EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compound feed 2023

EURL-PT-POP_2302-CF

FEED

Report

PFAS

(Report Version 1.0)

21 June 2024

EURL for halogenated POPs in Feed and Food
c/o State Institute for Chemical and Veterinary Analysis Freiburg
Bissierstraße 5
79114 Freiburg
D-Germany
contact@eurl-pops.eu
+49 761 8855 500
eurl-pops.eu

Summary

Test sample	**FEED:** Compound feed [2302-CF]
Analytes of interest **Optional** for NRLs:	**Main PFAS** (L-PFOS, PFOA, PFNA, PFHxS, Sum of L-PFOS, PFOA, PFNA, PFHxS) **Other PFAS** (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 feed business operators
Statistical evaluation	ISO 13528:2022 [1], IUPAC Protocol [2]
Report of final results	21 June 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 **PFAS** in **compound feed** was organized by European Union Reference Laboratory (EURL) for halogenated persistent organic pollutants (POPs) in Feed and Food to be performed between September and November 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 **compound feed**.

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 feed business operators were invited to participate in this proficiency test.

The evaluated results were discussed by representatives of European Commission, NRLs and the EURL at the EURL/NRL workshop on 29 and 30 November 2023.

1.1. Samples and coding

The test sample was prepared from commercially available feed, naturally contaminated with PCDD/Fs, PCBs and PFASs and fortified with analytes of interest using technical mixtures of PBDEs and HBCDDs. Each participant received about **90 g** of the test sample in a HDPE bottle.

Compound feed	**Sample no. 2302-CF-xxx**

1.2. Analytes of interest

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

- 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

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

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

1.4. Coding of laboratories and confidentiality

The identity of participating laboratories will be kept confidential and will not be revealed to other participants.
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, relative to a feed with a moisture content of 12 %.**

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

117 laboratories registered for this proficiency test and 40 reported results for at least one PFAS parameter. One laboratory reported 2 sets of results.

Table 1: Participating laboratories

Participating laboratories	Region	No. of participants in this PT	No. of participants reporting results for PFAS
National Reference Laboratories	European Union	27	15
	Europe	3	1
	Americas	1	-
	Oceania	1	1
Official Laboratories	European Union	56	18
	Other European Countries	-	-
	Americas	3	-
	Oceania	-	-
Commercial Laboratories	European Union	19	4
	Other European Countries	1	-
	Americas	5	1
	Oceania	1	-
	Total	**117**	**40**

2.1. Number of reported results

Table 2: Reported results for individual PFCAs and PFSAs for compound feed (2302-CF) of all laboratories

Analyte	Abbreviation	2302-CF
Perfluorobutanoic acid	(PFBA)	24
Perfluoropentanoic acid	(PFPeA)	26
Perfluorohexanoic acid	(PFHxA)	31
Perfluoroheptanoic acid	(PFHpA)	33
Perfluorooctanoic acid	(PFOA)	41
Perfluorononanoic acid	(PFNA)	41
Perfluorodecanoic acid	(PFDA)	32
Perfluoroundecanoic acid	(PFUnDA)	32
Perfluorododecanoic acid	(PFDoDA)	30
Perfluorotridecanoic acid	(PFTrDA)	27
Perfluorotetradecanoic acid	(PFTeDA)	26
Perfluorobutanesulfonic acid	(PFBS)	30
Perfluoropentanesulfonic acid	(PFPeS)	27
Perfluorohexanesulfonic acid	(PFHxS)	39
Perfluoroheptanesulfonic acid	(PFHpS)	28
Linear Perfluorooctanesulfonic acid	(L-PFOS)	33
Perfluorononanesulfonic acid	(PFNS)	26
Perfluorodecanesulfonic acid	(PFDS)	27
Perfluoroundecanesulfonic acid	(PFUnDS)	11
Perfluorododecanesulfonic acid	(PFDoDS)	18
Perfluorotridecanesulfonic acid	(PFTrDS)	10

Table 3: Reported results for PFAS sum parameters for compound feed (2302-CF) of all laboratories

Analyte	Abbreviation	2302-CF
Sum of branched perfluorooctanesulfonic acids	(br-PFOS)	24
Sum of branched & linear perfluorooctanesulfonic acids	(Total-PFOS)	37
Sum of Total-PFOS, PFOA, PFNA, PFHxS (upper bound)	-	40
Sum of Total-PFOS, PFOA, PFNA, PFHxS (lower bound)	-	41

Table 4: Reported results for other PFAS for compound feed (2302-CF) of all laboratories

Analyte	Abbreviation	2302-CF
Perfluorooctane sulphonamide	(FOSA)	12
2,2,3-Trifluoro-3-[1,1,2,2,3,3-hexafluor-3-(trifluoromethoxy)propoxy]-propionic acid	(DONA)	16
2,3,3,3-tetrafluoro-2-(heptafluoropropoxy)-propanoic acid	(GenX)	16
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)	12
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)	3

2.2. Accreditation

Table 5: Reported accreditation according to ISO/IEC 17025 by participants for PFAS

Compound feed	PFAS
Accreditation	18
No accreditation	18

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. High resolution mass spectrometry (Orbitrap HRMS) was also applied 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 2302-CF were analyzed in duplicate for individual PFAS. The test for sufficient homogeneity was performed for the individual substances and sum parameters. The test materials showed sufficient homogeneity for PFAS in this proficiency test. The stability check of the analytes of interest applying room temperature storage was performed according to DIN ISO ISO 13528:2022 [1]. The test material showed sufficient stability for PFAS.

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 DIN 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 is 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 individual PFAS compounds, and sum parameters (including limits of quantification (LOQs)), if possible. Additionally the median of all values was calculated.

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

Assigned values were calculated for PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA and PFDA, for L-PFOS and total PFOS (sum of branched and linear Perfluorooctanesulfonic acids) and for the sum parameter sum of total-PFOS, PFOA, PFNA, PFHxS in the test sample "compound feed" (2302-CF), including limits of quantification (LOQs). Assigned values could not be calculated for all other perfluoroalkylcarboxylic and perfluoroalkylsulfonic acids and other PFAS substances due to the limited number of reported results above the LOQ, but median values of participants' results are given in this report.

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 the results of the participants. Additionally the results of all participants reporting results and the results of participants having accreditation according to ISO/IEC 17025 were compared for PFOA, PFNA, L-PFOS, total-PFOS and for the sum parameter sum of total-PFOS, PFOA, PFNA, PFHxS. 18 of 40 reporting laboratories were accredited according to ISO/IEC 17025 for PFAS in feed. No significant differences (3 %) between the assigned values calculated for both data sets for PFAS were observed for most of the analytes (Table 6).

Table 6: Comparison of assigned values for 2302-CF for all participants and participants with reported accreditation according to ISO/IEC 17025 for PFAS

Sum parameters	Assigned value All participants	Assigned value ISO/IEC 17025 accreditation	Deviation
	µg/kg product (12% moisture content)		%
PFOA	1.84	1.81	<2
PFNA	0.580	0.570	<2
L-PFOS	0.747	0.724	3
Total PFOS	0.802	0.799	<1
Sum of total PFOS, PFOA, PFNA, PFHxS (ub)	3.35	3.37	<1
Sum of total PFOS, PFOA, PFNA, PFHxS (lb)	3.21	3.28	-2

4.1. Assigned values of individual substances

The assigned values for the test sample 2302-CF were calculated as consensus of participants' results for individual PFAS, taking into account the calculation criteria described above (Table 7 and 8 ; tabular summary see annex 1; Figure 1 and 2).

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

Compound feed (2302-CF)	Assigned value PFCAs µg/kg product (12% moisture content)
PFBA	12.2
PFPeA	19.3
PFHxA	3.78
PFHpA	1.03
PFOA	1.84
PFNA	0.580
PFDA	1.22

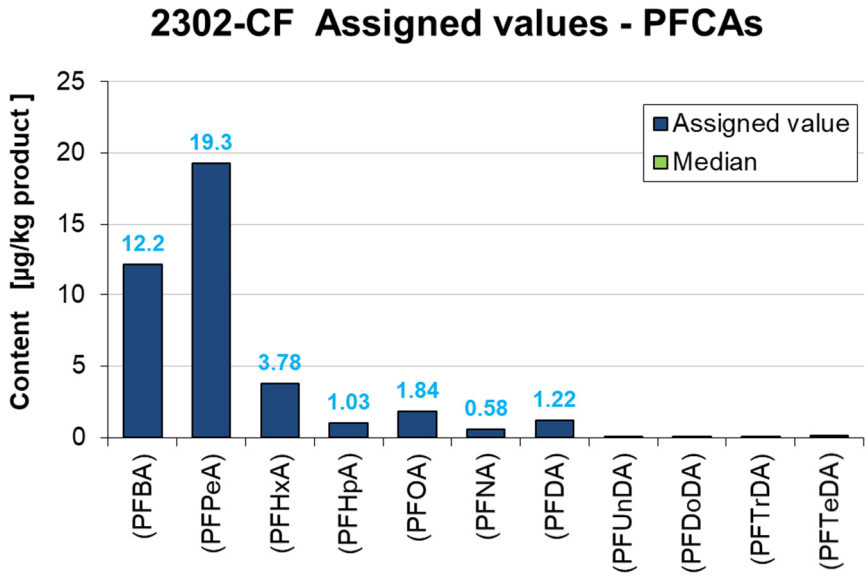

Figure 1: Assigned values (blue) and median values (green) for PFCAs individual substances for compound feed (2302-CF) [µg/kg product (12% moisture content)]

Table 8: Assigned values for Perfluoroalkylsulfonic acids (rounded to three significant figures)

Compound feed (2302-CF)	Assigned value PFSAs µg/kg product (12% moisture content)
L-PFOS	0.747

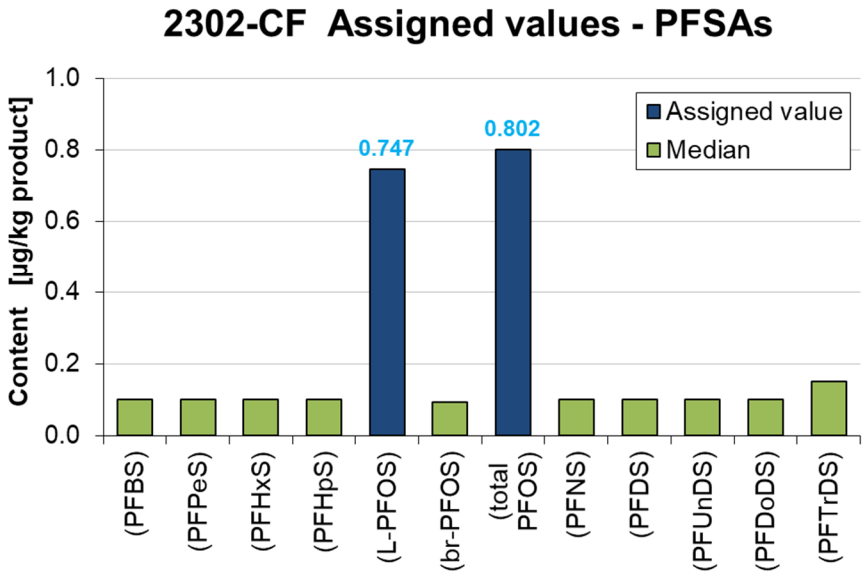

Figure 2: Assigned values (blue) and median values (green) for PFSAs individual substances for compound feed (2302-CF) [µg/kg product (12% moisture content)]

4.2. Assigned values of sum parameter

The assigned values for the test sample 2302-CF were calculated as consensus of participants' results for sum parameters, taking into account the calculation criteria described above (Table 9 ; tabular summary see annex 1; Figure 2 and 3).

Table 9: Assigned values for sum parameters (rounded to three significant figures)

Compound feed (2302-CF)	Assigned value µg/kg product (12% moisture content)
Sum of branched & linear perfluorooctanesulfonic acids (Total-PFOS)	0.802
Sum of Total PFOS, PFOA, PFNA, PFHxS (ub)	3.35
Sum of Total PFOS, PFOA, PFNA, PFHxS (lb)	3.21

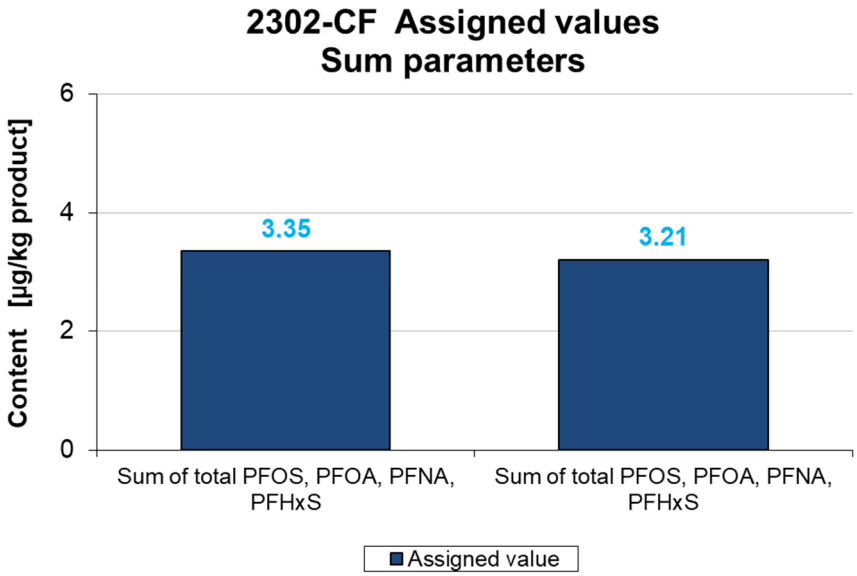

Figure 3: Assigned values for sum parameters of total-PFOS, PFOA, PFNA and PFHxS for compound feed (2302-CF) [µg/kg product (12% moisture content)]

5. Scoring of results – Z-scores

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)*

5.1. Participants' z-scores of individual substances

Z-scores for individual substances were calculated and ranged between ± 2 for 78 - 94% of all participants (Table 10 and 11; tabular summary see annex 3; Figure 4 and 5).

Table 10: Distribution of participants' z-scores for PFCAs for compound feed (2302-CF)

Percentage of participants' results PFCAs	|z-score| ≤ 2	2 < |z-score| < 3	|z-score| ≥ 3
PFBA	78%	-	22%
PFPeA	81%	4%	15%
PFHxA	81%	6%	13%
PFHpA	88%	6%	6%
PFOA	80%	10%	10%
PFNA	90%	5%	5%
PFDA	94%	-	6%

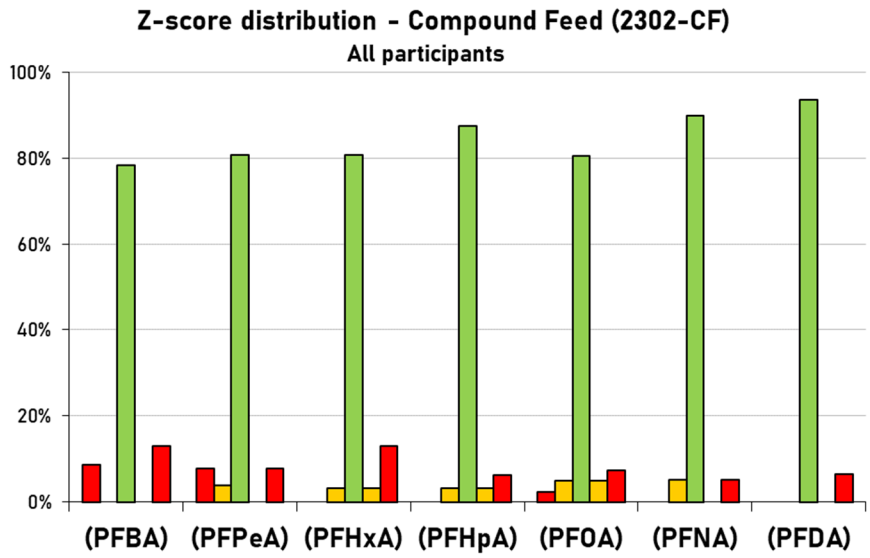

Figure 4: Distribution of participants' z-scores for individual PFCAs for compound feed (2302-CF) [Green bars: -2 ≤ z-score ≤ 2, orange bars: -3 < z-score < -2, 2 < z-score < 3, red bars: z-score ≤ -3, z-score ≥ 3]

Table 11: Distribution of participants' z-scores for PFSAs for compound feed (2302-CF)

Percentage of participants' results PFSAs	|z-score| ≤ 2	2 <|z-score| < 3	|z-score| ≥ 3
L-PFOS	81%	3%	16%

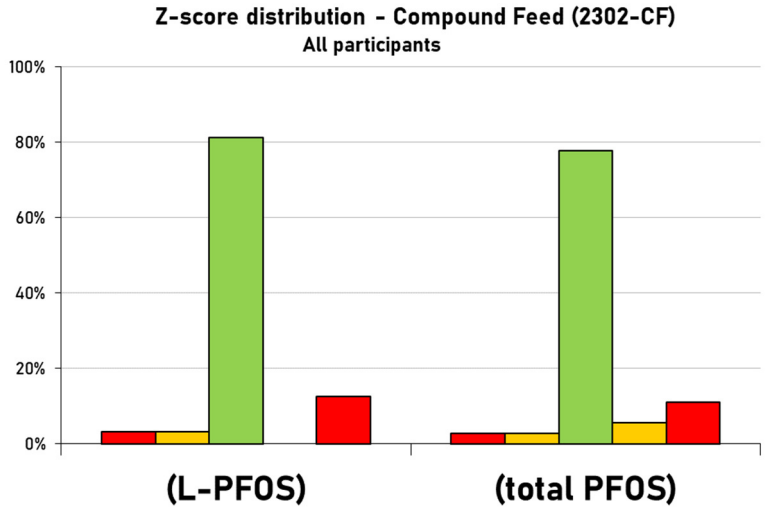

Figure 5: Distribution of participants' z-scores for individual PFSAs and sum parameter total-PFOS for compound feed (2302-CF) [Green bars: -2 ≤ z-score ≤ 2, orange bars: -3 < z-score < -2, 2 < z-score < 3, red bars: z-score ≤ -3, z-score ≥ 3]

5.2. Participants' z-scores of sum parameters

Z-scores for sum parameters were calculated and ranged between ± 2 for 78 % of all participants (Table 12; tabular summary see annex 3; Figure 5 and 6).

Table 12: Distribution of participants' z-scores for sum parameters for compound feed (2302-CF)

Percentage of participants' results Sum parameters	|z-score| ≤ 2	2 <|z-score| < 3	|z-score| ≥ 3
Total-PFOS	78%	8%	14%
Sum of total-PFOS, PFOA, PFNA, PFHxS (ub)	77%	8%	15%
Sum of total-PFOS, PFOA, PFNA, PFHxS (lb)	78%	5%	17%

6. Participants' feedback

A questionnaire for feedback from participants of this EURL proficiency test was available as online survey between 22 November 2023 and 19 January 2024. 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, 4 laboratories replied to this survey.

Table 13: Participating laboratories in the feedback survey

Type of laboratory	Answers
National Reference Laboratory (NRL)	2
Official Laboratory (OFL)	1
Commercial laboratory	1
Other (e.g. research and development)	0
No Answer	0

General aspect

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".

Did the proficiency test meet expectations?

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?	

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

Choice of matrix 

Level of contamination 

Comments from participants:

- Information on ingredients would be nice to have (e.g. exact list of ingredients with quantity labeling); *Comment EURL: Natural contamination of the test material with all analytes of interest is preferred, therefore different materials have to be mixed and no information about the exact list of ingredients of the test material is possible*
- More sample material, if more than one group of analytes have to be analyzed (e.g. PCDD/Fs + PCBs and PFAS); *Comment EURL: The quantity of the sample is listed in the announcement; if more material is required, this can be ordered directly at the time of registration or later*
- Annexes linked as individual pdf files in the document e.g. like in previous PT reports. *Comment EURL: As too many participants had problems opening the pdf files linked in the document the structure of the report was changed*

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 PFAS results for the PT test sample compound feed (2302-CF) is 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.

10. Annex

Compound feed – 2302-CF	
Annex 1	Assigned values – PFCAs, PFSAs and sum parameters
Annex 2	Participants' results – Tables – PFCAs, PFSAs, sum parameters and other PFAS
Annex 3	Participants' z-scores – Tables – PFCAs, PFSAs and sum parameters
Annex 4	Participants' z-scores – Charts – PFCAs, PFSAs and sum parameters
Annex 5	Test for sufficient homogeneity – PFAS
Annex 6	Overview participants' methods – Weighed sample, internal and recovery standards and comments
Annex 7	Overview participants' methods – Extractions, clean-up and detection
Annex 8	Overview participants' methods – Measurement uncertainty and Limit of Quantification

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

Coordinator: Theresa Zwickel
(Senior scientist at EURL POPs)
Phone: +49 761 8855 500 E-Mail: pt@eurl-pops.eu

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EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food
21 June 2024

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

Test sample - Compound Feed (2302-CF)

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 Compound Feed 2023 [EURL-PT-POP_2302-CF]

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

Compound Feed (2302-CF)

Perfluoroalkylcarboxylic acids (PFCAs) - Assigned values

Analyte		Result µg/kg product (12% moisture content)	**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)		12.2	2.9	0.87	18	11.9
Perfluoropentanoic acid	(PFPeA)		19.3	4.2	1.1	21	19.1
Perfluorohexanoic acid	(PFHxA)		3.78	1.0	0.25	26	3.8
Perfluoroheptanoic acid	(PFHpA)		1.03	0.23	0.052	30	1.05
Perfluorooctanoic acid	(PFOA)		1.84	0.32	0.067	36	1.88
Perfluorononanoic acid	(PFNA)		0.580	0.13	0.027	39	0.600
Perfluorodecanoic acid	(PFDA)		1.22	0.21	0.048	29	1.23
Perfluoroundecanoic acid	(PFUnDA)						0.0765
Perfluorododecanoic acid	(PFDoDA)						0.0950
Perfluorotridecanoic acid	(PFTrDA)						0.100
Perfluorotetradecanoic acid	(PFTeDA)						0.126

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]

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

Compound Feed (2302-CF)

Perfluoroalkylsulfonic acids (PFSAs) - Assigned values

Analyte		Result µg/kg product (12% moisture content)	**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)						0.100
Perfluoropentanesulfonic acid	(PFPeS)						0.100
Perfluorohexanesulfonic acid	(PFHxS)						0.100
Perfluoroheptanesulfonic acid	(PFHpS)						0.100
Linear Perfluorooctanesulfonic acid	(L-PFOS)		0.747	0.14	0.034	27	0.754
Sum of branched Perfluorooctanesulfonic acids	(br-PFOS)						0.093
Sum of branched and linear perfluorooctanesulfonic acids	(total PFOS)		0.802	0.14	0.033	30	0.820
Perfluorononanesulfonic acid	(PFNS)						0.100
Perfluorodecanesulfonic acid	(PFDS)						0.100
Perfluoroundecane sulfonic acid	(PFUnDS)						0.100
Perfluorododecane sulfonic acid	(PFDoDS)						0.100
Perfluorotridecane sulfonic acid	(PFTrDS)						0.151

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]

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

Compound Feed (2302-CF)

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

Analyte		Result µg/kg product (12% moisture content)	**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)		3.35	0.61	0.13	32	3.60
Sum of total PFOS, PFOA, PFNA, PFHxS	(lb)		3.21	0.58	0.13	33	3.38

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]

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

Compound Feed (2302-CF)

Other PFASs - Assigned values

Analyte		Result µg/kg product (12% moisture content)	**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)						0.135
2,2,3-Trifluoro-3-[1,1,2,2,3,3-hexafluor-3-(trifluoromethoxy)propoxy]-propionic acid	(DONA)						0.0750
2,3,3,3-tetrafluoro-2-(heptafluoropropoxy)-propanoic acid	(GenX)						0.350
Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonate	(major component of F-53B)						0.0750
Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonate	(minor component of F-53B)						0.0717
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)						0.0650
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)						0.100

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food
21 June 2024

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

Test sample - Compound Feed (2302-CF)

* 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 Compound Feed 2023 [EURL-PT-POP_2302-CF]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Compound Feed (2302-CF)
Perfluoroalkylcarboxylic acids (PFCAs) - Results

LC	Sample	Result µg/kg product (12% moisture content)	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)
4	2302-CF			5.67	9.37	0.999	1.57	0.654		< 0.08	< 0.045	< 0.044	< 0.055
5	2302-CF		14.3	23.1	4.41	1.13	1.91	0.657	1.21	0.048	0.023	0.027	0.025
8	2302-CF		12.1	19.8	3.44	1.06	1.68	0.418	1.1	< 0.239		< 0.239	< 0.239
10	2302-CF					0.93	1.7	0.55	1.4	0.085	0.025	0.047	< 0.1
11B	2302-CF		0.762	1.15	3.24	1.22	4.05	0.733	2.03	< 0.381	< 0.381	< 0.381	< 0.381
12	2302-CF						1.5	0.42					
14	2302-CF				4.83	1.15	2.13	0.757	1.41	< 0.02	< 0.02	< 0.05	
18	2302-CF		7.86	18.1	3	0.921	1.58	0.501	1.13	0.067	< 0.05	< 0.151	< 0.151
22	2302-CF		< 0.1	16.5	2.3	1.1	2.3	0.6	1.3	< 0.1	< 0.1	< 0.1	< 0.1
27	2302-CF					1.46	1.89	0.783	1	0.0803			
28	2302-CF		9.37	13.5	3.12	0.789	1.51	0.447	0.977	< 0.288	< 0.288	< 0.288	< 0.288
30	2302-CF		10.7	21.5	4.86	1.31	2.14	0.626	1.16	0.06	< 0.03	0.03	< 0.03
36	2302-CF						1.88	0.64					
39	2302-CF		11	21	3.7	1.2	1.9	0.65	1.3	< 0.5	< 0.5	< 1	< 1
40	2302-CF						0.966	0.334					
41	2302-CF		15.3	26.7	5.03	1.15	2.06	0.629	1.38	0.052	< 0.05	< 0.05	< 0.05
43	2302-CF		430	62	5	0.76	2.2	< 0.6	1.1	< 0.6	< 0.6	< 0.6	< 0.6
47	2302-CF		16	12.9	3.84	0.643	1.84	0.628	1.3	0.253	0.213	0.298	0.368
53	2302-CF			23.2	2.71	1.83	2.81	1.15	1.96	< 1	< 0.2	< 2	< 2
55	2302-CF		25.4	24.3	5.15	1.2	2.07	0.66	1.28	0.073	< 0.09	0.031	< 0.05
59	2302-CF		11.6	15.3	3.34	0.9	1.61	0.52	1.09	0.053	< 0.32	< 0.27	< 0.24
60	2302-CF				7.22	0.781	1.88	0.651	1.52				
64	2302-CF						7.3	2.28					
67	2302-CF						1.28	0.48					
78	2302-CF				2.54	1.67	2.12	0.73	1.46	0.07	0.02		
80	2302-CF		0.291	9.44	1.81	0.596	0.881	0.379	0.871	0.068	0.024	0.021	0.044
83	2302-CF				2.92	1	1.62	0.54	1.1	0.07	0.021		
84	2302-CF				2.37	0.75	1.45	0.516	1.01	0.051	0.022	< 0.04	< 0.04
87	2302-CF		14.7	14.8	3.5	1.35	1.81	0.64	1.65	< 0.25	< 0.25	< 0.25	< 0.25
88	2302-CF						0.63	0.3					
91	2302-CF		9.9	17	4.1	0.98	2.1	0.79	1.5	0.062	0.023		
92	2302-CF		10.9	18.4	3.5	0.99	1.46	0.53	1.04	0.05	< 0.05		
93	2302-CF		25	44.4	10.6	< 0.1	4.26	< 0.5	< 0.5	< 1	< 1.5	< 1.5	< 1.5
99	2302-CF		15.8	20.6	4.36	1.05	2.04	0.558	1.24	0.0716	< 0.0647	< 0.0262	< 0.0372
107	2302-CF		9.91	15.8	3.23	0.78	1.28	0.4	0.89	< 0.05	< 0.1	< 0.5	< 0.5
108	2302-CF		15	24	10	1.4	2.6	0.79	1.4	0.059	< 0.1	0.074	< 0.1
109	2302-CF		13.2	21.1	4.4	1.05	1.89	0.544	1.22	< 0.1	< 0.1	< 0.1	< 0.1
113	2302-CF						2	0.76					
114	2302-CF		8.4	17.2	4.17	1.16	1.78	0.54	1.14	< 0.1	< 0.1	< 0.3	< 0.3
115	2302-CF						2.02	0.515					
10A	2302-CF		13	22	5.5	1.1	1.8	0.55	1.3	< 0.1	< 0.1	< 0.1	< 0.1

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Compound Feed (2302-CF)
Perfluoroalkylsulfonic acids (PFSAs) - Results

LC	Sample	Result µg/kg product (12% moisture content)	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)
4	2302-CF		< 0.138		< 0.167		0.937				< 0.51			
5	2302-CF		< 0.05	0.053	< 0.05	< 0.05	0.744		0.82	< 0.2	< 0.2			
8	2302-CF		< 0.239	< 0.239	< 0.239	< 0.239	0.583		0.583	< 0.239	< 0.239	< 0.478	< 0.478	< 0.717
10	2302-CF		< 0.1	< 0.1	< 0.1	< 0.1	0.84	0.11	0.95	< 0.1	< 0.1		< 0.1	
11B	2302-CF		< 0.381	< 0.381	< 0.381	< 0.381			0.628	< 0.381	< 0.381	< 0.381	< 0.381	< 0.381
12	2302-CF				0.88				0.93					
14	2302-CF		< 0.02	< 0.05	0.061	< 0.02	0.803	0.12	0.921	< 0.05	< 0.05		< 0.1	
18	2302-CF		< 0.094	< 0.047	< 0.095	< 0.096	0.685	0.051	0.736	< 0.019	< 0.019	< 0.02	< 0.049	< 0.049
22	2302-CF		< 0.1	< 0.1	< 0.1	< 0.1	0.8		0.8	< 0.1	< 0.1		< 0.1	
27	2302-CF						0.615							
28	2302-CF		< 0.962	< 0.288	< 0.288	< 0.288	0.584	0.087	0.633	< 0.288	< 0.288		< 0.962	
30	2302-CF		< 0.03	< 0.03	< 0.03	< 0.03	0.754	0.073	0.827	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03
36	2302-CF				< 0.097				0.73					
39	2302-CF		< 0.5	< 0.5	< 0.5	< 0.5	< 1	< 1	< 1	< 0.5	< 0.5		< 1	
40	2302-CF				0.0105		0.463	0.0395	0.503					
41	2302-CF		< 0.025	< 0.025	< 0.025	< 0.025	0.829	0.036	0.891	< 0.025	< 0.025	< 0.025	< 0.025	< 0.025
43	2302-CF		< 0.6	< 0.6	< 0.6	< 0.6	1.22	< 0.6	1.26	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6
47	2302-CF		0.168	0.163	0.103	3.99			3.48	< 0.1	0.16			
53	2302-CF		0.06	< 0.05	< 0.1	< 0.2	1.23	0.06	1.29	< 0.05	< 0.05	< 0.2	< 2	< 2
55	2302-CF		< 0.02	< 0.02	< 0.02	< 0.07	0.88		0.89	< 0.04	< 0.05	< 0.05	< 0.05	< 0.1
59	2302-CF		< 0.06	< 0.2	< 0.08	< 0.25	0.66	0.09	0.75	< 0.1	< 0.24		< 0.2	
60	2302-CF						0.778	< 0.2	0.83					
64	2302-CF				1.05				8.29					
67	2302-CF				0.12		0.61							
78	2302-CF		< 0.004		0.09		0.92	0.08	1					
80	2302-CF		< 0.05	0.266	0.231	0.249	0.242	0.083	0.325	< 0.02	0.216			
83	2302-CF		< 0.02	< 0.05	< 0.015	< 0.07	0.69	0.054	0.747					
84	2302-CF				< 0.02	< 0.02	0.653	0.113	0.766					
87	2302-CF		< 0.25	< 0.25	< 0.25	< 0.25			0.91	< 0.25	< 0.25			
88	2302-CF				< 0.2		0.37	< 0.2	0.37					
91	2302-CF		< 0.05	< 0.05	< 0.05	< 0.05	0.8	< 0.05	0.8	< 0.05	< 0.1			
92	2302-CF		< 0.05		< 0.05				0.69					
93	2302-CF		< 1	< 1	< 0.1	< 0.1	1.62		1.62	< 0.1	< 0.1			
99	2302-CF		< 0.136	< 0.315	< 0.0815	< 0.18	0.708	< 0.0968	0.708	< 0.0524	< 0.0414	< 0.0894		< 0.201
107	2302-CF		< 0.05	< 0.1	< 0.05	< 0.1	0.49	< 0.05	0.49	< 0.1	< 0.1			
108	2302-CF		< 0.1	< 0.1	< 0.1	< 0.1	0.88	< 0.1	0.88	< 0.1	< 0.1	< 0.1	< 0.1	
109	2302-CF		< 0.1	< 0.1	< 0.1	< 0.1	0.745			< 0.1	< 0.1		< 0.1	
113	2302-CF				0.11		0.93	< 0.1	0.92					
114	2302-CF		< 0.1	< 0.1	< 0.1	< 0.1	0.698	< 0.1	0.707	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
115	2302-CF				< 0.1				0.803					
10A	2302-CF		< 0.1	< 0.1	< 0.1	< 0.1	1.2	< 0.1	1.2	< 0.1	< 0.1		< 0.1	

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Compound Feed (2302-CF)
Sum of PFOS, PFOA, PFNA, PFHxS - Results

LC	Sample	Results µg/kg product (12% moisture content)	Sum of L-PFOS, PFOA, PFNA, PFHxS **(ub)**	Sum of L-PFOS, PFOA, PFNA, PFHxS **(lb)**
4	2302-CF		20.2	19.2
5	2302-CF		3.44	3.39
8	2302-CF		2.92	2.68
10	2302-CF		3.3	3.2
11B	2302-CF		5.79	5.41
12	2302-CF		3.73	3.73
14	2302-CF		3.87	3.87
18	2302-CF		2.91	2.81
22	2302-CF		3.8	3.7
27	2302-CF			
28	2302-CF		2.88	2.59
30	2302-CF		3.63	3.6
36	2302-CF			3.25
39	2302-CF		4.1	2.6
40	2302-CF		1.81	1.81
41	2302-CF		3.6	3.58
43	2302-CF		4.65	3.45
47	2302-CF		6.04	6.04
53	2302-CF		6.35	6.25
55	2302-CF		3.64	3.62
59	2302-CF		2.96	2.88
60	2302-CF		3.56	3.65
64	2302-CF		18.9	18.9
67	2302-CF		2.49	2.49
78	2302-CF		3.95	3.95
80	2302-CF		1.82	1.82
83	2302-CF		2.93	2.91
84	2302-CF		2.73	2.73
87	2302-CF		3.61	3.36
88	2302-CF		1.5	1.31
91	2302-CF		3.7	3.7
92	2302-CF		2.73	2.68
93	2302-CF		6.48	5.88
99	2302-CF		3.49	3.31
107	2302-CF		2.22	2.17
108	2302-CF		4.37	4.27
109	2302-CF		3.28	3.18
113	2302-CF		3.8	3.8
114	2302-CF		3.12	3.03
115	2302-CF		3.44	3.34
10A	2302-CF		3.7	3.6
4*	2302-CF		3.33	3.16

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]
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Compound Feed (2302-CF)
Other PFAS - Results

LC	Sample	Result µg/kg product (12% moisture content)	Perfluorooctane sulphonamide **FOSA**	2,2,3-Trifluoro-3-[1,1,2,2,3,3-hexafluor-3-(trifluoromethoxy)propoxy]-propionic acid **DONA**	2,3,3,3-tetrafluoro-2-(heptafluoropropoxy)-propanoic acid **GenX**	Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonate **(major component of F-53B)**	Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonate **(minor component of F-53B)**	1-Propanaminium, N,N-dimethyl-N-oxide-3-[[(3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluorooctyl)sulfonyl]amino]-, hydroxide **Capstone A**	1-Propanaminium, N-(carboxymethyl)-N,N-dimethyl-3-[[(3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluorooctyl)sulfonyl]amino]-, hydroxide **Capstone B**
4	2302-CF								
5	2302-CF			< 0.05	< 0.05	< 0.05	< 0.05		
8	2302-CF			< 0.239		< 0.239	< 0.239		
10	2302-CF		< 0.05	< 0.2	< 0.5	< 0.2	< 0.2		
11B	2302-CF			< 0.571	< 0.571	0.533	1.07		
12	2302-CF								
14	2302-CF								
18	2302-CF					< 0.047	< 0.048		
22	2302-CF								
27	2302-CF								
28	2302-CF		< 0.288						
30	2302-CF		0.044	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03
36	2302-CF								
39	2302-CF		< 0.5	< 0.05	< 0.05	< 0.05	< 0.05		
40	2302-CF								
41	2302-CF		< 0.2	< 0.05	< 0.2	< 0.05	< 0.05		
43	2302-CF		< 0.6	< 0.6	< 0.6				
47	2302-CF								
53	2302-CF		< 2	< 0.1	< 2	< 0.1	< 0.1	< 0.1	< 0.1
55	2302-CF		0.05	< 0.025	< 0.043	< 0.039	< 0.034		< 0.56
59	2302-CF			< 0.2	< 0.1				
60	2302-CF								
64	2302-CF								
67	2302-CF								
78	2302-CF								
80	2302-CF		0.047	0.011	0.886	< 0.1			
83	2302-CF								
84	2302-CF								
87	2302-CF								
88	2302-CF								
91	2302-CF			< 0.05	< 0.1	< 0.05			
92	2302-CF				< 0.05				
93	2302-CF								
99	2302-CF		< 0.172	< 0.0422	< 3.34	< 0.16	< 0.0933		
107	2302-CF								
108	2302-CF		0.098	< 0.1	< 1				
109	2302-CF								
113	2302-CF								
114	2302-CF		< 0.02	< 0.1	< 0.5	< 0.5	< 0.5		
115	2302-CF								
10A	2302-CF								

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

Test sample - Compound Feed (2302-CF)

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 Compound Feed 2023 [EURL-PT-POP_2302-CF]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Compound Feed (2302-CF)
Perfluoroalkylcarboxylic acids (PFCAs) - Z-scores

LC	Sample	Z-score [σ_p = 20 %]	Perfluorobutanoic acid **(PFBA)**	Perfluoropentanoic acid **(PFPeA)**	Perfluorohexanoic acid **(PFHxA)**	Perfluoroheptanoic acid **(PFHpA)**	Perfluorooctanoic acid **(PFOA)**	Perfluorononanoic acid **(PFNA)**	Perfluorodecanoic acid **(PFDA)**
4	2302-CF			-3.5	7.4	-0.2	-0.7	0.6	
5	2302-CF		0.9	1.0	0.8	0.5	0.2	0.7	0.0
8	2302-CF		0.0	0.1	-0.4	0.1	-0.4	-1.4	-0.5
10	2302-CF					-0.5	-0.4	-0.3	0.7
11B	2302-CF		-4.7	-4.7	-0.7	0.9	6.0	1.3	3.3
12	2302-CF						-0.9	-1.4	
14	2302-CF				1.4	0.6	0.8	1.5	0.8
18	2302-CF		-1.8	-0.3	-1.0	-0.5	-0.7	-0.7	-0.4
22	2302-CF			-0.7	-2.0	0.3	1.3	0.2	0.3
27	2302-CF					2.1	0.1	1.8	-0.9
28	2302-CF		-1.2	-1.5	-0.9	-1.2	-0.9	-1.1	-1.0
30	2302-CF		-0.6	0.6	1.4	1.4	0.8	0.4	-0.2
36	2302-CF						0.1	0.5	
39	2302-CF		-0.5	0.4	-0.1	0.8	0.2	0.6	0.3
40	2302-CF						-2.4	-2.1	
41	2302-CF		1.3	1.9	1.7	0.6	0.6	0.4	0.7
43	2302-CF		171	11	1.6	-1.3	1.0		-0.5
47	2302-CF		1.6	-1.7	0.1	-1.9	0.0	0.4	0.3
53	2302-CF			1.0	-1.4	3.9	2.6	4.9	3.0
55	2302-CF		5.4	1.3	1.8	0.8	0.6	0.7	0.2
59	2302-CF		-0.2	-1.0	-0.6	-0.6	-0.6	-0.5	-0.5
60	2302-CF				4.6	-1.2	0.1	0.6	1.2
64	2302-CF						14.8	14.7	
67	2302-CF						-1.5	-0.9	
78	2302-CF				-1.6	3.1	0.8	1.3	1.0
80	2302-CF		-4.9	-2.6	-2.6	-2.1	-2.6	-1.7	-1.4
83	2302-CF				-1.1	-0.1	-0.6	-0.3	-0.5
84	2302-CF				-1.9	-1.4	-1.1	-0.6	-0.9
87	2302-CF		1.0	-1.2	-0.4	1.6	-0.1	0.5	1.8
88	2302-CF						-3.3	-2.4	
91	2302-CF		-0.9	-0.6	0.4	-0.2	0.7	1.8	1.1
92	2302-CF		-0.5	-0.2	-0.4	-0.2	-1.0	-0.4	-0.7
93	2302-CF		5.2	6.5	9.0		6.6		
99	2302-CF		1.5	0.3	0.8	0.1	0.5	-0.2	0.1
107	2302-CF		-0.9	-0.9	-0.7	-1.2	-1.5	-1.6	-1.4
108	2302-CF		1.1	1.2	8.2	1.8	2.1	1.8	0.7
109	2302-CF		0.4	0.5	0.8	0.1	0.1	-0.3	0.0
113	2302-CF						0.4	1.6	
114	2302-CF		-1.6	-0.5	0.5	0.6	-0.2	-0.3	-0.3
115	2302-CF						0.5	-0.6	
10A	2302-CF		0.3	0.7	2.3	0.3	-0.1	-0.3	0.3

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Compound Feed (2302-CF)
Perfluoroalkylsulfonic acids (PFSAs) - Z-scores

LC	Sample	Z-score [σ_p = 20 %]	Linear Perfluorooctanesulfonic acid **(L-PFOS)**	Sum of branched Perfluorooctanesulfonic acid **(br-PFOS)**	Sum of branched and linear Perfluorooctanesulfonic acid **(total PFOS)**
4	2302-CF		1.3		
5	2302-CF		0.0		0.1
8	2302-CF		-1.1		-1.4
10	2302-CF		0.6		0.9
11B	2302-CF				-1.1
12	2302-CF				0.8
14	2302-CF		0.4		0.7
18	2302-CF		-0.4		-0.4
22	2302-CF		0.4		0.0
27	2302-CF		-0.9		
28	2302-CF		-1.1		-1.1
30	2302-CF		0.0		0.2
36	2302-CF				-0.4
39	2302-CF				
40	2302-CF		-1.9		-1.9
41	2302-CF		0.5		0.6
43	2302-CF		3.2		2.9
47	2302-CF				16.7
53	2302-CF		3.2		3.0
55	2302-CF		0.9		0.5
59	2302-CF		-0.6		-0.3
60	2302-CF		0.2		0.2
64	2302-CF				46.7
67	2302-CF		-0.9		
78	2302-CF		1.2		1.2
80	2302-CF		-3.4		-3.0
83	2302-CF		-0.4		-0.3
84	2302-CF		-0.6		-0.2
87	2302-CF				0.7
88	2302-CF		-2.5		-2.7
91	2302-CF		0.4		0.0
92	2302-CF				-0.7
93	2302-CF		5.8		5.1
99	2302-CF		-0.3		-0.6
107	2302-CF		-1.7		-1.9
108	2302-CF		0.9		0.5
109	2302-CF		0.0		
113	2302-CF		1.2		0.7
114	2302-CF		-0.3		-0.6
115	2302-CF				0.0
10A	2302-CF		3.0		2.5

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]

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

Compound Feed (2302-CF)

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)**
4	2302-CF		25	25
5	2302-CF		0.1	0.3
8	2302-CF		-0.6	-0.8
10	2302-CF		-0.1	0.0
11B	2302-CF		3.6	3.4
12	2302-CF		0.6	0.8
14	2302-CF		0.8	1.0
18	2302-CF		-0.7	-0.6
22	2302-CF		0.7	0.8
27	2302-CF			
28	2302-CF		-0.7	-1.0
30	2302-CF		0.4	0.6
36	2302-CF			0.1
39	2302-CF		1.1	-1.0
40	2302-CF		-2.3	-2.2
41	2302-CF		0.4	0.6
43	2302-CF		1.9	0.4
47	2302-CF		4.0	4.4
53	2302-CF		4.5	4.7
55	2302-CF		0.4	0.6
59	2302-CF		-0.6	-0.5
60	2302-CF		0.3	0.7
64	2302-CF		23.2	24.4
67	2302-CF		-1.3	-1.1
78	2302-CF		0.9	1.2
80	2302-CF		-2.3	-2.2
83	2302-CF		-0.6	-0.5
84	2302-CF		-0.9	-0.7
87	2302-CF		0.4	0.2
88	2302-CF		-2.8	-3.0
91	2302-CF		0.5	0.8
92	2302-CF		-0.9	-0.8
93	2302-CF		4.7	4.2
99	2302-CF		0.2	0.2
107	2302-CF		-1.7	-1.6
108	2302-CF		1.5	1.7
109	2302-CF		-0.1	0.0
113	2302-CF		0.7	0.9
114	2302-CF		-0.3	-0.3
115	2302-CF		0.1	0.2
10A	2302-CF		0.5	0.6
4*	2302-CF		0.0	-0.1

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]
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Annex 4: Participants' z-scores of PFCAs, PFSAs, sum of PFOS, PFOA, PFNA, PFHxS - charts

Test sample - Compound Feed (2302-CF)

Z-scores of individual substances and sum parameters

Calculation of z-score on basis of assigned value

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

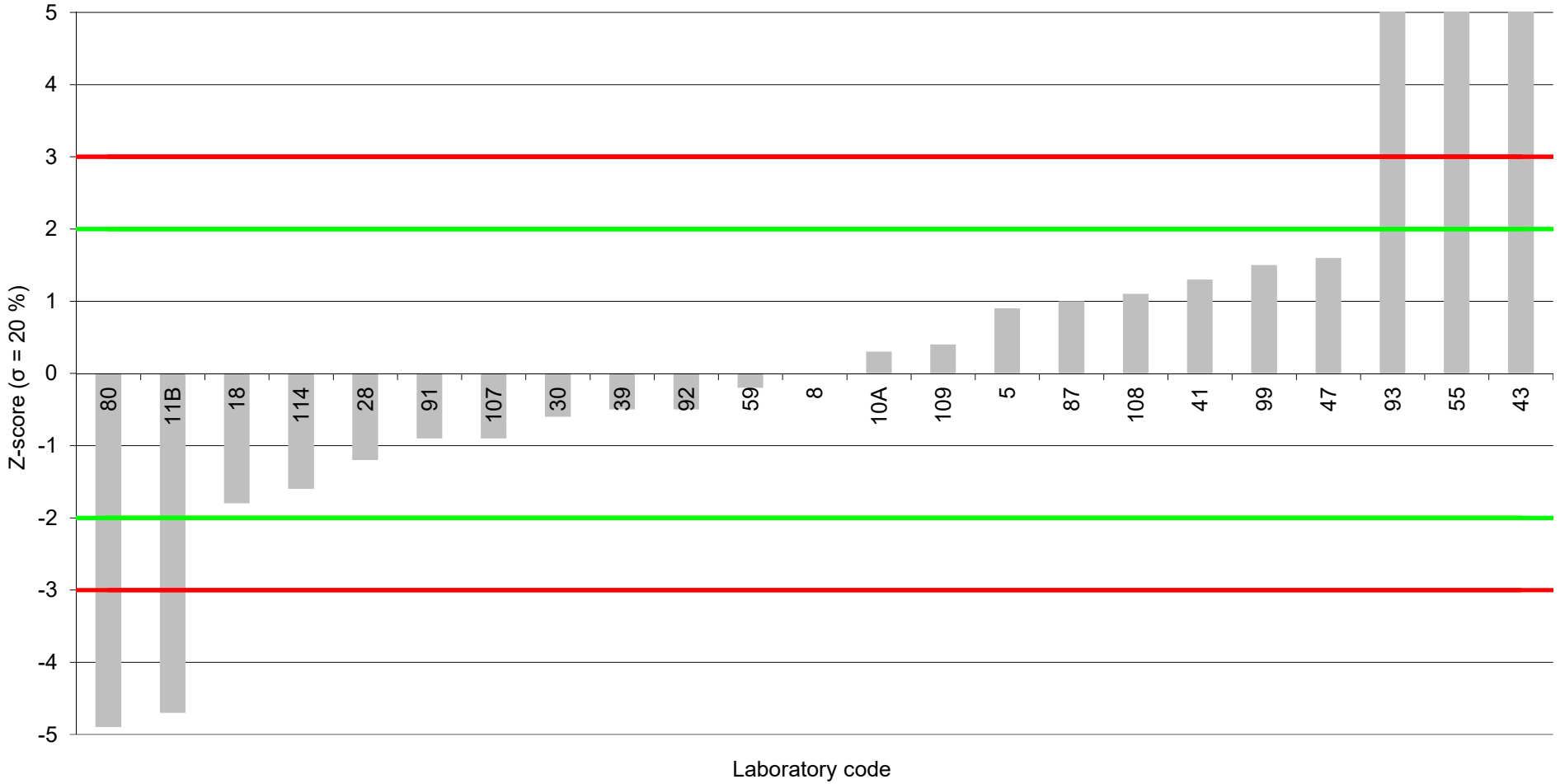
x_a: assigned value
x: participant's result
σ_p: fitness-for-purpose-based standard deviation for proficiency assessment
20%: Evaluated individual substances and sum parameters

± 2 z-scores:
± 3 z-scores:

Compound Feed (2302-CF)

Perfluorobutanoic acid

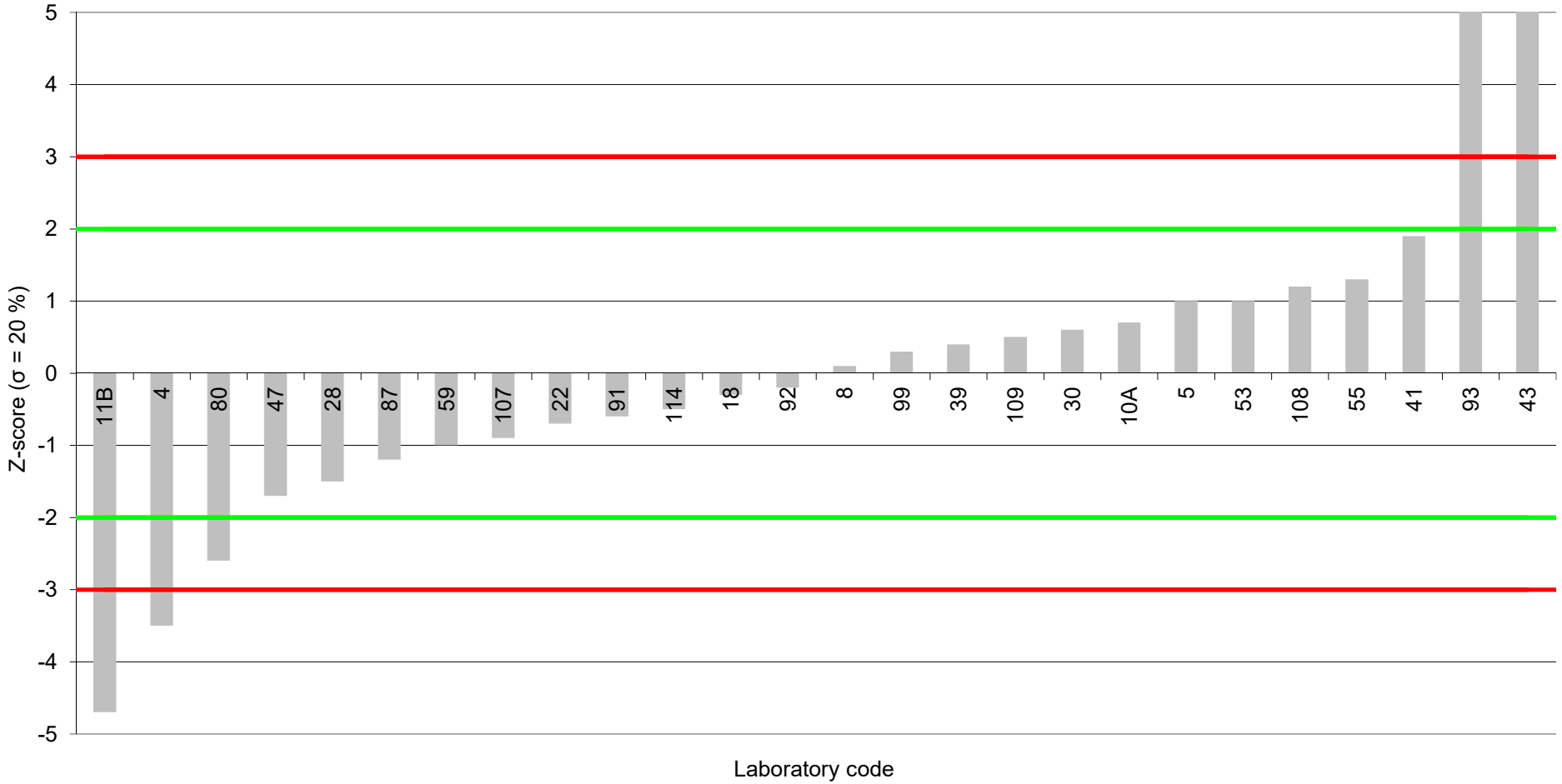
Assigned value: 12.2 µg/kg (12% moisture content)



Compound Feed (2302-CF)

Perfluoropentanoic acid

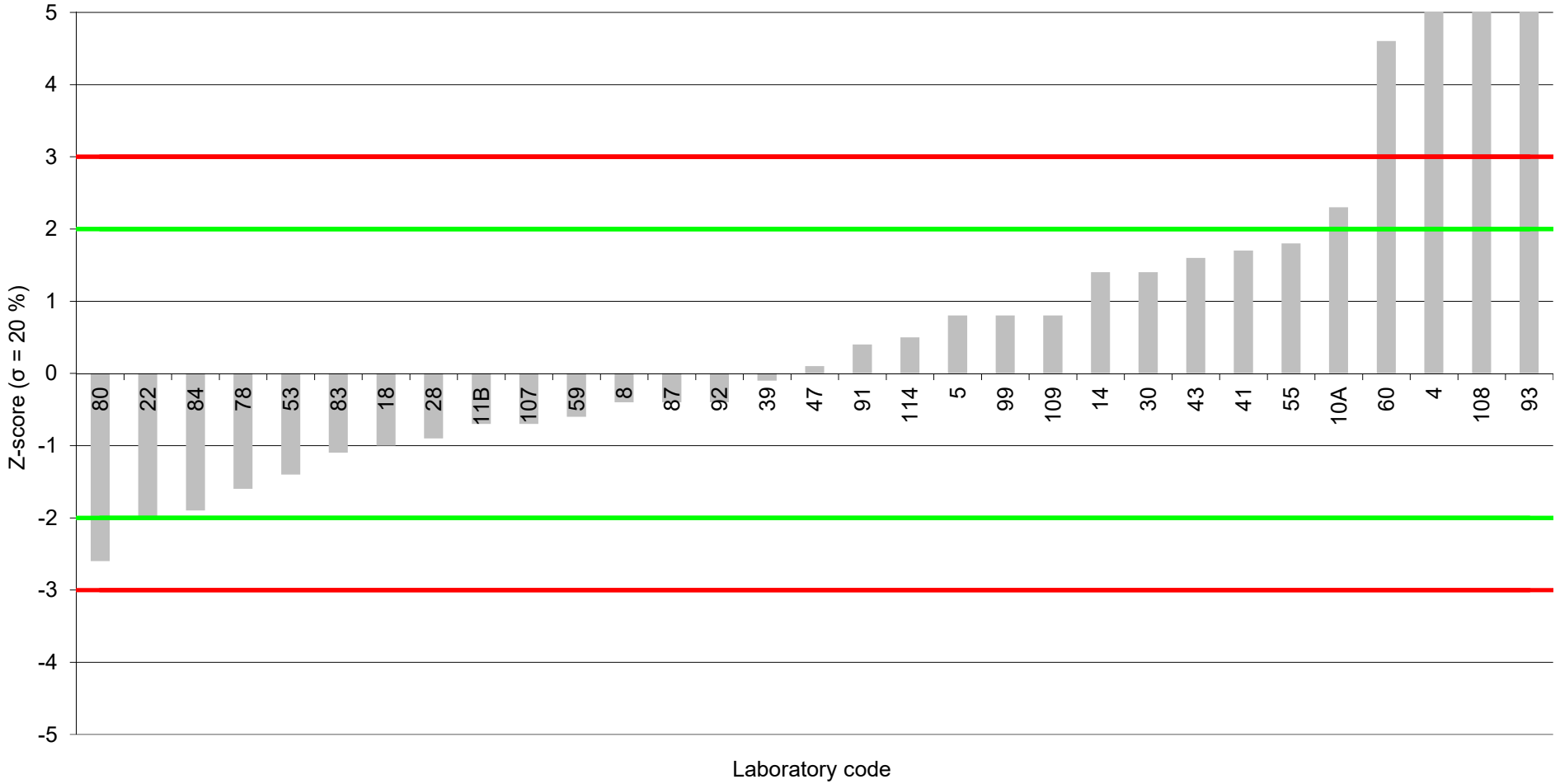
Assigned value: 19.3 µg/kg (12% moisture content)



Compound Feed (2302-CF)

Perfluorohexanoic acid

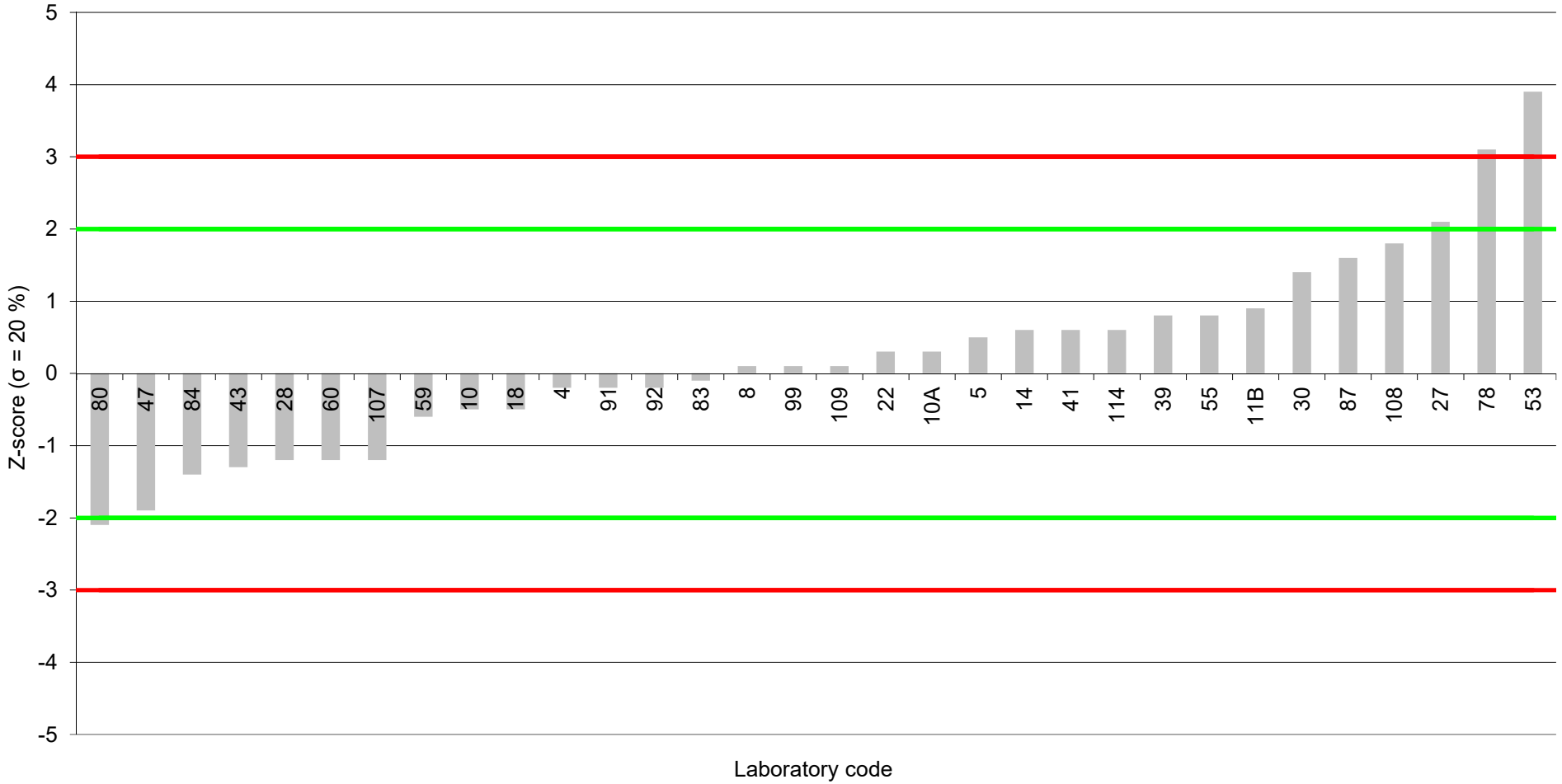
Assigned value: 3.78 µg/kg (12% moisture content)



Compound Feed (2302-CF)

Perfluoroheptanoic acid

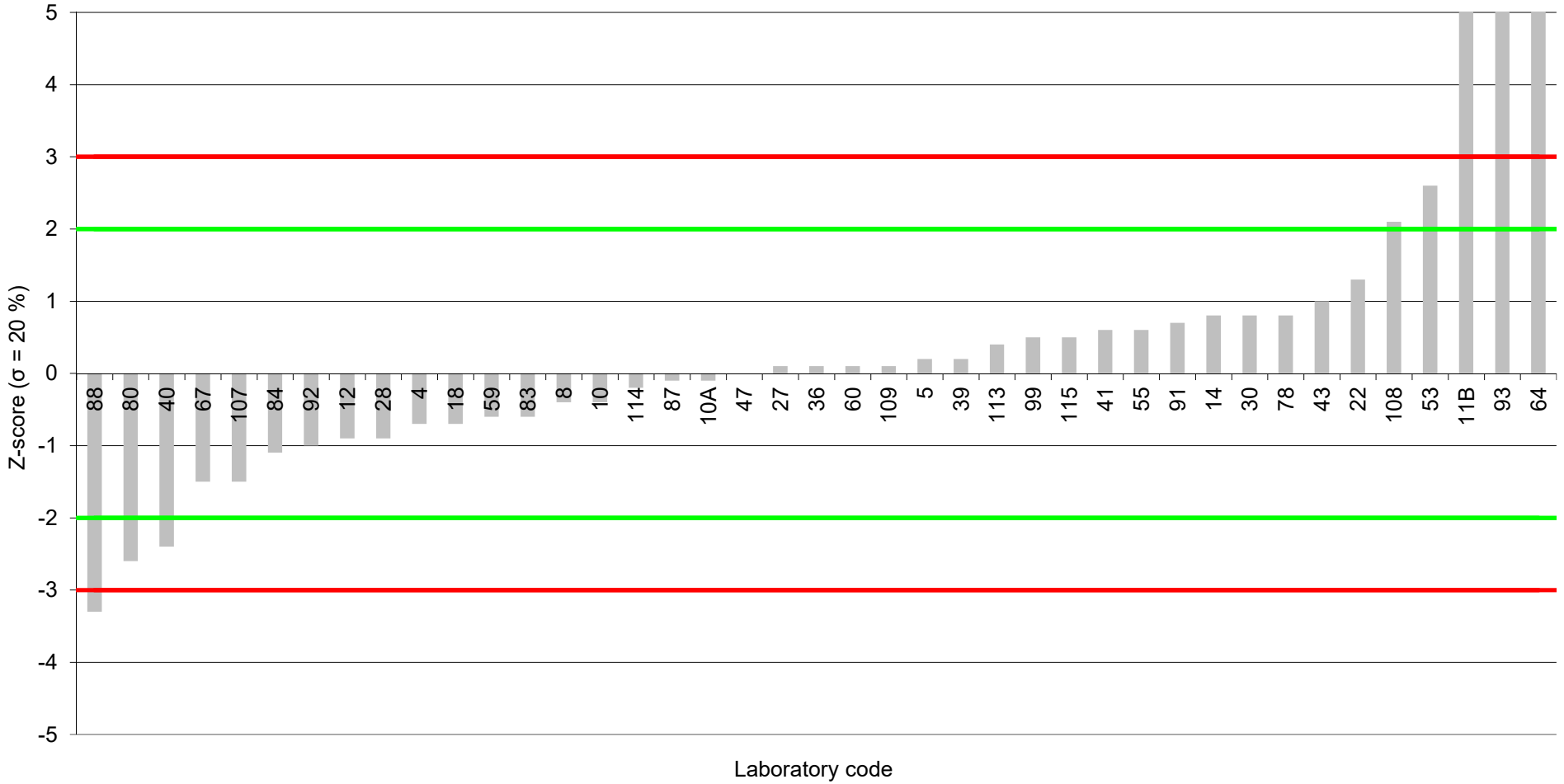
Assigned value: 1.03 µg/kg (12% moisture content)



Compound Feed (2302-CF)

Perfluorooctanoic acid

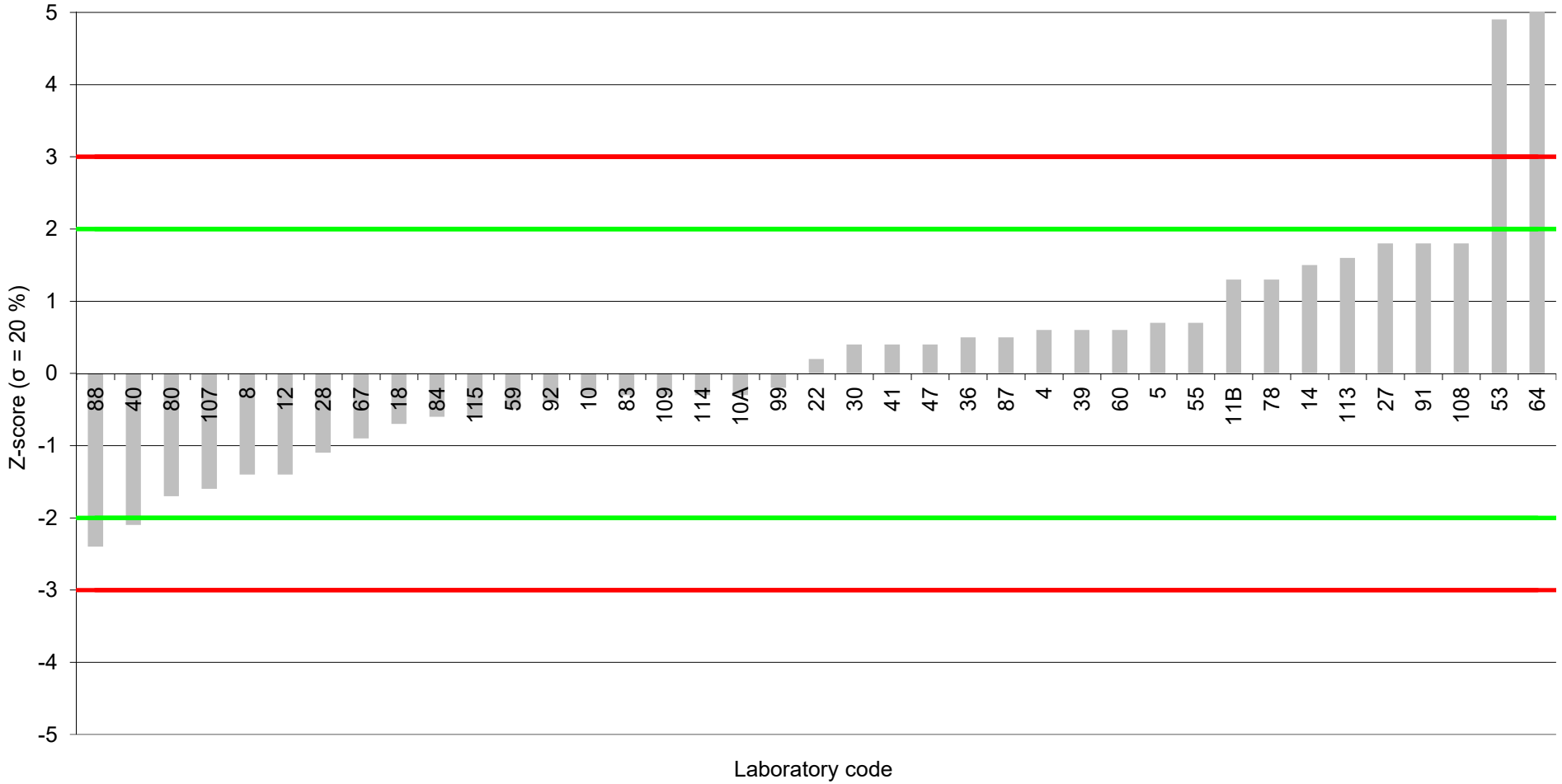
Assigned value: 1.84 µg/kg (12% moisture content)



Compound Feed (2302-CF)

Perfluorononanoic acid

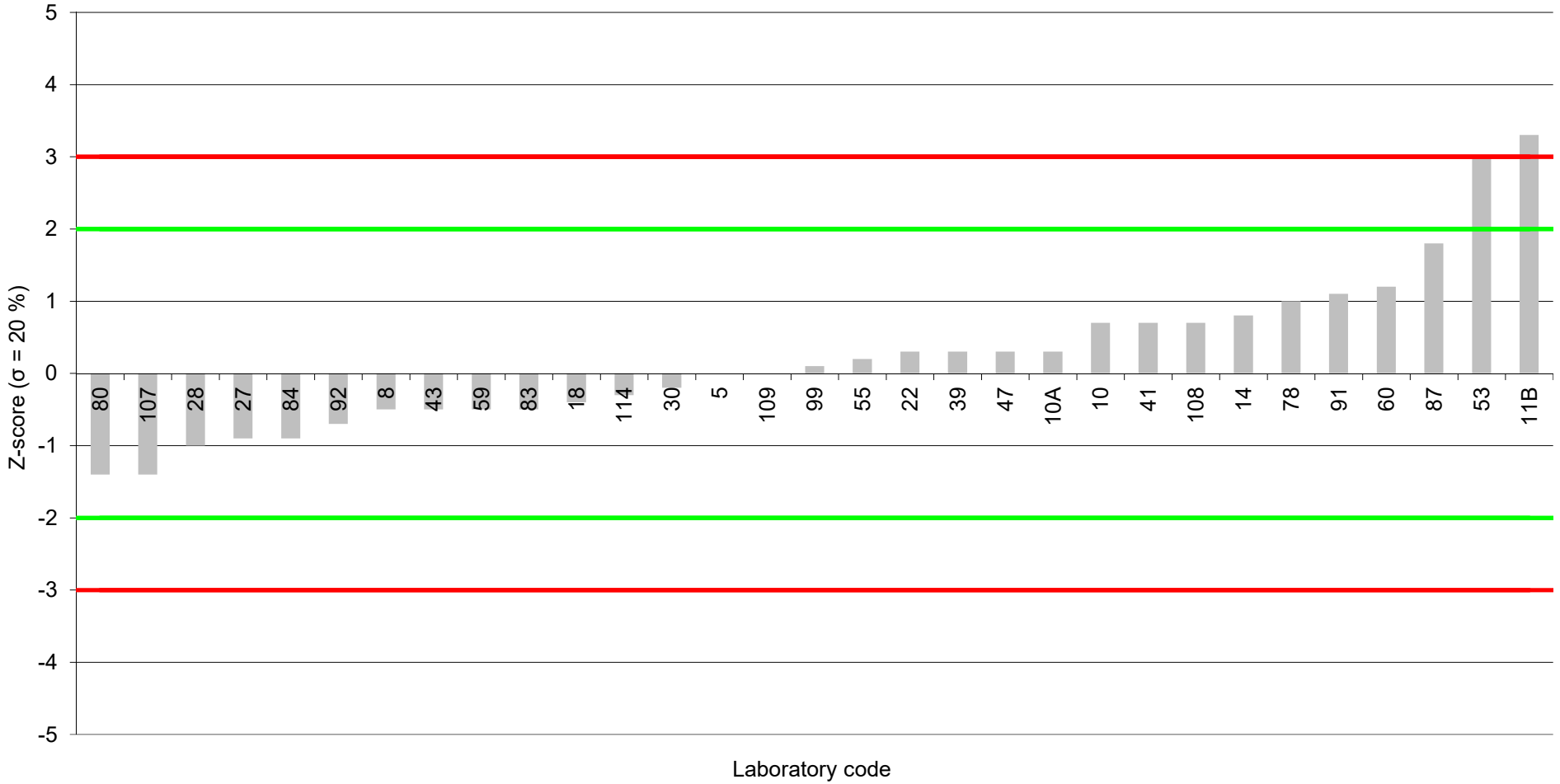
Assigned value: 0.58 µg/kg (12% moisture content)



Compound Feed (2302-CF)

Perfluorodecanoic acid

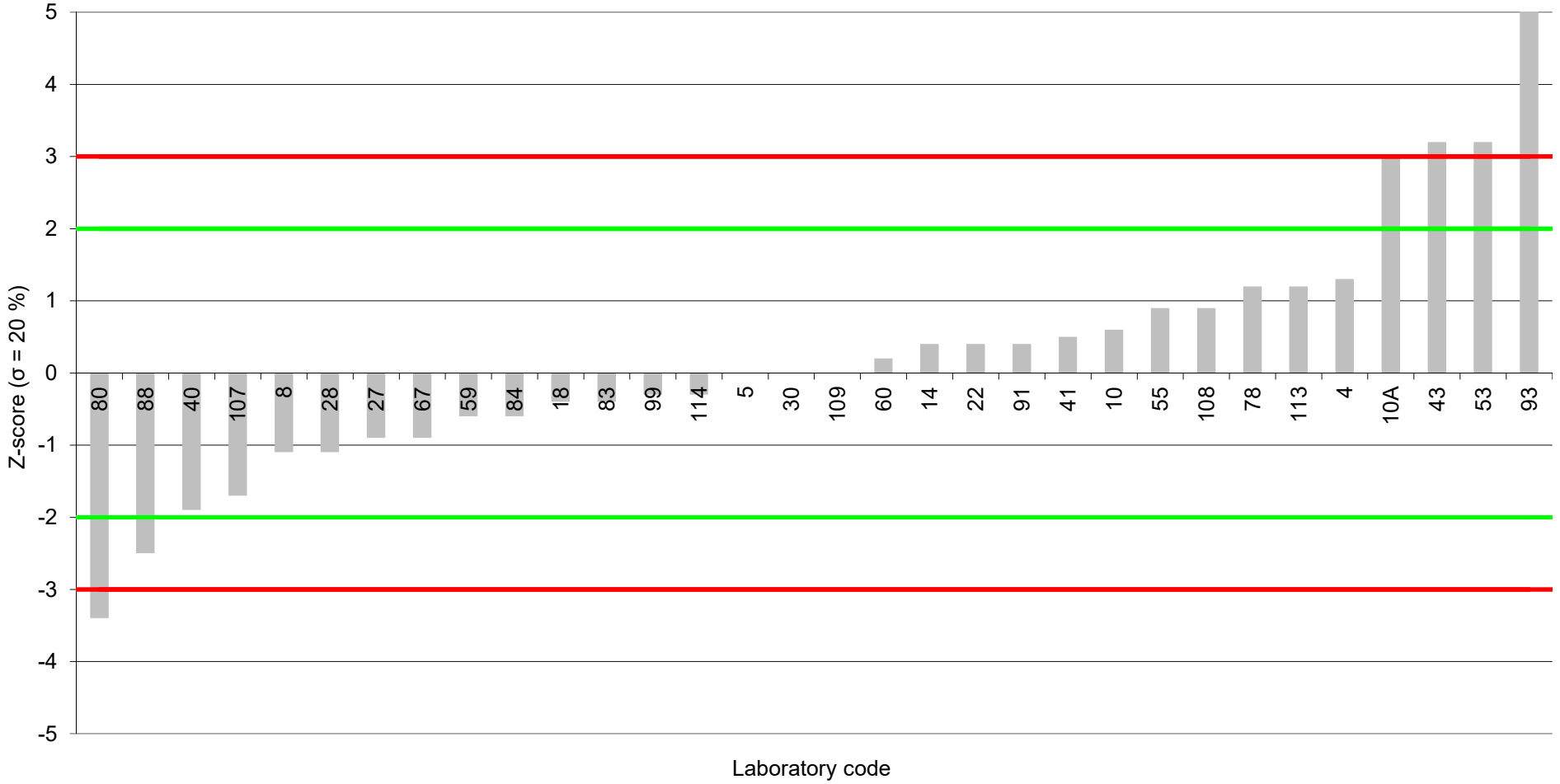
Assigned value: 1.22 µg/kg (12% moisture content)



Compound Feed (2302-CF)

Linear Perfluorooctanesulfonic acid

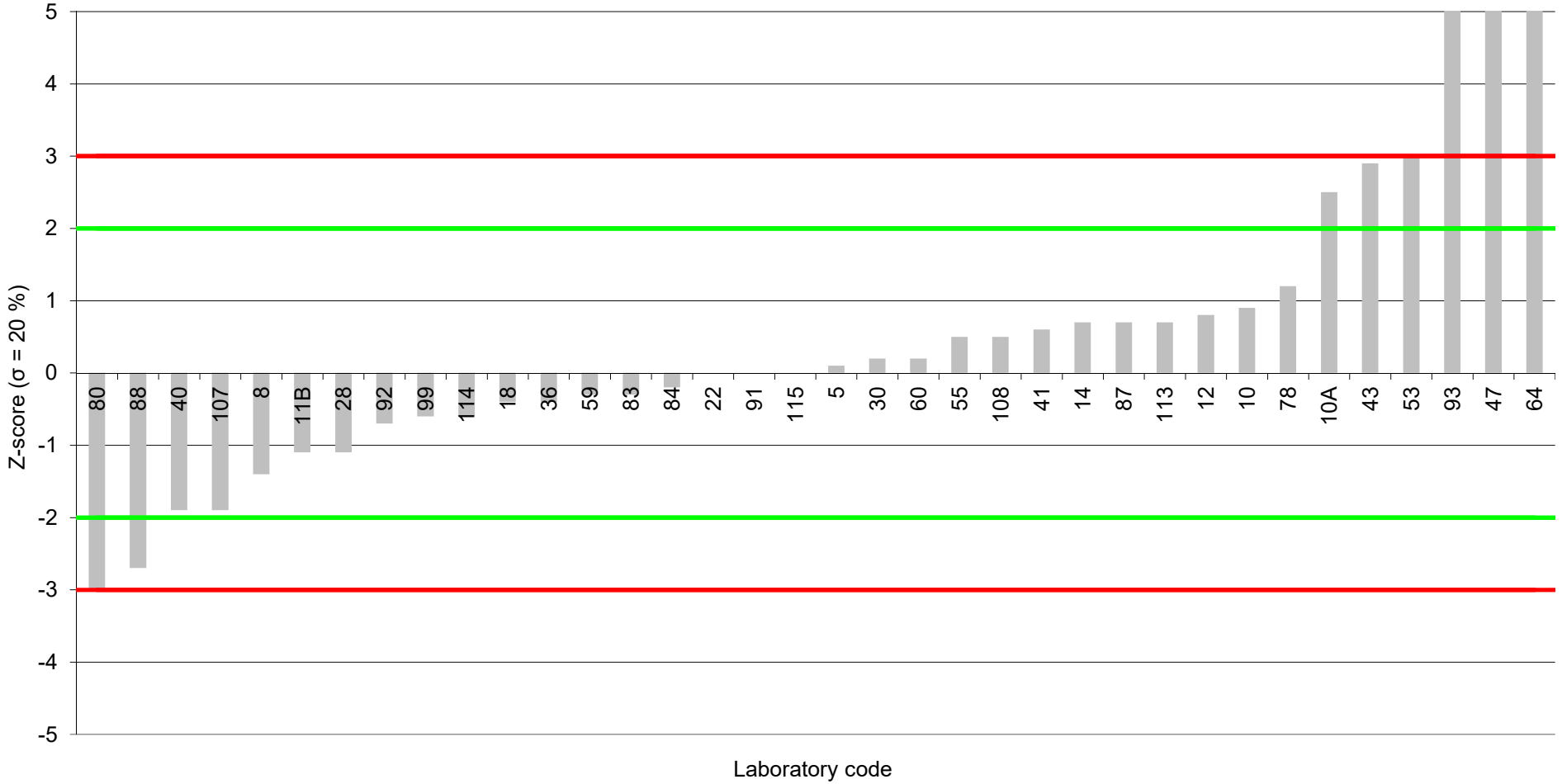
Assigned value: 0.747 µg/kg (12% moisture content)



Compound Feed (2302-CF)

Sum of branched and linear Perfluorooctanesulfonic acid

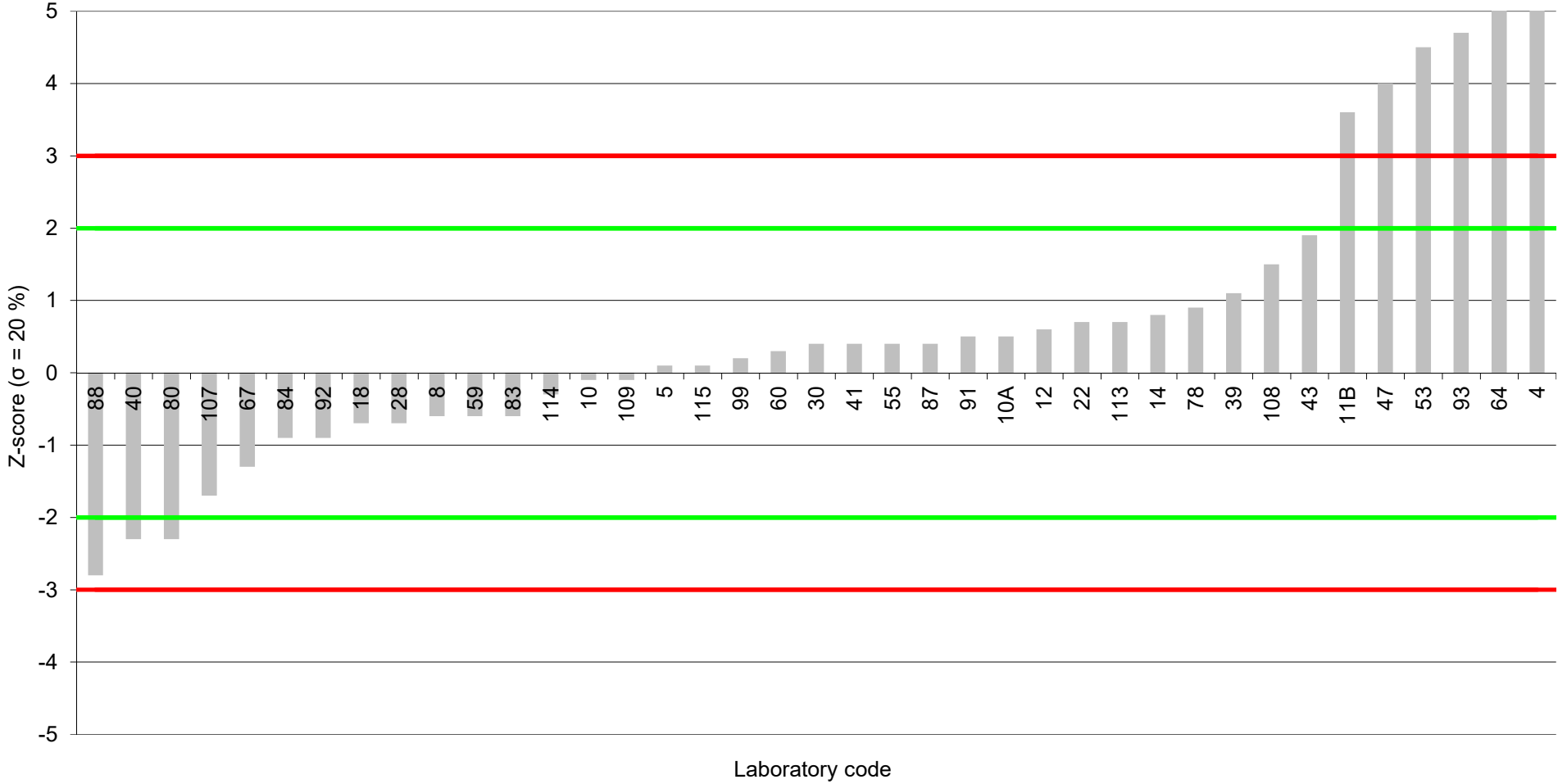
Assigned value: 0.802 µg/kg (12% moisture content)



Compound Feed (2302-CF)

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

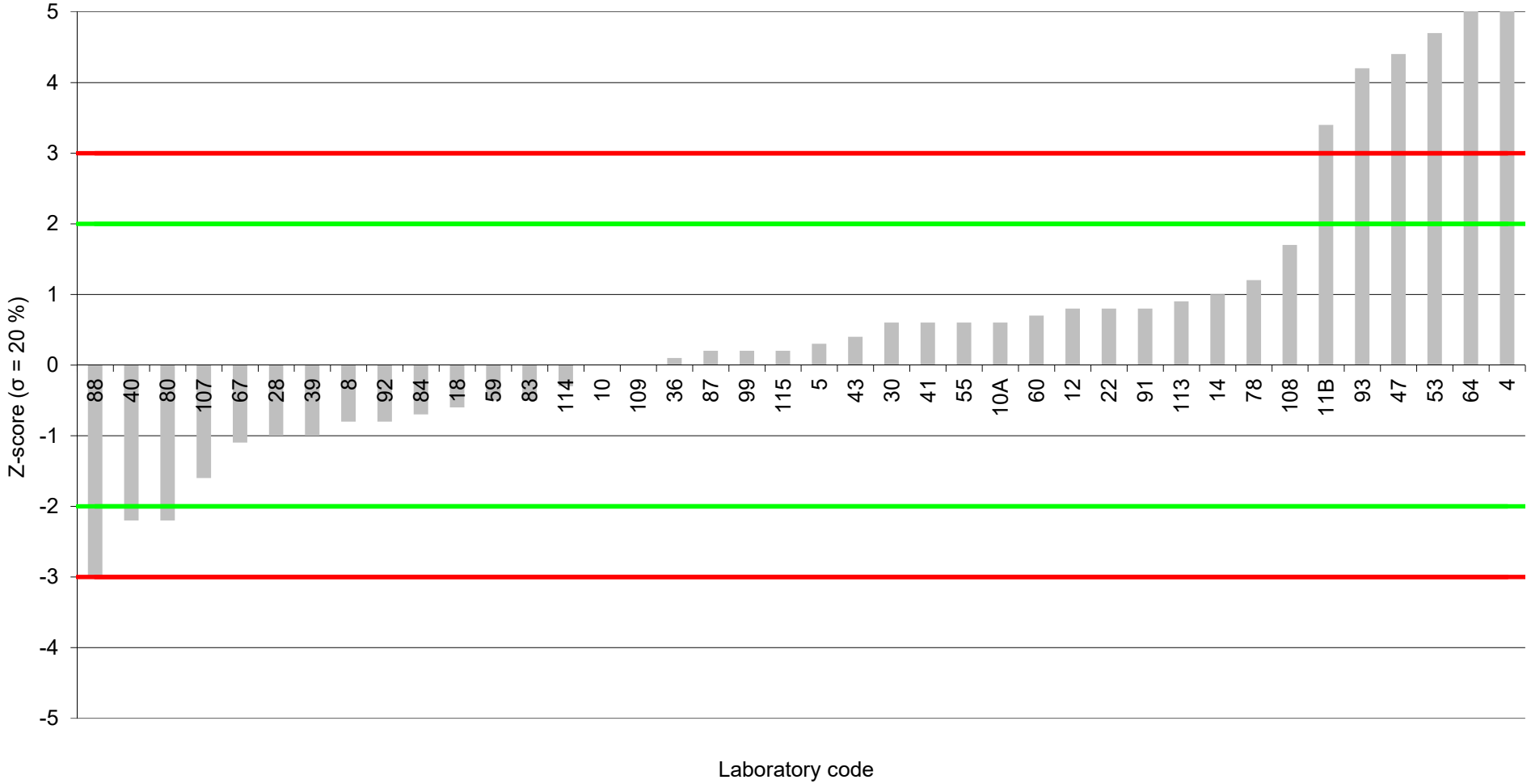
Assigned value: 3.35 µg/kg (12% moisture content)



Compound Feed (2302-CF)

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

Assigned value: 3.21 µg/kg (12% moisture content)



EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food
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Annex 5: Test for sufficient homogeneity for PFAS

Test sample - Compound Feed (2302-CF)

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Compound Feed (2302-CF)
PFCAs, PFSAs - Homogeneity test - Data

Analyte	Result µg/kg product (12% Moisture Content)	**Mean** (n = 10, duplicate analysis)	**Median** (n = 10, duplicate analysis)	**Relative standard deviation** [%]
Perfluoroheptanoic acid (PFHpA)		1.63	1.62	2%
Perfluorooctanoic acid (PFOA)		3.05	3.05	3%
Perfluorononanoic acid (PFNA)		0.780	0.786	7%
Perfluorodecanoic acid (PFDA)		1.76	1.78	3%
Sum of branched Perfluorooctanesulfonic acid (br-PFOS)		0.0869	0.0822	10%
Linear Perfluorooctanesulfonic acid (L-PFOS)		1.02	1.02	6%
Sum of branched and linear Perfluorooctanesulfonic acid (total PFOS)		1.11	1.11	6%

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Compound Feed (2302-CF)
Selected compounds - Homogeneity test - Data

Sample	Replicate	Result µg/kg product (12% Moisture Content)	L-PFOS	PFNA	PFHpA
13	1		0.973	0.745	1.58
	2		1.10	0.790	1.65
43	1		0.985	0.745	1.63
	2		1.10	0.746	1.55
45	1		0.944	0.776	1.54
	2		1.00	0.807	1.56
60	1		1.11	0.803	1.59
	2		1.06	0.777	1.77
129	1		0.879	0.786	1.59
	2		1.05	0.835	1.84
132	1		0.981	0.803	1.65
	2		0.940	0.835	1.56
143	1		1.03	0.731	1.65
	2		1.11	0.765	1.62
169	1		1.01	0.665	1.58
	2		0.970	0.735	1.62
172	1		1.02	0.855	1.65
	2		1.07	0.814	1.65
196	1		1.06	0.807	1.64
	2		1.03	0.776	1.64
Cochran's C-test					
C			0.390	0.312	0.530
$C_{critical}$ (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 $\bar{x}$			1.02	0.780	1.6
Standard deviation of sample averages s_x			0.046	0.040	0.0468
Wthin-sample standard deviation s_w			0.061	0.028	0.0752
Between-sample standard deviation s_s			0.015	0.035	0
Standard deviation for proficiency assessment σ_{PT}			0.20	0.16	0.325
s_s / σ_{PT}			0.072	0.224	0
Test for homogeneity ($s_s \leq 0.3\ \sigma_{PT}$)			passed	passed	passed

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-C
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Compound Feed (2302-CF)
Selected compounds - Stability test - Data

Sample	Replicate	Result µg/kg product (12% Moisture Content)	**PFHpA**
58	1		1.57
	2		1.55
111	1		1.48
	2		1.62
184	1		1.54
	2		1.53
Stability test			
General average (stability test) $\bar{y}$			1.55
General average (homogeneity test) $\bar{x}$			1.63
Standard deviation for proficiency assessment σPT			0.33
$\lvert \bar{y} - \bar{x} \rvert$			0.0790
Test for stability ($\lvert \bar{y} - \bar{x} \rvert \leq 0.3$ σPT)			passed

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]
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Annex 6: Overview participants' methods – Weighed sample, internal and recovery standards and comments

Test sample - Compound Feed (2302-CF)

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]

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

Compound Feed (2302-CF)

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)	Other PFAS (yes/no)	Other internal standards	Use of recovery / injection standard(s) (yes/no)	Matrix calibration (yes/no)
4	2302-CF	2	yes	yes			no	no
5	2302-CF	0,5 g / 1 g	yes	yes			yes	no
8	2302-CF	1	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
10	2302-CF	1	YES	YES	YES		YES	NO
11B	2302-CF	2	yes	yes	yes		no	no
12	2302-CF	3	yes	yes	no		no	no
14	2302-CF	2 g	yes	yes	no		yes	no
18	2302-CF	1 g	yes	yes			yes	no
22	2302-CF		yes	yes			Yes	
27	2302-CF	~1	Y -MPFAC-C-ES - wellington	Y -MPFAC-C-ES wellington		MPFAC-C-ES - wellington	Y- MPFAc-C-IS wellington	no
28	2302-CF	1.04 g	Yes	Yes	Yes		Yes	No
30	2302-CF	0.8	yes	yes (except PFDoDS)	yes (except Capstone A)		no	no (except PFDoDS and Capstone A)
36	2302-CF	2	yes	yes			no	no
39	2302-CF	1	Yes	Yes	Yes		Yes	No
40	2302-CF	1	yes	yes			no	no
41	2302-CF	1	yes	yes	yes		no	yes
43	2302-CF	0.5	yes	yes	yes	13C Genx	yes	no
47	2302-CF	1	yes	yes			no	no
53	2302-CF	1,0g	yes	yes	yes	M2-6FTS, M2-6FTS, M2-8FTS, d3-N-MeFOSAA, d5-N-EtFOSAA, M8FOSA	yes	yes
55	2302-CF	1	yes	yes	yes and no		no	no
59	2302-CF	2	yes	yes	yes	M3-HFPO-DA, M2-4:2 FTS; M2-6:2 FTS; M2-8:2 FTS	yes	no
60	2302-CF	1	yes	yes			no	no
64	2302-CF	5	no	no	no		no	no
67	2302-CF	2	Yes	Yes			No	No
78	2302-CF	2	yes	yes			no	no- solvent standard
80	2302-CF	1	no	no	no		no	yes
83	2302-CF	4	yes	yes			yes	no
84	2302-CF	0.5	Yes	Yes			No	Yes
87	2302-CF	2	yes	yes			yes	no
88	2302-CF	2	yes	yes	no	no	no	no
91	2302-CF	2	yes	yes			yes/no	no
92	2302-CF	5	yes	yes	yes		no	no
93	2302-CF	2	yes	yes	yes		yes / no	no
99	2302-CF	0,5 g	yes	yes			yes	no
107	2302-CF	1	yes	yes	no		no	no
108	2302-CF	2	yes	yes	yes		no	no
109	2302-CF	1	yes	yes	no	no	yes	no
113	2302-CF	5	yes	yes	no			
114	2302-CF		yes	yes			yes	yes
115	2302-CF	1 g	yes	yes			yes	no
10A	2302-CF	1	YES	YES	YES		YES	NO

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]

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

Compound Feed (2302-CF)

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)**
4	2302-CF		M5PFPeA	MPFHxA	MPFOA	MPFOA	MPFNA		MPFUnDA	MPFDoDA	MPFDoDA	MPFDoDA
5	2302-CF	13C4-PFBA	13C5-PFPeA	13C5-PFHxA	13C4-PFHpA	13C8-PFOA	13C9-PFNA	13C6-PFDA	13C7-PFUnDA	13C2-PFDoDA	13C2-PFDoDA	13C2-PFTeDA
8	2302-CF	MPFBA	MPFPeA	MPFHxA	MPFHpA	MPFOA	MPFNA	MPFDA	MPFUnDA		MPFDoDA/MPFTeDA	MPFTeDA
10	2302-CF				13C4-PFHpA	13C8-PFOA	13C9-PFNA	13C6-PFDA	13C7-PFUnDA	13C2-PFDoDA	13C2-PFDoDA	13C2-PFTeDA
11B	2302-CF	M3PFBA	M3PFPeA	PFHxA-13C2	M4PFHpA	PFOA-13C4	PFNA-13C5	PFDA-13C2	PFUdA-13C2	PFDoA-13C2	M2PFTeDA	M2PFTeDA
12	2302-CF					13C-PFOA	13C-PFNA					
14	2302-CF			13C5-PFHxA	13C4-PFHpA	13C8-PFOA	13C9-PFNA	13C6-PFDA	13C7-PFUnDA	13C2-PFDoDA	13C2-PFDoDA	
18	2302-CF	13C4 PFBA	13C3 PFPeA	13C2 PFHxA	13C4 PFHpA	13C4 PFOA	13C5 PFNA	13C2 PFDA	13C2 PFUnDA	13C2 PFDoDA	13C2 PFDoDA	13C2 PFDoDA
22	2302-CF	PFBA-13C3	PFPeA-13C5	PFHxA-13C5	PFHpA--13C4	PFOA-13C8	PFNA-13C9	PFDA-13C6	PFUnDA-13C7	PFDoDA-13C2	PFTrDA-13C2	PFTeDA-13C2
27	2302-CF				13C PFHpA	13C PFOA	13C PFNA	13C PFDA	13C PFUnDA			
28	2302-CF	13C3-PFBA	13C3-PFBA	13C2-PFOA	13C2-PFOA	13C2-PFOA	13C4-PFOS	13C2-PFDA	13C2-PFDA	13C2-PFDA	13C2-PFDA	13C2-PFDA
30	2302-CF	MPFBA	MPFPeA	MPFHxA	MPFOS	MPFOA	MPFNA	MPFDA	MPFUnDA	MPFDoDA	MPFDoDA	MPFTeDA
36	2302-CF					PFOA-13C4	PFNA-13C5					
39	2302-CF	13C4 PFBA	13C5 PFPeA	13C5 PFHxA	13C4 PFHpA	13C8 PFOA	13C9 PFNA	13C6 PFDA	13C7 PFUnDA	13C2 PFDoDA	13C2 PFTeDA	13C2 PFTeDA
40	2302-CF					13C PFOA	13C PFNA					
41	2302-CF	13C4-PFBA	13C5-PFPeA	13C5-PFHxA	13C4-PFHpA	13C8-PFOA	13C9-PFNA	13C6-PFDA	13C7-PFUdA	13C2-PFDoA	13C2-PFTeDA	13C2-PFTeDA
43	2302-CF	MPFBA	M5PFPeA	13C2 -PFHA	M4PFHpA	13C4 PFOA	13C PFNA	13C2 PFDA	13C2 PFUnDA	13C2 PFDoDA	13C2 PFDoDA	M2 PFTeDA
47	2302-CF	PFBA-C13	PFPeA-C13	PFHxA-C13	PFOA-C13	PFOA-C13	PFNA-C13	PFDA-C13	PFDoDA-C13	PFDoDA-C13	PFOS-C13	PFDoDA-C13
53	2302-CF		M5PFPeA.IS	M5PFPHxA.IS	M4PFHpA.IS	M8PFOA.IS	M9PFNA.IS	M6PFDA.IS	M7PFUdA.IS	MPFDoA.IS	M8PFOS.IS	M2PFTeDA.IS
55	2302-CF	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-(13C3)tetradecansäure (M3PFTeDA)
59	2302-CF	13C4-PFBA	13C5-PFPeA	13C5-PFHxA	13C4-PFHpA	13C8-PFOA	13C9-PFNA	13C6-PFDA	13C7-PFUnDA	13C2-PFDoA	13C2-PFTeA	13C2-PFTeA
60	2302-CF	MPFBA	M3PFPeA	MPFHxA	M4PFHpA	MPFOA	MPFNA	MPFDA	MPFUdA	MPFDoA	M2PFTeDA	M2PFTeDA
64	2302-CF											
67	2302-CF					13C4-PFOA	13C5-PFNA					
78	2302-CF			[13C5]-PFHxA	[13C4]-PFHpA	[13C8]-PFOA	[13C9]-PFNA	[13C6]-PFDA	[13C7]-PFUnDA	[13C9]-PFNA		
80	2302-CF											
83	2302-CF			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		
84	2302-CF			MPFHxA	MPFHpA	MPFOA	MPFNA	MPFDA	MPFUnDA	MPFDoDA	MPFDoDA	MPFTeDA
87	2302-CF	PFBA-13C4	PFBA-13C4	PFHxA-13C2	PFOA-13C8	PFOA-13C8	PFOA-13C8	PFOA-13C8	PFOA-13C8	PFOA-13C8	PFOA-13C8	PFOA-13C8
88	2302-CF					PFOA-C8	PFNA-C9					
91	2302-CF	PFBA-13C2	PFPeA-13C5	PFHxA-13C2	PFOA-13C4	PFOA-13C4	PFNA-13C5	PFDA-13C2	PFUnDA-13C2	PFDoDA-13C2		
92	2302-CF	perfluoro-n-[13C4] butanoic acid (MPFBA)	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-[1,2,3,4,5,6,7–13 C7] undecanoic acid (M7PFUdA)	perfluoro-n-[1,2–13C2] dodecanoic acid (M2PFDoA)		
93	2302-CF											
99	2302-CF	13C4-PFBA	13C5-PFPeA	13C5-PFHxA	13C4-PFHpA	13C8-PFOA	13C9-PFNA	13C6-PFDA	13C7-PFUnDA	13C2-PFDoDA	Average 13C2-PFDoDA + 13C2-PFTeDA	13C2-PFTeDA
107	2302-CF	M4PFBA	M5PFPeA	MPFHxA	M4PFHpA	M4PFOA	M5PFNA	M6PFDA	M7PFUnDA	M2PFDoDA	M2PFDoDA	M2PFTeDA
108	2302-CF	13C4-PFBA	13C5-PFPeA	13C5-PFHxA	13C4-PFHpA	13C4-PFOA	13C5-PFNA	13C6-PFDA	13C7-PFUnDA	13C2-PFDoDA	13C2-PFDoDA	13C2-PFTeDA
109	2302-CF	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUnDA	PFDoDA	PFDoDA	PFTeDA
113	2302-CF					ILIS PFOA	ILIS PFNA					
114	2302-CF	13C4-PFBA	13C5-PFPeA	13C2-PFHxA	13C4-PFHpA	13C8-PFOA	13C5-PFNA	13C2-PFDA	13C2-PFUnA	13C2-PFDoA	13C2-PFTeDA	13C2-PFTeDA
115	2302-CF					13C4 - PFOA	13C9 - PFNA					
10A	2302-CF	13C4-PFBA	13C5-PFPeA	13C5-PFHxA	13C4-PFHpA	13C8-PFOA	13C9-PFNA	13C6-PFDA	13C7-PFUnDA	13C2-PFDoDA	13C2-PFDoDA	13C2-PFTeDA

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]

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

Compound Feed (2302-CF)

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)**
4	2302-CF												
5	2302-CF		13C3-PFBA		13C2-PFHxA		13C4-PFOA	13C5-PFNA	13C2-PFDA				
8	2302-CF		MPFBA injection	MPFBA injection	MPFBA injection	MPFBA injection	MPFOA injection	MPFOA injection	MPFDA injection	MPFDA injection		MPFDA injection	MPFDA injection
10	2302-CF					13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS
11B	2302-CF												
12	2302-CF												
14	2302-CF				13C5-PFNA	13C5-PFNA	13C5-PFNA	13C5-PFNA	13C5-PFNA	13C5-PFNA	13C5-PFNA	13C5-PFNA	
18	2302-CF		13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS
22	2302-CF		PFOA-13C2	PFOA-13C2	PFOA-13C2	PFOA-13C2	PFOA-13C2	PFOA-13C2	PFDA-13C2	PFDA-13C2	PFDA-13C2	PFDA-13C2	PFDA-13C2
27	2302-CF					RS-PFOA	RS-PFOA	RS-PFOA	RS-PFDA	RS-PFDA			
28	2302-CF		13C4-PFBA	13C5-PFPeA	13C5-PFHxA	13C4-PFHpA	13C8-PFOA	13C9-PFNA	13C6-PFDA	13C7-PFUnDA	13C2-PFDoDA	13C2-PFDoDA	13C2-PFTeDA
30	2302-CF												
36	2302-CF												
39	2302-CF		13C3 PFBA	13C3 PFBA	13C2 PFHxA	13C2 PFHxA	13C4 PFOA	13C5 PFNA	13C2 PFDA	13C2 PFDA	13C2 PFDA	13C2 PFDA	13C2 PFDA
40	2302-CF						none	none					
41	2302-CF		No	No	No	No	No	No	No	No	No	No	No
43	2302-CF		0.24	0.52	0.48	0.545	0.57	0.57	0.76	0.58	0.83	0.83	0.77
47	2302-CF												
53	2302-CF												
55	2302-CF												
59	2302-CF												
60	2302-CF		none	none	none	none	none	none	none	none	none	none	none
64	2302-CF												
67	2302-CF												
78	2302-CF												
80	2302-CF												
83	2302-CF				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		
84	2302-CF												
87	2302-CF												
88	2302-CF												
91	2302-CF												
92	2302-CF												
93	2302-CF		MPFBA	M5PFPeA	M5PFHxA	M4PFHpA	M8PFOA	M9PFNA	M6PFDA	M7PFUnDA	MPFDoA	M2PFTeDA	M2PFTeDA
99	2302-CF		R-13C3-PFBA	R-13C2-PFOA	R-13C2-PFOA	R-13C2-PFOA	R-13C2-PFOA	R-13C2-PFDA	R-13C2-PFDA	R-13C2-PFDA	R-13C2-PFDA	R-13C2-PFDA	R-13C2-PFDA
107	2302-CF		-	-	-	-	-	-	-	-	-	-	-
108	2302-CF												
109	2302-CF		PFBA	PFOA	PFOA	PFOA	PFOA	PFOA	PFDA	PFDA	PFDA	PFDA	PFDA
113	2302-CF						ILIS PFOA						
114	2302-CF		13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA
115	2302-CF												
10A	2302-CF		13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS

Compound Feed (2302-CF)

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)**
4	2302-CF			Only one MS/MS transition for identification						Only one MS/MS transition for identification		
5	2302-CF	confirmed via HILIC separation	confirmed via HILIC separation									
8	2302-CF											
10	2302-CF											
11B	2302-CF								result <0,40	result <0,40	result <0,40	result <0,40
12	2302-CF											
14	2302-CF	Not Analyzed	Not Analyzed									
18	2302-CF	1 transition / no confirmation	1 transition / no confirmation									
22	2302-CF		only one MS/MS transition									
27	2302-CF				Not validated for feed. No LOQ established. MU based on food method	Not validated for feed. No LOQ established. MU based on food method	Not validated for feed. No LOQ established. MU based on food method	Not validated for feed. No LOQ established. MU based on food method	Not validated for feed. No LOQ established. MU based on food method			
28	2302-CF	213.00>169.00	263.00>219.00									
30	2302-CF	A second chromatographic separation was used for identification.	A second chromatographic separation was used for identification.									
36	2302-CF											
39	2302-CF	one MRM used, no confirmation method	one MRM used, no confirmation method									
40	2302-CF											
41	2302-CF	HRMS	HRMS									
43	2302-CF	Due to a low recovery of the internal standard, and the use of only one MS/MS transition used for confirmation, the result should be considered indicative	Due to the use of only one MS/MS transition used for confirmation, the result should be considered indicative	Due to matrixeffects, there was interference in the qualifier ion peak. The result should be considered indicative.								
47	2302-CF											
53	2302-CF	Analyte not tested	confirmation with second chromatographic run									
55	2302-CF	no second transition, no confirmation	Identification via second MS/MS transition									
59	2302-CF	only one MS/MS transition	only one MS/MS transition									
60	2302-CF											
64	2302-CF											
67	2302-CF											
78	2302-CF											
80	2302-CF	one transition LC-MS/MS	two transition LC-MS/MS									
83	2302-CF											
84	2302-CF	not analysed	not analysed									
87	2302-CF											
88	2302-CF											
91	2302-CF											
92	2302-CF											
93	2302-CF	Uncertainty expressed as %RSD	Uncertainty expressed as %RSD	Uncertainty expressed as %RSD		Uncertainty expressed as %RSD						
99	2302-CF	only transition m/z 213 -> 169 ; result was confirmed by a second chromotographic seperation	only transition m/z 213,0 -> 169, ; result was confirmed by a second chromotographic seperation									
107	2302-CF	only one MS/MS transition and no additional confirmation	only one MS/MS transition and no additional confirmation									
108	2302-CF	Two transitions	Two transitions									
109	2302-CF	Identification and quantification were performed by HRMS	Identification and quantification were performed by HRMS									
113	2302-CF											
114	2302-CF											
115	2302-CF					after recalculation from 93,37% dry matter to 88%	after recalculation from 93,37% dry matter to 88%					
10A	2302-CF	Confirmed by a second chromatographic separation	Confirmed by a second chromatographic separation									

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]

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

Compound Feed (2302-CF)

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)
4	2302-CF		MPFBS		MPFHxS		MPFHcX				MPFOS			
5	2302-CF			13C3-PFHxS			13C8-PFOS		13C8-PFOS					
8	2302-CF		MPFBS	MPFHxS	MPFHxS	MPFOS	MPFOS		MPFOS	MPFHxS	MPFHxS	MPFHxS	MPFHxS	MPFHxS
10	2302-CF		13C3-PFBS	13C3-PFHxS	13C3-PFHxS	13C3-PFHxS	13C8-PFOS	13C8-PFOS		13C8-PFOS	13C6-PFDA		13C2-PFDoDA	
11B	2302-CF		M3PFBS	M3PFBS	PFHxS-18O2	PFHxS-18O2			PFOS ISTD	PFOS ISTD	PFOS ISTD	PFOS ISTD	PFOS ISTD	PFOS ISTD
12	2302-CF				13C-PFHxS				13C-PFOS					
14	2302-CF		13C3-PFBS	13C8-PFOS	13C3-PFHxS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C8-PFOS	-	13C8-PFOS	-
18	2302-CF		13C3 PFBS	13C3 PFBS	18O2 PFHxS	18O2 PFHxS	13C4 PFOS	13C4 PFOS	13C4 PFOS	13C2 PFUnDA	13C2 PFUnDA	13C2 PFUnDA	13C2 PFUnDA	13C2 PFUnDA
22	2302-CF		PFBS-13C3	PHHxS-13C3	PFHxS-13C3	PFOS-13C8	PFOS-13C8	PFOS-13C8	PFOS-13C8	PFOS-13C8	PFOS-13C8		PFOS-13C8	
27	2302-CF						13C PFOS							
28	2302-CF		13C3-PFBA	13C3-PFBA	13C2-PFOA	13C2-PFOA			13C4-PFOS	13C4-PFOS	13C4-PFOS		13C4-PFOS	
30	2302-CF		MPFHxS	MPFOS	MPFHxS	MPFHxS	MPFOS	MPFOS	MPFOS	MPFHxS	MPFHxS	MPFHxS		MPFTeDA
36	2302-CF				PFHxS-13C3				PFOS-13C4					
39	2302-CF		13C3 PFBS	13C3 PFBS	13C3 PFHxS	13C3 PFHxS	13C8 PFOS	13C8 PFOS		13C8 PFOS	13C8 PFOS		13C8 PFOS	
40	2302-CF				13C PFHxS		13C PFOS	13C PFOS	13C PFOS					
41	2302-CF		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	13C7-PFUdA	13C7-PFUdA
43	2302-CF		M3 PFBS	18O-PFHxS	18O-PFHxS	18O-PFHxS	13C4 PFOS	13C4 PFOS	13C4 PFOS	13C4 PFOS	13C4 PFOS	13C4 PFOS	13C4 PFOS	13C4 PFOS
47	2302-CF													
53	2302-CF		M3PFBS.IS	M4PFHpA.IS	M3PFHxS.IS	M4PFHpA.IS	M8PFOS.IS	M8PFOS.IS	M8PFOS.IS	M9PFNA	M2PFTeDA	MPFDoA.IS	MPFDoA.IS	MPFDoA.IS
55	2302-CF		Perfluoro-1-(2,3,4-13C3)butansulfonsäure (Na.salz) (M3PFBS)	Perfluoro-n-[1,2,3,4-13C4]heptansäure (M4PFHpA)	Perfluoro-1-(2,3,4-13C3)hexansulfonsäure (Na.salz) (M3PFHxS)	Perfluoro-n-(13C9)nonansäure (M9PFNA)	Perfluoro-n-(13C8)octansulfonsäure (Natriumsalz) (M8PFOS)		Perfluoro-n-(13C8)octansulfonsäure (Natriumsalz) (M8PFOS)	external calibration	external calibration	external calibration	external calibration	external calibration
59	2302-CF		13C3-PFBS	13C3-PFHxS	13C3-PFHxS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C7-PFUnDA	13C7-PFUnDA		13C2-PFTeA	
60	2302-CF		M3PFBS	M3PFBS	MPFHxS	MPFHxS	MPFOS	MPFOS	MPFOS	MPFOS	MPFOS			
64	2302-CF													
67	2302-CF				18O2-PFHxS		13C4-PFOS							
78	2302-CF		[13C3]-PFBS		[13C3]-PFHxS		[13C8]-L PFOS	[13C8]-L PFOS	[13C8]-L PFOS					
80	2302-CF													
83	2302-CF		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.					
84	2302-CF				MPFHxS	MPFOS	MPFOS	MPFOS	MPFOS					
87	2302-CF		PFHxA-13C2	PFHxA-13C2	PFHxA-13C2	PFOS-13C4			PFOS-13C4	PFOS-13C4	PFOS-13C4			
88	2302-CF				PFHxS-C3		PFOS-C8	PFOS-C8						
91	2302-CF				PFHxS-18O2	PFOS-13C4	PFOS-13C4	PFOS-13C4						
92	2302-CF		perfluoro-1-[1,3,4–13C3] butansolfonate (M3PFBS)		perfluoro-1-[1,3–13C3] esanosolfonate (M3PFHxS)				perfluoro-1-[13C8] octansolfonate (M8PFOS)					
93	2302-CF													
99	2302-CF		13C3-PFBS	13C5-PFPeA	13C3-PFHxS	13C4-PFHpA	13C8-PFOS m/z 80	13C8-PFOS m/z 80		13C7-PFUnDA	13C8-PFOS m/z 80	13C2-PFDoDA		13C2-PFTeDA
107	2302-CF		M3PFBS	M3PFBS	M3PFHxS	M4PFOS	M4PFOS	M4PFOS	M4PFOS	M4PFOS	M4PFOS			
108	2302-CF		13C3-PFBS	13C3-PFBS	18O2-PFHxS	18O2-PFHxS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C2-PFDoA
109	2302-CF		PFBS	PFBS	PFHxS	PFHxS	PFOS	PFOS		PFOS	PFOS		PFOS	
113	2302-CF				ILIS PFHxS		ILIS L-PFOS	ILIS L-PFOS	ILIS L-PFOS					
114	2302-CF		13C3-PFBS	13C3-PFBS	18O2-PFHxS	18O2-PFHxS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C8-PFOA	13C2-PFUnA	13C2-PFDoA	13C2-PFDoA	13C2-PFTeDA
115	2302-CF				13C3 - PFHxS				13C4 - PFOS					
10A	2302-CF		13C3-PFBS	13C3-PFHxS	13C3-PFHxS	13C3-PFHxS	13C8-PFOS	13C8-PFOS		13C8-PFOS	13C6-PFDA		13C2-PFDoDA	

Compound Feed (2302-CF)

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)
4	2302-CF													
5	2302-CF			18O2-PFHxS			13C4-PFOS		13C4-PFOS					
8	2302-CF		MPFBA injection	MPFBA injection	MPFBA injection	MPFOS injection	MPFOS injection		MPFOS injection	MPFBA injection	MPFBA injection	MPFBA injection	MPFBA injection	MPFBA injection
10	2302-CF		13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS		13C4-PFOS	13C4-PFOS		13C4-PFOS	
11B	2302-CF													
12	2302-CF													
14	2302-CF		13C5-PFNA	13C5-PFNA	13C5-PFNA	13C5-PFNA	13C5-PFNA	13C5-PFNA	13C5-PFNA	13C5-PFNA	13C5-PFNA	-	13C5-PFNA	-
18	2302-CF		13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS
22	2302-CF		PFOS-13C3	PFOS-13C3	PFOS-13C3	PFOS-13C3	PFOS-13C3	PFOS-13C3	PFOS-13C3	PFOS-13C3	PFOS-13C3		PFOS-13C3	
27	2302-CF						RS-PFOS							
28	2302-CF		13C3-PFBS	13C3-PFBS	13C3-PFHxS	13C3-PFHxS			13C8-PFOS	13C8-PFOS	13C8-PFOS		13C8-PFOS	
30	2302-CF													
36	2302-CF													
39	2302-CF		18O2 PFHxS	18O2 PFHxS	18O2 PFHxS	18O2 PFHxS	13C4 PFOS	13C4 PFOS		13C4 PFOS	13C4 PFOS		13C4 PFOS	
40	2302-CF				none		none	none	none					
41	2302-CF		No	No	No	No	No	No	No	No	No	No	No	No
43	2302-CF		1.21	1.50	1.50	1.495	1.08	1.08	1.08	1.08	1.08	1.08	1.08	1.08
47	2302-CF													
53	2302-CF													
55	2302-CF													
59	2302-CF													
60	2302-CF		none	none	none	none	none	none	none	none	none			
64	2302-CF													
67	2302-CF													
78	2302-CF													
80	2302-CF													
83	2302-CF		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					
84	2302-CF													
87	2302-CF													
88	2302-CF													
91	2302-CF													
92	2302-CF													
93	2302-CF		M3PFBS	M3PFBS	M3PFHxS	M3PFHxS	M8PFOS	M8PFOS	M8PFOS	M8PFOS	M8PFOS			
99	2302-CF		R-13C2-PFOA	R-13C2-PFOA	R-13C4-PFOS	R-13C2-PFOA	R-13C4-PFOS	R-13C4-PFOS		R-13C2-PFDA	R-13C4-PFOS	R-13C2-PFDA		R-13C2-PFDA
107	2302-CF		-	-	-	-	-	-	-	-	-	-	-	-
108	2302-CF													
109	2302-CF		PFOS	PFOS	PFOS	PFOS	PFOS	PFOS		PFOS	PFOS		PFOS	
113	2302-CF													
114	2302-CF		13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA
115	2302-CF													
10A	2302-CF		13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS		13C4-PFOS	13C4-PFOS		13C4-PFOS	

Compound Feed (2302-CF)
Methods Perfluoroalkylsulfonic acids (PFSAs) - Comments

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)
4	2302-CF					Only one MS/MS transition for identification							
5	2302-CF					L-PFOS, 499>80		L-PFOS, 499>80					
8	2302-CF												
10	2302-CF						L-PFOS; transition: 499>80	L-PFOS; transition: 499>80					
11B	2302-CF	result <0,40	result <0,40	result <0,40	result <0,40				result <0,40	result <0,40	result <0,40	result <0,40	result <0,40
12	2302-CF												
14	2302-CF	> LOD				L-PFOS 489.9 -> 80	br-PFOS = total - n-PFOS	L-PFOS 489.9 -> 80			Not Analyzed		Not Analyzed
18	2302-CF						L-PFOS / 498,9 > 80	L-PFOS / 498,9 > 80					
22	2302-CF						same MS transition of linear PFOS	498,9>80,1 498,9>99,1			not analized		not analized
27	2302-CF					Not validated for feed. No LOQ established. MU based on food method							
28	2302-CF						499.00>80.00; 463.00>169.00	br-PFOS and l-PFOS					
30	2302-CF						Reference standard: L-PFOS; mass transitions: m/z 499 > 80	Reference standard: L-PFOS; mass transitions: m/z 499 > 80					
36	2302-CF							L-PFOS; 499/80; 499/99					
39	2302-CF						br-PFOSK used for quantification, 499>99	LOQ raised due to lab Blank			Not tested		Not tested
40	2302-CF												
41	2302-CF						13C8-L-PFOS	13C8-L-PFOS					
43	2302-CF					Due to matrixeffects, there was interference in the qualifier ion peak. The result should be considered indicative.	L-PFOS; MRM 499 > 99 for quantification, 499 > 80 for qualification	Due to matrixeffects, there was interference in the qualifier ion peak. The result should be considered indicative.					
47	2302-CF										not analyzed	not analyzed	not analyzed
53	2302-CF						498,8/98,8 L-PFOSK branched (PFAC30PAR)						
55	2302-CF							L-PFOS was used to quantify total PFOS					
59	2302-CF						L-PFOS (499,0 -> 80,1)	L-PFOS (499,0 -> 80,1)					
60	2302-CF						L-PFOS for quantification	L-PFOS for quantification					
64	2302-CF												
67	2302-CF												
78	2302-CF			Linear isomer		498.9>80.1/99.1 L- and Br- Peaks are separated	498.9>80.1/99.1 L- and Br- Peaks are separated	498.9>80.1/99.1					
80	2302-CF						L-PFOS, 498.3>89.6,98.8	L-PFOS, 498.3>89.6,98.8					
83	2302-CF												
84	2302-CF	not analysed	not analysed						not analysed	not analysed	not analysed	not analysed	not analysed
87	2302-CF												
88	2302-CF												
91	2302-CF												
92	2302-CF							transition 499>99 for quantification					
93	2302-CF					Uncertainty expressed as %RSD		Uncertainty expressed as %RSD					
99	2302-CF						m/z 499,0 -> 80,0	m/z 499,0 -> 80,0				method not suitable for the sample --> apparent recovery < 65 %	
107	2302-CF						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
108	2302-CF						L-PFOS (499/99)	L-PFOS (499/99)					Not able to analyze.
109	2302-CF												
113	2302-CF						below LOQ (~0.075); T-PFOS 499.0 / 79.9	T-PFOS 499.0 / 79.9					
114	2302-CF												
115	2302-CF							after recalculation from 93,37% dry matter to 88%					
10A	2302-CF						L-PFOS; transition: 499>80	L-PFOS; transition: 499>80					

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Compound Feed (2302-CF)

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)**
4	2302-CF			
5	2302-CF			
8	2302-CF			
10	2302-CF			
11B	2302-CF			
12	2302-CF			
14	2302-CF			
18	2302-CF			
22	2302-CF			
27	2302-CF			
28	2302-CF			
30	2302-CF			
36	2302-CF			Reg (EU) 2023/915
39	2302-CF			
40	2302-CF			
41	2302-CF			
43	2302-CF		Due to matrixeffects, there was interference in the qualifier ion peak for PFOS. The result should be considered indicative.	Due to matrixeffects, there was interference in the qualifier ion peak for PFOS. The result should be considered indicative.
47	2302-CF			
53	2302-CF			
55	2302-CF			
59	2302-CF			
60	2302-CF			
64	2302-CF			
67	2302-CF			
78	2302-CF			
80	2302-CF			
83	2302-CF			
84	2302-CF			
87	2302-CF			
88	2302-CF		Expanded uncertainty was calculated According to REGULATION (EU) 2022/1428; U= 0.28 ug/kg	Expanded uncertainty was calculated According to REGULATION (EU) 2022/1428 U= 0.27 ug/kg
91	2302-CF			
92	2302-CF			
93	2302-CF			
99	2302-CF			
107	2302-CF			
108	2302-CF			
109	2302-CF			
113	2302-CF			
114	2302-CF			
115	2302-CF			
10A	2302-CF			
4*	2302-CF		Total PFOS is only linear PFOS	Total PFOS is only linear PFOS

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

Compound Feed (2302-CF)

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**
4	2302-CF								
5	2302-CF								
8	2302-CF			MPFOA		MPFHxS	MPFHxS		
10	2302-CF		13C8PFOSA		GenX-13C3				
11B	2302-CF			M3HFPO-DA	M4PFHpA	PFOS ISTD	PFOS ISTD		
12	2302-CF								
14	2302-CF								
18	2302-CF					M13C2 PFUnDA	M13C2 PFUnDA		
22	2302-CF								
27	2302-CF								
28	2302-CF		13C2-PFDA						
30	2302-CF		MFOSA	MPFHxS	MGENX	MPFHxS	MPFHxS		MPFHxS
36	2302-CF								
39	2302-CF		13C8 PFOSA	13C3 HFPO-DA	13C3 HFPO-DA	13C8 PFOS	13C8 PFOS		
40	2302-CF								
41	2302-CF		M8PFOSA	13C5-PFHxA	13C3-M3HFPO-DA	13C7-PFUdA	13C7-PFUdA		
43	2302-CF		13C8 PFOSA	13C GENX	13C GENX				
47	2302-CF								
53	2302-CF		M8FOSA.IS	M4PFHpA.IS	M2-4:2FTS3.IS	M9PFNA.IS	M7PFUdA.IS	no	no
55	2302-CF		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)	external calibration	external calibration		external calibration
59	2302-CF			13C4-PFHpA	13C3-HFPO-DA				
60	2302-CF								
64	2302-CF								
67	2302-CF								
78	2302-CF								
80	2302-CF								
83	2302-CF								
84	2302-CF								
87	2302-CF								
88	2302-CF								
91	2302-CF				GenX-13C3				
92	2302-CF				perfluoro-n-[1,2,3,4–13C4] heptanoic acid (M4PFHpA)				
93	2302-CF								
99	2302-CF		13C8-PFOSA	13C4-PFHpA	13C3-GenX	13C8-PFOS m/z 80	13C8-PFOS m/z 80		
107	2302-CF								
108	2302-CF		13C8-FOSA	13C4-PFHpA	13C3-HFPO-DA				
109	2302-CF								
113	2302-CF								
114	2302-CF		13C8-PFOSA	13C4-PFHpA	13C3-HFPO-DA	13C4-PFOS	13C2-PFDoA		
115	2302-CF								
10A	2302-CF								

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Compound Feed (2302-CF)
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**
4	2302-CF								
5	2302-CF								
8	2302-CF			MPFOA inj		MPFBA inj	MPFBA inj		
10	2302-CF		13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS		
11B	2302-CF								
12	2302-CF								
14	2302-CF								
18	2302-CF					M13C8 PFOS	M13C8 PFOS		
22	2302-CF								
27	2302-CF								
28	2302-CF		13C8-FOSA						
30	2302-CF								
36	2302-CF								
39	2302-CF		13C4 PFOS	13C4 PFOS	13C4 PFOS	13C4 PFOS	13C4 PFOS		
40	2302-CF								
41	2302-CF		No	No	No	No	No		
43	2302-CF		1.49	0.80	0.80				
47	2302-CF								
53	2302-CF								
55	2302-CF								
59	2302-CF								
60	2302-CF								
64	2302-CF								
67	2302-CF								
78	2302-CF								
80	2302-CF								
83	2302-CF								
84	2302-CF								
87	2302-CF								
88	2302-CF								
91	2302-CF								
92	2302-CF								
93	2302-CF								
99	2302-CF		R-13C2-PFOA	R-13C2-PFOA	R-13C2-PFDA	R-13C4-PFOS	R-13C4-PFOS		
107	2302-CF								
108	2302-CF								
109	2302-CF								
113	2302-CF								
114	2302-CF		13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA		
115	2302-CF								
10A	2302-CF								

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Compound Feed (2302-CF)
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**
4	2302-CF								
5	2302-CF								
8	2302-CF								
10	2302-CF								
11B	2302-CF			result <0,50	result <0,60				
12	2302-CF								
14	2302-CF		Not Analyzed	Not Analyzed	Not Analyzed	Not Analyzed	Not Analyzed	Not Analyzed	Not Analyzed
18	2302-CF								
22	2302-CF								
27	2302-CF								
28	2302-CF								
30	2302-CF								
36	2302-CF								
39	2302-CF							Not tested	Not tested
40	2302-CF								
41	2302-CF								
43	2302-CF								
47	2302-CF								
53	2302-CF								
55	2302-CF								
59	2302-CF								
60	2302-CF								
64	2302-CF								
67	2302-CF								
78	2302-CF								
80	2302-CF								
83	2302-CF								
84	2302-CF		not analysed	not analysed	not analysed	not analysed	not analysed	not analysed	not analysed
87	2302-CF								
88	2302-CF								
91	2302-CF								
92	2302-CF								
93	2302-CF								
99	2302-CF							method under development	method under development
107	2302-CF		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
108	2302-CF					Have not been analyzed.	Have not been analyzed.	Have not been analyzed.	Have not been analyzed.
109	2302-CF								
113	2302-CF								
114	2302-CF								
115	2302-CF								
10A	2302-CF								

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EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food
21 June 2024

Annex 7: Overview participants' methods – Extractions, clean-up and detection

Test sample - Compound Feed (2302-CF)

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]

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

Compound Feed (2302-CF)

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

LC	Sample	Pre-treatment and extraction				
4	2302-CF	Duplicate extraction with 5 mL acetonitrile	Sonification, 15 min	Centrifugation		
5	2302-CF	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, 250 mg carbon	Shaking by hand rapidly; Centrifugation
8	2302-CF	Quechers extraction using Water, Acetonitrile, NaCl and MgSO4 salts.	After centrifugation, organic phase is taken for clean-up steps.			
10	2302-CF	EXTRACTION WITH ACETONITRILE				
11B	2302-CF	extraction: 2x 5 ml acetonitrile, 10 min. ultrasonic bath, centrifugation 5 min./ 5000 rpm				
12	2302-CF	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.		
14	2302-CF	solid liquid extraction with 20 mL 0.1 % NH3 in acetonitrile + 2 mL water				
18	2302-CF	Extraction with MeOH/KOH				
22	2302-CF	extraction with NaOH 0,05M in methanol				
27	2302-CF	Addition of internal std	Alkaline digestion	Ext in Acetonitrile - shake and sonicate x 2		
28	2302-CF	A protion of a homogenized food sample is fortified with isotopically -labeled extraction standards and	extracted with acidified acetonitrile. After extraction, a dispersive Quechers cleanup is performed,	followed by solid phase extraction (SPE) cleanup. The extract is concentrated to approximately 400-500 ul	with nitrogen in a heated water bath. Extract is then reconstituted to 1mL with methanol.	Isotopically-labeled injection standards are added to the sample extract.
30	2302-CF	No pre-treatments.	Addition of internal standards.	Extraction: shaking with water/acetonitrile at room temperature.		
36	2302-CF	hydrolysis and solvent extraction				
39	2302-CF	Sample spiked with internal standards	Added Methanol with Potassium Hydroxide	Tumbled 8 hours	Centrifuged @ 3000 rpm	
40	2302-CF	addition of 1ml 200 mM NaOH	adddition of ISTD	extraction with 5 mL MeOH; shaking and UAE	centrifugation	addition of 4M HCl dilution with 12 mL of H2O
41	2302-CF	Two extractions with acetonitrile, after the addition of 5 mL of water to 1 g of sample feed				
43	2302-CF	Quechers method	Extracted in acetonitrile and water	AOAC Quechers salts added and sample centrifuged	Sample transfered to dSPE cartridge (900 mg MgSO4 and 300mg PSA) and shaken	Centrifuged and diluted in UPLC vial
47	2302-CF	Extraction with Acetonitrile/ Water (50:50, v/v) shaken for 20 min on a mechanical shaker	Addition of QUEChERS-Mix	centrifugation	Addition of the salt mixture to the sample resulted in a well-defined phase separation.	Organic supernatant was mixed with 2 ml of demineralized water
53	2302-CF	1,0g Sample, add 3mL Water, add 10mL ACN, add 0,5gNaCL+0,5gMgSO4, vortex, zentrifuge				
55	2302-CF	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	
59	2302-CF	2 g are extracted with 25 mL MeOH	Centrifugation Step	Evaporate the supernatant to a volume of 3 mL	dilute with water and adjust the pH value to 3,0	
60	2302-CF	Adding ISTD	Adding 20 mL methanol	Shaking for 15 min	Extract for 60 min with ultra sonic	Centrifugate for 15 min
64	2302-CF	Quechers extraction (salts) EMR				
67	2302-CF	After the adition of internal standards sample was extracted by acetonitrile				
78	2302-CF	Basic methanol liquid extraction				
80	2302-CF	1 g sample	10 mL MilliQ water, vortex and shaker 15 min	10 mL ACN and 150 uL formic acid	Vortex and shaker Ultrasound bath	6 g MgSO4 and 1,5 g NaCl Vortex and shaker; Centrifuge
83	2302-CF	Extraction was done using 10 ml of methanol/potassium hydroxide for 17 hours.				
84	2302-CF	Extracted with acetonitrile				
87	2302-CF	2 g were extracted with 10 ml Methanol				
88	2302-CF	Two grams of homogenised sample was extracted with 10 mL of ACN. The extraction was repeated twice.				
91	2302-CF	ultrasonic extraction using acetonitrile				
92	2302-CF	Quechers Extraction EN method (4g MgSO4 - 1g NaCl - 1g Trisodium Citrate dihydrate - 0,5 g disodium hydrogencitrate sesquihydrate Disodico Sesquidrato				
93	2302-CF	1) sample introduction into 50 ml PP tubes and spiking with 10 uL mix surrogate IS and left to equilibrium for 20 min	2) Additon of 10 ml of Methanol with 10 mM of NaOH and homogenization for 5 min in USAE	3) Orbital digestion (120 rpm, 2h) 4) centrifugation (2000 rpm, 10 min)	5) supernatant transferred to 50 ml PP new tubes 6) Evaporation till 2 ml under nitrogen current	7) Re-dilution in 45 ml of HPLC water, homogenization in vortex
99	2302-CF	# addition of 10 mL dest. water to 0,5 g	# extraction with 10 mL ACN and 60 µL HCl (6 MOL), shaking (20 min) and ultrasonication (20 min)	# addtion of 4 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), add 1 mL ACN	# addition of 8 mL 25mM NH4Ac buffer (pH = 6) # centrifugation and transfer to SPE cartridge
107	2302-CF	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
108	2302-CF	Modified QuEChERS. The sample is extracted two times with acetonitrile, MgSO4 and NaCl is added.				
109	2302-CF	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.			
113	2302-CF	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	
114	2302-CF	Quechers-Extraction				
115	2302-CF	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	
10A	2302-CF	QuEChERS EXTRACTION WITH ACETONITRILE	(MgSO4=6g; NaCl=1,5g, H2O=10 mL, HCOOH=0,2 mL)			

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

Compound Feed (2302-CF)
Methods Perfluoroalkylcarboxylic acids (PFCAs), Perfluoroalkylsulfonic acids (PFSAs) and Other PFAS - Clean-up

LC	Sample	Clean-up				
4	2302-CF	Freezing of sample	Dispersive SPE (MgSO4, NaCl, C18, ENVI-carb)			
5	2302-CF	SPE using Oasis WAX 6 cc, 150 mg Sorbent, 30 µm particle size	Conditioning with 4 mL 0.1% NH3 in MeOH, 4 mL MeOH, 4 mL 25 mM CH3COONH4 in H2O (pH 4 using acetic acid); 6 mL extract + 4 mL H2O	Washing with 4 mL H2O, 4 mL Acetone/ACN (50/50), 4 mL MeOH; Elution with 2x 1.5 mL 0.1% NH3 in MeOH	Keeper (ethylene glycole, 50 µL) was used during evaporation	Reconstitution with MeOH/H2O (50/50)
8	2302-CF	Purification on SPE Envicarb	Evaporation to 10 ml	Mix with water and pH adapted to 3	Purification on SPE Agilent polymeric weak anion exchange 500mg	Evaporation to 0,1 ml, recovery standards added and MeOH added for total volume of 0,5ml; Injection in Polypropelyne vial
10	2302-CF	SPE CLEAN-UP WITH STRATA X-AW 200 mg - dSPE-Envicarb 80mg				
11B	2302-CF	extract cleaning: 2 g MgSO4, 0.5g NaCl, 0.1 g C 18, 0.1 g activated carbon, centrifugation 5 min./ 5000 rpm	evaporation to dryness: N2, 45°C	dissolv. in 250 ul of 0,5 % NH4OH in MeOH, vortex, 10 min. ultrasonic bath, centrifugation 5 min./ 15000 rpm		
12	2302-CF	Clean-up 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		
14	2302-CF	automated solid-phase extraction with two different SPE cartidges (1. ENVI-Carb and 2. STRATA-X AW)	Strata-X-GCB as a not retaining SPE, Strata-X-AW as retaining SPE			
18	2302-CF	SPE chromabond PFAS	SPE Envi Carb 500 mg			
22	2302-CF	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
27	2302-CF	2D SPE - OASIS WAX + STRATA GCB via ASPEC	Evaporation and reconsitution	Addition of recovery std.		
28	2302-CF	See above.				
30	2302-CF	Clean up with quechers salts and degreasing by freezing.				
36	2302-CF	PSA				
39	2302-CF	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.		
40	2302-CF	WAX columns coditioned with 0.1% NH4OH in MeOH and with water	after sample load elution with 4 mL MeOH nd 4mL 0.1% NH4OH in MeOH			
41	2302-CF	Weak anionic exchange (WAX) SPE plus graphitized carbon black (Envicarb)				
43	2302-CF	Quechers method				
47	2302-CF	SPE Clean Up (Waters Oasis WAX Cartriges)	The eluate was concentrated under a gentle stream of nitrogen by drying	the residue dissolved in 250 µL of methanol and water (50:50 v/v)		
53	2302-CF	pass the upper ACN phase trough a 250mgGCB SPE Cartriche	evaporate the ACN 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 2mL 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)
55	2302-CF	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 (4ml extract diluted with about 26 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
59	2302-CF	Clean-up with SPE (Strata X-AW)				
60	2302-CF	Clean up with Strata X SPE	Elution wit 0.1 % NH4OH in methanol	Adding 5 µL glycerol to eluate	evaporate t dryness	reconstitute in LC Eluents (90/10)
64	2302-CF	Filter				
67	2302-CF	Clean-up was performed by the QUECHERS method described in the EURL guidance document				
78	2302-CF	SPE WAX				
80	2302-CF	7 mL from upper layer; 300 mg PSA, 150 mg GCB	Vortex and shaker; Freeze for 30 min; Centrifuge; 1 mL for supernantant and dilution to 10 mL Water	Cleaning with SPE column 200 mg Sepra ZT WAX\ ; Condition with 0,3% NH3/MeOH, Water	Load sample and drying for 1 h	Elution 0,3% NH3/MeOH, evaporation and reconstitution with 200 uL MeOH-H2O (80/20, 0,3 % formic acid)
83	2302-CF	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.			
84	2302-CF	Cleaned with Envicarb				
87	2302-CF	0,5 ml were added to 2 ml acidified water on WAX cartridge (200 mg)	cleaning with 2 ml MeOH/water, 2 ml MeOH, 4 ml ACN/Aceton/acetic acid (50:50:1), 2 ml MeOH	elution with 1,6 ml 0,5 m NH3 in MeOH	evaporation to dryness and dissolve in 200 µl MeOH	
88	2302-CF	Strata X AW SPE cartridges were conditioned with 4 mL of MeOH followed by 4 mL of water and rinsed three times with the eluting mixture (3 × 6 mL of 2% NH4OH in MeOH).	The SPE cartridges were then re-conditioned again with 4 mL of MeOH and 4 mL of water. After sample loading, the cartridges were washed with 4 mL of sodium acetate 25 mM (pH = 4), 4 mL of MeOH 40% and 4 mL MeOH and dried under vacuum.	Finally, the analytes were eluted with 4 mL of 2% NH4OH in MeOH into a 15 mL tube containing 80 mg of d-SPE Envicarb and 100 µL of acetic acid.	After shaking and centrifugation (10 min at 3900 rpm), the supernatant was transferred into a 15 mL tube. After evaporation near-dryness in a current of nitrogen (at 40 °C), 0.2 mL of 80:20 MeOH/ammonium acetate 4 mM were added.	
91	2302-CF	degreasing using n-hexane	dispersive SPE using activated carbon			
92	2302-CF	Quechers Dispersive (150mg PSA - 900mg MgSO4 - 150mg C18 EC)				
93	2302-CF	1) Pre-conditioning of Oasis WAX 3cc with: 2 x 2.5 ml of Methanol and 2 x 2.5 ml of water under gravity conditions	2) Sample loading under vaccuum conditions 3) dryness under vacuum 4) Elution with 2 x 2.5 ml of methanol 0.1% NH4OH	5) Evaporation under nitrogen stream till 0.5 ml	6) Transfer to LC vial equipped with inserto and further evaporation till dryness under nitrogen stream	7) Reconstitution in 0.1 ml of methanol:water (1:9)
99	2302-CF	# SPE (Strata X-AW, 200 mg) # GCB 250 mg	# elution using 3 x 3 mL 1% 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)
107	2302-CF	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
108	2302-CF	After MgSO4 and NaCl is added, clean-up by using fluorisil and ENVI-carb, centrifugated multiple times	supernatant transfered multiple times, evaporated and reconstituted in methanol before injection on the	instrument.		
109	2302-CF	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.
113	2302-CF	frozen out in freezer (-80°C, 30 min), centrifugated	3ml of ACN phase was added to salt mix III (0.45gMgSO4, 0.075g d-SPE)	shaken, centrifugated and filled in ALS-Vials for chromatrography		
114	2302-CF	SPE Cleanup				
115	2302-CF	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
10A	2302-CF	SPE CLEAN-UP WITH STRATA X-AW 200 mg - dSPE-Envicarb 80mg				

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Compound Feed (2302-CF)
Methods Perfluoroalkylcarboxylic acids (PFCAs), Perfluoroalkylsulfonic acids (PFSAs) and Other PFAS - Chromatographic separation and detection

LC	Sample	**Chromatographic separation and detection**					
4	2302-CF	HPLC-column (retention): Luna Omega Polar C18 (1.6 µm, 100*2.1 mm)	HPLC-column (analytical): Acquity CSH C18 (1.7 µm, 100*2.1 mm)	Gradient with Methanol and Ammoniumacetate (2 mM)	MS/MS detection		
5	2302-CF	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	
8	2302-CF	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	
10	2302-CF	CHROMATOGRAPHIC COLUMN: Phenomenex 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
11B	2302-CF	A: 5mM NH4Ac in water, B: MeoH, column: Poroshell 120 EC-C18, delay column: Poroshell 120 EC-C18	MS: TSQ Endura triple quadrupole, MRM, only one ion transition for: PFBA, PFPeA, 9Cl-PF3ONS and	11Cl-PF3OUdS			
12	2302-CF	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	
14	2302-CF	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
18	2302-CF	LC-(ESI-)-MS/MS - Agilent 6495C	Hypersil Gold (100 x 2,1 mm) 1,9 µm				
22	2302-CF	HPLC-MS/MS, column BEH C18 100x2,1mm*1,7um,	mobile phase water/methanol with ammonium acetate				
27	2302-CF	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 -
28	2302-CF	Sample extracts are analyzed by LC/MS/MS operated in negative electrospray ionization (ESI) mode	for detection and quantification of analytes. Quantitative analysis is perfromed using isotope dilution.				
30	2302-CF	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)	
36	2302-CF	LC-MSMS					
39	2302-CF	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	
40	2302-CF	Luna Omega Polar 100x 2.1mm, 3um, precolunn Polar C18 4x2mm	A: 10mM NH4OH	B: 10mM NH4OH in methanol	flow 0.3 mL/min	injection volume 40 ul	
41	2302-CF	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-)			
43	2302-CF	UPLC-MS/MS; Aqcuity BeH column C18 1.7 um, 2.1 x100mm column for chromatographic separation; XEVO TQ-XS MS system for detection; MRM transitions used in ESI- mode.					
47	2302-CF	Alliance e2695 separation module coupled to a Xevo-TQD tandem-MS	using an UniSpray ionization source (Waters, Eschborn, Germany)				
53	2302-CF	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	
55	2302-CF	LC-MS/MS, reversed phase, column: Gemini 3µ C18 110Å, 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			
59	2302-CF	Chromatographic separation on a C18-RP-Column with a binary gradient elution	Detection on a triple quadrupole mass spectrometer				
60	2302-CF	Use of autosampler programm	use of delay column between pump an autosampler (C18, XBridge, 50 x 2.1 mm, 3,5 µm)	Eluent A: 2 mM ammoniumacetat and 5% acetonitril; Eluent B: acetonitril/methanol (60/40, v/v)	Flow: 0.25 mL/min; Column temperature: 50°C; Injection volume: 20 µL	Column: C18, XBridge, 150 x 2.1 mm, 3,5 µm	Gradient: 0 min: 70%A, 9 min: 25% A, 12 min: 5% A, 15 min: 2% A, 19,5 min: 2% A, 20,5 min: 90% A, 24 min: 90% A
64	2302-CF						
67	2302-CF	Sample extracts were analyzed by LC-MS/MS with ESI operating in negative mode. HPLC equiped with a C18 column was coupled to triple quadrupole.					
78	2302-CF	LC-MS/MS C18 reverse phase column -Ve mode.					
80	2302-CF	Delay Column: XBridgeTM C18 2.5 um 2.1x50 mm	Column: Xterra® MS C18 3.5 um 2.1x100 mm	Pre-Column: SecurityGuardTM Guard Cartridge Kit, Phenomenex	Modile phase: MeOH/Buffer 5 mM Ammonium Acetate, 5 mM 1-methyl-piperidine	Flow 0.3 mL/min	LC system: EXCION LC; MS system: AB Sciex QTRAP 6500+
83	2302-CF	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		
84	2302-CF	LC-MS/MS					
87	2302-CF	LC-MSMS API 4000 Sciex, MeOH/10 mmol NH4acetate gradient, column Gemini NX 100x3, 3µm	6point calibration				
88	2302-CF	Chromatographic separation was carried out on a Luna Omega PS C18 column 1.6 mm, 100 x 2.1 mm (Phenomenex). A delay column was installed downstream of the pumping system (PFAS delay COLUMN 2.1 × 50 mm C18 (Phenomenex)					
91	2302-CF	seperation on reversed phase C18	detection in multiple reaction monitoring mode (ESI neg)				
92	2302-CF	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		
93	2302-CF	Column: Hypersil GOLD PFP 50 x 3 (3 um)	Mobile phases: water 20mM NH4Ac / methanol 20 mM NH4Ac	Injection volume: 10 ul	Gradient conditions during 13 min	Ionisation: ESI negative	Analyser: QExactive; full scan (100 - 1000 Da) at 70,000 FWHM: Data dependant scan of the most intense ions
99	2302-CF	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
107	2302-CF	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
108	2302-CF	Ammoniumacetate as buffer, methanol as the organic mobile phase.	8 min separation on column going from 100% water to 95 % methanol and back.				
109	2302-CF	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-)			
113	2302-CF	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
114	2302-CF	Measurement: HPLC	Injection method: standard	Injection volume (µl): 5	Stationary phase: C18-Phase	Detector: MS/MS	Scan mode: MRM
115	2302-CF	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
10A	2302-CF	CHROMATOGRAPHIC COLUMN: Phenomenex 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

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food
21 June 2024

Annex 8: Overview participants' methods – Measurement uncertainty and Limit of Quantification

Test sample - Compound Feed (2302-CF)

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]

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

Compound Feed (2302-CF)

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
4	2302-CF	Calculated form triplicate analysis of the compound feed sample. LOQ corresponding to 10*S/N of result. The sample was analysed with and without addition of 1 µg/kg of PFAS.	Calculated from triplicate analysis of the compound feed sample.	
5	2302-CF	According to Guidance Document	Es gelten die extrapolierten Analysenspielräume des VDLUFA	
8	2302-CF	Lowest validated level of a similar procedure using Quecher extraction. Adapted for 1g matrix.	U= k*u=((2x CVrw) + biais)	
10	2302-CF	Spiked samples at the LOQ	From validation data including reproducibility and trueness contributions	
11B	2302-CF			PFAS were analyzed at the Water Research Institute (WRI) in the accredited (ISO/IEC 17025) National Water Reference Laboratory of Slovakia.
12	2302-CF			
14	2302-CF	Calibration curve, lowest calibation point	according to the guidance document	
18	2302-CF	S/n =10		
22	2302-CF	0,1 ppb		
27	2302-CF	NA - feed not validated - food method applied.	NA - feed not validated - food method applied.	
28	2302-CF	LOQ Based on level 1 of the initial calibration standard	2 times the standard deviation of the last 12 months of LCS/LCSD data results.	
30	2302-CF	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,	
36	2302-CF			
39	2302-CF	Performance of calibration standards; Batch blank level	Top down estimation from reproducibility of spiked samples	
40	2302-CF			
41	2302-CF		Application of Horwitz-Thompson equation	
43	2302-CF			
47	2302-CF			
53	2302-CF			
55	2302-CF	estimation of LOD and LOQ according to DIN 32645 (calibration method)		
59	2302-CF	DIN 32645	Nord-Test-approach	
60	2302-CF			
64	2302-CF			
67	2302-CF		Control chart of reference material	
78	2302-CF	10x baseline noise		
80	2302-CF			
83	2302-CF	LOD and LOQ were estimated based on analysis of 10 blank spiked samples	MU were estimated based on precision and truenes from fortyfication experiment	
84	2302-CF			
87	2302-CF	lowest validation level	according to the procedure of SANTE document for pesticide	overlaying substances at transitions of PFHxS, PFNS 1.transition, PFOS 1. transition
88	2302-CF	5 independent samples fortified at 0,20 µg/kg were analysed. 0,20 µg/kg fortification level met the identification requirements and accuracy and accuracy criteria, so it was set as LOQ of method.	A first assessment of the uncertainty value was obtained by combining the intermediate precision and trueness data.	
91	2302-CF			
92	2302-CF			
93	2302-CF	Spiking real matrix with the mixture of internal standards		
99	2302-CF	The LOQ was estimated using a S/N of 3 for the less intensive mrm transition.	top-down approach as discribed in the "Guidance document on measurment uncertainty for laboratories performing PCDD/F and PCB analysis" ; Sum parameter: U = square root of sum of squares of MU of indivitual PFAS	
107	2302-CF	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	
108	2302-CF			
109	2302-CF	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.	
113	2302-CF	during validation of PFAS in fish matrix	during validation of PFAS in fish matrix	
114	2302-CF			
115	2302-CF			
10A	2302-CF	Spiked samples at the LOQ	Horwitz	

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]

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

Compound Feed (2302-CF)

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)**
4	2302-CF		20	6	8	10	12			20		
5	2302-CF	88	88	88	88	88	88	88	88	88	88	88
8	2302-CF	30	30	30	30	30	30	30	30		30	30
10	2302-CF				31	33	35	36	44	40	59	43
11B	2302-CF											
12	2302-CF					20	20					
14	2302-CF			30	30	25	25	30	30	30	30	
18	2302-CF					18	12					
22	2302-CF											
27	2302-CF				35	35	35	50	50			
28	2302-CF	23	21	25	27	19	24	25	26	30	35	26
30	2302-CF	45	45	45	45	45	45	45	45	45	45	45
36	2302-CF					50	50					
39	2302-CF	30	30	30	30	30	30	30	30	30	30	30
40	2302-CF					20	20					
41	2302-CF	44	44	44	44	44	44	44	44			
43	2302-CF					20	23					
47	2302-CF	50	50	30	40	40	40	40	40	40	50	50
53	2302-CF											
55	2302-CF	40	40	40	40	40	40	40	40	40	40	40
59	2302-CF	20	20	20	20	20	20	20	20	20	20	20
60	2302-CF	50	50	50	50	50	50	50	50	50	50	50
64	2302-CF					40	40					
67	2302-CF					26	26					
78	2302-CF											
80	2302-CF											
83	2302-CF			29	30	34	29	34	28	30		
84	2302-CF			40	40	40	40	40	40	40	40	40
87	2302-CF	33.7	10	19.7	9.5	12.5	14.1	7.2	19.8	8.4	12.1	9.7
88	2302-CF					34	34					
91	2302-CF	45	45	20	20	25	20	25	45	45		
92	2302-CF	44	44	44	44	44	44	44	44			
93	2302-CF	11	6.75	24		10						
99	2302-CF	36	23	12	21	22	16	25	23			
107	2302-CF	11	30	20	32	34	22	25	31	41	22	6
108	2302-CF	37	37	37	37	37	37	37	37	37	37	37
109	2302-CF	45	30	31	48	27	43	29	27	39	27	32
113	2302-CF					36	38					
114	2302-CF	30	30	30	30	30	30	30	30	30	30	30
115	2302-CF					18	25					
10A	2302-CF	44	44	44	44	44	44	44	44	44	44	44

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]

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

Compound Feed (2302-CF)

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

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)**
4	2302-CF			0.294	0.962	0.136	0.116	0.234		0.08	0.045	0.044	0.055
5	2302-CF		0.4	0.05	0.2	0.05	0.2	0.05	0.05				
8	2302-CF		0.239	0.239	0.239	0.239	0.239	0.239	0.239	0.239		0.239	0.239
10	2302-CF												0.1
11B	2302-CF		0.381	0.381	0.381	0.381	0.381	0.381	0.381	0.381	0.381	0.381	0.381
12	2302-CF						0.01	0.01					
14	2302-CF				0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.05	
18	2302-CF										0.05	0.151	0.151
22	2302-CF		0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
27	2302-CF												
28	2302-CF		0.962	0.288	0.288	0.288	0.288	0.288	0.288	0.288	0.288	0.288	0.288
30	2302-CF		0.06	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03
36	2302-CF						0.097	0.097					
39	2302-CF		0.5	0.5	0.5	0.5	0.3	0.5	0.5	0.5	0.5	1	1
40	2302-CF						0.01	0.005					
41	2302-CF		0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
43	2302-CF		0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6
47	2302-CF		0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
53	2302-CF			0.1	0.1	0.1	0.1	0.1	1	1	0.2	2	2
55	2302-CF		0.16	0.3	0.05	0.06	0.06	0.03	0.12	0.07	0.09	0.03	0.05
59	2302-CF		0.12	0.1	0.05	0.05	0.05	0.05	0.05	0.05	0.32	0.27	0.24
60	2302-CF				0.2	0.2	0.2	0.2	0.2				
64	2302-CF						0.5	0.5					
67	2302-CF												
78	2302-CF												
80	2302-CF		0.25	0.5	0.05	0.01	0.02	0.1	0.01	0.05	0.01	0.015	0.02
83	2302-CF				0.01	0.01	0.01	0.01	0.01	0.01	0.01		
84	2302-CF				0.04	0.02	0.02	0.02	0.02	0.02	0.02	0.04	0.04
87	2302-CF									0.25	0.25	0.25	0.25
88	2302-CF						0.2	0.2					
91	2302-CF		0.1	0.05	0.03	0.03	0.03	0.04	0.02	0.03	0.02		
92	2302-CF		0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05		
93	2302-CF		1	ERROR	0.1	0.1	0.1	0.5	0.5	1	1.5	1.5	1.5
99	2302-CF										0.0647	0.0262	0.0372
107	2302-CF		0.5	0.5	0.1	0.05	0.05	0.1	0.1	0.05	0.1	0.5	0.5
108	2302-CF		0.3	0.1	0.1	0.01	0.01	0.005	0.01	0.01	0.1	0.01	0.1
109	2302-CF		1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
113	2302-CF						0.1	0.1					
114	2302-CF		0.3	0.3	0.1	0.1	0.3	0.3	0.1	0.1	0.1	0.3	0.3
115	2302-CF						0.1	0.1					
10A	2302-CF									0.1	0.1	0.1	0.1

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]

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

Compound Feed (2302-CF)

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)**
4	2302-CF						10							
5	2302-CF			88			88		88					
8	2302-CF		30	30	30	30	30		30	30	45	45	60	60
10	2302-CF		40	34	53	43	43	55	39	60	41		58	
11B	2302-CF													
12	2302-CF				20				20					
14	2302-CF		30	30	25	30	25	25	25	30	30		30	
18	2302-CF				18		23		19					
22	2302-CF													
27	2302-CF													
28	2302-CF		28	24	25	24			27	23	25		35	
30	2302-CF		45	45	45	45	45	45	45	45	45	45	45	45
36	2302-CF				50				50					
39	2302-CF		30	30	30	40	30	30	30	40	40		40	
40	2302-CF				20		20	20	20					
41	2302-CF						44	44	44					
43	2302-CF				36		39							
47	2302-CF		40	50	40	50			40	50	50			
53	2302-CF													
55	2302-CF		40	40	40	40	40		40	40	40	40	40	40
59	2302-CF		20	20	20	20	20	20	20	25	20		20	
60	2302-CF		50	50	50	50	50	50	50	50	50			
64	2302-CF				40				40					
67	2302-CF				26		26							
78	2302-CF													
80	2302-CF													
83	2302-CF		22	28	41	17	20	20	29					
84	2302-CF				40	40	40	40	40					
87	2302-CF		14.8	24.6	16.7	13.4			4.6	12.9	13.2			
88	2302-CF				34		34	34						
91	2302-CF						40							
92	2302-CF		44		44				44					
93	2302-CF						19.83		19.83					
99	2302-CF						20							
107	2302-CF		26	7	34	6	26	26	26	8	33			
108	2302-CF		37	37	37	37	37	37	37	37	37	37	37	37
109	2302-CF		29	32	33	33	27			26	31		45	
113	2302-CF				34				32					
114	2302-CF		30	30	30	30	30	30	30	30	30	30	30	30
115	2302-CF				15				16					
10A	2302-CF		44	44	44	44	44	44	44	44	44		44	

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

Compound Feed (2302-CF)

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

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)**
4	2302-CF		0.138		0.167		0.561				0.51			
5	2302-CF		0.05	0.05	0.05	0.05	0.05		0.05	0.2	0.2			
8	2302-CF		0.239	0.239	0.239	0.239	0.239		0.239	0.239	0.239	0.478	0.478	0.717
10	2302-CF		0.1	0.1	0.1	0.1		0.1		0.1	0.1		0.1	
11B	2302-CF		0.381	0.381	0.381	0.381			0.381	0.381	0.381	0.381	0.381	0.381
12	2302-CF				0.01				0.01					
14	2302-CF		0.02	0.05	0.02	0.02	0.02	0.02	0.02	0.05	0.05		0.1	
18	2302-CF		0.094	0.047	0.095	0.096				0.019	0.019	0.02	0.049	0.049
22	2302-CF		0.1	0.1	0.1	0.1	0.1		0.1	0.1	0.1		0.1	
27	2302-CF													
28	2302-CF		0.962	0.288	0.288	0.288			0.288	0.288	0.288		0.962	
30	2302-CF		0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03
36	2302-CF				0.097				0.097					
39	2302-CF		0.5	0.5	0.5	0.5	1	1	1	0.5	0.5		1	
40	2302-CF				0.01		0.01	0.01	0.01					
41	2302-CF		0.025	0.025	0.025	0.025	0.025	0.025	0.025	0.025	0.025	0.025	0.025	0.025
43	2302-CF		0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6
47	2302-CF		0.1	0.1	0.1	0.1			0.1	0.1	0.1			
53	2302-CF		0.05	0.05	0.1	0.2	0.02	0.05		0.05	0.05	0.2	2	2
55	2302-CF		0.02	0.02	0.02	0.07	0.04		0.04	0.04	0.05	0.05	0.05	0.1
59	2302-CF		0.06	0.2	0.08	0.25	0.05	0.05	0.05	0.1	0.24		0.2	
60	2302-CF						0.2	0.2	0.2					
64	2302-CF				0.5				0.5					
67	2302-CF													
78	2302-CF		0.004											
80	2302-CF		0.05	0.02	0.02	0.02	0.05	0.05	0.05	0.02	0.02			
83	2302-CF		0.02	0.05	0.015	0.07	0.01	0.01						
84	2302-CF				0.02	0.02	0.02	0.05	0.02					
87	2302-CF		0.25	0.25	0.25	0.25				0.25	0.25			
88	2302-CF				0.2		0.2	0.2						
91	2302-CF		0.05	0.05	0.05	0.05	0.05	0.05		0.05	0.1			
92	2302-CF		0.05		0.05				0.05					
93	2302-CF		1	1	0.1	0.1	0.1			0.1	0.1			
99	2302-CF		0.136	0.315	0.0815	0.18		0.0968		0.0524	0.0414	0.0894		0.201
107	2302-CF		0.05	0.1	0.05	0.1	0.05	0.05	0.05	0.1	0.1			
108	2302-CF		0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	
109	2302-CF		0.1	0.1	0.1	0.1	0.1			0.1	0.1		0.1	
113	2302-CF				0.1		0.1	0.1	0.1					
114	2302-CF		0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
115	2302-CF				0.1				0.1					
10A	2302-CF		0.1	0.1	0.1	0.1				0.1	0.1		0.1	

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]

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

Compound Feed (2302-CF)

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)**
4	2302-CF		13	13
5	2302-CF		88	88
8	2302-CF		20	20
10	2302-CF		21	22
11B	2302-CF			
12	2302-CF		20	20
14	2302-CF		25	25
18	2302-CF		12	12
22	2302-CF			
27	2302-CF			
28	2302-CF		27	27
30	2302-CF		30	30
36	2302-CF			50
39	2302-CF		30	30
40	2302-CF		20	20
41	2302-CF		28.4	28.6
43	2302-CF			
47	2302-CF		40	40
53	2302-CF			
55	2302-CF		40	40
59	2302-CF		20	20
60	2302-CF		50	50
64	2302-CF		40	40
67	2302-CF		26	26
78	2302-CF			
80	2302-CF			
83	2302-CF		67	67
84	2302-CF		40	40
87	2302-CF		4.2	4.2
88	2302-CF			
91	2302-CF			
92	2302-CF		44	44
93	2302-CF			
99	2302-CF			34
107	2302-CF		34	34
108	2302-CF		37	37
109	2302-CF		18.4	18.9
113	2302-CF		22	22
114	2302-CF			
115	2302-CF		38	38
10A	2302-CF		27	28

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]

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

Compound Feed (2302-CF)

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

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**
4	2302-CF								
5	2302-CF			0.05	0.05	0.05	0.05		
8	2302-CF			0.24		0.24	0.24		
10	2302-CF		0.05	0.20	0.50	0.20	0.20		
11B	2302-CF			0.57	0.57	0.48	0.48		
12	2302-CF								
14	2302-CF								
18	2302-CF					0.05	0.05		
22	2302-CF								
27	2302-CF								
28	2302-CF		0.29						
30	2302-CF		0.03	0.03	0.03	0.03	0.03	0.03	0.03
36	2302-CF								
39	2302-CF		0.50	0.05	0.05	0.05	0.05		
40	2302-CF								
41	2302-CF		0.20	0.05	0.20	0.05	0.05		
43	2302-CF		0.60	0.60	0.60				
47	2302-CF								
53	2302-CF		2.00	0.10	2.00	0.10	0.10	0.10	0.10
55	2302-CF		0.04	0.03	0.04	0.04	0.03		0.56
59	2302-CF			0.20	0.10				
60	2302-CF								
64	2302-CF								
67	2302-CF								
78	2302-CF								
80	2302-CF		0.04	0.01	0.05	0.10			
83	2302-CF								
84	2302-CF								
87	2302-CF								
88	2302-CF								
91	2302-CF			0.05	0.10	0.05			
92	2302-CF				0.05				
93	2302-CF								
99	2302-CF		0.17	0.04	3.34	0.16	0.09		
107	2302-CF								
108	2302-CF		0.01	0.10	1.00				
109	2302-CF								
113	2302-CF								
114	2302-CF		0.02	0.10	0.50	0.50	0.50		
115	2302-CF								
10A	2302-CF								

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Compound Feed 2023 [EURL-PT-POP_2302-CF]

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

Compound Feed (2302-CF)

Methods Other PFAS - Measurement Uncertainty MU [%]

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**
4	2302-CF								
5	2302-CF								
8	2302-CF			30		30	50		
10	2302-CF		44	44	44	44	44		
11B	2302-CF								
12	2302-CF								
14	2302-CF								
18	2302-CF								
22	2302-CF								
27	2302-CF								
28	2302-CF		24						
30	2302-CF		45	45	45	45	45	45	45
36	2302-CF								
39	2302-CF		30	30	30	30	30		
40	2302-CF								
41	2302-CF								
43	2302-CF								
47	2302-CF								
53	2302-CF								
55	2302-CF		40	40	40	40	40		40
59	2302-CF			20	20				
60	2302-CF								
64	2302-CF								
67	2302-CF								
78	2302-CF								
80	2302-CF								
83	2302-CF								
84	2302-CF								
87	2302-CF								
88	2302-CF								
91	2302-CF								
92	2302-CF								
93	2302-CF								
99	2302-CF								
107	2302-CF								
108	2302-CF		37	37	37				
109	2302-CF								
113	2302-CF								
114	2302-CF								
115	2302-CF								
10A	2302-CF								