EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024

EURL-PT-DPB_2402-BT

FEED

Report

PBDEs and HBCDDs

(Report Version 1.0)

26 August 2025

Funded by the European Union

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Summary

Test sample	**FEED:** Bentonite [2402-BT]
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	26 August 2025 (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 and HBCDDs in **Bentonite** was organized by the EURL for halogenated POPs in Feed and Food to be performed between August and November 2024. The objective is to assess analytical performance of laboratories and interlaboratory comparability of results from analyses of PCDD/Fs, PCBs, PBDEs and HBCDDs in one sample of **Bentonite**.

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 2024. 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 26 and 27 November 2024.

1.1. Samples and coding

The test sample was prepared from contaminated feed and fortified with some analytes of interest using analytical standards or technical mixtures of PCBs, PBDEs and HBCDDs.

Bentonite	**Sample no. 2402-BT-xxx**

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

1.2. Analytes of interest

Participants 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.
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

93 laboratories registered for this proficiency test. For PBDE and HBCDD, 27 and 19 sets of results were reported, respectively. One laboratory submitted two sets of data

Table 1: Participating laboratories

Participating laboratories	Region	No. all participants	No. participants reporting PBDE/HBCDD results
National Reference Laboratories	European Union	25	19
	Other Countries	4	1
Official Laboratories	European Union	41	6
	Other European Countries	-	-
	Africa	-	-
	Americas	2	-
	Asia	-	-
	Oceania	-	-
Commercial Laboratories	European Union	16	3
	Other European Countries	-	-
	Africa	-	-
	Americas	4	-
	Asia	-	-
	Oceania	1	-
	Total	**93**	**29**

2.1. Number of reported results

Table 2: Reported results for PBDEs and moisture content for Bentonite (2402-BT)

Reported results (2402-BT)	All laboratories
BDE-28, 47, 99, 100, 153, 154, 183	27
BDE-49, 209	26, 20
Sum of 8 PBDEs (without BDE-209) (ub)	27
Sum of 8 PBDEs (without BDE-209) (lb)	26
Sum of 9 PBDEs (with BDE-209) (ub)	20
Sum of 9 PBDEs (with BDE-209) (lb)	19
Moisture content	26

Table 3: Reported results for HBCDDs for Bentonite (2402-BT)

Reported results (2402-BT)	All laboratories
α-, β-, γ-HBCDD	19
Sum of α-, β-, γ-HBCDD (ub)	19
Sum of α-, β-, γ-HBCDD (lb)	18
Total HBCDD (using GC methods)	2

2.2. Accreditation

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

Bentonite	PBDEs	HBCDDs
Accreditation	21	9
No accreditation	6	10

2.3. Detection methods

The following detection methods were applied:

- GC-HRMS-, GC-MS/MS-methods for PBDEs
- GC-HRMS-, GC-MS/MS-, 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 Bentonite (2402-BT)

Detection methods	PBDEs	HBCDDs
GC-HRMS	17	1
GC-MS/MS	10	1
GC-NCI-MS	-	-
LC-MS/MS	-	15
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 2402-BT 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 ISO 13528:2022 [1] and the International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [2].
The determination of the assigned value was performed according [1] by estimating of the assigned value as the consensus of participants' results (using only results of physico-chemical methods). The Huber robust mean was taken as assigned value after excluding extreme outliers (outside the range of ± 50% of the median of all reported results) and examination of the distribution of the remaining results using histogram and Kernel density estimation, if necessary.

Assigned values were calculated for 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/diastereomers (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:

- BDE-28 and 49
- (+/–)-α- HBCDD and (+/–)-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 29 participating laboratories were accredited according to ISO/IEC 17025 for PBDEs. No significant differences (0 to 3 %) between the assigned values calculated for both data sets for PBDEs were observed. For HBCDDs only 9 out of 29 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)	0.385	0.385	<1
Sum of PBDE without BDE-209 (lb)	0.378	0.378	<1
Sum of PBDE including BDE-209 (ub)	0.611	0.597	2
Sum of PBDE including BDE-209 (lb)	0.566	0.549	3
γ-HBCDD	0.907	-*	
Sum of α-, β-, γ-HBCDD (ub)	0.988	-*	
Sum of α-, β-, γ-HBCDD (lb)	0.939	-*	

*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 2402-BT 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)

Bentonite (2402-BT)	Assigned value µg/kg product (12 % moisture content)
BDE-47	0.0711
BDE-99	0.0920
BDE-100	0.0188
BDE-153	0.0320
BDE-154	0.0109
BDE-183	0.154
BDE-209	0.257
Sum of 8 PBDEs (without BDE-209) (ub)	0.385
Sum of 8 PBDEs (without BDE-209) (lb)	0.378
Sum of 9 PBDEs (with BDE-209) (ub)	0.611
Sum of 9 PBDEs (with BDE-209) (lb)	0.566

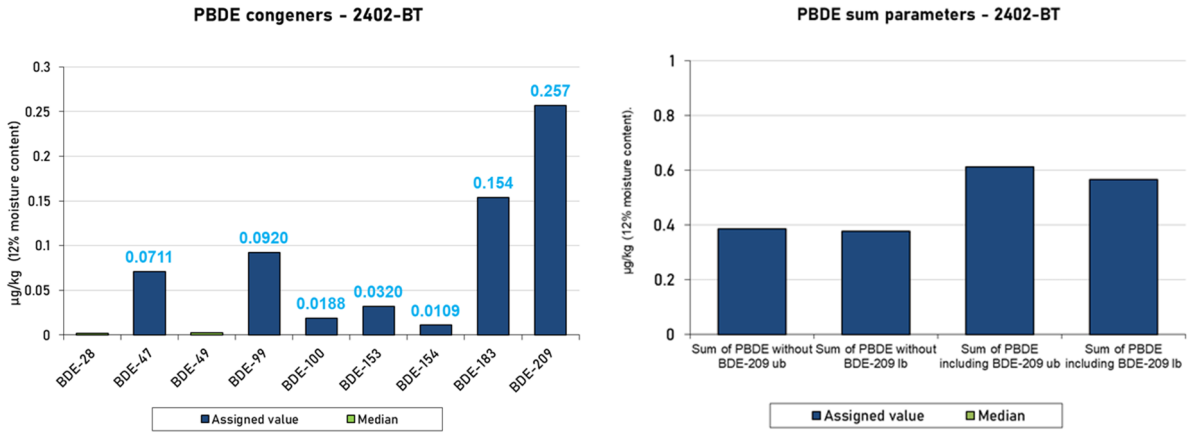

Figure 1: Assigned values (blue) for PBDE individual congeners and sum parameters for Bentonite (2402-BT) [µg/kg product (12% moisture content)]

4.2. HBCDDs – individual stereoisomers and sum parameter

The assigned values for the test sample 2402-BT were calculated as consensus of participants' results for individual HBCDDs and sum parameters, taking into account the calculation criteria described above (Table 9; tabular summary see annex 1; Figure 2).

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

Bentonite (2402-BT)	Assigned value µg/kg product (12 % moisture content)
(+/–)-γ -HBCDD	0.907
Sum of α-, β-, γ-HBCDD (ub)	0.988
Sum of α-, β-, γ-HBCDD (lb)	0.939

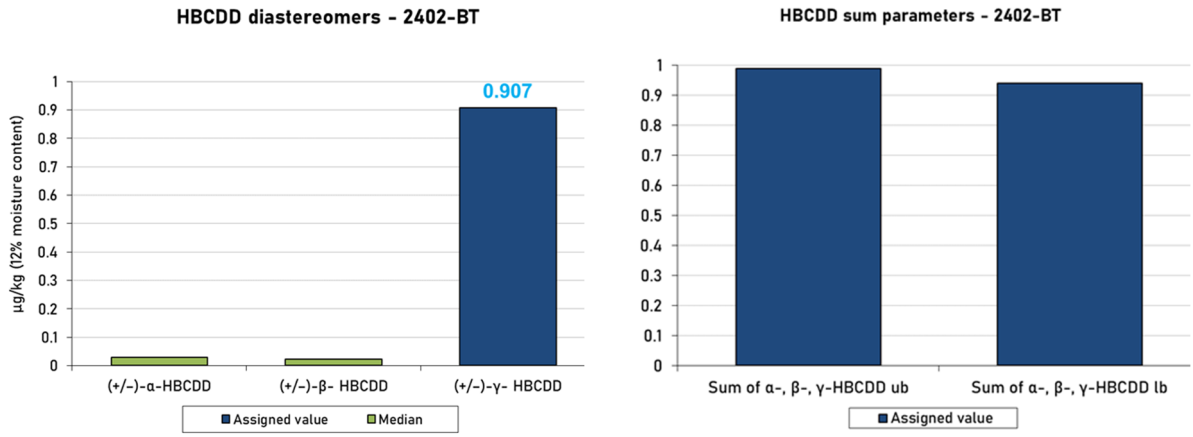

Figure 2: Assigned values (blue) and median values (green) for HBCDD individual congeners and sum parameters for Bentonite (2402-BT) [µg/kg product (12% moisture content)]

4.3. Moisture content

For the moisture content an assigned value of 9.26 % for the test sample 2402-BT was calculated as a consensus of the participants' results, taking into account the calculation criteria described above.

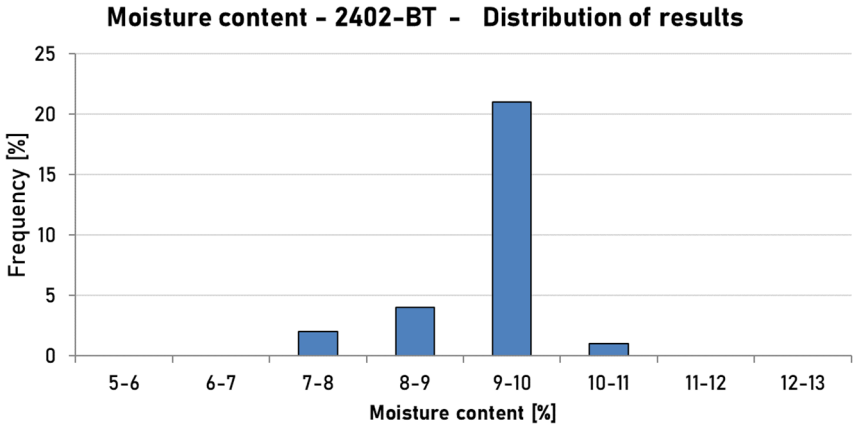

Figure 3: Distribution of participant's results of the moisture content in % for Bentonite (2402-BT)

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:

Assigned values for BDE-47, -99, -153, -183 and -209 were three to twenty-five times higher compared to the recommended LOQs (Table 7). But for BDE-100 and BDE-154 the assigned values were 0.0188 and 0.0109 µg/kg product, respectively, which were within the range of the recommended LOQ of 0.01 µg/kg product. No assigned values could be calculated for BDE-28 and BDE-49. For BDE-28, less than two-thirds of all reported results were above the LOQs (see calculation criteria in Section 4). For BDE-49, a high variation in the participants' results was observed, resulting in a higher percentage of outliers than the 33% limit. It should be noted that BDE-49 does not have its own internal C13-labeled standard and therefore the values may be more scattered. Nevertheless, the median values (0.002 µg/kg) for both analytes were at the target LOQ, demonstrating that some laboratories are capable of analyzing BDE congeners in this low concentration range in the test sample bentonite (2402BT).

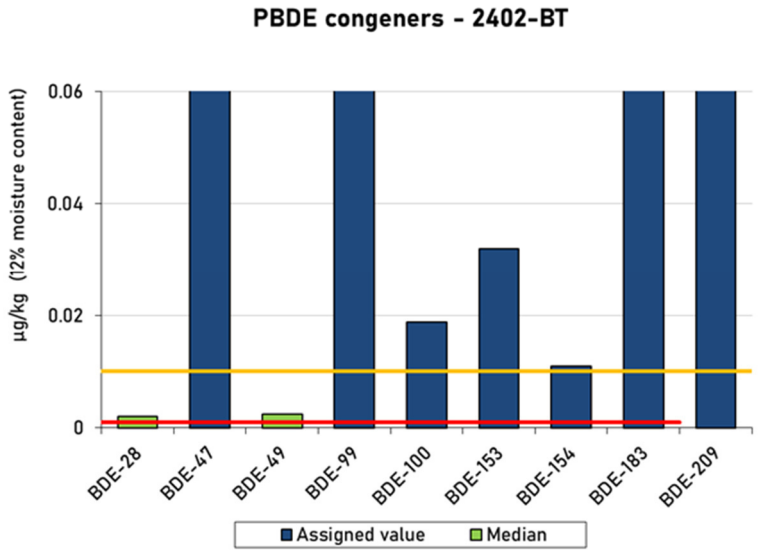

Figure 4: Comparison of assigned values for PBDE congeners with recommended LOQs (yellow line at 0.01 µg/kg product and red line at 0.001 µg/kg product) in Bentonite (2402-BT)

HBCDDs:

For alpha- and gamma-HBCDD no assigned values could be calculated, because less than two-thirds 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 values for alpha-HBCDD (0.03 µg/kg) and for beta-HBCDD (0.02 µg/kg) were both in the range of the recommended LOQ of 0.01 µg/kg product, showing that the not all participating laboratories could reliably report results for alpha- and beta-HBCDDs in Bentonite in this low concentration range.

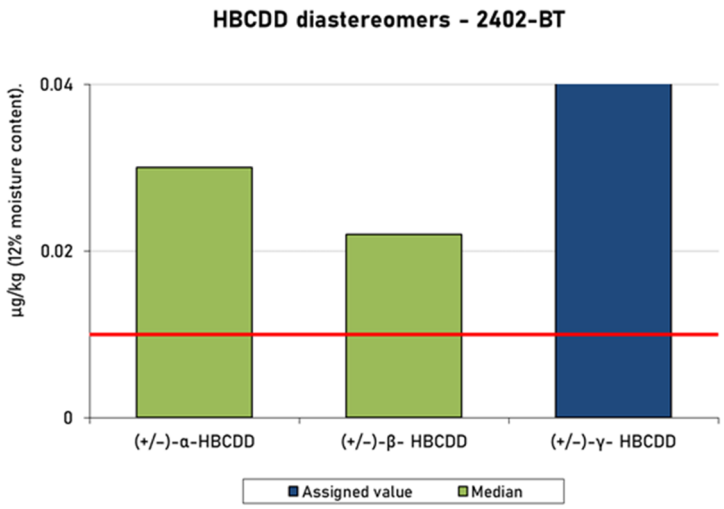

Figure 5: Comparison of assigned values for HBCDD stereoisomers with recommended LOQs (red line at 0.01 µg/kg product) in Bentonite (2402-BT)

5. Scoring of results – Z-scores

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

$$z = \frac{(x - x_a)}{\sigma_{p_{rel}} * x_a}$$

x: *participant's result*

x_a: *assigned value*

$\sigma_{p_{rel}}$: *relative fitness-for-purpose-based "standard deviation for proficiency assessment"*

For individual PBDE congeners, individual HBCDD diastereomers and PBDE and HBCDD sum parameters, the relative standard deviation for proficiency assessment $\sigma_{p_{rel}}$ 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)*

5.1. PBDEs - Participants' z-scores

Table 10: Distribution of participants' z-scores for PBDEs for Bentonite (2402-BT)

Percentage of participants' results	|z-score| ≤ 2	2 < |z-score| < 3	|z-score| ≥ 3
BDE-47	89 %	-	11 %
BDE-99	89 %	-	11 %
BDE-100	88 %	-	12 %
BDE-153	88 %	-	12 %
BDE-154	83 %	-	17 %
BDE-183	92 %	-	8 %
BDE-209	86 %		14 %
Sum of 8 PBDEs without BDE-209 (ub)	78 %	7 %	15 %
Sum of 8 PBDE including BDE-209 (lb)	89 %	-	11 %
Sum of 9 PBDE including BDE-209 (ub)	65 %	-	35 %
Sum of 9 PBDE including BDE-209 (lb)	84 %	-	16 %

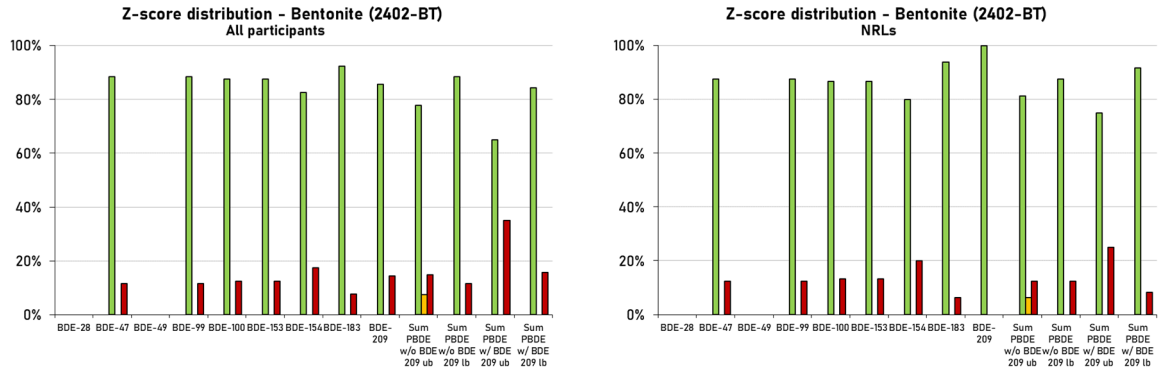

Figure 7: Distribution of participants' z-scores and NRLs only for PBDE congeners / sum parameters for Bentonite (2402-BT) [Green bars: -2 ≤ z-score ≤ 2, orange bars: -3 < z-score < -2, 2 < z-score < 3, red bars: z-score ≤ -3, z-score ≥ 3]

5.2. 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-HBCDD (using LC separation) was taken for comparison.

Table 11: Distribution of participants' z-scores for HBCDD for Bentonite (2402-BT)

Percentage of participants' results	|z-score| ≤ 2	2 < |z-score| < 3	|z-score| ≥ 3
(+/–)-γ -HBCDD	100 %	-	-
Sum of α-, β-, γ-HBCDD (ub)	90 %	-	10 %
Sum of α-, β-, γ-HBCDD (lb)	94 %	-	6 %
Total HBCDD*	100%	-	-

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

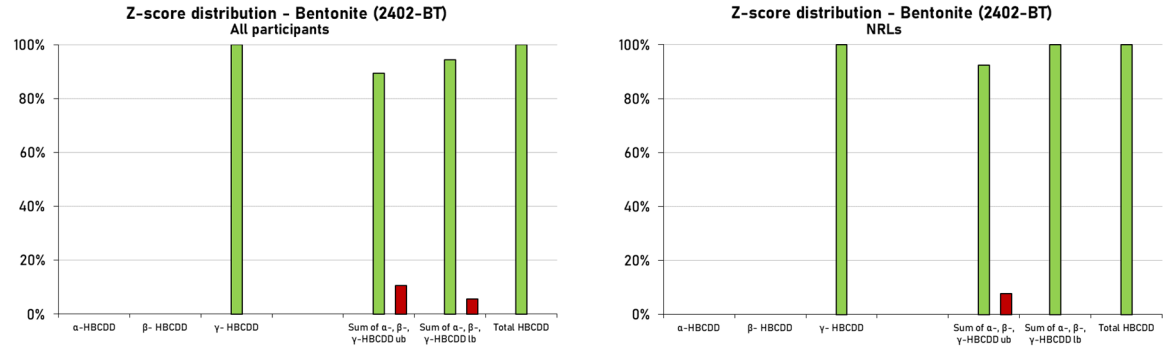

Figure 8: Distribution of participants' z-scores and NRLs only for HBCDD stereoisomers / sum parameters for Bentonite (2402-BT) [Green bars: -2 ≤ z-score ≤ 2, orange bars: -3 < z-score <-2, 2 < z-score < 3, red bars: z-score ≤ -3, z-score ≥ 3]

6. Participants' feedback

A questionnaire for feedback from participants of this EURL proficiency test was available as online survey between 18 November 2024 and 17 January 2025. 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, 3 laboratories replied to this survey.

Table 12: Participating laboratories in the feedback survey

Type of laboratory	Answers
National Reference Laboratory (NRL)	0
Official Laboratory (OFL)	3
Commercial laboratory	0
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".

Announcement 

Instructions 

Sample shipment 

Reporting of results 

Preliminary report 

Did the proficiency test meet expectations? 8 out of 10 rating points

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

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).

All homogeneity and stability testing was performed under accreditation according to DIN EN ISO/IEC 17025:2018.

8. Results of participants

An overview of the PBDE and HBCDD results for the PT test sample Bentonite (2402-BT) 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

Bentonite – 2402-BT	
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	Test for sufficient homogeneity and stability – PBDEs
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 and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food
26 August 2024

Annex 1: Assigned values of PBDEs and HBCDDs

Test sample - Bentonite (2402-BT)

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 and HBCDDs in Bentonite 2024 [EURL-PT-DPB-2402-BT]

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

Bentonite (2402-BT)

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						0.00200
BDE-47	2,2′,4,4′-tetrabromodiphenyl ether		0.0711	0.0078	0.0020	23	0.0730
BDE-49	2,2′,4,5′-tetrabromodiphenyl ether						0.00241
BDE-99	2,2′,4,4′,5-pentabromodiphenyl ether		0.0920	0.013	0.0034	23	0.0951
BDE-100	2,2′,4,4′,6-pentabromodiphenyl ether		0.0188	0.0023	0.00062	21	0.0193
BDE-153	2,2′,4,4′,5,5′-hexabromodiphenyl ether		0.0320	0.003	0.00081	21	0.0329
BDE-154	2,2′,4,4′,5,6′-hexabromodiphenyl ether		0.0109	0.0012	0.00035	19	0.0116
BDE-183	2,2′,3,4,4′,5′,6-heptabromodiphenyl ether		0.154	0.025	0.0065	24	0.155
BDE-209	2,2′,3,3′,4,4′,5,5′,6,6′-decabromodiphenyl ether		0.257	0.057	0.021	12	0.296
Sum of 8 PBDE	without BDE-209 (ub)		0.385	0.041	0.011	22	0.390
Sum of 8 PBDE	without BDE-209 (lb)		0.378	0.036	0.0095	23	0.387
Sum of 9 PBDE	including BDE-209 (ub)		0.611	0.090	0.031	13	0.690
Sum of 9 PBDE	including BDE-209 (lb)		0.566	0.13	0.042	16	0.641

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB-2402-BT]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Bentonite (2402-BT)
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)						0.0300
(+/–)-β- HBCDD	(1,2,5,6,9,10-hexabromo-(1R,2S,5R,6R,9R,10S)-rel-cyclododecane)						0.0222
(+/–)-γ- HBCDD	(1,2,5,6,9,10-hexabromo-(1R,2R,5R,6S,9S,10R)-rel-cyclododecane)		0.907	0.10	0.029	18	0.903
Sum of α-, β-, γ-HBCDD	(ub)		0.988	0.095	0.029	17	1.030
Sum of α-, β-, γ-HBCDD	(lb)		0.939	0.13	0.038	17	0.969
Total HBCDD	(using GC-methods)						0.772

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB-2402-BT]

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

Bentonite (2402-BT)

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		9.26	0.286	0.068	28	9.25

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]

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

26 August 2024

Annex 2: Participants' results of PBDEs and HBCDDs

Test sample - Bentonite (2402-BT)

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB-2402-BT]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Bentonite (2402-BT)
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)
3	2402-BT		0.00755	0.187	0.00473	0.155	0.034	0.0576	0.0223	0.215			0.683	0.683		
4	2402-BT		0.00158	0.0715	0.0019	0.0893	0.02	0.0328	0.0127	0.154			0.384	0.384		
9	2402-BT		0.0052	0.073	< 0.003	0.106	0.019	0.029	0.0099	0.17			0.42	0.41		
20	2402-BT		< 1.3	66.5	1.73	83.7	17.5	23.3	7.79	94.6	< 339		296	295	635	295
27	2402-BT		< 0.02	0.078	< 0.02	0.077	0.017	0.025	< 0.02	0.104			0.36	0.3		
32	2402-BT		< 0.00136	0.0756	0.00155	0.0963	0.0199	0.0344	0.0116	0.142	0.308		0.383	0.382	0.69	0.689
34	2402-BT		0.00125	0.0601	0.00138	0.0825	0.0181	0.0292	0.0101	0.14	0.213		0.342	0.342	0.556	0.556
37	2402-BT		0.002	0.055	0.0015	0.11	0.022	0.035	0.011	0.13	0.2		0.37	0.37	0.57	0.57
45	2402-BT		< 0.00148	0.0684	0.00241	0.0867	0.0118	0.0329	0.00864	0.142	0.157		0.354	0.353	0.511	0.509
46	2402-BT		0.00198	0.0679	0.0024	0.0824	0.018	0.044	0.0177	0.169	0.294		0.403	0.403	0.698	0.698
47	2402-BT															
51	2402-BT		0.00153	0.0615	0.00165	0.0723	0.0155	0.0295	0.0103	0.15			0.342	0.342		
52	2402-BT		0.00158	0.0751	0.00195	0.0865	0.0183	0.0336	0.0118	0.169	0.273		0.397	0.397	0.67	0.67
53	2402-BT		0.0019	0.08	0.0024	0.103	0.023	0.036	0.014	0.18	0.263		0.44	0.44	0.703	0.703
55	2402-BT		0.0013	0.066	0.0017	0.084	0.018	0.029	0.01	0.13	0.35		0.34	0.34	0.57	0.57
58	2402-BT															
59	2402-BT															
64	2402-BT		0.0015	0.0719	0.0016	0.0896	0.0188	0.0326	0.0116	0.162	0.298		0.39	0.39	0.69	0.69
65	2402-BT		0.023	1.03	0.021	0.653	0.161	0.353	0.128	1.25	1.8		3.62	3.62	5.42	5.42
69	2402-BT		< 0.0191	0.0752	< 0.0191	0.108	< 0.0381	< 0.0572	< 0.0572	0.196	< 1.91		0.568	0.377	2.47	0.377
70	2402-BT		0.00108	0.0659	0.00186	0.0951	0.0193	0.0344	0.0105	0.161	0.252		0.39	0.39	0.641	0.641
72	2402-BT		< 100	< 100	< 100	< 100	< 100	< 100	< 100	< 100	< 100		800		900	
78	2402-BT		< 0.01	0.077		0.098	0.021	0.032	0.012	0.178			0.428	0.418		
80	2402-BT		< 0.00103	0.0562	0.00142	0.0767	0.0149	0.0281	0.00961	0.127	0.201		0.315	0.315	0.516	0.516
83	2402-BT		< 0.01	0.071	0.079	0.097	0.021	0.032	0.012	0.151	< 0.01		0.47	0.46	0.48	0.46
84	2402-BT		0.00153	0.0699	0.000438	0.0881	0.019	0.0298	0.0114	0.155	0.267		0.375	0.375	0.642	0.642
85	2402-BT		< 0.0036	0.068	< 0.0031	0.0815	0.017	0.0325	0.0105	0.15	< 0.77		0.37	0.36	1.1	0.36
86	2402-BT		0.0025	0.0745	0.006	0.0861	0.0184	0.0303	0.0095	0.124			0.35	0.35		
94	2402-BT		< 0.0195	0.085	< 0.0195	0.109	< 0.039	< 0.0585	< 0.0585	0.214	< 1.95		0.593	0.398	2.54	0.398
37A	2402-BT		0.0035	0.098	0.0031	0.11	0.021	0.034	0.011	0.13	0.7		0.41	0.41	1.1	1.1

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB-2402-BT]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Bentonite (2402-BT)
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)
3	2402-BT								
4	2402-BT		0.0097	0.0047	0.879		0.894	0.894	
9	2402-BT								
20	2402-BT		0.0128	0.0057	1.03		1.05	1.05	
27	2402-BT		0.01	< 0.01	0.784		0.805	0.795	
32	2402-BT								
34	2402-BT		0.009	0.005	1.04		1.05	1.05	
37	2402-BT								
45	2402-BT		0.109	< 0.05	0.881		1.04	0.99	
46	2402-BT								
47	2402-BT		< 0.007	< 0.006	0.949		0.962	0.949	
51	2402-BT		0.121	0.0222	0.966		1.11	1.11	
52	2402-BT		0.01	< 0.01	0.978		0.998	0.988	
53	2402-BT		< 0.005	< 0.005	0.74		0.741	0.74	
55	2402-BT		< 0.2	< 0.05	0.83		1.08	0.83	
58	2402-BT		< 0.03	< 0.03	1		1.06	1	
59	2402-BT		< 0.039	< 0.039	0.844		0.922	0.844	
64	2402-BT		0.0104	0.00754	1.01		1.03	1.03	
65	2402-BT								
69	2402-BT		< 0.0572	< 0.0572	0.795		0.909	0.795	
70	2402-BT		0.153	0.038	0.903		1.09	1.09	
72	2402-BT		< 100	< 100	< 100		300		< 100
78	2402-BT								
80	2402-BT								
83	2402-BT		0.0122	0.0055	0.917		0.935	0.935	
84	2402-BT								0.772
85	2402-BT		0.15	0.58	0.9		1.63	1.63	
86	2402-BT								
94	2402-BT		< 0.0585	< 0.0585	0.855		0.972	0.855	

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB-2402-BT]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Bentonite (2402-BT)
Moisture content - Results

LC	Sample	Result %	Moisture content	Moisture content	Moisture content
			PBDE	HBCDD	Mean
3	2402-BT		9.2		9.2
4	2402-BT		9.2	9.2	9.2
9	2402-BT		9.5		9.5
20	2402-BT		9.4	9.4	9.4
27	2402-BT		8.6	8.6	8.6
32	2402-BT		7.4		7.4
34	2402-BT		9.0	9.0	9.0
37	2402-BT		9.2		9.2
45	2402-BT		9.2	9.2	9.2
46	2402-BT		9.4		9.4
47	2402-BT			9.4	9.4
51	2402-BT		9.2	9.2	9.2
52	2402-BT		9.4	9.4	9.4
53	2402-BT		9.1	9.1	9.1
55	2402-BT		8.8	8.7	8.8
58	2402-BT				
59	2402-BT			9.2	9.2
64	2402-BT		9.1	9.5	9.3
65	2402-BT		9.4		9.4
69	2402-BT		9.7	9.7	9.7
70	2402-BT		9.4	9.4	9.4
72	2402-BT				
78	2402-BT		7.9		7.9
80	2402-BT		9.0		9.0
83	2402-BT		9.3	9.3	9.3
84	2402-BT		10.3	10.3	10.3
85	2402-BT		9.3	9.3	9.3
86	2402-BT		9.6		9.6
94	2402-BT		9.7	9.7	9.7
37A	2402-BT		9.2		9.2

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]

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

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

Test sample - Bentonite (2402-BT)

Z-scores of sum parameters and individual results

Calculation of z-score on basis of assigned value

$$z = (x - x_a) / x_a * \sigma_{prel}$$

x_a: assigned value

x: participant's result

σ_{prel}: fitness-for-purpose-based relative 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 and HBCDDs in Bentonite 2024 [EURL-PT-DPB-2402-BT]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Bentonite (2402-BT)
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)
3	2402-BT			8.2		3.4	4.0	4.0	5.2	2.0			3.9	4.0		
4	2402-BT			0.0		-0.1	0.3	0.1	0.8	0.0			0.0	0.1		
9	2402-BT			0.1		0.8	0.1	-0.5	-0.5	0.5			0.5	0.4		
20	2402-BT			4671.5		4543.9	4649.3	3635.6	3568.4	3066.4			3839.2	3897.1	5191.4	2601.0
27	2402-BT			0.5		-0.8	-0.5	-1.1		-1.6			-0.3	-1.0		
32	2402-BT			0.3		0.2	0.3	0.4	0.3	-0.4	1.0		0.0	0.1	0.6	1.1
34	2402-BT			-0.8		-0.5	-0.2	-0.4	-0.4	-0.5	-0.9		-0.6	-0.5	-0.5	-0.1
37	2402-BT			-1.1		1.0	0.9	0.5	0.0	-0.8	-1.1		-0.2	-0.1	-0.3	0.0
45	2402-BT			-0.2		-0.3	-1.9	0.1	-1.0	-0.4	-1.9		-0.4	-0.3	-0.8	-0.5
46	2402-BT			-0.2		-0.5	-0.2	1.9	3.1	0.5	0.7		0.2	0.3	0.7	1.2
47	2402-BT															
51	2402-BT			-0.7		-1.1	-0.9	-0.4	-0.3	-0.1			-0.6	-0.5		
52	2402-BT			0.3		-0.3	-0.1	0.3	0.4	0.5	0.3		0.2	0.3	0.5	0.9
53	2402-BT			0.6		0.6	1.1	0.6	1.4	0.8	0.1		0.7	0.8	0.8	1.2
55	2402-BT			-0.4		-0.4	-0.2	-0.5	-0.4	-0.8	1.8		-0.6	-0.5	-0.3	0.0
58	2402-BT															
59	2402-BT															
64	2402-BT			0.1		-0.1	0.0	0.1	0.3	0.3	0.8		0.1	0.2	0.6	1.1
65	2402-BT			67.4		30.5	37.8	50.2	53.7	35.6	30.0		42.0	42.9	39.4	42.9
69	2402-BT			0.3		0.9				1.4			2.4	0.0	15.2	-1.7
70	2402-BT			-0.4		0.2	0.1	0.4	-0.2	0.2	-0.1		0.1	0.2	0.2	0.7
72	2402-BT												10384.6		7360.0	
78	2402-BT			0.4		0.3	0.6	0.0	0.5	0.8			0.6	0.5		
80	2402-BT			-1.0		-0.8	-1.0	-0.6	-0.6	-0.9	-1.1		-0.9	-0.8	-0.8	-0.4
83	2402-BT			0.0		0.3	0.6	0.0	0.5	-0.1			1.1	1.1	-1.1	-0.9
84	2402-BT			-0.1		-0.2	0.1	-0.3	0.2	0.0	0.2		-0.1	0.0	0.3	0.7
85	2402-BT			-0.2		-0.6	-0.5	0.1	-0.2	-0.1			-0.2	-0.2	4.0	-1.8
86	2402-BT			0.2		-0.3	-0.1	-0.3	-0.6	-1.0			-0.5	-0.4		
94	2402-BT			1.0		0.9				1.9			2.7	0.3	15.8	-1.5
37A	2402-BT			1.9		1.0	0.6	0.3	0.0	-0.8	8.6		0.3	0.4	4.0	4.7

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB-2402-BT]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Bentonite (2402-BT)
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)
3	2402-BT								
4	2402-BT				-0.2		-0.5	-0.2	
9	2402-BT								
20	2402-BT				0.7		0.3	0.6	
27	2402-BT				-0.7		-0.9	-0.8	
32	2402-BT								
34	2402-BT				0.7		0.3	0.6	
37	2402-BT								
45	2402-BT				-0.1		0.3	0.3	
46	2402-BT								
47	2402-BT				0.2		-0.1	0.1	
51	2402-BT				0.3		0.6	0.9	
52	2402-BT				0.4		0.1	0.3	
53	2402-BT				-0.9		-1.3	-1.1	
55	2402-BT				-0.4		0.5	-0.6	
58	2402-BT				0.5		0.4	0.3	
59	2402-BT				-0.3		-0.3	-0.5	
64	2402-BT				0.6		0.2	0.5	
65	2402-BT								
69	2402-BT				-0.6		-0.4	-0.8	
70	2402-BT				0.0		0.5	0.8	
72	2402-BT						1513.2		
78	2402-BT								
80	2402-BT								
83	2402-BT				0.1		-0.3	0.0	
84	2402-BT								-0.9
85	2402-BT				0.0		3.2	3.7	
86	2402-BT								
94	2402-BT				-0.3		-0.1	-0.4	

* 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 and HBCDDs in Bentonite 2024 [EURL-PT-DPB-2402-BT]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Bentonite (2402-BT)
Moisture content - Z-scores

LC	Sample	Z-score [σ_p = 10 %]	Moisture content	Moisture content	Moisture content
			PBDE	HBCDD	Mean
3	2402-BT		-0.1		-0.1
4	2402-BT		0.0	0.0	0.0
9	2402-BT		0.3		0.3
20	2402-BT		0.2	0.2	0.2
27	2402-BT		-0.7	-0.7	-0.7
32	2402-BT		-2.1		-2.1
34	2402-BT		-0.3	-0.3	-0.3
37	2402-BT		-0.1		-0.1
45	2402-BT		-0.1	-0.1	-0.1
46	2402-BT		0.2		0.2
47	2402-BT			0.2	0.2
51	2402-BT		0.0	0.0	0.0
52	2402-BT		0.1	0.2	0.1
53	2402-BT		-0.2	-0.2	-0.2
55	2402-BT		-0.5	-0.6	-0.6
58	2402-BT				
59	2402-BT			-0.1	-0.1
64	2402-BT		-0.2	0.2	0.0
65	2402-BT		0.1		0.1
69	2402-BT		0.5	0.5	0.5
70	2402-BT		0.1	0.1	0.1
72	2402-BT				
78	2402-BT		-1.5		-1.5
80	2402-BT		-0.3		-0.3
83	2402-BT		0.0	0.0	0.0
84	2402-BT		1.1	1.1	1.1
85	2402-BT		0.0	0.0	0.0
86	2402-BT		0.4		0.4
94	2402-BT		0.5	0.5	0.5
37A	2402-BT		-0.1		-0.1

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]

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

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

Test sample - Bentonite (2402-BT)

Z-scores of sum parameters and individual results

Calculation of z-score on basis of assigned value

$z = (x - x_a) / xa * \sigma_{prel}$

x_a: assigned value

x: participant's result

σ_{prel}: fitness-for-purpose-based relative standard deviation for proficiency assessment

20%: Evaluated individual PBDE congeners and HBCDD diastereomers and sum

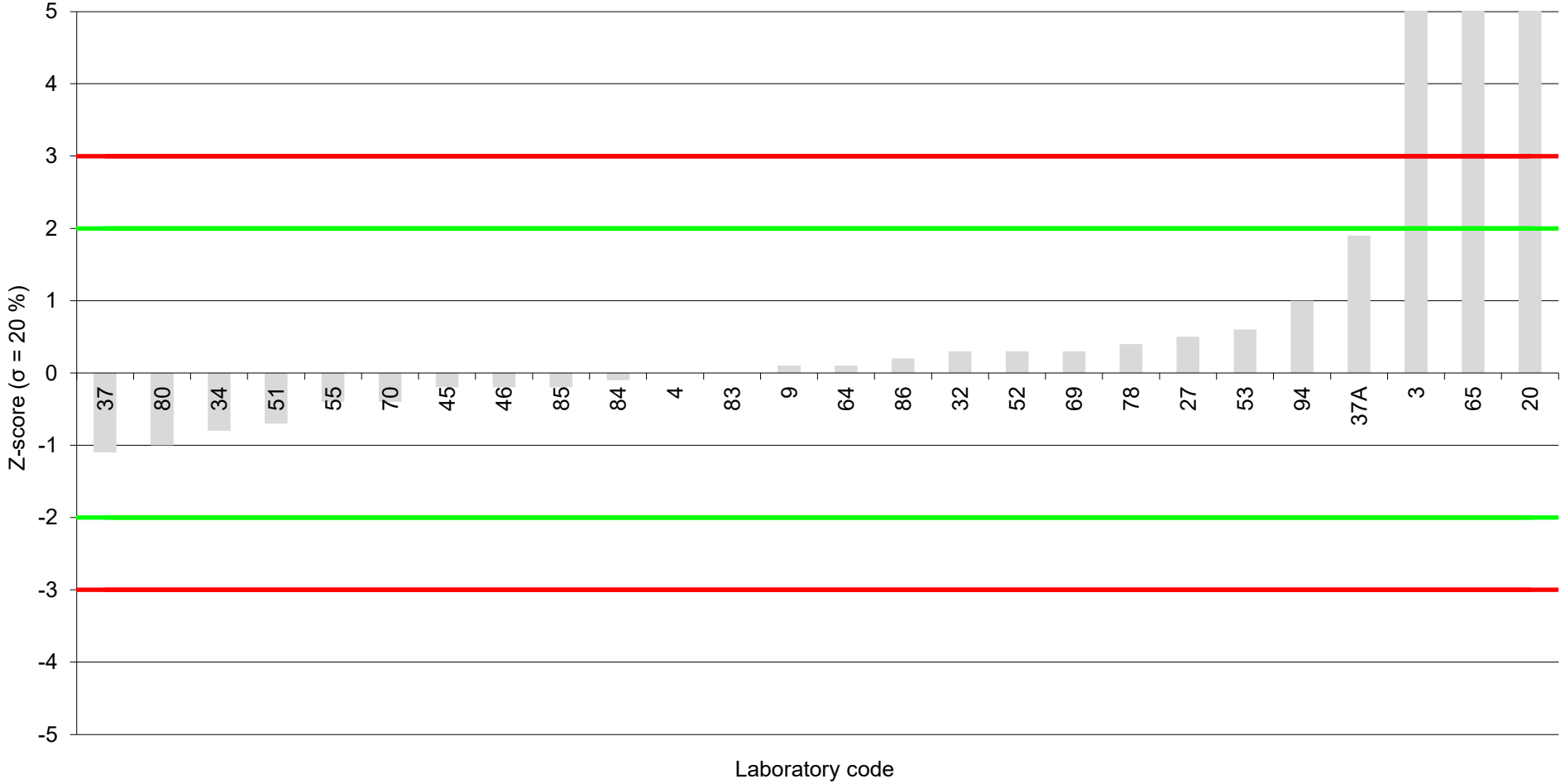
± 2 z-scores:

± 3 z-scores:

Bentonite (2402-BT)

BDE-47

Assigned value: 0.0711 µg/kg product (12 % moisture content)



Bentonite (2402-BT)

BDE-99

Assigned value: 0.092 µg/kg product (12 % moisture content)

Bentonite (2402-BT)

BDE-100

Assigned value: 0.0188 µg/kg product (12 % moisture content)

Bentonite (2402-BT)

BDE-153

Assigned value: 0.032 µg/kg product (12 % moisture content)

Bentonite (2402-BT)

BDE-154

Assigned value: 0.0109 µg/kg product (12 % moisture content)

Bentonite (2402-BT)

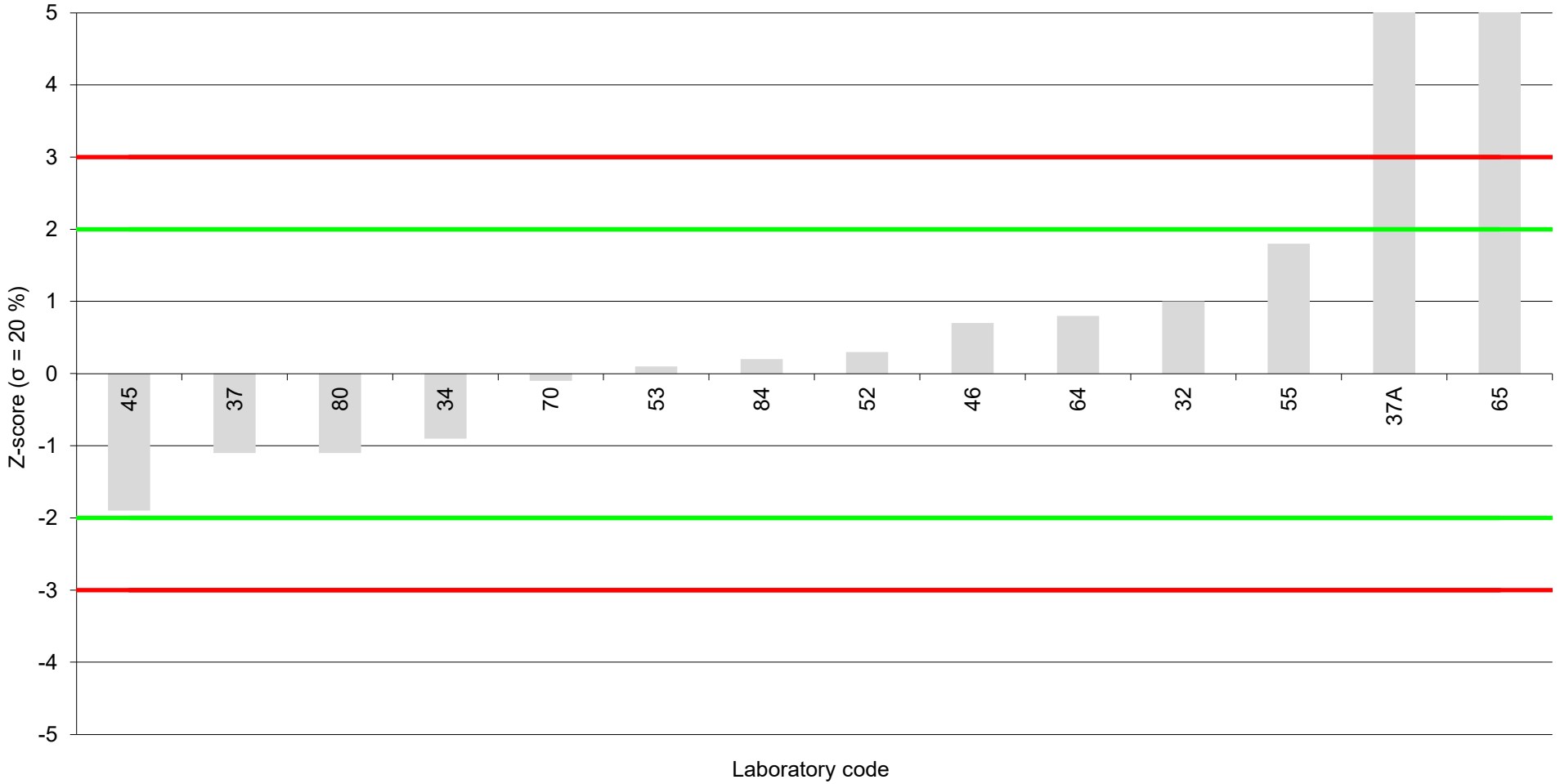
BDE-183

Assigned value: 0.154 µg/kg product (12 % moisture content)

Bentonite (2402-BT)

BDE-209

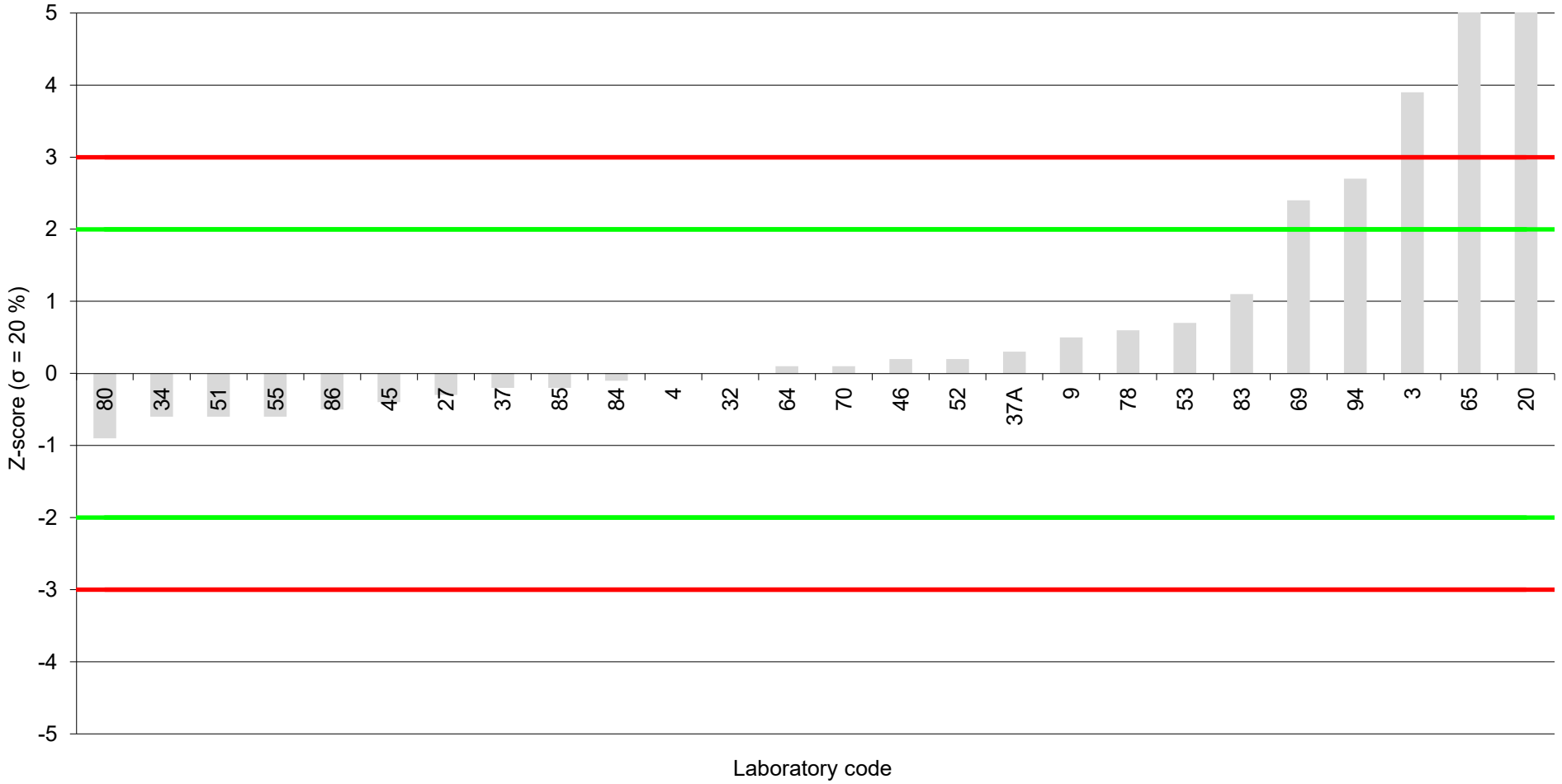
Assigned value: 0.257 µg/kg product (12 % moisture content)



Bentonite (2402-BT)

Sum of PBDE without BDE-209 ub

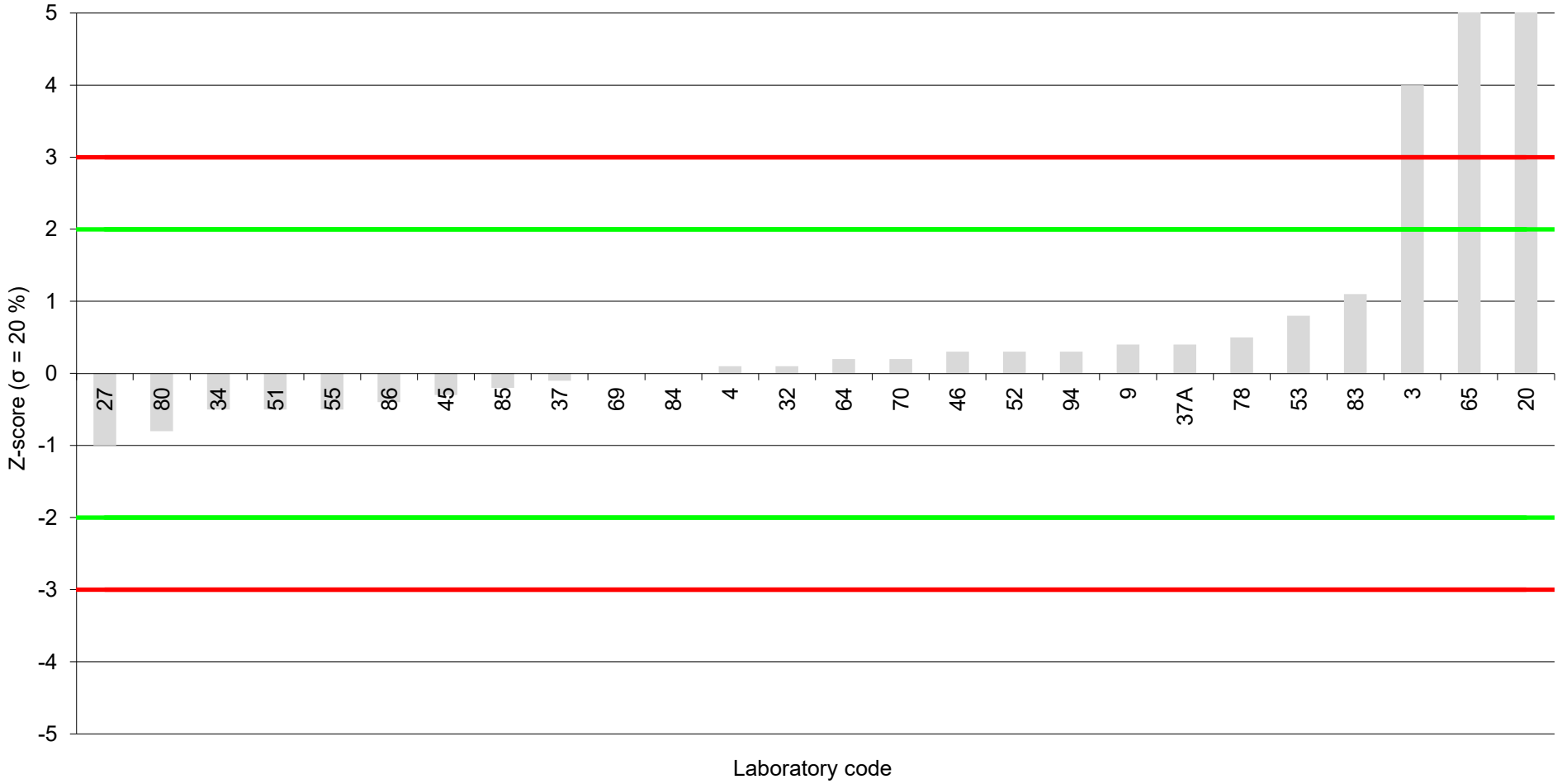
Assigned value: 0.385 µg/kg product (12 % moisture content)



Bentonite (2402-BT)

Sum of PBDE without BDE-209 lb

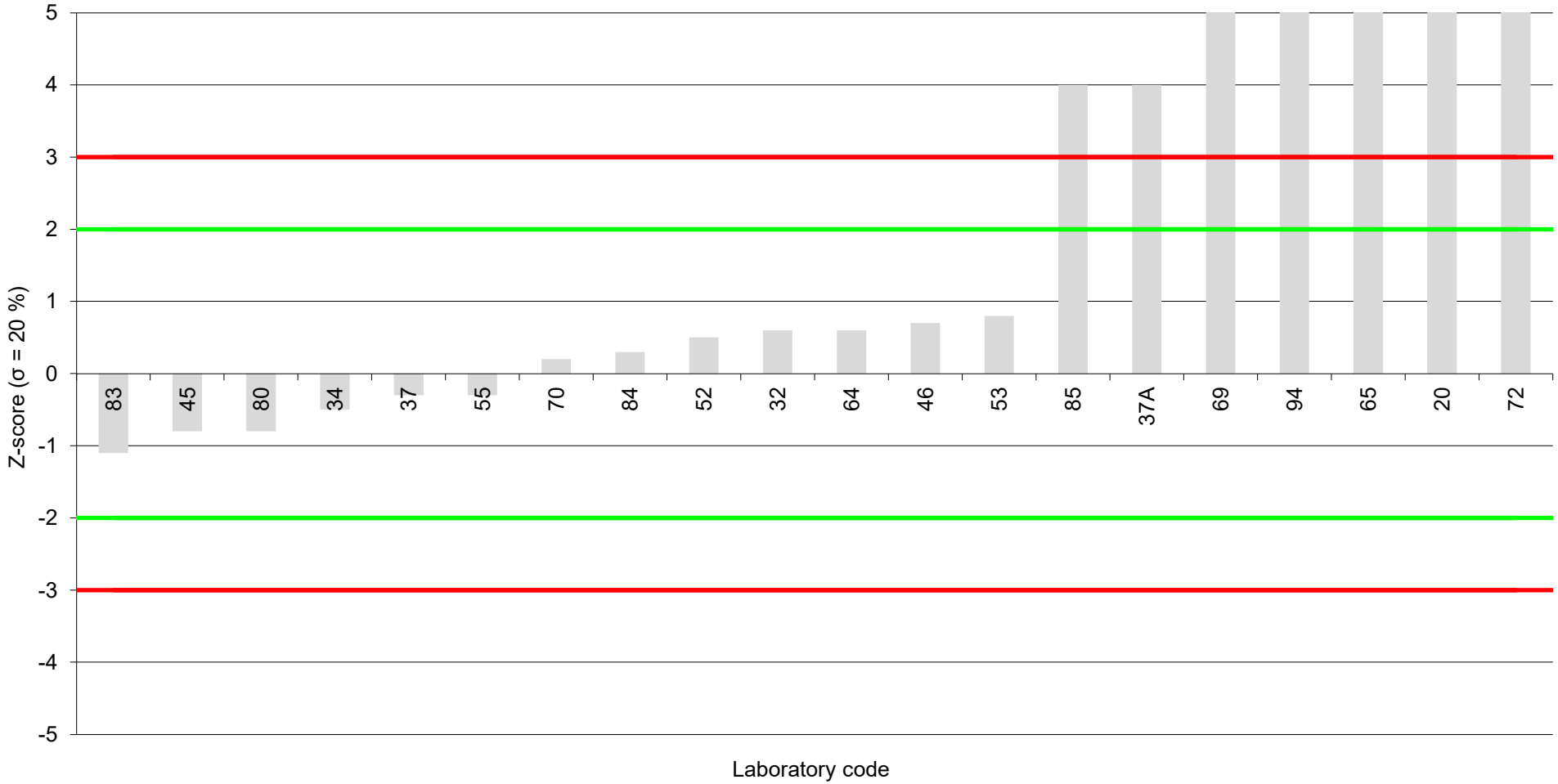
Assigned value: 0.378 µg/kg product (12 % moisture content)



Bentonite (2402-BT)

Sum of PBDE including BDE-209 ub

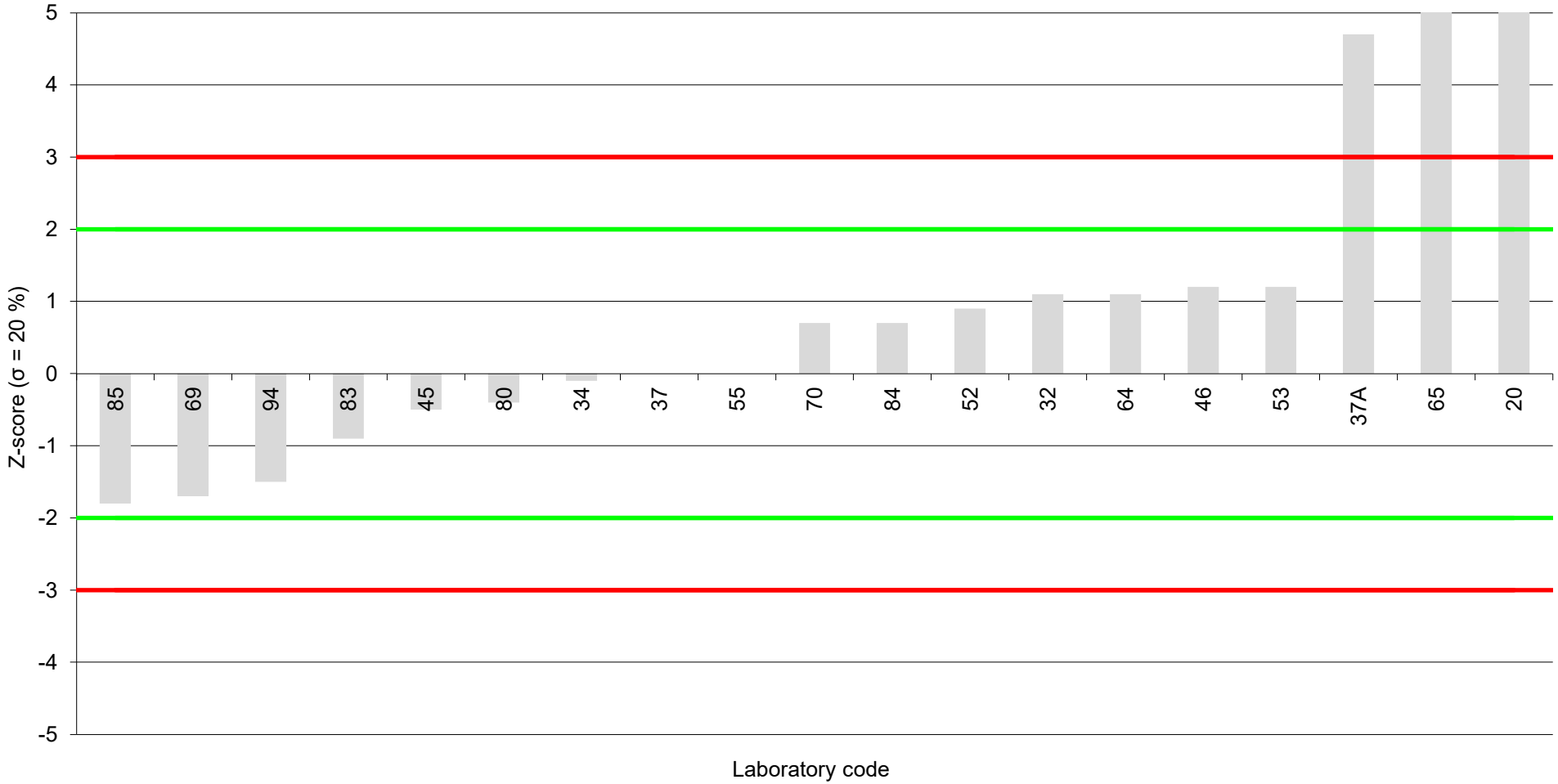
Assigned value: 0.611 µg/kg product (12 % moisture content)



Bentonite (2402-BT)

Sum of PBDE including BDE-209 lb

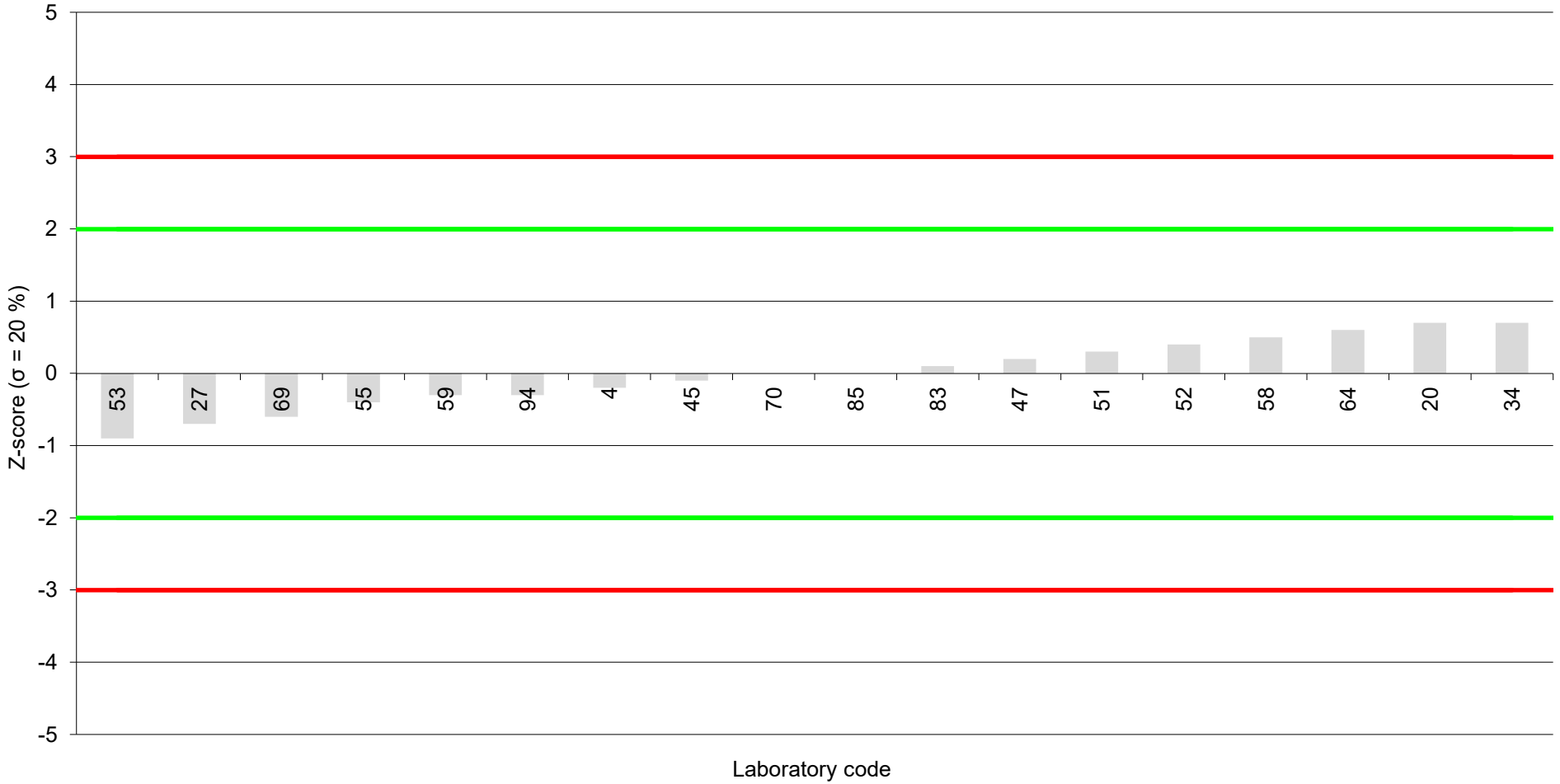
Assigned value: 0.566 µg/kg product (12 % moisture content)



Bentonite (2402-BT)

(+/–)-γ- HBCDD

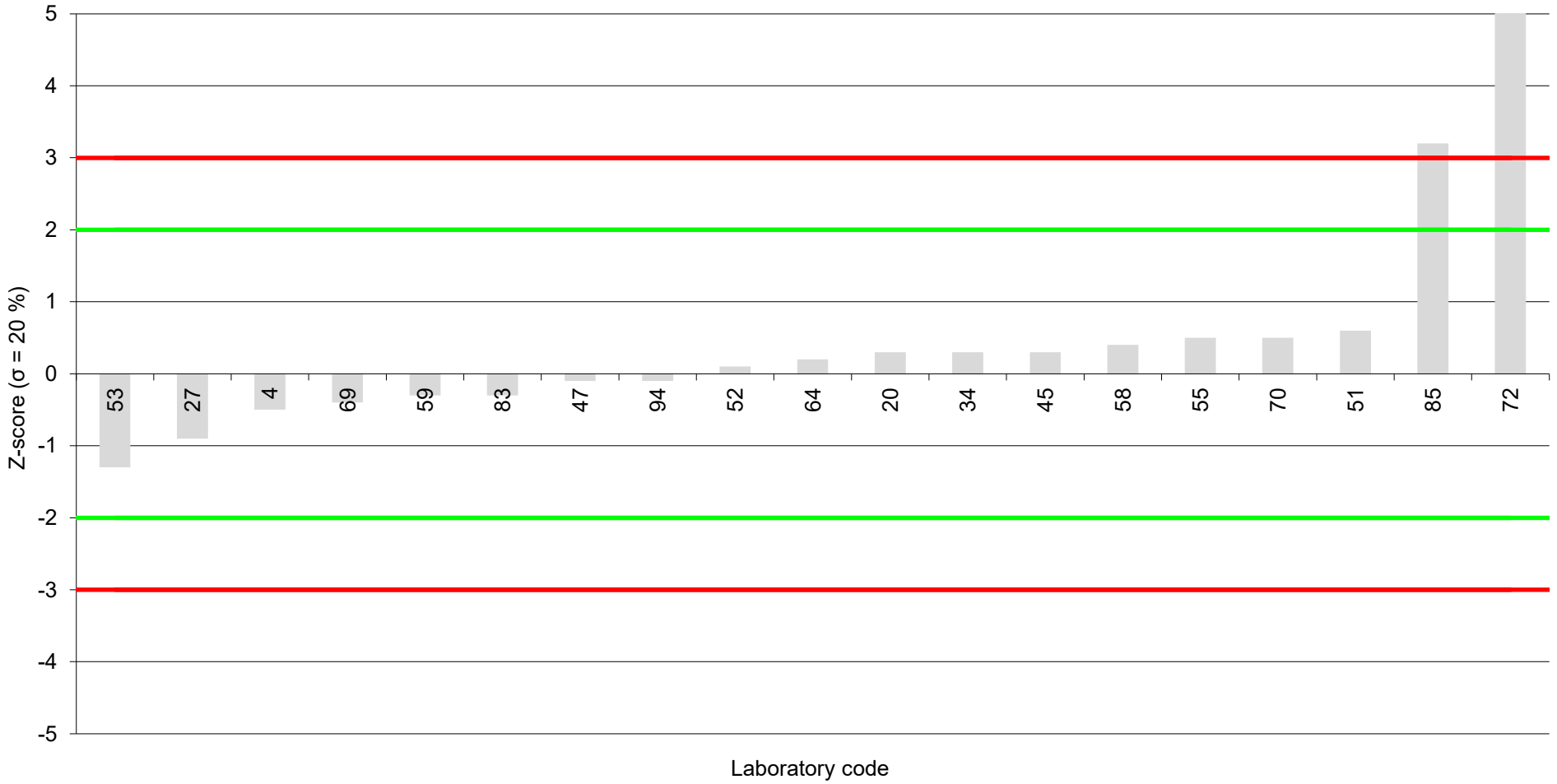
Assigned value: 0.907 µg/kg product (12 % moisture content)



Bentonite (2402-BT)

Sum of α-, β-, γ-HBCDD ub

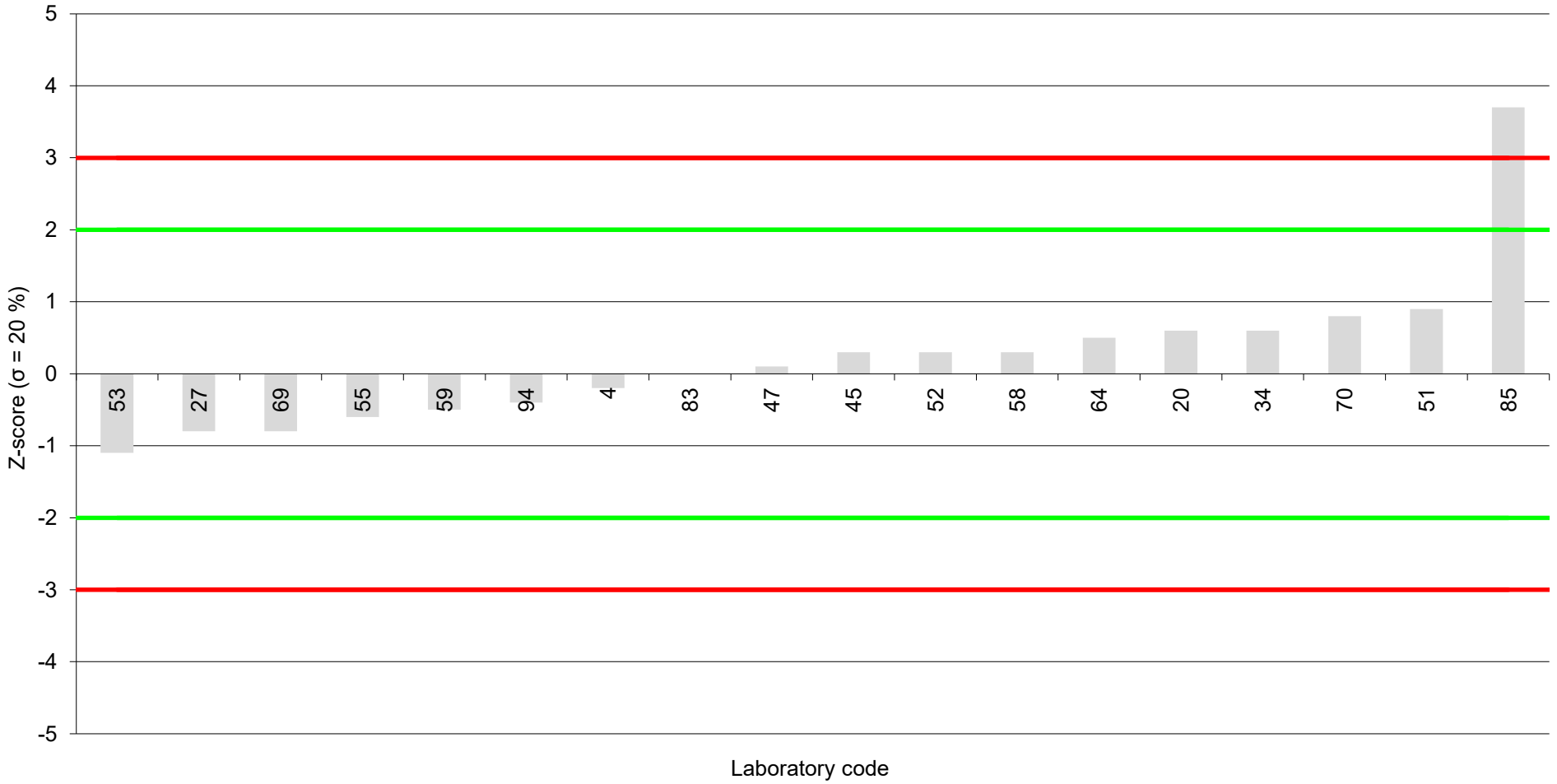
Assigned value: 0.988 µg/kg product (12 % moisture content)



Bentonite (2402-BT)

Sum of α-, β-, γ-HBCDD lb

Assigned value: 0.939 µg/kg product (12 % moisture content)



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Annex 5: Test for sufficient homogeneity and stability for PBDEs

Test sample - Bentonite (2402-BT)

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Bentonite (2402-BT)
PBDE - Homogeneity test - Data

Analyte	Result µg/kg 12% moisture content	**Mean** (n = 10, duplicate analysis)	**Median** (n = 10, duplicate analysis)	**Relative standard deviation** [%]
Sum of 8 PBDE - without BDE-209 (lb)		0.342	0.342	3%
Sum of 8 PBDE - without BDE-209 (ub)		0.342	0.342	3%
Sum of 9 PBDE - including BDE-209 (lb)		0.633	0.610	7%
Sum of 9 PBDE - including BDE-209 (ub)		0.633	0.610	7%
BDE-28		0.00158	0.00172	18%
BDE-47		0.0640	0.0643	5%
BDE-49		0.00174	0.00170	13%
BDE-99		0.0833	0.0856	12%
BDE-100		0.0187	0.0187	6%
BDE-153		0.0281	0.0281	5%
BDE-154		0.0108	0.0106	10%
BDE-183		0.131	0.130	4%
BDE-209		0.290	0.272	15%

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Bentonite (2402-BT)
Selected PBDE congeners - Homogeneity test - Data

Sample	Replicate	Result µg/kg 12% moisture content	**BDE-99**
8	1		0.0884
	2		0.0876
38	1		0.0856
	2		0.0644
51	1		0.0943
	2		0.0896
58	1		0.0677
	2		0.0877
80	1		0.0841
	2		0.0632
82	1		0.0927
	2		0.0682
105	1		0.0842
	2		0.0901
144	1		0.0795
	2		0.0947
195	1		0.0806
	2		0.0856
204	1		0.0848
	2		0.0924
Cochran's C-test			
C			0.265
$C_{critical}$ (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 $\bar{x}$			0.0833
Standard deviation of sample averages s_x			0.0063
Wthin-sample standard deviation s_w			0.0106
Between-sample standard deviation s_s			0
Standard deviation for proficiency assessment σ_{PT}			0.0167
s_s / σ_{PT}			0
Test for homogeneity ($s_s \leq 0.3\ \sigma_{PT}$)			passed

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Bentonite (2402-BT)
Selected congeners - Stability test - Data

Sample	Replicate	Result µg/kg (12% moisture content)	**BDE-99**
47	1		0.0810
	2		0.0893
51	1		0.0880
	2		0.0873
61	1		0.0823
	2		0.0955
Stability test			
General average (stability test) $\bar{y}$			0.0872
General average (homogeneity test) $\bar{x}$			0.0833
Standard deviation for proficiency assessment σPT			0.0167
$\lvert \bar{y} - \bar{x} \rvert$			0.00395
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 and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]
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Annex 6: Overview participants' methods – Weighed sample, internal and recovery standards and comments

Test sample - Bentonite (2402-BT)

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]

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

Bentonite (2402-BT)

Physico-chemical Methods PBDEs - Internal standards

LC	Sample	Weighed sample [g]	Use of isotope-labelled internal standards for … PBDE congeners (yes/no)
3	2402-BT	3.01	Yes
4	2402-BT	20	yes
9	2402-BT	5.2	YES (15)
20	2402-BT	10.0	yes
27	2402-BT	4	yes
32	2402-BT	35.0	yes
34	2402-BT	5.0	YES
37	2402-BT	5.0	yes
45	2402-BT	10	yes
46	2402-BT	5.03	YES
47	2402-BT		
51	2402-BT	10.0	yes
52	2402-BT	10	yes
53	2402-BT	10	yes
55	2402-BT	15.0	yes
58	2402-BT		
59	2402-BT		
64	2402-BT	10.34	yes
65	2402-BT	15.0	yes
69	2402-BT	0.5	yes
70	2402-BT	19.9576	yes
72	2402-BT	5	no
78	2402-BT	5.0	yes
80	2402-BT	10.0	yes
83	2402-BT	10	yes
84	2402-BT	20	yes
85	2402-BT	5.0	yes
86	2402-BT	31.6	yes (except for BDE-49)
94	2402-BT	0.5	yes
37A	2402-BT	5	yes

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]

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

Bentonite (2402-BT)

Methods PBDEs - Internal Standards

LC	Sample	BDE 28	BDE 47	BDE 49	BDE 99	BDE 100	BDE 153	BDE 154	BDE 183	BDE 209
3	2402-BT	13C-BDE-28	13C-BDE-47	13C-BDE-47	13C-BDE-99	13C-BDE-99	13C-BDE-153	13C-BDE-154	13C-BDE-183	
4	2402-BT	13C12-PBDE 28	13C12-PBDE 47	13C12-PBDE 47	13C12-PBDE 99	13C12-PBDE 100	13C12-PBDE 153	13C12-PBDE 154	13C12-PBDE 183	-
9	2402-BT	2,4,4'-Tribromo[13C12]diphenyl ether	2,2',4,4'-Tetrabromo[13C12]diphenyl ether	2,2',4,4'-Tetrabromo[13C12]diphenyl ether	2,2',4,4',5-Pentabromo[13C12]diphenyl ether	2,2',4,4',6-Pentabromo[13C12]diphenyl ether	2,2',4,4',5,5'-Hexabromo[13C12]biphenyl	2,2',4,4',5,6'-Hexabromo[13C12]diphenyl ether	2,2',3,4,4',5',6-Heptabromo[13C12]cliphenyl ether	
20	2402-BT	13C-BDE28	13C-BDE47	13C-BDE47	13C-BDE99	13C-BDE100	13C-BDE153	13C-BDE154	13C-BDE183	13C-BDE209
27	2402-BT	BDE-28-13C12	BDE-47-13C12	BDE-47-13C12	BDE-99-13C12	BDE-100-13C12	BDE-153-13C12	BDE-154-13C12	BDE-183-13C12	
32	2402-BT	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
34	2402-BT	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
37	2402-BT	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
45	2402-BT									
46	2402-BT	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
47	2402-BT									
51	2402-BT	BDE-28	BDE-47	BDE-47	BDE-99	BDE-100	BDE-153	BDE-154	BDE-183	
52	2402-BT	MBDE-28	MBDE-47	MBDE-47	MBDE-99	MBDE-100	MBDE-153	MBDE-154	MBDE-183	MBDE-209
53	2402-BT	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
55	2402-BT	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
58	2402-BT									
59	2402-BT									
64	2402-BT	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
65	2402-BT	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
69	2402-BT	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
70	2402-BT	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
72	2402-BT	PCB 209	PCB 209	PCB 209	PCB 209	PCB 209	PCB 209	PCB 209	PCB 209	PCB 209
78	2402-BT	2,2',4-TriBDE (13C12)	2,2',4,4'-TetraBDE (13C12)		2,2',4,4',5-PentaBDE (13C12)	2,2',4,4',6-PentaBDE (13C12)	2,2',4,4',5,5'-HexaBDE (13C12)	2,2',4,4',5',6-HexaBDE (13C12)	2,2',3,4,4',5',6-HeptaBDE (13C12)	
80	2402-BT	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
83	2402-BT	c13 bde-28	c13 bde-47	c13 bde-47	c13 bde-99	c13 bde-100	c13 bde-153	c13 bde-154	c13 bde-183	c13 bde-209
84	2402-BT	BDE-28	BDE-47	BDE-47	BDE-99	BDE-100	BDE-153	BDE-154	BDE-183	BDE-209
85	2402-BT	13C-BDE28	13C-BDE47	13C-BDE47	13C-BDE99	13C-BDE100	13C-BDE153	13C-BDE154	13C-BDE183	13C-BDE209
86	2402-BT	BDE-28	BDE-47	BDE-47	BDE-99	BDE-100	BDE-153	BDE-154	BDE-183	
94	2402-BT	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
37A	2402-BT	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

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

Bentonite (2402-BT)

Methods PBDEs - Recovery Standards

LC	Sample		BDE 28	BDE 47	BDE 49	BDE 99	BDE 100	BDE 153	BDE 154	BDE 183	BDE 209
3	2402-BT		13C-PCB-80	13C-PCB-80	13C-PCB-80	13C-PCB-80	13C-PCB-80	13C-PCB-80	13C-PCB-80	13C-PCB-80	
4	2402-BT		13C12-PCB 52	13C12-PCB 52	13C12-PCB 52	13C12-PCB 138	13C12-PCB 138	13C12-PCB 138	13C12-PCB 138	13C12-PCB 138	-
9	2402-BT		3,3',4,5'-Tetrabromo[13C12]diphenyl ether	3,3',4,5'-Tetrabromo[13C12]diphenyl ether	3,3',4,5'-Tetrabromo[13C12]diphenyl ether	3,3',4,5'-Tetrabromo[13C12]diphenyl ether	3,3',4,5'-Tetrabromo[13C12]diphenyl ether	2,2',3,4,4',6-Hexabromo[13C12]diphenyl ether	2,2',3,4,4',6-Hexabromo[13C12]diphenyl ether	2,2',3,4,4',5,5'-Heptabromo[13C12]diphenyl ether	
20	2402-BT		13C-BDE126	13C-BDE126	13C-BDE126	13C-BDE126	13C-BDE126	13C-BDE126	13C-BDE126	13C-BDE126	13C-BDE126
27	2402-BT		BDE-77-13C12	BDE-77-13C12	BDE-77-13C12	BDE-77-13C12	BDE-77-13C12	BDE-138-13C12	BDE-138-13C12	BDE-138-13C12	
32	2402-BT		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
34	2402-BT		13C BDE-77	13C BDE-77	13C BDE-77	13C BDE-77	13C BDE-77	13C BDE-138	13C BDE-138	13C BDE-138	
37	2402-BT		13C12-BDE-118	13C12-BDE-118	13C12-BDE-118	13C12-BDE-118	13C12-BDE-118	13C12-BDE-118	13C12-BDE-118	13C12-BDE-118	13C12-BDE-118
45	2402-BT										
46	2402-BT		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
47	2402-BT										
51	2402-BT		BDE-79	BDE-79	BDE-79	BDE-79	BDE-79	BDE-138	BDE-138	BDE-138	
52	2402-BT		MBDE-79	MBDE-79	MBDE-79	MBDE-139	MBDE-139	MBDE-139	MBDE-139	MBDE-139	MBDE-206
53	2402-BT		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 209
55	2402-BT		13C-BDE-77	13C-BDE-77	13C-BDE-77	13C-BDE-77	13C-BDE-77	13C-BDE-139	13C-BDE-139	13C-BDE-180	13C-BDE-206
58	2402-BT										
59	2402-BT										
64	2402-BT		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
65	2402-BT		13C6-TCDD	13C6-TCDD	13C6-TCDD	13C6-TCDD	13C6-TCDD	13C6-TCDD	13C6-TCDD	13C6-TCDD	13C6-TCDD
69	2402-BT		13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138
70	2402-BT		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
72	2402-BT										
78	2402-BT										
80	2402-BT		13C12 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
83	2402-BT		yes	yes	yes	yes	yes	yes	yes	yes	yes
84	2402-BT		PCB-138	PCB-138	PCB-138	PCB-138	PCB-138	PCB-138	PCB-138	PCB-138	PCB-138
85	2402-BT		13C-BDE118	13C-BDE118	13C-BDE118	13C-BDE118	13C-BDE118	13C-BDE118	13C-BDE118	13C-BDE118	13C-BDE208
86	2402-BT		BDE-77	BDE-77	BDE-77	BDE-77	BDE-77	BDE-138	BDE-138	BDE-138	
94	2402-BT		13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138
37A	2402-BT		13C12-BDE-118	13C12-BDE-118	13C12-BDE-118	13C12-BDE-118	13C12-BDE-118	13C12-BDE-118	13C12-BDE-118	13C12-BDE-118	13C12-BDE-118

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]

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

Bentonite (2402-BT)

Methods PBDEs - Comments

LC	Sample	BDE 28	BDE 47	BDE 49	BDE 99	BDE 100	BDE 153	BDE 154	BDE 183	BDE 209
3	2402-BT									
4	2402-BT									not analysed
9	2402-BT									Not Determined
20	2402-BT									
27	2402-BT									BDE 209 WAS NOT REPORTED BECAUSE THE SAMPLE WAS STRONGLY INTERFERED
32	2402-BT									
34	2402-BT									
37	2402-BT									
45	2402-BT									
46	2402-BT									
47	2402-BT									
51	2402-BT									
52	2402-BT									
53	2402-BT									
55	2402-BT									
58	2402-BT									
59	2402-BT									
64	2402-BT									
65	2402-BT									
69	2402-BT									
70	2402-BT									
72	2402-BT									
78	2402-BT			not determined						not determined
80	2402-BT									
83	2402-BT									
84	2402-BT									
85	2402-BT									
86	2402-BT									
94	2402-BT									
37A	2402-BT									

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]

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

Bentonite (2402-BT)

Physico-chemical Methods HBCDDs - Internal standards

LC	Sample		**Weighed sample** [g]		**Use of isotope-labelled internal standards for …** HBCDD diastereomers (yes/no)
3	2402-BT				
4	2402-BT		5		yes
9	2402-BT				
20	2402-BT		10		yes
27	2402-BT		4		yes
32	2402-BT				
34	2402-BT		5		YES
37	2402-BT				
45	2402-BT		5		yes
46	2402-BT				
47	2402-BT		10		yes
51	2402-BT		10		yes
52	2402-BT		10		yes
53	2402-BT		10		yes
55	2402-BT		10		yes
58	2402-BT		10		Yes
59	2402-BT		5		yes
64	2402-BT		10		yes
65	2402-BT				
69	2402-BT		0.5		yes
70	2402-BT		19.9576		yes
72	2402-BT		5		no
78	2402-BT				
80	2402-BT				
83	2402-BT		10		yes
84	2402-BT		20		yes
85	2402-BT		5		yes
86	2402-BT				
94	2402-BT		0.5		yes

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]

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

Bentonite (2402-BT)

Methods HBCDDs - Internal Standards

LC	Sample		(+/–)-α-HBCDD	(+/–)-β- HBCDD	(+/–)-γ- HBCDD
4	2402-BT		alfa-HBCDD (13C12)	beta-HBCDD (13C12)	gamma-HBCDD (13C12)
9	2402-BT				
20	2402-BT		13C-a-HBCD	13C-B-HBCD	13C-j-HBCD
27	2402-BT		alfa-HBCD-13C12	gamma-HBCD-13C12	gamma-HBCD-13C12
32	2402-BT				
34	2402-BT		13C-α-HBCDD	13C-β-HBCDD	13C-γ-HBCDD
37	2402-BT				
45	2402-BT				
46	2402-BT				
47	2402-BT		13C12-a-HBCDD	13C12-a-HBCDD	13C12-a-HBCDD
51	2402-BT		alpha-HBCDD	beta-HBCDD	gamma-HBCDD
52	2402-BT		13C12-alpha-HBCDD	13C12-beta-HBCDD	13C12-gamma-HBCDD
53	2402-BT		13C-(+/–)-α-HBCDD	13C-(+/–)-β- HBCDD	13C-(+/–)-γ- HBCDD
55	2402-BT		13C-alpha-HBCDD	13C-beta-HBCDD	13C-gamma-HBCDD
58	2402-BT		13C-A-HBCD	13C-B-HBCD	13C-Y-HBCD
59	2402-BT		alpha- HBCDD (13C)	beta- HBCDD (13C)	gamma- HBCDD (13C)
64	2402-BT		13C alphaHBCDD	13C betaHBCDD	13C gammaHBCDD
65	2402-BT				
69	2402-BT		13C12-α-HBCD	13C12-β-HBCD	13C12-γ-HBCD
70	2402-BT		13C-α-HBCD	13C-β-HBCD	13C-γ-HBCD
72	2402-BT		PCB 209	PCB 209	PCB 209
78	2402-BT				
80	2402-BT				
83	2402-BT		yes	yes	yes
84	2402-BT				
85	2402-BT		13C-α-HBCDD	13C-β-HBCDD	13C-γ-HBCDD
86	2402-BT				
94	2402-BT		13C12-α-HBCD	13C12-β-HBCD	13C12-γ-HBCD

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]

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

Bentonite (2402-BT)

Methods HBCDDs - Recovery Standards

LC	Sample		(+/–)-α-HBCDD	(+/–)-β- HBCDD	(+/–)-γ- HBCDD
4	2402-BT				
9	2402-BT				
20	2402-BT		none	none	none
27	2402-BT		beta-HBCD-13C12	beta-HBCD-13C12	beta-HBCD-13C12
32	2402-BT				
34	2402-BT		d18-β-HBCDD	d18-β-HBCDD	d18-β-HBCDD
37	2402-BT				
45	2402-BT				
46	2402-BT				
47	2402-BT				
51	2402-BT		D18-beta-HBCDD	D18-beta-HBCDD	D18-beta-HBCDD
52	2402-BT		alpha-HBCDD-d18	beta-HBCDD-d18	beta-HBCDD-d18
53	2402-BT				
55	2402-BT		-	-	-
58	2402-BT		D18-B-HBCD	D18-B-HBCD	D18-B-HBCD
59	2402-BT		D- beta-HBCDD	D- beta-HBCDD	D- beta-HBCDD
64	2402-BT		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
65	2402-BT				
69	2402-BT		d18-β-HBCD	d18-β-HBCD	d18-β-HBCD
70	2402-BT		13C-δ-HBCD	13C-δ-HBCD	13C-δ-HBCD
72	2402-BT				
78	2402-BT				
80	2402-BT				
83	2402-BT		yes	yes	yes
84	2402-BT				
85	2402-BT		2H-α-HBCDD	2H-β-HBCDD	2H-γ-HBCDD
86	2402-BT				
94	2402-BT		d18-β-HBCD	d18-β-HBCD	d18-β-HBCD

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]

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

Bentonite (2402-BT)

Methods HBCDDs - Comments

LC	Sample		(+/–)-α-HBCDD	(+/–)-β- HBCDD	(+/–)-γ- HBCDD
4	2402-BT		commonly used LOQ is 0.05, but in this case we tried to headed to lower LOQ	commonly used LOQ is 0.05, but in this case we tried to headed to lower LOQ	
9	2402-BT				
20	2402-BT				
27	2402-BT				
32	2402-BT				
34	2402-BT				
37	2402-BT				
45	2402-BT				
46	2402-BT				
47	2402-BT				
51	2402-BT				
52	2402-BT				
53	2402-BT				
55	2402-BT				
58	2402-BT				
59	2402-BT				
64	2402-BT				
65	2402-BT				
69	2402-BT				
70	2402-BT				
72	2402-BT				
78	2402-BT		not determined	not determined	not determined
80	2402-BT				
83	2402-BT				
84	2402-BT				
85	2402-BT				
86	2402-BT				
94	2402-BT				

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food
26 August 2024

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

Test sample - Bentonite (2402-BT)

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Bentonite (2402-BT)
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]
3	2402-BT	No	Soxhlet	Toluene:Ethanol 70:30	18	Boiling point	
4	2402-BT	thorough homogenization	Soxhlet (ultrasonic bath, agitate)	toluene:methanol 2:1	21	109	0.1
9	2402-BT	No sample pre-treatment, used directly from container supplied.	ASE	Toluene	40 minutes	150	11.7
20	2402-BT		LL Extraction/Adding silica/H2SO4 40% and then extraction with 10 ml hexane (2x). Evaporated to dryness.	Ethylacetate/Hexane	over night	Roomtemp	
27	2402-BT	no	QuEChERs like-extraction	Ethyl acetate	no	no	no
32	2402-BT		Speed-Extractor	Toluene / Ethanol (30/70)	1h30	150	10
34	2402-BT	drying	PLE	Toluene/Acetone			
37	2402-BT	no	Soxhlet	Toluene			
45	2402-BT	105 C	Soxhlet	Toluene	24h	reflux	ambient
46	2402-BT	NO	SOXHLET	TOLUENE/ETHANOL (3/7)	24H		
47	2402-BT						
51	2402-BT	Mixed with sodium sulphate	ASE	Toluene/Acetone (70:30)	1	125	10.3
52	2402-BT		ASE	1.Toluol; 2. Toluol/EtOH (9/1)	1 h	100 °C	10
53	2402-BT		Soxhlet	toluene/acetone 7/3			
55	2402-BT	already dried material, combined with small amount of hydromatrix during extraction	Twisselmann	ethanol/toluene (70/30, v/v)	6	boiling point	
58	2402-BT						
59	2402-BT						
64	2402-BT	no	ASE	toluene:acetone (2:1)	4 x 5 min static time	100	100
65	2402-BT	no	Soxtherm	Ethanol / Toluene 70:30	1	80	
69	2402-BT	drying	Soxhlet	Toluol	8 h		
70	2402-BT	Soxhlet	Toluene /ethanol 50 / 50	Step 1: toluene /ethanol 50 / 50 - step 2: toluene	24 for each step (48 in total)	120	atm
72	2402-BT	the weighed sample is mixed with Na2SO4 for drying	ultrasonic bath	Ethylacetate / Acetone 1:1	1h	60 °C	environmental preassure
78	2402-BT	homogenisation	Soxhlet	toluene	6		
80	2402-BT	-	ASE	(1) Toluene - (2) Toluene:Ethanol 90:10	0.25	100°C	10.3
83	2402-BT	no	Soxhlet	EtOH/Toluene 2/1	8		
84	2402-BT	homogenisation, drying (only for determination of moisture content)	ASE, Soxhlet	toluene, ethanol (ASE), toluene (Soxhlet)	30 min/sample (ASE), 16 h (Soxhlet)	100 °C (ASE)	10 MPa (ASE)
85	2402-BT		soxhlet	toluene/ethanol 70/30	12	bp	ambient
86	2402-BT	sample mixed with sodium sulphate before extraction	Soxhlet	Ethanol:Toluene (70:30)	24 h		
94	2402-BT	drying	Soxhlet	Toluol	8 h		
37A	2402-BT	no	Soxhlet	Toluene			

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]

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

Bentonite (2402-BT)

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
3	2402-BT	No	Yes	No	Yes	No		50
4	2402-BT	no	yes	no	yes	yes	basic silica, silver nitrate silica	50
9	2402-BT	No	Yes	No	Yes	Yes	Sample extract partitioned with concentrated sulfuric acid to remove bulk organic material residues	30
20	2402-BT	no	yes	no	no	no	no	250 µl
27	2402-BT	yes	yes	no	no	no	no	250
32	2402-BT	no	yes	no	yes	no	acidic treatment	50
34	2402-BT	YES	YES	YES	NO	YES		50
37	2402-BT	no	yes	no	yes	yes	Silica/AgNO3	100
45	2402-BT	yes	yes	yes	no	yes	acidic treatment	50
46	2402-BT	NO	YES	NO	YES	NO	NO	20
47	2402-BT							
51	2402-BT	no	yes	no	yes	no	acid hydrolysis with sulphuric acid	40
52	2402-BT	no	yes	no	yes	yes	MIURA	50
53	2402-BT	no	yes	no	yes	yes		100
55	2402-BT	no	yes	no	yes	no	further clean-up of final extract with sulphuric acid silica column	300
58	2402-BT							
59	2402-BT							
64	2402-BT	no	yes	yes	yes	no	no	100
65	2402-BT	no	yes	no	yes	no	no	50
69	2402-BT	no	yes	no	yes	no		100
70	2402-BT	no	yes	no	yes	no		25
72	2402-BT	no	no	no	no	no	no	1000
78	2402-BT	no	yes	no	yes	no	silica	50
80	2402-BT	no	yes	no	yes	yes	-	100
83	2402-BT	yes	yes	no	no	yes		
84	2402-BT	no	yes	no	no	yes	no	20
85	2402-BT	no	yes	no	yes	yes	no	500
86	2402-BT	no	yes	no			basic set of "power-prep system" columns	40
94	2402-BT	no	yes	no	yes	no		100
37A	2402-BT	no	yes	no	yes	yes	Silica/AgNO3	100

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]

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

Bentonite (2402-BT)

Physico-chemical Methods PBDEs - Chromatographic separation and detection method

		Chromatographic separation and detection method			
LC	Sample	GC injection	Injected volume [µl]	Chromatographic separation: Stationary phase	Detector
3	2402-BT	Splitless	1.5	DB-5MS	HRMS, DFS
4	2402-BT	pulsed splitless	1	DB-5MS (60m x 0,25mm x 0,10 µm)	HRMS (R>10000)
9	2402-BT	Splitless	1µL	5%-Phenyl-methylPolysiloxane (DZB5) column 10m×0.1mm×0.1µm	Thermo DFS HRMS (>10,000 Mass resolution)
20	2402-BT	PTV	10	Rtx-CIPesticides	GC-HRMS
27	2402-BT	PTV	10	DB5HT 15 m x 0.25 mm; 0.1 um	MS/MS
32	2402-BT	splitless	2.0	DB-5HT	HRMS (Autospec Ultima Waters)
34	2402-BT	Splitless	2	HT8PCB	GC-HRMS
37	2402-BT	PTV	1	ZB-Semivolatile 20m, 0.18mm, 0.18µm	MS/MS
45	2402-BT	SSL	1	Rtx-1614	HRMS
46	2402-BT	SPLITLESS	1	DB-5ms 40m x 0,18 mm x 0,18 um	HRMS
47	2402-BT				
51	2402-BT	PTV mode: Splitless	2	Rtx-1614 30m x 0.25mm x 0.1µm	HRMS
52	2402-BT	large volume	4	Rtx-1614 (15m x 0,25 mm x 0,1 µm) + 2m retention gap (uncoated)	MS/MS
53	2402-BT	splittless	2	DB 5 MS	HRMS
55	2402-BT	PTV	5	Rtx-1614	HRMS
58	2402-BT				
59	2402-BT				
64	2402-BT	PTV	1.5	RTX-1614	HRMS
65	2402-BT	splitless	1	CP-5MS	MS/MS
69	2402-BT	pulsed splitless	2	Diphenyl-/dimethylpolysiloxan (5%/95%)	MS/MS
70	2402-BT	splitless	1	Rtx-1614	HRMS
72	2402-BT	PTV	2	DB-5MS 10m	Agilent 7000
78	2402-BT	splitless	1	DB5-MS	HRMS
80	2402-BT	PTV	5	RTX-1614 (15x0.25x0.10)	HRMS
83	2402-BT	splitless	2	Phenomenex ZB-SemiVolatiles 20m	GC MS/MS
84	2402-BT	splitless	2	DB-5ht	HRMS
85	2402-BT	splitless	5	(5%-phenyl)-methylpolysiloxane	MSMS
86	2402-BT	Splitless	1	DB-5MS (30 m, 0.25 mm id, 0,25 mm film)	HRMS (Mat-95 XP)
94	2402-BT	pulsed splitless	2	Diphenyl-/dimethylpolysiloxan (5%/95%)	MS/MS
37A	2402-BT	PTV	1	ZB-Semivolatile 20m, 0.18mm, 0.18µm	MS/MS

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Bentonite (2402-BT)
Physico-chemical Methods HBCDDs - Pre-treatment and extraction

		Pre-treatment and extraction					
LC	Sample	Sample preparation/pre-treatment	Extraction technique	Extraction solvent	Extraction time [h]	Extraction temperature [ºC]	Extraction pressure [MPa]
3	2402-BT						
4	2402-BT	drying	shaking with dichlormethan : acetone 2:1 for 90 min	100 ml of dichlormethan : acetone 2:1	90 min	ambient	0.10
9	2402-BT						
20	2402-BT	Sodium Sulfate	ASE	Hexane/Acetone 1:1	30 minutes	100	1500 psi
27	2402-BT		QuEChERS-like	Ethyl Acetate			
32	2402-BT						
34	2402-BT	drying	PLE	Toluene/Acetone			
37	2402-BT						
45	2402-BT	NA	Ultrasound + Mechanical Shaking	DCM:N-Hex (1:1, v/v)	1h	Room temp.	ambient
46	2402-BT						
47	2402-BT		Soxleth	Acetone/Hexane (1/1)	6		
51	2402-BT	Mixed with sodium sulphate	ASE	Toluene/Acetone (70:30)	1	125	10.3
52	2402-BT	slurry with Aceton/Water (4/1) and than mix with sodiumfulfate	hot extraction (Randall)	Hexane/Dichlormethane/Ethanol (5/2/1)	2	95	ambient
53	2402-BT		Soxhlet	toluene/acetone 7/3			
55	2402-BT	freeze drying	Twisselmann	Cyclohexan / Toluol (1:1, v/v)	6	boiling point	
58	2402-BT	None	ASE	50:50 Acetone:Hexane	0.5	100	10.3
59	2402-BT	no	solid/liquid extraction	Hexane/Dichloromethane 50/50	0.33	20	Ambiant
64	2402-BT		ASE 350	toluene / acetone (70/30)	0.35	100	10
65	2402-BT						
69	2402-BT	drying	Soxhlet	Toluol	8 h		
70	2402-BT	Soxhlet	Toluene /ethanol 50 / 50	Step 1: toluene /ethanol 50 / 50 - step 2: toluene	24 for each step (48 in total)	120	atm
72	2402-BT	the weighed sample is mixed with Na2SO4 for drying	ultrasonic bath	Ethylacetate / Acetone 1:1	1h	60 °C	environmental preassure
78	2402-BT						
80	2402-BT						
83	2402-BT	no	Soxhlet	EtOH/Tol 2/1	8		
84	2402-BT	homogenisation, drying (only for determination of moisture content)	ASE, Soxhlet	toluene, ethanol (ASE), toluene (Soxhlet)	30 min/sample (ASE), 16 h (Soxhlet)	100 °C (ASE)	10 MPa (ASE)
85	2402-BT		soxhlet	toluene/ethanol 70/30	12	bp	ambient
86	2402-BT						
94	2402-BT	drying	Soxhlet	Toluol	8 h		

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Bentonite (2402-BT)
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
3	2402-BT								
4	2402-BT		yes	yes	no	no	no		500
9	2402-BT								
20	2402-BT		no	yes	no	no	no		500 µl
27	2402-BT		yes	no	no	no	no	Extrelut NT-3 column, acidic for H2SO4 tandem Si 1g/6 mL column	250
32	2402-BT								
34	2402-BT		YES	YES	NO	NO	NO	liquid liquid extraction	50
37	2402-BT								
45	2402-BT		No	Yes	Yes	No	No	No	50
46	2402-BT								
47	2402-BT		no	no	no	no	no	Conc. sulphuricacid	200
51	2402-BT		no	yes	no	no	no	acid hydrolysis with sulphuric acid	50
52	2402-BT		no	yes	no	no	no	no	400
53	2402-BT		yes	no	no	no	no	silica gel column	50
55	2402-BT		no	yes	yes	no	no	no	100
58	2402-BT		No	Yes	Yes	No	No	None	100
59	2402-BT		no	yes	no	no	no		200
64	2402-BT		no	yes	no	no	no		50
65	2402-BT								
69	2402-BT		no	yes	no	no	no		1000
70	2402-BT		no	yes	no	no	no		1000
72	2402-BT		no	no	no	no	no	no	1000
78	2402-BT								
80	2402-BT								
83	2402-BT		yes	yes	no	no	yes		30
84	2402-BT		no	s (elution with a solvent mixture of hexane and dichloromethar	no	no	no	no	20
85	2402-BT		no	yes	no	no	no		80
86	2402-BT								
94	2402-BT		no	yes	no	no	no		1000

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

Bentonite (2402-BT)

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
3	2402-BT					
4	2402-BT		normal	10	C18	MS/MS
9	2402-BT					
20	2402-BT			10	C18 (Waters symmetry), 150 mm x 2.1 mm x 3.5 µm	LCMSMS
27	2402-BT			20 µL	KINETEX 2.6um XB-C18 100A (100 x 2.1 mm) (PHENOMENEX)	LC-MS/MS
32	2402-BT					
34	2402-BT			15	Hypersil Gold	MS/MS
37	2402-BT					
45	2402-BT		Liquid injection	5	LC - C18	HRMS (Orbitrap Q-Exactive)
46	2402-BT					
47	2402-BT		HPLC injection	5	Acquity BEH C18 (1.7 µm, 2.1*100 mm)	MS/MS
51	2402-BT			5	BEH C18 (50 mm x 2.1 mm; 1.7 µm)	HRMS
52	2402-BT			15	C18	MS/MS
53	2402-BT			10	RP 18	MS/MS
55	2402-BT		HPLC	2	C18	MS/MS
58	2402-BT			10	C18	HRMS (Hybrid Quadrupole/Orbitrap)
59	2402-BT		UPLC	5	Eclipse Plus C18, 2,1 x 100mm	MS/MS
64	2402-BT			10	Hypersil Gold C18, 100x2,1mm, 1,9µm	MS/MS
65	2402-BT					
69	2402-BT		standard	10	C18	MS/MS
70	2402-BT		LC		Acquity BEH C18 150 mm	MS/MS
72	2402-BT		PTV	2	DB-5MS 10m	Agilent 7000
78	2402-BT					
80	2402-BT					
83	2402-BT			70	Luna PFP	LC MS/MS
84	2402-BT		splitless	2	DB-5ht	HRMS
85	2402-BT		Autosampler	7	C18	MS(MS
86	2402-BT					
94	2402-BT		standard	10	C18	MS/MS

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26 August 2024

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

Test sample - Bentonite (2402-BT)

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]

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

Bentonite (2402-BT)

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
3	2402-BT	The LOQ was defined as the concentration that produced an instrumantal resposse with a signal-to-noise ration of 10		
4	2402-BT			
9	2402-BT			BDE 209 Not determined
20	2402-BT	according to NEN 7777	according to NEN 7777 expanded measurement uncertainty	
27	2402-BT	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	
32	2402-BT			
34	2402-BT	S/N > 3		
37	2402-BT	Mean of Blank + 3s	multiple analysis of reference material	
45	2402-BT			
46	2402-BT			
47	2402-BT			
51	2402-BT	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	
52	2402-BT		top-down approach as discribed in the "Guidance document on measurment uncertainty for laboratories performing PCDD/F and PCB analysis"	
53	2402-BT			
55	2402-BT			
58	2402-BT			
59	2402-BT			
64	2402-BT	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.	
65	2402-BT			
69	2402-BT			
70	2402-BT			
72	2402-BT			
78	2402-BT	4-Point-Calibration	quality control chart of different matrices, but fat (food): halibut, turkey, milk powder, egg	
80	2402-BT	Signal to noise in the validation & derived from blanks	From old PT material using the EURL excel sheet for dioxins	Co-extraction with the dioxins and pcbs
83	2402-BT			
84	2402-BT	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	We did not observed impurities that could affect the HRMS analysis especially for PBDEs. The problem occurs more often in feed materials of oily or vegetable origin containing waxes. Therefore, we didnt apply clean-up on florisil or GPC.
85	2402-BT			
86	2402-BT			
94	2402-BT			
37A	2402-BT	Mean of Blank + 3s	multiple analysis of reference material	

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]

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

Bentonite (2402-BT)

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

LC	Sample	BDE 28	BDE 47	BDE 49	BDE 99	BDE 100	BDE 153	BDE 154	BDE 183	BDE 209
3	2402-BT	0.001937259	0.001937259	0.001937259	0.001937259	0.001937259	0.001937259	0.001937259	0.001937259	
4	2402-BT									
9	2402-BT	0.001	0.03	0.003	0.02	0.001	0.002	0.001	0.03	
20	2402-BT	1.3	1.1	1.1	2.8	1.9	7.4	4.5	5	339
27	2402-BT	0.02		0.02				0.02		
32	2402-BT	0.001363512								
34	2402-BT									
37	2402-BT	0.0013	0.034	0.0015	0.0084	0.0025	0.0016	0.0018	0.0033	0.13
45	2402-BT	0.00148	0.00102	0.00152	0.00151	0.00167	0.00209	0.0024	0.05678	0.08963
46	2402-BT	0.000148231	0.002035692	0.000140423	0.001569788	0.000353288	0.025682962	0.008803577	0.047570808	0.0419764
47	2402-BT									
51	2402-BT	0.001	0.025	0.0009	0.015	0.0017	0.0017	0.0024	0.0008	
52	2402-BT									
53	2402-BT									
55	2402-BT	0.0001	0.0001	0.0001	0.0002	0.0002	0.0001	0.0001	0.0003	0.006
58	2402-BT									
59	2402-BT									
64	2402-BT	0.00068	0.0005	0.0006	0.0016	0.0013	0.0009	0.0013	0.0021	0.0058
65	2402-BT	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.1
69	2402-BT	0.0191	0.0191	0.0191	0.0381	0.0381	0.0572	0.0572	0.0953	1.91
70	2402-BT									
72	2402-BT	100	100	100	100	100	100	100	100	100
78	2402-BT	0.01	0.01		0.01	0.01	0.01	0.01	0.05	
80	2402-BT	0.001029743	0.018838336	0.000540168	0.003688198	0.002451277	0.000632397	0.001259717	0.004215687	0.066032393
83	2402-BT	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
84	2402-BT	6.26488E-05	6.28937E-05	6.28937E-05	0.000163133	0.000168867	7.70689E-05	6.23147E-05	0.000127939	0.005686567
85	2402-BT	0.0036		0.0031						0.77
86	2402-BT	0.00001	0.00001	0.00005	0.00002	0.00001	0.00002	0.00001	0.00008	
94	2402-BT	0.0195	0.0195	0.0195	0.039	0.039	0.0585	0.0585	0.0975	1.95
37A	2402-BT	0.0025	0.032	0.0017	0.011	0.003	0.0054	0.0024	0.025	0.18

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]

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

Bentonite (2402-BT)

Methods PBDEs - Measurement Uncertainty [%]

LC	Sample	BDE 28	BDE 47	BDE 49	BDE 99	BDE 100	BDE 153	BDE 154	BDE 183	BDE 209
3	2402-BT	15	15	15	15	15	15	15	15	
4	2402-BT	30	30	30	30	30	30	30	30	-
9	2402-BT	15	15	15	15	15	15	15	15	
20	2402-BT	25	25	25	25	25	30	25	25	50
27	2402-BT	34	34	34	34	34	34	34	22	
32	2402-BT	30	30	60	30	30	30	30	30	30
34	2402-BT									
37	2402-BT	20	20	20	20	20	20	20	20	70
45	2402-BT									
46	2402-BT									
47	2402-BT									
51	2402-BT	28	41	34	34	27	27	28	36	
52	2402-BT	29.6	19.1	56	18.5	16.51	16.3	19.3	17.3	24.7
53	2402-BT									
55	2402-BT									
58	2402-BT									
59	2402-BT									
64	2402-BT	29	21	28	22	23	28	27	23	29
65	2402-BT	50	50	50	50	50	50	50	50	50
69	2402-BT	30	30	30	30	30	30	30	30	30
70	2402-BT									
72	2402-BT	50	50	50	50	50	50	50	50	50
78	2402-BT	40	30		30	30	30	30	30	
80	2402-BT	26.3	20	29.9	20.2	22.1	27.8	20	25.3	23
83	2402-BT	20	20	20	20	20	20	20	20	20
84	2402-BT	67.9	20.3	20.3	17.5	19.4	20.2	23.8	9.68	31.4
85	2402-BT		50		50	50	50	50	50	
86	2402-BT									
94	2402-BT	30	30	30	30	30	30	30	30	30
37A	2402-BT	20	20	20	20	20	20	20	20	70

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Bentonite (2402-BT)
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
3	2402-BT			
4	2402-BT			
9	2402-BT			
20	2402-BT	according to validation.	according to NEN 7777	
27	2402-BT	Samples spiked at LOQ level (10 pg/g) within the batch	From validation data with a semplified bottom up approach	
32	2402-BT			
34	2402-BT			
37	2402-BT			
45	2402-BT			
46	2402-BT			
47	2402-BT	From analysis of the bentonite sample with addition of analyte LOQ estimated as 10*S/N of calculated concentration	Combined intralaboratory reproducibility and method bias	
51	2402-BT	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	
52	2402-BT			
53	2402-BT			
55	2402-BT			
58	2402-BT	lowest validated level		
59	2402-BT	The LOQs corespond to the lowest level of the calibration curve	U= k*u=((2x CVrw) + biais) for validated matrices	
64	2402-BT	LOD and LOQ were estimated based on analysis of 10 blank spiked samples	MU were estimated based on precision and truenes from fortyfication experiment	
65	2402-BT			
69	2402-BT			
70	2402-BT			
72	2402-BT			
78	2402-BT			
80	2402-BT			
83	2402-BT			
84	2402-BT	LOQ = 3 x LOD; LOD evaluated as 3 times of noise		
85	2402-BT	The lowest calibration curve point.		
86	2402-BT			
94	2402-BT			

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]

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

Bentonite (2402-BT)

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

LC	Sample	(+/–)-α-HBCDD	(+/–)-β- HBCDD	(+/–)-γ- HBCDD	Total HBCDD (using GC-methods)
4	2402-BT	0.009	0.004	0.05	
9	2402-BT				
20	2402-BT	0.0045	0.003	0.0092	
27	2402-BT		0.01		
32	2402-BT				
34	2402-BT				
37	2402-BT				
45	2402-BT	0.05	0.05	0.05	
46	2402-BT				
47	2402-BT	0.007	0.006	0.005	
51	2402-BT	0.01	0.01	0.01	
52	2402-BT		0.01		
53	2402-BT	0.005	0.005		
55	2402-BT	0.2	0.05	0.05	
58	2402-BT	0.03	0.03	0.03	
59	2402-BT	0.039	0.039	0.039	
64	2402-BT	0.001	0.005	0.011	
65	2402-BT				
69	2402-BT	0.0572	0.0572	0.0572	
70	2402-BT				
72	2402-BT	100	100	100	
78	2402-BT				
80	2402-BT				
83	2402-BT	0.002	0.002	0.002	
84	2402-BT				0.005
85	2402-BT	0.1	0.1	0.1	
86	2402-BT				
94	2402-BT	0.0585	0.0585	0.0585	

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Bentonite 2024 [EURL-PT-DPB_2402-BT]

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

Bentonite (2402-BT)

Methods HBCDDs - Measurement Uncertainty [%]

LC	Sample		(+/–)-α-HBCDD	(+/–)-β- HBCDD	(+/–)-γ- HBCDD
4	2402-BT		23	18	16
9	2402-BT				
20	2402-BT		13	7.5	13
27	2402-BT		49	49	36
32	2402-BT				
34	2402-BT				
37	2402-BT				
45	2402-BT				
46	2402-BT				
47	2402-BT				22
51	2402-BT		27	30	32
52	2402-BT				
53	2402-BT		30	30	30
55	2402-BT				
58	2402-BT		30	30	30
59	2402-BT		40	40	40
64	2402-BT		35	22	24
65	2402-BT				
69	2402-BT		30	30	30
70	2402-BT				
72	2402-BT		50	50	50
78	2402-BT				
80	2402-BT				
83	2402-BT		20	20	20
84	2402-BT				
85	2402-BT		40	40	40
86	2402-BT				
94	2402-BT		30	30	30