EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2022

EURL-PT-POP_2203-FM

FEED

Report

PFASs

(Report Version 1.0)

03 August 2023

EURL for halogenated POPs in Feed and Food
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Summary

Test sample	**FEED:** Fish Meal [2203-FM]
Analytes of interest	**Main PFASs** (L-PFOS, PFOA, PFNA, PFHxS, Sum of L-PFOS, PFOA, PFNA, PFHxS) **Other PFASs** (perfluoroalkylcarboxylic acids, perfluoroalkylsulfonic acids, perfluoroalkane sulfonamides)
Methods	Any kind of method
Participants	NRLs, OFLs, other official laboratories, commercial laboratories performing the analysis of samples taken by feed business operators
Statistical evaluation	ISO 13528:2022 [1], IUPAC Protocol [2]
Report of final results	03 August 2023 (Version 1.0)
Publication	EURL POPs reserves all rights to publish and present the anonymised results of the interlaboratory study in scientific journals and/or during conferences.

1. Structure of the ILS, test material and analytes

This proficiency test (PT) on the determination of **PCDD/Fs, PCBs, PBDEs, HBCDDs** and **PFASs** in **fish meal** 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 2022. 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 **fish meal**.

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 2022. 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 2022.

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.

Fish Meal	**Sample no. 2203-FM-xxx**

1.2. Analytes of interest

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

- **Main PFASs**
  - Linear perfluoroctanesulfonic acid (L-PFOS), perfluorooctanoic acid (PFOA), perfluorononanoic acid (PFNA), perfluorohexanesulfonic acid (PFHxS)
  - Sum of L-PFOS, PFOA, PFNA, PFHxS
  - Sum of total[1] PFOS, PFOA, PFNA, PFHxS
  - Total[1] PFOS (sum of L- and br-PFOS)

- **Other PFASs**
  - **Perfluoroalkylsulfonic acids (PFSAs):** perfluorobutanesulfonic acid (PFBS), perfluoropentanesulfonic acid (PFPeS), perfluoroheptanesulfonic acid (PFHpS), branched perfluoroctanesulfonic acids (br-PFOS), perfluorononanesulfonic acid (PFNS), perfluorodecanesulfonic acid (PFDS), perfluoroundecane sulfonic acid (PFUnDS), perfluorododecane sulfonic acid (PFDoDS), perfluorotridecane sulfonic acid (PFTrDS)
  - **Perfluoroalkylcarboxylic acids (PFCAs):** perfluorobutanoic acid (PFBA), perfluoropentanoic acid (PFPeA), perfluorohexanoic acid (PFHxA), perfluoroheptanoic acid (PFHpA), perfluorodecanoic acid (PFDA), perfluoroundecanoic acid (PFUnDA), perfluorododecanoic acid (PFDoDA), perfluorotridecanoic acid (PFTrDA), perfluorotetradecanoic acid (PFTeDA)
  - Perfluorooctane sulphonamide **(FOSA)**

1.3. Methods

All kinds of detection and quantification methods could be applied.

1.4. Coding of laboratories and confidentiality

The laboratory code of the participating laboratories will be kept confidential and will not be revealed to other participants. The confidentiality between NRLs and their OFLs will be kept unless a Member State initiated a cooperation between the NRL, OFLs and the EURL.
For NRLs, the “Protocol for management of underperformance in comparative testing and/or lack of collaboration of National Reference Laboratories (NRLs) with Community reference laboratories (CRLs) activities” will be observed. The confidentiality of NRLs will be kept according to this protocol.

[1] Sum of all PFOSs, whether they are chromatographically separated or not

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

113 laboratories registered for this proficiency test and 34 reported results for at least one PFAS parameter.

Table 1: Participating laboratories

Participating laboratories	Region	No. of participants
National Reference Laboratories	European Union	11
	Other Countries	2
Official Laboratories	European Union	16
	Other European Countries	-
	Africa	-
	Americas	-
	Asia	-
	Oceania	1
Commercial Laboratories	European Union	3
	Other European Countries	-
	Africa	-
	Americas	1
	Asia	-
	Oceania	-
	Total	**34**

2.1. Number of reported results

Table 2: Reported results for individual PFASs for fish meal (2203-FM) of all laboratories

Analyte	Abbreviation	2203-FM
Perfluorobutanoic acid	(PFBA)	24
Perfluoropentanoic acid	(PFPeA)	26
Perfluorohexanoic acid	(PFHxA)	29
Perfluoroheptanoic acid	(PFHpA)	30
Perfluorooctanoic acid	(PFOA)	33
Perfluorononanoic acid	(PFNA)	33
Perfluorodecanoic acid	(PFDA)	30
Perfluoroundecanoic acid	(PFUnDA)	30
Perfluorododecanoic acid	(PFDoDA)	29
Perfluorotridecanoic acid	(PFTrDA)	26
Perfluorotetradecanoic acid	(PFTeDA)	26
Perfluorobutanesulfonic acid	(PFBS)	29
Perfluoropentanesulfonic acid	(PFPeS)	25
Perfluorohexanesulfonic acid	(PFHxS)	33
Perfluoroheptanesulfonic acid	(PFHpS)	28
Linear Perfluorooctanesulfonic acid	(L-PFOS)	31
Perfluorononanesulfonic acid	(PFNS)	22
Perfluorodecanesulfonic acid	(PFDS)	27
Perfluoroundecanesulfonic acid	(PFUnDS)	10
Perfluorododecanesulfonic acid	(PFDoDS)	17
Perfluorotridecanesulfonic acid	(PFTrDS)	9
Perfluorooctane sulphonamide	(FOSA)	14

Table 3: Reported results for PFASs sum parameters for fish meal (2203-FM) of all laboratories

Analyte	2203-FM
Sum of branched perfluorooctanesulfonic acids (br-PFOS)	25
Sum of branched & linear perfluorooctanesulfonic acids (Total-PFOS)	28
Sum of L-PFOS, PFOA, PFNA, PFHxS (upper bound)	31
Sum of L-PFOS, PFOA, PFNA, PFHxS (lower bound)	30
Sum of Total-PFOS, PFOA, PFNA, PFHxS (upper bound)	26
Sum of Total-PFOS, PFOA, PFNA, PFHxS (lower bound)	25

2.2. Accreditation

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

Fish Meal	PFASs
Accreditation	18
No accreditation	13

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 2203-FM were analyzed in duplicate for individual PFASs. The test for sufficient homogeneity was performed for the individual substances and sum parameters. The test materials showed sufficient homogeneity for PFASs 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 PFASs.

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 the perfluoroalkylcarboxylic acids PFOA, PFNA, PFDA, PFUnDA, PFDoDA and PFTrDA, for the perfluoroalkylsulfonic acids PFHxS, L-PFOS and total PFOS (sum of branched and linear Perfluorooctanesulfonic acids), for perfluorooctane sulphonamide (FOSA) and for the sum parameters sum of L-PFOS, PFOA, PFNA, PFHxS and sum of total-PFOS, PFOA, PFNA, PFHxS in the test sample "fish meal" (2203-FM), including limits of quantification (LOQs). Assigned values could not be calculated for all other perfluoroalkylcarboxylic acids and perfluoroalkylsulfonic acids 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 L-PFOS, PFOA, PFNA, PFHxS and sum parameters (sum of branched and linear Perfluorooctanesulfonic acids, sum of L-PFOS, PFOA, PFNA, PFHxS and sum of Total-PFOS, PFOA, PFNA, PFHxS). Only 18 of 34 reporting laboratories were accredited according to ISO/IEC 17025 for PFAS in feed. After eliminating outliers, 14 to 16 results contributed to the calculation of the assigned values from the ISO/IEC 17025 group. No significant differences (3 - 7 %) between the assigned values calculated for both data sets for PFASs were observed for most of the analytes. Only for PFOA and PFHxS slightly higher deviations of 13% were observed, due to the low contamination level (Table 5).

Table 5: Comparison of assigned values for 2203-FM 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	0.374	0.327	-13
PFNA	1.03	0.958	-7
PFHxS	0.140	0.122	-13
L-PFOS	4.84	4.68	-3
Total PFOS	5.13	4.92	-4
Sum of L-PFOS, PFOA, PFNA, PFHxS (ub)	6.56	6.21	-5
Sum of L-PFOS, PFOA, PFNA, PFHxS (lb)	6.52	6.31	-3
Sum of total PFOS, PFOA, PFNA, PFHxS (ub)	6.87	6.40	-7
Sum of total PFOS, PFOA, PFNA, PFHxS (lb)	6.75	6.50	-4

PFASs – individual substances and sum parameter

The assigned values for the test sample 2203-FM were calculated as consensus of participants' results for individual PFASs and sum parameters, taking into account the calculation criteria described above (Table 6 - 9 ; tabular summary see annex 1; Figure 1-3).

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

Fish meal (2203-FM)	Assigned value PFCAs µg/kg product (12% moisture content)
PFOA	0.374
PFNA	1.03
PFDA	0.282
PFUnDA	0.455
PFDoDA	0.117
PFTrDA	0.240

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

Fish meal (2203-FM)	Assigned value PFSAs µg/kg product (12% moisture content)
PFHxS	0.140
L-PFOS	4.84
Total-PFOS	5.13

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

Fish meal (2203-FM)	Assigned value µg/kg product (12% moisture content)
Sum of L-PFOS, PFOA, PFNA, PFHxS (ub)	6.56
Sum of L-PFOS, PFOA, PFNA, PFHxS (lb)	6.52
Sum of Total PFOS, PFOA, PFNA, PFHxS (ub)	6.87
Sum of Total PFOS, PFOA, PFNA, PFHxS (lb)	6.75

Table 9: Assigned values for Perfluorooctane sulphonamide (FOSA; rounded to three significant figures)

Fish meal (2203-FM)	Assigned value µg/kg product (12% moisture content)
FOSA	0.445

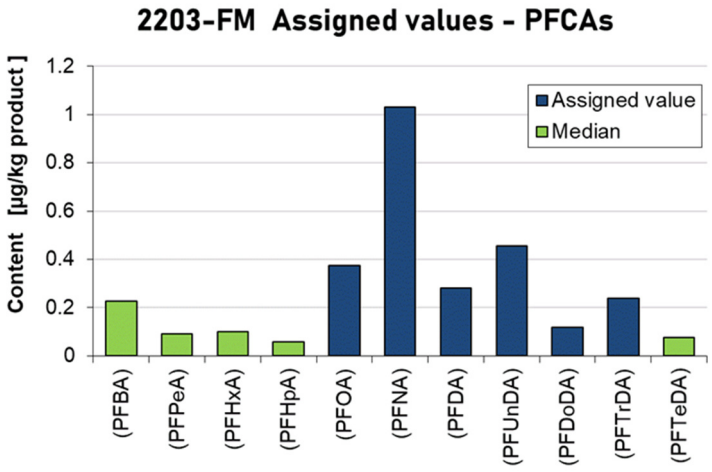

Figure 1: Assigned values (blue) and median values (green) for PFCAs individual substances for fish meal (2203-FM) [µg/kg product (12% moisture content)]

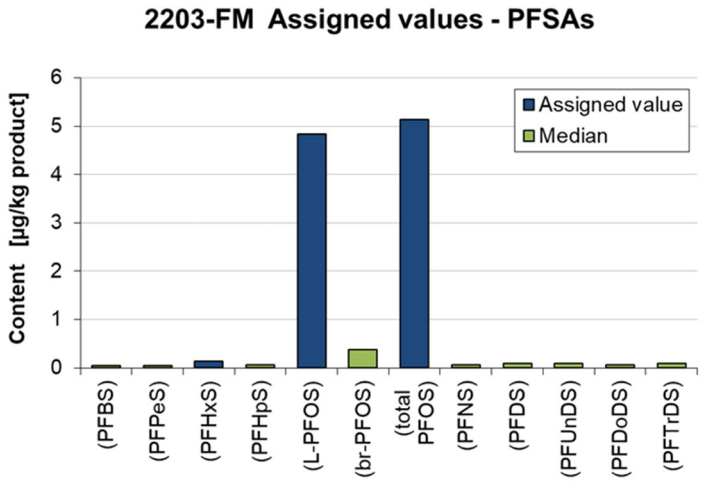

Figure 2: Assigned values (blue) and median values (green) for PFSAs individual substances for fish meal (2203-FM) [µg/kg product (12% moisture content)]

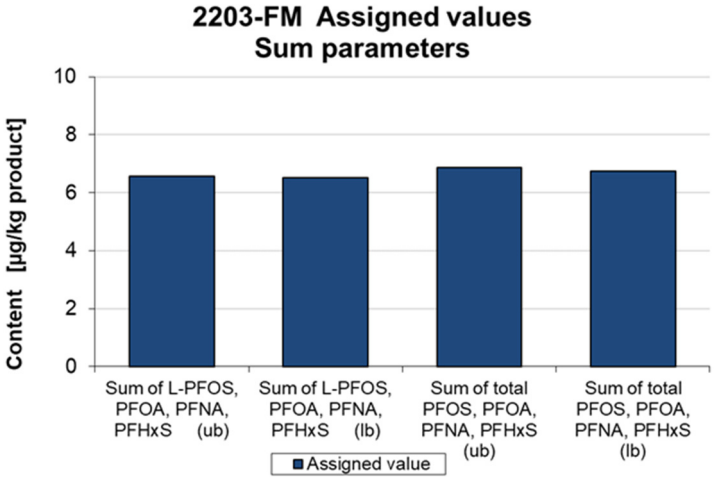

Figure 3: Assigned values for sum parameters of L-PFOS, PFOA, PFNA and PFHxS and total-PFOS, PFOA, PFNA and PFHxS for fish meal (2203-FM) [µg/kg product (12% moisture content)]

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)*

PFASs - Participants' z-scores

Z-scores for individual substances and sum parameters were within the range of ± 2 for 82% of all participants (Table 10 - 13; tabular summary see annex 3; Figure 4-6).

Table 10: Distribution of participants' z-scores for PFCAs for fish meal (2203-FM)

Percentage of participants' results PFCAs	$\lvert z\text{-score} \rvert \leq 2$	$2 < \lvert z\text{-score} \rvert < 3$	$\lvert z\text{-score} \rvert \geq 3$
PFOA	94%	3%	3%
PFNA	88%	9%	3%
PFDA	89%	7%	4%
PFUnDA	97%	-	3%
PFDoDA	90%	10%	-
PFTrDA	65%	18%	17%

Table 11: Distribution of participants' z-scores for PFSAs for fish meal (2203-FM)

Percentage of participants' results PFSAs	|z-score| ≤ 2	2 < |z-score| < 3	|z-score| ≥ 3
PFHxS	88%	4%	8%
L-PFOS	87%	7%	6%
Total-PFOS	75%	14%	11%

Table 12: Distribution of participants' z-scores for sum parameters for fish meal (2203-FM)

Percentage of participants' results Sum parameters	|z-score| ≤ 2	2 < |z-score| < 3	|z-score| ≥ 3
Sum of L-PFOS, PFOA, PFNA, PFHxS (ub)	90%	3%	7%
Sum of L-PFOS, PFOA, PFNA, PFHxS (lb)	90%	3%	7%
Sum of total-PFOS, PFOA, PFNA, PFHxS (ub)	84%	12%	4%
Sum of total-PFOS, PFOA, PFNA, PFHxS (lb)	84%	12%	4%

Table 13: Distribution of participants' z-scores for FOSA for fish meal (2203-FM)

Percentage of participants' results PFSAs	|z-score| ≤ 2	2 < |z-score| < 3	|z-score| ≥ 3
FOSA	90%	-	10%

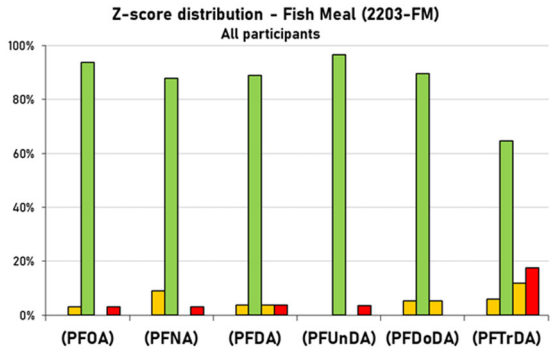

Figure 4: Distribution of participants' z-scores for individual PFCAs for fish meal (2203-FM) [Green bars: -2 ≤ z-score ≤ 2, orange bars: -3 < z-score < -2, 2 < z-score < 3, red bars: z-score ≤ -3, z-score ≥ 3]

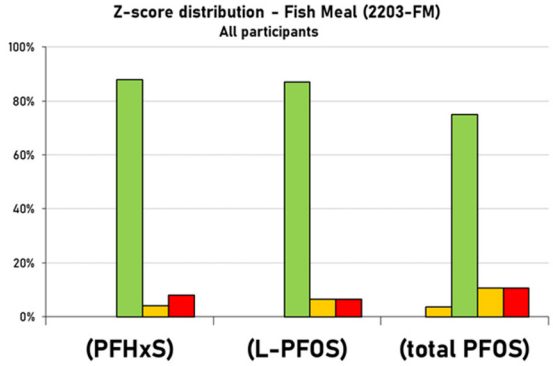

Figure 5: Distribution of participants' z-scores for individual PFSAs for fish meal (2203-FM) [Green bars: -2 ≤ z-score ≤ 2, orange bars: -3 < z-score < -2, 2 < z-score < 3, red bars: z-score ≤ -3, z-score ≥ 3]

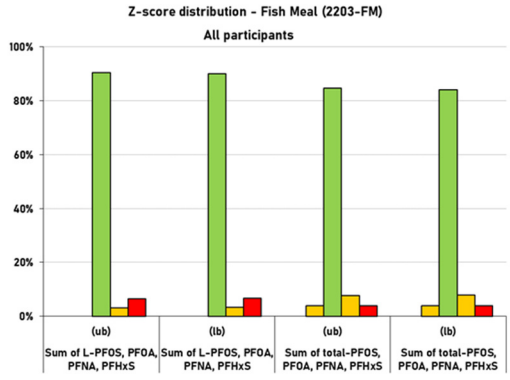

Figure 6: Distribution of participants' z-scores for sum parameters for fish meal (2203-FM) [Green bars: -2 ≤ z-score ≤ 2, orange bars: -3 < z-score < -2, 2 < z-score < 3, red bars: z-score ≤ -3, z-score ≥ 3]

6. Participants' feedback

A questionnaire for feedback from participants of this EURL proficiency test was available as online survey between 28 November 2022 and 23 January 2023. The survey was anonymous, but participants could also give their laboratory name. The identity of the laboratories is kept confidential. The survey included several questions related to different topics (participants' information, organization of the proficiency test, PT test samples and evaluation of results and summary of data) and a possibility to include comments and further suggestions.

In total, 9 laboratories (8 % of all PT participants) replied to this survey.

Table 14: Participating laboratories in the feedback survey

Type of laboratory	Answers
National Reference Laboratory (NRL)	2
Official Laboratory (OFL)	5
Commercial laboratory	2
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	

The following comments or suggestions for improvements were submitted:
"To the EUSurvey webtool: the person-related application is impractical; it would be better to create an access option for several people in a laboratory."; "timing to perform test was very good (enough time); timing to send the preliminary results was very good (short)"

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 fish meal (2203-FM) 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

Fish meal – 2203-FM	
Annex 1	Assigned values – PFCAs, PFSAs, sum parameters and FOSA
Annex 2	Participants' results – Tables – PFCAs, PFSAs, sum parameters and FOSA
Annex 3	Participants' z-scores – Tables – PFCAs, PFSAs, sum parameters and FOSA
Annex 4	Participants' z-scores – Charts – PFCAs, PFSAs, sum parameters and FOSA
Annex 5	Test for sufficient homogeneity – PFASs
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
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EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2022 [EURL-PT-POP_2203-FM]

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

03 August 2023

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

Test sample - Fish Meal (2203-FM)

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 Fish Meal 2022 [EURL-PT-POP_2203-FM]

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

Fish Meal (2203-FM)

Perfluoroalkylcarboxylic acids (PFCAs) - Assigned values

Analyte		Result µg/kg (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)						0.226
Perfluoropentanoic acid	(PFPeA)						0.0903
Perfluorohexanoic acid	(PFHxA)						0.100
Perfluoroheptanoic acid	(PFHpA)						0.0587
Perfluorooctanoic acid	(PFOA)		0.374	0.077	0.018	30	0.374
Perfluorononanoic acid	(PFNA)		1.03	0.27	0.059	32	1.05
Perfluorodecanoic acid	(PFDA)		0.282	0.060	0.015	27	0.290
Perfluoroundecanoic acid	(PFUnDA)		0.455	0.11	0.026	28	0.475
Perfluorododecanoic acid	(PFDoDA)		0.117	0.031	0.0085	21	0.122
Perfluorotridecanoic acid	(PFTrDA)		0.240	0.074	0.021	19	0.255
Perfluorotetradecanoic acid	(PFTeDA)						0.0760

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2022 [EURL-PT-POP_2203-FM]

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

Fish Meal (2203-FM)

Perfluoroalkylsulfonic acids (PFSAs) - Assigned values

Analyte		Result µg/kg (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)						0.0500
Perfluoropentanesulfonic acid	(PFPeS)						0.0500
Perfluorohexanesulfonic acid	(PFHxS)		0.140	0.039	0.010	26	0.140
Perfluoroheptanesulfonic acid	(PFHpS)						0.0670
Linear Perfluorooctanesulfonic acid	(L-PFOS)		4.84	0.76	0.18	29	5.00
Sum of branched Perfluorooctanesulfonic acids	(br-PFOS)						0.380
Sum of branched and linear perfluorooctanesulfonic acids	(total PFOS)		5.13	1.1	0.27	25	5.32
Perfluorononanesulfonic acid	(PFNS)						0.0590
Perfluorodecanesulfonic acid	(PFDS)						0.0980
Perfluoroundecane sulfonic acid	(PFUnDS)						0.0990
Perfluorododecane sulfonic acid	(PFDoDS)						0.0700
Perfluorotridecane sulfonic acid	(PFTrDS)						0.100

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2022 [EURL-PT-POP_2203-FM]

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

Fish Meal (2203-FM)

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

Analyte		Result µg/kg (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 L-PFOS, PFOA, PFNA, PFHxS	(ub)		6.56	1.2	0.28	29	6.79
Sum of L-PFOS, PFOA, PFNA, PFHxS	(lb)		6.52	1.0	0.25	28	6.69
Sum of total PFOS, PFOA, PFNA, PFHxS	(ub)		6.87	1.6	0.41	25	6.89
Sum of total PFOS, PFOA, PFNA, PFHxS	(lb)		6.75	1.3	0.34	23	6.79

Fish Meal (2203-FM)

FOSA - Assigned values

Analyte		Result µg/kg (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)		0.445	0.073	0.029	10	0.445

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2022 [EURL-PT-POP_2203-FM]

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

03 August 2023

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

Test sample - Fish Meal (2203-FM)

* 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 Fish Meal 2022 [EURL-PT-POP_2203-FM]

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

Fish Meal (2203-FM)

Perfluoroalkylcarboxylic acids (PFCAs) - Results

LC	Sample	Result µg/kg (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)
1	2203-FM		0.27	< 0.03	< 0.03	0.06	0.36	1.15	0.29	0.53	0.138	0.38	0.073
3	2203-FM		2.96	< 0.05	< 0.03	0.0536	0.232	0.695	0.188	0.317	0.0681		
4	2203-FM			< 0.05	< 0.05	< 0.05	0.295	1.07	0.214	0.295	< 0.05	0.259	< 0.05
5	2203-FM		2.78	< 0.0493	< 0.0493	0.12	0.456	1.45	0.535	0.872	0.169	0.444	0.079
7	2203-FM		< 0.227	< 0.0806	< 0.155	0.0573	0.331	0.99	0.264	0.415	0.1	0.211	0.0431
8	2203-FM		< 0.008	0.822	1.75	< 0.033	0.48	0.602	< 0.069	0.28		< 0.13	< 0.14
16	2203-FM			< 0.049	< 0.049	0.056	0.392	1.2	0.351	0.528	0.119	< 0.293	
20	2203-FM		< 0.3	< 0.1	< 0.1	< 0.01	0.38	1	0.29	0.47	0.11	0.25	0.058
23	2203-FM		< 2		< 1	< 0.5	< 0.7	1.4	0.4	< 1	< 0.5	< 0.5	< 0.2
25	2203-FM						0.51	1.18					
26	2203-FM		< 0.07	< 0.07	< 0.07	< 0.07	0.28	0.784	0.264	0.402	< 0.07	< 0.07	< 0.07
28	2203-FM		0.107	0.0551	< 0.0437	< 0.0437	0.376	1.19	0.335	0.502	0.13	0.401	< 0.0873
29	2203-FM		< 0.44	< 0.88	< 0.18	0.0389	0.284	0.965	0.278	0.456	0.109	0.133	< 0.06
43	2203-FM		< 0.225	< 0.0676	< 0.0676	< 0.0676	0.353	0.779	0.232	0.349	0.112	0.161	< 0.0676
46	2203-FM				< 0.03	0.053	0.411	1.2	0.31	0.549	0.137		
49	2203-FM						2.08	0.99					
56	2203-FM		< 5	< 2	< 0.5	< 0.5	0.451	1.3	< 0.5	0.634	< 0.5	< 1	< 1
59	2203-FM					< 0.3	0.401	2.06	0.201	0.587	< 0.25		< 0.25
62	2203-FM			< 0.75	0.629	< 0.05	0.389	1.39	0.302	0.592	< 0.05		
63	2203-FM				< 0.1	< 0.1	0.5	0.97	0.24	0.49	< 0.1	0.3	< 0.1
67	2203-FM						0.372	1.36					
68	2203-FM		< 0.3	< 0.3	< 0.3	< 0.3	0.34	1.05	< 0.3	0.44	< 0.3	< 0.3	< 0.3
72	2203-FM		0.021	< 0.02	< 0.02	0.057	0.332	0.902	0.254	0.414	0.122	0.436	0.047
75	2203-FM		< 0.1	< 0.1	< 0.1	0.043	0.19	0.75	0.214	0.32	0.095	0.19	0.037
76	2203-FM		< 0.2	< 0.2	< 0.2	< 0.2	0.356	1.18	0.373	0.564	< 0.2	< 0.2	< 0.2
80	2203-FM		< 0.5	< 0.5	< 0.1	0.052	0.28	0.87	0.24	0.39	0.12	< 0.5	< 0.5
83	2203-FM		< 0.2	< 0.2	< 0.2	0.077	0.363	1.02	0.285	0.444	0.126	0.249	< 0.2
84	2203-FM		< 1.25	< 0.5	< 0.025	0.068	0.338	0.981	0.3	0.482	0.12	0.248	0.047
85	2203-FM		< 0.05	< 0.01	< 0.01	0.0387	0.261	0.601	0.225	0.33	0.0813	0.19	< 0.05
92	2203-FM		< 0.1	< 0.025	< 0.025	0.029	0.18	0.616	0.163	0.282	0.072	0.198	0.03
93	2203-FM		< 0.05	< 0.025	< 0.055	0.039	0.374	1.23	0.334	0.572	0.155	0.345	0.047
94	2203-FM		< 0.2	< 0.2	0.63	< 0.2	0.5	0.55	0.29	0.41	< 0.2	< 0.2	< 0.2
106	2203-FM		8.1	< 0.5	< 0.1	< 0.1	0.41	1.1	0.33	0.48	0.14	0.23	< 0.1
110	2203-FM												

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2022 [EURL-PT-POP_2203-FM]

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

Fish Meal (2203-FM)

Perfluoroalkylsulfonic acids (PFSAs) - Results

LC	Sample	Result µg/kg (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)
1	2203-FM		< 0.03	< 0.03	0.164	0.043	5.4	0.49	5.9	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03
3	2203-FM		< 0.05	< 0.05	0.12	< 0.05	3.85	0.131	3.98	< 0.05	< 0.1			
4	2203-FM		< 0.05	< 0.05	0.115	< 0.05	4.97	0.225	5.2	< 0.05	< 0.05		< 0.05	
5	2203-FM		< 0.0493	< 0.0098	< 0.0098	< 0.0098	6.21	2.1	8.31	< 0.0493	< 0.0493			
7	2203-FM		< 0.0219	< 0.0715	0.139	0.0404	4.43	0.199	4.63	< 0.0162	< 0.0133	< 0.0157	< 0.06	< 0.0324
8	2203-FM		0.051	< 0.043	< 0.065	0.073			2.86		< 0.45			
16	2203-FM		< 0.049	< 0.049	0.14	< 0.049	5.14	0.489	5.63	< 0.049	< 0.098	< 0.098	< 0.488	
20	2203-FM		< 0.01	< 0.01	0.12	0.043	5.3	0.37	5.7	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
23	2203-FM		< 2		< 1		5				< 0.2			
25	2203-FM				0.17		4.94							
26	2203-FM		< 0.07	< 0.07	< 0.07	< 0.07	7.44	< 0.07	7.44	< 0.07	< 0.07	< 0.07	< 0.07	< 0.07
28	2203-FM		< 0.0437	< 0.0437	0.166	0.0686	13.8	0.2	14	1.19	< 0.0437	0.502	0.13	0.4
29	2203-FM		< 0.16	< 0.83	0.106	< 0.08	4.57	0.38	4.95	< 0.04	< 0.04	< 0.02	< 0.02	< 0.04
43	2203-FM		< 0.225	< 0.0676	0.181	< 0.0676			4.54	< 0.0676	< 0.0676		< 0.225	
46	2203-FM		0.029	< 0.02	0.113	0.024	3.5	0.227	3.72					
49	2203-FM				0.18		10.8							
56	2203-FM		< 0.5	< 0.5	< 0.5	< 0.5	5.28	< 0.5	5.28	< 1	< 1		< 1	
59	2203-FM		< 1		0.145	< 0.06	5.53	< 1	6.14		< 0.1			
62	2203-FM		< 5.5		< 0.5	< 0.75	4.57	< 0.1						
63	2203-FM				0.1	< 0.1	5.02	0.34	5.36		< 0.1			
67	2203-FM				0.161		7.44	0.485	7.93					
68	2203-FM		< 0.3	< 0.3	0.26	< 0.3	4.49	0.46	4.95	< 0.3	< 0.3	< 0.3	< 0.3	< 0.3
72	2203-FM		< 0.02	< 0.02	0.105	0.067	5.45	0.416	4.91		< 0.02			
75	2203-FM		< 0.02		0.124		2.78		4.53					
76	2203-FM		< 0.2	< 0.2	< 0.2	< 0.2	5.26	0.485	5.53	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
80	2203-FM		< 0.05	< 0.1	0.11	< 0.1	4.22	0.4	4.62	< 0.1	< 0.1			
83	2203-FM		< 0.02	< 0.2	0.14	0.089	5.05	0.45	5.5	< 0.2	< 0.2			
84	2203-FM		< 0.025	< 0.025	0.358	0.031	3.35			< 0.025	< 0.025		< 0.025	
85	2203-FM		< 0.01	< 0.01	0.09	< 0.01	3.8	< 0.05	3.8	< 0.01	< 0.01		< 0.05	
92	2203-FM		< 0.025	< 0.025	0.106	0.026	3	0.197	3.2	< 0.025	< 0.025		< 0.025	
93	2203-FM		< 0.015	< 0.002	0.136	0.031	5.19	0.491	5.69	< 0.002	< 0.002		< 0.002	
94	2203-FM		< 0.2	< 0.2	< 0.2	< 0.2	4.49			< 0.2	< 0.2			
106	2203-FM		< 0.1	< 0.1	0.15	< 0.1	4.8	0.2	5	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
110	2203-FM								47.2					

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2022 [EURL-PT-POP_2203-FM]

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

Fish Meal (2203-FM)

Sum of PFOS, PFOA, PFNA, PFHxS - Results

LC	Sample	l33l µg/kg (12% moisture content)	Sum of L-PFOS, PFOA, PFNA, PFHxS **(ub)**	Sum of L-PFOS, PFOA, PFNA, PFHxS **(lb)**		Sum of total-PFOS, PFOA, PFNA, PFHxS **(ub)**	Sum of total-PFOS, PFOA, PFNA, PFHxS **(lb)**
1	2203-FM		7.1	7.1			
3	2203-FM		4.9	4.9		5.0	5.0
4	2203-FM		6.45	6.45		6.7	6.7
5	2203-FM		8.15	8.11		10.3	10.2
7	2203-FM		5.89	5.89		6.1	6.1
8	2203-FM					4.0	3.9
16	2203-FM		6.88	6.88		7.4	7.4
20	2203-FM		6.8	6.8		7.2	7.2
23	2203-FM		8	7			
25	2203-FM		6.8	6.8			
26	2203-FM		8.58	8.51		8.7	8.5
28	2203-FM		15.5	15.5		15.7	15.7
29	2203-FM		5.93	5.93		6.3	6.3
43	2203-FM						
46	2203-FM		5.22	5.22		5.4	5.4
49	2203-FM		14.1	14.1			
56	2203-FM		7.54	7.04		8.0	7.0
59	2203-FM		8.14	8.14		8.8	8.8
62	2203-FM		6.85	6.35		6.9	6.4
63	2203-FM		6.59	6.59		6.9	6.9
67	2203-FM		9.33	9.33		9.8	9.8
68	2203-FM		6.14	6.14		6.6	6.6
72	2203-FM		6.79	6.79		6.3	6.3
75	2203-FM		3.85	3.85		5.6	5.6
76	2203-FM		7.27	7.07		7.0	6.8
80	2203-FM		5.48	5.48		5.9	5.9
83	2203-FM		6.58	6.58		7.0	7.0
84	2203-FM		5.03	5.03			
85	2203-FM		4.75			4.8	
92	2203-FM		3.9	3.9		4.1	4.1
93	2203-FM		6.94	6.94		7.4	7.4
94	2203-FM		5.74	5.54			
106	2203-FM		6.4	6.4		6.7	6.7
110	2203-FM						

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2022 [EURL-PT-POP_2203-FM]

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

Fish Meal (2203-FM)

FOSA - Results

LC	Sample	Result µg/kg (12% moisture content)	Perfluorooctane sulphonamide **(FOSA)**
1	2203-FM		0.440
3	2203-FM		
4	2203-FM		
5	2203-FM		
7	2203-FM		0.423
8	2203-FM		1.310
16	2203-FM		
20	2203-FM		0.460
23	2203-FM		< 2
25	2203-FM		
26	2203-FM		< 0.07
28	2203-FM		0.718
29	2203-FM		
43	2203-FM		0.549
46	2203-FM		
49	2203-FM		
56	2203-FM		0.579
59	2203-FM		
62	2203-FM		
63	2203-FM		0.420
67	2203-FM		
68	2203-FM		0.440
72	2203-FM		
75	2203-FM		
76	2203-FM		
80	2203-FM		
83	2203-FM		
84	2203-FM		
85	2203-FM		0.372
92	2203-FM		0.351
93	2203-FM		
94	2203-FM		
106	2203-FM		0.450
110	2203-FM		

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2022 [EURL-PT-POP_2203-FM]

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

03 August 2023

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

Test sample - Fish Meal (2203-FM)

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 Fish Meal 2022 [EURL-PT-POP_2203-FM]

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

Fish Meal (2203-FM)

Perfluoroalkylcarboxylic acids (PFCAs) - Z-scores

LC	Sample	Z-score [σ_p = 20 %]	Perfluorooctanoic acid **(PFOA)**	Perfluorononanoic acid **(PFNA)**	Perfluorodecanoic acid **(PFDA)**	Perfluoroundecanoic acid **(PFUnDA)**	Perfluorododecanoic acid **(PFDoDA)**	Perfluorotridecanoic acid **(PFTrDA)**
1	2203-FM		-0.2	0.6	0.1	0.8	0.9	2.9
3	2203-FM		-1.9	-1.6	-1.7	-1.5	-2.1	
4	2203-FM		-1.1	0.2	-1.2	-1.8		0.4
5	2203-FM		1.1	2.0	4.5	4.6	2.2	4.3
7	2203-FM		-0.6	-0.2	-0.3	-0.4	-0.7	-0.6
8	2203-FM		1.4	-2.1		-1.9		
16	2203-FM		0.2	0.8	1.2	0.8	0.1	
20	2203-FM		0.1	-0.1	0.1	0.2	-0.3	0.2
23	2203-FM			1.8	2.1			
25	2203-FM		1.8	0.7				
26	2203-FM		-1.3	-1.2	-0.3	-0.6		
28	2203-FM		0.0	0.8	0.9	0.5	0.6	3.4
29	2203-FM		-1.2	-0.3	-0.1	0.0	-0.3	-2.2
43	2203-FM		-0.3	-1.2	-0.9	-1.2	-0.2	-1.6
46	2203-FM		0.5	0.8	0.5	1.0	0.9	
49	2203-FM		22.8	-0.2				
56	2203-FM		1.0	1.3		2.0		
59	2203-FM		0.4	5.0	-1.4	1.5		
62	2203-FM		0.2	1.7	0.4	1.5		
63	2203-FM		1.7	-0.3	-0.7	0.4		1.3
67	2203-FM		0.0	1.6				
68	2203-FM		-0.5	0.1		-0.2		
72	2203-FM		-0.6	-0.6	-0.5	-0.5	0.2	4.1
75	2203-FM		-2.5	-1.4	-1.2	-1.5	-0.9	-1.0
76	2203-FM		-0.2	0.7	1.6	1.2		
80	2203-FM		-1.3	-0.8	-0.7	-0.7	0.1	
83	2203-FM		-0.1	0.0	0.1	-0.1	0.4	0.2
84	2203-FM		-0.5	-0.2	0.3	0.3	0.1	0.2
85	2203-FM		-1.5	-2.1	-1.0	-1.4	-1.5	-1.0
92	2203-FM		-2.6	-2.0	-2.1	-1.9	-1.9	-0.9
93	2203-FM		0.0	1.0	0.9	1.3	1.6	2.2
94	2203-FM		1.7	-2.3	0.1	-0.5		
106	2203-FM		0.5	0.3	0.9	0.3	1.0	-0.2
110	2203-FM							

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2022 [EURL-PT-POP_2203-FM

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

Fish Meal (2203-FM)

Perfluoroalkylsulfonic acids (PFSAs) - Z-scores

LC	Sample	Z-score [σ_p = 20 %]	Perfluorohexanesulfonic acid **(PFHxS)**	Linear Perfluorooctanesulfonic acid **(L-PFOS)**	Sum of branched and linear Perfluorooctanesulfonic acid **(total PFOS)**
1	2203-FM		0.9	0.6	0.8
3	2203-FM		-0.7	-1.0	-1.1
4	2203-FM		-0.9	0.1	0.1
5	2203-FM			1.4	3.1
7	2203-FM		0.0	-0.4	-0.5
8	2203-FM				-2.2
16	2203-FM		0.0	0.3	0.5
20	2203-FM		-0.7	0.5	0.6
23	2203-FM			0.2	
25	2203-FM		1.1	0.1	
26	2203-FM			2.7	2.3
28	2203-FM		0.9	9.3	8.6
29	2203-FM		-1.2	-0.3	-0.2
43	2203-FM		1.5		-0.6
46	2203-FM		-1.0	-1.4	-1.4
49	2203-FM		1.4	6.2	
56	2203-FM			0.5	0.1
59	2203-FM		0.2	0.7	1.0
62	2203-FM			-0.3	
63	2203-FM		-1.4	0.2	0.2
67	2203-FM		0.8	2.7	2.7
68	2203-FM		4.3	-0.4	-0.2
72	2203-FM		-1.3	0.6	-0.2
75	2203-FM		-0.6	-2.1	-0.6
76	2203-FM			0.4	0.4
80	2203-FM		-1.1	-0.6	-0.5
83	2203-FM		0.0	0.2	0.4
84	2203-FM		7.8	-1.5	
85	2203-FM		-1.8	-1.1	-1.3
92	2203-FM		-1.2	-1.9	-1.9
93	2203-FM		-0.1	0.4	0.5
94	2203-FM			-0.4	
106	2203-FM		0.4	0.0	-0.1
110	2203-FM				41.0

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2022 [EURL-PT-POP_2203-FM]

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

Fish Meal (2203-FM)

Sum of PFOS, PFOA, PFNA, PFHxS - Z-scores

LC	Sample	Z-score [σ_p = 20 %]	Sum of L-PFOS, PFOA, PFNA, PFHxS **(ub)**	Sum of L-PFOS, PFOA, PFNA, PFHxS **(lb)**		Sum of total-PFOS, PFOA, PFNA, PFHxS **(ub)**	Sum of total-PFOS, PFOA, PFNA, PFHxS **(lb)**
1	2203-FM		0.4	0.4			
3	2203-FM		-1.3	-1.2		-1.3	-1.3
4	2203-FM		-0.1	-0.1		-0.1	-0.1
5	2203-FM		1.2	1.2		2.5	2.6
7	2203-FM		-0.5	-0.5		-0.6	-0.5
8	2203-FM					-2.1	-2.1
16	2203-FM		0.2	0.3		0.4	0.5
20	2203-FM		0.2	0.2		0.2	0.3
23	2203-FM		1.1	0.4			
25	2203-FM		0.2	0.2			
26	2203-FM		1.5	1.5		1.3	1.3
28	2203-FM		6.8	6.9		6.4	6.6
29	2203-FM		-0.5	-0.5		-0.4	-0.3
43	2203-FM						
46	2203-FM		-1.0	-1.0		-1.0	-1.0
49	2203-FM		5.7	5.8			
56	2203-FM		0.7	0.4		0.9	0.2
59	2203-FM		1.2	1.2		1.4	1.5
62	2203-FM		0.2	-0.1		0.0	-0.3
63	2203-FM		0.0	0.1		0.0	0.1
67	2203-FM		2.1	2.2		2.1	2.3
68	2203-FM		-0.3	-0.3		-0.2	-0.1
72	2203-FM		0.2	0.2		-0.5	-0.4
75	2203-FM		-2.1	-2.0		-0.9	-0.8
76	2203-FM		0.5	0.4		0.1	0.0
80	2203-FM		-0.8	-0.8		-0.7	-0.6
83	2203-FM		0.0	0.0		0.1	0.2
84	2203-FM		-1.2	-1.1			
85	2203-FM		-1.4			-1.5	
92	2203-FM		-2.0	-2.0		-2.0	-2.0
93	2203-FM		0.3	0.3		0.4	0.5
94	2203-FM		-0.6	-0.8			
106	2203-FM		-0.1	-0.1		-0.1	0.0
110	2203-FM						

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2022 [EURL-PT-POP_2203-FM]

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

Fish Meal (2203-FM)

FOSA - Z-scores

LC	Sample	Z-score [σ_p = 20 %]	Perfluorooctane sulphonamide **(FOSA)**
1	2203-FM		-0.1
3	2203-FM		
4	2203-FM		
5	2203-FM		
7	2203-FM		-0.2
8	2203-FM		9.7
16	2203-FM		
20	2203-FM		0.2
23	2203-FM		
25	2203-FM		
26	2203-FM		
28	2203-FM		3.1
29	2203-FM		
43	2203-FM		1.2
46	2203-FM		
49	2203-FM		
56	2203-FM		1.5
59	2203-FM		
62	2203-FM		
63	2203-FM		-0.3
67	2203-FM		
68	2203-FM		-0.1
72	2203-FM		
75	2203-FM		
76	2203-FM		
80	2203-FM		
83	2203-FM		
84	2203-FM		
85	2203-FM		-0.8
92	2203-FM		-1.1
93	2203-FM		
94	2203-FM		
106	2203-FM		0.1
110	2203-FM		

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2022 [EURL-PT-POP_2203-FM]

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

03 August 2023

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

Test sample - Fish Meal (2203-FM)

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

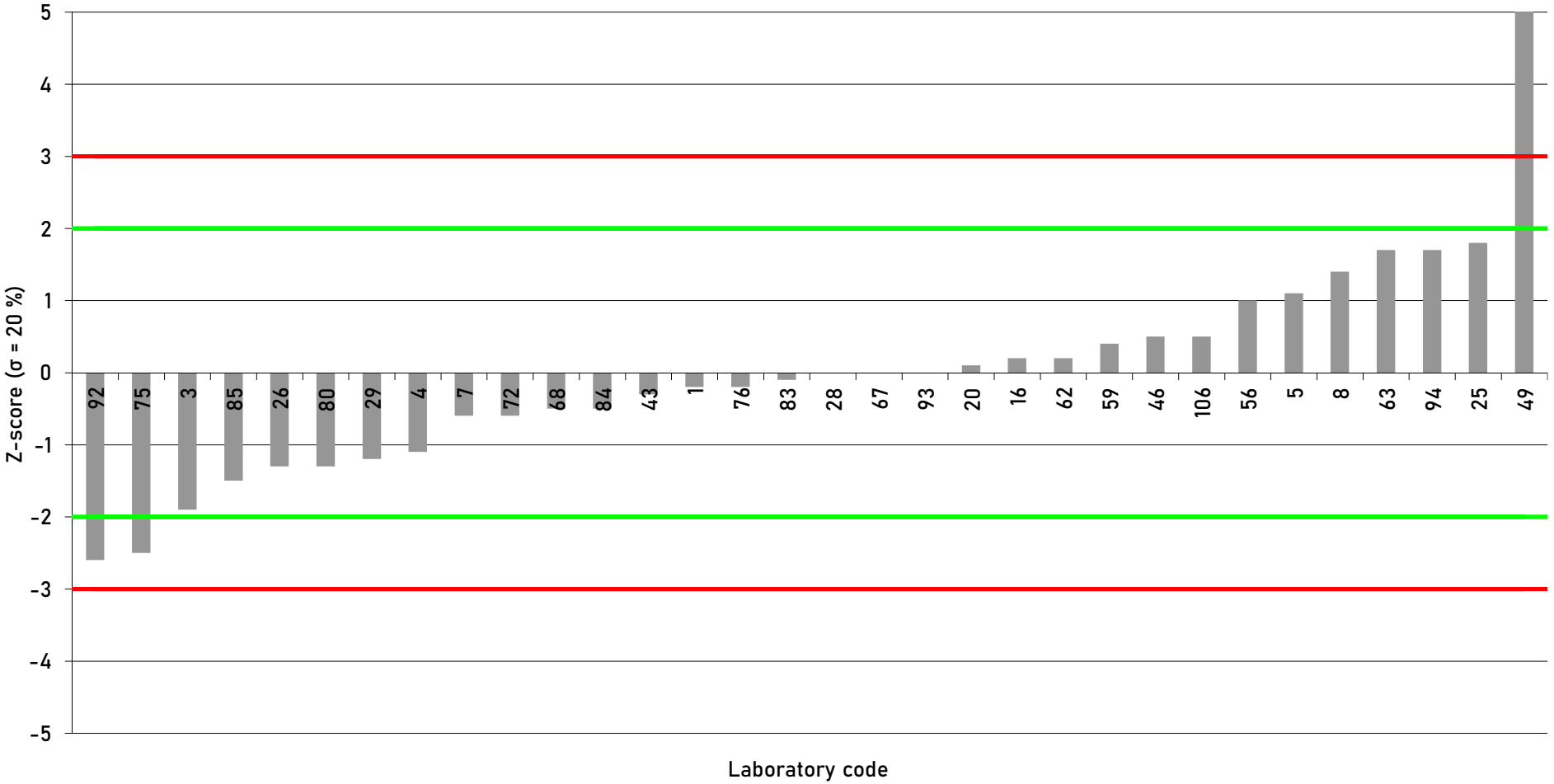
± 2 z-scores:

± 3 z-scores:

Fish Meal (2203-FM)

Perfluorooctanoic acid

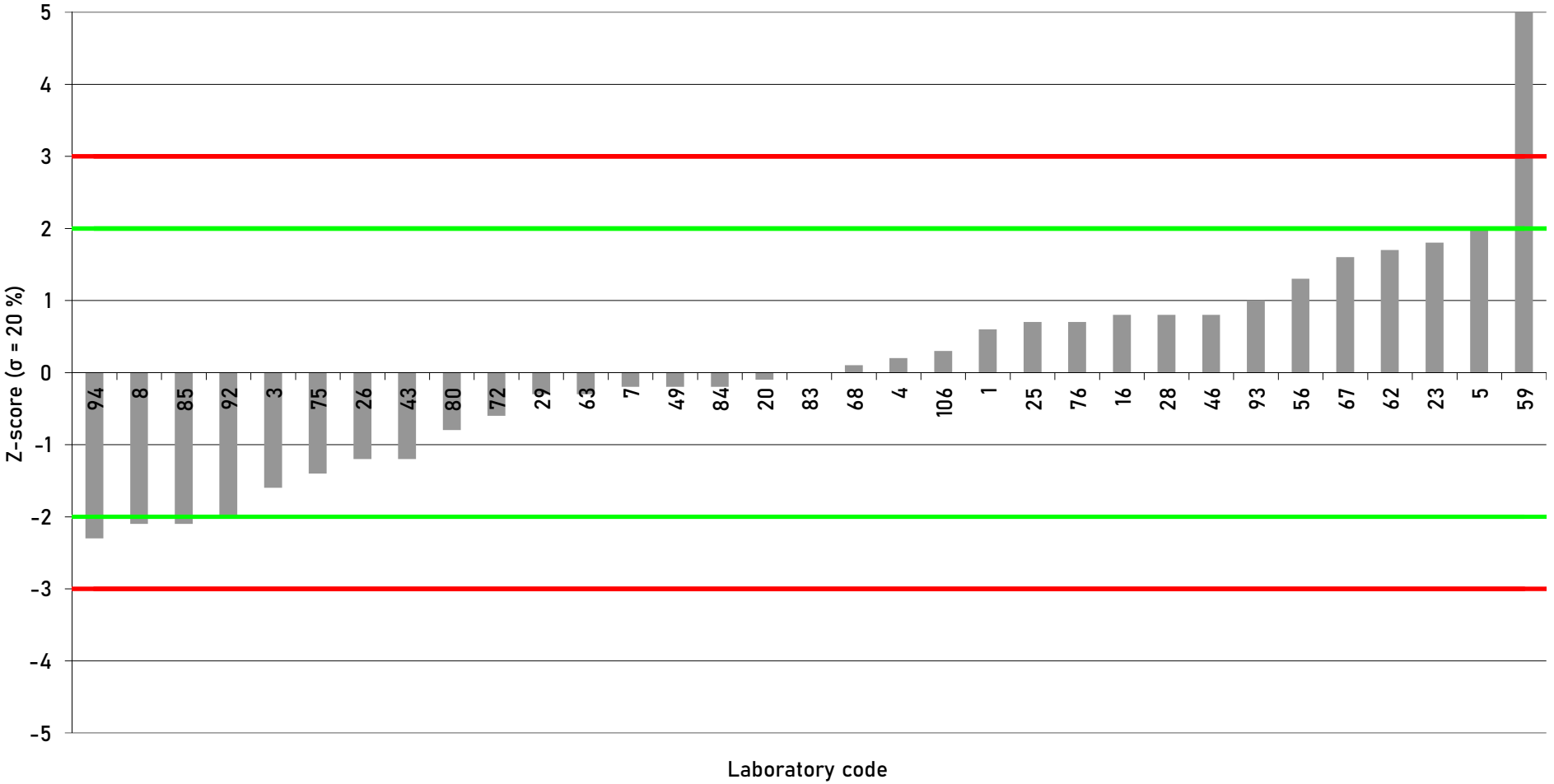
Assigned value: 0.374 µg/kg (12% moisture content)



Fish Meal (2203-FM)

Perfluorononanoic acid

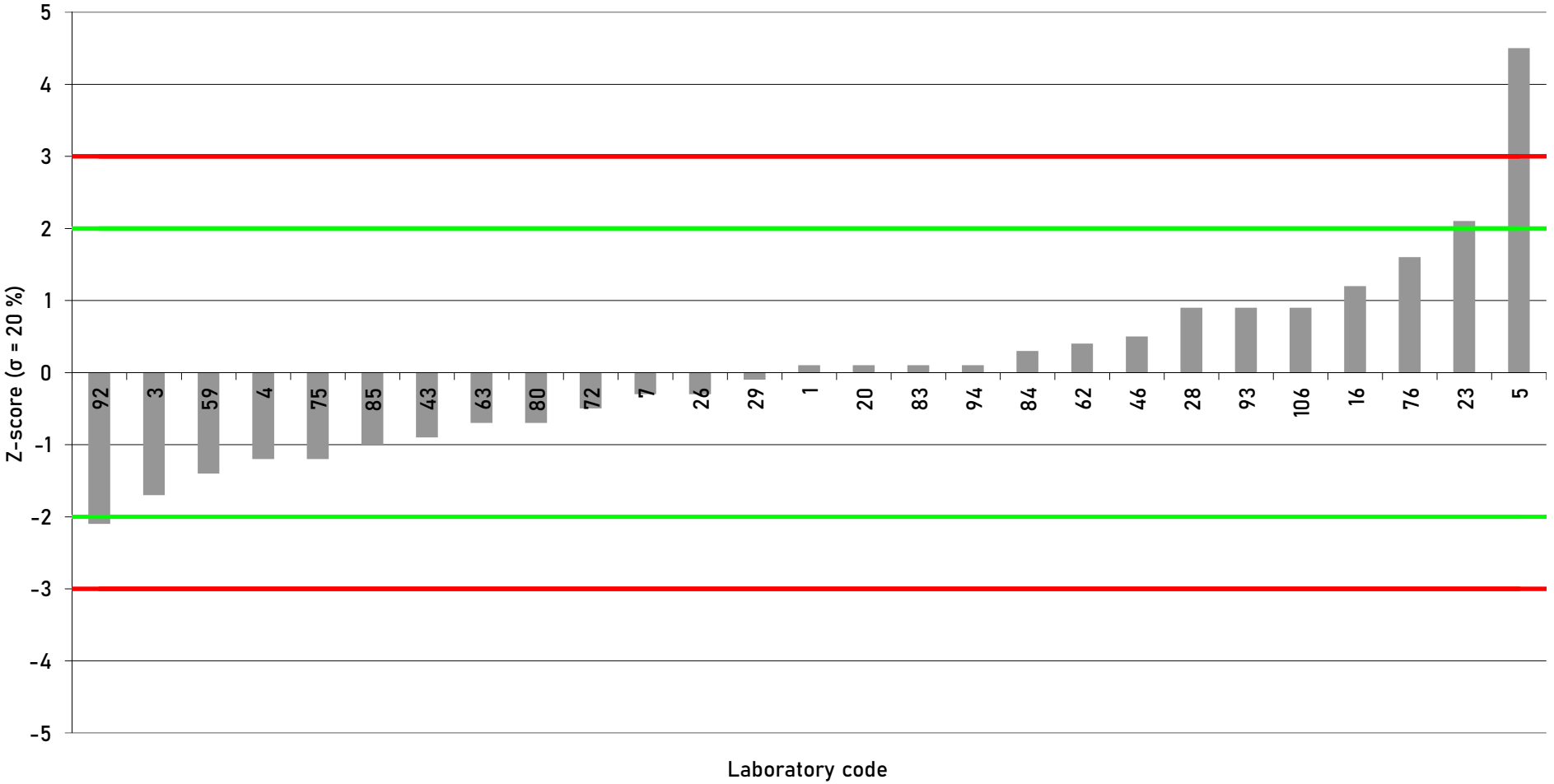
Assigned value: 1.03 µg/kg (12% moisture content)



Fish Meal (2203-FM)

Perfluorodecanoic acid

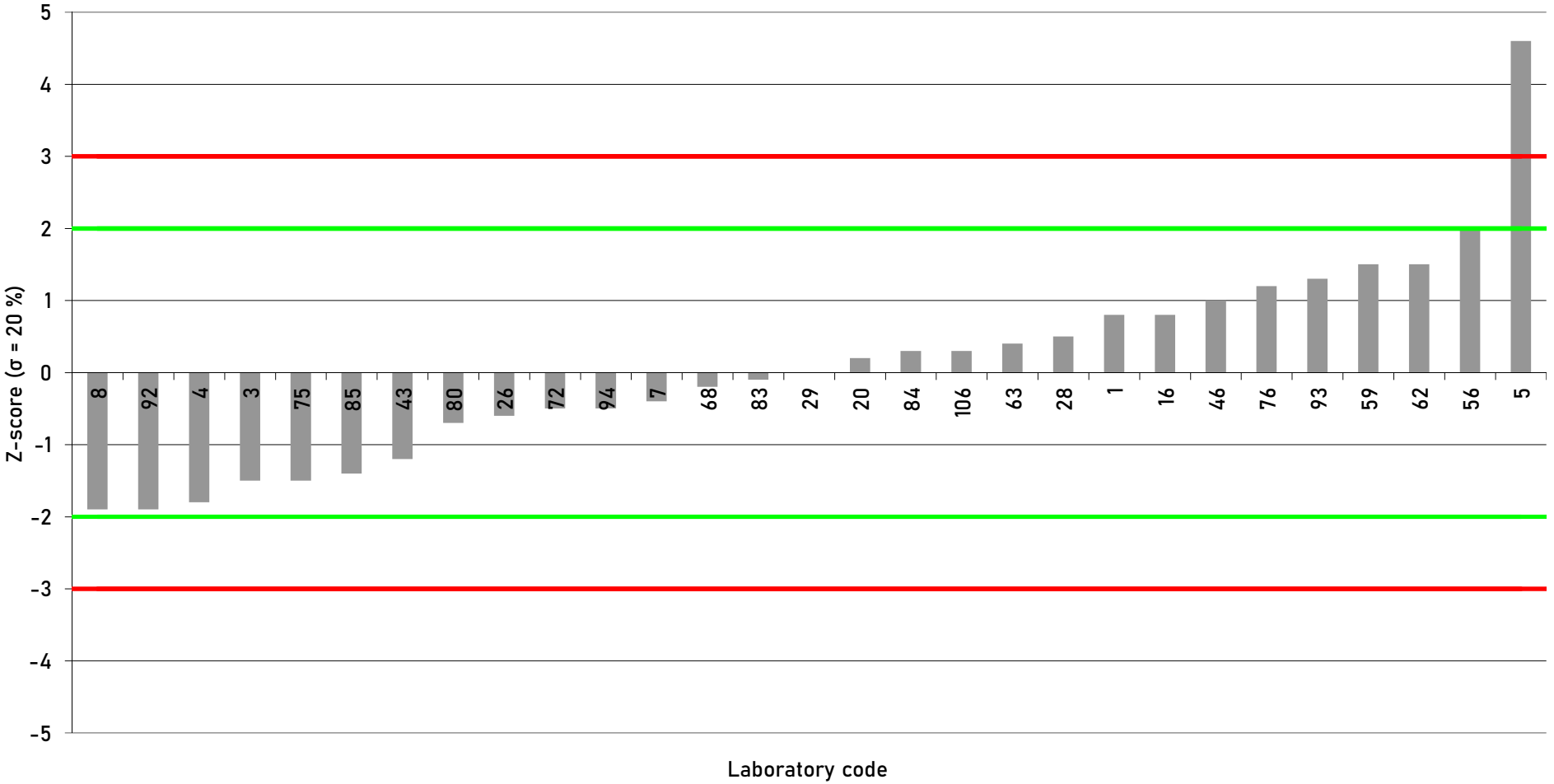
Assigned value: 0.28 µg/kg (12% moisture content)



Fish Meal (2203-FM)

Perfluoroundecanoic acid

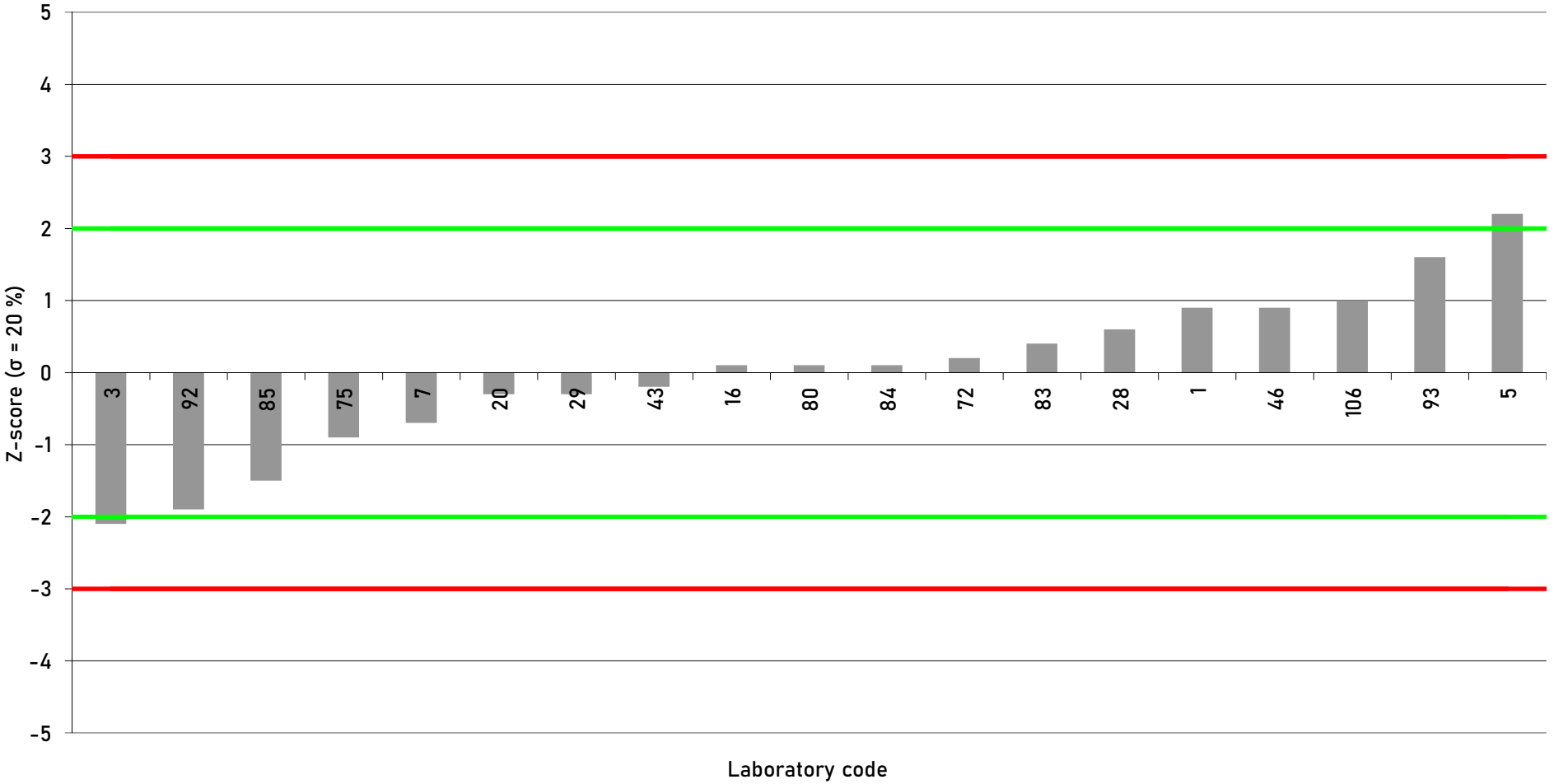
Assigned value: 0.46 µg/kg (12% moisture content)



Fish Meal (2203-FM)

Perfluorododecanoic acid

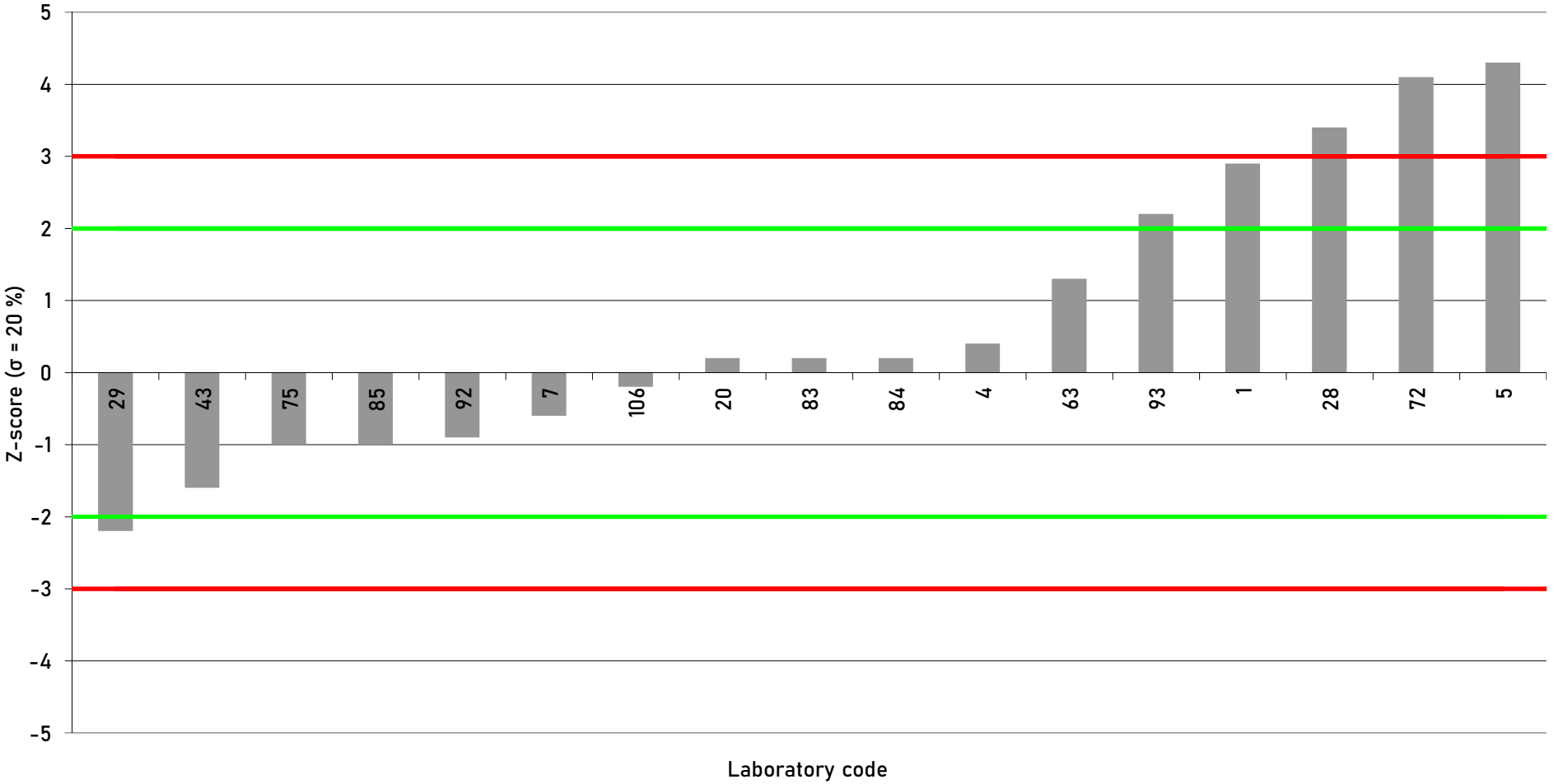
Assigned value: 0.117 µg/kg (12% moisture content)



Fish Meal (2203-FM)

Perfluorotridecanoic acid

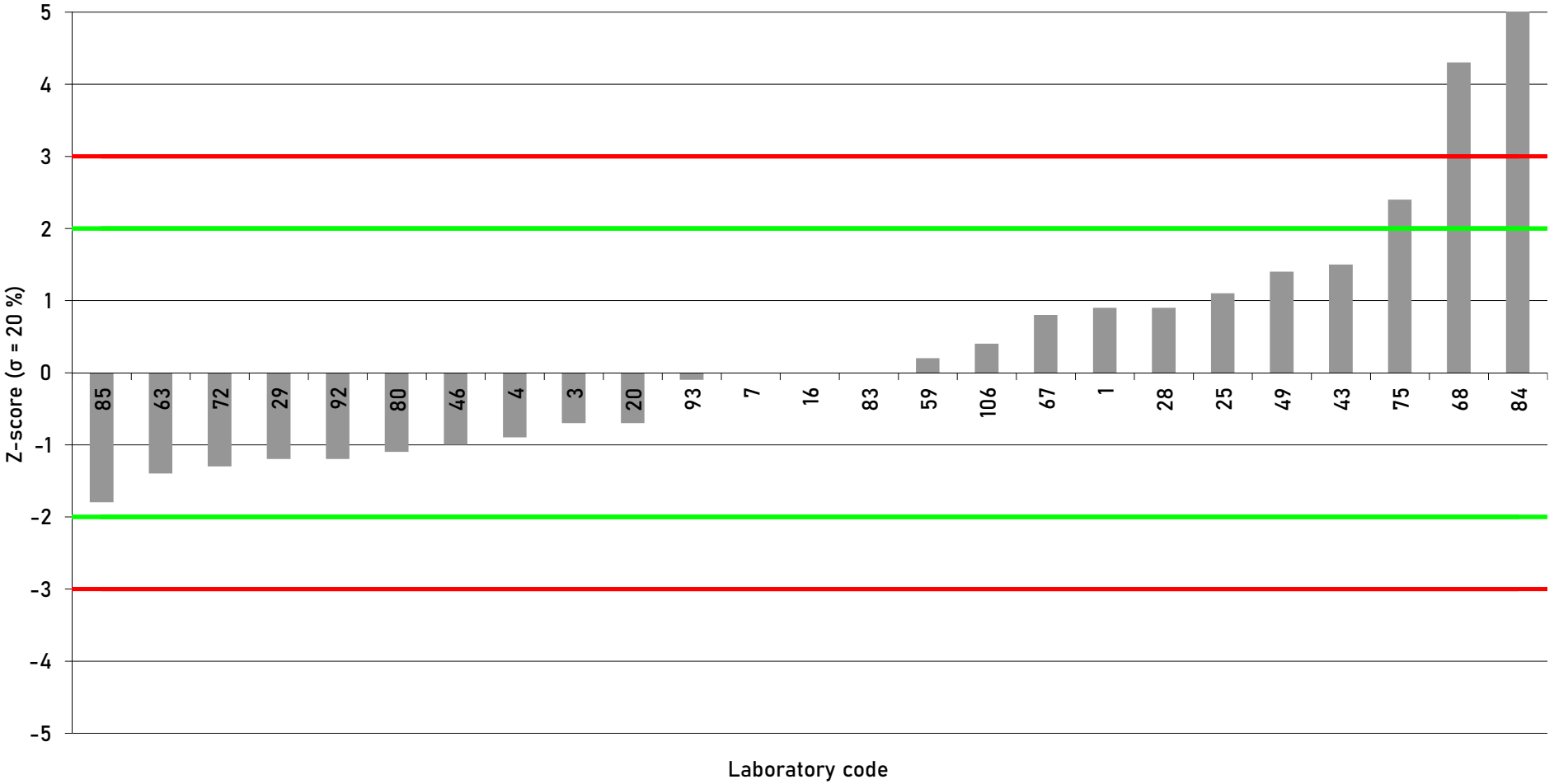
Assigned value: 0.24 µg/kg (12% moisture content)



Fish Meal (2203-FM)

Perfluorohexanesulfonic acid

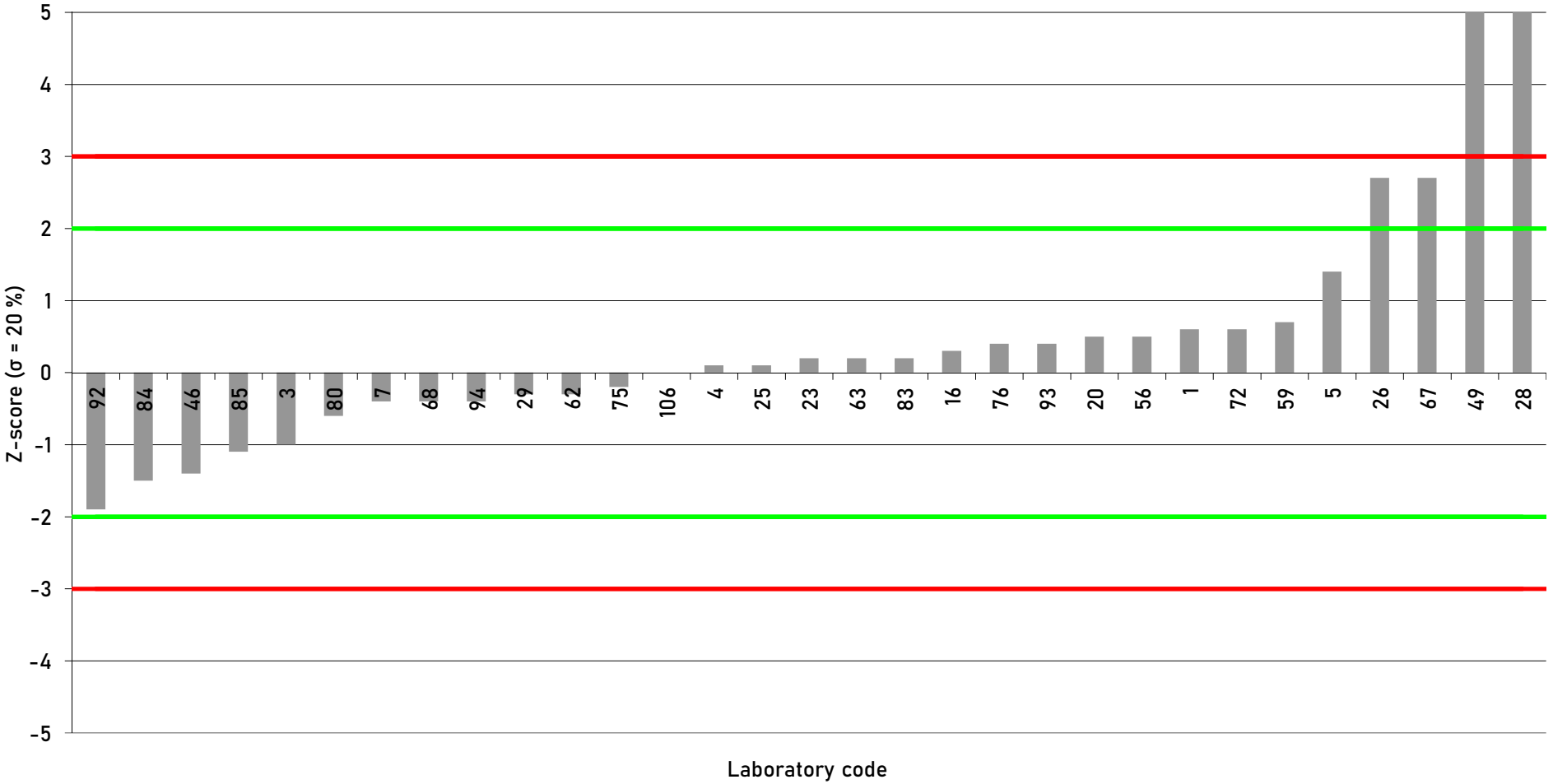
Assigned value: 0.14 µg/kg (12% moisture content)



Fish Meal (2203-FM)

Linear Perfluorooctanesulfonic acid

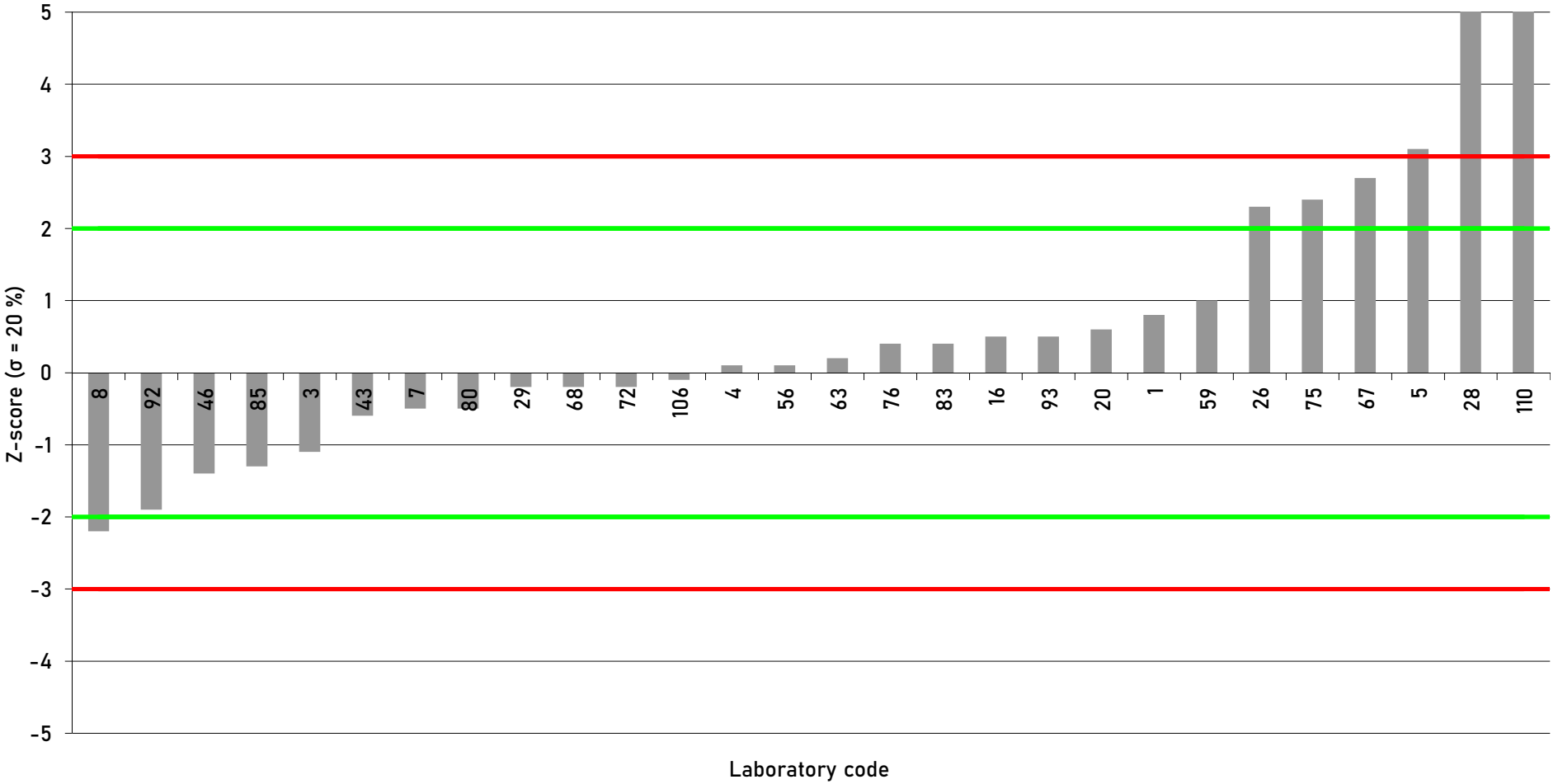
Assigned value: 4.84 µg/kg (12% moisture content)



Fish Meal (2203-FM)

Sum of branched and linear Perfluorooctanesulfonic acid

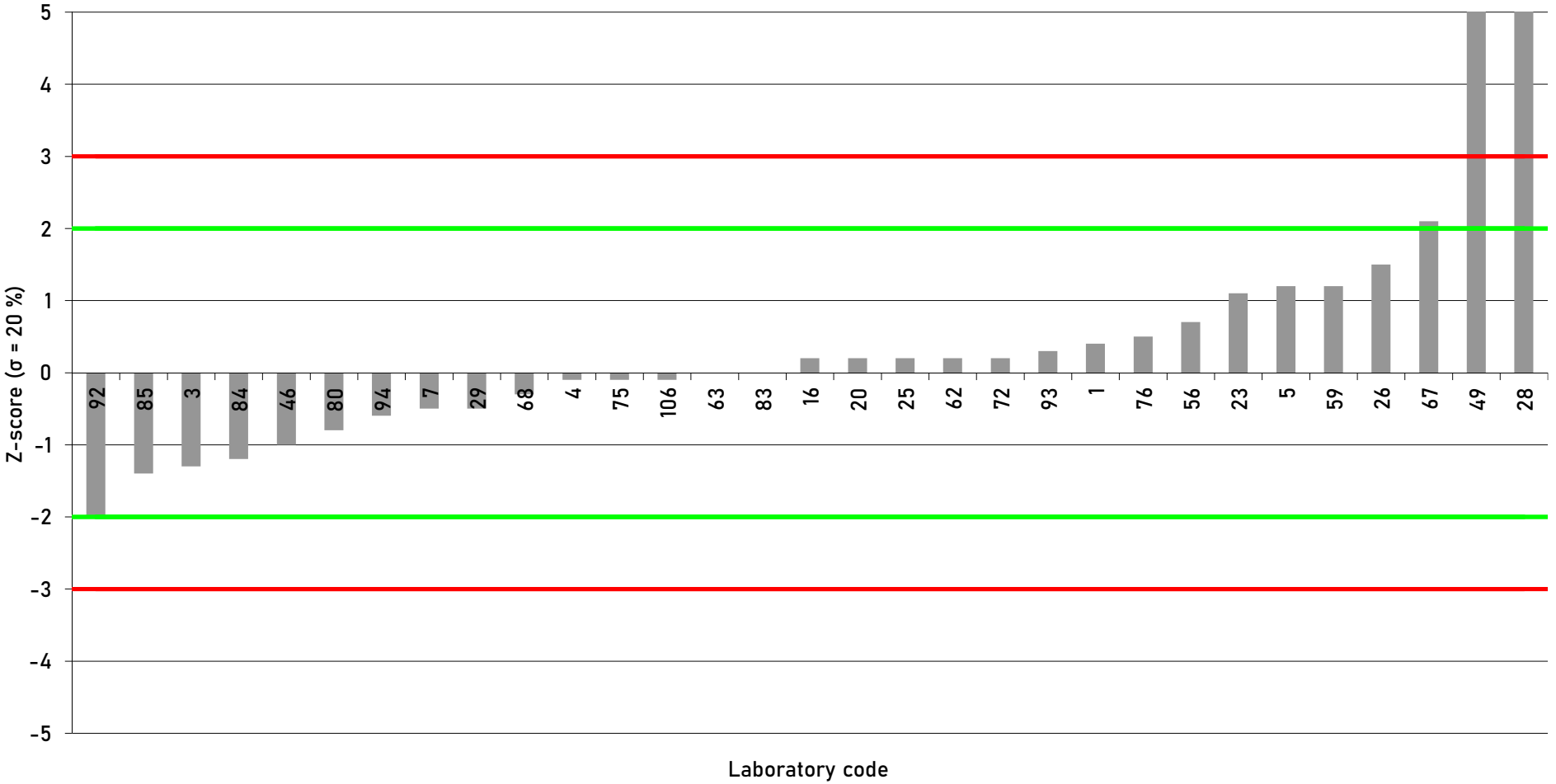
Assigned value: 5.13 µg/kg (12% moisture content)



Fish Meal (2203-FM)

Sum of L-PFOS, PFOA, PFNA, PFHxS (ub)

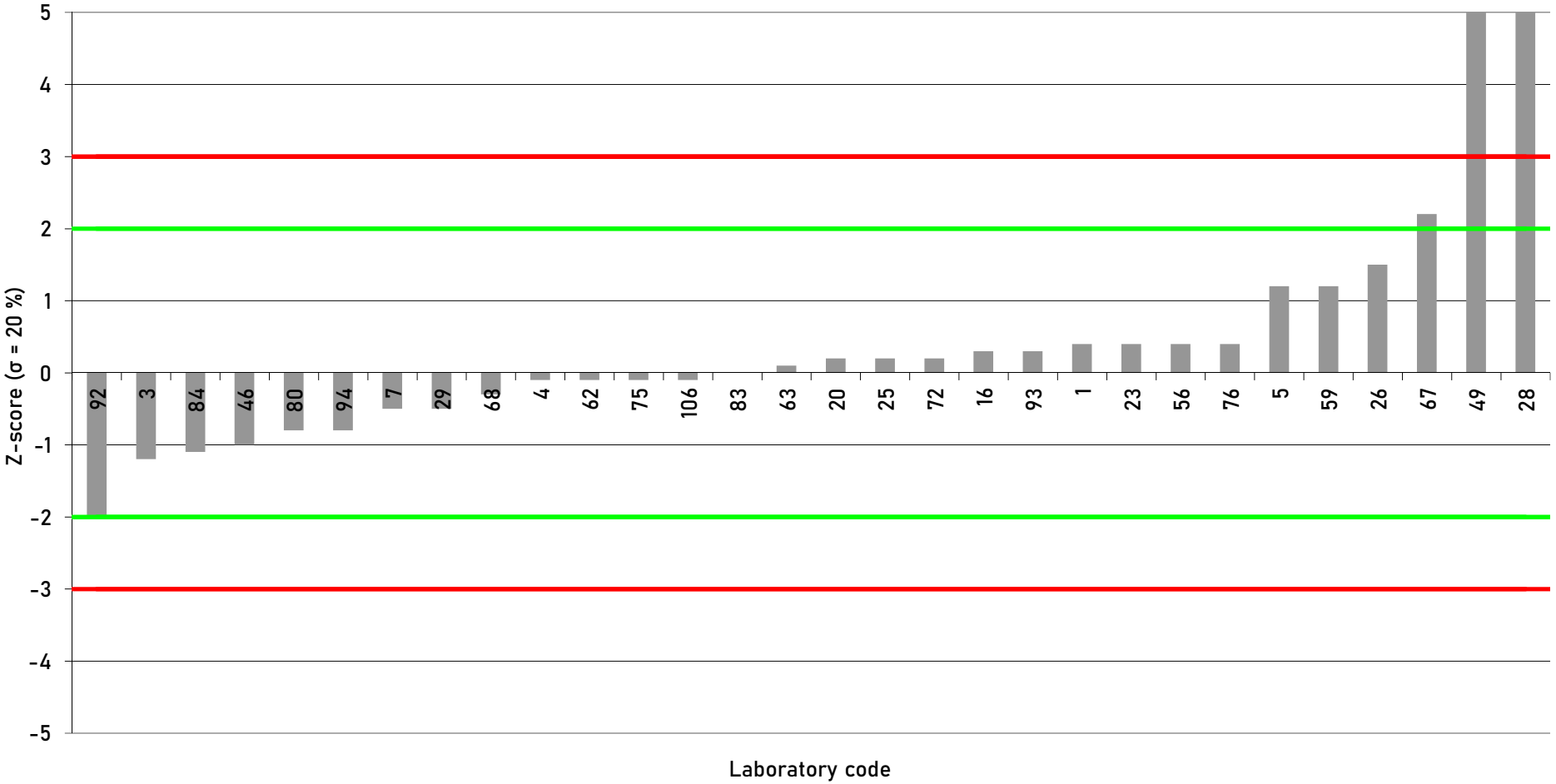
Assigned value: 6.56 µg/kg (12% moisture content)



Fish Meal (2203-FM)

Sum of L-PFOS, PFOA, PFNA, PFHxS (lb)

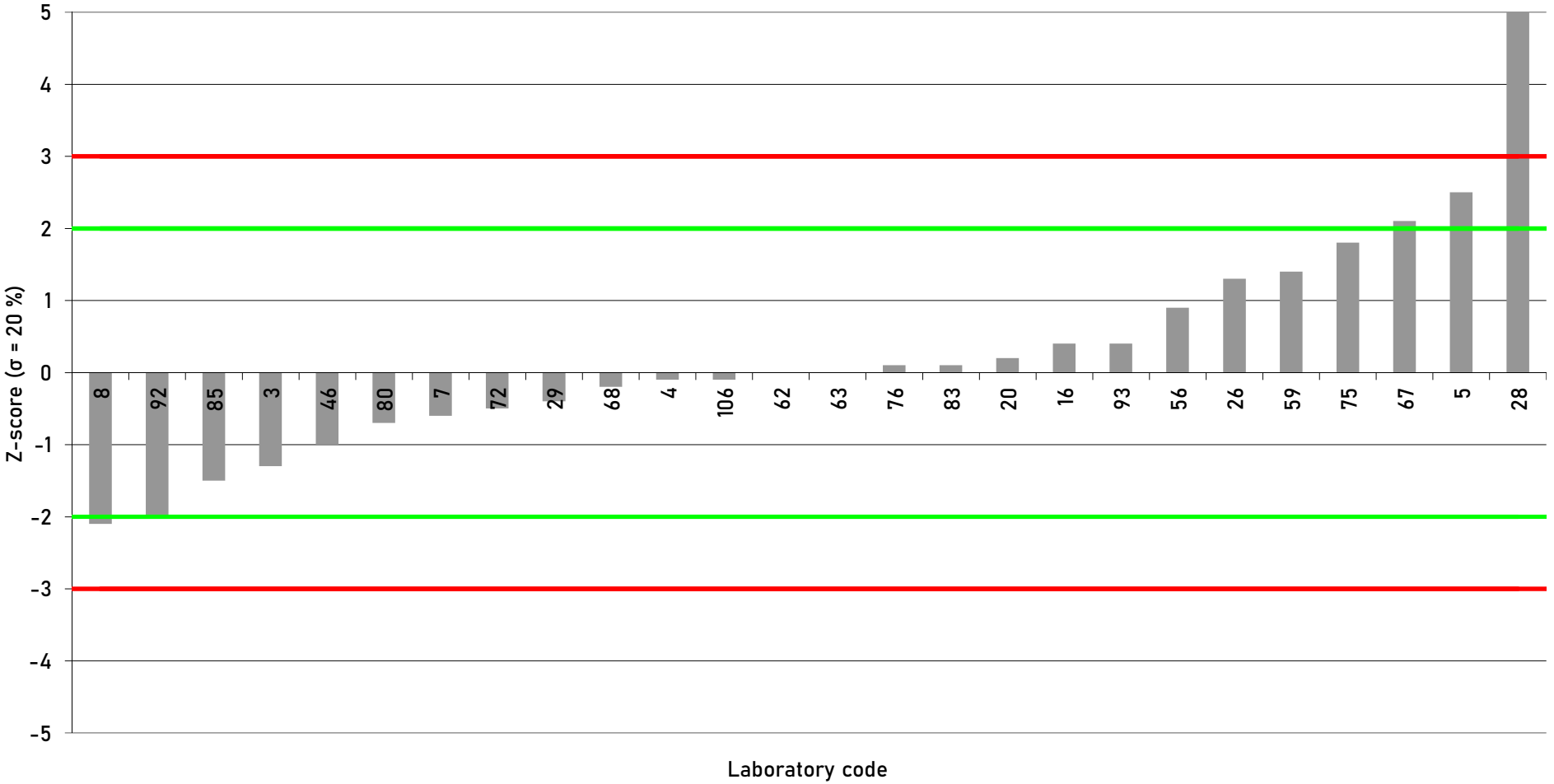
Assigned value: 6.52 µg/kg (12% moisture content)



Fish Meal (2203-FM)

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

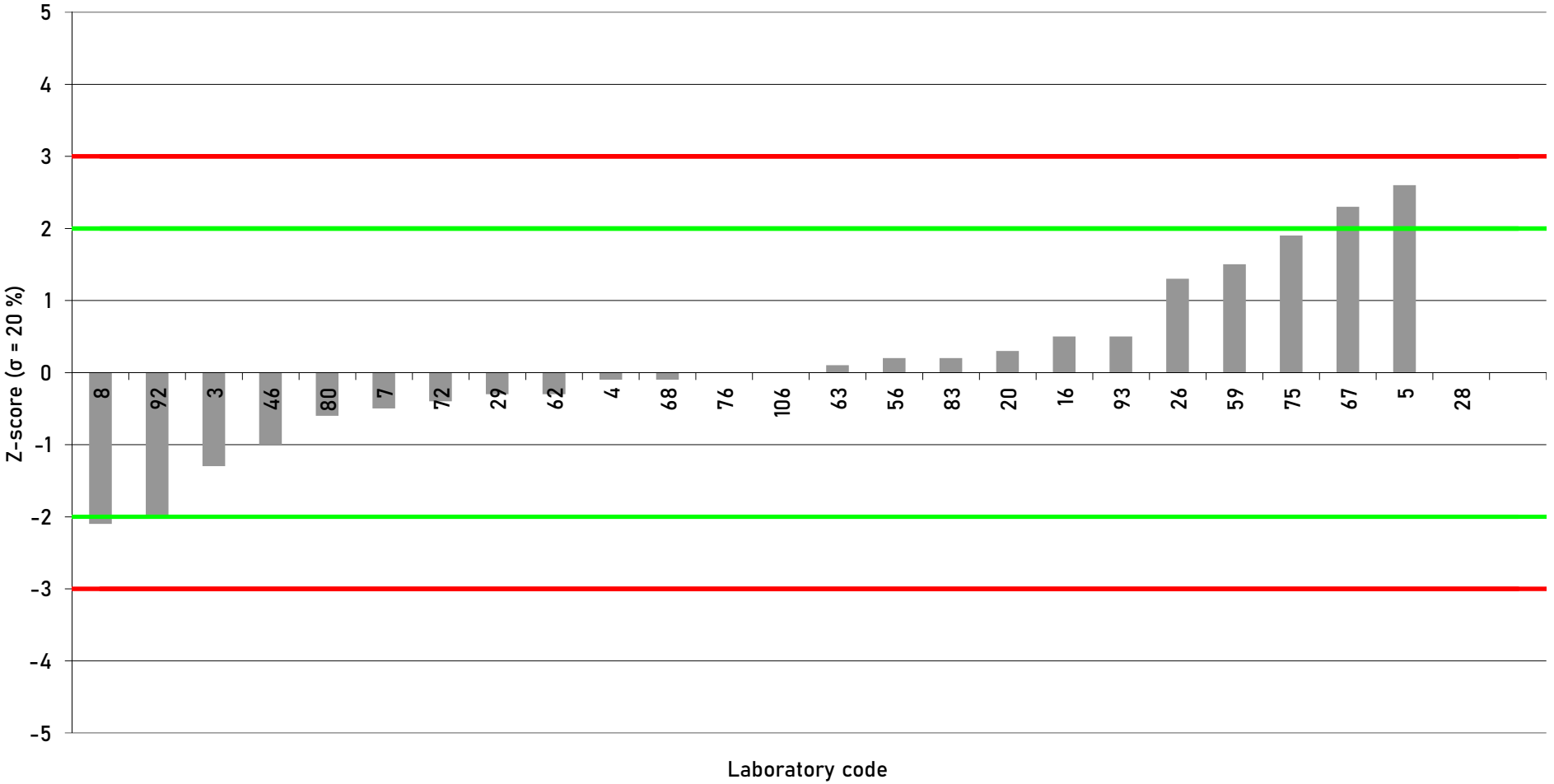
Assigned value: 6.87 µg/kg (12% moisture content)



Fish Meal (2203-FM)

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

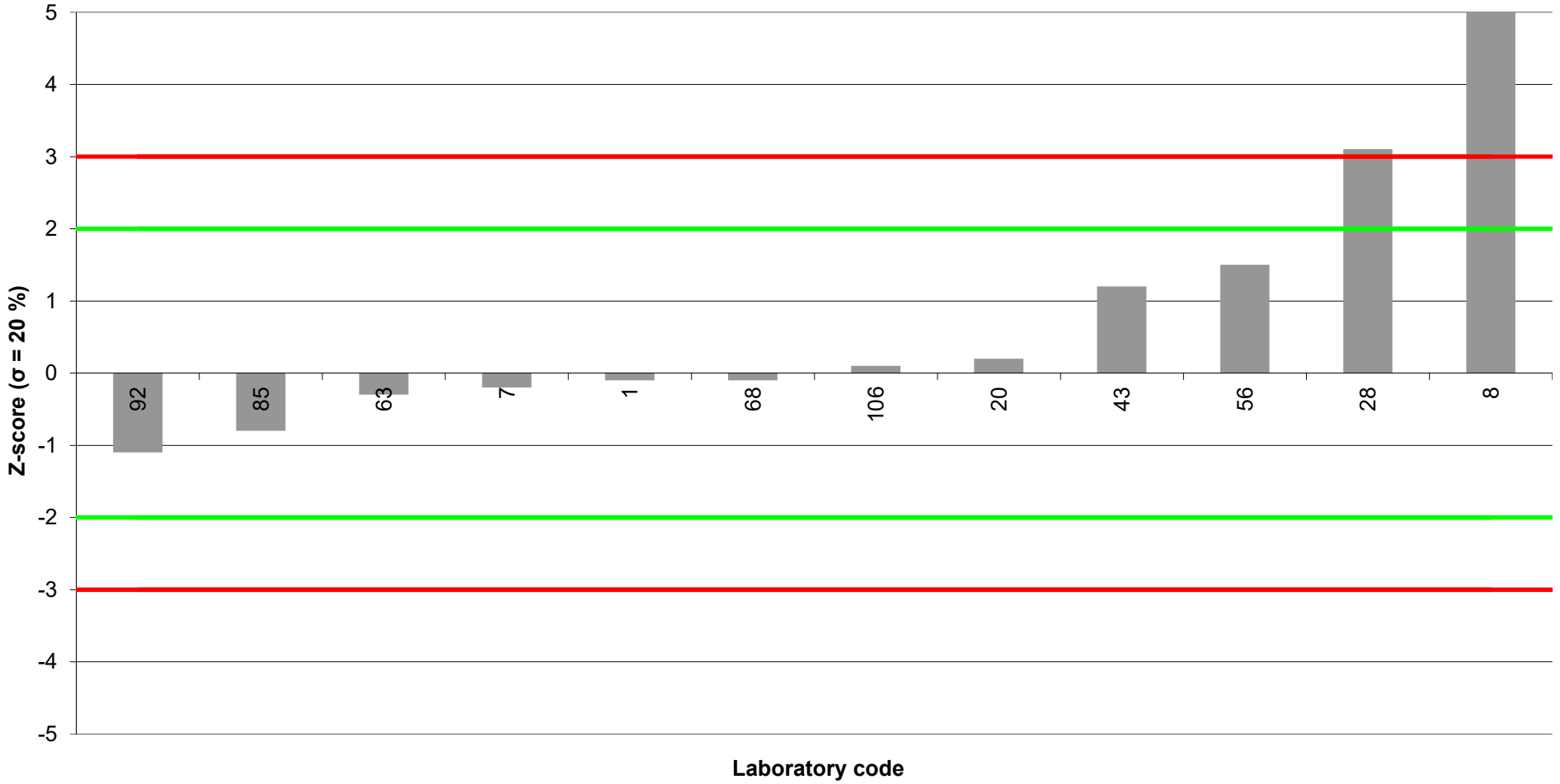
Assigned value: 6.87 µg/kg (12% moisture content)



Fish Meal (2203-FM)

Perfluorooctane sulphonamide (FOSA)

Assigned value: 0.445 µg/kg (12% moisture content)



EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2022 [EURL-PT-POP_2203-FM]

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

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Annex 5: Test for sufficient homogeneity for PFASs

Test sample - Fish Meal (2203-FM)

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2022 [EURL-PT-POP_2203-FM]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Fish Meal (2203-FM)
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** [%]
Perfluorooctanoic acid (PFOA)		0.347	0.349	2%
Perfluorodecanoic acid (PFDA)		0.260	0.261	2%
Perfluorohexanesulfonic acid (PFHxS)		0.128	0.127	1%
Linear Perfluorooctanesulfonic acid (L-PFOS)		4.64	4.67	20%
Perfluorooctane sulphonamide (FOSA)		0.362	0.368	2%

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2022 [EURL-PT-POP_2203-FM]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Fish Meal (2203-FM)
Selected compounds - Homogeneity test - Data

Sample	Replicate	Result µg/kg product (12% moisture content)	PFOA	L-PFOS	PFDA
33	1		0.349	4.48	0.253
	2		0.372	4.70	0.270
69	1		0.349	4.72	0.264
	2		0.382	4.73	0.264
95	1		0.370	4.85	0.254
	2		0.335	4.50	0.271
119	1		0.329	4.87	0.284
	2		0.311	4.58	0.247
137	1		0.358	4.67	0.269
	2		0.334	4.32	0.259
162	1		0.344	4.88	0.272
	2		0.310	4.32	0.246
166	1		0.371	4.84	0.265
	2		0.356	4.71	0.258
193	1		0.354	4.41	0.240
	2		0.321	4.36	0.226
221	1		0.371	4.62	0.292
	2		0.344	5.24	0.262
224	1		0.356	4.68	0.257
	2		0.320	4.41	0.247
Cochran's C-test					
C			0.156	0.332	0.354
$C_{critical}$ (α = 0.05, m = 2, n = 10)			0.602	0.602	0.602
$C_{critical}$ (α = 0.01, m = 2, n = 10)			0.718	0.718	0.718
$C < C_{critical}$			yes	yes	yes
Outliers			no evidence for analytical outliers	no evidence for analytical outliers	no evidence for analytical outliers
Homogeneity test					
General average $\bar{x}$			0.347	4.644	0.260
Standard deviation of sample averages s_x			0.016	0.156	0.0114
Wthin-sample standard deviation s_w			0.020	0.242	0.0142
Between-sample standard deviation s_s			0.006	0.000	0.005
Standard deviation for proficiency assessment σ_{PT}			0.07	0.93	0.052
s_s / σ_{PT}			0.100	0.0	0.100
Test for homogeneity ($s_s \leq 0.3\ \sigma_{PT}$)			passed	passed	passed

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2022 [EURL-PT-POP_2203-FM]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Fish Meal (2203-FM)
Selected compounds - Stability test - Data

Sample	Replicate	Result µg/kg product (12% moisture content)	**PFOA**	**PFDA**
33	1		0.352	0.284
	2		0.320	0.267
162	1		0.325	0.225
	2		0.318	0.250
221	1		0.320	0.213
	2		0.307	0.252
Stability test				
General average (stability test) $\bar{y}$			0.324	0.25
General average (homogeneity test) $\bar{x}$			0.347	0.26
Standard deviation for proficiency assessment σPT			0.07	0.05
$\lvert \bar{y} - \bar{x} \rvert$			0.02	0.01
Test for stability ($\lvert \bar{y} - \bar{x} \rvert \leq 0.3$ σPT)			passed	passed

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2022 [EURL-PT-POP_2203-FM]

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

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Annex 6: Overview participants' methods – Weighed sample, internal and recovery standards and comments

Test sample - Fish Meal (2203-FM)

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2023 [EURL-PT-POP_2203-FM]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Fish Meal (2203-FM)
Methods Perfluoroalkylcarboxylic acids (PFCAs), Perfluoroalkylsulfonic acids (PFSAs) and Perfluorooctanesulfonamide (FOSA) - Weighed sample and internal standards

LC	Sample	Weighed sample [g]	Use of isotope-labelled internal standards for … PFCAs (yes/no)	PFSAs (yes/no)	FOSA (yes/no)	Other internal standards	Use of recovery / injection standard(s) (yes/no)	Matrix calibration (yes/no)
1	2203-FM	1.5	yes	yes	yes		no	no
3	2203-FM	2 and 1	yes	yes			no	no
4	2203-FM	5	yes	yes	no			
5	2203-FM	2	Yes	Yes	No		Yes	No
7	2203-FM	1	yes	yes	yes		yes	no
8	2203-FM	2	yes	yes	yes		yes	yes
16	2203-FM	5	yes : MPFAC-C-ES from Wellington Laboratories	yes : MPFAC-C-ES from Wellington Laboratories			yes : MPFAC-C-IS from Wellington Laboratories	no
20	2203-FM	2	Yes	Yes	Yes		No	No
23	2203-FM	1	yes	yes	yes		no	no
25	2203-FM	2	yes	yes			no	no
26	2203-FM	1	yes	yes	yes		no	no
28	2203-FM	2	yes	yes	yes		yes	no
29	2203-FM	1	YES	YES			YES	NO
43	2203-FM	5	Yes	Yes	Yes		Yes	Yes
46	2203-FM	2	yes	yes			yes	no
49	2203-FM	1	Yes	Yes			Yes	
56	2203-FM	1	yes	yes	yes		yes	no
59	2203-FM	5	yes	yes			yes	yes
62	2203-FM	0.5	Y	Y	Y		Y	N
63	2203-FM	0.3	yes	tes	yes		no	yes
67	2203-FM	5	yes	yes	no			
68	2203-FM	0.75	yes	yes	yes		yes	no
72	2203-FM	2	yes	yes	-	-	no	no
75	2203-FM	0.1 g / 0.5 g / 2 g	yes	yes			no	no
76	2203-FM	1	yes	yes	no		no	no
80	2203-FM	1	yes	yes	no		no	no
83	2203-FM	1	yes	yes	yes		yes	no
84	2203-FM	2	yes	yes			yes	no
85	2203-FM	2	yes	yes	yes		no	no
92	2203-FM	2	YES	YES	YES		YES	NO
93	2203-FM	2	yes	yes	no		no	yes
94	2203-FM	2	Yes	Yes	no		Yes	No
106	2203-FM	5 g	Yes	Yes	Yes	no	no	no
110	2203-FM	0.118	No	Yes			yes	no

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2023 [EURL-PT-POP_2203-FM]
EU-RL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Fish Meal (2203-FM)
Methods Perfluoroalkylcarboxylic acids (PFCAs) - Internal Standards

LC	Sample		Perfluorobutanoic acid **(PFBA)**	Perfluoropentanoic acid **(PFPeA)**	Perfluorohexanoic acid **(PFHxA)**	Perfluoroheptanoic acid **(PFHpA)**	Perfluorooctanoic acid **(PFOA)**	Perfluorononanoic acid **(PFNA)**	Perfluorodecanoic acid **(PFDA)**	Perfluoroundecanoic acid **(PFUnDA)**	Perfluorododecanoic acid **(PFDoDA)**	Perfluorotridecanoic acid **(PFTrDA)**	Perfluorotetradecanoic acid **(PFTeDA)**
1	2203-FM		MPFBA	MPFHxS	MPFHxA	MPFOS	MPFOA	MPFNA	MPFDA	MPFUnDA	MPFDoDA	MPFDoDA	MPFTeDA
3	2203-FM		PFBA-13C2	PFPeA-13C5	PFHxA-13C2	PFOA-13C4	PFOA-13C4	PFNA-13C5	PFDA-13C2	PFUnDA-13C2	PFDoDA-13C2		
4	2203-FM			PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUnDA	PFDoDA	PFDoDA	PFTeDA
5	2203-FM		13C4-PFBA	13C5-PFPeA	13C5-PFHxA	13C4 PFHpA	13C8-PFOA	13C9-PFNA	13C6-PFDA	13C7-PFUnDA	13C7-PFUnDA	13C2-PFTeDA	13C2-PFTeDA
7	2203-FM		13C-PFBA	13C-PFPeA	13C-PFHxA	13C-PFHpA	13C-PFOA	13C-PFNA	13C-PFDA	13C-PFDoDA	13C-PFDoDA	13C-PFOS	13C-PFTeDA
8	2203-FM												
16	2203-FM			MPFPeA	MPFHxA	MPFHpA	MPFOA	MPFNA	MPFDA	MPFUnDA	MPFDoDA	MPFDoDA	
20	2203-FM		Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
23	2203-FM		MPFBA		MPFHxA	M4PFHpA	MPFOA	MPFNA	MPFDA	MPFUdA	MPFDoDA	MPFDoDA	M2PFTeDA
25	2203-FM						13C8-PFOA	13C5-PFNA					
26	2203-FM		13C4-PFBA	13C5-PFPeA	13C5-PFHxA	13C4-PFHpA	13C4-PFOA	13C4-PFOA	13C6-PFDA	13C6-PFDA	13C6-PFDA	13C6-PFDA	13C6-PFDA
28	2203-FM		13C4-PFBA	13C5-PFPeA	13C2-PFHxA	13C4-PFHpA	13C8-PFOA	13C5-PFNA	13C2-PFDA	13C2-PFUnA	13C2-PFDoA	13C2PFTeDA	13C2PFTeDA
29	2203-FM		13C PFBA	13C PFPA	13C PFHxA	13C PFHpA	13C PFOA	13C PFNA	13C PFDA	13C PFUnDA	13C PFDoDA	13C PFDoDA	13C PFDoDA
43	2203-FM		13C4PFBA	13C5PFPeA	13C5PFHxA	13C4PFHpA	13C8PFOA	13C9PFNA	13C6PFDA	13C7PFUnA	13C2PFDoDA	13C2PFDoDA	13C2PFTeDA
46	2203-FM				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		
49	2203-FM						13C8-PFOA	13C9-PFNA					
56	2203-FM		13C4PFBA	13C5PFPeA	13C5PFHxA	13C4PFHpA	13C8PFOA	13C9PFNA	13C6PFDA	13C7PFUnDA	13C2PFDoA	13C2PFDoA	13C2PFTeDA
59	2203-FM					13C-PFHpA	13C-PFOA	13C-PFNA	13C-PFDA	13C-PFUnDA	13C-PFDoDA		13C-PFTeDA
62	2203-FM		13C PFBA	13C PFPeA	13C PFHxA	13C PFHpA	13C PFOA	13C PFNA	13C PFDA	13C PFUnDA	13C PFDoDA	13C PFTeDA	13C PFTeDA
63	2203-FM				MPFHxA	MPFHpA	MPFOA	MPFNA	MPFDA	MPFUnDA	MPFDoA	MPFDoA	MPFTeA
67	2203-FM						ILIS PFOA	ILIS PFNA					
68	2203-FM		13C-PFBA	13C-PFPeA	13C-PFHxA	13C-PFHpA	13C-PFOA	13C-PFNA	13C-PFDA	13C-PFUnDA	13C-PFDoDA	13C-PFDoDA	13C-PFTeDA
72	2203-FM		x	x	x	x	x	x	x	x	x	x	x
75	2203-FM		13C4-PFBA	13C5-PPeA	13C5-PFHxA	13C4-PFHpA	13C8-PFOA	13C9-PFNA	13C6-PFDA	13C7-PFUnDA	13C2-PFDoDA	13C2-PFTeDA	13C2-PFTeDA
76	2203-FM		MPFBA	M3PFPeA	MPFHxA	M4PFHpA	MPFOA	MPFNA	MPFDA	MPFUdA	MPFDoA	M2PFTeDA	M2PFTeDA
80	2203-FM		M4PFBA	M5PFPeA	MPFHxA	M4PFHpA	M4PFOA	M5PFNA	M6PFDA	M7PFUnDA	M2PFDoDA	M2PFDoDA	M2PFTeDA
83	2203-FM		13C-PFBA	13C-PFPeA	13C-PFHxA	13C-PFHpA	13C-PFOA	13C-PFNA	13C-PFDA	13C-PFUnDA	13C-PFDoDA	13C-PFTeDA	13C-PFTeDA
84	2203-FM		13C4-PFBA	13C5-PFPeA	1,2,3,4,6-13C5-PFHxA	1,2,3,4-13C4-PFHpA	13C8-PFOA	13C9-PFNA	1,2,3,4,5,6-13C6-PFDA	1,2,3,4,5,6,7-13C7-PFUnDA	1,2-13C2-PFDoDA	1,2-13C2-PFDoDA	1,2-13C2-PFTeDA
85	2203-FM		MPFBA	MPFPeA	MPFHxA	MPFHpA	MPFOA	MPFNA	MPFDA	MPFUnDa	MPFDoDA	MPFTeDA	MPFTeDA
92	2203-FM		13C4-PFBA	13C5-PFPeA	13C5-PFHxA	13C4-PFHpA	13C8-PFOA	13C9-PFNA	13C6-PFDA	13C7-PFUnDA	13C2-PFDoDA	13C2-PFDoDA	13C2-PFTeDA
93	2203-FM		13C4-PFBA	13C5-PFPeA	13C5-PFHxA	13C4-PFHpA	13C8-PFOA	13C9-PFNA	13C6-PFDA	13C7-PFUdA	13C2-PFDoA	13C2-PFTeDA	13C2-PFTeDA
94	2203-FM		13C3-PFBA	13C5-PFPeA	13C5-PFHxA	13C4PFHpA	13C8-PFOA	13C9-PFNA	13C6-PFDA	13C7-PFUnDA	13C-PFDoDA	13C12-PFDods	13C2-PFTeDA
106	2203-FM		MPFBA	M5PFPeA	13C2-PFHxA	M4PFHpA	13C4-PFOA	13C-PFNA	13C2-PFDA	13C2-PFUnDA	13C2-PFDoDA	13C2-PFDoDA	M2PFTeDA
110	2203-FM												

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

Fish Meal (2203-FM)
Methods Perfluoroalkylcarboxylic acids (PFCAs) - Recovery Standards

LC	Sample	Perfluorobutanoic acid **(PFBA)**	Perfluoropentanoic acid **(PFPeA)**	Perfluorohexanoic acid **(PFHxA)**	Perfluoroheptanoic acid **(PFHpA)**	Perfluorooctanoic acid **(PFOA)**	Perfluorononanoic acid **(PFNA)**	Perfluorodecanoic acid **(PFDA)**	Perfluoroundecanoic acid **(PFUnDA)**	Perfluorododecanoic acid **(PFDoDA)**	Perfluorotridecanoic acid **(PFTrDA)**	Perfluorotetradecanoic acid **(PFTeDA)**
1	2203-FM											
3	2203-FM											
4	2203-FM		PFBA	PFBA	PFOA	PFOA	PFOA	PFDA	PFDA	PFDA	PFDA	PFDA
5	2203-FM	13C3-PFBA	13C3-PFBA	13C2-PFOA	13C2-PFOA	13C2-PFOA	13C2-PFOA	13C2-PFDA	13C2-PFDA	13C2-PFDA	13C2-PFDA	13C2-PFDA
7	2203-FM	R-PFBA	R-PFOS	R-PFOA	R-PFOA	R-PFOA	R-PFOA	R-PFDA	R-PFDA	R-PFDA		R-PFDA
8	2203-FM											
16	2203-FM		MPFOA inj	MPFOA inj	MPFOA inj	MPFOA inj	MPFOA inj	MPFDA inj	MPFDA inj	MPFDA inj	MPFDA inj	
20	2203-FM											
23	2203-FM											
25	2203-FM					no	no					
26	2203-FM											
28	2203-FM	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA
29	2203-FM	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS
43	2203-FM	13C3PFBA	13C3PFBA	13C3PFOA	13C3PFOA	13C3PFOA	13C4PFOS	13C2PFDA	13C2PFDA	13C2PFDA	13C2PFDA	13C2PFDA
46	2203-FM			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		
49	2203-FM					13C2-PFOA	13C2-PFOA					
56	2203-FM	13C3 PFBA	13C3 PFBA	13C2 PFHxA	13C2 PFHxA	13C4 PFOA	13C5 PFNA	13C2 PFDA	13C2 PFDA	13C2 PFDA	13C2 PFDA	13C2 PFDA
59	2203-FM					13C8-PFOA						
62	2203-FM	M2PFOA	M2PFOA	M2PFOA	M2PFOA	M2PFOA	M2PFOA	M2PFDA	M2PFDA	M2PFDA	M2PFDA	M2PFDA
63	2203-FM											
67	2203-FM											
68	2203-FM											
72	2203-FM	-	-	-	-	-	-	-	-	-	-	-
75	2203-FM											
76	2203-FM	none	none	none	none	none	none	none	none	none	none	none
80	2203-FM	-	-	-	-	-	-	-	-	-	-	-
83	2203-FM	13C2-PFHxA	13C2-PFHxA	13C2-PFHxA	13C2-PFHxA	13C5-PFNA	13C5-PFNA	13C5-PFNA	13C5-PFNA	13C5-PFNA		13C5-PFNA
84	2203-FM	2,3,4-13C3-PFBA	2,3,4-13C3-PFBA	1,2-13C2-PFOA	1,2-13C2-PFOA	1,2-13C2-PFOA	1,2-13C2-PFOA	1,2-13C2-PFDA	1,2-13C2-PFDA	1,2-13C2-PFDA	1,2-13C2-PFDA	1,2-13C2-PFDA
85	2203-FM											
92	2203-FM	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS
93	2203-FM	No	No	No	No	No	No	No	No	No	No	No
94	2203-FM											
106	2203-FM	none	none	none	none	none	none	none	none	none	none	none
110	2203-FM											

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

Fish Meal (2203-FM)
Methods Perfluoroalkylcarboxylic acids (PFCAs) - Comments

LC	Sample	Perfluorobutanoic acid **(PFBA)**	Perfluoropentanoic acid **(PFPeA)**	Perfluorohexanoic acid **(PFHxA)**	Perfluoroheptanoic acid **(PFHpA)**	Perfluorooctanoic acid **(PFOA)**	Perfluorononanoic acid **(PFNA)**	Perfluorodecanoic acid **(PFDA)**	Perfluoroundecanoic acid **(PFUnDA)**	Perfluorododecanoic acid **(PFDoDA)**	Perfluorotridecanoic acid **(PFTrDA)**	Perfluorotetradecanoic acid **(PFTeDA)**
1	2203-FM					only linear form	only linear form					
3	2203-FM											
4	2203-FM											
5	2203-FM	Only 1 MRM monitored	Matrix interference present	Matrix interference present								
7	2203-FM	Content could not be confirmed by HILIC method; MU not determined	MU not determined	MU not determined								
8	2203-FM											
16	2203-FM	Not analysed										Not analysed
20	2203-FM											
23	2203-FM		Not analysed									
25	2203-FM											
26	2203-FM	not detected	not detected	not detected	not detected					not detected	not detected	not detected
28	2203-FM											
29	2203-FM											
43	2203-FM											
46	2203-FM											
49	2203-FM											
56	2203-FM											
59	2203-FM											
62	2203-FM			New LOQ								
63	2203-FM	not analysed	not analysed									
67	2203-FM											
68	2203-FM											
72	2203-FM		Wert gefunden jedoch < LOQ	Wert gefunden jedoch < LOQ								
75	2203-FM					Blank concentrations were subtracted						
76	2203-FM											
80	2203-FM											
83	2203-FM											
84	2203-FM											
85	2203-FM											
92	2203-FM											
93	2203-FM											
94	2203-FM											
106	2203-FM											
110	2203-FM											

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

Fish Meal (2203-FM)
Methods Perfluoroalkylsulfonic acids (PFSAs) - Internal Standards

LC	Sample		Perfluorobutanesulfonic acid (PFBS)	Perfluoropentanesulfonic acid (PFPeS)	Perfluorohexanesulfonic acid (PFHxS)	Perfluoroheptanesulfonic acid (PFHpS)	Linear Perfluorooctanesulfonic acid (L-PFOS)	Sum of branched PFOS acids (br-PFOS)	Total-Perfluorooctanesulfonic acids (total PFOS)	Perfluorononanesulfonic acid (PFNS)	Perfluorodecanesulfonic acid (PFDS)	Perfluoroundecane-sulfonic acid (PFUnDS)	Perfluorododecane-sulfonic acid (PFDoDS)	Perfluorotridecane-sulfonic acid (PFTrDS)
1	2203-FM		MPFHxS	MPFOS	MPFHxS	MPFHxS	MPFOS	MPFOS	MPFOS	MPFHxS	MPFHxS	MPFHxS	MPFBA	MPFHxS
3	2203-FM				PFHxS-18O2	PFOS-13C4	PFOS-13C4	PFOS-13C4						
4	2203-FM		PFBS	PFBS	PFHxS	PFHxS	PFOS	PFOS		PFOS	PFOS		PFOS	
5	2203-FM		13C3-PFBS	13C3-PFBS	13C3-PFBS	13C3-PFBS	13C8-PFOS	13C8-PFOS	13C8-PFOS	13C3-PFHxS	13C3-PFHxS			
7	2203-FM		13C-PFBS	13C-PFHpA	13C-PFHxS	13C-PFOA	13C-PFOS	13C-PFOS		13C-PFOS	13C-PFUnDA	13C-PFDoDA	13C-PFTeDA	13C-PFDoDA
8	2203-FM													
16	2203-FM		MPFBS	MPFHxS	MPFHxS	MPFHxS	MPFOS	MPFOS	MPFOS	MPFHxS	MPFHxS	MPFHxS	MPFHxS	MPFHxS
20	2203-FM		Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
23	2203-FM		MPFHxS		MPFHxS		MPFOS				MPFOS			
25	2203-FM				13C6-PFHxS		13C8-PFOS							
26	2203-FM													
28	2203-FM		13C3-PFBS	13C3-PFBS	18O2-PFHxS	18O2-PFHxS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C5-PFNA	13C2-PFDA	13C2PFUnA	13C2-PFDoA	13C2-PFTeDA
29	2203-FM		13C PFBS	13C PFBS	13C PFHxS	13C PFHxS	13C PFOS	13C PFOS	13C PFOS	13C PFUnDA	13C PFUnDA	13C PFUnDA	13C PFUnDA	13C PFUnDA
43	2203-FM		13C3PFBS	13C3PFBS	13C3PFHxS	13C3PFHxS			13C8PFOS	13C8PFOS	13C8PFOS		13C8PFOS	
46	2203-FM		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.					
49	2203-FM				13C3-PFHxS		13C8-PFOS							
56	2203-FM		13C3 PFBS	13C3 PFBS	13C3 PFHxS	13C3 PFHxS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS		13C8 PFOS	
59	2203-FM		13C-PFBS		13C-PFHxS	13C-PFHxS	13C-PFOS	13C-PFOS	13C-PFOS		13C-PFDoDA			
62	2203-FM		13C PFBS		13C PFHxS	13C PFOS	13C PFOS	13C PFOS	13C PFOS					
63	2203-FM				MPFHxS	MPFOS	MPFOS	MPFOS	MPFOS		MPFOS			
67	2203-FM				ILIS PFHxS		ILIS L-PFOS	ILIS L-PFOS						
68	2203-FM		13C-PFBS	13C-PFHpA	13C-PFHxS	13C-PFNA	13C-PFOS	13C-PFOS		2H-N-EtFOSAA	13C-PFDoDA	13C-PFDoDA	13C-PFTeDA	13C-PFTeDA
72	2203-FM		x	x	x	x	x	x	x		x			
75	2203-FM				13C3-PFHxS		13C8-PFOS		13C8-PFOS					
76	2203-FM		M3PFBS		MPFHxS				MPFOS					
80	2203-FM		M3PFBS	M3PFBS	M3PFHxS	M4PFOS	M4PFOS	M4PFOS	M4PFOS	M4PFOS	M4PFOS			
83	2203-FM		13C-PFBS	13C-PFBS	13C-PFHxS	13C-PFHxS	13C-PFOS	13C-PFOS	13C-PFOS	13C-PFOS	13C-PFOS			
84	2203-FM		2,3,4-13C3-PFBS	1,2,3-13C3-PFHxS	1,2,3-13C3-PFHxS	1,2,3-13C3-PFHxS	13C8-PFOS			13C8-PFOS	13C8-PFOS		13C8-PFOS	
85	2203-FM		MPFBS	MPFHxS	MPFHxS	MPFHxS	MPFOS	MPFOS		MPFHxS	MPFOS		MPFDoDA	
92	2203-FM		13C3-PFBS	13C3-PFHxS	13C3-PFHxS	13C3-PFHxS	13C8-PFOS	13C8-PFOS		13C8-PFOS	13C6-PFDA		13C2-PFDoDA	
93	2203-FM		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	
94	2203-FM		13C3-PFBS	13C3-PFHxS	13C3-PFHxS	13C8-PFOS	13C8-PFOS			13C8-PFOS	13C8-PFOS			
106	2203-FM		M3PFBS	18O-PFHxS	18O-PFHxS	18O-PFHxS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS
110	2203-FM													

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Fish Meal (2203-FM)
Methods Perfluoroalkylsulfonic acids (PFSAs) - Recovery Standards

LC	Sample		Perfluorobutanesulfonic acid **(PFBS)**	Perfluoropentanesulfonic acid **(PFPeS)**	Perfluorohexanesulfonic acid **(PFHxS)**	Perfluoroheptanesulfonic acid **(PFHpS)**	Linear Perfluorooctanesulfonic acid **(L-PFOS)**	Sum of branched PFOS acids **(br-PFOS)**	Total-Perfluorooctanesulfonic acids **(total PFOS)**	Perfluorononanesulfonic acid **(PFNS)**	Perfluorodecanesulfonic acid **(PFDS)**	Perfluoroundecane-sulfonic acid **(PFUnDS)**	Perfluorododecane-sulfonic acid **(PFDoDS)**	Perfluorotridecane-sulfonic acid **(PFTrDS)**
1	2203-FM													
3	2203-FM													
4	2203-FM		PFOS	PFOS	PFOS	PFOS	PFOS	PFOS		PFOS	PFOS		PFOS	
5	2203-FM		13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS			
7	2203-FM		R-PFOS		R-PFOS		R-PFOS							
8	2203-FM													
16	2203-FM		MPFOA inj	MPFOA inj	MPFOA inj	MPFOA inj	MPFOS inj	MPFOS inj	MPFOS inj	MPFOA inj	MPFOA inj	MPFOA inj	MPFOA inj	
20	2203-FM													
23	2203-FM													
25	2203-FM				no		no							
26	2203-FM													
28	2203-FM		13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA
29	2203-FM		13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS	13C8 PFOS
43	2203-FM		13C3PFBA	13C3PFBA	13C2PFOA	13C2PFOA			13C4PFOS	13C4PFOS	13C4PFOS		13C4PFOS	
46	2203-FM		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					
49	2203-FM				13C4-PFOS		13C4-PFOS							
56	2203-FM		PFHxS18O2	PFHxS18O2	PFHxS18O2	PFHxS18O2	13C4 PFOS	13C4 PFOS	13C4 PFOS	13C4 PFOS	13C4 PFOS		13C4 PFOS	
59	2203-FM						13C8-PFOS	13C8-PFOS	13C8-PFOS					
62	2203-FM		MPFOS		MPFOS	MPFOS	MPFOS	MPFOS	MPFOS					
63	2203-FM													
67	2203-FM													
68	2203-FM													
72	2203-FM		-	-	-	-	-	-	-		-			
75	2203-FM													
76	2203-FM		none	none	none	none	none	none	none	none	none	none	none	none
80	2203-FM		-	-	-	-	-	-	-	-	-	-	-	-
83	2203-FM		13C2-PFHxA	13C2-PFHxA	13C2-PFHxA	13C2-PFHxA	13C5-PFNA			13C5-PFNA	13C5-PFNA			
84	2203-FM		1,2,3,4-13C4-PFOS	1,2,3,4-13C4-PFOS	1,2,3,4-13C4-PFOS	1,2,3,4-13C4-PFOS	1,2,3,4-13C4-PFOS			1,2,3,4-13C4-PFOS	1,2,3,4-13C4-PFOS		1,2,3,4-13C4-PFOS	
85	2203-FM													
92	2203-FM		13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS		13C4-PFOS	13C4-PFOS		13C4-PFOS	
93	2203-FM		No	No	No	No	No	No	No	No	No		No	
94	2203-FM													
106	2203-FM		none	none	none	none	none	none	none	none	none	none	none	none
110	2203-FM								107.00					

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Fish Meal (2203-FM)
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)
1	2203-FM							Reference standard: L-PFOS; mass transitions: m/z 499 > 80	Reference standard: L-PFOS; mass transitions: m/z 499 > 80					
3	2203-FM													
4	2203-FM													
5	2203-FM		Matrix interference present	Matrix interference present	Matrix interference present	Matrix interference present						Not measured	Not measured	Not measured
7	2203-FM		MU not determined	ion ratio not correct, content could not be confirmed by HILIC chromatography; MU not determined	ion ratio not correct content was confirmed by HILIC chromatography			L-PFOS; m/z 499 > 80	MU not determined	MU not determined	MU not determined	MU not determined	MU not determined	MU not determined
8	2203-FM													
16	2203-FM													Not analysed
20	2203-FM													
23	2203-FM			Not analysed		Not analysed		Not analysed		Not analysed		Not analysed	Not analysed	Not analysed
25	2203-FM													
26	2203-FM		not detected	not detected	not detected	not detected		not detected		not detected	not detected	not detected	not detected	not detected
28	2203-FM													
29	2203-FM													
43	2203-FM								Native PDS Mix - 499.00>80.0			Do not calibrate for this compound		Do not calibrate for this compound
46	2203-FM													
49	2203-FM													
56	2203-FM							Branched PFOS (Wellington) calibration standards used, m/z 499 > 99				Not tested		Not tested
59	2203-FM							L-PFOS used to quantify br-PFOS (mass transitions 499>99 used as quantifier and 499>80 as qualifier)						
62	2203-FM													
63	2203-FM		not analysed	not analysed						not analysed		not analysed	not analysed	not analysed
67	2203-FM							L-PFOS & techn. Mixture						
68	2203-FM								calculated by adding l-PFOS + br-PFOS					
72	2203-FM													
75	2203-FM						L-PFOS, 499>80		L-PFOS, 499>80					
76	2203-FM													
80	2203-FM							reference Standard: L-PFOS; mass transition: 499 > 80	branched PFOS content is not validated in our method, because we normally only measure L-PFOS			we do not measure this compound	we do not measure this compound	we do not measure this compound
83	2203-FM													
84	2203-FM													
85	2203-FM				Anion		Anion							
92	2203-FM							br-PFOSK Wellington lab; transition: 499>80						
93	2203-FM													
94	2203-FM							No Reference Standard				Not analyzed	Not analyzed	Not analyzed
106	2203-FM							L-PFOS; 498.9 > 98.7, 498.9 > 79.7						
110	2203-FM													

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Fish Meal (2203-FM)
Methods Perfluorooctanesulfonamide (FOSA) - Intern and recovery standards and comments

LC	Sample	Perfluorooctane sulphonamide **(FOSA)**		
		Internal standard	**Recovery standard**	**Comments**
1	2203-FM	MPFBA		only linear form
3	2203-FM			
4	2203-FM			
5	2203-FM			Not measured
7	2203-FM	13C-PFOSA	R-PFOA	
8	2203-FM			
16	2203-FM			
20	2203-FM	Yes		
23	2203-FM	MPFOSA		
25	2203-FM			
26	2203-FM	13C8-PFOSA		not detected
28	2203-FM	13C8-PFOSA	13C4-PFOA	
29	2203-FM			
43	2203-FM	13C8PFOSA	13C2PFDA	
46	2203-FM			
49	2203-FM			
56	2203-FM	13C8 PFOSA	13C4 PFOS	
59	2203-FM			
62	2203-FM			
63	2203-FM	MFOSA		
67	2203-FM			
68	2203-FM	13C-FOSA		
72	2203-FM			
75	2203-FM			
76	2203-FM			
80	2203-FM			we do not measure this compound
83	2203-FM			
84	2203-FM			
85	2203-FM	MFOSA		
92	2203-FM	13C8PFOSA	13C4-PFOS	
93	2203-FM			
94	2203-FM			Not analyzed
106	2203-FM	13C8-PFOSA	none	
110	2203-FM			

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

Test sample - Fish Meal (2203-FM)

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Fish Meal (2203-FM)
Methods Perfluoroalkylcarboxylic acids (PFCAs), Perfluoroalkylsulfonic acids (PFSAs) and Perfluorooctanesulfonamide (FOSA) - Pre-treatment and extraction

LC	Sample	Pre-treatment and extraction					
1	2203-FM	No pre-treatments.	Addition of internal standards.	Extraction: shaking with water/acetonitrile at room temperature.			
3	2203-FM	ultrasonic extraction using aconitrile					
4	2203-FM	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.				
5	2203-FM	The wet sample was extracted using 0.02 M sodium hydroxide in methanol	A portion of the extract was dilured in water and pH adjusted to acidic conditions				
7	2203-FM	# 1 g of sample was extracted by shaking (20 min) and ultrasonication (20 min) using 1,5 mL water and	13 mL 1 % NH3 in ACN	# centrifugation	# evaporation to 1 mL using nitrogen (50 °C)	# addition of 9 mL 25mM NH4Ac buffer (pH = 6)	# centrifugation and transfer to SPE cartridge
8	2203-FM	2 g sample	10 mL Acetonitrile (5+5 mL, 2 times)	Ultrasonic (15 min)	Centrifugation	collection of supernatant to 15 mL centrifuge tube	
16	2203-FM	Quecher extraction (acetonitrile - Nh4OH)	Vortex 1 minute	Centrifugation at 10000 rpm			
20	2203-FM	Quechers					
23	2203-FM	homogenization, weighing and addition of internal standard	extraction with Methanol, shaking, ultrasonic bath, sentrifugation	filtration of supernatant	addition of MilliQ water		
25	2203-FM	Extraction with NaOH methanolic solution					
26	2203-FM	1 g of sample + HCOOH + water + acetonitrile	shaking	addition of NaCl and MgSO4	shaking and centrifugation	removing of aliquot of organic layer for further purification	
28	2203-FM	Quechers-Extraktion					
29	2203-FM	Extraction with MeOH/KOH					
43	2203-FM	In a 50ml centrifuge tube, ~5 grams of PT was weighed. For QC weigh 10mls of MilliQwater in 50ml tubes	Add labeled extraction to all tubes. Add Native PFAS spike to LCS/LCSD	15 mls of MilliQwater was added, vortexed. 10ml of Acetontile and 150 ul of Formic Acid added. Vortexed.	Add 1 packet of Quechers extraction salts, 6000 mg MgSO4/1500 mg NaCL, to each tube vortex.	Place tubes on a benchtop vortexer, vortex for 5 min. Centrifuge for 15 min at 3000 rpm	Transfer supernatant to a 15 ml centrifuge tube, add 100ul MilliQ water. Vortex. Concentrate on NEVAP at 40 degree until 50-100ul remain. Reconsitute to 10mls with MilliQ water, vortex.
46	2203-FM	Extraction was done using 10 ml of methanol/potassium hydroxide for 17 hours.					
49	2203-FM	Ultrasonic extraction with Acetonitrile					
56	2203-FM	Sample spiked with internal standards	Added Methanol with Potassium Hydroxide	Tumbled 8 hours	Centrifuged @ 3000 rpm		
59	2203-FM	Extraction with 200mM sodiumhydroxide and MeOH	Addition of formic acid	Vortex, head-over-head, centrifugation	Dilution with MQ		
62	2203-FM	Sample is fortified with isotopically labelled analogues of the method analytes, which function as isotope dilution standards.	The sample undergoes an alkali digestion followed by extraction with acetonitrile				
63	2203-FM	Liquid extraction with methanol.					
67	2203-FM	to 5g of sample 10ml water was added and let it soaked for 10 minutes	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		
68	2203-FM	Pre-treatment: sample was gently warmed and then slowly stirred prior to weighing		Extraction method: extraction according to a modified version of the QuEChERs method	addition of ACN and Citrat-Extraction-Tube (CET) for QuEChERs	sample measured in ACN-MeOH-H2O mix	
72	2203-FM	Homogenisation	Extraction with methanol				
75	2203-FM	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
76	2203-FM	ad 10 ml water and 10 ml Acetonitril to 1.0 g sample	mechanically shake 5 min, 15 min US-bath				
80	2203-FM	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
83	2203-FM	Exrtraction: ACN with NH4					
84	2203-FM	2 g sample + 5 ml water in polypropylen tubes; addition of internal standard (100 µl á 10 ng/ml)	addition of 10 ml ACN - extraction 5 min shaker and 5 min ultrasonic; addition of 1 ml NaOH (1M) - extraction 15 min shaker and 15 min ultrasonic	phase separation with 0,5 g NaCl - extraction 5 min shaker and 5 min ultrasonic - centrifugation; organic phase in new polyproylen tube	addition of 10 ml ACN to sample - extraction 15 min shaker and 15 min ultrasonic - centrifugation	combine organic phase from second extraction with organic phase from first extraction in polyproylen tube	addition of 10 µl formic acid (98%) to raw extrakt (pH ca. 5-6)
85	2203-FM	10 ml acetonitrile					
92	2203-FM	EXTRACTION WITH ACETONITRILE					
93	2203-FM	Double exctraction with acetonitrile					
94	2203-FM	Alkaline Extraction: sodium hydroxide in methanol					
106	2203-FM	The sample was homogenised	5g of sample was weighed	10 mL of acetonitrile was added followed by 150 uL formic acid. The sample was shaken.	Quecher salt was added and the sample shaken and centrifuged.	5mL supertanatant was transferred to a 15mL dSPE tube, shaken and centrifuged.	The sample was diluted in a 1:1 ratio sample: water and added to an analytical vial.
110	2203-FM	stirring extraction of 0.1 g of fish with 5 ml of methanol					

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Fish Meal (2203-FM)
Methods Perfluoroalkylcarboxylic acids (PFCAs), Perfluoroalkylsulfonic acids (PFSAs) and Perfluorooctanesulfonamide (FOSA) - Clean-up

LC	Sample	Clean-up					
1	2203-FM	Clean up with quechers salts and degreasing by freezing.					
3	2203-FM	degreasing using n-hexane	dispersive SPE using activated carbon				
4	2203-FM	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.	
5	2203-FM	WAX solid phase extraction cartridges					
7	2203-FM	# SPE (Strata X-AW)	# ENVI-Carb 250 mg, 3 mL	# elution using 4 x 1 mL methanol (neutral PFAS) and 4 x 1 mL 3% ammonia in methanol	# evaporation to dryness under nitrogen stream (50 °C)	# addition of recovery standard and MeOH/ 1% formic acid; final volume of 500 µL	# freeze over night and syringe filtration
8	2203-FM	Cleaning extract with dSPE salts (MgSO4, C18, NaCl, GCB)	collection of supernatant	Evaporation to dryness	Reconstitution to 250 uL	Analysis to LC-MS/MS	
16	2203-FM	Mix supernatant with water and pH adaptation at 3	Purification on SPE Strata-X-AW 33µm polymeric weak anion 200mg	Evaporation to 1 ml	Purification on SPE SupelClean Envicarb	Evaporation 0,1 ml, recovery standards addition and MeOH addition up to 0,5 ml	Injection in PP vial
20	2203-FM	Quechers					
23	2203-FM	SPE OASIS WAX	eluting with 1% (V/V) Ammonium Hydroxide in Methanol				
25	2203-FM	extract dilution with water; concentration and clean-up using WAX column					
26	2203-FM	Z-Sep+, C18 and MgSO4	shaking and cetrifugation	removing of aliquot for evaporation	reconsitution in MeOH	microfiltation before UHPLC-MS/MS	
28	2203-FM	SPE CleanUp					
29	2203-FM	SPE Oasis WAX	SPE Envi Carb				
43	2203-FM	Condition Solid Phase Extraction (SPE) cartridges with 0.3% NH4OH in MeOH, MeOH, MilliQwater	Load QC and sample to corresponding SPE Cartridge. If sample won't go through by gravity apply light vacuum.	After full volume passes through, Dry cartridges with vacuum for 5 minutes. Place 15 ml centrifuge under each SPE cartridge.	Add 5 ml of 1:1 ACN:MeOH to each tube vortex, transfer to SPE cartridges and let soak. While soaking add 5 ml of 0.3% NH4OH/MeOH to each tube, vortex.	After 5ml of 1:1 ACN:MeOH goes through, add the next 5 mls of 0.3% and allow it to pass through by gravity Apply vacuum after elutions has gone through to collect any solvent left on SPE cartridges	Concentrate on NEVAP at 40 degree until 400-500 uL remain. Reconstitute to 1ml with 100% MeOH. Vortex. Centrifuge for full cycle. Transfer 400ul of extract into autosampler vial with 20ul labeled internal standard spike
46	2203-FM	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.				
49	2203-FM	Dispersive SPE C18 EnviCarb					
56	2203-FM	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.			
59	2203-FM	SPE (Strata-X-AW, 200mg/6ml, 33um, Phenomenex)	Condition with MeOH and 0.04M HCl in MQ	Washing with 25mM sodium acetate buffer and 0.04M HCl in MeOH	Elution with 2% NH4OH in ACN		
62	2203-FM	The supernatant is then cleaned up by automated solid phase extracction(ASPEC)	The resulting aliquot is evaporated & reconstituted	Additional isotopically labeled standards (recovery standards) are added			
63	2203-FM	None					
67	2203-FM	frozen out with liquid N2, 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			
68	2203-FM						
72	2203-FM	phenomenex: Strata TM -X-AW 33 µm	100 mg 6ml				
75	2203-FM	SPE with Strata HR-XAW	Conditioning with 3 mL 0.1% NH3 in MeOH, 2 mL MeOH, 2 mL 25 mM CH3COONH4 in H2O (pH 4 using acetic acid)	6 mL extract + 6 mL H2O	Washing with 4mL 25 mM CH3COONH4 in H2O (pH 4 using acetic acid), 4 mL Acetone/ACN (50/50), 4 mL MeOH	Elution with 2 mL 0.1% NH3 in MeOH	Keeper (ethylene glycole) was used during evaporation reconstitution with MeOH/H2O (50/50)
76	2203-FM	add first QuCHERS Salt	shake 1 min manually	centrifuge	evaporate 4 ml of supernatant with 200 µl water and 5 µl glycol added to 200 µl total volume	reconstitute with 400 µl ACN/MeOH (10/90) in US bath	centrifuge and pass through filter in vial
80	2203-FM	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
83	2203-FM	SPE: EnviCarb and StrataX					
84	2203-FM	freeze out raw extrakt (-20°C, > 2h) - centrifugation	d-SPE with 900 mg MgSO4 and 250 mg ENVI-Carb (graphitized carbon black)	evaporate extract to 0.3 ml (Buchi Syncore Plus Analyst)	tranfer evaportated extract to centrifugation-tube (Eppendorf-tube)	addition of recovery standard (100 µl á 10 ng/ml)	fill up extract with acid MeOH (0,3% formic acid) to 1 ml (volume is also used for rinsing the evaporation tubes)
85	2203-FM	d-SPE C18 and ENVI-carb					
92	2203-FM	SPE CLEAN-UP WITH STRATA X-AW 200 mg - dSPE-Envicarb 80mg					
93	2203-FM	Weak anionic exchange (WAX) SPE plus graphitized carbon black (Envicarb)					
94	2203-FM	SPE OASIS WAX					
106	2203-FM	Quecher salts were used as a clean up method.					
110	2203-FM	no					

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Fish Meal (2203-FM)
Methods Perfluoroalkylcarboxylic acids (PFCAs), Perfluoroalkylsulfonic acids (PFSAs) and Perfluorooctanesulfonamide (FOSA) - Chromatographic separation and detection

LC	Sample	Chromatographic separation and detection				
1	2203-FM	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)
3	2203-FM	seperation on reversed phase C18	detection in multiple reaction monitoring mode (ESI neg)			
4	2203-FM	Chromatographic column: Acquity UPLC BEH Shield RP18 COLUMN (1,7 um 2,1x 100 mm)	Mobile phase: (A) Water 10 mM of ammonium acetate	(B) Methanol:Acetonitrile (1:1)	LC-Q-ORBITRAP (ESI-)	
5	2203-FM	Acquity HSS T3 C18 100x2.1 1.8micron		MPA = 0.2 mM ammonium acetate	MPB = methanol	LC-MS/MS (Xevo TQ Absolute); ESI negative; Isotope dilution using solvent standards was used for quantify the sample
7	2203-FM	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
8	2203-FM	Waters Xterra C18 (100x2.1 mm, 3.5 um)	Mobile Phase A: MeOH	Mobile Phase B: 5 mM Ammonium Acetate	Gradient Program of elution	Liquid Chromatography: EXION LC AD pump; Mass Spectrometer:SCIEX QTRAP 6500+
16	2203-FM	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
20	2203-FM	Gradient with MS/MS detection				
23	2203-FM	LC- MSMS				
25	2203-FM	HPLC using C18-phase, Orbitrap-MS detection in PRM mode				
26	2203-FM	UHPLC-MS/MS analysis	5 ul injection, (A) 5 mM ammonium acetate in water and (B) MeOH	BEH C18 (100 × 2,1 mm; 1,7 µm); gradient elution	tandem mass spectrometry (triple quad, MRM mode), electrospray ionization in negative mode	
28	2203-FM	Measurement: HPLC	Injection method: standard	Injection volume (µl): 5	Stationary phase: C18-Phase	Detector: MS/MS ; Scan mode: MRM
29	2203-FM	LC-(ESI-)-MS/MS	Hypersil Gold (100 x 2,1 mm) 1,9 µm			
43	2203-FM	After the isotopically-labeled internal standands are added to the sample, it is analyzed by LC/MS/MS operated	in negative electrospray ionization (ESI) mode for detection and quantification of the analytes.	Quantitative analysis is performed using isotope dilution		
46	2203-FM	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	
49	2203-FM	LC-MS/MS				
56	2203-FM	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 calibration standards
59	2203-FM	LC-MS/MS	Column: Acquity UPLC BEH C18 (50mm*2.1mm i.d., 1.7 um) + Symmetry C18 (50mm x 2.1mm i.d., 1.7um) as isolator-column	Mobile phase A: 2mM ammonium acetate in MQ	Mobile phase B: ACN	
62	2203-FM	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	
63	2203-FM	LC-MS/MS				
67	2203-FM	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
68	2203-FM	LC-MS/MS, reversed phase, column: Gemini, MRM measurement,	mobile phase 1: 95 % water + 5% ACN + 2mM ammonium acetate	mobile phase 2: 40 % MeOH + 60 % ACN		
72	2203-FM	HPLC Agilent 1260 Infinity II	Infinity Lab PFC Delay Column	Infinity Lab PFC Poroshel 120 EC C18		MS Agilent 6495
75	2203-FM	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
76	2203-FM	column: C18, Gemini® 3 µm C18 110Å, 150 x 2 mm	retention column: C18, z. B. Accucore C18 2.1 x 100 mm 2.6 µm	Eluent A: 20 mM Ammoniumacetate in Water; Eluent B: MeOH	Flow 0,3 ml/min	Gradient: 0 min, 10%B / 2 min, 10%B / 9min,85%B / 12min,98%B / 25min,98%B / 30min, 10%B
80	2203-FM	LCQQQ (Agilent Technologies 1290 Infinity, Agilent Technologies 6490)	Pre-Column and Column Zorbax Eclipse XDB-C18	Flow rate: 0,3 mL/min, Column Temp: 40 °C, injection volume: 1 µL	Eluent A: 0,1 mmol NH4OAc in 0,1% HCOOH (Milli Q water); Eluent B: 0,1 mmol NH4OAc 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
83	2203-FM	HPLC on a C18 column; Detektion: MS/MS				
84	2203-FM	LC-MS/MS is equpied with online-SPE (Gerstel) - automated enrichment of sample on miniaturized WAX-cartriges	LC-column: Poroshell 12 EC-C18 (3,0mm x 100 mm / 2,7 µm)	detection of analytes and internal standards with dMRM ; calibration in solevent (MeOH)	quantification about response ratios of analytes/internal standards calculation recovery about response ratio of internal standards/recovery standards (calibration =100%)	verification of PFBA/PFPeA with Raptor polar X
85	2203-FM	LC-MS/MS reversed phase chromatography				
92	2203-FM	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
93	2203-FM	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-)		
94	2203-FM	Mobile phase: ammonium acetate 2mM In water and ammonium acetate 2mM in methanol	Column: BEH C18 2*10mm 1,7um	Detection by MS/MS		
106	2203-FM	Chromatographic separation and detection was achieved using a combination of UPLC-MS/MS	Column: Acquity UPLC BeH C18 1.7 um, 2.1x100 mm column	Detection: XEVO TQ-XS ESI		
110	2203-FM	HPLCMS witch colum C18.				

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

Test sample - Fish Meal (2203-FM)

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2023 [EURL-PT-POP_2203-FM]
EU-RL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Fish Meal (2203-FM)
Methods Perfluoroalkylcarboxylic acids (PFCAs), Perfluoroalkylsulfonic acids (PFSAs) and Perfluorooctanesulfonamide (FOSA) - LOQ determination, measurement uncertainty calculation and additional information

LC	Sample	Limit of quantification (LOQ) approach	Measurement Uncertainty (MU) approach	Additional Information
1	2203-FM	By spiking samples at LOQ concentration level.		
3	2203-FM			
4	2203-FM	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”.	
5	2203-FM	The lowest standard meeting the method performance (S:N, ion ratio, RT tolerance) included in the batch	Not calculated as the method is still under development and not validated	This method is under development and not previously tested in fish meal matrix
7	2203-FM	The LOQ was estimated using a S/N of 3 for the less intensive mrm transition. LOQs will likely be higher ones estimated during validation	U = k * u` (k = 2); The measurement uncertainty was determined using four fortified sample of the PT sample (Bias) and six replicates of the PT sample (precision). Sum parameter: U = square root of sum of squares of expanded MU of indivitual PFAS	
8	2203-FM	Spiking in matrix blank with low concentration standard solution	Combination of precision (intermediated precision) and bias component; Expanded Uncertainty multiplying by factor k=2 for 95 % confidence level	
16	2203-FM	The LOQs corespond to the lowest validated level for validated matrices	U= k*u=((2x CVrw) + biais) for validated matrices	
20	2203-FM			
23	2203-FM			
25	2203-FM	first calibration level (provisional first validation level)		
26	2203-FM	as lowest calibration point where S/N > 10 (quantification transition) and S/N > 3 (confirmation transition)		
28	2203-FM			
29	2203-FM			
43	2203-FM			
46	2203-FM	LOD and LOQ were estimated based on analysis of 10 blank spiked samples	MU were estimated based on precision and truenes from fortyfication experiment	
49	2203-FM			
56	2203-FM	Performance of calibration standards; Batch blank level	Top down estimation from reproducibility of spiked samples	
59	2203-FM			
62	2203-FM			
63	2203-FM			
67	2203-FM	during validation of PFAS in fish matrix	during validation of PFAS in fish matrix	
68	2203-FM			
72	2203-FM	calculated by matrixkurve (validation)	Nord Test	
75	2203-FM	S/N-ratio according to the Guidance Document; PFOA: 3 x blank concentrations		
76	2203-FM	MSMS-Triple Quadrupol 6495 Agilent, Turbo Ionspray (ESI) mit Agilent Jet Strea; ESI negative	According to SANTE/11312/2021	
80	2203-FM	Measurement of different spiked samples; LOQ is the value were the following criteria are fulfilled: Recovery: 70-120%; RSD </=20%	Analysis of the QM-samples over a year; Multiply the uncertainty with 2 to get the MU	
83	2203-FM	Calibration curve, lowest calibationpoint	according to the guidance document	
84	2203-FM	method is actually validated for fresh meat, fish, egg, milk (food); LOQ is lowest validated level - fresh fish ca. 0,01 µg/kg; weight of 2 g fish meal instead of 5 g fresh fish -> factor 2,5 for LOQ	individuall calculation for PFOS, PFOA, PFNA, PFHxS at levels > 0,1 µg/kg for fresh samples calculation with PT-results, validation results, qualitty control samples; (calculation with AQS-form)	
85	2203-FM	lowest validated level	According EURL Guidance Document PFAS	
92	2203-FM	Spiked samples at the LOQ	From validation data with a semplified bottom up approach	
93	2203-FM		Application of Horwitz-Thompson equation	
94	2203-FM			
106	2203-FM			For the compound FOSA a recovery-correction for matrix effects was performed. The spike of FOSA to the sample gave a reproducible (n = 8) recovery of 155% and the result; was corrected for the observed recovery: measured value of 0.72 µg/kg (88%dw) was corrected to 0.45 µg/kg (88%dw)
110	2203-FM			

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

Fish Meal (2203-FM)
Methods Perfluoroalkylcarboxylic acids (PFCAs) - Measurement Uncertainty [%]

LC	Sample		Perfluorobutanoic acid **(PFBA)**	Perfluoropentanoic acid **(PFPeA)**	Perfluorohexanoic acid **(PFHxA)**	Perfluoroheptanoic acid **(PFHpA)**	Perfluorooctanoic acid **(PFOA)**	Perfluorononanoic acid **(PFNA)**	Perfluorodecanoic acid **(PFDA)**	Perfluoroun- decanoic acid **(PFUnDA)**	Perfluorodo- decanoic acid **(PFDoDA)**	Perfluorotri- decanoic acid **(PFTrDA)**	Perfluorotetra- decanoic acid **(PFTeDA)**
1	2203-FM		45			45	45	45	45	45	45	45	45
3	2203-FM		45			30	25	25	25	25	25		
4	2203-FM			32	34	26	32	23	32	33	33	34	21
5	2203-FM												
7	2203-FM					5	5	8	6	9	11	38	9
8	2203-FM		30	34	28	29	23	22	25	24		36	37
16	2203-FM			30	30	30	30	30	30	30	30	30	
20	2203-FM		37	37	37	37	37	37	37	37	37	37	37
23	2203-FM		35		60	50	35	35	40	40	40	80	80
25	2203-FM												
26	2203-FM						25	20	25	25			
28	2203-FM												
29	2203-FM												
43	2203-FM		24	26	24	23	22	23	23	23	23	25	23
46	2203-FM				26	34	21	30	28	31	26		
49	2203-FM						10	10					
56	2203-FM		30	30	30	30	30	30	30	30	30	40	30
59	2203-FM												
62	2203-FM		35	35	35	35	35	35	35	35	35	35	35
63	2203-FM				30	30	30	30	30	30	40	40	40
67	2203-FM						44	34					
68	2203-FM		40	40	40	40	40	40	40	40	40	40	40
72	2203-FM		20	20	20	20	20	20	20	20	20	20	20
75	2203-FM												
76	2203-FM		50	50	50	50	50	50	50	50	50	50	50
80	2203-FM		11	30	20	32	34	22	25	31	41	22	6
83	2203-FM		40	40	30	25	25	25	25	25	25	40	40
84	2203-FM		30	30	30	30	30	30	30	30	30	30	30
85	2203-FM		20	30	25	20	30	20	20	25	20	30	25
92	2203-FM		38	40	32	36	35	36	38	44	40	59	43
93	2203-FM					44	44	44	44	44	44	44	44
94	2203-FM												
106	2203-FM		30	30	30	30	30	30	30	30	30	30	30
110	2203-FM												

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

Fish Meal (2203-FM)

Methods Perfluoroalkylcarboxylic acids (PFCAs) - Limit of detection (LOQ) in µg/kg product (12% moisture content)

LC	Sample		Perfluorobutanoic acid **(PFBA)**	Perfluoropentanoic acid **(PFPeA)**	Perfluorohexanoic acid **(PFHxA)**	Perfluoroheptanoic acid **(PFHpA)**	Perfluorooctanoic acid **(PFOA)**	Perfluorononanoic acid **(PFNA)**	Perfluorodecanoic acid **(PFDA)**	Perfluoroun-decanoic acid **(PFUnDA)**	Perfluorodo-decanoic acid **(PFDoDA)**	Perfluorotri- decanoic acid **(PFTrDA)**	Perfluorotetra-decanoic acid **(PFTeDA)**
1	2203-FM		0	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03
3	2203-FM		0	0.05	0.03	0.03	0.03	0.04	0.02	0.03	0.02		
4	2203-FM			0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
5	2203-FM		0	0.05	0.05	0.01	0.01	0.01	0.01	0.01	0.05	0.05	0.05
7	2203-FM		0	0.08	0.16								
8	2203-FM		0	0.01	0.01	0.03	0.03	0.07	0.07	0.06		0.13	0.14
16	2203-FM			0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.29	
20	2203-FM		0.30	0.10	0.10	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
23	2203-FM		2.00		1.00	0.50	0.70	0.50	0.20	1.00	0.50	0.50	0.20
25	2203-FM						0.10	0.10					
26	2203-FM		0.07	0.07	0.07	0.07	0.07	0.07	0.07	0.07	0.07	0.07	0.07
28	2203-FM		0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.09
29	2203-FM		0.44	0.88	0.18								0.06
43	2203-FM		0.23	0.07	0.07	0.07	0.07	0.07	0.07	0.07	0.07	0.07	0.07
46	2203-FM				0.03	0.01	0.01	0.01	0.01	0.01	0.01		
49	2203-FM						0.10	0.10					
56	2203-FM		5.00	2.00	0.50	0.50	0.30	0.50	0.50	0.50	0.50	1.00	1.00
59	2203-FM					0.30	0.10	0.10	0.10	0.25	0.25		0.25
62	2203-FM			0.75	0.05	0.05	0.05	0.05	0.05	0.05	0.05		
63	2203-FM				0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10
67	2203-FM						0.10	0.10					
68	2203-FM		0.30	0.30	0.30	0.30	0.20	0.30	0.30	0.30	0.30	0.30	0.30
72	2203-FM		0.02	0.02	0.02	0.02	0.01	0.01	0.02	0.02	0.02	0.02	0.02
75	2203-FM		0.10	0.10	0.10	0.02	0.10	0.01	0.01	0.01	0.01	0.01	0.01
76	2203-FM		0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20
80	2203-FM		0.50	0.50	0.10	0.05	0.05	0.10	0.10	0.05	0.10	0.50	0.50
83	2203-FM		0.20	0.20	0.20								0.20
84	2203-FM		1.25	0.50	0.03	0.03	0.05	0.03	0.03	0.03	0.03	0.03	0.03
85	2203-FM		0.05	0.01	0.01	0.01	0.05	0.01	0.01	0.05	0.05	0.10	0.05
92	2203-FM		0.10	0.03	0.03								
93	2203-FM		0.05	0.03	0.06	0.02	0.03	0.03	0.02	0.10	0.01	0.05	0.03
94	2203-FM		0.20	0.20		0.20					0.20	0.20	0.20
106	2203-FM		0.10	0.50	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10
110	2203-FM												

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

Fish Meal (2203-FM)
Methods Perfluoroalkylsulfonic acids (PFSAs) - Measurement Uncertainty [%]

LC	Sample		Perfluorobutane-sulfonic acid (PFBS)	Perfluoropentane-sulfonic acid (PFPeS)	Perfluorohexane-sulfonic acid (PFHxS)	Perfluoroheptane-sulfonic acid (PFHpS)	Linear Perfluorooctane-sulfonic acid (L-PFOS)	Sum of branched Perfluorooctane-sulfonic acids (br-PFOS)	Total-Perfluorooctane-sulfonic acids (total PFOS)	Perfluorononane-sulfonic acid (PFNS)	Perfluorodecane-sulfonic acid (PFDS)	Perfluoroundecane-sulfonic acid (PFUnDS)	Perfluorododecane-sulfonic acid (PFDoDS)	Perfluorotridecane-sulfonic acid (PFTrDS)
1	2203-FM				45	45	45	45	45					
3	2203-FM		30		30	30	30	100		30	30			
4	2203-FM		32	26	29	28	32			31	31		28	
5	2203-FM													
7	2203-FM				7	7	12	12						
8	2203-FM		20	22	24	20			27		31			
16	2203-FM		30	30	30	30	30	30	30	30	30	30	30	
20	2203-FM		37	37	37	37	37	37	37	37	37	37	37	37
23	2203-FM		80		40		20				60			
25	2203-FM													
26	2203-FM						15		15					
28	2203-FM													
29	2203-FM													
43	2203-FM		22	22	23	23			22	23	24		25	
46	2203-FM		29	32	22	27	30	30	43					
49	2203-FM				10		10							
56	2203-FM		30	30	30	40	30	30	30	40	40	40	40	
59	2203-FM													
62	2203-FM		35		35	35	35	35	35					
63	2203-FM				30	30	30	30	30		40			
67	2203-FM													
68	2203-FM		40	40	40	40	40	40	40	40	40	40	40	40
72	2203-FM		20	20	20	20	20	20	20		20			
75	2203-FM													
76	2203-FM		50	50	50	50	50	50	50	50	50	50	50	50
80	2203-FM		26	7	34	6	26	26	26	8	33			
83	2203-FM		25	40	25	40	25	40	25	40	40			
84	2203-FM		30	30	30	30	30			30	30		30	
85	2203-FM		35	20	30	25	30	30		30	20		20	
92	2203-FM		40	34	53	43	43	55	40	44	44		44	
93	2203-FM				44	44	44	44	44					
94	2203-FM													
106	2203-FM		30	30	30	30	30	30	30	30	30	30	30	30
110	2203-FM								39					

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2023 [EURL-PT-POP_2203-FM]

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

Fish Meal (2203-FM)

Methods Perfluoroalkylsulfonic acids (PFSAs) - Limit of detection (LOQ) in µg/kg product (12% moisture content)

LC	Sample		Perfluorobutane-sulfonic acid (PFBS)	Perfluoropentane-sulfonic acid (PFPeS)	Perfluorohexane-sulfonic acid (PFHxS)	Perfluoroheptane-sulfonic acid (PFHpS)	Linear Perfluorooctane-sulfonic acid (L-PFOS)	Sum of branched Perfluorooctane-sulfonic acids (br-PFOS)	Total-Perfluorooctane-sulfonic acids (total PFOS)	Perfluorononane-sulfonic acid (PFNS)	Perfluorodecane-sulfonic acid (PFDS)	Perfluoroundecane-sulfonic acid (PFUnDS)	Perfluorododecane-sulfonic acid (PFDoDS)	Perfluorotridecane-sulfonic acid (PFTrDS)
1	2203-FM		0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03
3	2203-FM		0.05	0.05	0.05	0.05	0.05	0.05		0.05	0.10			
4	2203-FM		0.05	0.05	0.05	0.05	0.05			0.05	0.05		0.05	
5	2203-FM		0.05	0.01	0.01	0.0098				0.05	0.05			
7	2203-FM		0.02	0.07						0.02	0.01	0.02	0.06	0.03
8	2203-FM		0.01	0.04	0.07	0.064			0.02		0.45			
16	2203-FM		0.05	0.05	0.05	0.049	0.05	0.05	0.05	0.05	0.10	0.10	0.49	
20	2203-FM		0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.10	0.10	0.10	0.10	0.10
23	2203-FM		2.00		1.00		1.00				0.20			
25	2203-FM				0.10		0.10							
26	2203-FM		0.07	0.07	0.07	0.07	0.07	0.07	0.14	0.07	0.07	0.07	0.07	0.07
28	2203-FM		0.04	0.04	0.04	0.0437	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04
29	2203-FM		0.16	0.83		0.08				0.04	0.04	0.02	0.02	0.04
43	2203-FM		0.23	0.07	0.07	0.0676			0.07	0.07	0.07		0.23	
46	2203-FM		0.01	0.02	0.01	0.01	0.01	0.01	0.02					
49	2203-FM				0.10		0.10							
56	2203-FM		0.50	0.50	0.50	0.5	0.30	0.50	0.50	1.00	1.00		1.00	
59	2203-FM		1.00		0.10	0.06	1.00	1.00	1.00		0.10			
62	2203-FM		5.50		0.50	0.75	0.03	0.10						
63	2203-FM				0.10	0.1	0.10	0.10	0.10		0.10			
67	2203-FM				0.10		0.10	0.10	0.10					
68	2203-FM		0.30	0.30	0.20	0.3	0.20	0.20	0.20	0.30	0.30	0.30	0.30	0.30
72	2203-FM		0.02	0.02	0.01	0.02	0.01	0.01	0.01		0.02			
75	2203-FM		0.02		0.07		0.03		0.06					
76	2203-FM		0.20	0.20	0.20	0.2	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20
80	2203-FM		0.05	0.10	0.05	0.1	0.05	0.05	0.05	0.10	0.10			
83	2203-FM		0.02	0.20						0.20	0.20			
84	2203-FM		0.03	0.03	0.03	0.025	0.03			0.03	0.03		0.03	
85	2203-FM		0.01	0.01	0.01	0.01	0.05	0.05		0.01	0.01		0.05	
92	2203-FM		0.03	0.03						0.03	0.03		0.03	
93	2203-FM		0.02	0.00	0.00	0.002	0.00	0.00	0.00	0.00	0.00		0.00	
94	2203-FM		0.20	0.20	0.20	0.2				0.20	0.20			
106	2203-FM		0.10	0.10	0.10	0.1	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10
110	2203-FM								5.00					

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

Fish Meal (2203-FM)

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

LC	Sample	Sum of L-PFOS, PFOA, PFNA, PFHxS (ub)	Sum of L-PFOS, PFOA, PFNA, PFHxS (lb)	Sum of total-PFOS, PFOA, PFNA, PFHxS (ub)	Sum of total-PFOS, PFOA, PFNA, PFHxS (lb)
1	2203-FM	37	37		
3	2203-FM				
4	2203-FM	25			
5	2203-FM				
7	2203-FM	34	34	42	42
8	2203-FM			48	48
16	2203-FM	60	60	60	60
20	2203-FM	37	37	37	37
23	2203-FM	67	67		
25	2203-FM	35	35		
26	2203-FM	15	15	15	15
28	2203-FM				
29	2203-FM				
43	2203-FM				
46	2203-FM	52	52	60	60
49	2203-FM	10	10		
56	2203-FM	30	30	30	30
59	2203-FM				
62	2203-FM	35	35	35	35
63	2203-FM				
67	2203-FM	34	34	32	32
68	2203-FM	40	40	40	40
72	2203-FM	40	40	40	40
75	2203-FM				
76	2203-FM	50	50	50	50
80	2203-FM	34	34	34	34
83	2203-FM	25	25	30	30
84	2203-FM	50	50		
85	2203-FM	24		24	
92	2203-FM	34	34	32	32
93	2203-FM	34	34	35	35
94	2203-FM				
106	2203-FM	30	30	30	30
110	2203-FM				

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Fish Meal 2023 [EURL-PT-POP_2203-FM]

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

Fish Meal (2203-FM)

Methods Perfluorooctanesulfonamide (FOSA) - Measurement Uncertainty MU [%] and Limit of detection (LOQ) in µg/kg product (12% moisture content)

LC	Sample	Perfluorooctane sulphonamide **(FOSA)**	
		MU [%]	**LOQ (µg/kg product)**
1	2203-FM	45	0.03
3	2203-FM		
4	2203-FM		
5	2203-FM		
7	2203-FM	5	
8	2203-FM	15	0.18
16	2203-FM		
20	2203-FM	37	0.01
23	2203-FM	80	2
25	2203-FM		
26	2203-FM		0.07
28	2203-FM		0.0437
29	2203-FM		
43	2203-FM	23	0.0676
46	2203-FM		
49	2203-FM		
56	2203-FM	30	0.5
59	2203-FM		
62	2203-FM		
63	2203-FM	50	0.1
67	2203-FM		
68	2203-FM	40	0.3
72	2203-FM		
75	2203-FM		
76	2203-FM		
80	2203-FM		
83	2203-FM		
84	2203-FM		
85	2203-FM	20	0.01
92	2203-FM	44	
93	2203-FM		
94	2203-FM		
106	2203-FM	30	0.1
110	2203-FM		