS	13C-PFOS	13C-PFOS	13C-PFOS	13C-PFOS					
34	2301-MP	13C3-PFBS	18O2-PFHxS	18O2-PFHxS	18O2-PFHxS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS
45	2301-MP	13C3-PFBS	13C3-PFHxS	13C3-PFHxS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C8-PFOS		13C7-PFUnDA		13C-PFTeA	
46	2301-MP	sodium perfluoro-1-[2,3,4-13C12] butanesulfonate	sodium perfluoro-1-hexane[18O2]sulfonate	sodium perfluoro-1-hexane[18O2]sulfonate	sodium perfluoro-1-hexane[18O2]sulfonate	Sodium perfluoro-1-[1,2,3,4-13C12] octanesulfonate.	Sodium perfluoro-1-[1,2,3,4-13C12] octanesulfonate.	Sodium perfluoro-1-[1,2,3,4-13C12] octanesulfonate.					
47	2301-MP	sodium perfluoro-1-hexane(18O2)sulfonate		sodium perfluoro-1-hexane(18O2)sulfonate				sodium perfluoro-1-(1,2,3,4-13C4)octanesulfonate		sodium perfluoro-1-(1,2,3,4-13C4)octanesulfonate			
49	2301-MP	MPFHxS	MPFOS	MPFHxS	MPFHxS	MPFOS	MPFOS	MPFOS	MPFHxS	MPFHxS	MPFHxS		MPFTeDA
51	2301-MP	13C3-PFBS	13C3-PFBS	18O2-PFHxS	18O2-PFHxS	13C4-PFOS	13C4-PFOS	13C4-PFOS		13C2-PFUnA			
53	2301-MP	MPFBS	MPFHxS	MPFHxS	MPFHxS	MPFOS	MPFOS		MPFHxS	MPFOS		MPFDoDA	
55	2301-MP	13C-PFBS		18O-PFHxS	18O-PFHxS	13C-PFOS 80	13C-PFOS 80	13C-PFOS 80		13C-PFDoDA			
57	2301-MP	PFBS-13C3	PFBS-13C3	PFHxS-18O2	PFHxS-18O2	PFOS-13C4	PFOS-13C4	PFOS-13C4	PFOS-13C8	PFOS-13C8			
58	2301-MP	13C3-PFBS	13C8-PFOS	13C3-PFHxS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C8-PFOS	-	13C8-PFOS	-
59	2301-MP	13C3-PFBS		18O2-PFHxS	18O2-PFHxS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C8-PFOS		13C8-PFOS	13C2-PFDoA
60	2301-MP	Sodium perfluoro-1-hexane(18O2)sulfonate	Sodium perfluoro-1-hexane(18O2)sulfonate	Sodium perfluoro-1-hexane(18O2)sulfonate	Sodium perfluoro-1-hexane(18O2)sulfonate	Sodium perfluoro-1-(1,2,3,4-13C4)octanesulfonate			Sodium perfluoro-1-(1,2,3,4-13C4)octanesulfonate	Sodium perfluoro-1-(1,2,3,4-13C4)octanesulfonate	Sodium perfluoro-1-(1,2,3,4-13C4)octanesulfonate	Sodium perfluoro-1-(1,2,3,4-13C4)octanesulfonate	Sodium perfluoro-1-(1,2,3,4-13C4)octanesulfonate
63	2301-MP			13C3 - PFHxS				13C4 - PFOS					
76	2301-MP	Sodium perfluoro-1-[2,3,4-13C3]butanesulfonate M3PFBS		Sodium perfluoro-1-[1,2,3-13C3]hexanesulfonate M3PFHxS		Sodium perfluoro-1-[13C8]octanesulfonate M8PFOS	Sodium perfluoro-1-[13C8]octanesulfonate M8PFOS						
77	2301-MP			13C3-PFHxS		13C8-PFOS	13C8-PFOS						
78	2301-MP			13C PFHxS		13C PFOS							
82	2301-MP			13C3-PFHxS		13C8-PFOS		13C8-PFOS					
84	2301-MP	M3PFBS		M3PFHxS		M8PFOS		M8PFOS					
93	2301-MP	MPFBS		MPFHxS		MPFOS	MPFOS	MPFOS		MPFOS			
95	2301-MP	Perfluoro-1-(2,3,4-13C3)butansulfonsäure (Na.salz) (M3PFBS)		Perfluoro-1-(2,3,4-13C3)hexansulfonsäure (Na.salz) (M3PFHxS)		Perfluoro-n-(13C8)octansulfonsäure (Natriumsalz) (M8PFOS)	Perfluoro-n-(13C8)octansulfonsäure (Natriumsalz) (M8PFOS)	Perfluoro-n-(13C8)octansulfonsäure (Natriumsalz) (M8PFOS)					
96	2301-MP	13C PFBS	13C PFBS	13C PFHxS	13C PFHxS	13C PFOS	13C PFOS	13C PFOS	13C PFUnDA	13C PFUnDA	13C PFUnDA	13C PFUnDA	13C PFUnDA
99	2301-MP	Sodium perfluoro-1-(2,3,4-13C3)butanesulfonate		Sodium perfluoro-1-(1,2,3-13C3)hexanesulfonate				Sodium perfluoro-1 -(13C8)octanesulfonate					
100	2301-MP	MPFHxS		MPFHxS		MPFOS	MPFOS	MPFOS		MPFOS			
107	2301-MP	M3PFBS.IS	M4PFHpA.IS	M3PFHxS.IS	M4PFHpA.IS	M8PFOS.IS	M8PFOS.IS	M8PFOS.IS	M9PFNA	M2PFTeDA	Analyte not tested	Analyte not tested	Analyte not tested
108	2301-MP	Isotope labeled analogue	Isotope labeled PFHpA	Isotope labeled analogue	Isotope labeled PFOA			Isotope labeled analogue	Isotope labeled PFDA	isotope labeled PFDoDA	isotope labeled PFDoDA	isotope labeled PFDoDA	Isotope labeled PFTeDA
109	2301-MP	13C3-L-PFBS	13C3-L-PFHxS	13C3-L-PFHxS	13C3-L-PFHxS	13C8-L-PFOS	13C8-L-PFOS	13C8-L-PFOS	13C6-PFDA	13C7-PFUdA		13C7-PFUdA	
110	2301-MP												
115	2301-MP	13C3-PFBS	13C3-PFHxS	13C3-PFHxS	13C3-PFHxS	13C8-PFOS	13C8-PFOS		13C8-PFOS	13C6-PFDA		13C2-PFDoDA	
119	2301-MP	sodium perfluoro1-[2,3,4-13C3] butansolfonato		sodium perfluoro-1-[1,2,3-13C3] esanosolfonato (M3PFHxS)		sodium perfluoro1-[13C8] octansolfonato							
122	2301-MP	M3PFBS	M3PFBS	M3PFHxS	M4PFOS	M4PFOS	M4PFOS	M4PFOS	M4PFOS	M4PFOS			
123	2301-MP	PFBS-13C3	PHHxS-13C3	PFHxS-13C3	PFOS-13C8	PFOS-13C8	PFOS-13C8	PFOS-13C8	PFOS-13C8	PFOS-13C8		PFOS-13C8	
128	2301-MP	13C3-PFBS	13C3-PFHxS	13C3-PFHxS	13C3-PFHxS	-	-	13C8-PFOS	13C8-PFOS	13C8-PFOS	-	13C8-PFOS	-
60*	2301-MP	Sodium perfluoro-1-hexane(18O2)sulfonate	Sodium perfluoro-1-hexane(18O2)sulfonate	Sodium perfluoro-1-hexane(18O2)sulfonate	Sodium perfluoro-1-hexane(18O2)sulfonate	Sodium perfluoro-1-(1,2,3,4-13C4)octanesulfonate			Sodium perfluoro-1-(1,2,3,4-13C4)octanesulfonate	Sodium perfluoro-1-(1,2,3,4-13C4)octanesulfonate	Sodium perfluoro-1-(1,2,3,4-13C4)octanesulfonate	Sodium perfluoro-1-(1,2,3,4-13C4)octanesulfonate	Sodium perfluoro-1-(1,2,3,4-13C4)octanesulfonate
128*	2301-MP												

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder 2023 [EURL-PT-POP_2301-MP]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Power (2301-MP)
Methods Perfluoroalkylsulfonic acids (PFSAs) - Recovery Standards

LC	Sample	Perfluorobutanesulfonic acid (PFBS)	Perfluoropentanesulfonic acid (PFPeS)	Perfluorohexanesulfonic acid (PFHxS)	Perfluoroheptanesulfonic acid (PFHpS)	Linear Perfluorooctanesulfonic acid (L-PFOS)	Sum of branched PFOS acids (br-PFOS)	Total-Perfluorooctanesulfonic acids (total PFOS)	Perfluorononanesulfonic acid (PFNS)	Perfluorodecanesulfonic acid (PFDS)	Perfluoroundecane-sulfonic acid (PFUnDS)	Perfluorododecane-sulfonic acid (PFDoDS)	Perfluorotridecane-sulfonic acid (PFTrDS)
1	2301-MP												
2	2301-MP	18O2 PFHxS	18O2 PFHxS	18O2 PFHxS	18O2 PFHxS	13C4 PFOS	13C4 PFOS		13C4 PFOS	13C4 PFOS		13C4 PFOS	
3	2301-MP	MPFBA injection	MPFBA injection	MPFBA injection	MPFOS injection	MPFOS injection	MPFOS injection	MPFOS injection	MPFBA injection	MPFBA injection	MPFBA injection	MPFBA injection	MPFBA injection
6	2301-MP												
8	2301-MP												
11	2301-MP	PFOS	PFOS	PFOS	PFOS	PFOS	PFOS		PFOS	PFOS		PFOS	
11A	2301-MP	PFOS	PFOS	PFOS	PFOS	PFOS			PFOS	PFOS		PFOS	
14	2301-MP												
15	2301-MP												
17	2301-MP												
18	2301-MP												
19	2301-MP	R-PFDA		R-PFOS		R-PFOS							
20	2301-MP												
24	2301-MP			d5-N-EtFOSA		d5-N-EtFOSA	d5-N-EtFOSA	d5-N-EtFOSA					
25	2301-MP												
27	2301-MP												
28	2301-MP	PFOS13C4		PFOS13C4	PFOS13C4	PFOS13C4	PFOS13C4						
31	2301-MP			MPFOS	MPFOS	MPFOS	MPFOS	MPFOS					
34	2301-MP												
45	2301-MP												
46	2301-MP	Sodium perfluoro-[13C8]octanesulfonate	Sodium perfluoro-[13C8]octanesulfonate	Sodium perfluoro-[13C8]octanesulfonate	Sodium perfluoro-[13C8]octanesulfonate	Sodium perfluoro-[13C8]octanesulfonate	Sodium perfluoro-[13C8]octanesulfonate	Sodium perfluoro-[13C8]octanesulfonate					
47	2301-MP												
49	2301-MP												
51	2301-MP	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA		13C4-PFOA			
53	2301-MP												
55	2301-MP	PFOA - M8		PFOA - M8	PFOA - M8	PFOS - M8	PFOS - M8	PFOS - M8		PFOA - M8			
57	2301-MP												
58	2301-MP	13C2-PFHxA	13C2-PFHxA	13C2-PFHxA	13C5-PFNA	13C5-PFNA	13C5-PFNA	13C5-PFNA	13C5-PFNA	13C5-PFNA		13C5-PFNA	
59	2301-MP												
60	2301-MP												
63	2301-MP												
76	2301-MP												
77	2301-MP			18O2-PFHxS		13C4-PFOS	13C4-PFOS						
78	2301-MP			none		none							
82	2301-MP			no recovery standard		no recovery standard		no recovery standard					
84	2301-MP	47.00		55.00		68.00		68.00					
93	2301-MP												
95	2301-MP												
96	2301-MP	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS
99	2301-MP												
100	2301-MP	n.a.		n.a.	n.a.	n.a.	n.a.	n.a.		n.a			
107	2301-MP												
108	2301-MP	Not used	Not used	Not used	Not used	Not used	Not used	Not used	Not used	Not used	Not used	Not used	Not used
109	2301-MP	No	No	No	No	No	No	No	No	No		No	
110	2301-MP												
115	2301-MP	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS		13C4-PFOS	13C4-PFOS		13C4-PFOS	
119	2301-MP			90.90		96.60							
122	2301-MP	-	-	-	-	-	-	-	-	-	-	-	-
123	2301-MP	PFOS-13C3	PFOS-13C3	PFOS-13C3	PFOS-13C3	PFOS-13C3	PFOS-13C3	PFOS-13C3	PFOS-13C3	PFOS-13C3		PFOS-13C3	
128	2301-MP	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	-	-	13C4-PFOS	13C4-PFOS	13C4-PFOS	-	13C4-PFOS	-
60*	2301-MP												
128*	2301-MP												

LC	Sample	Perfluorobutanesulfonic acid (PFBS)	Perfluoropentanesulfonic acid (PFPeS)	Perfluorohexanesulfonic acid (PFHxS)	Perfluoroheptanesulfonic acid (PFHpS)	Linear Perfluorooctanesulfonic acid (L-PFOS)	Sum of branched PFOS acids (br-PFOS)	Total-Perfluorooctanesulfonic acids (total PFOS)	Perfluorononanesulfonic acid (PFNS)	Perfluorodecanesulfonic acid (PFDS)	Perfluoroundecane-sulfonic acid (PFUnDS)	Perfluorododecane-sulfonic acid (PFDoDS)	Perfluorotridecane-sulfonic acid (PFTrDS)
1	2301-MP						br-PFOSK from a technical mixture; MS/MS: 499.0/79.9 and 499.0/99.0	br-PFOSK from a technical mixture; MS/MS: 499.0/79.9 and 499.0/99.0; MU calculated by quare root of square sum of L- and br-PFOS; no LOQ because sumparameter					
2	2301-MP												
3	2301-MP						L-PFOS, HRMS						
6	2301-MP												
8	2301-MP						L-PFOS, di-PFOS and mono-PFOS chromatographically separated	L-PFOS, di-PFOS and mono-PFOS chromatographically separated					
11	2301-MP						L-PFOS - HRMS (FullScan ddMS2)	sum of L-PFOS and br-PFOS					
11A	2301-MP					L-PFOS was quantified using a calibration curve with L-PFOS and mass-labelled PFOS.	br-PFOS was calculated as difference between Total PFOS and L-PFOS	A 3-point calibration curve was built-up using a mixture of linear and branched PFOS (br-PFOSK - Wellington laboratories) and mass-labelled PFOS was used as IS. The calculated RF was applied to quantify Total PFOS in the sample.					
14	2301-MP						br-pfos 499>80						
15	2301-MP	Below our LOQ	not part of our scope		Detected below our LOQ		not part of our scope		not part of our scope	Below our LOQ	not part of our scope	not part of our scope	not part of our scope
17	2301-MP												
18	2301-MP			30		30	30						
19	2301-MP					m/z 499 > 80	L-PFOS; m/z 499 > 80						
20	2301-MP												
24	2301-MP												
25	2301-MP	not detected	not detected		not detected	PFOSK: linear isomer; quantification: 499 -> 80, confirmation: 499 -> 99	PFOSK: sum of branched isomers; quantification: 499 -> 80, confirmation: 499 -> 99	sum of branched and linear isomers; chromatographically separated	not detected	not detected	not detected	not detected	not detected
27	2301-MP												
28	2301-MP		not analyzed						not analyzed	not analyzed	not analyzed	not analyzed	not analyzed
31	2301-MP			L- PFHxS only. LOQ in milk = 0.01ug/kg (used for routine samples), LOQ in FD milk = 0.08ug/kg	LOQ in milk = 0.01ug/kg (used for routine samples), LOQ in FD milk = 0.08ug/kg	499>99 used for quan. LOQ in milk = 0.01ug/kg (used for routine samples), LOQ in FD milk = 0.08ug/kg	499>99 used for quan. Br quanitified from Br std in curve. Std = UNEP.PFAS.SOLN.A from wellington. COA available from State Lab if required. Contains 78.8% L PFOS and 21.1% Br PFOS isomers, as established by NMR. LOQ in milk = 0.01ug/kg (used for routine samples), LOQ in FD milk = 0.08ug/kg	TOTal PFOS = L + Br total LOQ in milk = 0.01ug/kg (used for routine samples), LOQ in FD milk = 0.08ug/kg					
34	2301-MP			Linear part reported			Total PFOS - Linear PFOS	498.8 -> 79.7 ; 498.8 -> 98.7					
45	2301-MP												
46	2301-MP												
47	2301-MP												
49	2301-MP			only linear form			Reference standard: L-PFOS; mass transitions: m/z 499 > 80	Reference standard: L-PFOS; mass transitions: m/z 499 > 80					
51	2301-MP												
53	2301-MP						L-PFOS 499->80; average 499-> 80 and 499->99 = 0,161 µg/kg						
55	2301-MP						quantified on l-PFOS, trans 99	see above					
57	2301-MP					L-PFOS used for quantification	L-PFOS used for quantification	L-PFOS used for quantification					
58	2301-MP						L-PFOS 489.9 -> 80	L-PFOS 489.9 -> 80			not analysed		not analysed
59	2301-MP												
60	2301-MP												
63	2301-MP							L-PFOS, 499.33-> 79.7 (98.9)					
76	2301-MP	Not yet validated	Not analyzed		Not analyzed		Quan by L-PFOS standards, MRM 499>80. Confirmed by a second chromatographic system		Not analyzed	Not yet validated	Not analyzed	Not analyzed	Not analyzed
77	2301-MP												
78	2301-MP												
82	2301-MP			L-PFHxS		L-PFOS, 499>80		L-PFOS, 499>80					
84	2301-MP	The uncertainty is the %RSD		The uncertainty is the %RSD		The uncertainty is the %RSD	not quantified	L-PFOS, HRMS; The uncertainty is the %RSD					
93	2301-MP						quantified with l-PFOS						
95	2301-MP	external calibration	external calibration	Content L-PFHxS; content of br-PFHxS: 0,017	external calibration		L-PFOS was used to quantify br-PFOS	L-PFOS was used to quantify br-PFOS	external calibration	external calibration	external calibration	external calibration	external calibration
96	2301-MP					L-PFOS / quantified using 498,9>80	L-PFOS / quantified using 498,9>80	L-PFOS / quantified using 498,9>80					
99	2301-MP						L-PFOS (499-79.93)						
100	2301-MP	< LOQ	not analyzed	near LOQ	< LOQ				not analyzed	< LOQ			
107	2301-MP			br-PFHxS 0,040 / L-PFHxS 0,062							not part of used Standardmix	not part of used Standardmix	not part of used Standardmix
108	2301-MP							HRMS					
109	2301-MP			sum of Linear and branched			13C8-L-PFOS	13C8-L-PFOS					
110	2301-MP												
115	2301-MP						L-PFOS; transition: 499>80	L-PFOS; transition: 499>80					
119	2301-MP												
122	2301-MP						reference Standard: L-PFOS; mass transition: 499 > 80	reference Standard: L-PFOS; mass transition: 499 > 80			we do not measure this compound	we do not measure this compound	we do not measure this compound
123	2301-MP						same MS transition of linear PFOS	498,9>80,1 498,9>99,1			not analized		not analized
128	2301-MP					not separated	not separated				not examined		not examined
60*	2301-MP												
128*	2301-MP												

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Milk Power (2301-MP)

Methods Sum of PFOS, PFOA, PFNA, PFHxS - Comments

LC	Sample		Sum of total-PFOS, PFOA, PFNA, PFHxS **(ub)**	Sum of total-PFOS, PFOA, PFNA, PFHxS **(lb)**
1	2301-MP		MU calculated by square root of square sum of individual MU's	MU calculated by square root of square sum of individual MU's
2	2301-MP			
3	2301-MP			
6	2301-MP			
8	2301-MP		See LOQ for linear and branched PFOS in tab A2	
11	2301-MP		For PFOS was considered the MU of L-PFOS	
11A	2301-MP		For PFOS was considered the MU of L-PFOS	
14	2301-MP			
15	2301-MP			
17	2301-MP			
18	2301-MP			
19	2301-MP			
20	2301-MP			
24	2301-MP			
25	2301-MP		sum of branched and linear isomers of PFOS; chromatographically separated	sum of branched and linear isomers of PFOS; chromatographically separated
27	2301-MP			
28	2301-MP			
31	2301-MP			no MU% possible. Absolute MU = 0.23
34	2301-MP			
45	2301-MP			
46	2301-MP			
47	2301-MP			
49	2301-MP			
51	2301-MP			
53	2301-MP			
55	2301-MP			
57	2301-MP			
58	2301-MP			
59	2301-MP			
60	2301-MP			
63	2301-MP			
76	2301-MP			
77	2301-MP			
78	2301-MP			
82	2301-MP		L-PFHxS	L-PFHxS
84	2301-MP		The uncertainty is the expanded %RSD	The uncertainty is the expanded %RSD
93	2301-MP			
95	2301-MP		only L-PFHxS content was used	only L-PFHxS content was used
96	2301-MP		square root of the sum of squares of the individual combined uncertainties	square root of the sum of squares of the individual combined uncertainties
99	2301-MP			
100	2301-MP			
107	2301-MP			
108	2301-MP			
109	2301-MP			
110	2301-MP			
115	2301-MP			
119	2301-MP			
122	2301-MP			
123	2301-MP			
128	2301-MP			
60*	2301-MP			

Milk Power (2301-MP)
Methods Other PFAS - Internal Standards

LC	Sample		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**
1	2301-MP								
2	2301-MP		13C8 PFOSA	13C3 HFPO-DA	13C3 HFPO-DA	13C8 PFOS	13C8 PFOS		
3	2301-MP				M3HFPO-DA	MPFHxS	MPFHxS		
6	2301-MP								
8	2301-MP		MPFOSA	MPFHpA	M3HFPO-DA	M8PFOS	MPFDoDA		
11	2301-MP								
11A	2301-MP								
14	2301-MP			PFHpA-13C2		lin-PFOS-13C8	lin-PFOS-13C8		
15	2301-MP		M8PFOA (perfluoro-n-(13C8) octanoic acid)						
17	2301-MP								
18	2301-MP		13C-PFOSA	13C-PFHpA					
19	2301-MP								
20	2301-MP			d5-N-EtFOSA					
24	2301-MP		13C8-PFOSA	13C8-PFOA	13C3-HFPO-DA	13C3-PFHxS	13C3-PFHxS		
25	2301-MP								
27	2301-MP				PFHxA13C5				
28	2301-MP								
31	2301-MP		13C8-PFOSA	13C3-HPFO-DA	13C3-HPFO-DA				
34	2301-MP			13C4-PFHpA	13C3-HFPO-DA				
45	2301-MP								
46	2301-MP		Perfluoro-n-(1,2,3,4-13C4)octanoic acid						
47	2301-MP		MFOSA	MPFHxS	MGENX	MPFHxS	MPFHxS		MPFHxS
49	2301-MP		13C8-PFOSA	13C4-PFHPA	13C3-HFPODA				
51	2301-MP		MFOSA	MPFOA	MPFOS	MPFOS	MPFOS		
53	2301-MP			13C-PFHpA					
55	2301-MP								
57	2301-MP			13C3-HFPO-DA	13C3-HFPO-DA				
58	2301-MP		13C8-FOSA	13C4-PFHpA	13C3-HFPO-DA	13C8-PFOS	13C8-PFOS		
59	2301-MP								
60	2301-MP		Perfluoro-1-[13C8]octanesulfonamide						
63	2301-MP								
76	2301-MP								
77	2301-MP			13C4-PFHpA					
78	2301-MP								
82	2301-MP		MPFOS						
84	2301-MP		Perfluoro-n-(13C8)octansulfonamid (M8FOSA)	Perfluoro-4,8-dioxa-3H-nonansäure-13C2-Carboxyl Ammoniumsalz	2,3,3,3-Tetrafluoro-2(1,1,2,2,3,3,3-heptafluoropropoxy)-13C3-prppanoic acid (M3HFPO- DA)				
93	2301-MP			M13C3 GenX	M13C3 GenX	M13C2 PFUnDA	M13C2 PFUnDA		
95	2301-MP								
96	2301-MP								
99	2301-MP		M8FOSA	4MPFHpA	M5PFHxA	M8PFOA	M8PFOA	M8FOSA	
100	2301-MP		Isotope labeled analogue	Isotope labeled PFHpA	Isotope labeled analogue	Isotope labeled PFUdA			
107	2301-MP			13C5-PFHxA	13C3-M3HFPO-DA	13C7-PFUdA	13C7-PFUdA		
108	2301-MP								
109	2301-MP		13C8PFOSA						
110	2301-MP				perfluoro-n- [1,2,3,4, -13C4] heptanoic acid				
115	2301-MP								
119	2301-MP								
122	2301-MP			-	-	-	-	-	-
123	2301-MP								
128	2301-MP								
60*	2301-MP								

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Milk Power (2301-MP)
Methods Other PFAS - Recovery Standards

LC	Sample		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**
1	2301-MP								
2	2301-MP		13C4 PFOS	13C4 PFOS	13C4 PFOS	13C4 PFOS	13C4 PFOS		
3	2301-MP				MPFOA inj	MPFBA inj	MPFBA inj		
6	2301-MP								
8	2301-MP								
11	2301-MP								
11A	2301-MP								
14	2301-MP								
15	2301-MP		-	-	-	-	-	-	-
17	2301-MP								
18	2301-MP		R-PFOA	X					
19	2301-MP								
20	2301-MP			d3-Me-FOSA					
24	2301-MP								
25	2301-MP								
27	2301-MP				PFBA13C3				
28	2301-MP								
31	2301-MP								
34	2301-MP								
45	2301-MP								
46	2301-MP								
47	2301-MP								
49	2301-MP		13C4-PFOA	13C4-PFOA	13C4-PFOA				
51	2301-MP								
53	2301-MP			PFOA - M8					
55	2301-MP								
57	2301-MP			13C5-PFNA	13C5-PFNA				
58	2301-MP								
59	2301-MP								
60	2301-MP								
63	2301-MP								
76	2301-MP								
77	2301-MP			no recovery standard					
78	2301-MP								
82	2301-MP								
84	2301-MP								
93	2301-MP			13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS		
95	2301-MP								
96	2301-MP								
99	2301-MP								
100	2301-MP								
107	2301-MP			NO	NO	NO	NO		
108	2301-MP								
109	2301-MP		13C4-PFOS						
110	2301-MP								
115	2301-MP								
119	2301-MP								
122	2301-MP			-	-	-	-	-	-
123	2301-MP								
128	2301-MP								
60*	2301-MP								

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Milk Power (2301-MP)
Methods Other PFAS - Comments

LC	Sample		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**
1	2301-MP								
2	2301-MP								
3	2301-MP								
6	2301-MP								
8	2301-MP								
11	2301-MP								
11A	2301-MP								
14	2301-MP								
15	2301-MP			not part of our scope	not part of our scope	not part of our scope	not part of our scope	not part of our scope	not part of our scope
17	2301-MP								
18	2301-MP				analyte not included in method	analyte not included in method	analyte not included in method	analyte not included in method	analyte not included in method
19	2301-MP								
20	2301-MP								
24	2301-MP		not detected		not detected	not detected	not detected	not analysed	not analysed
25	2301-MP								
27	2301-MP		not analyzed	not analyzed		not analyzed	not analyzed	not analyzed	not analyzed
28	2301-MP								
31	2301-MP								
34	2301-MP								
45	2301-MP								
46	2301-MP								
47	2301-MP								
49	2301-MP								
51	2301-MP								
53	2301-MP								
55	2301-MP								
57	2301-MP		not analysed			not analysed	not analysed	not analysed	not analysed
58	2301-MP								
59	2301-MP								
60	2301-MP			Not analyzed	Not analyzed	Not analyzed	Not analyzed	Not analyzed	Not analyzed
63	2301-MP								
76	2301-MP								
77	2301-MP								
78	2301-MP								
82	2301-MP								
84	2301-MP					external calibration	external calibration		external calibration
93	2301-MP								
95	2301-MP								
96	2301-MP								
99	2301-MP								not part of used Standardmix
100	2301-MP								
107	2301-MP								
108	2301-MP								
109	2301-MP								
110	2301-MP								
115	2301-MP		we do not measure this compound	we do not measure this compound	we do not measure this compound	we do not measure this compound	we do not measure this compound	we do not measure this compound	we do not measure this compound
119	2301-MP								
122	2301-MP		not examined; by washing the online SPE, FOSA can not be detected	not examined	not examined	not examined	not examined	not examined	not examined
123	2301-MP								
128	2301-MP								
60*	2301-MP								

EURL Proficiency Test on Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Milk Powder 2023 [EURL-PT-POP_2301-MP]

EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

23 February 2024

Annex 7: Participants' methods for PFASs - Extraction, clean-up and detection

Test sample - Milk Powder (2301-MP)

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder 2023 [EURL-PT-POP_2301-MP]
EU-RL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Power (2301-MP)
Methods Perfluoroalkylcarboxylic acids (PFCAs), Perfluoroalkylsulfonic acids (PFSAs) and Other PFAS - Pre-treatment and extraction

LC	Sample	Pre-treatment and extraction				
1	2301-MP	to 5g of sample 10ml water was added	ILIS, 10ml ACN was added and shaken	salt mix I (4g MgSO4, 1g NaCl, 0.5g Na2H citrate, 1g Na3 citrate) was added	shaken, centrifugated	
2	2301-MP	Sample spiked with internal standards	Added Methanol with Potassium Hydroxide	Tumbled 8 hours	Centrifuged @ 3000 rpm	
3	2301-MP	Quechers extraction using Water, Acetonitrile, NaCl and MgSO4 salts.	After centrifugation, organic phase is taken for clean-up steps.			
6	2301-MP	Ultrasonic extraction with methanol & acetonitrile				
8	2301-MP	5 mL of acetonitrile is added and sample is vortexed and then shaked for 20 min at 250 rpm.	Sample is centrifudged and supernatant is poured in a new tube.	5 mL of fresh acetonitrile is added to the original tube and the precipitate is dislocated.	Sample is vortexed for 30s and then shaked for 20 min at 250 rpm. Sample is centrifudged and supernatant is poured in the tube containing the previous extract. Sample is blown down to 3-4 mL under a stream of N2 at 50°C	
11	2301-MP	Addition of 10 mL water and 10 mL acetonitrile. Sample was shaken for 15 min.	QuEChERS extraction packet was added to the extract and shaken again.			
11A	2301-MP	Addition of 10 mL water and 10 mL acetonitrile. Sample was shaken for 15 min.	QuEChERS extraction packet was added to the extract and shaken again.			
14	2301-MP	PFAS were extracted with acetonitrile				
15	2301-MP	Weigh 5 ± 0.01 grams of the sample into a 50 ml centrifuge tube, add 100 µl of Internal Standard.	Add 10 ml Acetonitrile with 5% Formic acid, Add 15 ml HPLC-Water for dry samples, Shake the collomix for 2 minutes.	Add 6.5 grams of QuEChERS salt, Shake in the collomix for 4 minutes.Centrifuge at 10000 rpm for 4 minutes		
17	2301-MP	Ion paring extraction in water + Na2CO3 + tetrabutylammonium and Methyl tert-butyl ether (MTBE)				
18	2301-MP	ultrasonic extraction using aconitrile				
19	2301-MP	# addition of 13,5 mL dest. water to 1,5 g	13 mL 1 % NH3 in ACN - # extraction with 10 mL ACN and 60 mL HCL (6 MOL), shaking (5 min) and ultrasonication (5 min)	# addtion of 6,5 g Mg2SO4 and 1 g NaCl # 15 min centrifugation, transfer of organic layer to 15 mL tube	# evaporation to 1 mL using nitrogen (50 °C) # addition of 9 mL 25mM NH4Ac buffer (pH = 6) # centrifugation and transfer to SPE cartridge	
20	2301-MP	1 g sample and 20 mL water and 10 mL acidified ACN with 0.15 % Formic acid	Added 4 g MgSO4 and 1 g NaCl	shaker for 5 min and 15 min ultrasound bath	Centrifugation for 15 min at 8000 rpm at 4o C	
24	2301-MP	The PFAS are extracted from the food samples using acetonitrile and formic acid. Following extraction, a modified QuEChERS extraction technique is performed. 6 g MgSO4 +1.5 NaCl				
25	2301-MP	5 g of sample + water+ HCOOH + acetonitrile - shaking -	addition of NaCl + MgSO4- shaking and centrifugation	removing of aliquot of organic layer for further purification		
27	2301-MP	Ultrasound assisted extraction with 1 mL of 0.2 M NaOH and 10 mL of acetonitrile.	After extraction sample was centrifuged and aliq. was dilluted to ~200 mL with Milli-Q grade water	and neutrilized with formic acid.		
28	2301-MP	Powder reconstitution after IS 13C addition	Quechers extraction with ACN and formic acid - centrifugation	concentration with nitrogen evaporation system	resume with water	
31	2301-MP	Addition of internal std	Alkaline digestion	Ext in Acetonitrile - shake and sonicate x 2		
34	2301-MP	Samples are spiked with internal standard. Afterwards 5mL of water is added, then 10mL acetonitrille and	150 ul formic acid. Samples are shaken for 1m.			
45	2301-MP	Extraction with acetonitrile				
46	2301-MP	Extraction was done using 10 ml of methanol/potassium hydroxide for 17 hours.				
47	2301-MP	soxhlet extraction with methanol				
49	2301-MP	No pre-treatments.	Addition of internal standards.	Extraction: shaking with water/acetonitrile at room temperature.		
51	2301-MP	Quechers-Extraction				
53	2301-MP	10 ml acetonitrile				
55	2301-MP	Centrifuge, afterwards remove fat layer by using a spoon to skim off the fat	Extraction with 10 mL MeOH	Addition of formic acid	Vortex, head-over-head, centrifugation	Dilution with MQ
57	2301-MP	add ISTD Mix to sample	add 10 mL water and vortex	add 10 mL acetonitrile and vortex	shake for 15 min	extract with ultra sonic for 15 min
58	2301-MP	2 mL H2O for PFAS analysis were added before extraction	solid.liquid extraction with 20 mL 0.1 % NH3 in acetonitrile			
59	2301-MP	Quechers				
60	2301-MP	The Sample is extracted using Acetonitril. The Acetonitrilphase is diluted with acidifed water and cleaned uped and concentrated via a weak anion exchange cartridge.				
63	2301-MP	1 g of sample	addition of internal standards + standard addition	extraction with water : acetonitrile 1:1 (10 + 10 ml)	vortex, shaking, vortex, shaking head over head	
76	2301-MP	Addition of 1,5 mL water. Extraction with ACN.				
77	2301-MP	Ultra sonic extraction with CAN				
78	2301-MP	1ml 200mM NaOH was added to the sample.	after that internal standards were added and the sample was thouroughly mixed.	Extraction was done with 5 ml MeOH and the mixture was shaked for 2 min, followed by 30min ultrasound.	after that, the sample was centrifuged and the supernatant was acidified with 4M HCl followed by shaking for 2 min.	After addition of 12ml water the sample was proccessed to clean up stage.
82	2301-MP	Sample + 10 mL H2O + 10 mL ACN	60 min ultrasonic extraction (shaking by hand every 15 min)	Cooling down to room temp	Addition of 4 g MgSO4, 1 g NaCl	Shaking by hand rapidly - Centrifugation
84	2301-MP	2 g of samples are introduced in a 50 ml PP tube and addition of 10 ul IS (ranging from 250 to 5000 ng/ml in methanol)	Agitation in a vortex and left to reach the equilibrium for 20 min	Addition of 10 ml Methanol with NaOH 10 mM and agitiation in a vortex and USAE 5 min	Orbital digestion for 2 h at 140 rpm, room temperature - Centrifugation at 4000 rpm for 10 min, room temperature	Supernatant introduced in a 50 ml PP tube - Evaporation till 1 ml under N2 current - Dilution with 50 ml ultra pure water (HPLC grade) and agitation
93	2301-MP	Weigh 2 g sample in 50 mL tube	Spike with standard and IS	Add 5 mL ACN and extract 3 min on genogrinder, 1500 rpm, ultrasonicate 15 min (ultrasonication bath)	Centrifuge 10 min (3500 rpm), transfer supernatant to 15 mL tube	Repeat extraction step - Freeze at -20 C - Defrost to 5 C, and centrifuge 5 min. at 3500 rpm
95	2301-MP	Pre-treatment: -		Extraction method: extraction according to a modified version of the QuEChERs method:	1g sample, addition of 10 ml water and 10 ml ACN , ultrasonic extraction, and shaking after addition of Citrat-Extraction-Tube (CET) for QuEChERs, centrifugation, clean up of the supernatant	
96	2301-MP	Extraction with MeOH/KOH				
99	2301-MP	2 ml 1% FA in water, shake. 8 ml 2% FA in MeOH shake. Centrifuge.				
100	2301-MP	addition of water	extraction with methanol			
107	2301-MP	weigh 2g of Sample (Milkpowder), add 5mL water, vortex				
108	2301-MP	Reconstitution of milk powder 2,5g in 20mL	Extraccion solvent Acetonitrile:Methanol (3:1)			
109	2301-MP	Double exctraction with acetonitrile				
110	2301-MP	quechers extraction : 10 mL of acetonitrile and 10 mL of water, 4g MgSO4+1g NaCl, vortex	30 min by ultrasonic bath	centrifugation at 4000 rpm at 2°C		
115	2301-MP	EXTRACTION WITH ACETONITRILE				
119	2301-MP	Quechers Extraction EN method (4g MgSO4 - 1g NaCl - 1g Trisodium Citrate dihydrate - 0,5 g disodium hydrogencitrate sesquihydrate Disodico Sesquidrato				
122	2301-MP	homogenization	addition of 10 mL water,10 mL acetonitrile and internal standard; agitate intensively	60 min ultrasonic bath (agitate all 15 min); addition of buffering salts for phase-separation	agitate intensively; centrifugation (4000 U/min, T<12°C)	5 mL aliquot; Add 5 mL water + mix; Use 80% (8 mL) for SPE
123	2301-MP	extraction with NaOH 0,05M in methanol				
128	2301-MP	Addition of acetonitrile, shake (5 min); Treatment in ultrasound bath (5 min); Addition of 1ml NaOH (1 M), shake (15 min); Treatment in ultrasound bath (15 min)	Addition of 0,5 g NaCl for phase separation, shake (5 min); Treatment in ultrasound bath (5 min), Centrifugation (3800 U/min, 7°C, 5 min)	Transfer supernatant to another centrifuge tube	second extraction step (10 ml) Acetonitril, shake (15 min); Treatment in ultrasound bath (15 min), Centrifugation (3800 U/min, 7°C, 5 min)	Addition of formic acid to adjust a pH value of 5-6.5
60*	2301-MP	The Sample is extracted using Acetonitril. The Acetonitrilphase is diluted with acidifed water and cleaned uped and concentrated via a weak anion exchange cartridge.				

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Milk Power (2301-MP)
Methods Perfluoroalkylcarboxylic acids (PFCAs), Perfluoroalkylsulfonic acids (PFSAs) and Other PFAS - Clean-up

LC	Sample	Clean-up				
1	2301-MP	frozen out with liquid N2, centrifugated	6ml of ACN phase was added to salt mix III (0.9g MgSO4, 0.15g d-SPE)	shaken and centrifugated	4ml was concentrated by evaporation to complete dryness and resolved in 400µl MeOH	filled in ALS-Vials for chromatrography
2	2301-MP	Eluant passed through activated carbon cartridge (ENVI-CARB 1000 mg), eluted with Methanol.	Evaporated under nitrogen, passed through 0.2 µm filter.	Transferred to polypropylene LC vial, recovery standard added.		
3	2301-MP	Purification on SPE Envicarb	Evaporation to 10 ml; Mix with water and pH adapted to 3	Purification on SPE Agilent polymeric weak anion exchange 200mg	Evaporation to 0,1 ml, recovery standards added and MeOH added for total volume of 0,5ml	Injection in Polypropelyne vial
6	2301-MP					
8	2301-MP	2 hexane clean up are performed.	The cleaned extract is then evaporate to almost dryness.	4 mL of methanol is added.	The extract is then blown down to 0.3 mL and 0.2 mL of water is added.	The 0.5 mL of final extract is vortexed, centrifudged and transferred in a PP HPLC vial
11	2301-MP	The extract was dissolved in 0.25 mL of 1 % acetic acid in methanol.	SPE cartridge (Strata-X-AW/Strata XL) was washed with 40 mL of 1 % NH4OH in methanol and conditioned	with 6 mL of methanol and 6 mL of water.	After loading the sample, cartridge was washed with 2 mL of 10 mM ammonium acetate, and compounds	eluted with 10 mL 1 % NH4OH in methanol.
11A	2301-MP	The extract was dissolved in 0.25 mL of 1 % acetic acid in methanol.	SPE cartridge (Strata-X-AW/Strata XL) was washed with 40 mL of 1 % NH4OH in methanol and conditioned	with 6 mL of methanol and 6 mL of water.	After loading the sample, cartridge was washed with 2 mL of 10 mM ammonium acetate, and compounds	eluted with 10 mL 1 % NH4OH in methanol.
14	2301-MP	SPE				
15	2301-MP	Add 7 ml of the extract to the dSPE tube (GCB, PSA, magnesiumsulfaat)	Shake in the Collomix for 2 minutes.Centrifuge at 4500 rpm for 6 minutes.	Evaporate 3 ml of the extract to 0.3 ml at 55 degrees Celsius.	Shake the extract in the vortex for 2 minutes, Transfer the sample to the vial.	
17	2301-MP	WAX SPE clean up				
18	2301-MP	degreasing using n-hexane	dispersive SPE using activated carbon			
19	2301-MP	# SPE (Strata X-AW, 200 mg)	# ENVI-Carb 250 mg, 3 mL	# elution using 4 x 1 mL methanol (neutral PFAS) and 4 x 1 mL 3% ammonia in methanol	# evaporation to dryness under nitrogen stream (50 °C)	# addition of recovery standard and MeOH/ 1% formic acid; final volume of 500 µL # freeze over night and syringe filtration (PES 0,2 µm)
20	2301-MP	Transfer 6 mL of upper layer to 15 mL centrifuge tube with PSA, C18 and Graphitize carbon black	Shaker for 5 min and centrifuge for 15 min at 8000 rpm at 4o C	Evaporate 5 mL of upper layer to dryeness with nitrogen at 40 oC	Reconstitution to 1 mL of MeOH	Run to LC-MS/MS
24	2301-MP	First step includes clean up with 900mg MgSO4, 300mg PSA and 150mf GCB	Second step includes SPE with WAX cartidges			
25	2301-MP	Z-Sep+, C18 and MgSO4	shaking and cetrifugation	removing of aliquot for evaporation	reconsitution in MeOH	
27	2301-MP	Cleanup with Strata-X-AW columns. Wash: 2% formic acid 1mL, Methanol 2 mL.	Elution with 1% NH4OH solution in methanol.	Evaporation under N2 in water bath (40 degrees) and reconstitution in 100 uL MeOH		
28	2301-MP	Purified with spe WAX activated WAX spe	sample in water addition wash with buffer	wash with Methanol; Methanol with NH4OH eluition	concentration with nitrogen evaporation system	spike recovery IS 13C and recostituited with methanol:water and acetic acid
31	2301-MP	2D SPE - OASIS WAX + STRATA GCB via ASPEC	Evaporation and reconsitution	Addition of recovery std.		
34	2301-MP	AOAC Quechers salts are added and sampels are shaken for 5m. Samples are centrifuged and 5mL of the	supernatant is transfered into a dSPE tube. Samples are shaken for 5m and centrifuged. Supertanas is	mixed with water (1:1) and analysed.		
45	2301-MP	Use of two different SPE-cartridges	1. Clean up with Capitiva EMR-Lipid	2. Clean up with Strata X-AW		
46	2301-MP	Solid phase extractions (SPE) cartridge Oasis WAX (150 mg, 6 ml) (Waters Corp., USA) and	ENVI Carb Solid Phase (500 mg, 6 mL) (Supelco, USA) were used.			
47	2301-MP	ion pair extraction	solvent exchange			
49	2301-MP	Clean up with quechers salts and degreasing by freezing.				
51	2301-MP	SPE Cleanup				
53	2301-MP	d-SPE C18 and ENVI-carb				
55	2301-MP	SPE (Strata-X-AW, 200mg/6ml, 33um, Phenomenex)	Condition with MeOH and 0.04M HCl in MQ	Washing with 25mM sodium acetate buffer and 0.04M HCl in MeOH	Elution with 2% NH4OH in ACN	
57	2301-MP	disperive SPE with Quechers salt kit 1	shake; centrifugation ad 4000 rpm for 10 min	take an 4mL aliquot, add 200 µL water, 5 µL glycerol and evaporate to 200 µL	reconstitute with acetonitril/water (10/90) to 400 µL and with use of ultra sonic	filter through syrigne filter after centrifugation
58	2301-MP	automated solid-phase extraction with two different SPE cartidges (1. ENVI-Carb and 2. STRATA-X AW)	ENVI-Carb as a not retaining SPE, Strata-X-AW as retaining SPE			
59	2301-MP	Quechers				
60	2301-MP	Weak anion exchange SPE (HR-XAW, Chromabond)				
63	2301-MP	addition of 4 g MgSO4 + 1 g NaCl - quick shaking + then head over head shaking for 5 mins	Centrifugation (4000 rpm, 10 min); 5 ml of acetonitrile layer transferred to new 15 ml centrifuge test-tube	addition of 150 mg MgSO4 + 50 mg PSA - shaking, vortex; Centrifugation (4000 rpm, 10 min)	2.5 ml of supernatant transferred to the new 15 ml centrifuge test-tube; addition of 12 ml water; vakuum SPE - Strata X-AW 200 mg 33 um Polymeric Weak Anion	4 ml placed to nitrogen concentrator (60°C, ca 2-3h) till one drop remain; dissolved in 500 ul of MeOH - vortex - centrifugation - transfer to vials
76	2301-MP	Dispersiv SPE, addition of MgSO4, NaCl, C18 dSPE and ENVI-carb dSPE.	Evaporation and disolution in 75% Methanol.			
77	2301-MP	Dispersive SPE				
78	2301-MP	WAX columns were first conditioned with NH4OH in methanol and with water.	After sample load, the analytes were eluted with 4 ml MeOH and 4ml 0.1% NH4OH in methanol.			
82	2301-MP					
84	2301-MP	SPE with Oasis WAX 3 cc:	1) Pre-conditioning with 2 x 2 ml of methanol and 2 x 2 ml water (spearately) 2) Loading of the sample at 1 ml/min under vaccuum conditions	3) Cartridge dried with vaccuum for 15 min 4) Elution with 2 x 2.5 ml of methanol 0.1% NH4OH in a 10 ml PP tube	Evaporation near to dryness under N2 current; Pass to LC-vial with inserto and further evaporation under N2 current till dryness;	Reconstitution with 100 ul of methanol:water (1:9)
93	2301-MP	Weigh a) 2 g MgSO4 + 0.5 g NaCl and b) 0.1 g C18 and 0.1 g ENVIcarb in 50 mL tube	Transfer supernatant extract to the tube	Shake 2 min.	Centrifuge 20 min, 400 rpm 5 C and transfer organic phase to 15 mL tube	Evaporate to 0.4 mL; add MeOH and evaporate to 0.5 mL
95	2301-MP	purification of the extracts using a mixed-mode weak anion-exchange sorbent combined with GCB SPE- tube (Strata PFAS (WAX/GCB); 200 mg / 50 mg in a 6 mL Tube)	SPE: condition: 4 ml 0,3% NH4OH/MeOH, 6 ml water	load: sample (8ml extract diluted with about 22 ml water (30 ml final volume))	wash: 2x 4 ml water; elute: 8 ml 0,3% NH4OH/MeOH	solvent evaporation to 100 µl water; addition of 100 µl ACN/MeOH-mix (10/90); sample measured in ACN-MeOH-H2O mix
96	2301-MP	SPE chromabond PFAS	SPE Envi Carb			
99	2301-MP	2.4 ml over EMR-L. wash 600 ul MeOH/H2O.				
100	2301-MP	SPE with Chromabond PFAS				
107	2301-MP	add IS mix, wait 10min, add 0,5g NaCL+0,5gMgSO4 and 10mL Acetonitrile.	Homogenize with UltaTurrax, zentrifuge 5min 14000g; evaporate upper ACN phase by nitrogenstream at 60°C to <0,5mL; reconstitiution of sample with 3ml 2,5% acetic acid	SPE with Strata W-AX 100mg/3mL; elution 2 times with 1% NH3 / MeOH; evaporate MeOH eluate by nitrogenstream at 60°C	reconstitiution of sample with 0,50 ml 0,05%FA in Water/Methanol 1:1	(all in plastic tubes)
108	2301-MP	Captiva EMR				
109	2301-MP	Weak anionic exchange (WAX) SPE plus graphitized carbon black (Envicarb)				
110	2301-MP	3 ml of supernatant was purified on dSPE	(PSA-Envicarb)	1 ml of extract was evapored to dryness	residue was dissolved in a solution of methanol and ammonium acetate 2mM	
115	2301-MP	SPE CLEAN-UP WITH STRATA X-AW 200 mg - dSPE-Envicarb 80mg				
119	2301-MP	Quechers Dispersive (150mg PSA - 900mg MgSO4 - 150mg C18 EC)				
122	2301-MP	SPE (Strata X-AW, 200 mg, 3 mL)	Wash: 2 mL 0,1% formic acid; 2 mL methanol	Elute: 4 mL methanol (w=0,1% ammonia); Dry	Dilute in 250 µL mixture of 0,1% formic acid : methanol, ratio 1 : 2	Ready for LCQQQ
123	2301-MP	SPE Oasis-WAX 200mg	sample in water pH<6	washing with water, methanol	elution with methanol 5% ammonia	evaporated on water bath at 50°C with nitrogen
128	2301-MP	Freezing out fats and non-polar components (-20°C)	Addition of 250 mg ENVI-Carb	online SPE (WAX weak anion exchange)		
60*	2301-MP	Weak anion exchange SPE (HR-XAW, Chromabond)				

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Milk Power (2301-MP)
Methods Perfluoroalkylcarboxylic acids (PFCAs), Perfluoroalkylsulfonic acids (PFSAs) and Other PFAS - Chromatographic separation and detection

LC	Sample	**Chromatographic separation and detection**					
1	2301-MP	LC: Agilent Technologies 1290 Infinity	MS: Sciex QTRAP 6500+	MRM-Mode	chromatographic column: EC 100/2 Nucleodur PFAS, 3µm - delay column: EC 50/2 Nucleodur PFAS Delay, 5µm	solvent A: 5mM NH4OAc in H2O - solvent B: 5mM NH4OAc in MeOH	gradient: 60:40 (v:v, A:B) to 5:95 (v:v, A:B) in 4 minutes - 4µl injection volume, 7.5 min total run, 45°C column temperature
2	2301-MP	2 µL of extract injected onto UPLC system, Waters Aquity BEH C18 2.1 x 50mm 1.7 µm.	Gradient of A) De-ionised water + ammonium acetate, B) Methanol + ammonuim acetate.	Detection by MSMS, Sciex 6500+ in Electron Spray negative mode, unit resolution	7 point calibration curve and internal standards used for quantitation	PFHxS and PFOS both used a combination of branched and linear certified calibration standards	
3	2301-MP	Injection on Orbitrap, ESI-	Mobile phase A : Water - acetate ammonium 20 mM/Methanol 96/4 v/v	Mobile phase B : Methanol/ Water - acetate ammonium 20 mM 96/4 v/v	Run time : 13 minutes	Column : Acquity UPLC BEH C18 1,7 µm	
6	2301-MP	LC-MS/MS					
8	2301-MP	Sample analysed by LC-MS/MS using an Agilent Model 1290 Infinity II HPLC and an ABSciex 6500+	tandem mass spectrometer.	Tandem mass spectrometer is operated in negative mode with acquisition in a scheduled MRM mode.	Trap column is used (between HPLC pump mixing unit and the multi-port autosampler valve)	Trap column Eclipse XDB-C18 4.6 mm x 20 mm x 1.8 µm - Zorbax XDB-C18 100 mm x 2.1 mm x 1.8 µm	Mobile phase A = 20 mM aqueous ammonium acetate, mobile phase B = methanol - Injection volume = 5 µL, flow rate = 300 µL/min, column temperature = 60°C - Gradient used
11	2301-MP	Chromatographic column: Luna Omega PS C18 (1,6 um 2,1x 100 mm)	Mobile phase: (A) Water 10 mM of ammonium acetate (B) Methanol:Acetonitrile (1:1)	LC-HRMS (ESI-)			
11A	2301-MP	Chromatographic column: Luna Omega PS C18 (1,6 um 2,1x 100 mm)	Mobile phase: (A) Water 10 mM of ammonium acetate (B) Methanol:Acetonitrile (1:1)	LC-HRMS (ESI-)			
14	2301-MP	LC-MS/MS separation using C18 column and triple-quadrupole as an MS detector					
15	2301-MP	Determination for PFAS in Milk by means of the Liquid Chromatography Mas Spectrometry (LC-MSMS).	Analytical Column: ZORBAX Eclipse Plus C18 RRHD (50mm 2.1mm 1.8µm)	Agilent UPLC 1290 infinity II, Agilent MS 6470 Triple Quad			
17	2301-MP	C18 HPLC column separation and ESI-MS/MS detection					
18	2301-MP	seperation on reversed phase C18					
19	2301-MP	Agilent InfinityLab Poroshell 120 2.7 µm column, EC-C18, 2,1 x 150 mm	Delay column: Agilent InfinityLab Poroshell 120 EC-C18, 3,0 x 50 mm, 2,7 µm LC-Säule	mobile phase A: 2 mmol/L ammonium acetate + 0,1 % acetic acid	mobile phase B: methanol/acetonitrile 60:40 v % - Flow: 0,25 mL/min	0-0,5 min: 20% B; 0,5-2 min to 55% B, 2-10 min to 80% B, 10-13 min to 98% B hold until 17,5 min,	17,5-18,5 min to 20% B - Injectionvolumn: 10 µL - MS/MS: 6495 B; resolution: unit to unit
20	2301-MP	LC-MS/MS	AB SCIEX 6500+	Gradient program	Total run 23 min	Mobile phase A: 20 mM ammonium Acetate - Mobile Phase B: Methanol	Scanning type: MRM - Column: ZorbaX Eclipse plus C18 2.1 x 100 mm, 1,8 µm
24	2301-MP	Waters BEH Shield C18 100x2,1um					
25	2301-MP	UHPLC-MS/MS analysis	5 ul injection, (A) 5 mM ammonium acetate in water and (B) MeOH	BEH C18 (100 × 2,1 mm; 1,7 µm); gradient elution	tandem mass spectrometry (triple quad, MRM mode), electrospray ionization in negative mode		
27	2301-MP	Kinetex 1.7u C18 (50x3 mm)	Instrument: Q-Exactive-Orbitrap-MS	Phases: 10 mM NH4FA in H2O/ACN (9:1) and MeOH/ACN (1:1)	Injection volume: 5 uL	Mode: PRM at 17500 FWHM resolution	
28	2301-MP	Column waters CSH Phenyl-Hexyl 1.7µm 2,1mm id 100mm l.	UPLC: mobile phase A: water 20mM acetic acid	UPLC: mobile phase B: Methanol 25mM ammonium Hydroxide	LC-MS/MS analysis		
31	2301-MP	The detection/confirmation is done by LCMSMS in MRM detection mode	The concentration of each analyte is calculated using the isotope dilution technique	The percent recoveries of the isotope dilution analogues are calculated using the integrated peak areas of the recovery standards	Quantitation is performed using a solvent curve & a stored calibration may be used	LC - ACQUITY UPLC I-Class with PFC kit- Column - ACQUITY CSH Phenyl hexyl 1.7µm, 2.1 x 100 mm	Injection vol 30 µL - MPA - 95:5 Water: methanol + 2mM ammonium acetate, MPB - Methanol + 2 mM ammonium acetate - MS - Xevo TQ-XS, ESI -
34	2301-MP	Chromatographic separation is acheived on a Acquity UPLC BEH C18 1.7 µm: 100 mm x 2.1 mm column.	Detection method is via MRM transitions (LC-MS/MS).				
45	2301-MP	separation on C18-reversed-phase with the usage of a binary solvent gradient	Detection: dynamic MRM analysis				
46	2301-MP	Mobile phases:20 mM ammonium acetate aqueous solution and methanol	Flow rate was 0.6 mL/min, and the injection volume of 10 µL.	Gemini C18 chromatographic column (3 µm, 50 x 2,0 mm).	Detection:LC-MS/MS using a Sciex 7500 system operated in the negative electrospray ionization		
47	2301-MP	biphenyl column 15 x 3,5 mm	binary gradient water/formiat methanol/carbonat				
49	2301-MP	Column C18 UPLC, 100 x 2.1 mm 1.6 µm (Waters)	Mobile phase: methanol /ammonium acetate	Injection volume: 5 µL	Flow rate 0.25 mL/min	Mass detector: triple quadrupole (SCIEX QTrap 7500)	
51	2301-MP	Measurement: HPLC	Injection method: standard	Injection volume (µl): 5	Stationary phase: C18-Phase	Detector: MS/MS	Scan mode: MRM
53	2301-MP	LC-MS/MS reversed phase chromatography					
55	2301-MP	LC-MS/MS	Column: Luna Omega 1.6u PS C18 100 A (100 x 21 mm)	Isolator column: Gemini 3µm C18 110A (50 x 3 mm)	Mobile phase A: 20 mM ammonium acetate in MQ	Mobile phase B: ACN	
57	2301-MP	use of delay column (XBridge C18, 50x2.1 mm, 3.5 µm, Waters)	separation column: XBridge C18, 100x2.1 mm, 3.5 µm, Waters	use of injector programm to clean autosampler parts, injection of 20 µL sample, 50°C column oven temp	Eluent A: 2 mM ammoniumacetate and 5% acetonitril; Eluent B: acetonitril/methanol (60/40, v/v)	flow: 0.25 mL/min - 0 min: 30% B, 9 min: 75% B, 12 min: 95% B, 15 min: 98% B, 19.5 min: 98% B, 20.5 min: 10% B, 24 min: 10% B	detection with agilent 6495 QQQ
58	2301-MP	LC-MS/MS	Analytical column: Acquity UPLC BEH C18, 150 x 3.0 mm, 2.7 µm	Trap column: InfinityLab PFC Delay Column 4.6 x 30 mm	HPLC gradient (Mobile phase A 2 mM ammonium acetate in water, 5 % acetonitrile,	mobile phase B: acetonitrile/ methanol 60/40 v/v)	Detection Triple quadrupole (ESI negative); dynamic multiple reaction monitoring
59	2301-MP	Gradient with MS/MS detection					
60	2301-MP	LC-MS/MS	delay column: Inifinity Lab PFC	pre-column: 2.1 mm x 5 mm Acquity UPLC BEH C18 VanGuard pre-column	Column: Acquity UPLC BEH C18(2.1 mm X 50 mm x 1.7 µm)		
63	2301-MP	delay column: HALO PFAS Delay (50 x 3.0 mm, 2.7 um, 160 A)	analytical column: Atlantis T3 (30 x 2.1 mm, 3 um)	MP: A - 2 mM Ammonium Acetate in MeOH	MP: B - 2 mM Ammonium Acetate in water	injection of 10 ul; temperature of column: 30 °C; gradient elution	LC-MS/MS detection (MRM) - 2 transitions: ESI negative
76	2301-MP	Detection LC-MS/MS, neg. Mode, MRM.	Waters Acquity UPLC BEH C18	Second chromatography system: Waters Acquity UPLC HSS T3			
77	2301-MP	UPLC-MS/MS (Waters system) Xevo TQ-XS/ACQUITY i-Class fitted with PFC kit					
78	2301-MP	Luna Omega Polar 100x2.1mm, 3um column, precolumn Polar C18 4x2mm	mobile phase A 10mM NH4OH	mobile phase B 10mM nH4OH in methanol	flow 0.3 ml/min	injection volume 40 ul	
82	2301-MP	HPLC-MS/MS (Dynamic MRM, ESI- with an Agilent 6470 mass spec)	Waters xBridge BEH C18 2.5 µm 2.1 x 150 mm using an Agilent UHPLC	Channel A: 100 % Water with 2 mM CH3COONH4	Channel B: 40 % ACN/60 % MeOH	2 min 90 % Channel A; 9 min 15 % Channel A; 12 min 2 % Channel A; 15 min 2 % Channel A	15.1 min 90 % Channel A; 20 min 90% Channel A
84	2301-MP	The analysis of PFASs was by means of liquid chromatography (LC) coupled to HRMS. Acquity LC (Waters, Milford, MA, USA) chromatography was equipped with an Hypersil GOLD PFP LC analytical column (50 x 3 µm) (Thermo Scientific).	The separation was achieved working under gradient conditions with (A) methanol 20 mM Ammonium acetate and (B) water 20 mM Ammonium acetate.	The system starts at 10% of A, maintained for 30 s and increased up to 90% A for 9.5 min.	Then, the gradient was maintained at this percentage for 3 min and returned to initial conditions within 1 min. The total run time was 14 min at a flow rate of 0.5 mL/min with an injection volume of 5 µL.	The chromatographic system was coupled to a QExactive (Thermo Fisher Scientific, San Jose, CA) mass spectrometer equipped with heated electrospray ionization source (HESI) operating under negative conditions with a spray voltage of 2500 V, capillary temperature of 350ºC, sheath gas at 50 a.u., auxiliary gas at 20 a.u. and S-Lens RF at 60 a.u.	The data was acquired in full scan (FS) mode from 66.7 to 1000 Da, at a resolving power of 70,000 FWHM.
93	2301-MP	LC-MS/MS Waters ACQUITY UPLC CSH C18 (130 Å, 1.7 µm, 100 x 2.1 mm) column;	Injection volume 5 µL, column oven temp.50°C, autosampler temp.10°C.	Eluent A: 2 mM ammonium acetate in Milli-Q water/methanol gradient			
95	2301-MP	LC-MS/MS, reversed phase, column: Gemini 3µ C18 110Ã, 100 x 2.0 , MRM measurement,	mobile phase 1: 95 % water + 5% ACN + 5mM ammonium acetate	mobile phase 2: 40 % MeOH + 60 % ACN + 5mM ammonium acetate			
96	2301-MP	LC-(ESI-)-MS/MS	Hypersil Gold (100 x 2,1 mm) 1,9 µm				
99	2301-MP	Delay column (C18), Zorbax Eclipse plus C18 column. Gradient MeOH/20 mm ammoniumacetate in H2O	Scheduled MRM (qualifier and quantifier transitions).				
100	2301-MP	HPLC with precolumn Phenomenex LUNA® PFP(2),	3µm, 100 A, 50 x 3 mm combined with Phenomenex Security Guard®	PFP(2)-Phase)	Column Phenomenex Kinetex® F5, 1.7 µm, 100 A, 50 x 2.1 mm	Graduent with 5 mM NH4Ac in Methanol and 5 mM Ammoniumacetat	HESI in positive mode - Multiple Reaction Monitoring
107	2301-MP	Luna Omega 3µ PS 150x2.1mm 00F-4756	Agilent_PFAS_Delay_Phase	Pump A: 0.05g NH4AC+AcOH 5%MeOH / Pump C: ACN / Pump D: MeoH	Gradient	Detection : Sciex Qtrap 6500+ MRM neg	
108	2301-MP	LC_HRMS (Q-Orbitrap)	Column: Hypersil GOLD C18 1,9µm 2,1x100mm	Delay column used	Gradient elution, mobile phase A: Water 2mM amonium acetate, B: Acetonitrile 2mM amonium acetate	Injection 10uL	Detection Full MS data dependant with inclusion list-ElectroSpraylonization ESI -
109	2301-MP	Reverse phase separation using XB column (100 x 2.1 mm, 3 µm-Phenomenex).	Mobile phases: water and MeOH both containing 5 mM of ammonium acetate	Acquisition: LC-Q-Orbitrap system using Full/SIM Scan mode (ESI-)			
110	2301-MP	The analysis of target PFASs was performed using an uHPLC coupled with a LRMS	Chromatographic separation was achieved with a Luna Omega Polar	PS"C18"column"(100"× 2.1"mm,"1.6"µm;"Phenomenex)	The mobile phase A was 2 mM ammonium acetate solution. The mobile phase B included ACN.	The flow rate was 0.2 mL/min, and column temperature was set at 40 °C. The injection volume was 10 µL.	
115	2301-MP	CHROMATOGRAPHIC COLUMN: Phenomex luna omega PS, C18,1.6 um (100 x 2.1 mm)	INJECTION MODE: PARTIAL LOOOP	INJECTION VOLUME: 20 microL	MOBILE PHASE: A) ACN; B) AMMONIUM ACETATE 2mM in H2O	FLOW: gradient from 0.1- 0.20 mL/min	DETECTION: LC-MS/MS; SOURCE: ESI
119	2301-MP	instrument: HPLC-MSMS	detection mode: MRM	Column: Aquity UPLC BEH Shield RP 18 1,7um	Mobile phases. A- ammonium acetate 10 mM; B Methanol + Acetonitrile and ammonium acetate 10 mM		
122	2301-MP	LCQQQ (Agilent Technologies 1290 Infinity, Agilent Technologies 6495C)	Pre-Column: Agilent InfinityLab PFC Delay Column, 4.6 x 30 mm	Column: Agilent Zorbax Eclipse XDB-C18, 1.8-Micron, 600Bar, 4,6 x 100mm	Flow rate: 0,3 mL/min, Column Temp: 40 °C, injection volume: 1 µL	Eluent A: 5 mmol NH4FA in H2O - Eluent B: 5 mmol NH4FA in MeOH	ESI, negative mode, capillary voltage:3000 V, nebulizer pressure: 20 psi, Sheath-Gas: T = 250 °C, flow rate: 11L/min, Gas flow rate: 14 L/min
123	2301-MP	HPLC-MS/MS, column BEH C18 100x2,1mm*1,7um,	mobile phase water/methanol with ammonium acetate				
128	2301-MP	LC-MS/MS	Eluent A 0,1% formic acid in water	Eluent B 0,25% NH3 and 0,05% formic acid in methanol	Pre-column and column Poroshell EC-C18	Delay-Column PFC Delay Column	
60*	2301-MP	LC-MS/MS	delay column: Inifinity Lab PFC	pre-column: 2.1 mm x 5 mm Acquity UPLC BEH C18 VanGuard pre-column	Column: Acquity UPLC BEH C18(2.1 mm X 50 mm x 1.7 µm)		

EURL Proficiency Test on Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Milk Powder 2023 [EURL-PT-POP_2301-MP]

EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

23 February 2024

Annex 8: Participants' methods for PFASs - Measurement uncertainty and Limit of Quantification

Test sample - Milk Powder (2301-MP)

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder 2023 [EURL-PT-POP_2301-MP]
EU-RL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Power (2301-MP)
Methods Perfluoroalkylcarboxylic acids (PFCAs), Perfluoroalkylsulfonic acids (PFSAs) and Other PFAS - LOQ determination, measurement uncertainty calculation and additional information

LC	Sample	Limit of quantification (LOQ) approach	Measurement Uncertainty (MU) approach	Additional Information
1	2301-MP	during validation of PFAS in milk matrix	during validation of PFAS in milk matrix square root of square sum of average of bias, standard deviation of bias and relative standard deviation of recovery	
2	2301-MP	Performance of calibration standards; Batch blank level	Top down estimation from reproducibility of spiked samples	
3	2301-MP	Lowest validated level of a similar procedure using Quecher extraction.	U= k*u=((2x CVrw) + biais)	
6	2301-MP			
8	2301-MP	LOQ was calculated as LOQ = 3 x LOD while LOD was calculated from the relationship LOD = 3 x S; Where S is the standard deviation of seven repicates of the representative liquid matrix fortified at a nominal concentration of 0.025 ug/kg. To calculate for powder the reconstitution ratio of 10 was taken into account.	The uncertainty was calculated as twice the standard deviation of the between day coefficient of variation.The between-day coefficient of variation was determined by analysing seven replicates of the representative liquid matrix fortified at three different concentrations (0.05 µg/kg, 1 µg/kg and 10 µg/kg) and analysed in three separate batches.	2g of milk powder reconstituted with 20 mL of DI water. Then 1g is taken for the extraction.
11	2301-MP	The LOQ was estimated as the lowest concentration of the sample fortified with acceptable precision and trueness, by applying the complete analytical method and identification criteria	The expanded measurement uncertainties were obtained using a top-down approach as reported in the “Guidance document on measurement uncertainty for laboratories performing PCDD/F and PCB analysis using isotope dilution mass spectrometry – 2017”. For the MU of the sum of four PFASs the RSS approach was used.	
11A	2301-MP	The LOQ was estimated as the lowest concentration of the sample fortified with acceptable precision and trueness, by applying the complete analytical method and identification criteria	The expanded measurement uncertainties were obtained using a top-down approach as reported in the “Guidance document on measurement uncertainty for laboratories performing PCDD/F and PCB analysis using isotope dilution mass spectrometry – 2017”.For the MU of the sum of four PFASs the RSS approach was used.	
14	2301-MP			
15	2301-MP	LOQ was determined according to NEN 7777:2011 & SANTE/11312/2021, by measuring 10 duplicates of blank material spiked at the LOD (limit of detection)	Measurement uncertainty was determined according to NEN 7777:2011 & SANTE/11312/2021,by measuring 10 duplicates of blank material spiked at 0.1 ug/kg and then calculated using the reproducibility, repeatability and the bias.	
17	2301-MP			
19	2301-MP		detection in multiple reaction monitoring mode (ESI neg)	
20	2301-MP	The LOQ was estimated using a S/N of 3 for the less intensive mrm transition.	U = k * u` (k = 2) The measurement uncertainty was determined using three fortified sample of the PT sample (Bias) and nine replicates of the PT sample (precision). Sum parameter: U = square root of sum of squares of MU of indivitual PFAS	
24	2301-MP			
25	2301-MP	at spiked samples (free of PFAS)		
27	2301-MP	as lowest calibration point where S/N > 10 (quantification transition) and S/N > 3 (confirmation transition if available)		
28	2301-MP			
31	2301-MP			If any additional details are required on method please contact hannah.smith@statelab.ie
34	2301-MP	Lowest validated level approach. Established by spiking milk samples and taking as the LOQ the lowest value which satisfied the validation criterea		
45	2301-MP			sample-no. 2301-MP-121
46	2301-MP			
47	2301-MP	LOD and LOQ were estimated based on analysis of 10 blank spiked samples	MU were estimated based on precision and truenes from fortyfication experiment	
49	2301-MP	spiked matrix S/N 10:1	repeatability of 10 spiked matrix samples	
51	2301-MP	By spiking samples at LOQ concentration level.	By combining the contributions from: the precision of the method, the systematic effects on mass and volume measurements, the recovery factor of each compound compared with the recovery of the respective internal reference material, the chromatographic response factor and the concentration of reference materials,	
53	2301-MP			
55	2301-MP	lowest validated level	According EURL Guidance Document PFAS	
57	2301-MP	lowest validated level method according to guidelines	validation - calculation based on accuracy, reproducibility	
58	2301-MP	lowest validated level		
59	2301-MP			
60	2301-MP			
76	2301-MP			

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EU-RL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Power (2301-MP)
Methods Perfluoroalkylcarboxylic acids (PFCAs), Perfluoroalkylsulfonic acids (PFSAs) and Other PFAS - LOQ determination, measurement uncertainty calculation and additional information

LC	Sample	Limit of quantification (LOQ) approach	Measurement Uncertainty (MU) approach	Additional Information
77	2301-MP			
78	2301-MP	Lowest validated level where criteria for identification, trueness and precision are met.	Combined measurement uncertainty Uc including bias and intermediere reproducibility x k (k=2) Bias calculated from PT and RSD from validation.	
82	2301-MP	lowest succesfully validated level	analysis of reference materials	
84	2301-MP	the LOQs were at first evaluated taking into account instrumental response and blanks. After that the LOQ was confirmed with fortification experiments.	MU was calculated taking into account within laboratory reproducibility and precision from fortification experiments.	
93	2301-MP	according to Guidance Document	Es gelten die extrapolierten Analysenspielräume des VDLUFA	
95	2301-MP	Experimentally. Blank fortified with a mixture of selected PFASs.	Use of replicates (n=3)	
96	2301-MP			
99	2301-MP	estimation of LOD and LOQ based on the analysis of spiked blank samples: Wenzl, T., Haedrich, J., Schaechtele, A., Robouch, P., Stroka, J., Guidance Document on the Estimation of LOD and LOQ for Measurements in the Field of Contaminants in Feed and Food; EUR 28099, Publications Office of the European Union, Luxembourg, 2016, ISBN 978-92-79-61768-3; doi:10.2787/8931		
100	2301-MP	S/n =10		
107	2301-MP	Method is under development. LOQ has been measured in Blank	RSD*2	
108	2301-MP	Smallest visible standard in typical matrix (standard addition)	Statistical evaluation (repeatability (n=6) in matrix and standard addition)	
109	2301-MP			
110	2301-MP			the analytical method being validated
115	2301-MP		Application of Horwitz-Thompson equation	
119	2301-MP			
122	2301-MP	Spiked samples at the LOQ	From validation data including reproducibility and trueness contributions	
123	2301-MP			
128	2301-MP	Measurement of different spiked samples; LOQ is the value were the following criteria are fulfilled: Recovery: 70-120%; RSD </=20%; Peak identification criteria fulfilled	Analysis of the QM-samples over a year Multiply the uncertainty with 2 to get the MU	branched and linear PFOS can not be detected separately
60*	2301-MP			

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EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Power (2301-MP)

Methods Perfluoroalkylcarboxylic acids (PFCAs) - Measurement Uncertainty [%]

LC	Sample		Perfluorobutanoic acid **(PFBA)**	Perfluoropentanoic acid **(PFPeA)**	Perfluorohexanoic acid **(PFHxA)**	Perfluoroheptanoic acid **(PFHpA)**	Perfluorooctanoic acid **(PFOA)**	Perfluorononanoic acid **(PFNA)**	Perfluorodecanoic acid **(PFDA)**	Perfluoroun- decanoic acid **(PFUnDA)**	Perfluorodo- decanoic acid **(PFDoDA)**	Perfluorotri- decanoic acid **(PFTrDA)**	Perfluorotetra- decanoic acid **(PFTeDA)**
1	2301-MP						48	46					
2	2301-MP		30	30	30	30	30	30	30	30	30	30	30
3	2301-MP			30	30	30	30	30	30	30	30	30	30
6	2301-MP												
8	2301-MP		10	9.2	19	11	28	7.8	12	12	17	46	11
11	2301-MP			30	31	48	27	43	29	27	39	27	32
11A	2301-MP			30	31	48	27	43	29	27	39	27	32
14	2301-MP												
15	2301-MP		34.7	24.4	29	20.9	20.3	37.1	29.8	26.7	30.1	26.6	37.6
17	2301-MP			0.5	50	50	50	50	50	50	50	50	50
18	2301-MP		30	30			25	25	25				
19	2301-MP			6		22	7	4	8				17
20	2301-MP												
24	2301-MP						30	30	30			30	
25	2301-MP		35	25			35	35	35			25	35
27	2301-MP												
28	2301-MP		37	32	36	37	36	34	32	39	38	40	45
31	2301-MP			35	35	35	35	35	35	35	35		35
34	2301-MP												
45	2301-MP		20	25	20	25	15	15	20	20	20	20	20
46	2301-MP				23	27	15	26	26	31	23		
47	2301-MP			26	17	32	25	15	26	17	28	28	30
49	2301-MP		45	45	45	45	45	45	45	45	45	45	45
51	2301-MP		30	30	30	30	30	30	30	30	30	30	30
53	2301-MP		20	30	25	20	30	20	20	25	20	30	25
55	2301-MP			19	20	18	42	33	12	15	24	24	39
57	2301-MP		50	50	50	50	50	50	50	50	50	50	50
58	2301-MP		25	25	25	25	20	20	25	25	25	50	50
59	2301-MP		37	37	37	37	37	37	37	37	37	37	37
60	2301-MP		45	45	40	40	30	35	40	40	40	45	45
63	2301-MP						18	25					
76	2301-MP			40			30	34	40			40	40
77	2301-MP						10	10					
78	2301-MP						20	20					
82	2301-MP		88	88	88	88	88	88	88	88	88	88	88
84	2301-MP				6.24		11.73	17.64	14.17				
93	2301-MP		25	14	14	20	7	6	10	6	7	7	7
95	2301-MP												
96	2301-MP						18	12					
99	2301-MP		20	22	30	30	30	34	16	20	20	26	
100	2301-MP		15.4	15.4	15.4	15.4	20.3	19	20.8			14.3	18.1
107	2301-MP												
108	2301-MP												
109	2301-MP		44	44			44	44	44			44	44
110	2301-MP												
115	2301-MP		28	28	40	40	28	28	28	40	40	28	28
119	2301-MP		44	44	44	44	44	44	44	44	44		
122	2301-MP		11	30	20	32	34	22	25	31	41	22	6
123	2301-MP												
128	2301-MP						30	30					
60*	2301-MP		45	45	40	40	30	35	40	40	40	45	45

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EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Power (2301-MP)

Methods Perfluoroalkylcarboxylic acids (PFCAs) - Limit of detection (LOQ) in µg/kg wet weight

LC	Sample		Perfluorobutanoic acid **(PFBA)**	Perfluoropentanoic acid **(PFPeA)**	Perfluorohexanoic acid **(PFHxA)**	Perfluoroheptanoic acid **(PFHpA)**	Perfluorooctanoic acid **(PFOA)**	Perfluorononanoic acid **(PFNA)**	Perfluorodecanoic acid **(PFDA)**	Perfluoroun-decanoic acid **(PFUnDA)**	Perfluorodo-decanoic acid **(PFDoDA)**	Perfluorotri- decanoic acid **(PFTrDA)**	Perfluorotetra-decanoic acid **(PFTeDA)**
1	2301-MP						0.01	0.02					
2	2301-MP		0.5	0.5	0.5	0.5	0.3	0.5	0.5	0.5	0.5	1	1
3	2301-MP			0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
6	2301-MP						0.05	0.05					
8	2301-MP		5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	5	5	5
11	2301-MP			0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
11A	2301-MP			0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
14	2301-MP		0.8	0.08	0.08	0.08	0.04	0.04	0.04	0.08	0.08	0.04	0.04
15	2301-MP		0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
17	2301-MP			0.03	0.012	0.012	0.006	0.006	0.012	0.012	0.012	0.03	0.06
18	2301-MP		0.1	0.05	0.03	0.03	0.03	0.04	0.02	0.03	0.02		
19	2301-MP				0.00954					0.174	0.0163		
20	2301-MP				0.015	0.022	0.036	0.015	0.044	0.063		0.069	0.093
24	2301-MP						0.05	0.05	0.05			0.05	
25	2301-MP		0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
27	2301-MP						0.01	0.01					
28	2301-MP		0.5		0.05	0.05				0.05	0.05	0.05	0.05
31	2301-MP			0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08		0.08
34	2301-MP		0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
45	2301-MP		0.025	0.001	0.00035	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001
46	2301-MP				0.009	0.01	0.006	0.002	0.006	0.008	0.02		
47	2301-MP			0.2	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
49	2301-MP		0.05	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02
51	2301-MP				0.1	0.1				0.1	0.1		
53	2301-MP		0.05	0.05	0.05	0.01	0.05	0.05	0.1	0.05	0.05	0.05	0.1
55	2301-MP				0.0025	0.0025	0.0005	0.0025	0.0005	0.0025	0.5	0.001	0.01
57	2301-MP		10	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
58	2301-MP		0.1	0.1	0.05	0.02	0.005	0.005	0.01	0.02	0.02	0.05	0.05
59	2301-MP		0.3	0.1	0.1	0.01	0.01	0.01	0.01	0.01		0.01	0.01
60	2301-MP		0.005	0.005	0.001	0.005	0.001	0.001	0.01	0.001	0.001	0.01	0.01
63	2301-MP						0.05	0.05					
76	2301-MP			0.1	0.1	0.1	0.05	0.05	0.1	0.1	0.1	0.1	0.1
77	2301-MP						0.1	0.1					
78	2301-MP						0.01	0.005					
82	2301-MP				0.2	0.05				0.05	0.05		
84	2301-MP		0.2	0.1	0.05	0.05	0.05	0.05	0.05	0.15	0.2	0.2	0.2
93	2301-MP		0.15	0.15	0.03	0.02	0.02	0.02	0.02	0.02	0.02	0.03	0.04
95	2301-MP		1.26	0.1	0.12	0.07	0.05	0.04	0.04	0.04	0.05	0.1	0.05
96	2301-MP				0.05	0.02				0.05	0.05		0.05
99	2301-MP		0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	
100	2301-MP		0.1	0.1	0.1	0.1	0.1	0.1	0.1			0.3	0.3
107	2301-MP		0.546	0.016	0.009	0.004	0.01	0.024	0.009	0.017		0.01	0.01
108	2301-MP												
109	2301-MP		0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
110	2301-MP				0.1	0.1				0.1	0.1	1.6	0.62
115	2301-MP				0.01	0.01				0.01	0.01		
119	2301-MP		0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05		
122	2301-MP		0.5	0.5	0.1	0.05	0.05	0.1	0.1	0.05	0.1	0.5	0.5
123	2301-MP		0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
128	2301-MP		0.5	0.05	0.01	0.01	0.01	0.005	0.005	0.005	0.005	0.005	0.005
60*	2301-MP		0.005	0.005	0.001	0.005	0.001	0.001	0.01	0.001	0.001	0.01	0.01

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder 2023 [EURL-PT-POP_2301-MP]

EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Power (2301-MP)

Methods Perfluoroalkylsulfonic acids (PFSAs) - Measurement Uncertainty [%]

LC	Sample		Perfluorobutane-sulfonic acid **(PFBS)**	Perfluoropentane-sulfonic acid **(PFPeS)**	Perfluorohexane-sulfonic acid **(PFHxS)**	Perfluoroheptane-sulfonic acid **(PFHpS)**	Linear Perfluorooctane-sulfonic acid **(L-PFOS)**	Sum of branched Perfluorooctane-sulfonic acids **(br-PFOS)**	Total-Perfluorooctane-sulfonic acids **(total PFOS)**	Perfluorononane-sulfonic acid **(PFNS)**	Perfluorodecane-sulfonic acid **(PFDS)**	Perfluoroundecane-sulfonic acid **(PFUnDS)**	Perfluorododecane-sulfonic acid **(PFDoDS)**	Perfluorotridecane-sulfonic acid **(PFTrDS)**
1	2301-MP				42		44	44	32					
2	2301-MP		30	30	30	40	30	30	30	40	40		40	
3	2301-MP		30	30	30	30			30	30	45	45	60	60
6	2301-MP													
8	2301-MP		14	9.9	5.7	8.7	14	12	14	12	18			
11	2301-MP		29	32	33	33	27			26	31		45	
11A	2301-MP		29	32	33	33	27			26	31		45	
14	2301-MP													
15	2301-MP		25.4		31.3	23.4	35.7				26			
17	2301-MP				50				50					
18	2301-MP													
19	2301-MP				7.97		7.61	9.43	12.1					
20	2301-MP													
24	2301-MP				30		30	30	30					
25	2301-MP				40		35	40	35					
27	2301-MP													
28	2301-MP		42		42	35	33	33	33					
31	2301-MP				35	35	35	35	35					
34	2301-MP													
45	2301-MP		20	20	20	20	15				20		20	
46	2301-MP		26	36	27	33	28	28	40					
47	2301-MP		54		32				22		26			
49	2301-MP		45	45	45	45	45	45	45	45	45	45	45	45
51	2301-MP		30	30	30	30	30	30	30		30			
53	2301-MP		35	20	30	25	30	30		30	20		20	
55	2301-MP		16		21	37	31	31	31		12			
57	2301-MP		50	50	50	50	50	50	50	50	50			
58	2301-MP		25	25	20	30	20	30	25	30	30		30	
59	2301-MP		37	37	37	37	37	37	37	37	37	37	37	37
60	2301-MP		40	40	30	40	30			40	40	40	40	40
63	2301-MP				15				16					
76	2301-MP		40		35		27	27			40			
77	2301-MP				10		10	10						
78	2301-MP				20		20							
82	2301-MP				88		88		88					
84	2301-MP		20		21		9.99		9.99					
93	2301-MP		6		14		10		10		21			
95	2301-MP													
96	2301-MP				18				19					
99	2301-MP		30	30	34	30			30	30	30	30	30	30
100	2301-MP		22.8		22.8	22.8	23	23	23		23			
107	2301-MP													
108	2301-MP													
109	2301-MP		44	44	44	44	44	44	44	44	44		44	
110	2301-MP													
115	2301-MP		40	40	28	40	28	28	20	40	40		40	
119	2301-MP		44		44	44	44							
122	2301-MP		26	7	34	6	26	26	26	8	33			
123	2301-MP													
128	2301-MP				30				30					
60*	2301-MP		40	40	30	40	30			40	40	40	40	40

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder 2023 [EURL-PT-POP_2301-MP]

EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Power (2301-MP)

Methods Perfluoroalkylsulfonic acids (PFSAs) - Limit of detection (LOQ) in µg/kg wet weight

LC	Sample		Perfluorobutane-sulfonic acid **(PFBS)**	Perfluoropentane-sulfonic acid **(PFPeS)**	Perfluorohexane-sulfonic acid **(PFHxS)**	Perfluoroheptane-sulfonic acid **(PFHpS)**	Linear Perfluorooctane-sulfonic acid **(L-PFOS)**	Sum of branched Perfluorooctane-sulfonic acids **(br-PFOS)**	Total-Perfluorooctane-sulfonic acids **(total PFOS)**	Perfluorononane-sulfonic acid **(PFNS)**	Perfluorodecane-sulfonic acid **(PFDS)**	Perfluoroundecane-sulfonic acid **(PFUnDS)**	Perfluorododecane-sulfonic acid **(PFDoDS)**	Perfluorotridecane-sulfonic acid **(PFTrDS)**
1	2301-MP				0.04		0.01	0.01						
2	2301-MP		0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	1		1	
3	2301-MP		0.05	0.05	0.05	0.05	0.05		0.05	0.05	0.1	0.1	0.1	0.1
6	2301-MP				0.05				0.05					
8	2301-MP		0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5			
11	2301-MP		0.05	0.05	0.05	0.05	0.05			0.05	0.05		0.05	
11A	2301-MP		0.05	0.05	0.05	0.05	0.05			0.05	0.05		0.05	
14	2301-MP		0.072	0.076	0.03	0.038	0.058	0.036		0.08	0.078			
15	2301-MP		0.01		0.01	0.01	0.01				0.01			
17	2301-MP				0.012				0.012					
18	2301-MP		0.05	0.05	0.05	0.05	0.05	0.05		0.05	0.1			
19	2301-MP		0.00838	0.0135		0.006				0.00486	0.00814	0.0107	0.00702	0.0091
20	2301-MP		0.01	0.01	0.023	0.14			0.014		0.035			
24	2301-MP				0.05		0.05	0.05	0.05					
25	2301-MP		0.01	0.01	0.01	0.01	0.01	0.02	0.03	0.01	0.01	0.01	0.01	0.01
27	2301-MP				0.01		0.01	0.01	0.01					
28	2301-MP		0.05			0.05								
31	2301-MP				0.08	0.08	0.08	0.08	0.08					
34	2301-MP		0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
45	2301-MP		0.0001	0.025	0.00035	0.00035	0.00035				0.00035		0.00035	
46	2301-MP		0.001	0.003	0.003	0.007	0.004	0.004	0.008					
47	2301-MP		0.05		0.05				0.05		0.05			
49	2301-MP		0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02
51	2301-MP		0.1	0.1		0.1					0.1			
53	2301-MP		0.01	0.01	0.01	0.1	0.05	0.05		0.01	0.01		0.05	
55	2301-MP		0.0005		0.01	0.0005	0.0025	0.0025	0.0025		0.001			
57	2301-MP		0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2			
58	2301-MP		0.01	0.01	0.005	0.01	0.005	0.025	0.01	0.01	0.05		0.1	
59	2301-MP		0.01		0.01	0.01	0.01	0.01	0.01	0.1	0.1		0.1	0.1
60	2301-MP		0.001	0.001	0.001	0.001	0.001		0.001	0.001	0.01	0.01	0.001	0.01
63	2301-MP				0.05				0.05					
76	2301-MP		0.1		0.05		0.05	0.05	0.05		0.1			
77	2301-MP				0.1		0.1	0.1						
78	2301-MP				0.01		0.01							
82	2301-MP		0.05	0.05		0.05				0.2	0.05			
84	2301-MP		0.0443		0.037		0.0366		0.0366					
93	2301-MP		0.04		0.04		0.03	0.03	0.03		0.05			
95	2301-MP		0.04	0.03	0.03	0.03	0.035			0.035	0.055	0.07	0.078	0.1
96	2301-MP		0.05	0.1		0.02				0.02	0.03	0.05	0.1	0.1
99	2301-MP		0.05	0.05	0.05	0.05			0.05	0.1	0.05	0.05	0.05	0.05
100	2301-MP		0.1		0.05	0.05	0.1	0.1	0.1	0.1	ERROR			
107	2301-MP		0.012	0.01	0.011	0.01	0.01	0.01	0.01	0.01	0.016			
108	2301-MP													
109	2301-MP		0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05		0.05	
110	2301-MP		0.1	0.1	0.1	0.1				0.1	0.1	0.1	0.1	0.1
115	2301-MP		0.01	0.01		0.01				0.01	0.01		0.01	
119	2301-MP		0.05		0.05		0.05							
122	2301-MP		0.05	0.1	0.05	0.1	0.05	0.05	0.05	0.1	0.1			
123	2301-MP		0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1		0.1	
128	2301-MP		0.005	0.01	0.005	0.005			0.005	0.005	0.005		0.005	
60*	2301-MP		0.001	0.001	0.001	0.001	0.001		0.001	0.001	0.01	0.01	0.001	0.01

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder 2023 [EURL-PT-POP_2301-MP]

EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Power (2301-MP)

Methods Sum of PFOS, PFOA, PFNA, PFHxS - Measurement Uncertainty [%]

LC	Sample	Sum of total-PFOS, PFOA, PFNA, PFHxS **(ub)**	Sum of total-PFOS, PFOA, PFNA, PFHxS **(lb)**
1	2301-MP	26	26
2	2301-MP	30	30
3	2301-MP	20	20
6	2301-MP		
8	2301-MP	14	14
11	2301-MP	16	
11A	2301-MP	15	
14	2301-MP		
15	2301-MP		
17	2301-MP	50	0.5
18	2301-MP		
19	2301-MP	17	17
20	2301-MP		
24	2301-MP	30	30
25	2301-MP	25	25
27	2301-MP	20	20
28	2301-MP	35	35
31	2301-MP		
34	2301-MP		
45	2301-MP	20	20
46	2301-MP	57	57
47	2301-MP	23	23
49	2301-MP	26	26
51	2301-MP	30	30
53	2301-MP	30	30
55	2301-MP		
57	2301-MP	50	50
58	2301-MP	25	25
59	2301-MP	37	37
60	2301-MP		
63	2301-MP	38	38
76	2301-MP	19	19
77	2301-MP	10	10
78	2301-MP	20	20
82	2301-MP	88	88
84	2301-MP	15.4	15.4
93	2301-MP	5	5
95	2301-MP		
96	2301-MP	11	11
99	2301-MP	39	39
100	2301-MP	21.2	21.2
107	2301-MP		
108	2301-MP		
109	2301-MP	24.9	24.9
110	2301-MP		
115	2301-MP	15	15
119	2301-MP	44	44
122	2301-MP	34	34
123	2301-MP		
128	2301-MP	50	50
60*	2301-MP		

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder 2023 [EURL-PT-POP_2301-MP]

EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Power (2301-MP)

Methods Other PFAS - Limit of detection (LOQ) in µg/kg wet weight

LC	Sample		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**
1	2301-MP								
2	2301-MP		0.50	0.30	0.30	0.30	0.30		
3	2301-MP				1.00	0.05	0.05		
6	2301-MP								
8	2301-MP		0.50	0.50	0.50	5.00	0.50		
11	2301-MP								
11A	2301-MP								
14	2301-MP			0.04		0.04	0.08		
15	2301-MP		0.01						
17	2301-MP								
19	2301-MP		0.03						
20	2301-MP								
24	2301-MP			0.05					
25	2301-MP		0.01	0.01	0.01	0.01	0.01		
27	2301-MP								
28	2301-MP				0.05				
31	2301-MP								
34	2301-MP		0.10	0.10	0.10				
45	2301-MP			0.00	0.03				
46	2301-MP								
47	2301-MP		0.05						
49	2301-MP		0.02	0.02	0.02	0.02	0.02	0.02	0.02
51	2301-MP		0.10		0.50				
53	2301-MP		0.01	0.05	1.00	0.50	0.50		
55	2301-MP			0.00					
57	2301-MP								
58	2301-MP			0.05	0.05				
59	2301-MP		0.01	0.10	0.10	0.01	0.01		
60	2301-MP		0.01						
76	2301-MP		0.10						
77	2301-MP								
78	2301-MP								
82	2301-MP				0.05	0.05	0.05		
84	2301-MP								
93	2301-MP		0.04						
95	2301-MP		0.07	0.01	0.03	0.03	0.05		0.80
96	2301-MP				0.20	0.05	0.05		
99	2301-MP			0.05		0.05	0.05		
100	2301-MP								
107	2301-MP		0.01	0.01	0.05	0.01	0.01	0.01	
108	2301-MP								
109	2301-MP			0.05	0.05	0.05	0.05		
110	2301-MP								
115	2301-MP		0.05						
119	2301-MP				0.10				
122	2301-MP								
123	2301-MP								
128	2301-MP								
60*	2301-MP		0.01						

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Milk Powder 2023 [EURL-PT-POP_2301-MP]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Milk Power (2301-MP)
Methods Other PFAS - Measurement Uncertainty MU [%]

LC	Sample		Perfluorooctane sulphonamide **FOSA**	2,2,3-Trifluoro-3-[1,1,2,2,3,3,3-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**
1	2301-MP								
2	2301-MP		30	30	30	30	30		
3	2301-MP				30	30	50		
6	2301-MP								
8	2301-MP		8	30	30	30	30		
11	2301-MP								
11A	2301-MP								
14	2301-MP								
15	2301-MP		18.2						
17	2301-MP								
19	2301-MP			5					
20	2301-MP								
24	2301-MP								
25	2301-MP			35					
27	2301-MP								
28	2301-MP				39				
31	2301-MP								
34	2301-MP								
45	2301-MP			20	20				
46	2301-MP								
47	2301-MP		26						
49	2301-MP		45	45	45	45	45	45	45
51	2301-MP		30	30	30				
53	2301-MP		20	20	20	15	15		
55	2301-MP			35					
57	2301-MP								
58	2301-MP			50	25				
59	2301-MP		37	37	37	37	37	37	37
60	2301-MP								
76	2301-MP								
77	2301-MP								
78	2301-MP								
82	2301-MP			88					
84	2301-MP								
93	2301-MP		20						
95	2301-MP								
96	2301-MP								
99	2301-MP			50		30	30		
100	2301-MP								
107	2301-MP								
108	2301-MP								
109	2301-MP			44	44	44	44		
110	2301-MP								
115	2301-MP		28						
119	2301-MP								
122	2301-MP								
123	2301-MP								
128	2301-MP								
60*	2301-MP								