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

EURL-PT-POP_2302-CF

FEED

Report

PBDEs and HBCDDs

(Report Version 1.0)

17 June 2024

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Summary

Test sample	**FEED:** Compound feed [2302-CF]
Analytes of interest **Mandatory** for NRLs:	**PBDEs** (BDE-28, -47, -49, -99, -100, -153, -154, -183, -209) **HBCDDs** (α-HBCDD, β-HBCDD, γ-HBCDD or total HBCDD)
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	17 June 2024 (Version 1.0)
Publication	EURL POPs reserves all rights to publish and present the anonymised results of the interlaboratory study in scientific journals and/or during conferences.

1. Structure of the PT, test material and analytes

This proficiency test (PT) on the determination of **PCDD/Fs, PCBs, PBDEs, HBCDDs** and **PFASs** in **compound feed** was organized by European Union Reference Laboratory (EURL) for halogenated persistent organic pollutants (POPs) in Feed and Food to be performed between September and November 2023. The objective was to assess analytical performance of laboratories and interlaboratory comparability of results from analyses of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in one sample of **compound feed**.

National Reference Laboratories (NRLs) for halogenated POPs in Feed and Food from EU member states were requested to participate as part of their work programme for 2023. NRLs were invited to encourage the participation of Official Laboratories (OFLs) from their member states as part of their duties following Article 101 of regulation (EU) 2017/625 of the European Parliament and of the Council of 15 March 2017. Furthermore, participation of OFLs allowed the extension of the data basis for calculation of assigned values and evaluation of results. **Other official laboratories** and **commercial laboratories** performing the analysis of samples taken by feed business operators were invited to participate in this proficiency test.

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

1.1. Samples and coding

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

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

1.2. Analytes of interest

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

Polybrominated diphenyl ethers (PBDEs)

- Individual congeners: BDE-28, -47, -49, -99, -100, -153, -154, -183, -209
- Sum of 8 PBDEs (without BDE-209)
- Sum of 9 PBDEs (with BDE-209)

Hexabromocyclododecanes (HBCDDs)

- α-HBCDD, β-HBCDD, γ-HBCDD stereoisomers
- Sum of α-, β-, γ-HBCDD (using HPLC methods)
- Total HBCDD (using GC methods)

1.3. Methods

All kinds of detection and quantification methods could be applied.

1.4. Coding of laboratories and confidentiality

The identity of participating laboratories will be kept confidential and will not be revealed to other participants. For NRLs of EU member states, the suggested “protocol for management of underperformance in comparative testing or lack of collaboration of National Reference Laboratories (NRLs)” will be followed. The confidentiality of NRLs will be kept according to this protocol. For OFLs of EU member states cooperating with NRL, the respective NRLs will inform the EURL for halogenated POPs about the participating OFLs and will receive the respective laboratory codes, invoices for participation fee and certificates of participation of the OFLs.

1.5. Results of PBDEs and HBCDDs

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 %,** for PBDEs and HBCDDs.

2. Participating laboratories

This proficiency test was open for participation of:

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

117 laboratories registered for this proficiency test. For PBDE and HBCDD, 26 and 21 sets of results were reported, respectively, for at least one parameter.

Table 1: Participating laboratories

Participating laboratories	Region	No. of participants
National Reference Laboratories	European Union Europe Americas Oceania	27 3 1 1
Official Laboratories	European Union Other European Countries Africa Americas Asia Oceania	56 - - 3 - -
Commercial Laboratories	European Union Other European Countries Africa Americas Asia Oceania	19 1 - 5 - 1
	Total	**117**

2.1. Number of reported results

Table 2: Reported results for PBDEs and moisture content for compound feed (2302-CF)

Reported results (2302-CF)	All laboratories
BDE-28, 47, 99, 100, 153, 154	26
BDE-49, 183, 209	25 / 25 / 21
Sum of 8 PBDEs (without BDE-209) (ub/lb)	26 / 26
Sum of 9 PBDEs (with BDE-209) (ub/lb)	21 / 21

Reported results (2302-CF)	All laboratories
Moisture content	25

Table 3: Reported results for HBCDDs and moisture content for compound feed (2302-CF)

Reported results (2302-CF)	All laboratories
α-, β-, γ-HBCDD	20
Sum of α-, β-, γ-HBCDD (ub/lb)	20 / 20
Total HBCDD (using GC methods)	1
Moisture content	22

2.2. Accreditation

Table 4: Reported accreditation according to ISO/IEC 17025 by participants for PBDEs and HBCDDs

Compound feed	PBDEs	HBCDDs
Accreditation	21	7
No accreditation	5	15

2.3. Detection methods

The following detection methods were applied:

- GC-HRMS-, GC-MS/MS, GC-NCI-MS-methods for PBDEs
- GC-HRMS-, LC-MS/MS-, LC-HRMS-methods for HBCDDs

Table 5: Overview of chromatographic separation and detection methods for the determination of PBDEs and HBCDDs for compound feed (2302-CF)

Detection methods	PBDEs	HBCDDs
GC-HRMS	18	2
GC-MS/MS	8	-
LC-MS/MS	-	18
LC-HRMS	-	2

3. Test for sufficient homogeneity and stability

The test for sufficient homogeneity was performed according to ISO 13528:2022 [1] and the International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [2]. Therefore, 10 portions of the test samples 2302-CF were analyzed in duplicate for PBDEs. The test for sufficient homogeneity was performed for individual congeners and sum parameters. The test materials showed sufficient homogeneity and stability for PBDEs for this proficiency test. Homogeneity can be concluded also for HBCDDs, due to similar physico-chemical properties. The stability check of the analytes of interest applying room temperature storage was performed according to ISO 13528:2022 [1]. The test material showed sufficient stability for this proficiency test.

4. Determination of the assigned value

Statistical evaluation of the PT results was performed by the EURL for halogenated POPs in feed and food according to 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 PBDE congeners, sum of 8 (without BDE-209) and sum of 9 (with BDE-209) PBDEs, for individual HBCDD diastereomers, sum of α-, β- and γ-HBCDD and total HBCDD (including limits of quantification (LOQs)), if possible. Additionally the median of all values was calculated.
For individual congeners (including LOQs) assigned values were only calculated according to the above mentioned procedure, if more than 2/3 of all results 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.
Due to high variation of participants' results in the range of the respective LOQ or too few results, no assigned values could be calculated for:

- (+/–)-β -HBCDD
- Total HBCDD (using GC-methods)

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 PBDE sum parameters. 21 of 26 participating laboratories were accredited according to ISO/IEC 17025 for PBDEs. No significant differences (<1 to 2 %)

between the assigned values calculated for both data sets for PBDEs were observed. For HBCDDs only 7 out of 20 participating laboratories were accredited according to ISO/IEC 17025. Therefore, no assigned values for HBCDDs could be calculated for this group of participants (Table 6).

Table 6: Comparison of assigned values for all participants and participants with reported accreditation according to ISO/IEC 17025 for PBDE and HBCDD sum parameters

Sum parameters	Assigned value All participants	Assigned value ISO/IEC 17025 accreditation	Deviation
	µg/kg product (12 % moisture content)		%
Sum of PBDE without BDE-209 (ub)	0.501	0.509	-1.6
Sum of PBDE without BDE-209 (lb)	0.504	0.509	< -1
Sum of PBDE including BDE-209 (ub)	0.892	0.895	<1
Sum of PBDE including BDE-209 (lb)	0.886	0.877	1
α-HBCDD	0.0421	-*	
γ-HBCDD	0.576	-*	
Sum of α-, β-, γ-HBCDD (ub)	0.661	-*	
Sum of α-, β-, γ-HBCDD (lb)	0.634	-*	

*only very few results from accredited laboratories to calculated an assigned value

4.1. PBDEs – individual congeners and sum parameter

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

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

Compound feed (2302-CF)	Assigned value µg/kg product (12 % moisture content)
BDE-28	0.00786
BDE-47	0.130
BDE-49	0.0153
BDE-99	0.112
BDE-100	0.0300
BDE-153	0.0361
BDE-154	0.0234
BDE-183	0.150

Compound feed (2302-CF)	Assigned value µg/kg product (12 % moisture content)
BDE-209	0.369
Sum of 8 PBDEs (without BDE-209) (ub)	0.501
Sum of 8 PBDEs (without BDE-209) (lb)	0.504
Sum of 9 PBDEs (with BDE-209) (ub)	0.892
Sum of 9 PBDEs (with BDE-209) (lb)	0.886

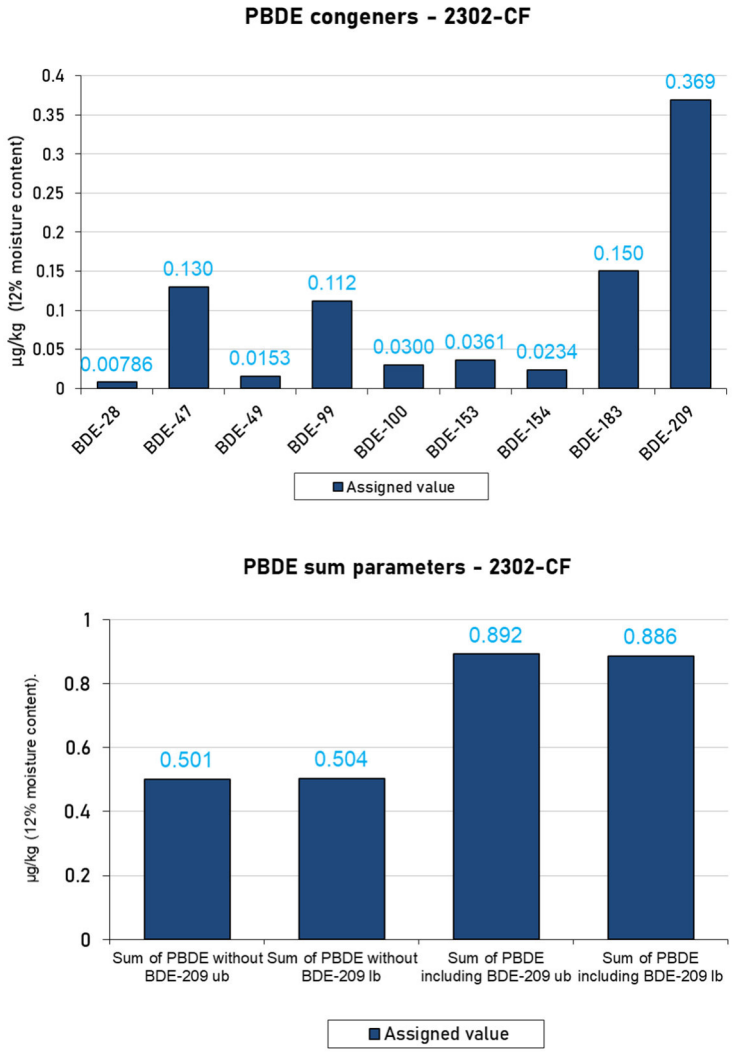

Figure 1: Assigned values (blue) for PBDE individual congeners and sum parameters for compound feed (2302-CF) [µg/kg product (12% moisture content)]

4.2. HBCDDs – individual stereoisomers and sum parameter

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

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

Compound feed (2302-CF)	Assigned value µg/kg product (12 % moisture content)
(+/–)-α-HBCDD	0.0421
(+/–)-γ -HBCDD	0.576
Sum of α-, β-, γ-HBCDD (ub)	0.661
Sum of α-, β-, γ-HBCDD (lb)	0.634

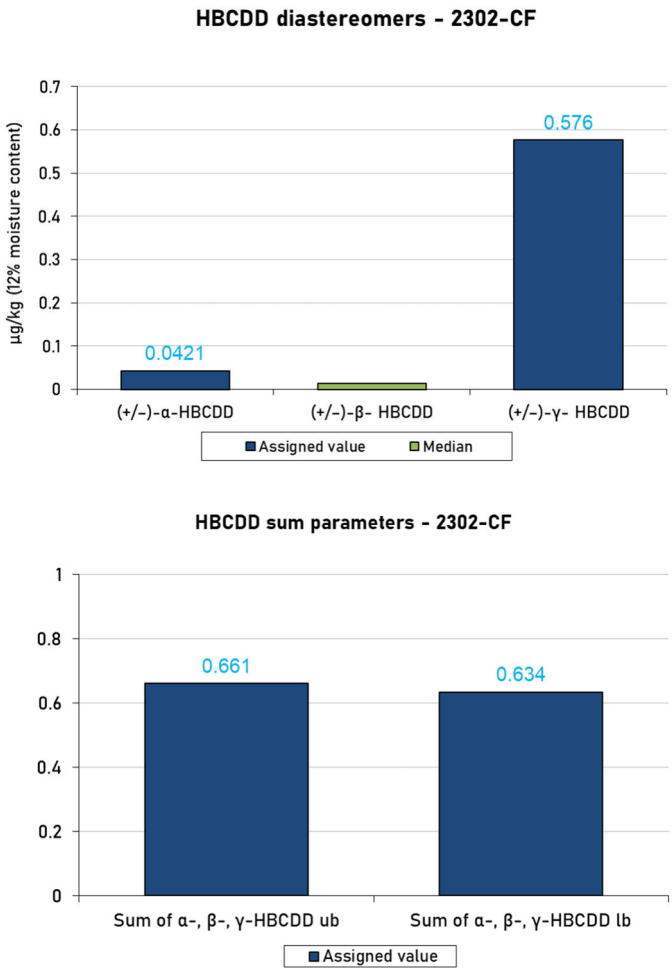

Figure 2: Assigned values (blue) and median values (green) for HBCDD individual congeners and sum parameters for compound feed (2302-CF) [µg/kg product (12% moisture content)]

4.3. Moisture content

For the moisture content an assigned value of 9.03 % for the test sample 2302-CF was calculated as a consensus of the participants' results, taking into account the calculation criteria described above.

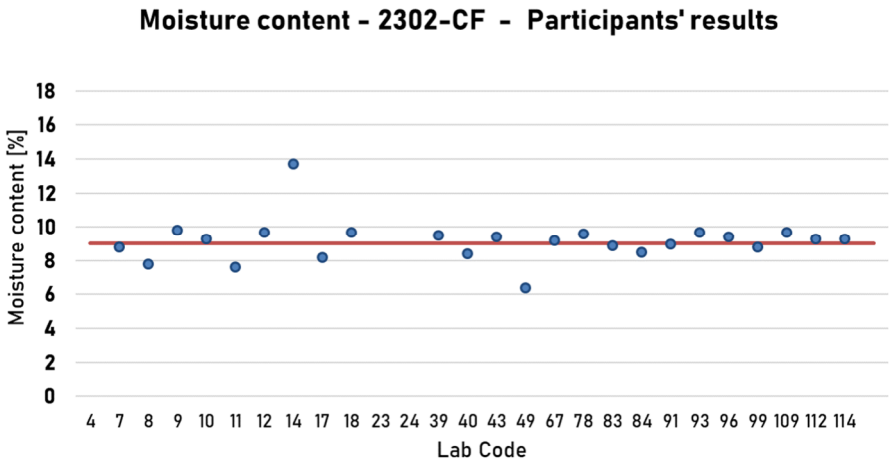

Figure 3: Distribution of participant's results of the moisture content in % for compound feed (2302-CF) – red line assigned value for the moisture content (9.03%)

4.4. Comparison of assigned values with recommended LOQs

The limits of quantification are currently based on the values specified in Commission Recommendation of 3 March 2014, on the monitoring of trace levels of brominated flame retardants in food (2014/118/EU). For PBDEs the recommended LOQ value is 0.01 µg/kg w.w. for individual congeners. However, it was discussed in the meetings of the core working group "Brominated Contaminants and PCNs" of the EURL/NRL network that for feed an LOQ value of 0.01 µg/kg product, compared to a feed with a moisture content of 12%, is preferable for all congeners and given that some feeds show concentrations below this an even lower targeted LOQ of 0.001 µg/kg product (12% moisture content), except for BDE-209 was recommended (Table 9). Valid data on the background contamination of feedstuffs with BFRs is particularly important for a reliable risk assessment. For HBCDDs the recommended LOQ value is at 0.01 µg/kg product, compared to a feed with a moisture content of 12% for α-, β- and γ-stereoisomers (Table 9). For total HBCDD measured by GC-MS, the corresponding LOQ value is 0.003 µg/kg product (12% moisture content), as cumulative response of all possible HBCDD diastereomers (Table 9).

Table 9: Analytical recommendations from "Guidance document on analytical parameters for the determination of organobromine contaminants in food and feed" (CWG "BCons and PCNs")

Undesirable Substances in Feed	Limit of quantification per congener/stereoisomer µg/kg product (12 % moisture content)
PBDEs	0.01 and 0.001 (all congeners except BDE-209)
HBCDDs	0.01 (sum of HBCDDs) and 0.003 (total HBCDD)

PBDEs:

The calculated assigned value for BDE-28 (0.00786 µg/kg product) was below the recommended LOQ of 0.01 µg/kg product. All other assigned values were in the range of the recommended LOQ (BDE-49, -100, -153, -154) or ten times higher (BDE-47, -99, -183), except for BDE-209. The assigned value for BDE-209 was almost 40 times higher than the recommended LOQ in this test sample compound feed (2302-CF).

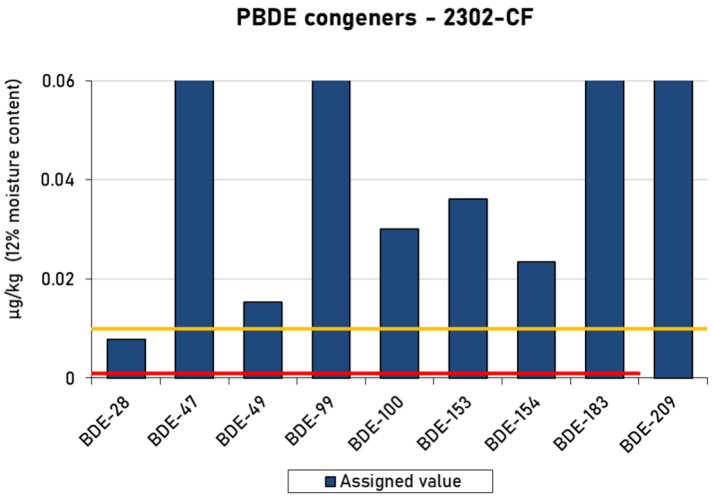

Figure 4: Comparison of assigned values for PBDE congeners with recommended LOQs (yellow line at 0.01 µg/kg product and red line at 0.001 µg/kg product) in compound feed (2302-CF)

HBCDDs:

For β-HBCDD no assigned value could be calculated, because less than 2/3 of all reported results were above the LOQs (see calculation criteria section 4). Therefore, the median value was taken for comparison with the recommended LOQ. The median value for β -HBCDD (0.0137 µg/kg) was in the range of the recommended LOQ of 0.01 µg/kg product.

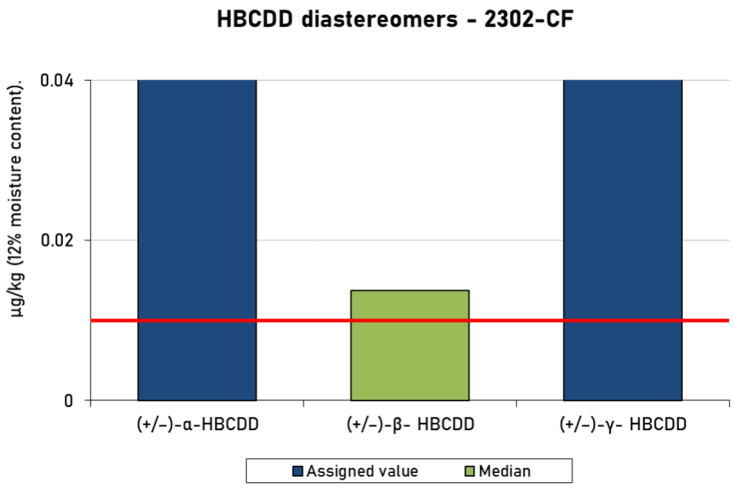

Figure 5: Comparison of assigned values for HBCDD stereoisomers with recommended LOQs (red line at 0.01 µg/kg product) in compound feed (2302-CF)

5. Evaluation of results

5.1. Z-scores calculation

Criteria for successful participation of laboratories were based on the evaluation of the results of individual congeners and sum parameters. For evaluation of results of physico-chemical methods 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 PBDE congeners, individual HBCDD diastereomers and PBDE and HBCDD sum parameters, the standard deviation for proficiency assessment σ_p is defined as 20 %.

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

Interpretation of z-scores:

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

5.2. PBDEs - Participants' z-scores

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

Percentage of participants' results	|z-score| ≤ 2	2 < |z-score| < 3	|z-score| ≥ 3
BDE-28	92%	4%	4%
BDE-47	96%	-	4%
BDE-49	88%	-	12%
BDE-99	96%	-	4%
BDE-100	96%	-	4%
BDE-153	92%	-	8%
BDE-154	96%	-	4%
BDE-183	96%	-	4%
BDE-209	84%	-	16%
Sum of 8 PBDEs without BDE-209 (ub)	96%	-	4%
Sum of 8 PBDE including BDE-209 (lb)	92%	-	8%
Sum of 9 PBDE including BDE-209 (ub)	86%	5%	9%
Sum of 9 PBDE including BDE-209 (lb)	95%	-	5%

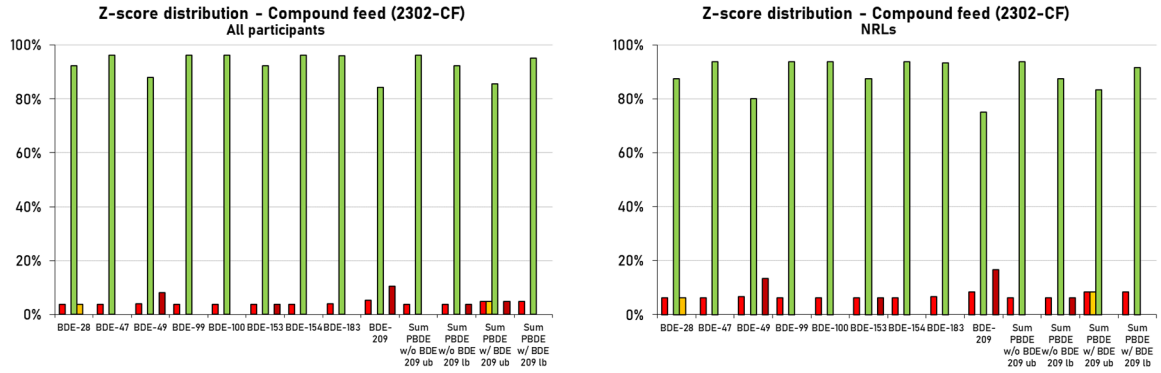

Figure 6: Distribution of all participants' z-scores and NRLs only for PBDE congeners / sum parameters for compound feed (2302-CF) [Green bars: -2 ≤ z-score ≤ 2, orange bars: -3 < z-score < -2, 2 < z-score < 3, red bars: z-score ≤ -3, z-score ≥ 3]

5.3. HBCDDs - Participants' z-scores

HBCDD stereoisomers undergo thermal isomerization at temperatures above 160 °C. With GC elution temperature of these compounds of normally above 160 °C a separation of HBCDD stereoisomers using GC analysis is not possible. Only one unresolved peak is obtained. Additional thermal decomposition of HBCDDs is reported for temperatures above 240 °C. Therefore, in case of applying GC-MS methods for HBCDD analysis determination of total HBCDD (as sum of all originally present HBCDD diastereomers) is possible only.

Due to the low numbers of results for total HBCDD the sum of α-, β-, γ-HBCDDs (lb) (using LC separation) was taken for comparison.

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

Percentage of participants' results	|z-score| ≤ 2	2 < |z-score| < 3	|z-score| ≥ 3
(+/–)-α-HBCDD	81%	-	19%
(+/–)-γ-HBCDD	85%	5%	10%
Sum of α-, β-, γ-HBCDD (ub)	85%	-	15%
Sum of α-, β-, γ-HBCDD (lb)	85%	5%	10%
Total HBCDD*	100%	-	-

*Comparison of participants' results for total HBCDD with assigned value for sum of α-, β-, γ-HBCDDs (lb)

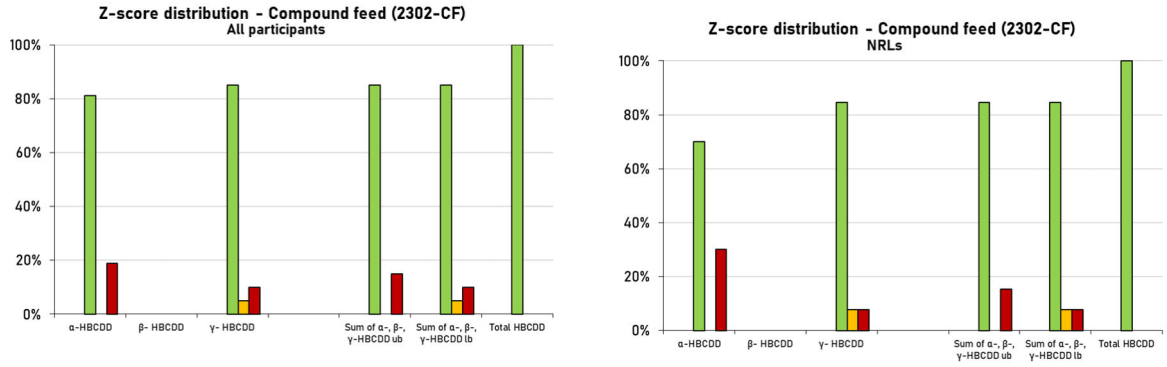

Figure 7: Distribution of all participants' z-scores and NRLs only for HBCDD stereoisomers / sum parameters for compound feed (2302-CF) [Green bars: -2 ≤ z-score ≤ 2, orange bars: -3 < z-score <-2, 2 < z-score < 3, red bars: z-score ≤ -3, z-score ≥ 3]

6. Participants' feedback

A questionnaire for feedback from participants of this EURL proficiency test was available as online survey between 22 November 2023 and 19 January 2024. The survey was anonymous, but participants could also give their laboratory name. The identity of the laboratories is kept confidential. The survey included several questions related to different topics (participants' information, organization of the proficiency test, PT test samples and evaluation of results and summary of data) and a possibility to include comments and further suggestions.

In total, 4 laboratories replied to this survey.

Table 12: Participating laboratories in the feedback survey

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

General aspect

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

Did the proficiency test meet expectations?

Specific aspects of this proficiency test

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

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

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

Choice of matrix 

Level of contamination 

Comments from participants:

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

7. Quality control

The Deutsche Akkreditierungsstelle GmbH attests that the provider of proficiency testing Chemisches und Veterinäruntersuchungsamt Freiburg, EU Reference Laboratory (EURL) for halogenated persistent organic pollutants (POPs) in feed and food is competent under the terms of DIN EN ISO/IEC 17043:2010 to carry out proficiency testing in the testing field of determination of halogenated persistent organic pollutants (POPs) in food and feed (Accreditation number: D-EP-18625-01-00).

8. Results of participants

An overview of the PBDE and HBCDD results for the PT test sample compound feed (2302-CF) are given in the following annexes. Laboratories are coded according to the laboratory codes sent after registration.

9. References

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

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

10. Annex

Compound feed – 2302-CF	
Annex-1	Assigned values – PBDEs, HBCDDs
Annex-2	Participants' results – Tables – PBDEs, HBCDDs
Annex-3	Participants' z-scores – Tables – PBDEs, HBCDDs
Annex-4	Participants' z-scores – Charts – PBDEs, HBCDDs
Annex-5	Homogeneity and stability test – PBDE
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

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

Annex 1: Assigned values of PBDEs and HBCDDs

Test sample - Compound Feed (2302-CF)

Assigned values of sum parameters and individual congeners

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

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

Compound Feed (2302-CF)
PBDE - 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]
BDE-28	2,2′,4-tribromodiphenyl ether		0.00786	0.0012	0.00032	24	0.00772
BDE-47	2,2′,4,4′-tetrabromodiphenyl ether		0.130	0.015	0.0037	25	0.130
BDE-49	2,2′,4,5′-tetrabromodiphenyl ether		0.0153	0.0032	0.00086	22	0.0150
BDE-99	2,2′,4,4′,5-pentabromodiphenyl ether		0.112	0.010	0.0026	25	0.110
BDE-100	2,2′,4,4′,6-pentabromodiphenyl ether		0.0300	0.0021	0.00053	25	0.0300
BDE-153	2,2′,4,4′,5,5′-hexabromodiphenyl ether		0.0361	0.0038	0.00098	24	0.0367
BDE-154	2,2′,4,4′,5,6′-hexabromodiphenyl ether		0.0234	0.0030	0.00075	25	0.0236
BDE-183	2,2′,3,4,4′,5′,6-heptabromodiphenyl ether		0.150	0.017	0.0043	24	0.151
BDE-209	2,2′,3,3′,4,4′,5,5′,6,6′-decabromodiphenyl ether		0.369	0.052	0.016	17	0.378
Sum of 8 PBDE	without BDE-209 (ub)		0.501	0.047	0.012	25	0.490
Sum of 8 PBDE	without BDE-209 (lb)		0.504	0.044	0.011	24	0.503
Sum of 9 PBDE	including BDE-209 (ub)		0.892	0.091	0.027	18	0.890
Sum of 9 PBDE	including BDE-209 (lb)		0.886	0.12	0.033	20	0.890

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

Compound Feed (2302-CF)
HBCDD - 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]
(+/–)-α-HBCDD	(1,2,5,6,9,10-hexabromo-(1R,2R,5S,6R,9R,10S)-rel-cyclododecane)		0.0421	0.0076	0.0026	13	0.0484
(+/–)-β- HBCDD	(1,2,5,6,9,10-hexabromo-(1R,2S,5R,6R,9R,10S)-rel-cyclododecane)						0.0137
(+/–)-γ- HBCDD	(1,2,5,6,9,10-hexabromo-(1R,2R,5R,6S,9S,10R)-rel-cyclododecane)		0.576	0.085	0.025	18	0.567
Sum of α-, β-, γ-HBCDD	(ub)		0.661	0.12	0.037	17	0.669
Sum of α-, β-, γ-HBCDD	(lb)		0.634	0.12	0.034	18	0.633
Total HBCDD	(using GC-methods)						0.606

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

Compound Feed (2302-CF)
Moisture content (BFR) - Assigned value

Analyte	Result %	**Assigned value** [outliers removed]	**Robust standard deviation** [outliers removed]	**Standard uncertainty** [outliers removed]	**No. of results contributing to assigned value**	**Median** [all values]
Moisture content		9.03	0.794	0.2	25	9.30

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

Annex 2: Participants' results of PBDEs and HBCDDs

Test sample - Compound Feed (2302-CF)

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

Compound Feed (2302-CF)
PBDE - Results

LC	Sample	Result µg/kg (12% moisture content)	2,2',4-tribromodiphenyl ether **BDE-28**	2,2',4,4'-tetrabromodiphenyl ether **BDE-47**	2,2',4,5'-tetrabromodiphenyl ether **BDE-49**	2,2',4,4',5-pentabromodiphenyl ether **BDE-99**	2,2',4,4',6-pentabromodiphenyl ether **BDE-100**	2,2',4,4',5,5'-hexabromodiphenyl ether **BDE-153**	2,2',4,4',5,6'-hexabromodiphenyl ether **BDE-154**	2,2',3,4,4',5',6-heptabromodiphenyl ether **BDE-183**	2,2',3,3',4,4',5,5',6,6'-decabromodiphenyl ether **BDE-209**	**Sum of 8 PBDE** without BDE-209 (ub)	**Sum of 8 PBDE** without BDE-209 (lb)	**Sum of 9 PBDE** including BDE-209 (ub)	**Sum of 9 PBDE** without BDE-209 (lb)
4	2302-CF														
7	2302-CF		0.007	0.105	0.013	0.092	0.027	0.022	0.015	0.133		0.414	0.414		
8	2302-CF														
9	2302-CF		0.0107	0.154	0.0207	0.13	0.0313	0.0419	0.0237	0.209	0.413	0.621	0.621	1.03	1.03
10	2302-CF		0.005	0.124	0.012	0.102	0.023	0.03	0.019	0.132	0.298	0.449	0.449	0.747	0.747
11	2302-CF		0.00788	0.136	0.0113	0.118	0.0306	0.036	0.025	0.161	0.409	0.526	0.526	0.935	0.935
12	2302-CF		0.00138	0.0214	0.00273	0.0166	0.00406	0.00539	0.00627	0.0219	0.0265	0.0797	0.0797	0.106	0.106
14	2302-CF		0.0084	0.143	0.018	0.124	0.035	0.037	0.026	0.171	0.375	0.562	0.562	0.937	0.937
17	2302-CF		0.00715	0.13	0.0146	0.111	0.0292	0.0394	0.0247	0.123	0.357	0.48	0.48	0.837	0.837
18	2302-CF		0.00721	0.123	0.0149	0.104	0.0303	0.0377	0.0222	0.151	0.747	0.49	0.49	1.24	1.24
23	2302-CF		0.012	0.147	0.027	0.133	0.038	0.043	0.031	0.124	0.33	0.56	0.56	0.89	0.89
24	2302-CF		0.00633	0.122	0.016	0.11	0.0284	0.0338	0.0239	0.136		0.477	0.477		
39	2302-CF		0.0087	0.136	0.018	0.125	0.033	0.037	0.026	0.171	< 1	0.555	0.555	1.56	0.555
40	2302-CF		0.01	0.146	0.015	0.113	0.0296	0.0303	0.0204	0.151	0.346	0.515	0.515	0.861	0.861
43	2302-CF		0.00815	0.13	0.0174	0.122	0.0303	0.0364	0.0234	0.163	< 0.295	0.53	0.53	0.826	0.53
49	2302-CF		0.00776	0.124	0.0136	0.111	0.0298	0.0322	0.0216	0.141	0.318	0.481	0.481	0.799	0.799
67	2302-CF		0.00535	0.112	0.0283	0.106	0.0284	0.0343	0.0166	0.141		0.471	0.471		
78	2302-CF		0.007	0.114	0.015	0.1	0.027	0.033	0.021	0.143	0.378	0.46	0.46	0.838	0.838
83	2302-CF		0.00753	0.134	0.0156	0.114	0.0294	0.0363	0.0225	0.166	0.406	0.525	0.525	0.931	0.931
84	2302-CF		0.00849	0.141	0.014	0.11	0.03	0.038	0.026	0.167	0.431	0.535	0.535	0.966	0.966
91	2302-CF		0.0092	0.13	0.018	0.12	0.031	0.041	0.026	0.16	0.39	0.54	0.54	0.93	0.93
93	2302-CF		0.011	0.136	0.0108	0.121	0.0331	0.0576	0.0286		0.644	0.398	1.04	0.398	1.04
96	2302-CF		0.0073	0.118	0.016	0.103	0.028	0.037	0.025	0.153		0.49	0.49		
99	2302-CF		0.00814	0.139	0.0193	0.11	0.0312	0.0371	0.0249	0.156	0.424	0.526	0.526	0.95	0.95
109	2302-CF		0.00917	0.151	0.0201	0.107	0.031	0.0331	0.0234	0.143	0.43	0.518	0.518	0.948	0.948
112	2302-CF		0.00712	0.107	0.0114	0.0962	0.0288	0.0372	0.021	0.136	0.34	0.444	0.444	0.784	0.784
114	2302-CF		0.00768	0.114	0.0123	0.108	0.0299	0.0401	0.0207	0.145	0.335	0.477	0.477	0.812	0.812
115	2302-CF		0.00731	0.125		0.109	0.0302	0.0352	0.0247	0.155		0.487	0.487		

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

Compound Feed (2302-CF)
HBCDD - Results

LC	Sample	Result µg/kg (12% moisture content)	(+/–)-α-HBCDD 1,2,5,6,9,10-hexabromo-(1R,2R,5S,6R,9R,10S)-rel-cyclododecane	(+/–)-β- HBCDD 1,2,5,6,9,10-hexabromo-(1R,2S,5R,6R,9R,10S)-rel-cyclododecane	(+/–)-γ- HBCDD 1,2,5,6,9,10-hexabromo-(1R,2R,5R,6S,9S,10R)-rel-cyclododecane		Sum of α-, β-, γ-HBCDD (ub)	Sum of α-, β-, γ-HBCDD (lb)	Total HBCDD (using GC-methods)
4	2302-CF		0.029	< 0.006	0.496		0.531	0.525	
7	2302-CF		0.257	0.054	0.548		0.859	0.859	
8	2302-CF		< 0.076	< 0.038	0.554		0.668	0.554	
9	2302-CF		< 0.27	< 0.15	0.674		1.09	0.674	
10	2302-CF		0.033	< 0.01	0.493		0.536	0.526	
11	2302-CF								0.606
12	2302-CF		0.0403	< 0.025	0.49		0.555	0.53	
14	2302-CF		0.073	0.014	0.842		0.929	0.929	
17	2302-CF								
18	2302-CF		0.049	0.008	0.612		0.67	0.67	
23	2302-CF								
24	2302-CF		0.0439	0.00955	0.555		0.608	0.608	
39	2302-CF								
40	2302-CF		0.0557	0.0134	1		1.07	1.07	
43	2302-CF		0.085	< 0.05	0.654		0.789	0.739	
49	2302-CF								
67	2302-CF								
78	2302-CF		0.05	< 0.01	0.75		0.81	0.8	
83	2302-CF		0.038	0.011	0.57		0.62	0.62	
84	2302-CF		< 0.1	< 0.1	0.52		0.72	0.52	
91	2302-CF		< 0.1	< 0.1	1.07		1.27	1.07	
93	2302-CF								
96	2302-CF								
99	2302-CF		0.04	0.01	0.65		0.7	0.7	
109	2302-CF		0.0477	< 0.01	0.458		0.516	0.506	
112	2302-CF		0.0372	< 0.03	0.602		0.646	0.646	
114	2302-CF		0.0385	< 0.03	0.564		0.609	0.609	
115	2302-CF		0.0455	0.00673	0.523		0.575	0.575	

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

Compound Feed (2302-CF)
Moisture content - Results

LC	Sample	Result %	Moisture content	Moisture content	Moisture content
			PBDE	HBCDD	Mean
4	2302-CF			0.0	0.0
7	2302-CF		8.8	8.8	8.8
8	2302-CF			7.8	7.8
9	2302-CF		9.8	9.8	9.8
10	2302-CF		9.3	9.3	9.3
11	2302-CF		7.6	7.6	7.6
12	2302-CF		9.7	9.7	9.7
14	2302-CF		13.7	13.7	13.7
17	2302-CF		8.2		8.2
18	2302-CF		9.7	9.7	9.7
23	2302-CF				
24	2302-CF		91.4	91.4	91.4
39	2302-CF		9.5		9.5
40	2302-CF		8.4	8.4	8.4
43	2302-CF		9.4	9.4	9.4
49	2302-CF		6.4		6.4
67	2302-CF		9.2		9.2
78	2302-CF		9.6	9.6	9.6
83	2302-CF		8.7	9.1	8.9
84	2302-CF		8.5	8.5	8.5
91	2302-CF		9.0	9.0	9.0
93	2302-CF		9.7	9.7	9.7
96	2302-CF		9.4		9.4
99	2302-CF		8.8	8.8	8.8
109	2302-CF		9.7	9.7	9.7
112	2302-CF		9.3	9.3	9.3
114	2302-CF		9.3	9.3	9.3
115	2302-CF		6.6	6.6	6.6

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

Annex 3: Participants' z-scores of PBDEs and HBCDDs - Tables

Test sample - Compound Feed (2302-CF)

Z-scores of sum parameters and individual results

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 PBDE congeners and HBCDD diastereomers and sum

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

Compound Feed (2302-CF)
PBDE - Z-scores

LC	Sample	Z-score [σ_p = 20 %]	2,2',4-tribromodiphenyl ether **BDE-28**	2,2',4,4'-tetrabromodiphenyl ether **BDE-47**	2,2',4,5'-tetrabromodiphenyl ether **BDE-49**	2,2',4,4',5-pentabromodiphenyl ether **BDE-99**	2,2',4,4',6-pentabromodiphenyl ether **BDE-100**	2,2',4,4',5,5'-hexabromodiphenyl ether **BDE-153**	2,2',4,4',5,6'-hexabromodiphenyl ether **BDE-154**	2,2',3,4,4',5',6-heptabromodiphenyl ether **BDE-183**	2,2',3,3',4,4',5,5',6,6'-decabromodiphenyl ether **BDE-209**		**Sum of 8 PBDE** without BDE-209 (ub)	**Sum of 8 PBDE** without BDE-209 (lb)	**Sum of 9 PBDE** including BDE-209 (ub)	**Sum of 9 PBDE** without BDE-209 (lb)
4	2302-CF															
7	2302-CF		-0.5	-1.0	-0.8	-0.9	-0.5	-2.0	-1.8	-0.6			-0.9	-0.9		
8	2302-CF															
9	2302-CF		1.8	0.9	1.8	0.8	0.2	0.8	0.1	2.0	0.6		1.2	1.2	0.8	0.8
10	2302-CF		-1.8	-0.2	-1.1	-0.4	-1.2	-0.8	-0.9	-0.6	-1.0		-0.5	-0.5	-0.8	-0.8
11	2302-CF		0.0	0.2	-1.3	0.3	0.1	0.0	0.3	0.4	0.5		0.2	0.2	0.2	0.3
12	2302-CF		-4.1	-4.2	-4.1	-4.3	-4.3	-4.3	-3.7	-4.3	-4.6		-4.2	-4.2	-4.4	-4.4
14	2302-CF		0.3	0.5	0.9	0.5	0.8	0.1	0.6	0.7	0.1		0.6	0.6	0.3	0.3
17	2302-CF		-0.5	0.0	-0.2	0.0	-0.1	0.5	0.3	-0.9	-0.2		-0.2	-0.2	-0.3	-0.3
18	2302-CF		-0.4	-0.3	-0.1	-0.4	0.1	0.2	-0.3	0.0	5.1		-0.1	-0.1	2.0	2.0
23	2302-CF		2.6	0.7	3.8	0.9	1.3	1.0	1.6	-0.9	-0.5		0.6	0.6	0.0	0.0
24	2302-CF		-1.0	-0.3	0.2	-0.1	-0.3	-0.3	0.1	-0.5			-0.2	-0.3		
39	2302-CF		0.5	0.2	0.9	0.6	0.5	0.1	0.6	0.7			0.5	0.5	3.7	-1.9
40	2302-CF		1.4	0.6	-0.1	0.0	-0.1	-0.8	-0.6	0.0	-0.3		0.1	0.1	-0.2	-0.1
43	2302-CF		0.2	0.0	0.7	0.4	0.1	0.0	0.0	0.4			0.3	0.3	-0.4	-2.0
49	2302-CF		-0.1	-0.2	-0.6	0.0	0.0	-0.5	-0.4	-0.3	-0.7		-0.2	-0.2	-0.5	-0.5
67	2302-CF		-1.6	-0.7	4.2	-0.3	-0.3	-0.2	-1.5	-0.3			-0.3	-0.3		
78	2302-CF		-0.5	-0.6	-0.1	-0.5	-0.5	-0.4	-0.5	-0.2	0.1		-0.4	-0.4	-0.3	-0.3
83	2302-CF		-0.2	0.2	0.1	0.1	-0.1	0.0	-0.2	0.5	0.5		0.2	0.2	0.2	0.3
84	2302-CF		0.4	0.4	-0.4	-0.1	0.0	0.3	0.6	0.6	0.8		0.3	0.3	0.4	0.5
91	2302-CF		0.9	0.0	0.9	0.4	0.2	0.7	0.6	0.3	0.3		0.4	0.4	0.2	0.2
93	2302-CF		2.0	0.2	-1.5	0.4	0.5	3.0	1.1		3.7		-1.0	5.3	-2.8	0.9
96	2302-CF		-0.4	-0.5	0.2	-0.4	-0.3	0.1	0.3	0.1			-0.1	-0.1		
99	2302-CF		0.2	0.3	1.3	-0.1	0.2	0.1	0.3	0.2	0.7		0.2	0.2	0.3	0.4
109	2302-CF		0.8	0.8	1.6	-0.2	0.2	-0.4	0.0	-0.2	0.8		0.2	0.1	0.3	0.3
112	2302-CF		-0.5	-0.9	-1.3	-0.7	-0.2	0.2	-0.5	-0.5	-0.4		-0.6	-0.6	-0.6	-0.6
114	2302-CF		-0.1	-0.6	-1.0	-0.2	0.0	0.6	-0.6	-0.2	-0.5		-0.2	-0.3	-0.4	-0.4
115	2302-CF		-0.3	-0.2		-0.1	0.0	-0.1	0.3	0.2			-0.1	-0.2		

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

Compound Feed (2302-CF)
HBCDD - Z-scores

LC	Sample	Z-score [σ_p = 20 %]	**(+/–)-α-HBCDD** 1,2,5,6,9,10-hexabromo-(1R,2R,5S,6R,9R,10S)-rel-cyclododecane	**(+/–)-β- HBCDD** 1,2,5,6,9,10-hexabromo-(1R,2S,5R,6R,9R,10S)-rel-cyclododecane	**(+/–)-γ- HBCDD** 1,2,5,6,9,10-hexabromo-(1R,2R,5R,6S,9S,10R)-rel-cyclododecane		**Sum of α-, β-, γ-HBCDD** (ub)	**Sum of α-, β-, γ-HBCDD** (lb)	**Total HBCDD*** (using GC-methods)
4	2302-CF		-1.6		-0.7		-1.0	-0.9	
7	2302-CF		25.5		-0.2		1.5	1.8	
8	2302-CF				-0.2		0.1	-0.6	
9	2302-CF				0.9		3.2	0.3	
10	2302-CF		-1.1		-0.7		-0.9	-0.9	
11	2302-CF								-0.2
12	2302-CF		-0.2		-0.7		-0.8	-0.8	
14	2302-CF		3.7		2.3		2.0	2.3	
17	2302-CF								
18	2302-CF		0.8		0.3		0.1	0.3	
23	2302-CF								
24	2302-CF		0.2		-0.2		-0.4	-0.2	
39	2302-CF								
40	2302-CF		1.6		3.7		3.1	3.4	
43	2302-CF		5.1		0.7		1.0	0.8	
49	2302-CF								
67	2302-CF								
78	2302-CF		0.9		1.5		1.1	1.3	
83	2302-CF		-0.5		-0.1		-0.3	-0.1	
84	2302-CF				-0.5		0.4	-0.9	
91	2302-CF				4.3		4.6	3.4	
93	2302-CF								
96	2302-CF								
99	2302-CF		-0.2		0.6		0.3	0.5	
109	2302-CF		0.7		-1.0		-1.1	-1.0	
112	2302-CF		-0.6		0.2		-0.1	0.1	
114	2302-CF		-0.4		-0.1		-0.4	-0.2	
115	2302-CF		0.4		-0.5		-0.7	-0.5	

* Z-scores for information only; calculation based on assigned value for sum of α-, β-, γ-HBCDD (lb)

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

Compound Feed (2302-CF)
Moisture content - Z-scores

LC	Sample	Z-score [σ_p = 10 %]	Moisture content	Moisture content	Moisture content
			PBDE	HBCDD	Mean
4	2302-CF			-10.0	-10.0
7	2302-CF		-0.3	-0.3	-0.3
8	2302-CF			-1.4	-1.4
9	2302-CF		0.9	0.9	0.9
10	2302-CF		0.3	0.3	0.3
11	2302-CF		-1.6	-1.6	-1.6
12	2302-CF		0.7	0.7	0.7
14	2302-CF		5.2	5.2	5.2
17	2302-CF		-0.9		-0.9
18	2302-CF		0.7	0.7	0.7
23	2302-CF				
24	2302-CF		91.2	91.2	91.2
39	2302-CF		0.5		0.5
40	2302-CF		-0.8	-0.8	-0.8
43	2302-CF		0.4	0.4	0.4
49	2302-CF		-2.9		-2.9
67	2302-CF		0.2		0.2
78	2302-CF		0.7	0.7	0.7
83	2302-CF		-0.3	0.1	-0.1
84	2302-CF		-0.6	-0.6	-0.6
91	2302-CF		0.0	-0.1	-0.1
93	2302-CF		0.7	0.7	0.7
96	2302-CF		0.4		0.4
99	2302-CF		-0.3	-0.3	-0.3
109	2302-CF		0.7	0.7	0.7
112	2302-CF		0.3	0.3	0.3
114	2302-CF		0.3	0.3	0.3
115	2302-CF		-2.7	-2.7	-2.7

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

Annex 4: Participants' z-scores of PBDEs and HBCDDs - Charts

Test sample - Compound Feed (2302-CF)

Z-scores of sum parameters and individual results

Calculation of z-score on basis of assigned value

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

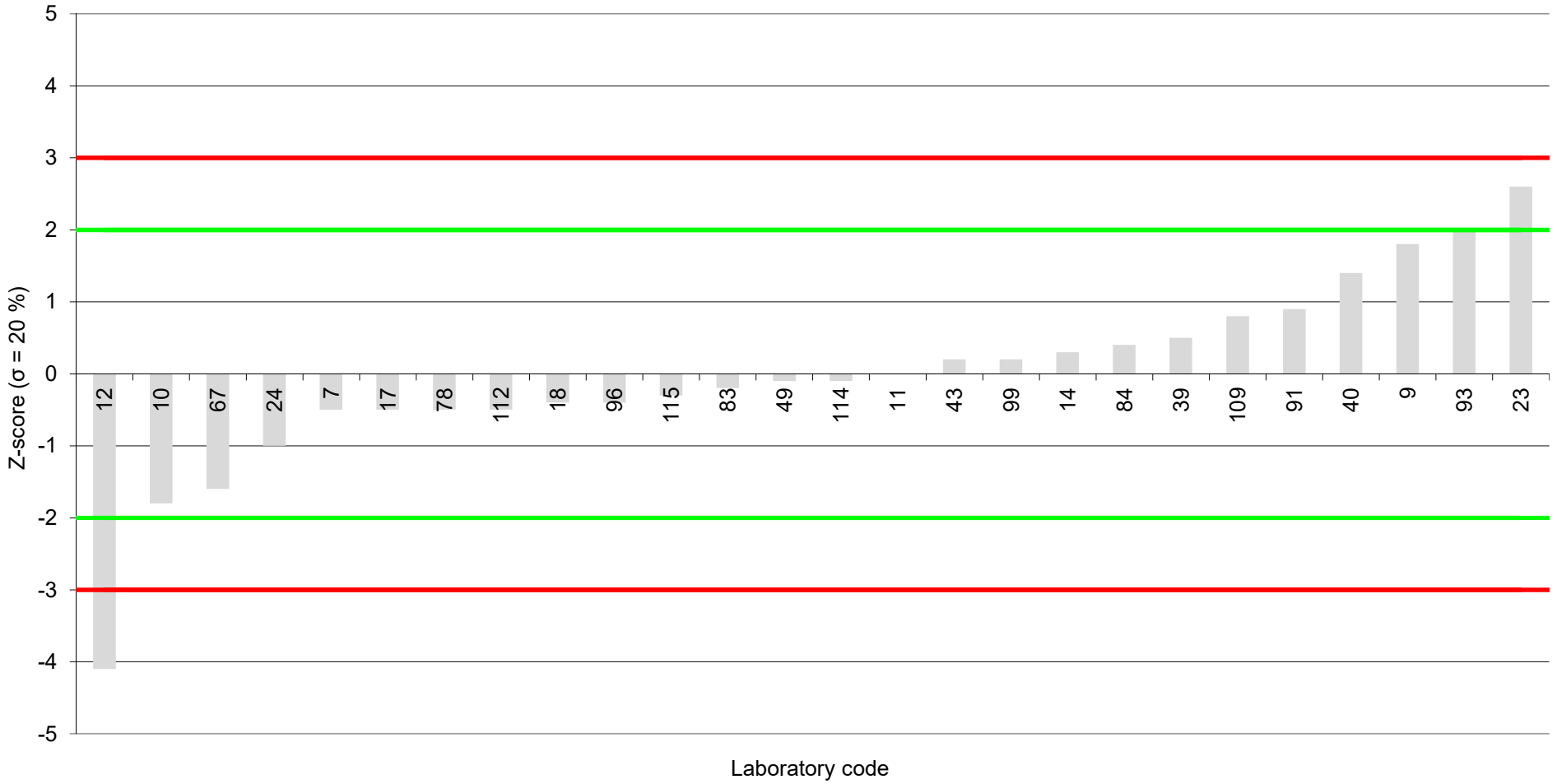
x_a: assigned value
x: participant's result
σ_p: fitness-for-purpose-based standard deviation for proficiency assessment
20%: Evaluated individual PBDE congeners and HBCDD diastereomers and sum

± 2 z-scores:
± 3 z-scores:

Compound Feed (2302-CF)

BDE-28

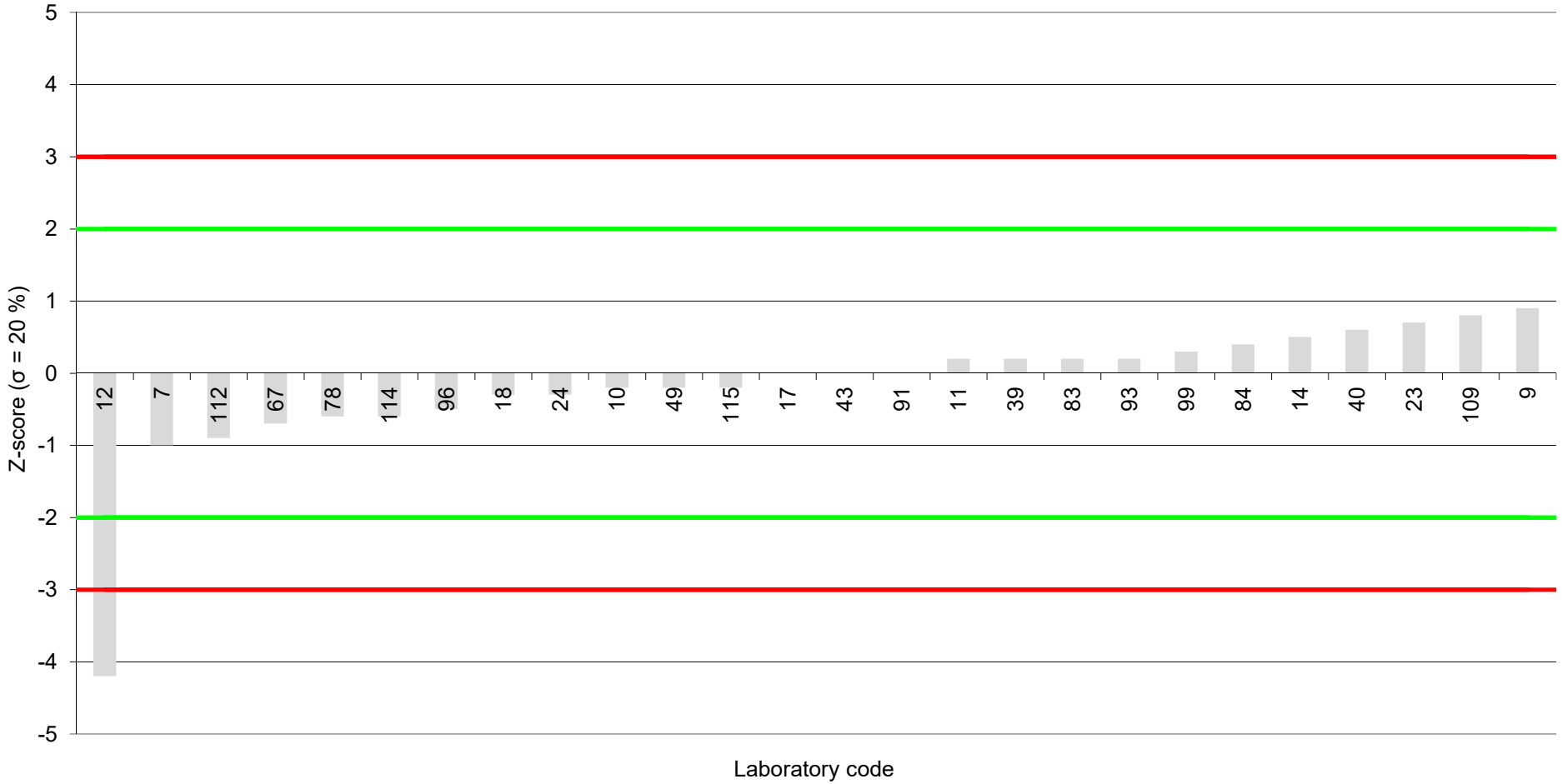
Assigned value: 0.00786 µg/kg product (12 % moisture content)



Compound Feed (2302-CF)

BDE-47

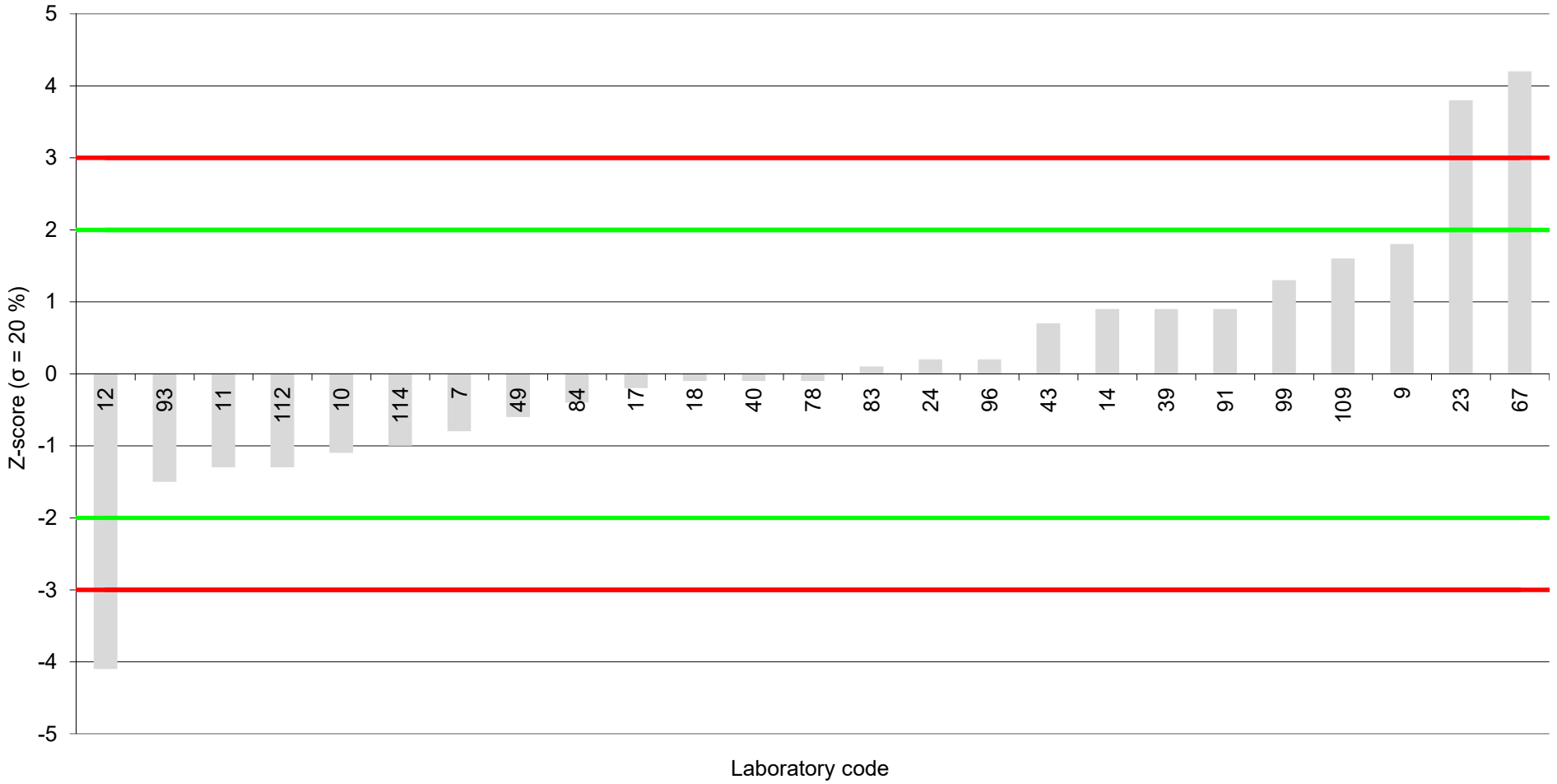
Assigned value: 0.13 µg/kg product (12 % moisture content)



Compound Feed (2302-CF)

BDE-49

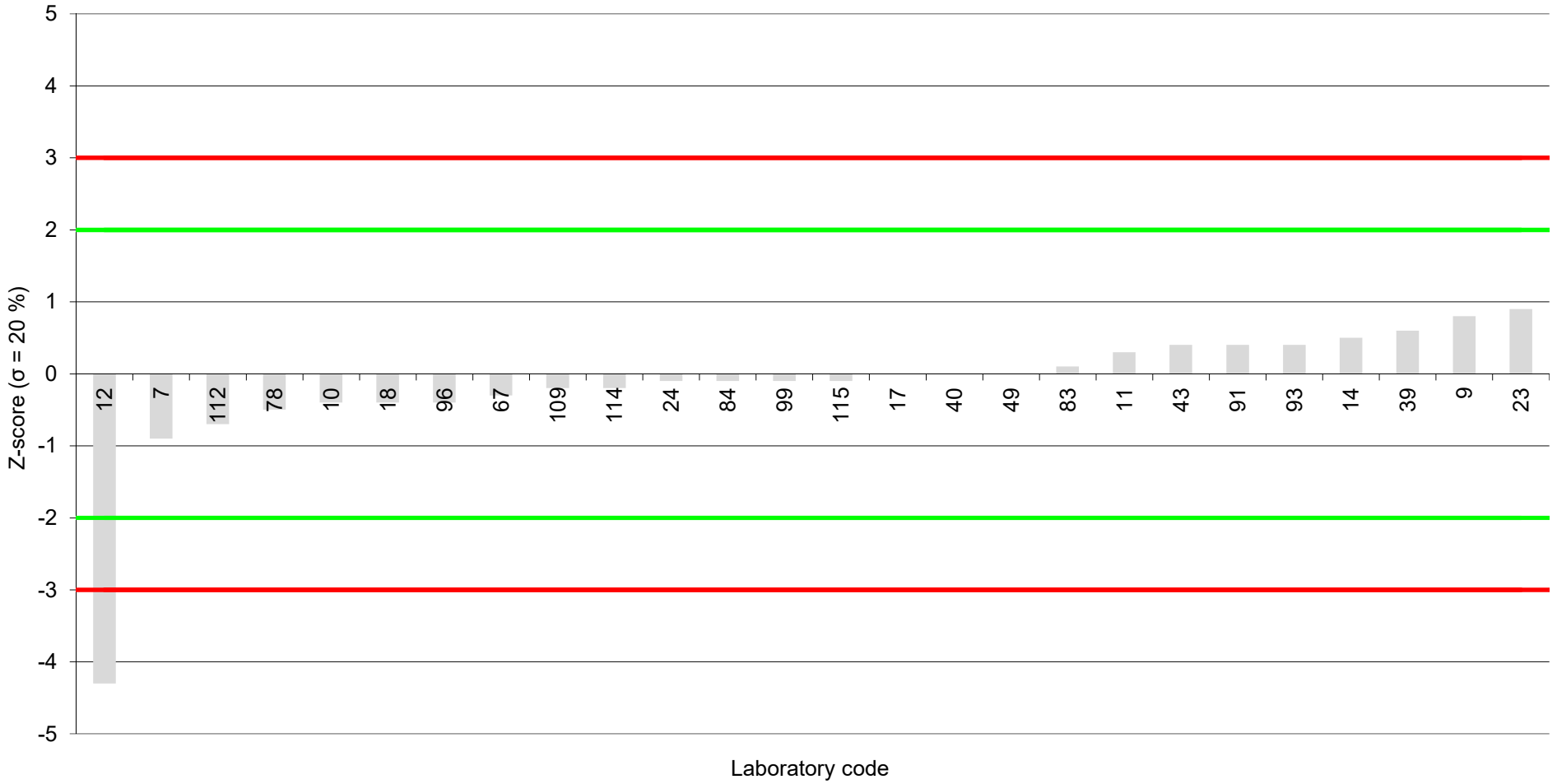
Assigned value: 0.0153 µg/kg product (12 % moisture content)



Compound Feed (2302-CF)

BDE-99

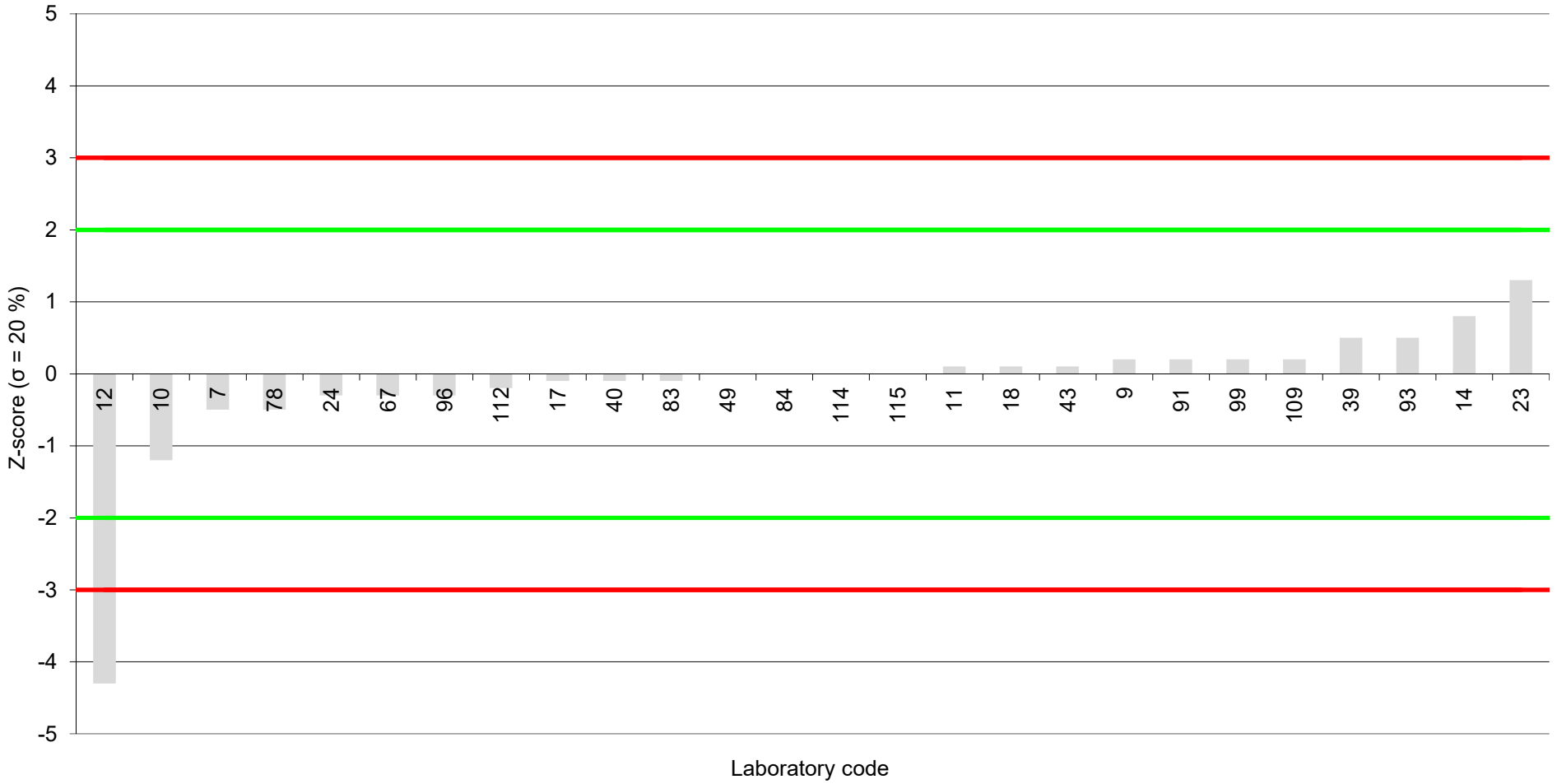
Assigned value: 0.112 µg/kg product (12 % moisture content)



Compound Feed (2302-CF)

BDE-100

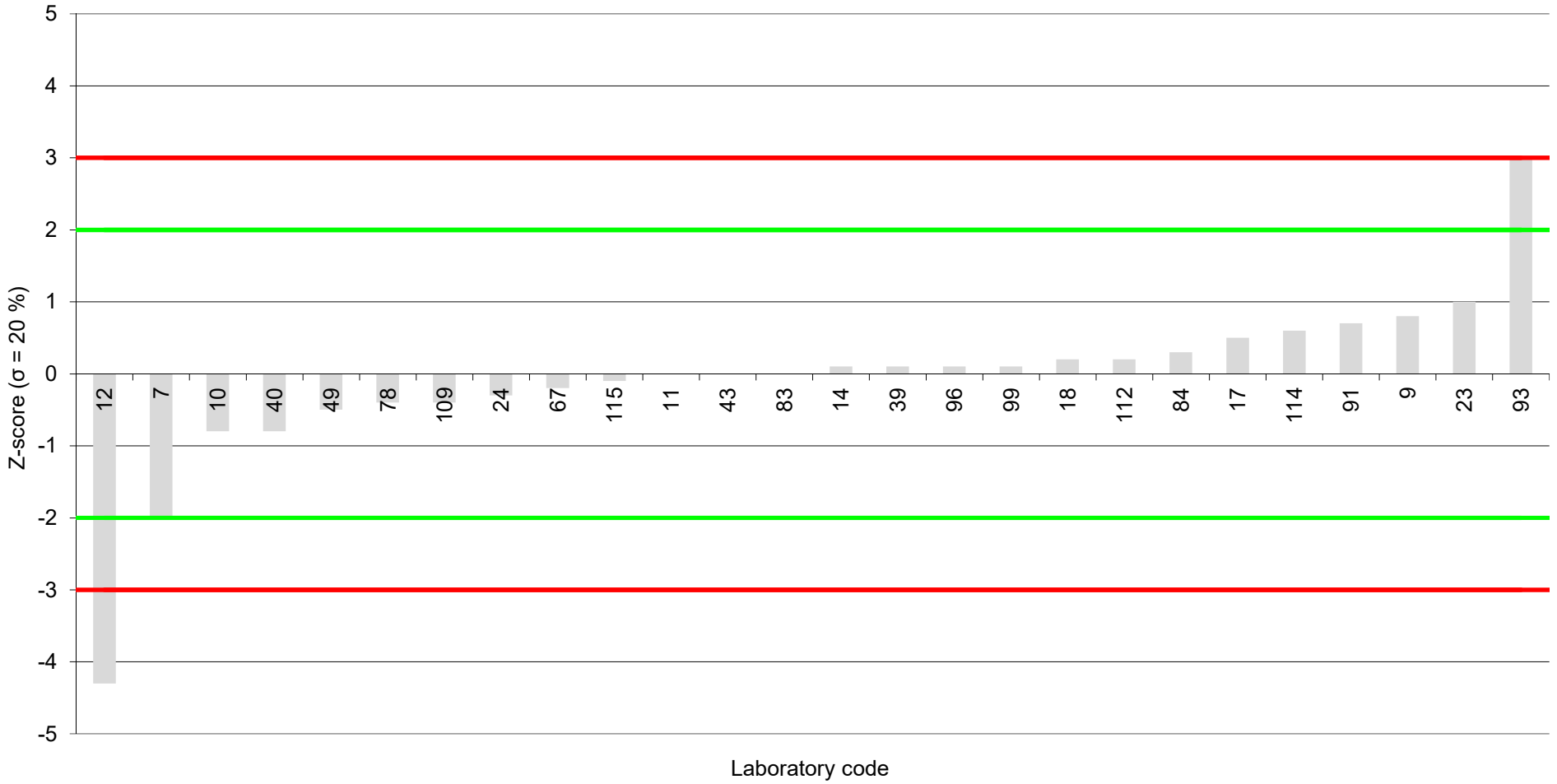
Assigned value: 0.03 µg/kg product (12 % moisture content)



Compound Feed (2302-CF)

BDE-153

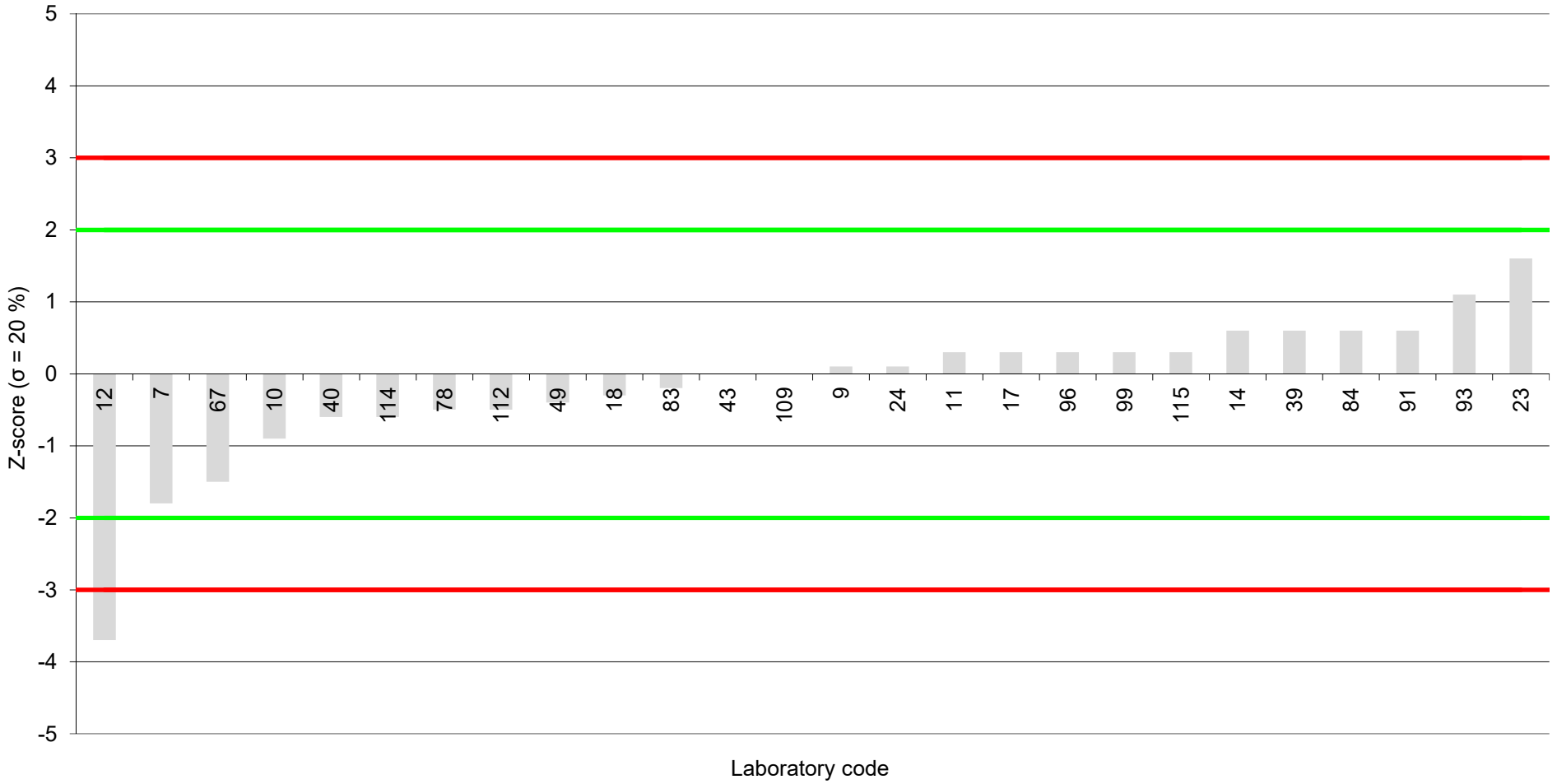
Assigned value: 0.0361 µg/kg product (12 % moisture content)



Compound Feed (2302-CF)

BDE-154

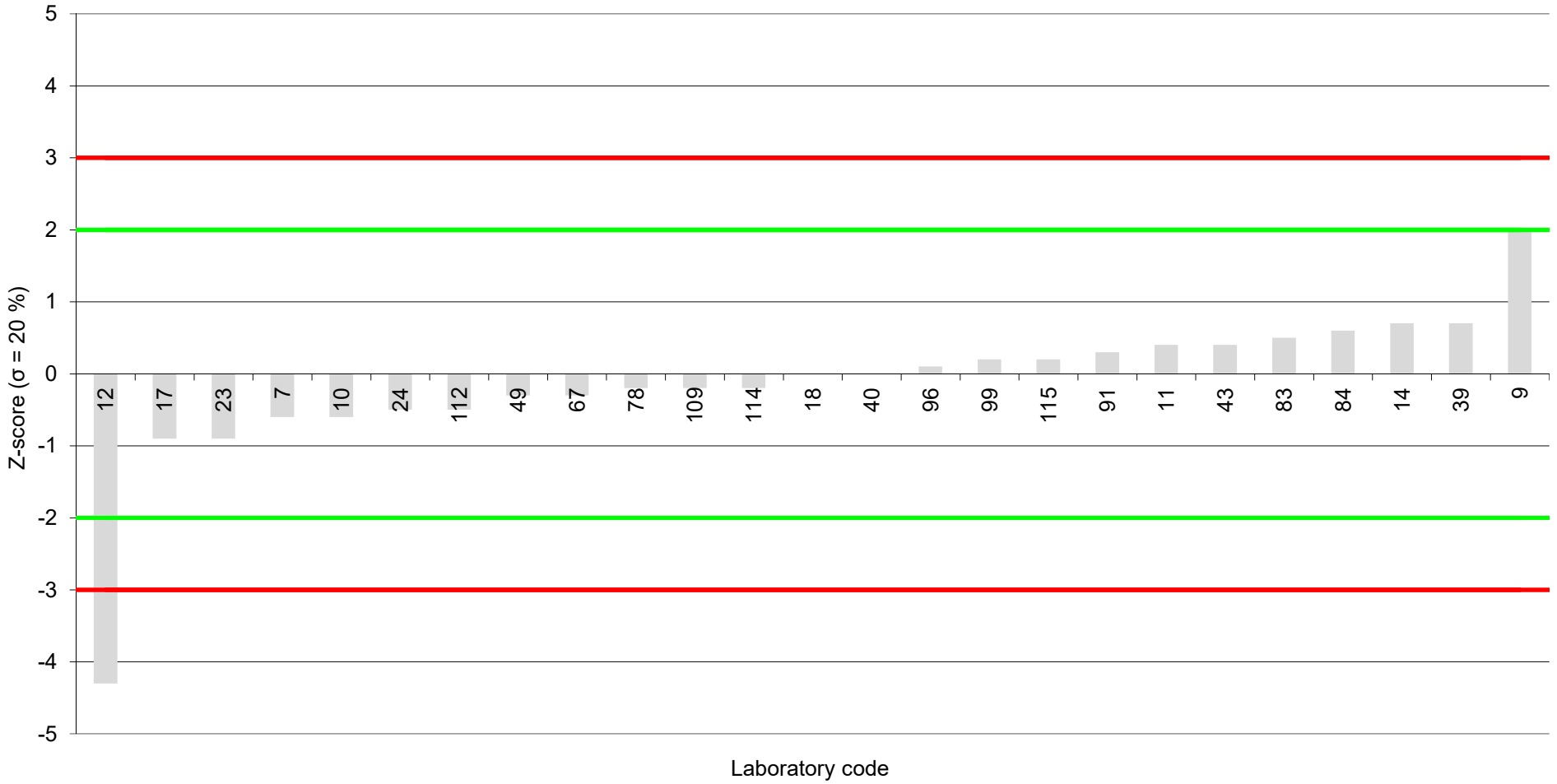
Assigned value: 0.0234 µg/kg product (12 % moisture content)



Compound Feed (2302-CF)

BDE-183

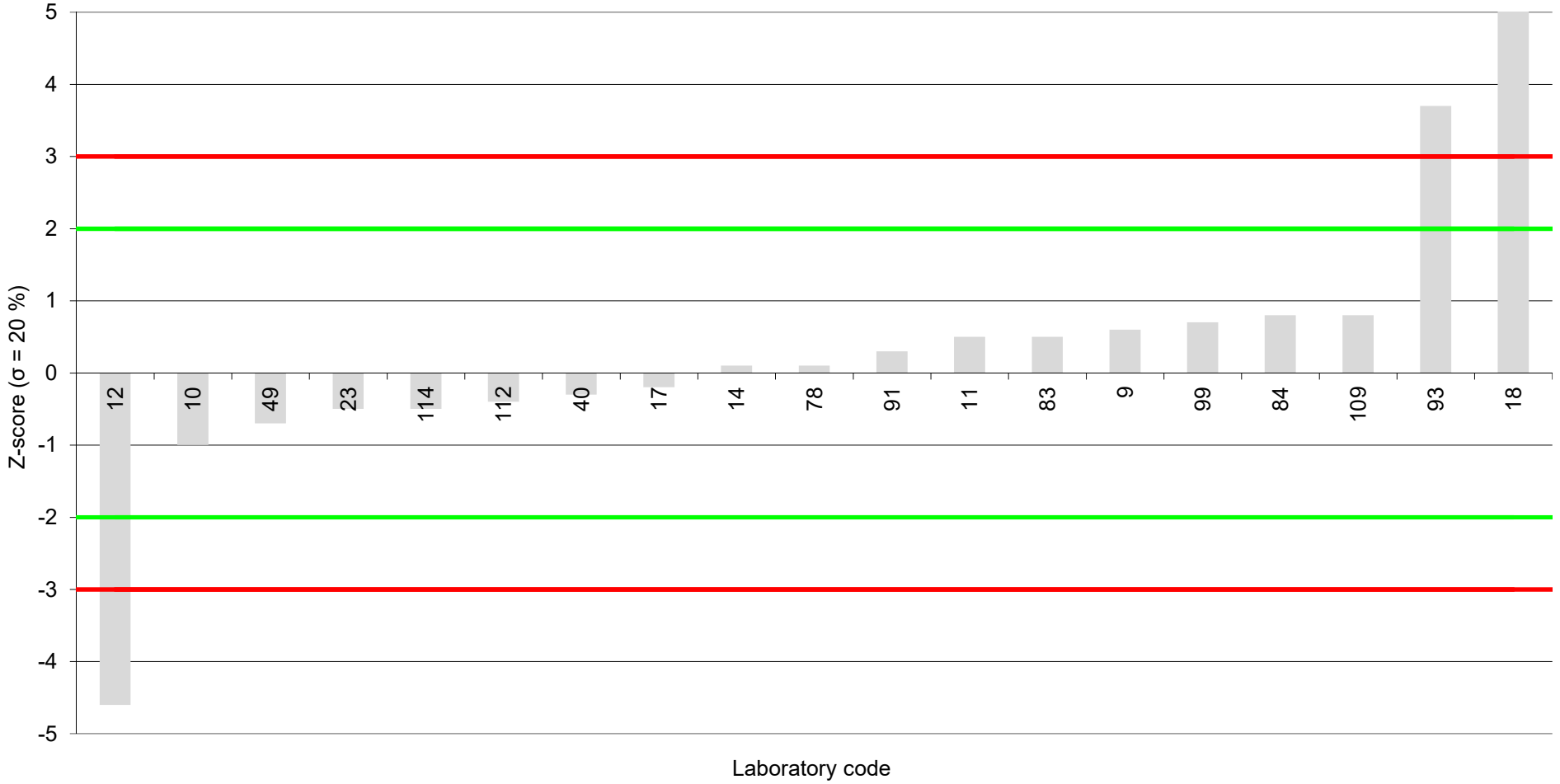
Assigned value: 0.15 µg/kg product (12 % moisture content)



Compound Feed (2302-CF)

BDE-209

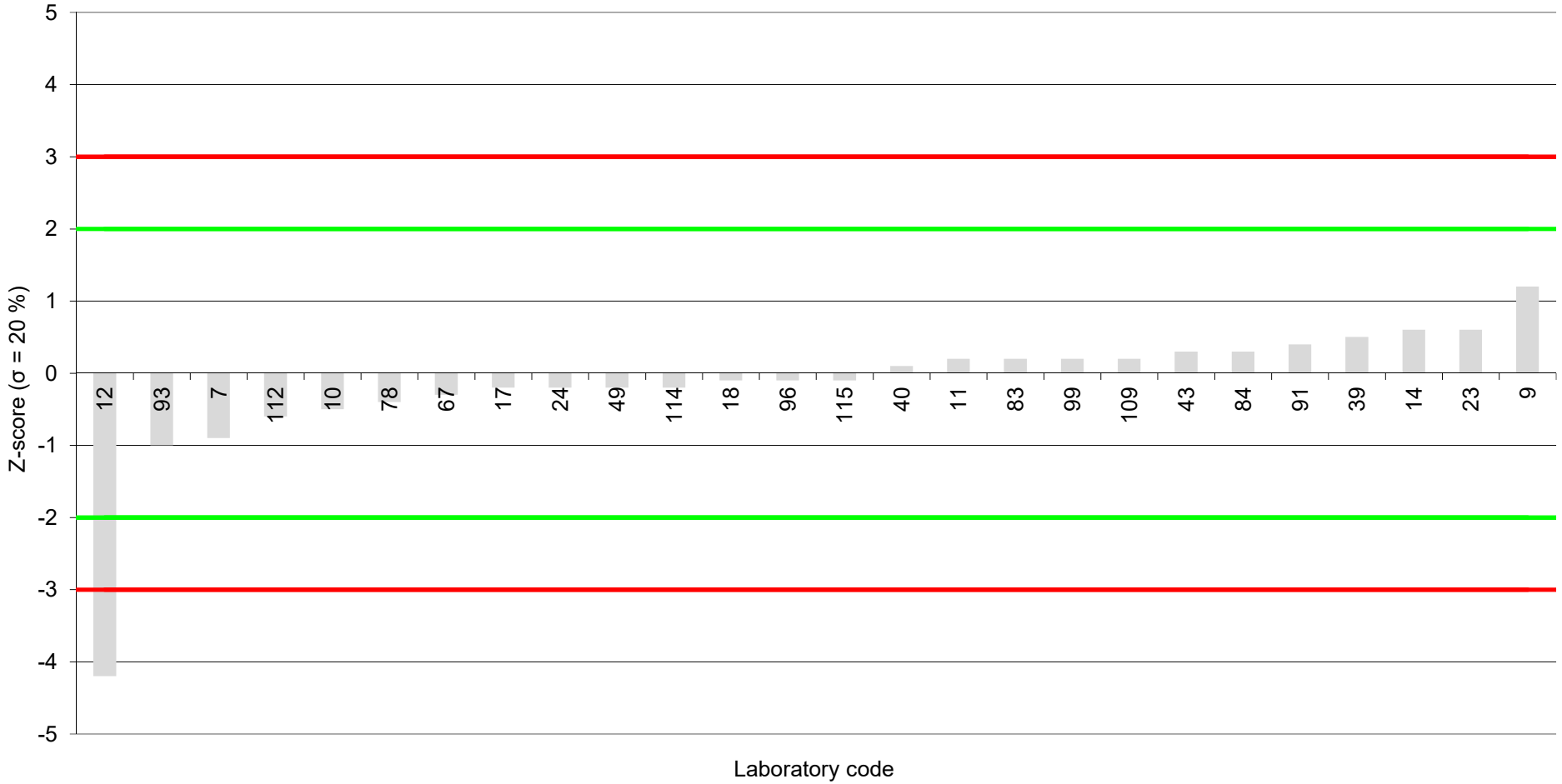
Assigned value: 0.369 µg/kg product (12 % moisture content)



Compound Feed (2302-CF)

Sum of PBDE without BDE-209 ub

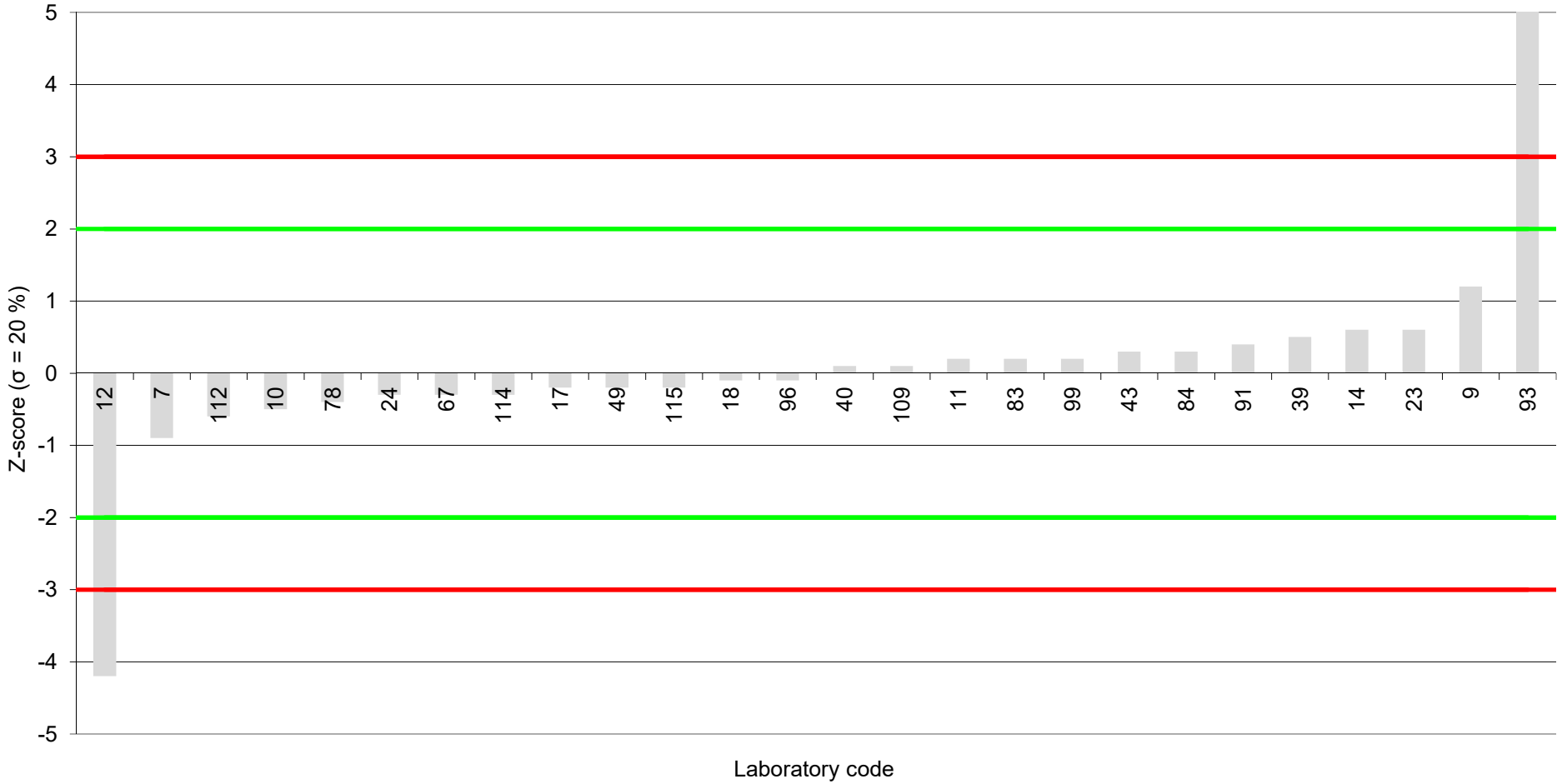
Assigned value: 0.501 µg/kg product (12 % moisture content)



Compound Feed (2302-CF)

Sum of PBDE without BDE-209 lb

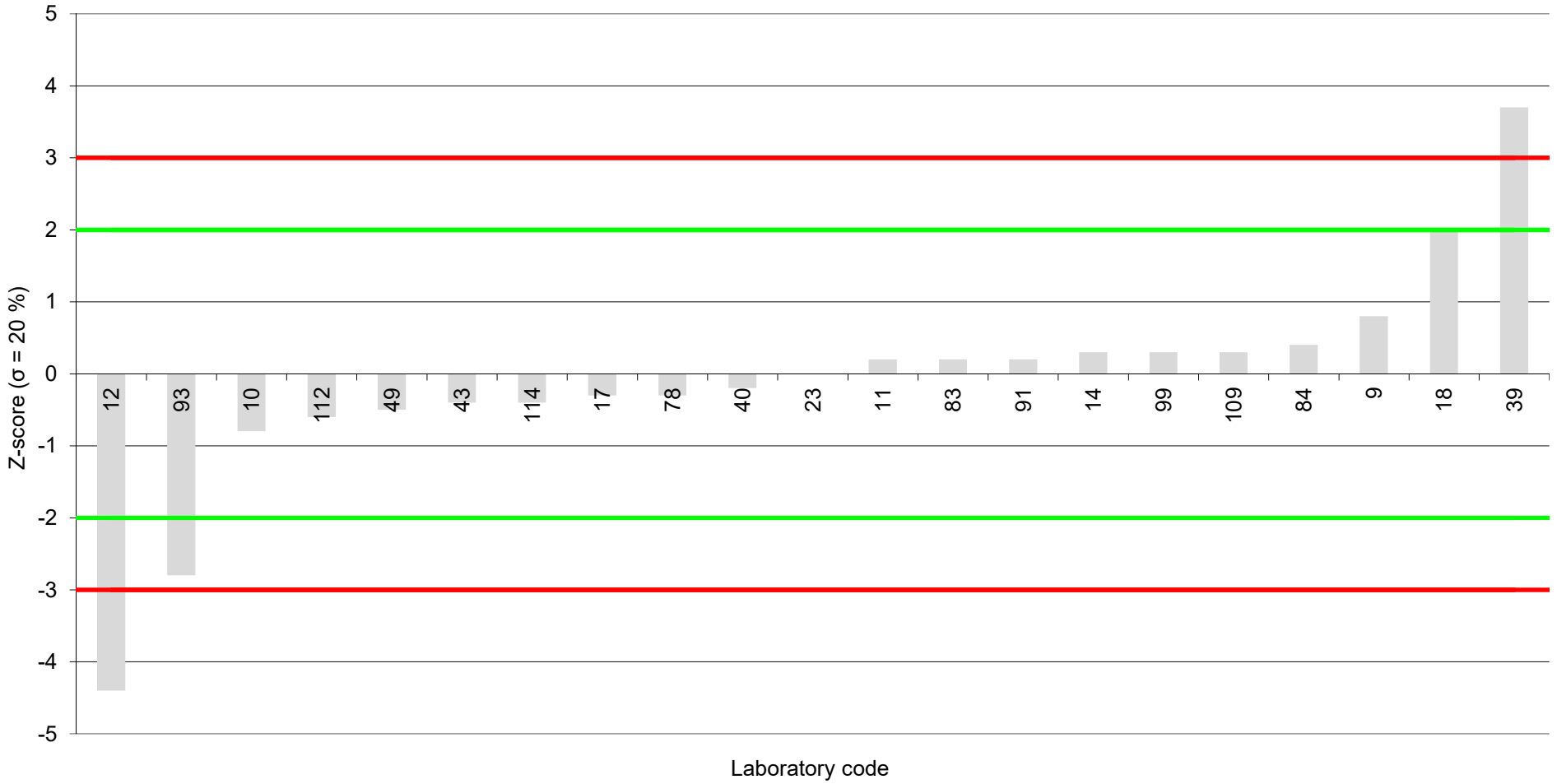
Assigned value: 0.504 µg/kg product (12 % moisture content)



Compound Feed (2302-CF)

Sum of PBDE including BDE-209 ub

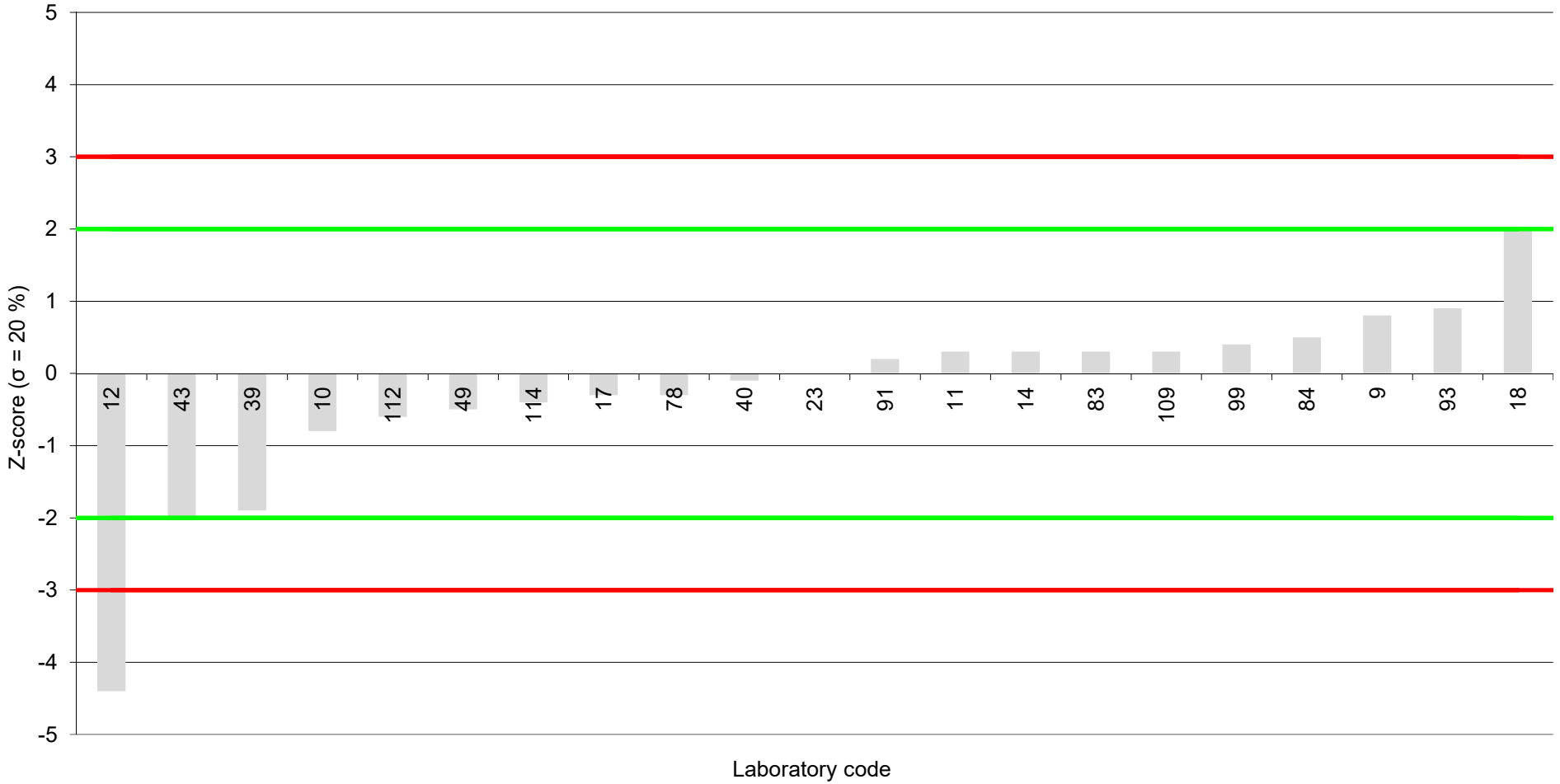
Assigned value: 0.892 µg/kg product (12 % moisture content)



Compound Feed (2302-CF)

Sum of PBDE including BDE-209 lb

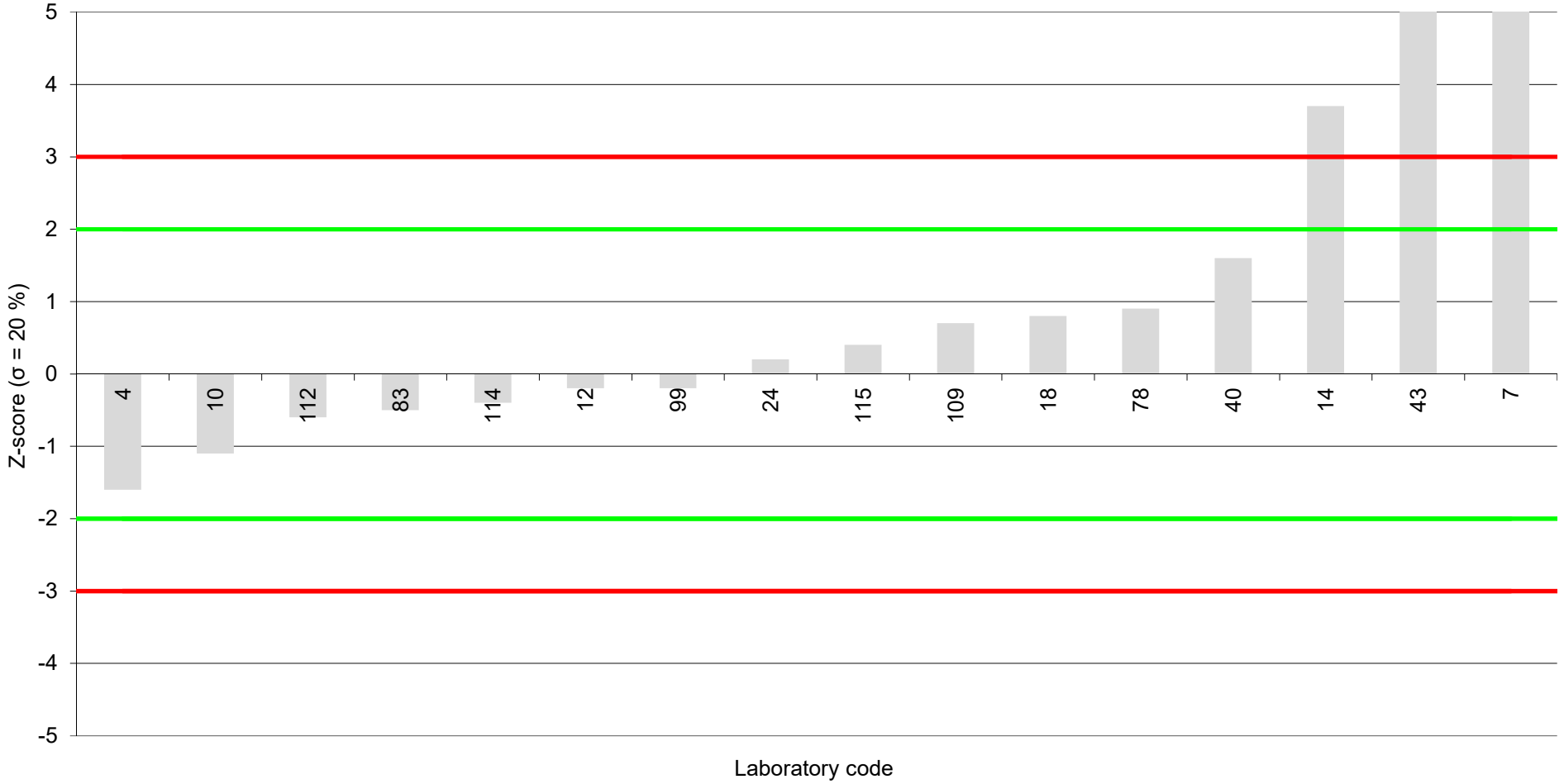
Assigned value: 0.886 µg/kg product (12 % moisture content)



Compound Feed (2302-CF)

(+/–)-α-HBCDD

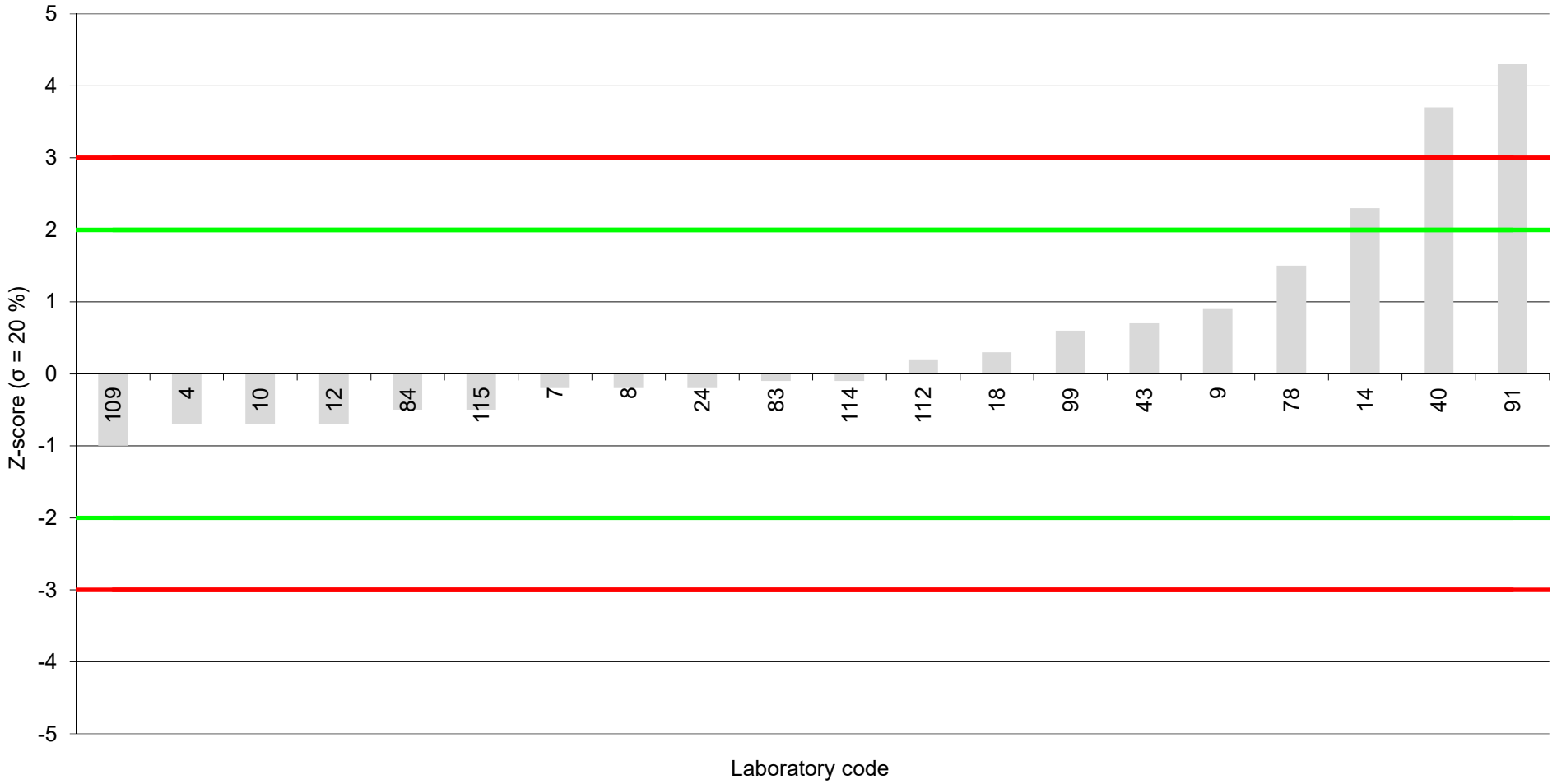
Assigned value: 0.0421 µg/kg product (12 % moisture content)



Compound Feed (2302-CF)

(+/–)-γ- HBCDD

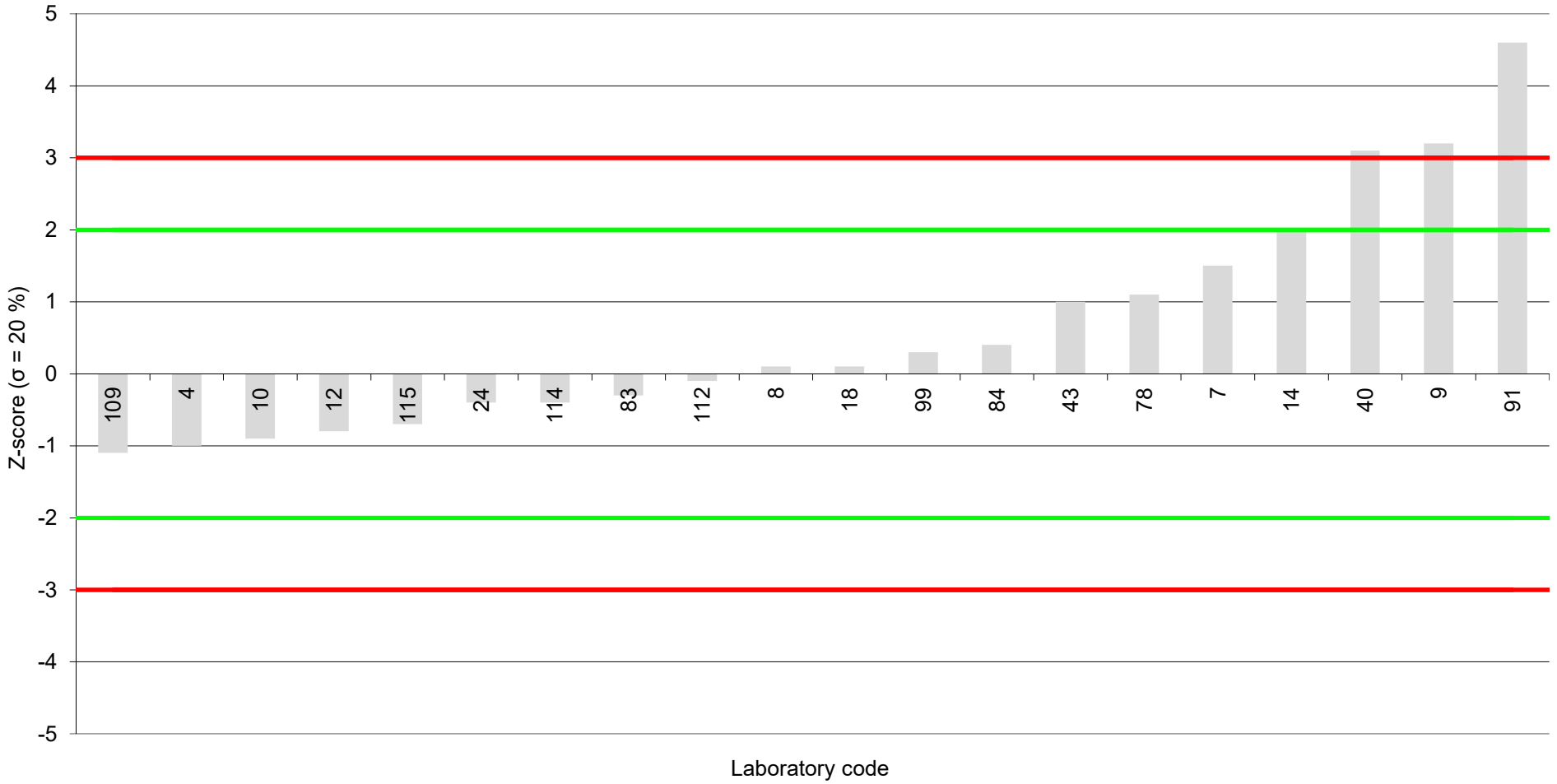
Assigned value: 0.576 µg/kg product (12 % moisture content)



Compound Feed (2302-CF)

Sum of α-, β-, γ-HBCDD ub

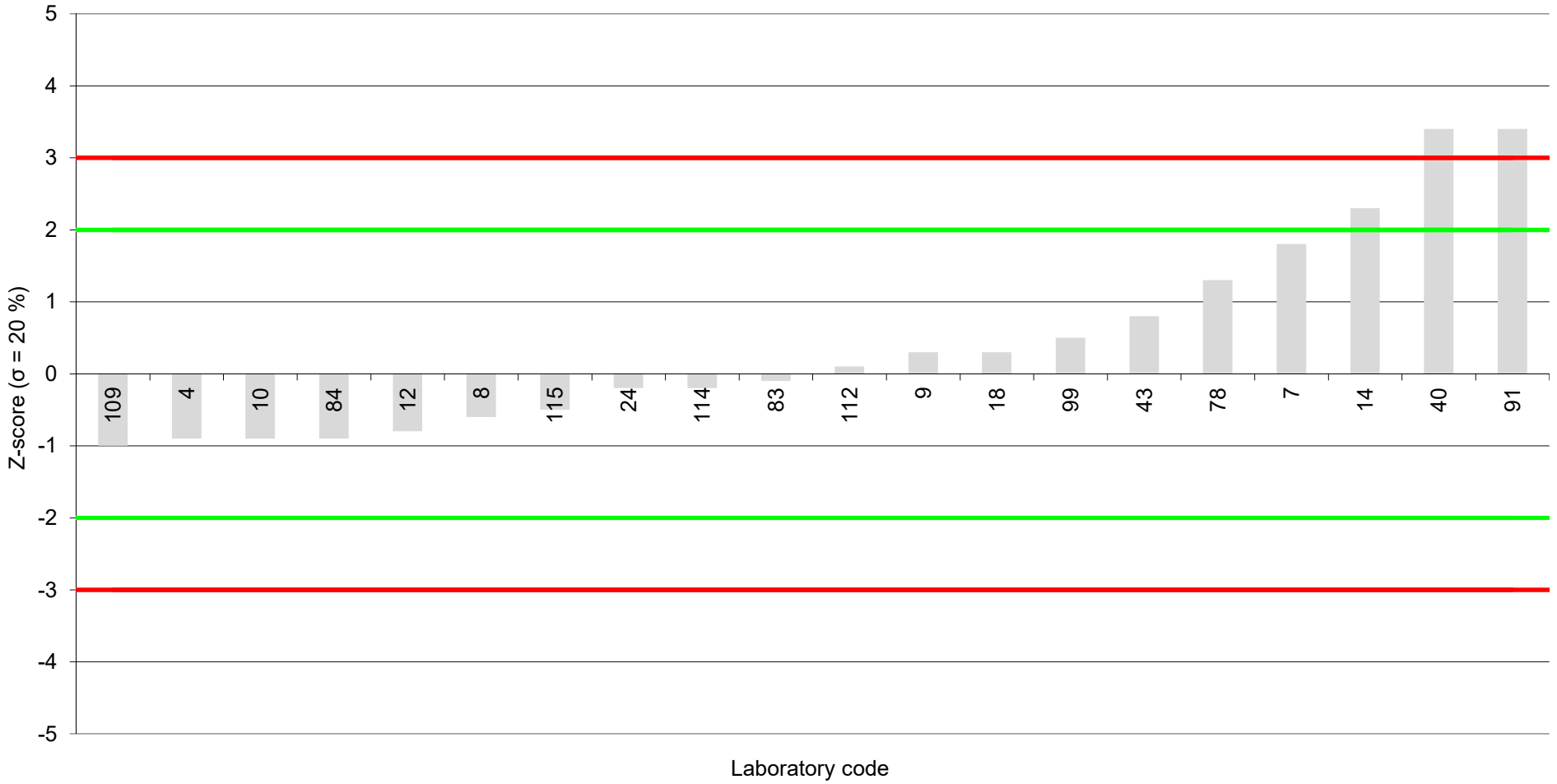
Assigned value: 0.661 µg/kg product (12 % moisture content)



Compound Feed (2302-CF)

Sum of α-, β-, γ-HBCDD lb

Assigned value: 0.634 µg/kg product (12 % moisture content)



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

Annex 5: Test for sufficient homogeneity and stability for PBDEs

Test sample - Compound Feed (2302-CF)

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compound Feed 2022 [EURL-PT-POP_2302-CF]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Compound Feed (2302-CF)
PBDE - Homogeneity test - Data

Analyte	Result µg/kg product (12% moisture content)	**Mean** (n = 10, duplicate analysis)	**Median** (n = 10, duplicate analysis)	**Relative standard deviation** [%]
Sum of PBDE without BDE-209 ub		0.442	0.443	5%
Sum of PBDE including BDE-209 ub*		1.09	1.15	14%
BDE-28		0.00813	0.00814	4%
BDE-47		0.107	0.108	5%
BDE-49		0.0194	0.0201	12%
BDE-99		0.0715	0.0712	5%
BDE-100		0.0250	0.0253	5%
BDE-153		0.0333	0.0331	6%
BDE-154		0.0238	0.0236	8%
BDE-183		0.154	0.155	7%
BDE-209*		0.665	0.712	22%

*only 10-fold determination due to blank issues

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compound Feed 2022 [EURL-PT-POP_2302-CF]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Compound Feed (2302-CF)
Selected PBDE congeners - Homogeneity test - Data

Sample	Replicate	Result µg/kg product (12% moisture content)	**BDE-153**
13	1		0.0324
	2		0.0349
45	1		0.0320
	2		0.0360
60	1		0.0313
	2		0.0324
74	1		0.0330
	2		0.0326
101	1		0.0321
	2		0.0344
129	1		0.0344
	2		0.0336
132	1		0.0319
	2		0.0338
143	1		0.0322
	2		0.0366
172	1		0.0288
	2		0.0333
196	1		0.0335
	2		0.0373
Cochran's C-test			
C			0.229
$C_{critical}$ (α = 0.05, m = 2, n = 10)			0.602
$C_{critical}$ (α = 0.01, m = 2, n = 10)			0.718
$C < C_{critical}$			yes
Outliers			no evidence for analytical outliers
Homogeneity test			
General average $\bar{x}$			0.0333
Standard deviation of sample averages s_x			0.00127
Wthin-sample standard deviation s_w			0.00209
Between-sample standard deviation s_s			0.0000
Standard deviation for proficiency assessment σ_{PT}			0.00666
s_s / σ_{PT}			0.0
Test for homogeneity ($s_s \leq 0.3\ \sigma_{PT}$)			passed

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compound Feed 2022 [EURL-PT-POP_2302-CF]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

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

Sample	Replicate	Result µg/kg product (12% moisture content)	**BDE-153**
58	1		0.0326
	2		0.0325
111	1		0.0330
	2		0.0351
184	1		0.0321
	2		0.0350
Stability test			
General average (stability test) $\bar{y}$			0.0334
General average (homogeneity test) $\bar{x}$			0.0333
Standard deviation for proficiency assessment σPT			0.0067
$\lvert \bar{y} - \bar{x} \rvert$			0.00007
Test for stability ($\lvert \bar{y} - \bar{x} \rvert \leq 0.3$ σPT)			passed

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

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

Test sample - Compound Feed (2302-CF)

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compounf Feed [EURL-PT-POP-2302-CF]

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

Compound Feed (2302-CF)

Physico-chemical Methods PBDEs - Internal standards

LC	Sample		**Weighed sample** [g]		**Use of isotope-labelled internal standards for …** PBDE congeners (yes/no)
4	2302-CF				
7	2302-CF		9.675		yes
8	2302-CF				
9	2302-CF		18.2		Yes
10	2302-CF		7		yes
11	2302-CF		30.0		yes
12	2302-CF		10.0		yes
14	2302-CF		10.0		
17	2302-CF		35		yes
18	2302-CF		5		YES
23	2302-CF		5.2		yes
24	2302-CF		30.0		yes
39	2302-CF		25.27		YES (15)
40	2302-CF		10.21		yes
43	2302-CF		20.8		yes
49	2302-CF		15.0		yes
67	2302-CF		40		Yes
78	2302-CF		30.06		yes
83	2302-CF		10.0		yes
84	2302-CF		10.0		yes
91	2302-CF		5g / 10g		yes
93	2302-CF		6		YES
96	2302-CF		36.2		yes (except for BDE-49)
99	2302-CF		10.0		yes
109	2302-CF		15		yes
112	2302-CF		5		yes
114	2302-CF		5.0		yes
115	2302-CF		20.0		yes

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compounf Feed [EURL-PT-POP-2302-CF]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Compound Feed (2302-CF)
Methods PBDEs - Internal Standards

LC	Sample	BDE 28	BDE 47	BDE 49	BDE 99	BDE 100	BDE 153	BDE 154	BDE 183	BDE 209
4	2302-CF									
7	2302-CF	13C-BDE28	13C-BDE47	13C-BDE47	13C-BDE99	13C-BDE100	13C-BDE153	13C-BDE154	13C-BDE183	
8	2302-CF									
9	2302-CF	13C12 BDE 28	13C12 BDE 47	13C12 BDE 47	13C12 BDE 99	13C12 BDE 100	13C12 BDE 153	13C12 BDE 154	13C12 BDE 183	13C12 BDE 209
10	2302-CF	BDE-28-13C12	BDE-47-13C12	BDE-47-13C12	BDE-99-13C12	BDE-100-13C12	BDE-153-13C12	BDE-154-13C12	BDE-183-13C12	BDE-209-13C12
11	2302-CF	BDE-28	BDE-47	BDE47	BDE-99	BDE-100	BDE-153	BDE-154	BDE-183	BDE-209
12	2302-CF	13C-BDE-28	13C-BDE-47	13C-BDE-47	13C-BDE-99	13C-BDE-100	13C-BDE-153	13C-BDE-154	13C-BDE-183	13C-BDE-209
14	2302-CF	13C-BDE-28	13C-BDE-47	13C-BDE-47	13C-BDE-99	13C-BDE-100	13C-BDE-153	13C-BDE-154	13C-BDE-183	13C-BDE-209
17	2302-CF	13C-BDE-28	13C-BDE-47	13C-BDE-47	13C-BDE-99	13C-BDE-100	13C-BDE-153	13C-BDE-154	13C-BDE-183	13C-BDE-209
18	2302-CF	13C BDE-28	13C BDE-47	13C BDE-47	13C BDE-99	13C BDE-100	13C BDE-153	13C BDE-154	13C BDE-183	13C BDE-209
23	2302-CF	PBDE 28 L	PBDE 47 L	PBDE 47 L	PBDE 99 L	PBDE 100 L	PBDE 153 L	PBDE 154 L	PBDE 183 L	PBDE 209 L
24	2302-CF	13C-BDE-28	13C-BDE-47	13C-BDE-47	13C-BDE-99	13C-BDE-100	13C-BDE-153	13C-BDE-154	13C-BDE-183	
39	2302-CF	28	47	47	99	100	153	154	183	209
40	2302-CF	13C12 BDE 28	13C12 BDE 47	13C12 BDE 47	13C12 BDE 99	13C12 BDE 100	13C12 BDE 153	13C12 BDE 154	13C12 BDE 183	13C12 BDE 209
43	2302-CF	13C-BDE-28	13C-BDE-47	13C-BDE-47	13C-BDE-99	13C-BDE-100	13C-BDE-153	13C-BDE-154	13C-BDE-183	13C-BDE-209
49	2302-CF									
67	2302-CF									
78	2302-CF	13C BDE-28	13C BDE-47	13C BDE-47	13C BDE-99	13C BDE-100	13C BDE-153	13C BDE-154	13C BDE-183	13C BDE-209
83	2302-CF	13C12-BDE-28	13C12-BDE-47	13C12-BDE-47	13C12-BDE-99	13C12-BDE-100	13C12-BDE-153	13C12-BDE-154	13C12-BDE-183	13C12-BDE-209
84	2302-CF	13C-BDE28	13C-BDE47	13C-BDE47	13C-BDE99	13C-BDE100	13C-BDE153	13C-BDE154	13C-BDE183	13C-BDE209
91	2302-CF	13C-BDE 28	13C-BDE 47	13C-BDE 47	13C-BDE 99	13C-BDE 100	13C-BDE 153	13C-BDE 154	13C-BDE 183	13C-BDE 209
93	2302-CF	13C-BDE-28	13C-BDE-47	13C-BDE-47	13C-BDE-99	13C-BDE-100	13C-BDE-153	13C-BDE-154	13C-BDE-183	13C-BDE-209
96	2302-CF	BDE-28	BDE-47	BDE-47	BDE-99	BDE-100	BDE-153	BDE-154	BDE-183	
99	2302-CF	MBDE-28	MBDE-47	MBDE-47	MBDE-99	MBDE-100	MBDE-153	MBDE-154	MBDE-183	MBDE-209
109	2302-CF	BDE-28	BDE-47	BDE-47	BDE-99	BDE-100	BDE-153	BDE-154	BDE-183	BDE-209
112	2302-CF	13C12-TriBDE 28	13C12-TetraBDE 47	13C12-TetraBDE 47	13C12-PentaBDE 99	13C12-PentaBDE 99	13C12-HexaBDE153	13C12-HexaBDE153	13C12-HeptaBDE 183	13C12-DecaBDE 209
114	2302-CF	13C12-TriBDE 28	13C12-TetraBDE 47	13C12-TetraBDE 47	13C12-PentaBDE 99	13C12-PentaBDE 99	13C12-HexaBDE153	13C12-HexaBDE153	13C12-HeptaBDE 183	13C12-DecaBDE 209
115	2302-CF	13C12-PBDE 28	13C12-PBDE 47	-	13C12-PBDE 99	13C12-PBDE 100	13C12-PBDE 153	13C12-PBDE 154	13C12-PBDE 183	-

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compounf Feed [EURL-PT-POP-2302-CF]

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

Compound Feed (2302-CF)

Methods PBDEs - Recovery Standards

LC	Sample		BDE 28	BDE 47	BDE 49	BDE 99	BDE 100	BDE 153	BDE 154	BDE 183	BDE 209
4	2302-CF										
7	2302-CF		13C-BDE126	13C-BDE126	13C-BDE126	13C-BDE126	13C-BDE126	13C-BDE126	13C-BDE126	13C-BDE126	
8	2302-CF										
9	2302-CF		13C12 BDE 79	13C12 BDE 79	13C12 BDE 79	13C12 BDE 139	13C12 BDE 139	13C12 BDE 139	13C12 BDE 139	13C12 BDE 180	13C12 BDE 206
10	2302-CF		BDE-77-13C12	BDE-77-13C12	BDE-77-13C12	BDE-77-13C12	BDE-77-13C12	BDE-138-13C12	BDE-138-13C12	BDE-138-13C12	BDE-138-13C12
11	2302-CF		PCB-138	PCB-138	PCB-138	PCB-138	PCB-138	PCB-138	PCB-138	PCB-138	PCB-138
12	2302-CF		13C-BDE-139	13C-BDE-139	13C-BDE-139	13C-BDE-139	13C-BDE-139	13C-BDE-139	13C-BDE-139	13C-BDE-139	13C-BDE-139
14	2302-CF		13C-BDE-139	13C-BDE-139	13C-BDE-139	13C-BDE-139	13C-BDE-139	13C-BDE-139	13C-BDE-139	13C-BDE-139	13C-BDE-206
17	2302-CF		13C-BDE-77	13C-BDE-77	13C-BDE-77	13C-BDE-126	13C-BDE-126	13C-BDE-126	13C-BDE-126	13C-BDE-126	13C-BDE-207
18	2302-CF		13C BDE-77	13C BDE-77	13C BDE-77	13C BDE-77	13C BDE-77	13C BDE-138	13C BDE-138	13C BDE-138	
23	2302-CF		PBDE 77 L	PBDE 77 L	PBDE 77 L	PBDE 77 L	PBDE 77 L	PBDE 77 L	PBDE 77 L	PBDE 77 L	4'-F-BDE 208
24	2302-CF		13C-BDE-77	13C-BDE-77	13C-BDE-77	13C-BDE-77	13C-BDE-77	13C-BDE-77	13C-BDE-77	13C-BDE-77	
39	2302-CF		79	79	79	79	79	139	139	180	206
40	2302-CF		13C12 BDE 139	13C12 BDE 139	13C12 BDE 139	13C12 BDE 139	13C12 BDE 139	13C12 BDE 139	13C12 BDE 139	13C12 BDE 139	13C12 BDE 139
43	2302-CF		13C-BDE-79	13C-BDE-79	13C-BDE-79	13C-BDE-138	13C-BDE-138	13C-BDE-138	13C-BDE-138	13C-BDE-138	13C-BDE-206
49	2302-CF										
67	2302-CF										
78	2302-CF		13C PCB-202	13C PCB-202	13C PCB-202	13C BDE-139	13C BDE-139	13C BDE-139	13C BDE-139	13C BDE-190	13C BDE-190
83	2302-CF		13C12-BDE-139	13C12-BDE-139	13C12-BDE-139	13C12-BDE-139	13C12-BDE-139	13C12-BDE-139	13C12-BDE-139	13C12-BDE-139	13C12-BDE-139
84	2302-CF		13C-BDE118	13C-BDE118	13C-BDE118	13C-BDE118	13C-BDE118	13C-BDE118	13C-BDE118	13C-BDE118	13C-BDE208
91	2302-CF		13C-BDE 118	13C-BDE 118	13C-BDE 118	13C-BDE 118	13C-BDE 118	13C-BDE 118	13C-BDE 118	13C-BDE 118	13C-BDE 118
93	2302-CF		13C-BDE-79	13C-BDE-79	13C-BDE-79	13C-BDE-79	13C-BDE-79	13C-BDE-138	13C-BDE-138	13C-BDE-138	13C-BDE-206
96	2302-CF		BDE-77	BDE-77	BDE-77	BDE-77	BDE-77	BDE-77	BDE-77	BDE-77	
99	2302-CF		MBDE-79	MBDE-79	MBDE-79	MBDE-139	MBDE-139	MBDE-139	MBDE-139	MBDE-139	MBDE-206
109	2302-CF		BDE-79	BDE-79	BDE-79	BDE-79	BDE-79	BDE-138	BDE-138	BDE-138	BDE-206
112	2302-CF		13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138
114	2302-CF		13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138
115	2302-CF		13C12-PCB 52	13C12-PCB 52	-	13C12-PCB 138	13C12-PCB 138	13C12-PCB 138	13C12-PCB 138	13C12-PCB 138	-

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compounf Feed [EURL-PT-POP-2302-CF]

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

Compound Feed (2302-CF)

Methods PBDEs - Comments

LC	Sample		BDE 28	BDE 47	BDE 49	BDE 99	BDE 100	BDE 153	BDE 154	BDE 183	BDE 209
4	2302-CF										
7	2302-CF										
8	2302-CF										
9	2302-CF		Co-elution with BDE-33								
10	2302-CF										
11	2302-CF										
12	2302-CF										
14	2302-CF										
17	2302-CF										
18	2302-CF										
23	2302-CF										
24	2302-CF										
39	2302-CF										<3x blank level
40	2302-CF										
43	2302-CF										
49	2302-CF										
67	2302-CF										
78	2302-CF										
83	2302-CF										
84	2302-CF										
91	2302-CF										
93	2302-CF										
96	2302-CF										
99	2302-CF										
109	2302-CF										
112	2302-CF										
114	2302-CF										
115	2302-CF				not analysed						not analysed

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compounf Feed [EURL-PT-POP-2302-CF]

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

Compound Feed (2302-CF)

Physico-chemical Methods HBCDDs - Internal standards

LC	Sample		**Weighed sample** [g]		**Use of isotope-labelled internal standards for …** HBCDD diastereomers (yes/no)
4	2302-CF		4		yes
7	2302-CF		4.065		yes
8	2302-CF		5		yes
9	2302-CF		1		Yes
10	2302-CF		7		yes
11	2302-CF		30		yes
12	2302-CF		3		Yes
14	2302-CF		10		yes
17	2302-CF				
18	2302-CF		5		YES
23	2302-CF				
24	2302-CF		30		yes
39	2302-CF				
40	2302-CF		10.21		yes
43	2302-CF		20.8228		yes
49	2302-CF				
67	2302-CF				
78	2302-CF		3.012		yes
83	2302-CF		10		yes
84	2302-CF		10		yes
91	2302-CF		1		yes
93	2302-CF		6		NO
96	2302-CF				
99	2302-CF		10		yes
109	2302-CF		15		yes
112	2302-CF		5		yes
114	2302-CF		5		yes
115	2302-CF		5		yes

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compounf Feed [EURL-PT-POP-2302-CF]

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

Compound Feed (2302-CF)

Methods HBCDDs - Internal Standards

LC	Sample		(+/–)-α-HBCDD	(+/–)-β- HBCDD	(+/–)-γ- HBCDD
4	2302-CF		13C12-a-HBCDD	13C12-a-HBCDD	13C12-a-HBCDD
7	2302-CF		13C-a-HBCD	13C-B-HBCD	13C-j-HBCD
8	2302-CF		a-Hexabromocyclododecane 13C12	b-Hexabromocyclododecane 13C12	g-Hexabromocyclododecane 13C12
9	2302-CF		alpha-HBCDD-13C12	alpha-HBCDD-13C12	alpha-HBCDD-13C12
10	2302-CF		alfa-HBCD-13C12	gamma-HBCD-13C12	gamma-HBCD-13C12
11	2302-CF				
12	2302-CF		13C12-α-HBCDD	13C12-β-HBCDD	13C12-γ-HBCDD
14	2302-CF		13C-α-HBCDD	13C-β-HBCDD	13C-γ-HBCDD
17	2302-CF				
18	2302-CF		13C-α-HBCDD	13C-β-HBCDD	13C-γ-HBCDD
23	2302-CF				
24	2302-CF		13C12-alpha-HBCDD	13C12-beta-HBCDD	13C12-gamma-HBCDD
39	2302-CF				
40	2302-CF		13C12 alfa-HBCDD	13C12 beta-HBCDD	13C12 gama-HBCDD
43	2302-CF		13C-α-HBCD	13C-β-HBCD	13C-γ-HBCD
49	2302-CF				
67	2302-CF				
78	2302-CF		13C-α-HBCDD	13C-β-HBCDD	13C-γ-HBCDD
83	2302-CF		13C alphaHBCDD	13C betaHBCDD	13C gammaHBCDD
84	2302-CF		13C -α- HBCDD	13C -β- HBCDD	13C -γ- HBCDD
91	2302-CF		alpha-HBCDD-C13	beta-HBCDD-C13	gamma-HBCDD-C13
93	2302-CF				
96	2302-CF				
99	2302-CF		13C12 alpha-HBCDD	13C12 beta-HBCDD	13C12 gamma-HBCDD
109	2302-CF		alpha-HBCDD	beta-HBCDD	gamma-HBCDD
112	2302-CF		13C12-α-HBCD	13C12-β-HBCD	13C12-γ-HBCD
114	2302-CF		13C12-α-HBCD	13C12-β-HBCD	13C12-γ-HBCD
115	2302-CF		alfa-HBCDD (13C12)	beta-HBCDD (13C12)	gamma-HBCDD (13C12)

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compounf Feed [EURL-PT-POP-2302-CF]

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

Compound Feed (2302-CF)

Methods HBCDDs - Recovery Standards

LC	Sample		(+/–)-α-HBCDD	(+/–)-β- HBCDD	(+/–)-γ- HBCDD
4	2302-CF				
7	2302-CF		none	none	none
8	2302-CF		18D-bHBCDD	18D-bHBCDD	18D-bHBCDD
9	2302-CF		4-nonylphenol-d8	4-nonylphenol-d8	4-nonylphenol-d8
10	2302-CF		beta-HBCD-13C12	beta-HBCD-13C12	beta-HBCD-13C12
11	2302-CF				
12	2302-CF		N/A	N/A	N/A
14	2302-CF				
17	2302-CF				
18	2302-CF		d18-β-HBCDD	d18-β-HBCDD	d18-β-HBCDD
23	2302-CF				
24	2302-CF				
39	2302-CF				
40	2302-CF				
43	2302-CF		13C-δ-HBCD	13C-δ-HBCD	13C-δ-HBCD
49	2302-CF				
67	2302-CF				
78	2302-CF		D18-β-HBCDD	D18-β-HBCDD	D18-β-HBCDD
83	2302-CF		d18-racbeta-1,2,5,6,9,10 - Hexabromocyklododecane	d18-racbeta-1,2,5,6,9,10 - Hexabromocyklododecane	d18-racbeta-1,2,5,6,9,10 - Hexabromocyklododecane
84	2302-CF		d18 -α- HBCDD	d18 -β- HBCDD	d18 -γ- HBCDD
91	2302-CF		alpha-HBCDD-d18	beta-HBCDD-d18	gamma-HBCDD-d18
93	2302-CF				
96	2302-CF				
99	2302-CF		alpha-HBCDD-d18	beta-HBCDD-d18	gamma-HBCDD-d18
109	2302-CF		D18-beta-HBCDD	D18-beta-HBCDD	D18-beta-HBCDD
112	2302-CF		d18-β-HBCD	d18-β-HBCD	d18-β-HBCD
114	2302-CF		d18-β-HBCD	d18-β-HBCD	d18-β-HBCD
115	2302-CF				

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compounf Feed [EURL-PT-POP-2302-CF]

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

Compound Feed (2302-CF)

Methods HBCDDs - Comments

LC	Sample		(+/–)-α-HBCDD	(+/–)-β- HBCDD	(+/–)-γ- HBCDD
4	2302-CF		Measurement uncertainty calculated for triplicate analysis of the sample		Measurement uncertainty calculated for triplicate analysis of the sample
7	2302-CF				
8	2302-CF				
9	2302-CF				
10	2302-CF				
11	2302-CF				
12	2302-CF				
14	2302-CF				
17	2302-CF				
18	2302-CF				
23	2302-CF				
24	2302-CF				
39	2302-CF				
40	2302-CF				
43	2302-CF				
49	2302-CF				
67	2302-CF				
78	2302-CF				
83	2302-CF				
84	2302-CF		Calculated result 0.024 ng/g; less than LOQ	Calculated result 0.005 ng/g; less than LOQ	
91	2302-CF				
93	2302-CF				
96	2302-CF				
99	2302-CF				
109	2302-CF				
112	2302-CF				
114	2302-CF				
115	2302-CF		commonly used LOQ is 0.05 ug/kg - for this purpose tried to use lower	commonly used LOQ is 0.05 ug/kg - for this purpose tried to use lower	

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

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

Test sample - Compound Feed (2302-CF)

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compounf Feed [EURL-PT-POP-2302-CF]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Compound Feed (2302-CF)
Physico-chemical Methods PBDEs - Pre-treatment and extraction

LC	Sample	Pre-treatment and extraction Sample preparation/pre-treatment	Extraction technique	Extraction solvent	Extraction time [h]	Extraction temperature [ºC]	Extraction pressure [MPa]
4	2302-CF						
7	2302-CF		Adding silica/H2SO4 44% and then extraction with 10 ml hexane (2x). Evaporated to dryness.		over night	Room temperature	
8	2302-CF						
9	2302-CF	n/a	soxhlet	Toluene	16	110	ambient
10	2302-CF	no	QuEChERs like-extraction	Ethyl acetate	no	no	no
11	2302-CF	drying (only for determination of moisture content)	ASE, Soxhlet	toluene, ethanol (ASE), toluene (Soxhlet)	30 min/sample (ASE), 8 h (Soxhlet)	100 °C (ASE)	10 MPa (ASE)
12	2302-CF		Soxhlet	Toluene	12h	boiling	
14	2302-CF		Soxhlet	toluene/acetone 7/3	6		
17	2302-CF		Soxhlet	Toluene / Ethanol (30/70)	12		
18	2302-CF	drying	PLE	Toluene/Acetone			
23	2302-CF		ASE	Hexane	5 min x2	100	10,3
24	2302-CF		Twisselmann	Ethanol/Toluol 70/30	6 h	boiling point	
39	2302-CF	No sample pre-treatment, used directly from container supplied.	ASE	Toluene	0.5	150	11.7
40	2302-CF	drying	Soxhlet	toluene:acetone (70:30), toluene	8	boiling point of solvent	normal
43	2302-CF	Homogenisation	Soxhlet	Toluene /ethanol 50 / 50	24	120	atm
49	2302-CF	-	ASE	(1) Toluene - (2) Toluene:Ethanol 90:10	0.25	100°C	10.3
67	2302-CF	Mixing with sodium sulfate	Soxhlet	Toluene	18	boiling	
78	2302-CF	ULTRA TURRAXING WITH HEXANE	SILICA GEL /SOLVENT EXTRACT- MANUAL	40:60 DCM:HEXANE	2-4HRS	AMBIENT	GRAVITY
83	2302-CF	freeze-dried	Accelerated Solvent Extraction (ASE)	toluene/acetone (70/30)	0.35	100	10
84	2302-CF	-	ASE	toluene (85%) / ethanol (15%)	20 min	120	10
91	2302-CF	none	Soxhlet	Toluene	8		
93	2302-CF	NO	SOXHLET	HEXANE/DCM (1:1)	24H		
96	2302-CF	sample mixed with sodium sulphate before extraction	Soxhlet	DCM:Hexane (50:50)	24 h		
99	2302-CF		ASE	Toluol Toluol/EtOH (9/1)	1 h	100 °C	10
109	2302-CF	Mixed with sodium sulphate	ASE	Hexane/Acetone (70:30)	1	125	10.3
112	2302-CF	homogenization, drying with polyacrylamide	cold extraction	hexane		room temperature	
114	2302-CF	homogenization, drying with polyacrylamide	cold extraction	hexane		room temperature	
115	2302-CF	thorough homogenization, drying with anhydrous Sodium Sulfate	Soxhlet (ultrasonic bath, agitate)	dichloromethane:acetone 3:1	21	109	0.1

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compounf Feed [EURL-PT-POP-2302-CF]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Compound Feed (2302-CF)
Physico-chemical Methods PBDEs - Clean-up

LC	Sample	Clean-up: Gelchromatography	Silica/sulfuric acid column	Florisil column	Alumina column	Carbon column	Others	Final volume [µl]: PBDE
4	2302-CF							
7	2302-CF	no	yes	no	no	no	no	250 µl
8	2302-CF							
9	2302-CF	No	Yes	No	Yes	No	No	45
10	2302-CF	yes	yes	no	no	no	no	250
11	2302-CF	no	yes	yes	yes	yes	PowerPrep FMS columns (basic-neutral silica, alumina, carbon)	20
12	2302-CF	yes	yes	yes	no	yes		50
14	2302-CF	no	yes	yes	yes	yes		100
17	2302-CF	no	yes	no	yes	yes	acidic treatment	50
18	2302-CF	YES	YES	YES	NO	YES		50
23	2302-CF	no	yes	no	yes	yes	Silver nitrate, AgNO3	65 ul
24	2302-CF	no	yes	no	yes	yes	no	100
39	2302-CF	Yes	Yes	No	Yes	Yes	Sample extract partitioned with concentrated sulfuric acid to remove bulk organic material residues	10µL
40	2302-CF	yes	yes	no	no	yes		20
43	2302-CF	no	yes	no	yes	no		25
49	2302-CF	no	yes	no	yes	yes	-	100
67	2302-CF	No	Yes	No	Yes	No	-	50
78	2302-CF	YES	YES	NO	YES	YES		25
83	2302-CF	no	yes	yes	yes	no	no	100
84	2302-CF	no	yes	no	yes	yes	no	500
91	2302-CF	no	yes	no	yes	yes	Silica/AgNO3	100
93	2302-CF	NO	YES	NO	YES	NO	NO	NO
96	2302-CF	no	yes	no			basic set of "power-prep system" columns	40
99	2302-CF	no	yes	no	yes	yes	MIURA	50
109	2302-CF	no	yes	no	yes	no	acid hydrolysis with sulphuric acid	40
112	2302-CF	no	yes	no	yes	no		100
114	2302-CF	no	yes	no	yes	no		100
115	2302-CF	no	yes	no	yes	yes	basic silica, silver nitrate silica	30

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compounf Feed [EURL-PT-POP-2302-CF]

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

Compound Feed (2302-CF)

Physico-chemical Methods PBDEs - Chromatographic separation and detection method

LC	Sample	Chromatographic separation and detection method			
		GC injection	Injected volume [µl]	Chromatographic separation: Stationary phase	Detector
4	2302-CF				
7	2302-CF	PTV	10	Rtx-CIPesticides	GC-HRMS
8	2302-CF				
9	2302-CF	Programmed Injection'	1	DB5	HRMS
10	2302-CF	PTV	10	DB5HT 15 m x 0.25 mm; 0.1 um	MS/MS
11	2302-CF	splitless	2	DB-5ht	HRMS
12	2302-CF	splitless	1	Rtx-1614	HRMS
14	2302-CF	splitless	2	DB-5MS	HRMS
17	2302-CF	splitless	2.0	DB-5HT	HRMS (Autospec Ultima Waters)
18	2302-CF	Splitless	2	HT8PCB	GC-HRMS
23	2302-CF	Tri-hepta PBDE: Pulsed splitless / Deca PBDE: PTV splitless	2	Tri-hepta PBDE: DB-XLB / Deca PBDE: Rtx-1614, 5% diphenyl, 95% dimethyl polysiloxane	Tri-hepta PBDE: MS/MS (EI) / Deca PBDE: Single quadropol MS (NCI)
24	2302-CF	PTV	5	Rtx-1614	HRMS
39	2302-CF	PTV	1µL	5%-Phenyl-Arylene-95% DimethylPolysiloxane (ZB5-MS) column 30m×0.1mm×0.1µm	Thermo DFS HRMS (>10,000 Mass resolution)
40	2302-CF	PTV	5	Zebron ZB-Semivolatiles	MS/MS (QQQ)
43	2302-CF	splitless	1	Rtx-1614	HRMS
49	2302-CF	PTV	5 µL	RTX-1614 (15x0.25x0.10)	HRMS
67	2302-CF	Splitless	1.5	DB-5 MS	HRMS, DFS
78	2302-CF	PTV	10	Rtx-1614	GC-HRMS
83	2302-CF	PTV Splitless	1	15 m RTX 1614	HRMS
84	2302-CF	splitless	5	(5%-phenyl)-methylpolysiloxane	MSMS
91	2302-CF	PTV	1	ZB - Semi Volatiles, 20m, 0.18 mm, 0.18µm	MS/MS
93	2302-CF	SPLITLESS	1	DB-5ms 13m x 0,18mm X 0,18um	HRMS
96	2302-CF	Splitless	1	DB-5MS (30 m, 0.25 mm id, 0,25 mm film)	HRMS (Mat-95 XP)
99	2302-CF		4	Rtx-1614 (15m x 0,25 mm x 0,1 µm) + 2m retention gap (uncoated)	MS/MS
109	2302-CF	splitless	1	Rtx-1614 30m x 0.25mm x 0.1µm	HRMS
112	2302-CF	pulsed splitless	2	Diphenyl-/dimethylpolysiloxan (5%/95%)	MS/MS
114	2302-CF	pulsed splitless	2	Diphenyl-/dimethylpolysiloxan (5%/95%)	MS/MS
115	2302-CF	pulsed splitless	1	DB-5MS (60m x 0,25mm x 0,10 µm)	HRMS (R>10000)

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compounf Feed [EURL-PT-POP-2302-CF]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Compound Feed (2302-CF)
Physico-chemical Methods HBCDDs - Pre-treatment and extraction

LC	Sample	Pre-treatment and extraction Sample preparation/pre-treatment	Extraction technique	Extraction solvent	Extraction time [h]	Extraction temperature [ºC]	Extraction pressure [MPa]
4	2302-CF		Soxleth	Acetone/Hexane (1/1)	6.00 XDR		
7	2302-CF	Sodium Sulfate	ASE	Hexane/Acetone 1:1	30 minutes	100.00 XDR	1500 psi
8	2302-CF	drying		hexane:dichloromethane 50/50	0.33 XDR	ambiente	ambiente
9	2302-CF	none	Soxhlet	DCM	16.00 XDR	40.00 XDR	ambient
10	2302-CF		QuEChERS-like	Ethyl Acetate			
11	2302-CF	drying (only for determination of moisture content)	ASE, Soxhlet	toluene, ethanol (ASE), toluene (Soxhlet)	30 min/sample (ASE), 8 h (Soxhlet)	100 °C (ASE)	10 MPa (ASE)
12	2302-CF	Drying using sodium sulfate	Ultrasound assisted extraction	DCM:Hex (1:1, v/v)	30 min	Room temp.	N/A
14	2302-CF		Soxhlet	toluene/acetone 7/3	6.00 XDR		
17	2302-CF						
18	2302-CF	drying	PLE	Toluene/Acetone			
23	2302-CF						
24	2302-CF		Twisselmann	Ethanol/Toluol 70/30	6 h	boiling point	
39	2302-CF						
40	2302-CF	drying	Soxhlet	toluene:acetone (70:30), toluene	8.00 XDR	boiling point of solvent	normal
43	2302-CF	Homogenisation	Soxhlet	Toluene /ethanol 50 / 50	24.00 XDR	120.00 XDR	atm
49	2302-CF						
67	2302-CF						
78	2302-CF	none	acidified silica homogenisation	40/60 DCM/hexane	0.20 XDR	ambient	n/a
83	2302-CF	no	ASE 350	toluene/acetone (70/30)	0.35 XDR	100.00 XDR	10.00 XDR
84	2302-CF	-	ASE	toluene (85%) / ethanol (15%)	20 min	120.00 XDR	10.00 XDR
91	2302-CF		Soxhlet	Toluene	12.00 XDR	unknown	ambient pressure
93	2302-CF	NO	SOXHLET	HEXANE/DCM (1/1)	24H		
96	2302-CF						
99	2302-CF		cold extraction	DCM/Hexan 1/1	roughly 2-3 hours	ambient	ambient
109	2302-CF	reconstitution of liquid milk by adding wate	liquid-liquid partitioning process	ethanol/diethylether/petroleum ether (1/2/2)			
112	2302-CF	homogenization	Quechers	acetonitrile/water (1/1)	10 min	room temperature	
114	2302-CF	homogenization	Quechers	acetonitrile/water (1/1)	10 min	room temperature	
115	2302-CF	drying	shaking with dichlormethan : acetone 2:1 for 90 min	100 ml of dichlormethan : acetone 2:1	90 min	ambient	0.10 XDR

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compounf Feed [EURL-PT-POP-2302-CF]

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

Compound Feed (2302-CF)

Physico-chemical Methods HBCDDs - Clean-up

LC	Sample		Clean-up Gelchromatography	Silica/sulfuric acid column	Florisil column	Alumina column	Carbon column	Others	Final volume [µl]: HBCDD
4	2302-CF		no	no	no	no	no	Conc. sulphuricacid	200
7	2302-CF		no	yes	no	no	no		500 µl
8	2302-CF		no	yes	no	no	no		200
9	2302-CF		No	Yes	No	No	No	n/a	500
10	2302-CF		yes	no	no	no	no	Extrelut NT-3 column, acidic for H2SO4 tandem Si 1g/6 mL column	250
11	2302-CF		no	yes	yes	yes	yes	PowerPrep FMS columns (basic-neutral silica, alumina, carbon)	20
12	2302-CF		No	Yes (both)	Yes	No	No	Direct H2SO4 treatment	50
14	2302-CF		yes	no	no	no	no	silica gel	500
17	2302-CF								
18	2302-CF		YES	YES	NO	NO	NO	liquid liquid extraction	50
23	2302-CF								
24	2302-CF		no	yes	yes	no	no	no	100
39	2302-CF								
40	2302-CF		yes	yes	no	no	yes		200
43	2302-CF		no	yes	no	no	no		1000
49	2302-CF								
67	2302-CF								
78	2302-CF		no	no	no	no	no	clean-up combined in extraction	150
83	2302-CF		no	yes	no	no	no		50
84	2302-CF		no	yes	no	no	no	no	500
91	2302-CF		no	yes	yes	no	no	no	500
93	2302-CF		NO	YES	NO	YES	NO	NO	20
96	2302-CF								
99	2302-CF		no	yes	no	no	no	no	400
109	2302-CF		no	yes	no	no	no	acid hydrolysis with sulphuric acid	100
112	2302-CF		no	yes	no	no	no		1000
114	2302-CF		no	yes	no	no	no		1000
115	2302-CF		yes	yes	no	no	no		500

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compounf Feed [EURL-PT-POP-2302-CF]

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

Compound Feed (2302-CF)

Physico-chemical Methods HBCDDs - Chromatographic separation and detection method

			Chromatographic separation and detection method			
LC	Sample		Injection	Injected volume [µl]	Chromatographic separation: Stationary phase	Detector
4	2302-CF		HPLC injection	5	Acquity BEH C18 (1.7 µm, 2.1*100 mm)	MS/MS
7	2302-CF			10	C18 (Waters symmetry), 150 mm x 2.1 mm x 3.5 µm	LCMSMS
8	2302-CF		UPLC	5	Eclipse Plus C18, 2,1 x 100mm	MS/MS
9	2302-CF		loop	5uL	ACQUITY UPLC BEH C18 1.7 um	MS/MS
10	2302-CF			20 µL	KINETEX 2.6um XB-C18 100A (100 x 2.1 mm) (PHENOMENEX)	LC-MS/MS
11	2302-CF		splitless	2	DB-5ht	HRMS
12	2302-CF		-	5	C18-RP-HPLC	HRMS
14	2302-CF			10	RP 18	LC-MS/MS
17	2302-CF					
18	2302-CF			15	Hypersil Gold	MS/MS
23	2302-CF					
24	2302-CF		HPLC	2	C18	MS/MS
39	2302-CF					
40	2302-CF			70	Luna PFP	MS/MS
43	2302-CF		LC		Acquity BEH C18 150 mm	MS/MS
49	2302-CF					
67	2302-CF					
78	2302-CF		Liquid	5	Agilent Zorbax RRHD Eclipse Plus C18 1.8 µm (150 x 2.1 mm)	LC-MS/MS
83	2302-CF			10	Hypersil Gold C18, 100x2,1mm, 1,9µm	MS/MS
84	2302-CF		ESI negative	5 µl	C18 column	MS/MS
91	2302-CF		direct injection	5, 10, 20, 40	reversed phase C18 (Eclipse XDB-C18 5µm 4,6x150mm)	MS/MS
93	2302-CF		SPLITLESS	1	DB-5ms 40m x 0,18 mm x 0,18 um	HRMS
96	2302-CF					
99	2302-CF			15	C18	MS/MS
109	2302-CF			5	BEH C18 (50 mm x 2.1 mm; 1.7 µm)	HRMS
112	2302-CF		standard	10	C18	MS/MS
114	2302-CF		standard	10	C18	MS/MS
115	2302-CF		normal	10	C18	MS/MS

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

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

Test sample - Compound Feed (2302-CF)

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compounf Feed [EURL-PT-POP-2302-CF]

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

Compound Feed (2302-CF)

Determination of Limit of Detection and Measurement Uncertainty (MU) - PBDEs

LC	Sample	**Methods apllied to determine** Limit of Quantification (LOQ)	Measurement Uncertainty (MU)	Additional Information
4	2302-CF	LOQ was obtained from blank samples made in the same batch as PT samples.	Obtained during method validation additional, MUs were compared with a standard deviation of PT sample, higher value was taken to report.	
7	2302-CF	Samples spiked at LOQ level (20 pg/g; 200ppg for BDE 209); for BDE 47 LOQ (35 pg/g) was estimated from procedural blanks run within the sample batch	Nordtest	
8	2302-CF			Calculation methodology as per USEPA Method 1614
9	2302-CF	low calibration standard level		
10	2302-CF			PBDEs have been analysed in the PCBs fraction of the dioxin protocol
11	2302-CF	Calculated by the software	Established from quality control charts (2*RSD)	
12	2302-CF	S/N	Control chart of reference material	
14	2302-CF			
17	2302-CF			
18	2302-CF			
23	2302-CF			
24	2302-CF			
39	2302-CF			
40	2302-CF			
43	2302-CF	blank + 3s		
49	2302-CF			
67	2302-CF	S/N > 3		
78	2302-CF			
83	2302-CF	reporting limit = 0.001 or 0.005 µg/kg 12% moisture content according to NEN 7777	according to NEN 7777 expanded measurement uncertainty	
84	2302-CF	LOQ = 3 x LOD LOD evaluated as 3 times of noise	Expanded U: Ue = k x Uc (k = 2). MU of type A is evaluated using internal RM. U of type B (includes i.a. uncertainity of weighing, volume measurements, etc.) Uc = combined uncertainty is summed uncertainty of type A and B according the law of propagation of measurement uncertainty	
91	2302-CF			
93	2302-CF			
96	2302-CF	The LOQs were estimated using the approach "Congener-based LOQs from S/N Calculations". This methodology is reported in the Guidance Document on the Estimation of LOD and LOQ for Measurements in the Field of Contaminants in Feed and Food.	The MU was estimated using the "semi-empirical approach" based on the ISO Guide to the expression of Uncertainty in Measurement applied to analytical chemistry by EURACHEM/CITAC. This approach is reported in the Guidance Document on Measurement Uncertanty for Laboratories performing PCDD/F and PCB Analysis using Isotope Dilution Mass Spectrometry	
99	2302-CF			
109	2302-CF			
112	2302-CF			
114	2302-CF			
115	2302-CF			

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compounf Feed [EURL-PT-POP-2302-CF]

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

Compound Feed (2302-CF)

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

LC	Sample	BDE 28	BDE 47	BDE 49	BDE 99	BDE 100	BDE 153	BDE 154	BDE 183	BDE 209
4	2302-CF									
7	2302-CF	0.001	0.001	0.001	0.005	0.005	0.005	0.005	0.005	
8	2302-CF									
9	2302-CF	0.001	0.001	0.001	0.0015	0.0015	0.002	0.002	0.0025	0.005
10	2302-CF									
11	2302-CF	0.00043	0.00016	0.00016	0.00097	0.00083	0.00073	0.00065	0.00145	0.01039
12	2302-CF	0.00011	0.00024	0.00035	0.00024	0.00015	0.00056	0.00032	0.00056	0.00404
14	2302-CF									
17	2302-CF									
18	2302-CF									
23	2302-CF	0.005	0.039	0.01	0.019	0.019	0.019	0.019	0.019	0.038
24	2302-CF									
39	2302-CF	0.001	0.006	0.001	0.003	0.001	0.001	0.001	0.001	1
40	2302-CF	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
43	2302-CF									0.295
49	2302-CF									
67	2302-CF	0.0019	0.0019	0.0019	0.0019	0.0019	0.0019	0.0019	0.0019	
78	2302-CF	0.002	0.002	0.002	0.002	0.002	0.002	0.002	0.002	0.027
83	2302-CF	0.0005	0.0005	0.0006	0.0013	0.0006	0.0007	0.0007	0.001	0.0063
84	2302-CF									
91	2302-CF	0.002	0.0095	0.0014	0.0034	0.00096	0.0016	0.0052	0.001	0.055
93	2302-CF	0.000083	0.00027	0.00055	0.00015	0.00011	0.00046	0.00031		0.0029
96	2302-CF	0.00003	0.00002	0.00003	0.00004	0.00004	0.00004	0.00005	0.00018	
99	2302-CF									
109	2302-CF	0.00049	0.034	0.00098	0.014	0.0049	0.00029	0.00052	0.00012	0.031
112	2302-CF	0.002	0.002	0.002	0.004	0.004	0.006	0.006	0.01	0.2
114	2302-CF	0.002	0.002	0.002	0.004	0.004	0.006	0.006	0.01	0.2
115	2302-CF									

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compounf Feed [EURL-PT-POP-2302-CF]

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

Compound Feed (2302-CF)

Methods PBDEs - Measurement Uncertainty [%]

LC	Sample	BDE 28	BDE 47	BDE 49	BDE 99	BDE 100	BDE 153	BDE 154	BDE 183	BDE 209
4	2302-CF									
7	2302-CF	25	25	25	25	25	25	25	25	
8	2302-CF									
9	2302-CF	20	20	25	20	20	20	20	20	20
10	2302-CF	59	42	59	42	59	59	59	42	42
11	2302-CF	64.5	20.4	20.4	17.6	19.7	19.95	23.7	9.68	31.4
12	2302-CF	20	20	20	20	20	20	20	20	30
14	2302-CF	25	25	25	25	25	25	25	30	30
17	2302-CF	30	30	60	30	30	30	30	30	30
18	2302-CF									
23	2302-CF	30	30	30	30	30	30	30	50	40
24	2302-CF									
39	2302-CF	±15	±15	±15	±15	±15	±15	±15	±15	
40	2302-CF	35	30	45	30	30	30	30	30	45
43	2302-CF									
49	2302-CF									
67	2302-CF	10	10	10	10	10	10	10	10	10
78	2302-CF									
83	2302-CF	29.2	24	24.8	23.8	37.9	26.9	24.8	19.3	28.4
84	2302-CF	70	30	50	30	50	50	50	50	50
91	2302-CF	30	30	30	30	30	30	30	30	40
93	2302-CF									
96	2302-CF									
99	2302-CF									
109	2302-CF	28	41	34	34	27	27	28	36	40
112	2302-CF	30	30	30	30	30	30	30	30	30
114	2302-CF	30	30	30	30	30	30	30	30	30
115	2302-CF	30	30	-	30	30	30	30	30	-

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compounf Feed [EURL-PT-POP-2302-CF]

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

Compound Feed (2302-CF)

Determination of Limit of Detection and Measurement Uncertainty (MU) - HBCDDs

LC	Sample		**Methods apllied to determine** Limit of Quantification (LOQ)	Measurement Uncertainty (MU)	Additional Information
4	2302-CF		From triplicate analysis of the compound feed sample LOQ estimated as 10*S/N of calculated concentration	From triplicate analysis uncertainty calculated as relative standarddeviation from the results	
7	2302-CF		3 x blank value	according to NEN 7777	
8	2302-CF		The LOQs corespond to the lowest level of the calibration curve	U= k*u=((2x CVrw) + biais) for validated matrices	
9	2302-CF		Low Calibration Std		
10	2302-CF		Samples spiked at LOQ level (10 pg/g) within the batch	From validation data with a semplified bottom up approach	
11	2302-CF		LOQ = 3 x LOD LOD evaluated as 3 times of noise		
12	2302-CF		Empirically determined as the lowest calibration point producing signal above S/N 10	Established from quality control charts (2*RSD)	
14	2302-CF				
17	2302-CF				
18	2302-CF				
23	2302-CF				
24	2302-CF				
39	2302-CF				
40	2302-CF				
43	2302-CF				
49	2302-CF				
67	2302-CF				
78	2302-CF				
83	2302-CF		LOD and LOQ were estimated based on analysis of 10 blank spiked samples	MU were estimated based on precision and truenes from fortyfication experiment	
84	2302-CF		LOQ = 8 x SD where, SD is the standard deviation of the lowest successfully validated level of HBCDD	U = k x uc where, Coverage factor k =2 is used, corresponding to a level of confidence of ~95% and, uc is combined standard uncertainty, calculated from the combination of the relative uncertainty components describing the random variations and the bias contribution	
91	2302-CF		calculated on base of standard with S/N 10:1 multiplied with the worst recovery rate	estimated value based on the RSD of multiple determination	Method according to Bichon et.al. 2018
93	2302-CF				
96	2302-CF				
99	2302-CF		ten point equidistant calibration in Matrix at the expected LOQ using DIN 32645		minimal traces of TBBPA were detected. The detected amounts were < LOQ
109	2302-CF		The LOQ was estimated following the "Calibration Standards" approach. This methodology is reported in the Guidance Document on the Estimation of LOD and LOQ for Measurements in the Field of Contaminants in Feed and Food.	The MU was estimated using the Top-Down approach reported in the Guidance Document on Measurement Uncertanty for Laboratories performing PCDD/F and PCB Analysis using Isotope Dilution Mass Spectrometry	
112	2302-CF				
114	2302-CF				
115	2302-CF				

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compounf Feed [EURL-PT-POP-2302-CF]

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

Compound Feed (2302-CF)

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

LC	Sample		(+/–)-α-HBCDD	(+/–)-β- HBCDD	(+/–)-γ- HBCDD
4	2302-CF		0.007	0.006	0.005
7	2302-CF		0.05	0.01	0.02
8	2302-CF		0.076	0.038	0.076
9	2302-CF		0.27	0.15	0.099
10	2302-CF			0.01	
11	2302-CF				
12	2302-CF		0.025	0.025	0.025
14	2302-CF				
17	2302-CF				
18	2302-CF				
23	2302-CF				
24	2302-CF				
39	2302-CF				
40	2302-CF		0.01	0.01	0.01
43	2302-CF			0.05	
49	2302-CF				
67	2302-CF				
78	2302-CF		0.01	0.01	0.01
83	2302-CF		0.006	0.005	0.005
84	2302-CF		0.1	0.1	
91	2302-CF		0.1	0.1	0.1
93	2302-CF				
96	2302-CF				
99	2302-CF				
109	2302-CF		0.01	0.01	0.01
112	2302-CF		0.03	0.03	0.03
114	2302-CF		0.03	0.03	0.03
115	2302-CF		0.01	0.005	0.05

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Compounf Feed [EURL-PT-POP-2302-CF]

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

Compound Feed (2302-CF)

Methods HBCDDs - Measurement Uncertainty [%]

LC	Sample		(+/–)-α-HBCDD	(+/–)-β- HBCDD	(+/–)-γ- HBCDD
4	2302-CF		0.18		0.1
7	2302-CF		13	7.5	13
8	2302-CF		50	50	50
9	2302-CF				20
10	2302-CF		36	49	36
11	2302-CF				
12	2302-CF		21	21	21
14	2302-CF		20	20	20
17	2302-CF				
18	2302-CF				
23	2302-CF				
24	2302-CF				
39	2302-CF				
40	2302-CF		30	30	30
43	2302-CF				
49	2302-CF				
67	2302-CF				
78	2302-CF				
83	2302-CF		30	20	18
84	2302-CF		0.3	0.3	0.3
91	2302-CF		30	30	30
93	2302-CF				
96	2302-CF				
99	2302-CF				
109	2302-CF		27	30	32
112	2302-CF		30	30	30
114	2302-CF		30	30	30
115	2302-CF		23	18	16