EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024

EURL-ILS-BCF_2403-PIM

FOOD

Final report

PBDEs and HBCDDs

(Report Version 1.0)

06 March 2026

Funded by the European Union

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Summary

Test sample	**Sample A:** Powdered Infant Milk [2403-PIM-A] **Sample B:** Powdered Infant Milk [2403-PIM-B] - Blank
Analytes of interest **Brominated**	**PBDEs** (Nine PBDE congeners) **HBCDDs** (Three stereoisomers and/or total HBCDD)
Methods	Any kind of method
Participants	NRLs, OFLs, other official laboratories, commercial laboratories performing the analysis of samples taken by food business operators, research institutes and universities
Statistical evaluation	ISO 13528:2022 [1], IUPAC Protocol [2]
Final Report	06 March 2026
Publication	EURL POPs reserves all rights to publish and present the anonymised results of the interlaboratory study in scientific journals and/or during conferences.

1. Structure of the ILS, test material and analytes

This interlaboratory study (ILS) on the determination of several **brominated contaminants (BCons), polychlorinated naphthalenes (PCNs), polychlorinated alkanes (PCAs –** main constituent of CPs) and **per- and polyfluorinated substances (PFAS)** in Powdered Infant Milk / Formula was organized by the EURL for halogenated POPs in Feed and Food to be performed between July and October 2024. The objective was to assess analytical performance of laboratories and interlaboratory comparability of results from analyses of **PBDEs, HBCDDs, TBBPA, eBFRs, PBDD/Fs, PXDD/Fs, PBB, PCNs, PCAs** and **PFAS** in a Powdered Infant Milk test material.

Official laboratories, research laboratories, Universities and commercial laboratories performing the analysis of samples taken by food business operators were invited to participate in this interlaboratory study.

1.1. Samples and coding

Test sample A was prepared from commercially available Powdered Infant Milk and fortified with analytes of interest. Test sample B was prepared from the same commercially available Powdered Infant Milk but not fortified with analytes of interest.

Powdered Infant Milk A	**Sample no. 2403-PIM-A-xxx**
Powdered Infant Milk B (Blank)	**Sample no. 2403-PIM-B-xxx**

Each participant received about **100 g** of each test sample in a HDPE bottle.

1.2. Analytes of interest

Participants were strongly encouraged to determine as many of the following analyte groups as possible, but at least one of the analyte groups (as many analytes as possible from each group):

Brominated Contaminants:

- **PBDE group**
  - 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)

- **HBCDD group**
  - 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 may 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 upper and lower bound results for sum parameters,
- 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 product**[1] and/or **µg/kg fat** 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

61 laboratories registered for this proficiency test. For PBDEs and HBCDDs, 22 and 16 sets of results were reported, respectively.

3. Test for sufficient homogeneity

The test for sufficient homogeneity was performed according to ISO 13528:2022 [1] and the International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [2]. Therefore, 10 portions of the test samples 2403-PIM-A were analysed in duplicate for PBDEs. The test for sufficient homogeneity was performed for the individual substances. The test materials showed sufficient homogeneity for PBDEs in this proficiency test. Due to similar physico-chemical properties this can also be concluded for HBCDDs.

[1] Product meaning dry powder as received (not reconstituted)

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 PBDEs and HBCDDs compounds, and sum parameters (including limits of quantification (LOQs)), if possible. Additionally the median of all values was calculated.

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

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

- BDE-28, BDE-49
- (+/-)-β-HBCDD, (+/-)-γ-HBCDD
- Total HBCDD (using GC-methods)

4.1. PBDEs – individual congeners and sum parameter

The assigned values for the test sample 2403-PIM-A were calculated as consensus of participants' results for individual PBDEs and sum parameters, taking into account the calculation criteria described above (Table 1; tabular summary see annex 1).

Table 1: Assigned values for PBDEs for 2403-PIM-A (rounded to three significant figures)

Powdered Infant Milk A (2403-PIM-A)	Assigned value µg/kg product	Assigned value µg/kg fat
BDE-47	0.107	0.418
BDE-99	0.144	0.562
BDE-100	0.0304	0.119
BDE-153	0.0351	0.135

Powdered Infant Milk A (2403-PIM-A)	Assigned value µg/kg product	Assigned value µg/kg fat
BDE-154	0.0158	0.0633
BDE-183	0.125	0.507
BDE-209	0.265	1.05
Sum of 8 PBDEs (without BDE-209) (ub)	0.457	1.82
Sum of 8 PBDEs (without BDE-209) (lb)	0.458	1.81
Sum of 9 PBDEs (including BDE-209) (ub)	0.697	2.85
Sum of 9 PBDEs (including BDE-209) (lb)	0.708	2.85

4.2. HBCDDs – individual stereoisomers and sum parameter

The assigned values for the test sample 2403-PIM-A were calculated as consensus of participants' results for individual HBCDDs and sum parameters, taking into account the calculation criteria described above (Table 2; tabular summary see annex 1).

Table 2: Assigned values for HBCDDs for 2403-PIM-A (rounded to three significant figures)

Powdered Infant Milk A (2403-PIM-A)	Assigned value µg/kg product	Assigned value µg/kg fat
(+/–)-α-HBCDD	0.0768	0.314
(+/–)-β-HBCDD	-	0.0421
Sum of α-, β-, γ-HBCDD (ub)	0.0983	0.396
Sum of α-, β-, γ-HBCDD (lb)	0.0844	0.354

4.3. Lipid content

For the lipid content an assigned value of 25.2 % for the test sample 2403-PIM-A was calculated as a consensus of the participants' results, taking into account the calculation criteria described above (tabular summary see annex 1).

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 (Table 3). In the EURL "Guidance document on analytical parameters

for the determination of organobromine contaminants in food and feed", a lower LOQ value of 0.001 µg/kg w.w. is targeted for all congeners except BDE-209, since some foods have concentrations below this value (Table 4; [3]). Valid data on the background contamination of foodstuffs with BFRs is particularly important for a reliable risk assessment. For HBCDDs the recommended LOQ value is 0.01 µg/kg w.w. for α-, β- and γ-stereoisomers (Table 3). For total HBCDD measured by GC-MS, the corresponding LOQ value is 0.003 µg/kg (as cumulative response of all possible HBCDD diastereomers, Table 4).

Table 3: Recommended LOQs for PBDEs and HBCDDs from COMMISSION RECOMMENDATION of 3 March 2014 on the monitoring of traces of brominated flame retardants in food (2014/118/EU)

Food	Limit of quantification per congener/stereoisomer µg/kg (wet weight)
PBDEs	≤ 0.01
HBCDDs	≤ 0.01

Table 41: Analytical recommendations from “Guidance document on analytical parameters for the determination of organobromine contaminants in food and feed” [3]

Food	Limit of quantification per congener/stereoisomer µg/kg (wet weight)
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 values for PBDEs on product basis were above the target LOQ of 0.001 µg/kg. For BDE-154 the assigned value (0.0158 µg/kg product) was in the range of the recommended LOQ of 0.01 µg/kg product.

HBCDDs:

For γ-HBCDD and total HBCDD (using GC methods) no assigned values could be calculated, and for β-HBCDD only for results on fat basis. The calculated median value of all participants’ results was taken for comparison with the recommended LOQ. The median values for β-HBCDD and γ-HBCDD were both in the range of the recommended LOQ of 0.01 µg/kg product. For β-HBCDD an assigned value could be calculated on fat basis showing that laboratories could possibly also analyze reliable in this concentration range.

5. Scoring of results – Z-scores

5.1. Z-scores calculation

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

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

x: *participant's result*

x_a: *assigned value*

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

For individual substances and sum parameters, the standard deviation for proficiency assessment $\sigma_{p_{rel}}$ is defined as 20 %.

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

Interpretation of z-scores:

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

5.2. PBDEs - Participants' z-scores

Table 5: Distribution of participants' z-scores for PBDEs for Powdered Infant Milk A (2403-PIM-A) based on results on ng/kg product basis

Percentage of participants' results (product basis)	$\lvert z\text{-score} \rvert \leq 2$	$2 < \lvert z\text{-score} \rvert < 3$	$\lvert z\text{-score} \rvert \geq 3$
BDE-47	68%	18%	14%
BDE-99	91%	5%	5%
BDE-100	86%	%%	9%
BDE-153	95%	-	5%
BDE-154	91%	-	9%
BDE-183	95%	-	5%
BDE-209	76%	6%	18%
Sum of 8 PBDEs	90%	-	10%

Percentage of participants' results (product basis)	|z-score| ≤ 2	2 < |z-score| < 3	|z-score| ≥ 3
without BDE-209 (ub)			
Sum of 8 PBDE including BDE-209 (lb)	90%	-	10%
Sum of 9 PBDE including BDE-209 (lb)	73%	20%	7%
Sum of 9 PBDE including BDE-209 (lb)	81%	13%	6%

Table 6: Distribution of participants' z-scores for PBDEs for Powdered Infant Milk A (2403-PIM-A) based on results on fat basis

Percentage of participants' results (fat basis)	|z-score| ≤ 2	2 < |z-score| < 3	|z-score| ≥ 3
BDE-47	79%	7%	14%
BDE-99	100%	-	-
BDE-100	87%	-	13%
BDE-153	87%	-	13%
BDE-154	93%	-	7%
BDE-183	93%	-	7%
BDE-209	70%	10%	20%
Sum of 8 PBDEs without BDE-209 (ub)	87%	-	13%
Sum of 8 PBDE including BDE-209 (lb)	87%	-	13%
Sum of 9 PBDE including BDE-209 (lb)	70%	10%	20%
Sum of 9 PBDE including BDE-209 (lb)	70%	10%	20%

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

Table 7: Distribution of participants' z-scores for HBCDD for Powdered Infant Milk A (2403-PIM-A) based on results on product basis

Percentage of participants' results (product basis)	|z-score| ≤ 2	2 < |z-score| < 3	|z-score| ≥ 3
(+/–)-α-HBCDD	92%	-	8%
Sum of α-, β-, γ-HBCDD (ub)	69%	15%	15%
Sum of α-, β-, γ-HBCDD (lb)	85%	-	15%
Total HBCDD*	50%	50%	-

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

Table 8: Distribution of participants' z-scores for HBCDD for Powdered Infant Milk A (2403-PIM-A) based on results on fat basis

Percentage of participants' results (fat basis)	|z-score| ≤ 2	2 < |z-score| < 3	|z-score| ≥ 3
(+/–)-α-HBCDD	90%		10%
(+/–)-β-HBCDD	100%		
Sum of α-, β-, γ-HBCDD (ub)	70%	10%	20%
Sum of α-, β-, γ-HBCDD (lb)	80%		20%
Total HBCDD*	100%		

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

6. 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).

7. Results of participants

An overview of the PBDE and HBCDD results for the PT test samples Powdered Infant Milk A and B (2403-PIM-A and 2403-PIM-B) is given in the following annexes. Laboratories are coded according to the laboratory codes sent after registration.

8. 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.

[3] EURL for halogenated POPs in feed and food (2021): Guidance Document on the Determination of Organobromine Contaminants - Analytical Parameters in food and feed, version 1.2 of 10 May 2023. Available online under https://eurl-pops.eu/news/guidance-document-bcon-parameters

9. Annex

Powdered Infant Milk – 2403-PIM	
1	2403-PIM-A - Assigned and median values – PBDEs, HBCDDs
2a	2403-PIM-A - Participants' results on product basis – PBDEs, HBCDDs, lipid content
2b	2403-PIM-A - Participants' results on fat basis – Tables – PBDEs, HBCDDs
3a	2403-PIM-A - Participants' z-scores on product basis – Tables – PBDEs, HBCDDs, lipid content
3b	2403-PIM-A - Participants' z-scores on fat basis – Tables – PBDEs, HBCDDs
4	2403-PIM-A - Participants' z-scores – Charts – PBDEs, HBCDDs
5	2403-PIM-A - Test for homogeneity for PBDEs
6a	2403-PIM-B - Participants' results on product basis – PBDEs, HBCDDs
6b	2403-PIM-B - Participants' results on fat basis – PBDEs, HBCDDs
7	Overview of participants' methods

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Annex 1: Assigned values of PBDEs and HBCDDs

Test sample - Powdered Infant Milk A (2403-PIM-A)

Assigned values of individual substances and sum parameters

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

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Powdered Infant Milk A (2403-PIM-A)

PBDE - Assigned values

Analyte		Result µg/kg product	**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						0.00180
BDE-47	2,2′,4,4′-tetrabromodiphenyl ether		0.107	0.019	0.0059	17	0.110
BDE-49	2,2′,4,5′-tetrabromodiphenyl ether						0.00350
BDE-99	2,2′,4,4′,5-pentabromodiphenyl ether		0.144	0.022	0.0066	18	0.143
BDE-100	2,2′,4,4′,6-pentabromodiphenyl ether		0.0304	0.0049	0.0014	18	0.0314
BDE-153	2,2′,4,4′,5,5′-hexabromodiphenyl ether		0.0351	0.0061	0.0017	19	0.0349
BDE-154	2,2′,4,4′,5,6′-hexabromodiphenyl ether		0.0158	0.0027	0.00079	18	0.0158
BDE-183	2,2′,3,4,4′,5′,6-heptabromodiphenyl ether		0.125	0.016	0.0045	19	0.126
BDE-209	2,2′,3,3′,4,4′,5,5′,6,6′-decabromodiphenyl ether		0.265	0.065	0.022	13	0.263
Sum of 8 PBDE	without BDE-209 (ub)		0.457	0.061	0.019	16	0.467
Sum of 8 PBDE	without BDE-209 (lb)		0.458	0.059	0.018	17	0.464
Sum of 9 PBDE	including BDE-209 (ub)		0.697	0.12	0.045	12	0.699
Sum of 9 PBDE	including BDE-209 (lb)		0.708	0.13	0.044	13	0.710

Analyte		Result µg/kg fat	**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						0.00595
BDE-47	2,2′,4,4′-tetrabromodiphenyl ether		0.418	0.068	0.025	11	0.429
BDE-49	2,2′,4,5′-tetrabromodiphenyl ether						0.0121
BDE-99	2,2′,4,4′,5-pentabromodiphenyl ether		0.562	0.10	0.035	13	0.573
BDE-100	2,2′,4,4′,6-pentabromodiphenyl ether		0.119	0.011	0.0040	12	0.123
BDE-153	2,2′,4,4′,5,5′-hexabromodiphenyl ether		0.135	0.016	0.0059	12	0.133
BDE-154	2,2′,4,4′,5,6′-hexabromodiphenyl ether		0.0633	0.011	0.0037	13	0.0639
BDE-183	2,2′,3,4,4′,5′,6-heptabromodiphenyl ether		0.507	0.060	0.022	12	0.511
BDE-209	2,2′,3,3′,4,4′,5,5′,6,6′-decabromodiphenyl ether		1.05	0.24	0.11	7	1.05
Sum of 8 PBDE	without BDE-209 (ub)		1.82	0.23	0.085	12	1.87
Sum of 8 PBDE	without BDE-209 (lb)		1.81	0.24	0.086	12	1.87
Sum of 9 PBDE	including BDE-209 (ub)		2.85	0.39	0.17	8	3.05
Sum of 9 PBDE	including BDE-209 (lb)		2.85	0.39	0.17	8	3.05

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Powdered Infant Milk A (2403-PIM-A)

HBCDD - Assigned values

Analyte		Result µg/kg product	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)		0.0768	0.0065	0.0023	12	0.0800
(+/–)-β- HBCDD	(1,2,5,6,9,10-hexabromo-(1R,2S,5R,6R,9R,10S)-rel-cyclododecane)						0.0105
(+/–)-γ- HBCDD	(1,2,5,6,9,10-hexabromo-(1R,2R,5R,6S,9S,10R)-rel-cyclododecane)						0.0105
Sum of α-, β-, γ-HBCDD	(ub)		0.0983	0.012	0.0046	11	0.100
Sum of α-, β-, γ-HBCDD	(lb)		0.0844	0.0087	0.0033	11	0.0842
Total HBCDD	(using GC-methods)						0.107

Analyte		Result µg/kg fat	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)		0.314	0.048	0.02	9	0.312
(+/–)-β- HBCDD	(1,2,5,6,9,10-hexabromo-(1R,2S,5R,6R,9R,10S)-rel-cyclododecane)		0.0421	0.008	0.0033	8	0.0455
(+/–)-γ- HBCDD	(1,2,5,6,9,10-hexabromo-(1R,2R,5R,6S,9S,10R)-rel-cyclododecane)						0.0435
Sum of α-, β-, γ-HBCDD	(ub)		0.396	0.069	0.03	8	0.410
Sum of α-, β-, γ-HBCDD	(lb)		0.354	0.053	0.023	8	0.373
Total HBCDD	(using GC-methods)						0.413

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Powdered Infant Milk A (2403-PIM-A)
Lipid 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]
Lipid content		25.2	2.09	0.58	20	25.0

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Annex 2a: Participants' results of PBDEs and HBCDDs on product basis

Test sample - Powdered Infant Milk A (2403-PIM-A)

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Powdered Infant Milk A (2403-PIM-A)

PBDE - Results

LC	Sample	Result µg/kg product	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** including BDE-209 (lb)
1	2403-PIM-A		0.0013	0.12	0.0035	0.17	0.035	0.036	0.019	0.15		0.53	0.53		
4	2403-PIM-A		< 0.01	0.115	< 0.01	0.145	0.0428	0.0434	0.0156	0.141	0.329		0.503		0.832
5	2403-PIM-A		0.001	0.082	0.092	0.124	0.027	0.03	0.011	0.104	0.235	0.47	0.47	0.71	0.71
6	2403-PIM-A		0.00145	0.0973	0.00151	0.133	0.0276	0.0283	0.014	0.113	0.254	0.416	0.416	0.671	0.671
9	2403-PIM-A		0.00138	0.0815	0.00231	0.111	0.0226	0.025	0.0113	0.0919	0.197	0.347	0.347	0.544	0.544
13	2403-PIM-A		< 1000	< 1000	< 1000	< 1000	< 1000	< 1000	< 1000	< 1000					
14	2403-PIM-A		< 0.00198	0.0931	< 0.00188	0.133	0.0246	0.0362	0.0148	0.124	0.215	0.432	0.426	0.647	0.641
15	2403-PIM-A		0.0065	0.16	0.003	0.2	0.043	0.047	0.019	0.13	0.35	0.61	0.61	0.95	0.96
16	2403-PIM-A		0.00128	0.108	0.00313	0.135	0.0319	0.0351	0.0157	0.133	0.271	0.464	0.464	0.735	0.735
20	2403-PIM-A		0.0052	0.114		0.148	0.0274	0.0349	0.031	0.124	0.29				
21	2403-PIM-A		0.00605	0.314	0.00728	0.184	0.0499	0.0302	0.0158	0.126	0.347	0.733	0.733	1.08	1.08
23	2403-PIM-A		0.0018	0.112	0.001	0.143	0.0305	0.0343	0.015	0.129		0.466	0.466		
36	2403-PIM-A		0.00101	0.0556	0.00269	0.0923	0.0288	0.0459	0.0214	0.145	0.471	0.393	0.393	0.864	0.864
37	2403-PIM-A		< 0.006	0.107	< 0.01	0.141	0.029	0.034	0.016	0.127	0.321	0.47	0.456	0.791	0.791
38	2403-PIM-A		1.49	117	3.15	160	33	31.4	14.7	144	261	505	505	766	766
41	2403-PIM-A		0.0014	0.1	0.00286	0.14	0.03	0.03	0.014	0.122	0.2	0.44	0.44	0.64	0.64
44	2403-PIM-A		< 0.0036	0.107		0.144	0.0314	0.033	0.0175	0.12		0.457	0.454		
49	2403-PIM-A		< 0.005	0.126	< 0.005	0.133	0.032	0.033	0.014	0.119	0.22	0.468	0.458	0.688	0.678
50	2403-PIM-A		0.0001	0.11	0.0027	0.152	0.0337	0.0342	0.0176	0.124	0.211	0.474	0.474	0.685	0.685
53	2403-PIM-A		< 0.00142	0.154	0.00396	0.173	0.0357	0.0463	0.0187	0.163		0.596	0.595		
58	2403-PIM-A		0.0011	0.0034	0.0446	0.0642	0.0247	0.0355	0.0141	0.0834	0.101	0.271	0.271	0.373	0.373
44A	2403-PIM-A		< 0.0036	0.104		0.143	0.032	0.0329	0.0154	0.119		0.449	0.446		
49A	2403-PIM-A		< 0.005	0.158	< 0.005	0.17	0.037	0.043	0.02	0.154	0.393	0.591	0.581	0.984	0.974

EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]

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

Powdered Infant Milk A (2403-PIM-A)

HBCDD - Results

LC	Sample	Result µg/kg product	**(+/–)-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)
5	2403-PIM-A		0.0735	0.0087	0.011		0.0932	0.0932	
6	2403-PIM-A								0.063
8	2403-PIM-A		0.067	0.009	0.005		0.081	0.081	
13	2403-PIM-A		< 1000	< 1000	< 1000				
14	2403-PIM-A		0.0745	0.00975	< 0.007		0.0913	0.0842	
16	2403-PIM-A		0.081	0.01	< 0.01		0.101	0.091	
19	2403-PIM-A		0.078	< 0.04	< 0.04		0.158	0.078	
20	2403-PIM-A								0.151
21	2403-PIM-A		0.0803	< 0.01	< 0.01		0.1	0.0803	
37	2403-PIM-A		0.0719	0.0117	0.0084		0.092	0.092	
38	2403-PIM-A		0.273	< 0.25	< 0.25		0.773	0.273	
43	2403-PIM-A		0.104	0.0133	0.032		0.149	0.149	
44	2403-PIM-A		0.068	< 0.01	< 0.01		0.088	0.068	
49	2403-PIM-A		0.08	< 0.01	< 0.01		0.1	0.08	
50	2403-PIM-A		0.081	0.011	0.011		0.103	0.103	
51	2403-PIM-A		0.08	< 0.03	< 0.03		0.14	0.08	

EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]

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

Powdered Infant Milk A (2403-PIM-A)

Lipid content - Results

LC	Sample	Result %	Lipid content	Lipid content	Lipid content
			PBDE	HBCDD	Mean
1	2403-PIM-A		26.8		26.8
4	2403-PIM-A				
5	2403-PIM-A		25.0	25.0	25.0
6	2403-PIM-A		24.0	24.0	24.0
8	2403-PIM-A			25.0	25.0
9	2403-PIM-A				
13	2403-PIM-A				
14	2403-PIM-A				
16	2403-PIM-A			26.8	26.8
15	2403-PIM-A		23.2		23.2
16	2403-PIM-A		29.3		29.3
19	2403-PIM-A			24.5	24.5
20	2403-PIM-A		23.3	23.3	23.3
21	2403-PIM-A		23.4	23.4	23.4
23	2403-PIM-A		25.6		25.6
36	2403-PIM-A		26.3		26.3
37	2403-PIM-A		25.8	23.6	24.7
38	2403-PIM-A		13.7	13.7	13.7
41	2403-PIM-A		23.8		23.8
43	2403-PIM-A			26.2	26.2
44	2403-PIM-A		27.1	27.1	27.1
49	2403-PIM-A				
50	2403-PIM-A		22.4	22.8	22.6
53	2403-PIM-A		28.4		28.4
58	2403-PIM-A				
44A	2403-PIM-A		27.1		27.1
49A	2403-PIM-A				

EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]

EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food
06 March 2026

Annex 2b: Participants' results of PBDEs and HBCDDs on fat basis

Test sample - Powdered Infant Milk A (2403-PIM-A)

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

EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]

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

Powdered Infant Milk (2403-PIM-A)

PBDE - Results

LC	Sample	Result µg/kg fat	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** including BDE-209 (lb)
1	2403-PIM-A		0.0049	0.45	0.013	0.64	0.13	0.14	0.072	0.553			2	2		
4	2403-PIM-A															
5	2403-PIM-A		0.006	0.328	0.368	0.497	0.107	0.12	0.046	0.415	0.939		1.89	1.89	2.82	2.82
6	2403-PIM-A		0.00587	0.443	0.0105	0.593	0.126	0.131	0.0633	0.509	1.15		1.88	1.88	3.03	3.03
9	2403-PIM-A		0.00639	0.378	0.0107	0.516	0.105	0.116	0.0523	0.427	0.87		1.61	1.61	2.48	2.48
13	2403-PIM-A															
14	2403-PIM-A															
15	2403-PIM-A															
16	2403-PIM-A		0.00436	0.0107	0.0103	0.369	0.109	0.461	0.0534	0.12	0.455		1.14	1.14	1.59	1.59
20	2403-PIM-A															
21	2403-PIM-A		0.0259	1.34	0.0311	0.786	0.213	0.129	0.0675	0.538	1.48		3.13	3.13	4.62	4.62
23	2403-PIM-A		0.0071	0.437	0.0039	0.557	0.119	0.134	0.0584	0.503			1.82	1.82		
36	2403-PIM-A		0.00391	0.216	0.0104	0.358	0.112	0.178	0.083	0.562	1.83		1.52	1.52	3.35	3.35
37	2403-PIM-A		< 0.021	0.416	< 0.039	0.548	0.113	0.131	0.062	0.493	1.25		1.82	1.77	3.07	3.07
38	2403-PIM-A		10.7	837	22.6	1150	237	225	106	1030	1880		3620	3620	5500	5500
41	2403-PIM-A		0.0059	0.42	0.012	0.588	0.127	0.126	0.059	0.513	0.842		1.85	1.85	2.69	2.69
44	2403-PIM-A		< 0.036	0.397		0.533	0.116	0.122	0.0645	0.443			1.71	1.67		
49	2403-PIM-A															
50	2403-PIM-A		0.00444	0.489	0.0121	0.68	0.15	0.152	0.0782	0.551	0.942		2.12	2.12	3.06	3.06
53	2403-PIM-A		< 0.005	0.542	0.0139	0.611	0.126	0.163	0.0658	0.573			2.1	2.09		
58	2403-PIM-A															
44A	2403-PIM-A		< 0.036	0.382		0.527	0.118	0.122	0.0567	0.441			1.68	1.65		
49A	2403-PIM-A															
16*	2403-PIM-A		0.00436	0.369	0.0107	0.461	0.109	0.12	0.0534	0.455	0.937		1.58	1.58	2.52	2.52

EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]

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

Powdered Infant Milk (2403-PIM-A)

HBCDD - Results

LC	Sample	Result µg/kg fat	**(+/–)-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)
5	2403-PIM-A		0.294	0.0348	0.044		0.373	0.373	
6	2403-PIM-A								0.413
8	2403-PIM-A		0.268	0.036	0.02		0.324	0.324	
13	2403-PIM-A								
14	2403-PIM-A								
16	2403-PIM-A		0.303	0.0374	0.0332		0.373	0.373	
19	2403-PIM-A		0.318	< 0.163	< 0.163		0.645	0.318	
20	2403-PIM-A								
21	2403-PIM-A		0.343	< 0.043	< 0.043		0.429	0.343	
37	2403-PIM-A		0.305	0.0495	0.0353		0.39	0.39	
38	2403-PIM-A		1.96	< 1.8	< 1.8		5.56	1.96	
43	2403-PIM-A		0.398	0.051	0.123		0.571	0.571	
44	2403-PIM-A		0.252	< 0.037	< 0.037		0.326	0.252	
49	2403-PIM-A								
50	2403-PIM-A		0.361	0.048	0.05		0.46	0.46	
51	2403-PIM-A								

EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]

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

06 March 2026

Annex 3a: Participants' z-scores of PBDEs and HBCDDs on product basis

Test sample - Powdered Infant Milk A (2403-PIM-A)

Z-scores of individual substances and sum parameters

Calculation of z-score on basis of assigned value

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

x_a: assigned value

x: participant's result

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

20%: Evaluated individual substances and sum parameters

EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]

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

Powdered Infant Milk A (2403-PIM-A)

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** including BDE-209 (lb)
1	2403-PIM-A			0.6		0.9	0.8	0.1	1.0	1.0		0.8	0.8		
4	2403-PIM-A			0.4		0.0	2.0	1.2	-0.1	0.6	1.2		0.5		0.9
5	2403-PIM-A			-1.2		-0.7	-0.6	-0.7	-1.5	-0.8	-0.6	0.1	0.1	0.1	0.0
6	2403-PIM-A			-0.5		-0.4	-0.5	-1.0	-0.6	-0.5	-0.2	-0.4	-0.5	-0.2	-0.3
9	2403-PIM-A			-1.2		-1.1	-1.3	-1.4	-1.4	-1.3	-1.3	-1.2	-1.2	-1.1	-1.2
13	2403-PIM-A														
14	2403-PIM-A			-0.6		-0.4	-1.0	0.2	-0.3	0.0	-0.9	-0.3	-0.3	-0.4	-0.5
15	2403-PIM-A			2.5		1.9	2.1	1.7	1.0	0.2	1.6	1.7	1.7	1.8	1.8
16	2403-PIM-A			0.0		-0.3	0.2	0.0	0.0	0.3	0.1	0.1	0.1	0.3	0.2
20	2403-PIM-A			0.3		0.1	-0.5	0.0	4.8	0.0	0.5				
21	2403-PIM-A			9.7		1.4	3.2	-0.7	0.0	0.0	1.5	3.0	3.0	2.7	2.6
23	2403-PIM-A			0.2		0.0	0.0	-0.1	-0.3	0.2		0.1	0.1		
36	2403-PIM-A			-2.4		-1.8	-0.3	1.5	1.8	0.8	3.9	-0.7	-0.7	1.2	1.1
37	2403-PIM-A			0.0		-0.1	-0.2	-0.2	0.1	0.1	1.1	0.1	0.0	0.7	0.6
38	2403-PIM-A			5462.3		5550.6	5422.6	4467.9	4646.9	5755.0	4919.5	5520.2	5508.1	5490.0	5404.6
41	2403-PIM-A			-0.3		-0.1	-0.1	-0.7	-0.6	-0.1	-1.2	-0.2	-0.2	-0.4	-0.5
44	2403-PIM-A			0.0		0.0	0.2	-0.3	0.5	-0.2		0.0	0.0		
49	2403-PIM-A			0.9		-0.4	0.3	-0.3	-0.6	-0.2	-0.8	0.1	0.0	-0.1	-0.2
50	2403-PIM-A			0.1		0.3	0.5	-0.1	0.6	0.0	-1.0	0.2	0.2	-0.1	-0.2
53	2403-PIM-A			2.2		1.0	0.9	1.6	0.9	1.5		1.5	1.5		
58	2403-PIM-A			-4.8		-2.8	-0.9	0.1	-0.5	-1.7	-3.1	-2.0	-2.0	-2.3	-2.4
44A	2403-PIM-A			-0.1		0.0	0.3	-0.3	-0.1	-0.2		-0.1	-0.1		
49A	2403-PIM-A			2.4		0.9	1.1	1.1	1.3	1.2	2.4	1.5	1.3	2.1	1.9

EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]

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

Powdered Infant Milk A (2403-PIM-A)

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)
5	2403-PIM-A		-0.2				-0.3	0.5	
6	2403-PIM-A								-1.8
8	2403-PIM-A		-0.6				-0.9	-0.2	
13	2403-PIM-A								
14	2403-PIM-A		-0.1				-0.4	0.0	
16	2403-PIM-A		0.3				0.1	0.4	
19	2403-PIM-A		0.1				3.0	-0.4	
20	2403-PIM-A								2.7
21	2403-PIM-A		0.2				0.1	-0.2	
37	2403-PIM-A		-0.3				-0.3	0.5	
38	2403-PIM-A		12.8				34.3	11.2	
43	2403-PIM-A		1.8				2.6	3.8	
44	2403-PIM-A		-0.6				-0.5	-1.0	
49	2403-PIM-A		0.2				0.1	-0.3	
50	2403-PIM-A		0.3				0.2	1.1	
51	2403-PIM-A		0.2				2.1	-0.3	

EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]

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

Powdered Infant Milk A (2403-PIM-A)

Lipid content - Z-scores

LC	Sample	Z-score [σ_p = 10 %]	Lipid content	Lipid content	Lipid content
			PBDE	HBCDD	Mean
1	2403-PIM-A		0.6		0.6
4	2403-PIM-A				
5	2403-PIM-A		-0.1	-0.1	-0.1
6	2403-PIM-A		-0.5	-0.5	-0.5
8	2403-PIM-A			-0.1	-0.1
9	2403-PIM-A				
13	2403-PIM-A				
14	2403-PIM-A				
16	2403-PIM-A			0.6	0.6
15	2403-PIM-A		-0.8		-0.8
16	2403-PIM-A		1.6		1.6
19	2403-PIM-A			-0.3	-0.3
20	2403-PIM-A		-0.8	-0.8	-0.8
21	2403-PIM-A		-0.7	-0.7	-0.7
23	2403-PIM-A		0.2		0.2
36	2403-PIM-A		0.4		0.4
37	2403-PIM-A		0.2	-0.6	-0.2
38	2403-PIM-A		-4.6	-4.6	-4.6
41	2403-PIM-A		-0.6		-0.6
43	2403-PIM-A			0.4	0.4
44	2403-PIM-A		0.8	0.8	0.8
49	2403-PIM-A				
50	2403-PIM-A		-1.1	-1.0	-1.0
53	2403-PIM-A		1.3		1.3
58	2403-PIM-A				
44A	2403-PIM-A		0.8		0.8
49A	2403-PIM-A				

EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]

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

06 March 2026

Annex 3b: Participants' z-scores of PBDEs and HBCDDs on fat basis

Test sample - Powdered Infant Milk A (2403-PIM-A)

Z-scores of individual substances and sum parameters

Calculation of z-score on basis of assigned value

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

x_a: assigned value

x: participant's result

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

20%: Evaluated individual substances and sum parameters

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

EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Powdered Infant Milk (2403-PIM-A)
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** including BDE-209 (lb)
1	2403-PIM-A			0.4		0.7	0.5	0.2	0.7	0.5		0.5	0.5		
4	2403-PIM-A														
5	2403-PIM-A			-1.1		-0.6	-0.5	-0.6	-1.4	-0.9	-0.5	0.2	0.2	-0.1	-0.1
6	2403-PIM-A			0.3		0.3	0.3	-0.1	0.0	0.0	0.5	0.2	0.2	0.3	0.3
9	2403-PIM-A			-0.5		-0.4	-0.6	-0.7	-0.9	-0.8	-0.9	-0.6	-0.6	-0.6	-0.6
13	2403-PIM-A														
14	2403-PIM-A														
15	2403-PIM-A														
16	2403-PIM-A			-4.9		-1.7	-0.4	12.1	-0.8	-3.8	-2.8	-1.9	-1.9	-2.2	-2.2
20	2403-PIM-A														
21	2403-PIM-A			11.0		2.0	3.9	-0.2	0.3	0.3	2.0	3.6	3.6	3.1	3.1
23	2403-PIM-A			0.2		0.0	0.0	0.0	-0.4	0.0		0.0	0.0		
36	2403-PIM-A			-2.4		-1.8	-0.3	1.6	1.6	0.5	3.7	-0.8	-0.8	0.9	0.9
37	2403-PIM-A			0.0		-0.1	-0.3	-0.1	-0.1	-0.1	1.0	0.0	-0.1	0.4	0.4
38	2403-PIM-A			10007.0		10226.3	9953.0	8328.3	8367.8	10152.8	8947.4	9940.1	9995.0	9644.1	9644.1
41	2403-PIM-A			0.0		0.2	0.3	-0.3	-0.3	0.1	-1.0	0.1	0.1	-0.3	-0.3
44	2403-PIM-A			-0.3		-0.3	-0.1	-0.5	0.1	-0.6		-0.3	-0.4		
49	2403-PIM-A														
50	2403-PIM-A			0.8		1.0	1.3	0.6	1.2	0.4	-0.5	0.8	0.9	0.4	0.4
53	2403-PIM-A			1.5		0.4	0.3	1.0	0.2	0.7		0.8	0.8		
58	2403-PIM-A														
44A	2403-PIM-A			-0.4		-0.3	0.0	-0.5	-0.5	-0.7		-0.4	-0.4		
49A	2403-PIM-A														
16*	2403-PIM-A			-0.6		-0.9	-0.4	-0.6	-0.8	-0.5	-0.5	-0.7	-0.6	-0.6	-0.6

EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]

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

Powdered Infant Milk (2403-PIM-A)

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)
5	2403-PIM-A		-0.3	-0.9		-0.3	0.3	
6	2403-PIM-A							0.2
8	2403-PIM-A		-0.7	-0.7		-0.9	-0.4	
13	2403-PIM-A							
14	2403-PIM-A							
16	2403-PIM-A		-0.2	-0.6		-0.3	0.3	
19	2403-PIM-A		0.1			3.1	-0.5	
20	2403-PIM-A							
21	2403-PIM-A		0.5			0.4	-0.2	
37	2403-PIM-A		-0.1	0.9		-0.1	0.5	
38	2403-PIM-A		26.2			65.2	22.7	
43	2403-PIM-A		1.3	1.1		2.2	3.1	
44	2403-PIM-A		-1.0			-0.9	-1.4	
49	2403-PIM-A							
50	2403-PIM-A		0.7	0.7		0.8	1.5	
51	2403-PIM-A							

EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]

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

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Annex 4: Participants' z-scores of PBDEs and HBCDDs on product basis and fat basis

Test sample - Powdered Infant Milk A (2403-PIM-A)

Z-scores of individual substances and sum parameters

Calculation of z-score on basis of assigned value

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

x_a: assigned value

x: participant's result

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

20%: Evaluated individual substances and sum parameters

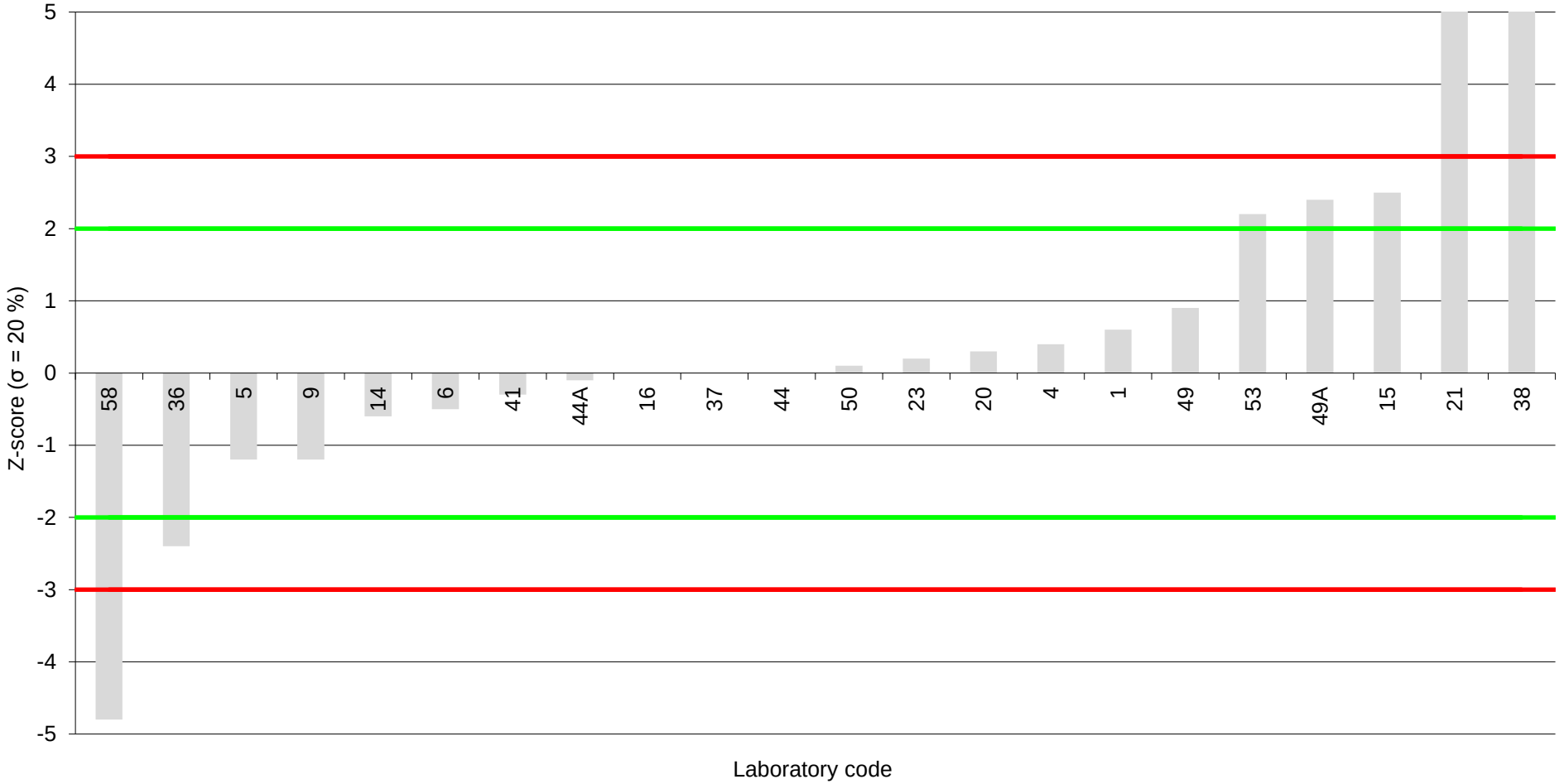
± 2 z-scores:

± 3 z-scores:

Powdered Infant Milk A (2403-PIM-A)

BDE-47

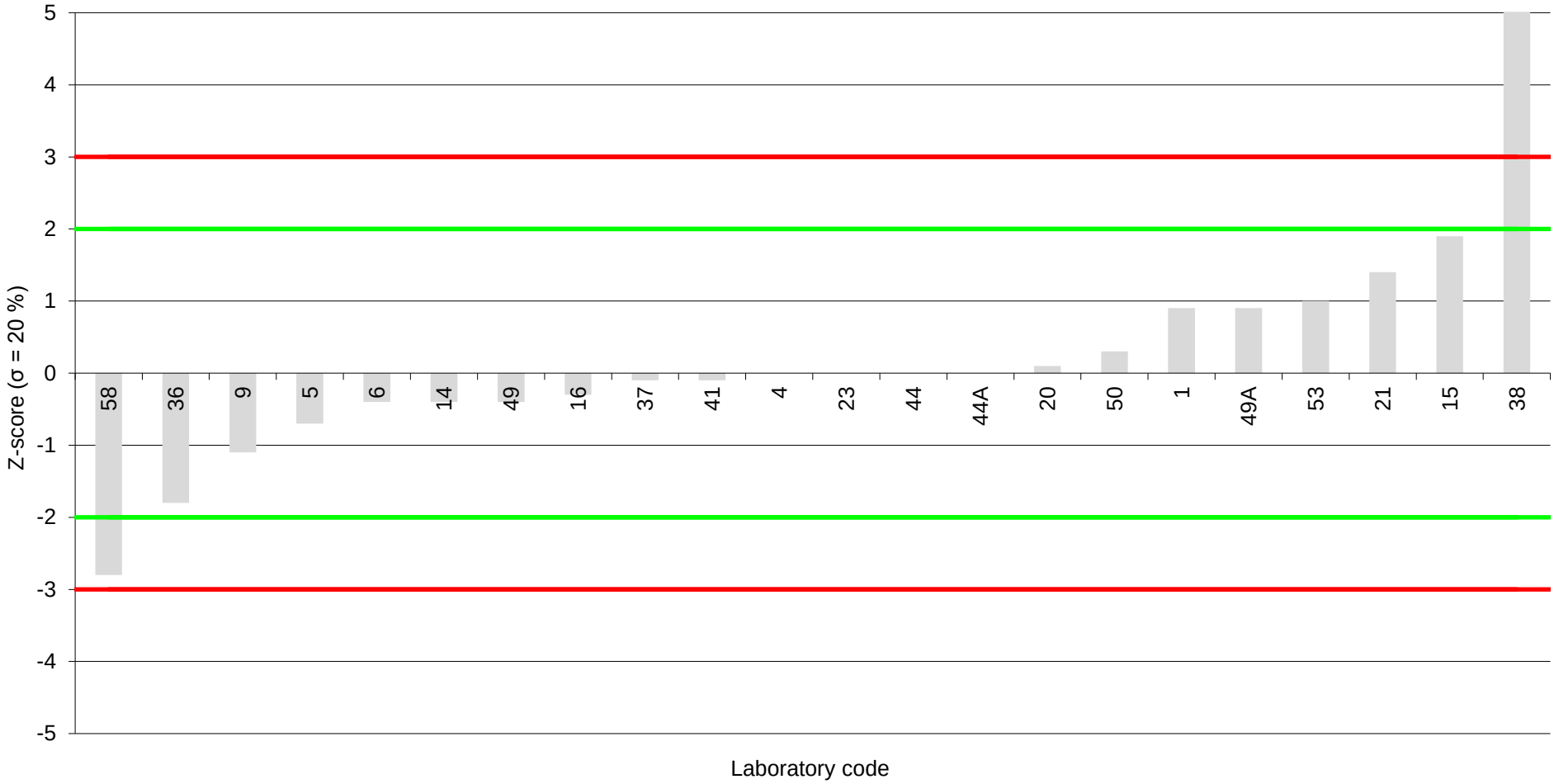
Assigned value: 0.107 µg/kg product



Powdered Infant Milk A (2403-PIM-A)

BDE-99

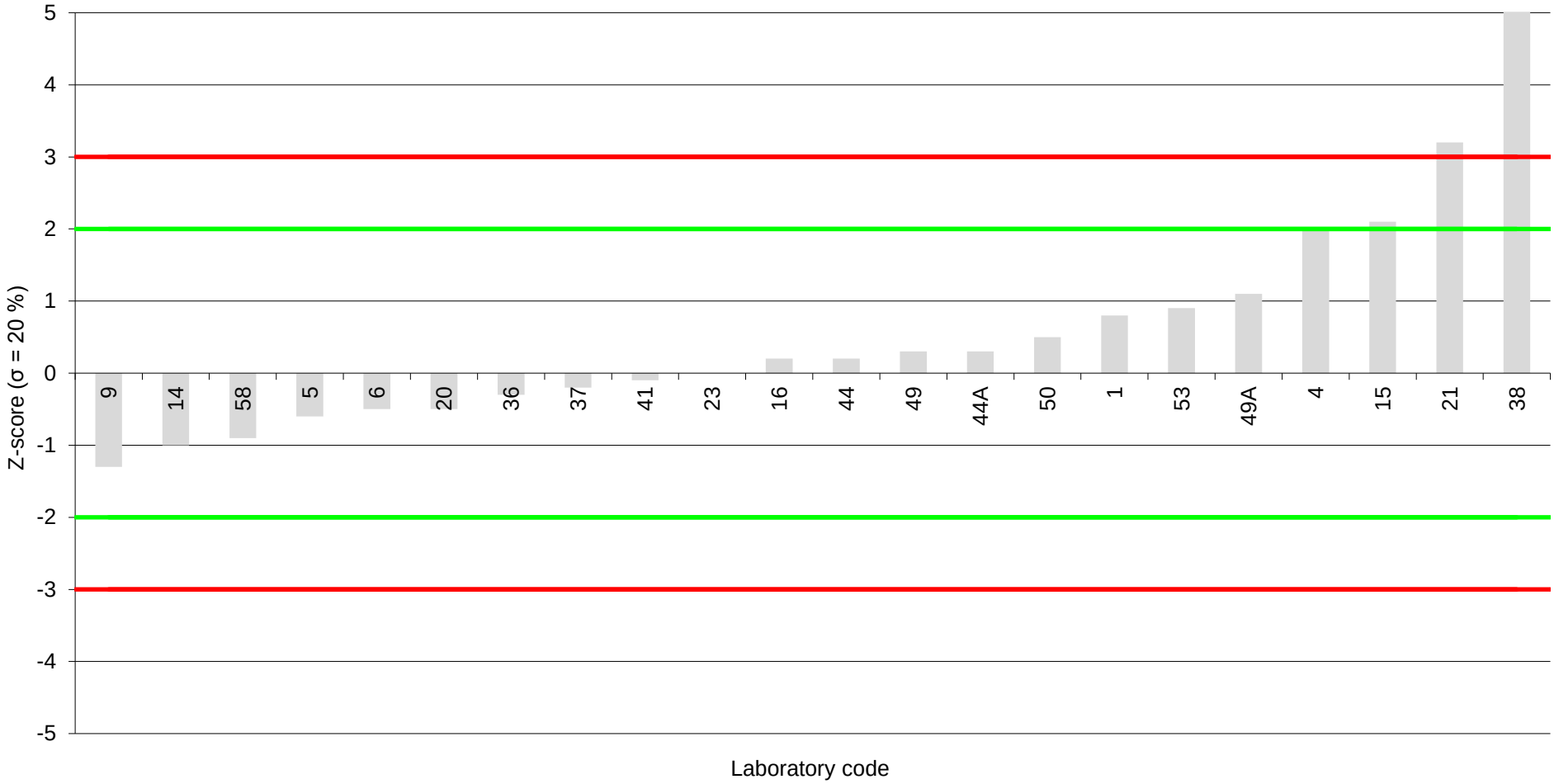
Assigned value: 0.144 µg/kg product



Powdered Infant Milk A (2403-PIM-A)

BDE-100

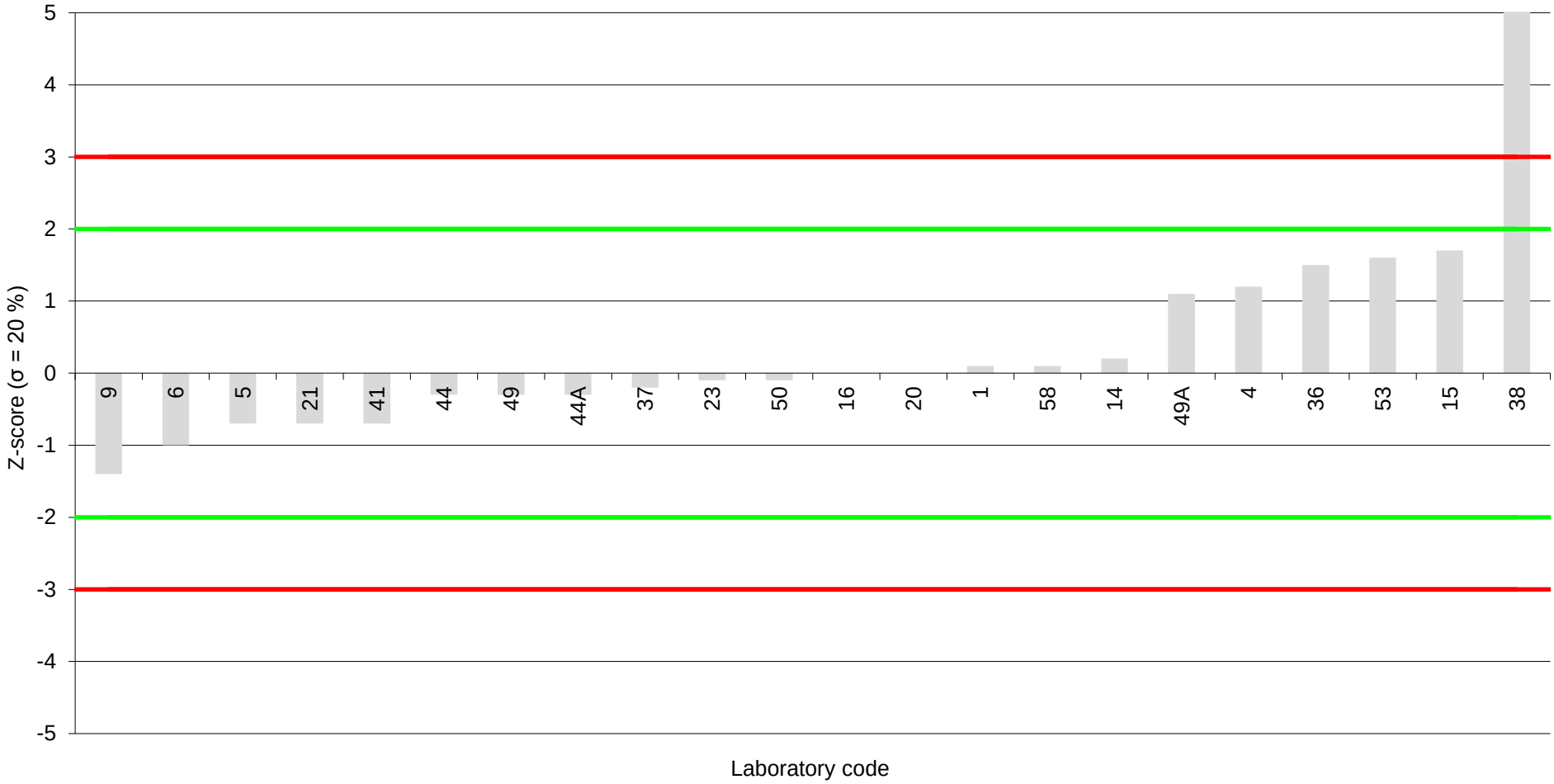
Assigned value: 0.0304 µg/kg product



Powdered Infant Milk A (2403-PIM-A)

BDE-153

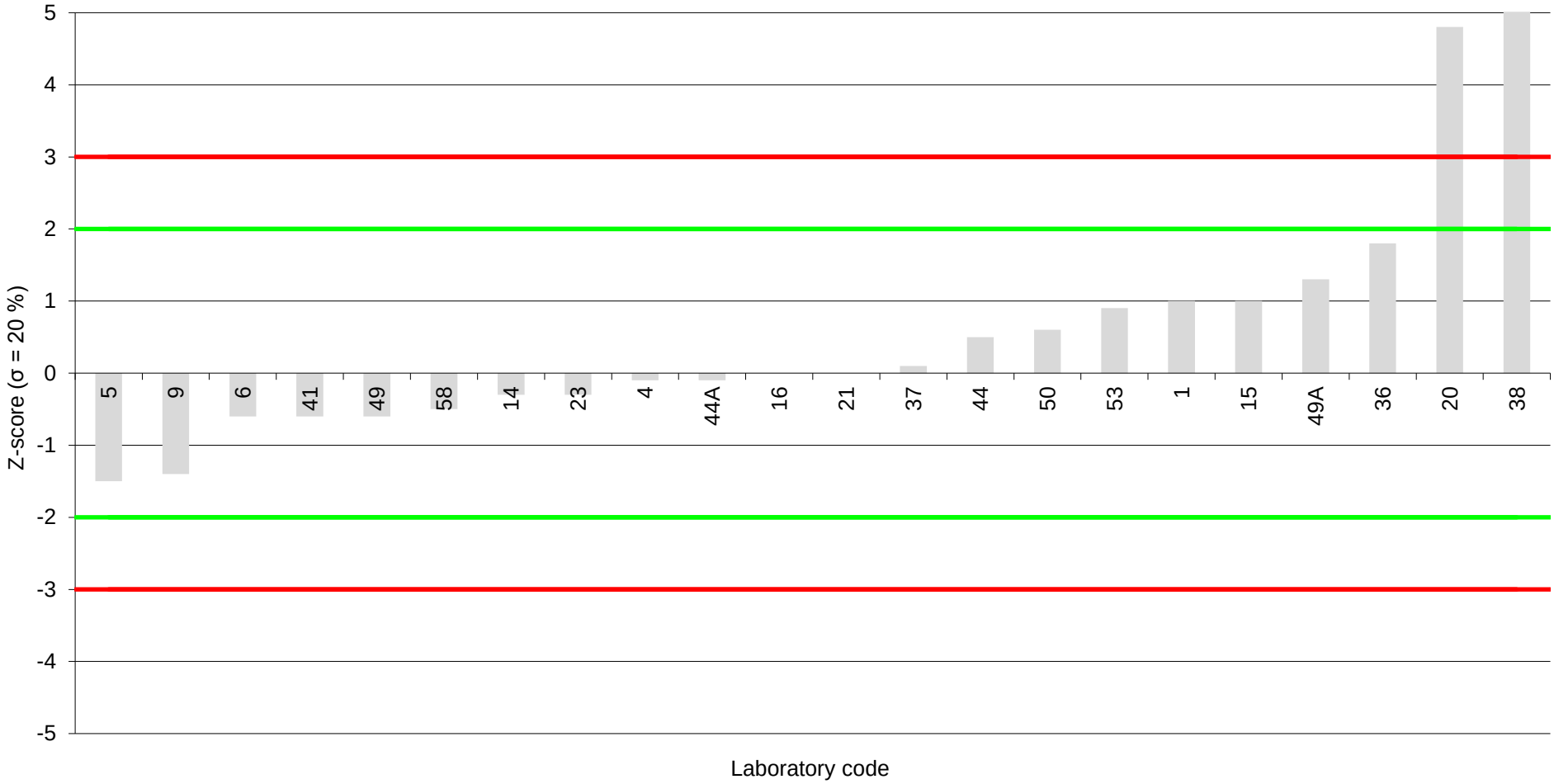
Assigned value: 0.0351 µg/kg product



Powdered Infant Milk A (2403-PIM-A)

BDE-154

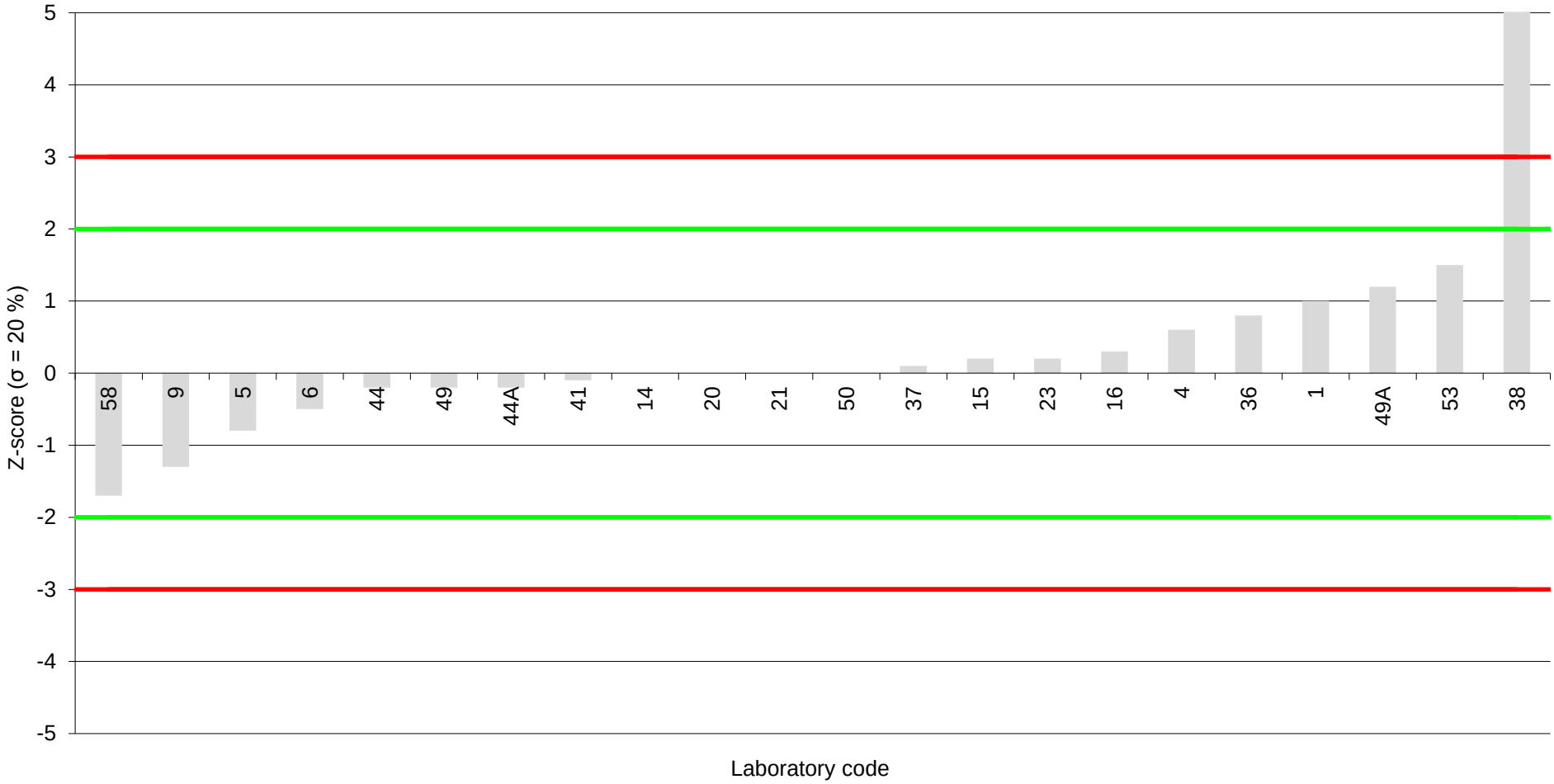
Assigned value: 0.0158 µg/kg product



Powdered Infant Milk A (2403-PIM-A)

BDE-183

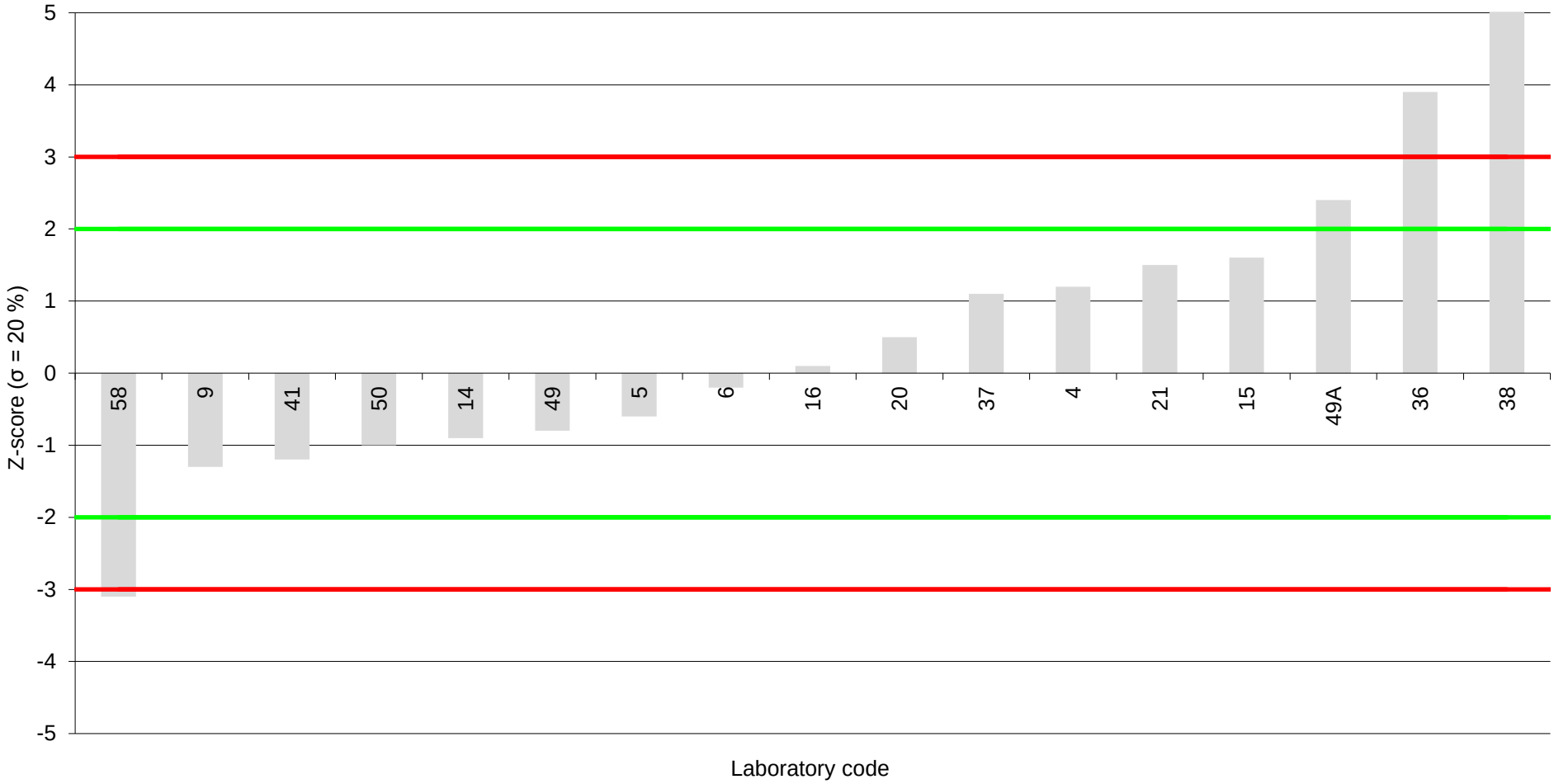
Assigned value: 0.125 µg/kg product



Powdered Infant Milk A (2403-PIM-A)

BDE-209

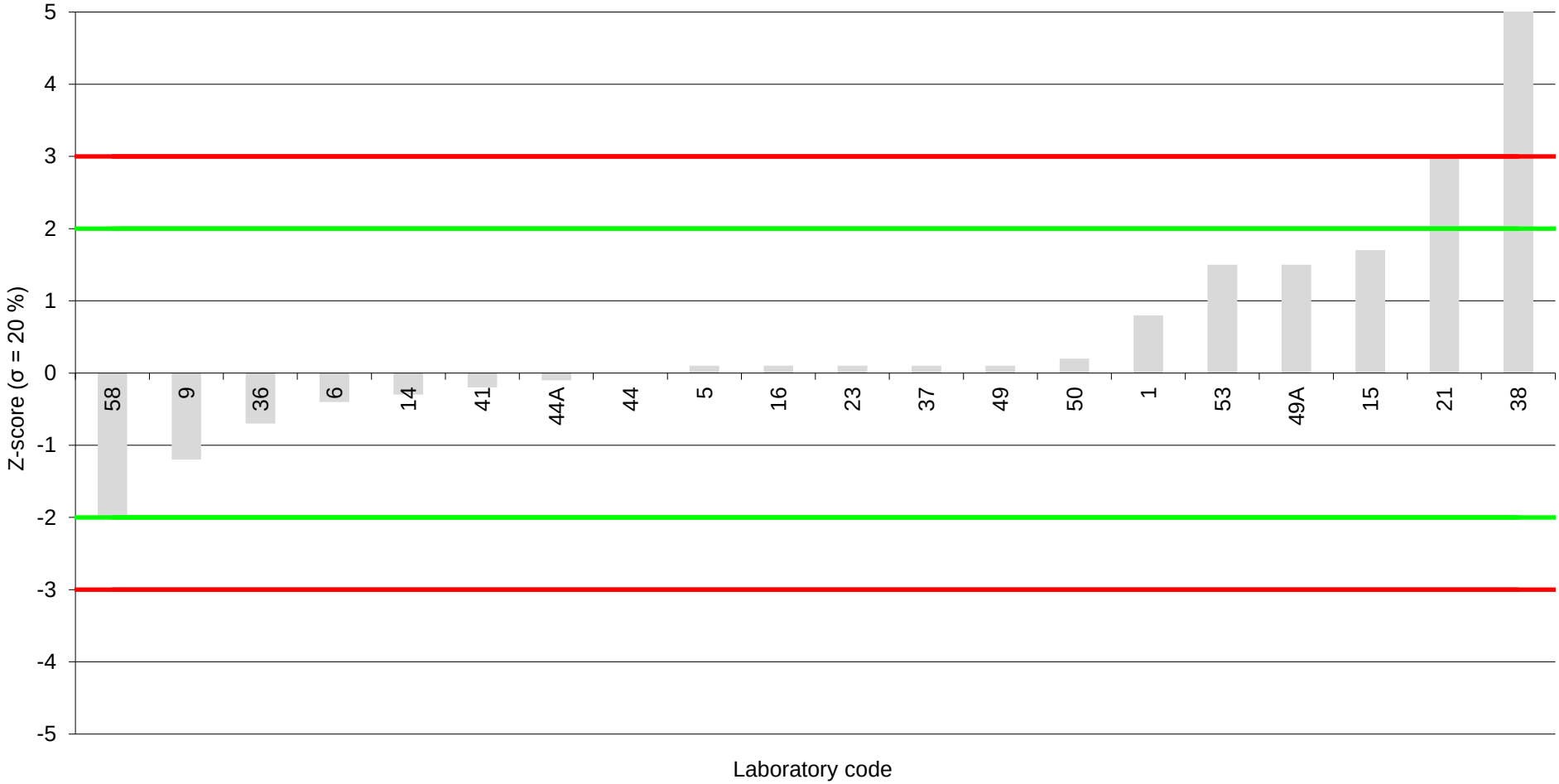
Assigned value: 0.265 µg/kg product



Powdered Infant Milk A (2403-PIM-A)

Sum of PBDE without BDE-209 ub

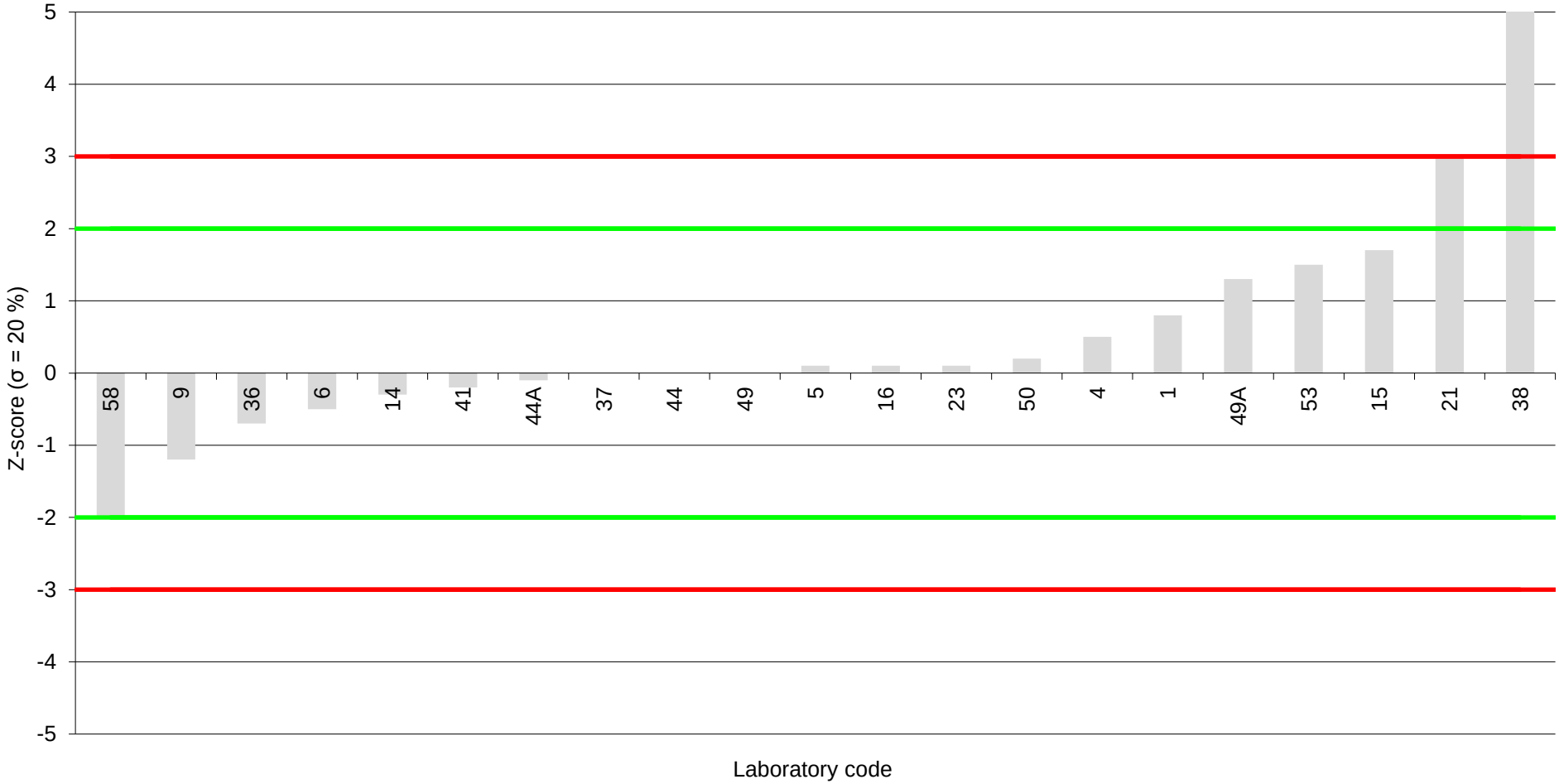
Assigned value: 0.457 µg/kg product



Powdered Infant Milk A (2403-PIM-A)

Sum of PBDE without BDE-209 lb

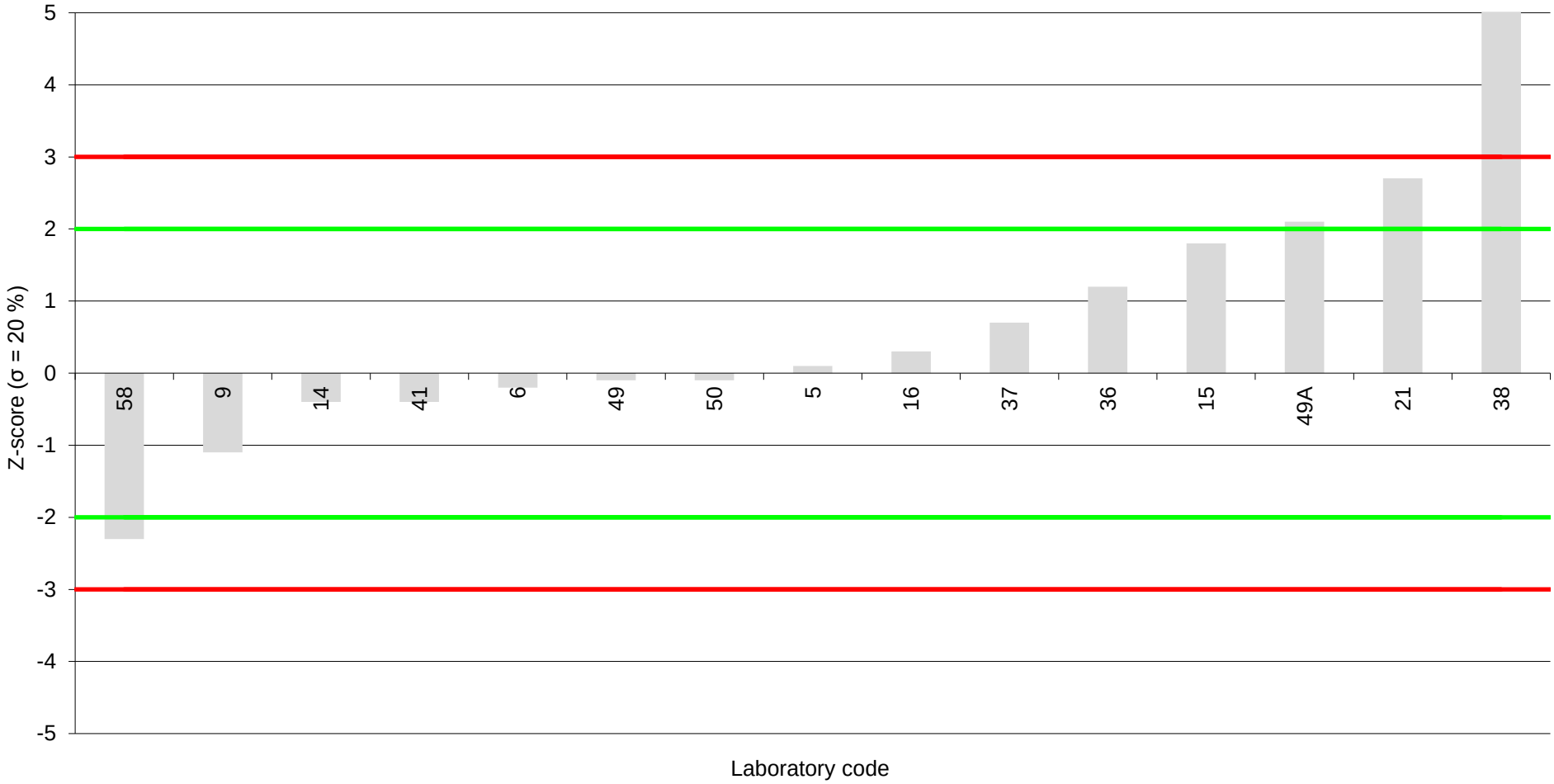
Assigned value: 0.458 µg/kg product



Powdered Infant Milk A (2403-PIM-A)

Sum of PBDE including BDE-209 ub

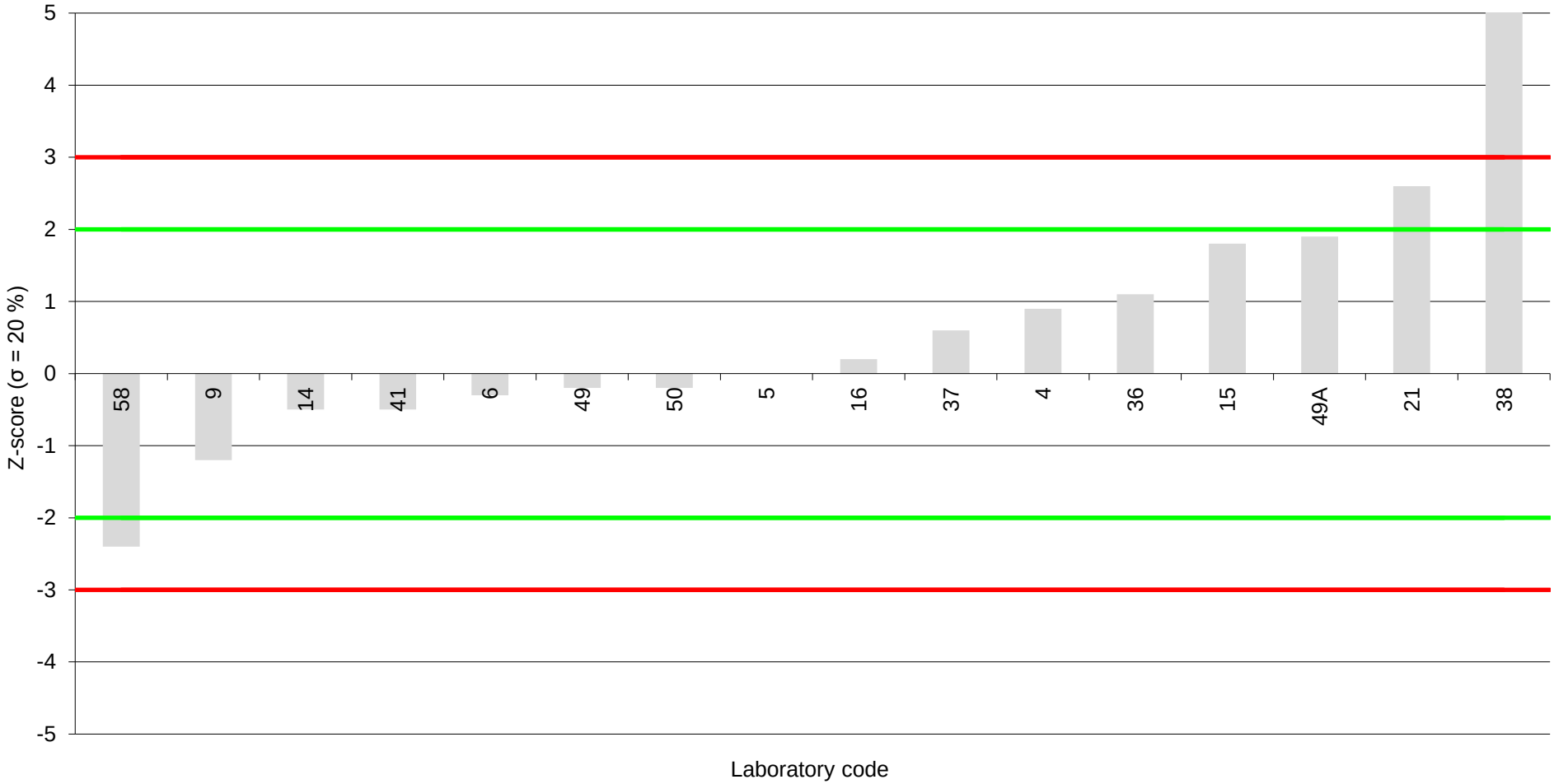
Assigned value: 0.697 µg/kg product



Powdered Infant Milk A (2403-PIM-A)

Sum of PBDE including BDE-209 lb

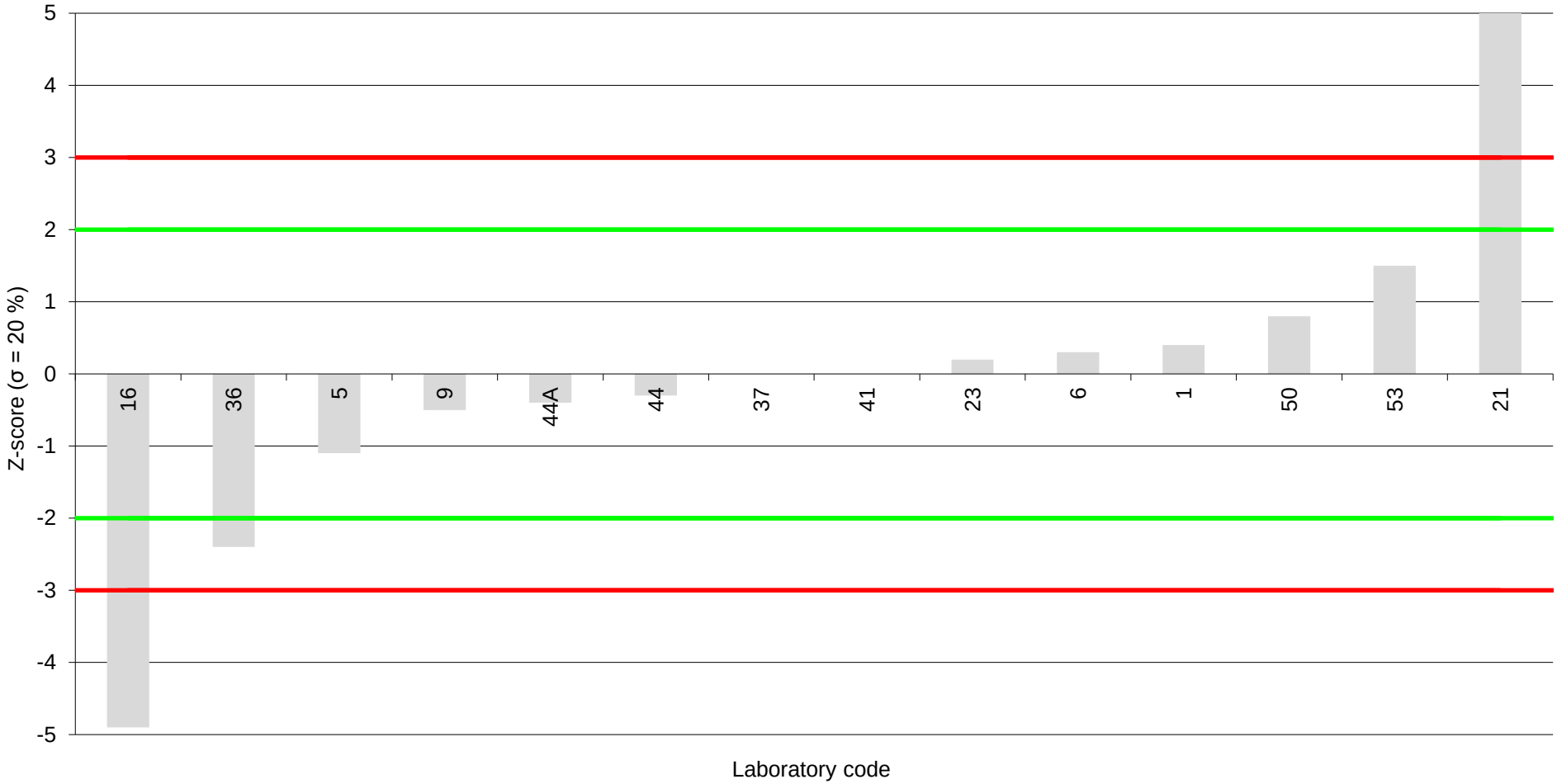
Assigned value: 0.708 µg/kg product



Powdered Infant Milk A (2403-PIM-A)

BDE-47

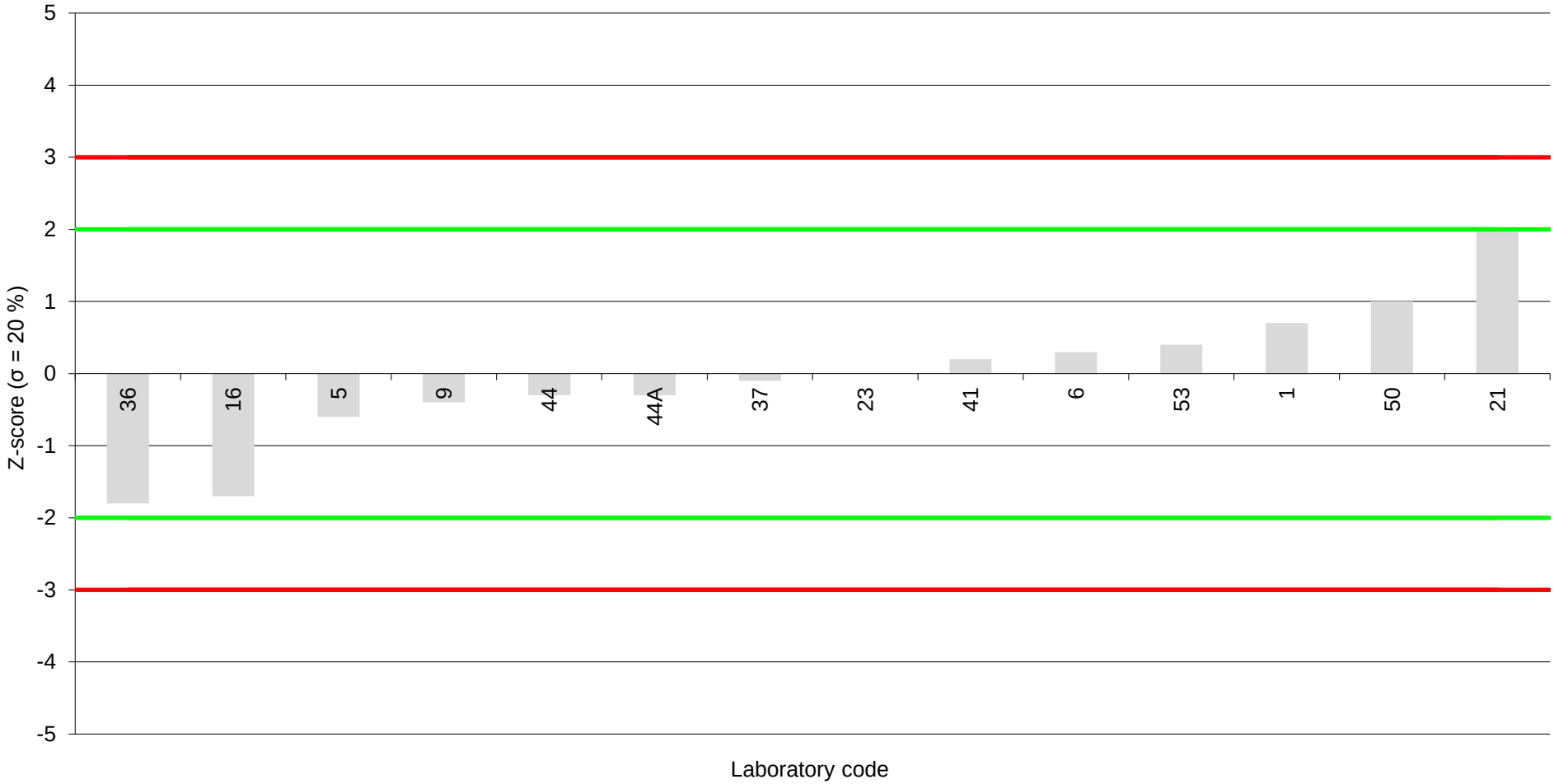
Assigned value: 0.418 µg/kg fat



Powdered Infant Milk A (2403-PIM-A)

BDE-99

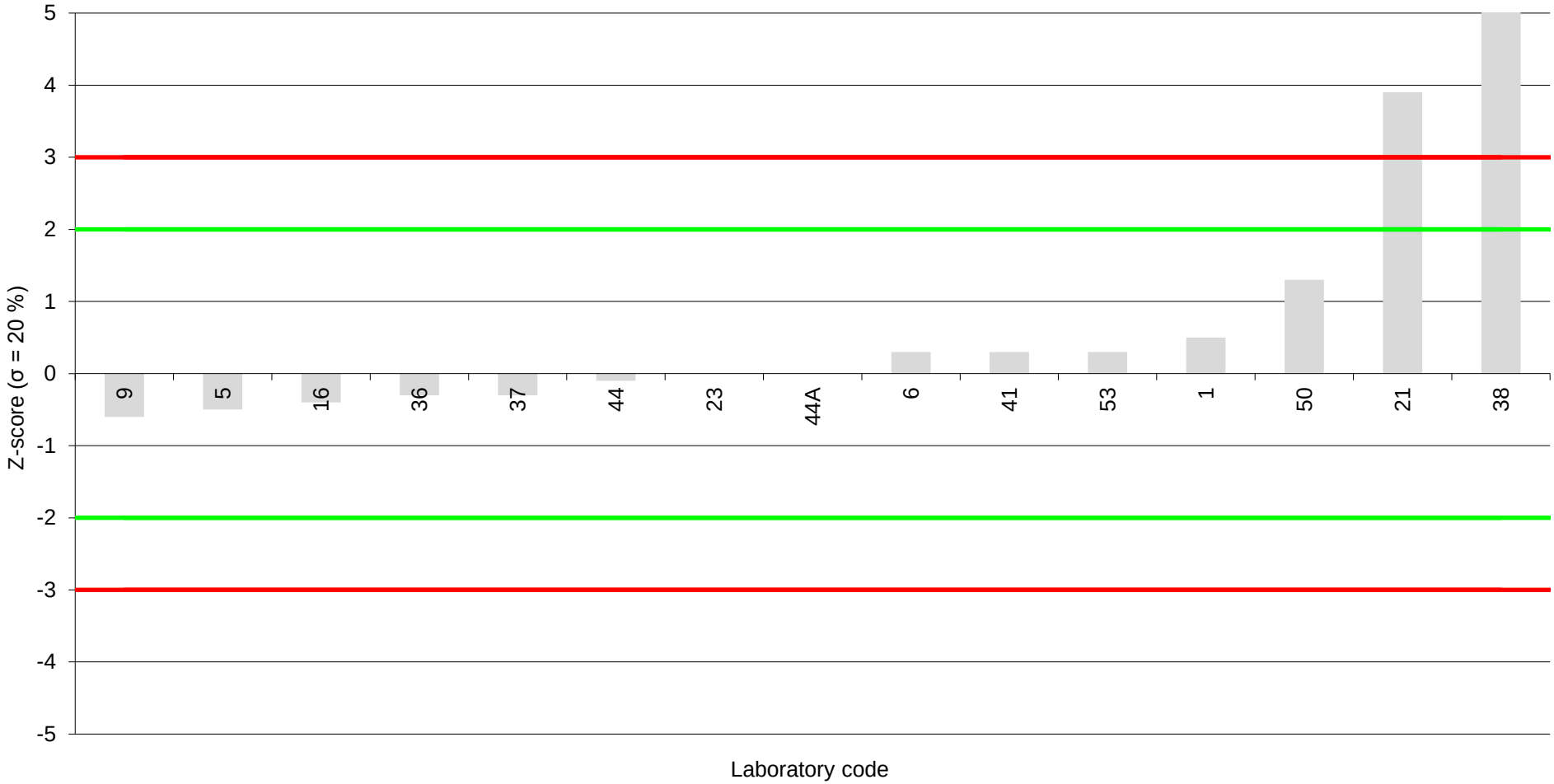
Assigned value: 0.562 µg/kg fat



Powdered Infant Milk A (2403-PIM-A)

BDE-100

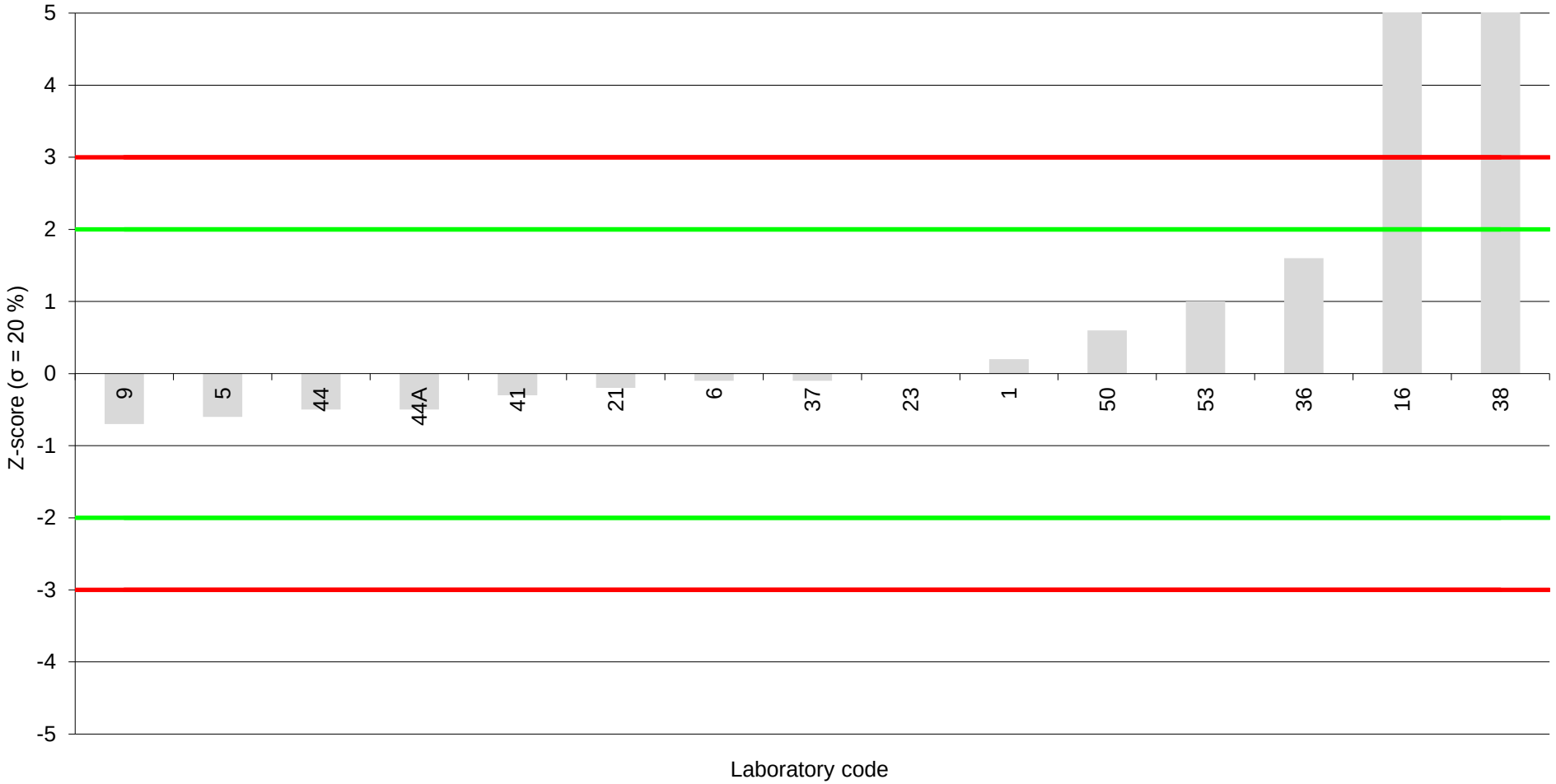
Assigned value: 0.119 µg/kg fat



Powdered Infant Milk A (2403-PIM-A)

BDE-153

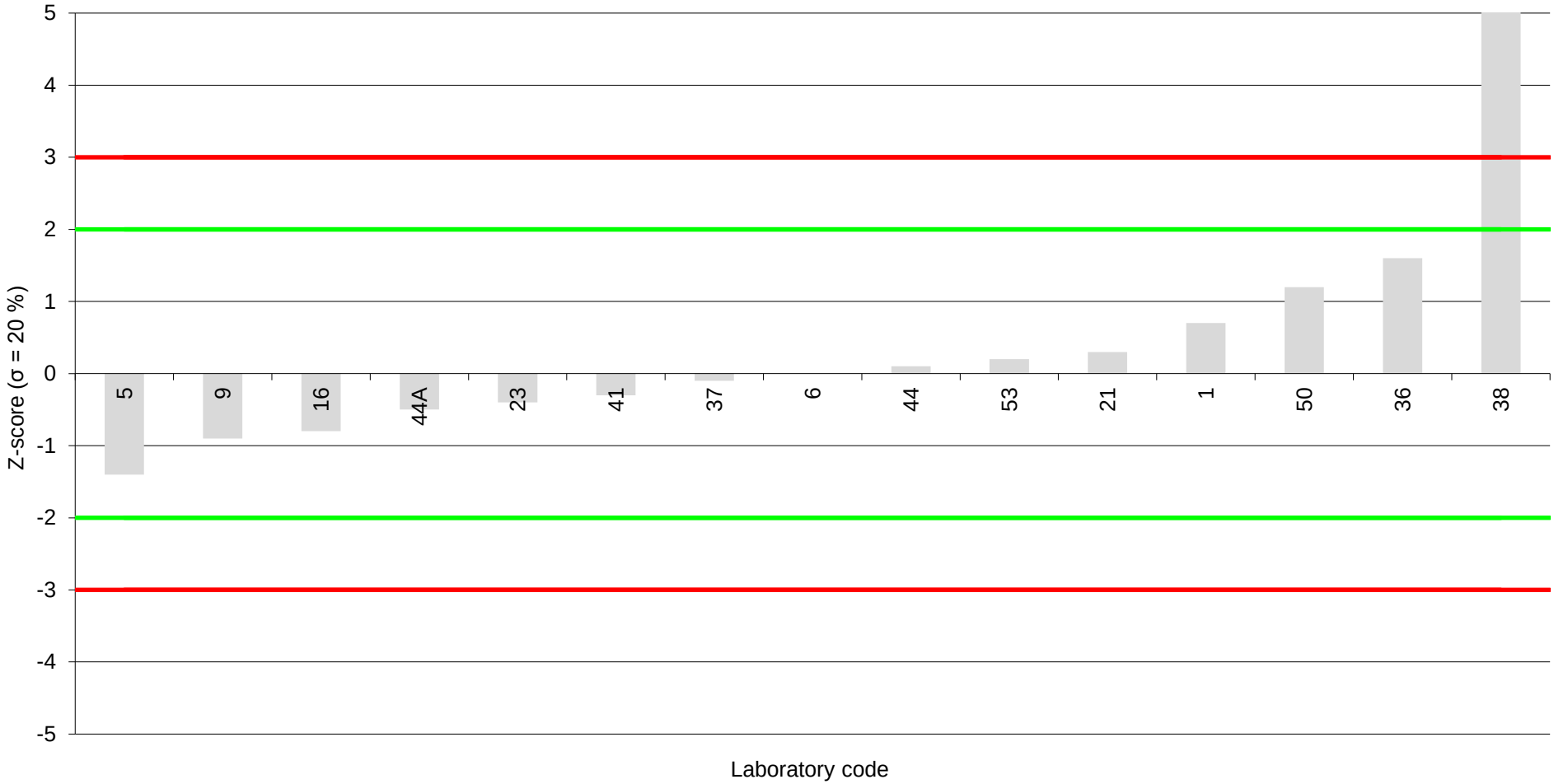
Assigned value: 0.135 µg/kg fat



Powdered Infant Milk A (2403-PIM-A)

BDE-154

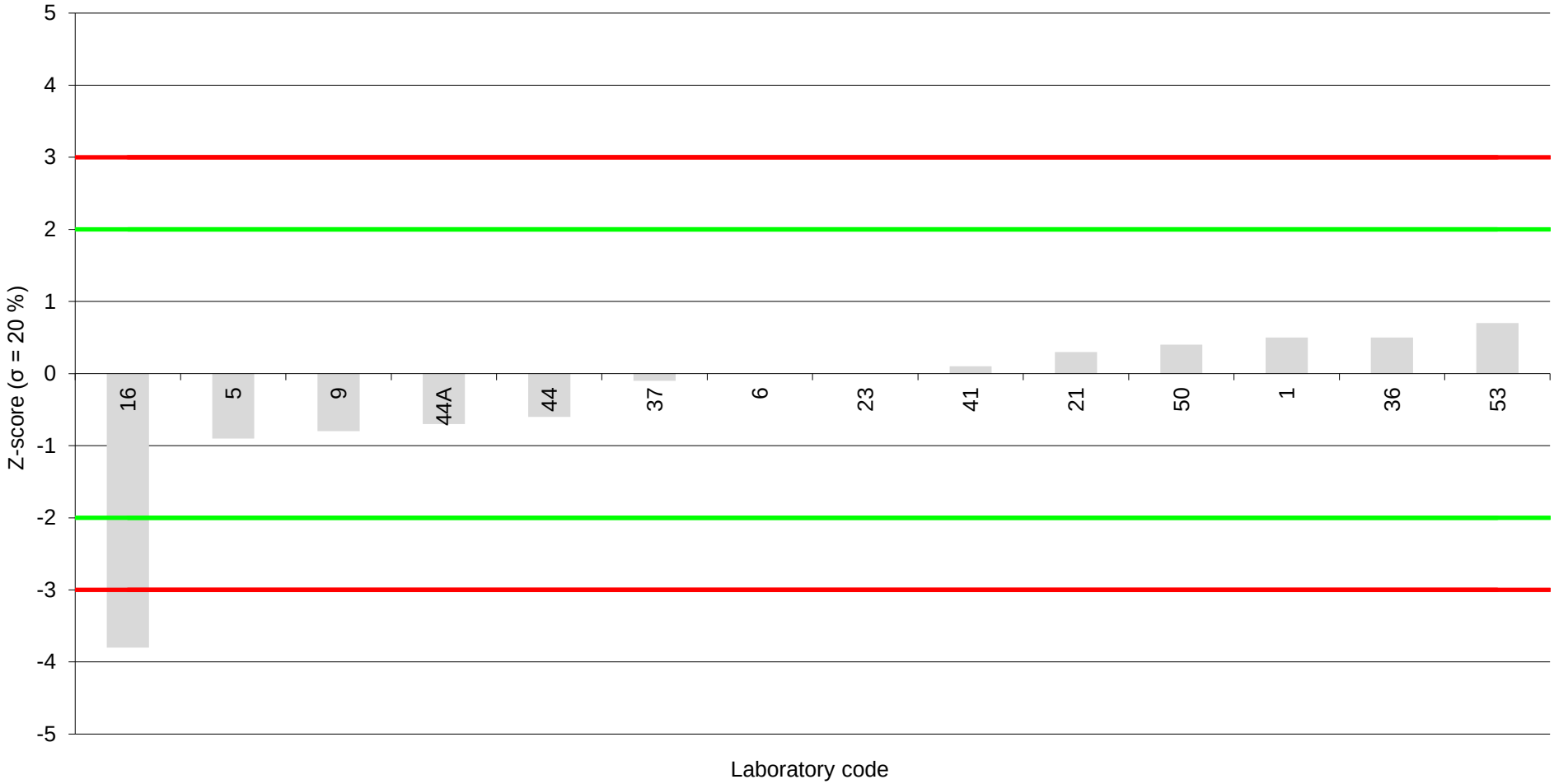
Assigned value: 0.0633 µg/kg fat



Powdered Infant Milk A (2403-PIM-A)

BDE-183

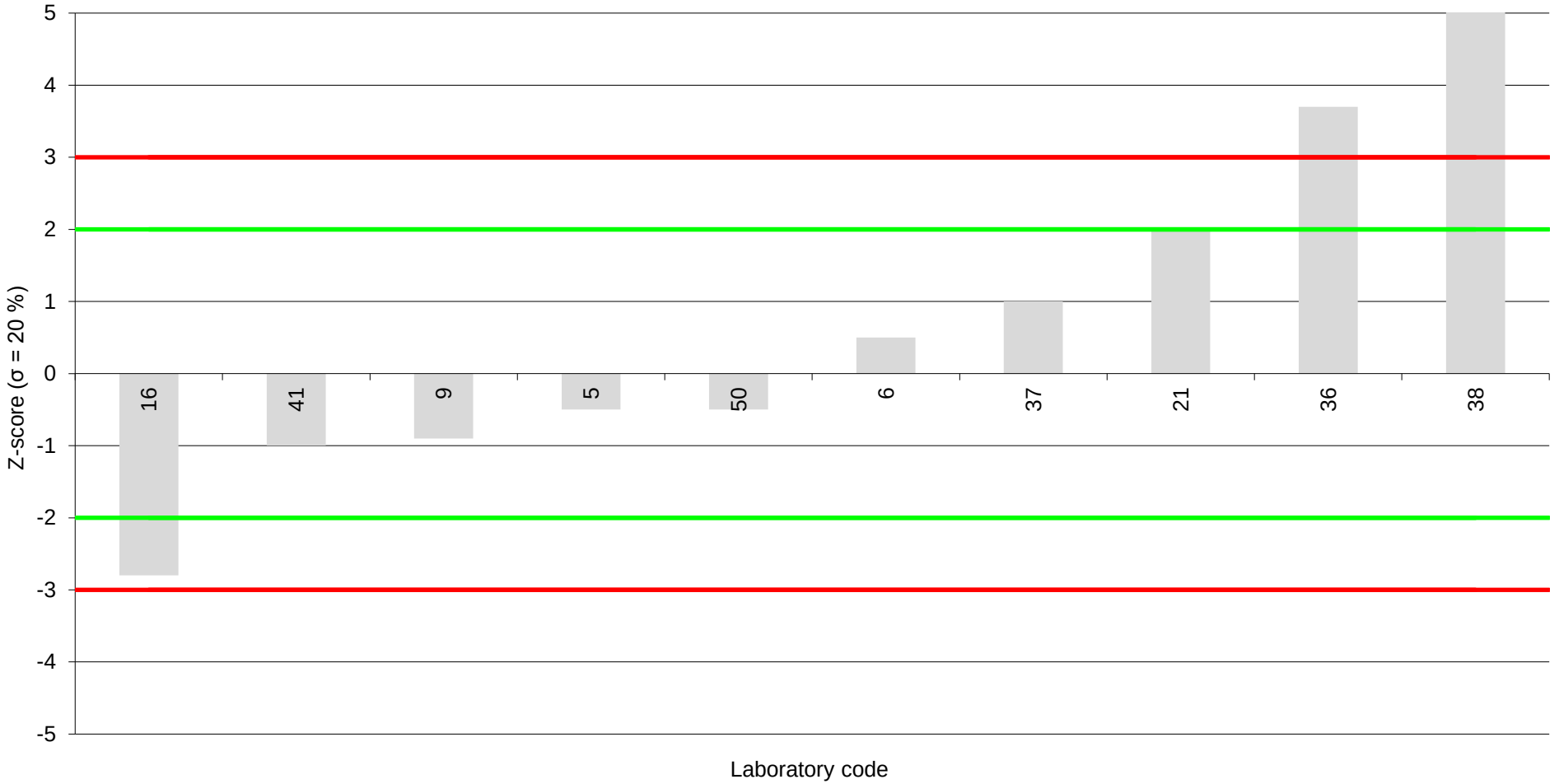
Assigned value: 0.507 µg/kg fat



Powdered Infant Milk A (2403-PIM-A)

BDE-209

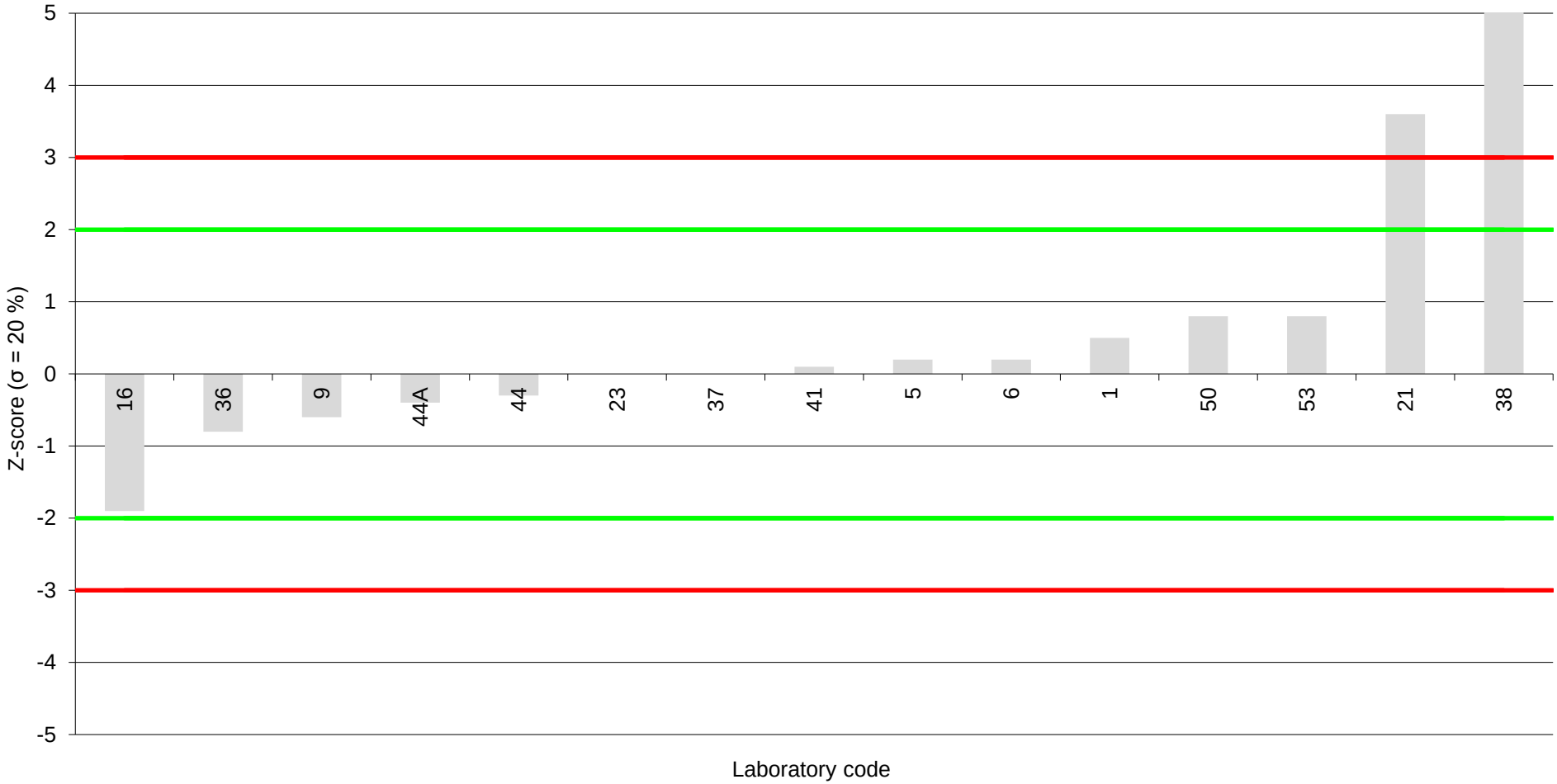
Assigned value: 1.05 µg/kg fat



Powdered Infant Milk A (2403-PIM-A)

Sum of PBDE without BDE-209 ub

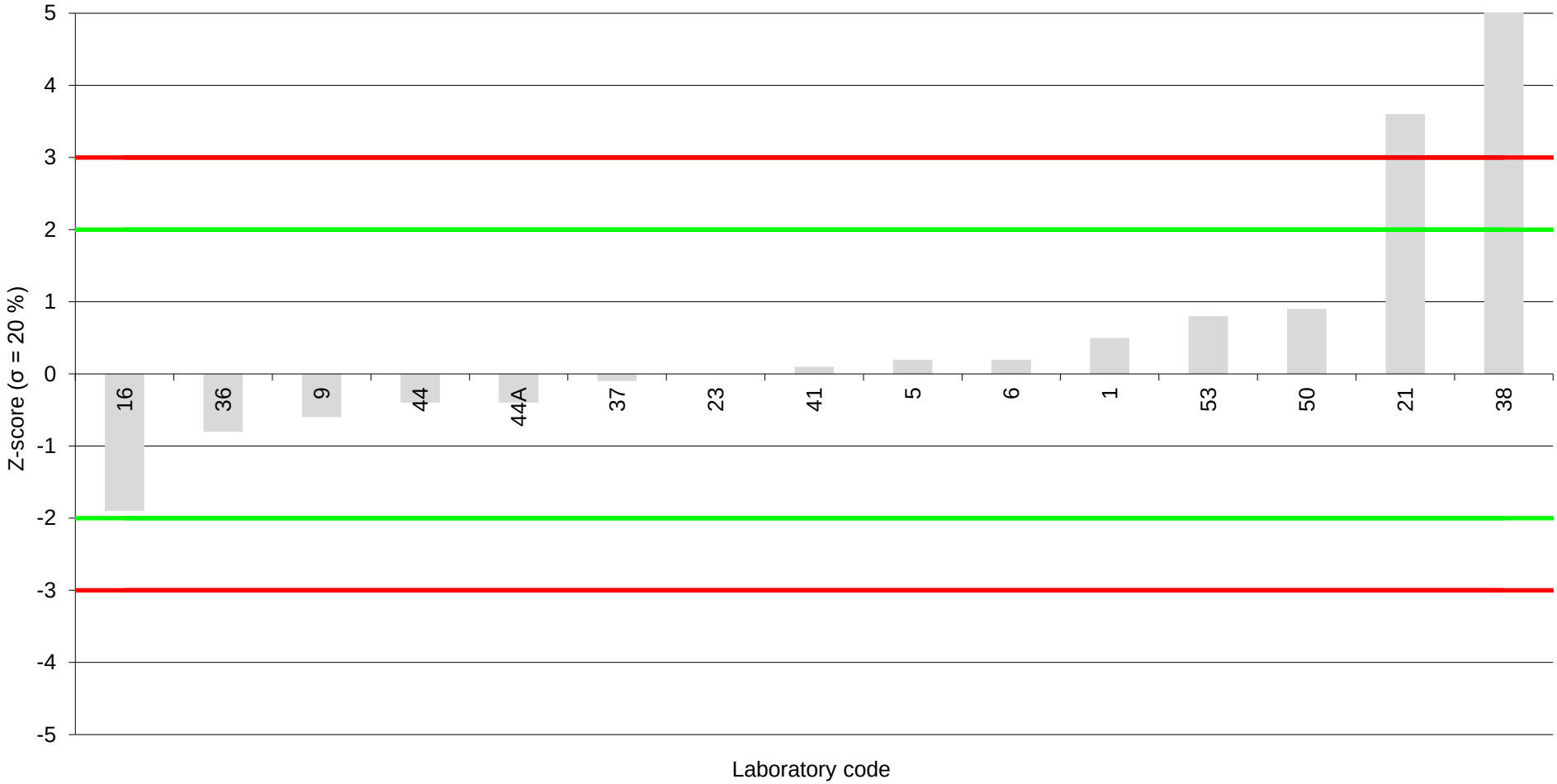
Assigned value: 1.82 µg/kg fat



Powdered Infant Milk A (2403-PIM-A)

Sum of PBDE without BDE-209 lb

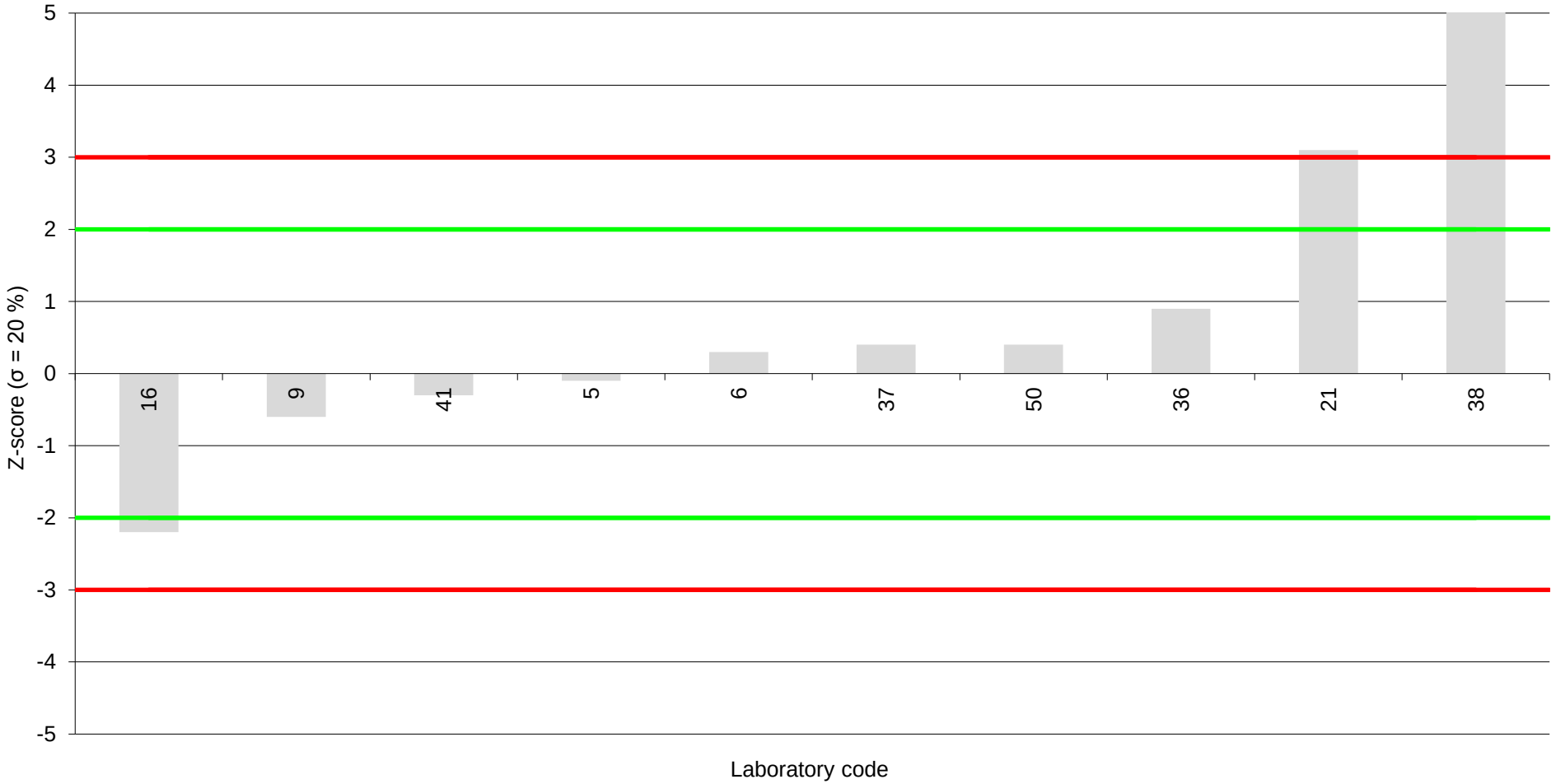
Assigned value: 1.81 µg/kg fat



Powdered Infant Milk A (2403-PIM-A)

Sum of PBDE including BDE-209 ub

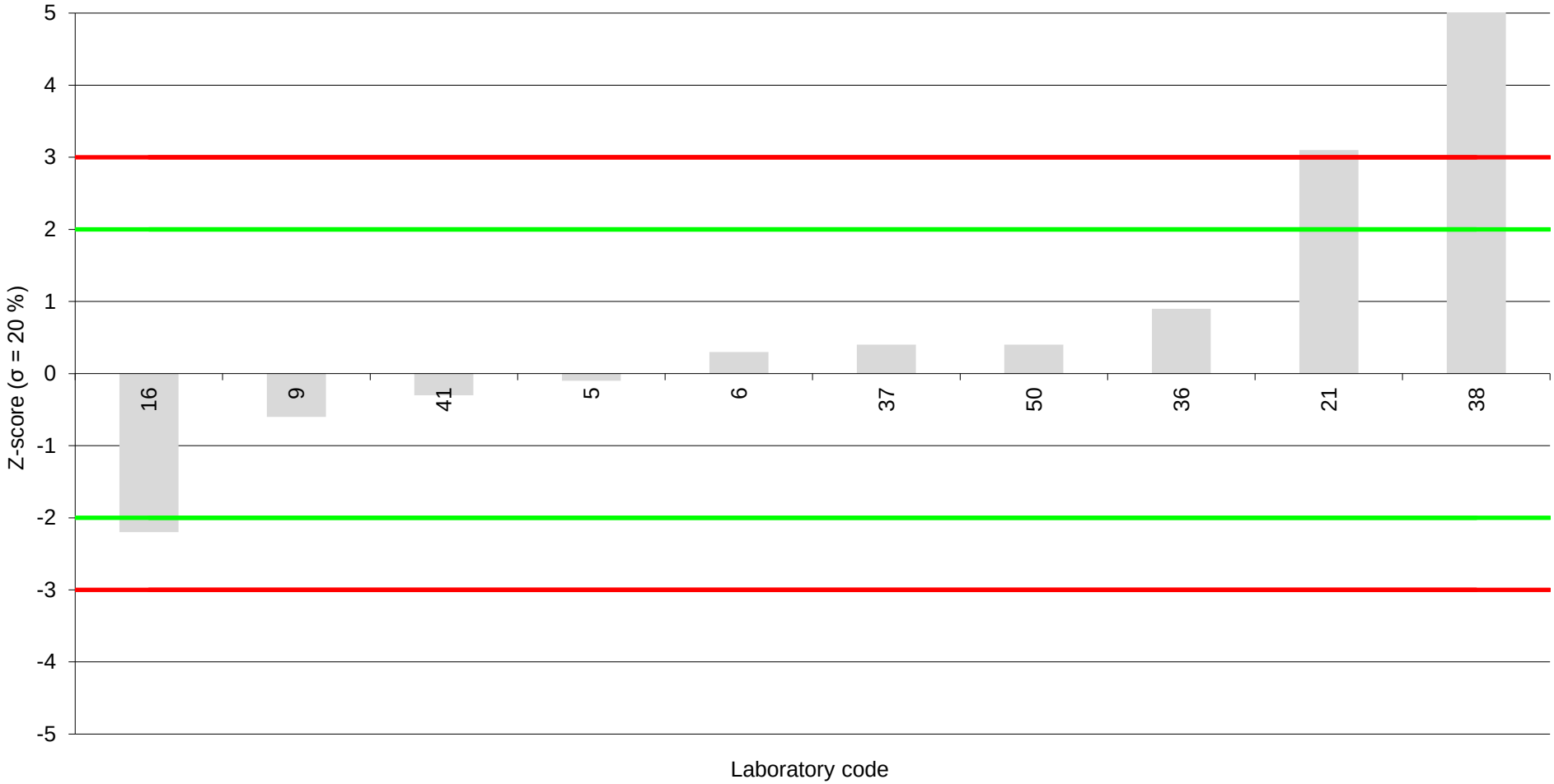
Assigned value: 2.85 µg/kg fat



Powdered Infant Milk A (2403-PIM-A)

Sum of PBDE including BDE-209 lb

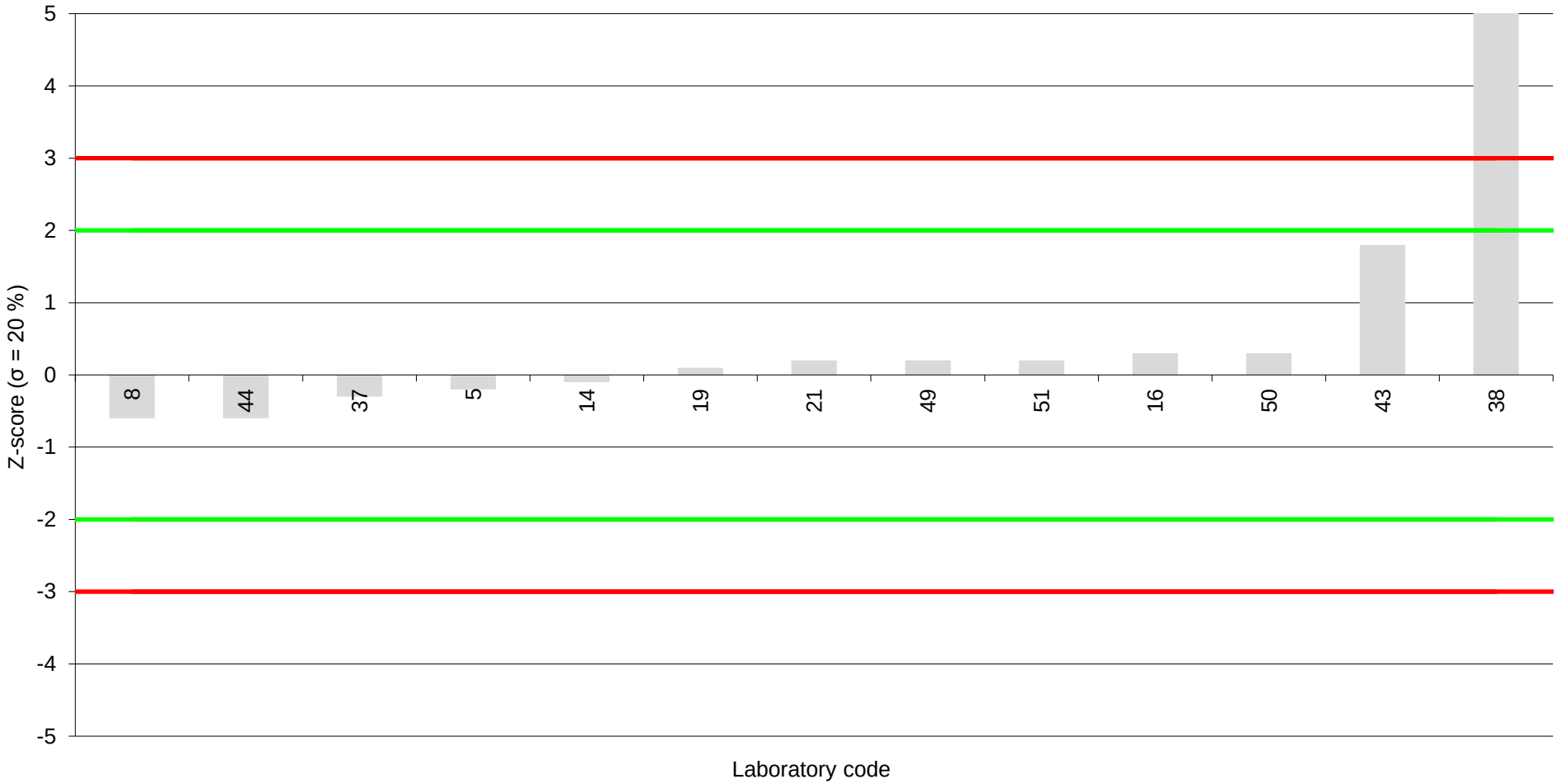
Assigned value: 2.85 µg/kg fat



Powdered Infant Milk A (2403-PIM-A)

(+/–)-α-HBCDD

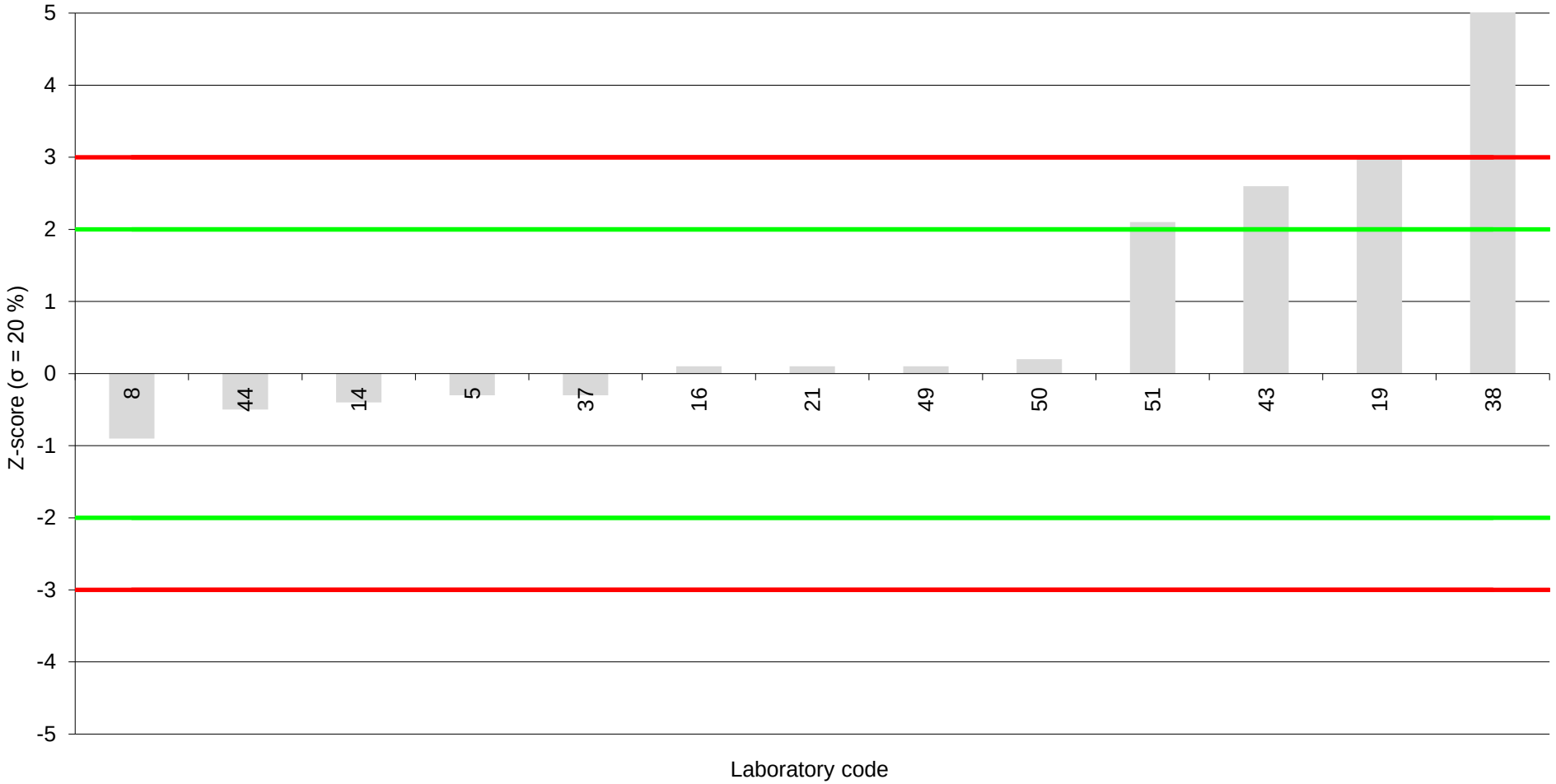
Assigned value: 0.0768 µg/kg product



Powdered Infant Milk A (2403-PIM-A)

Sum of α-, β-, γ-HBCDD ub

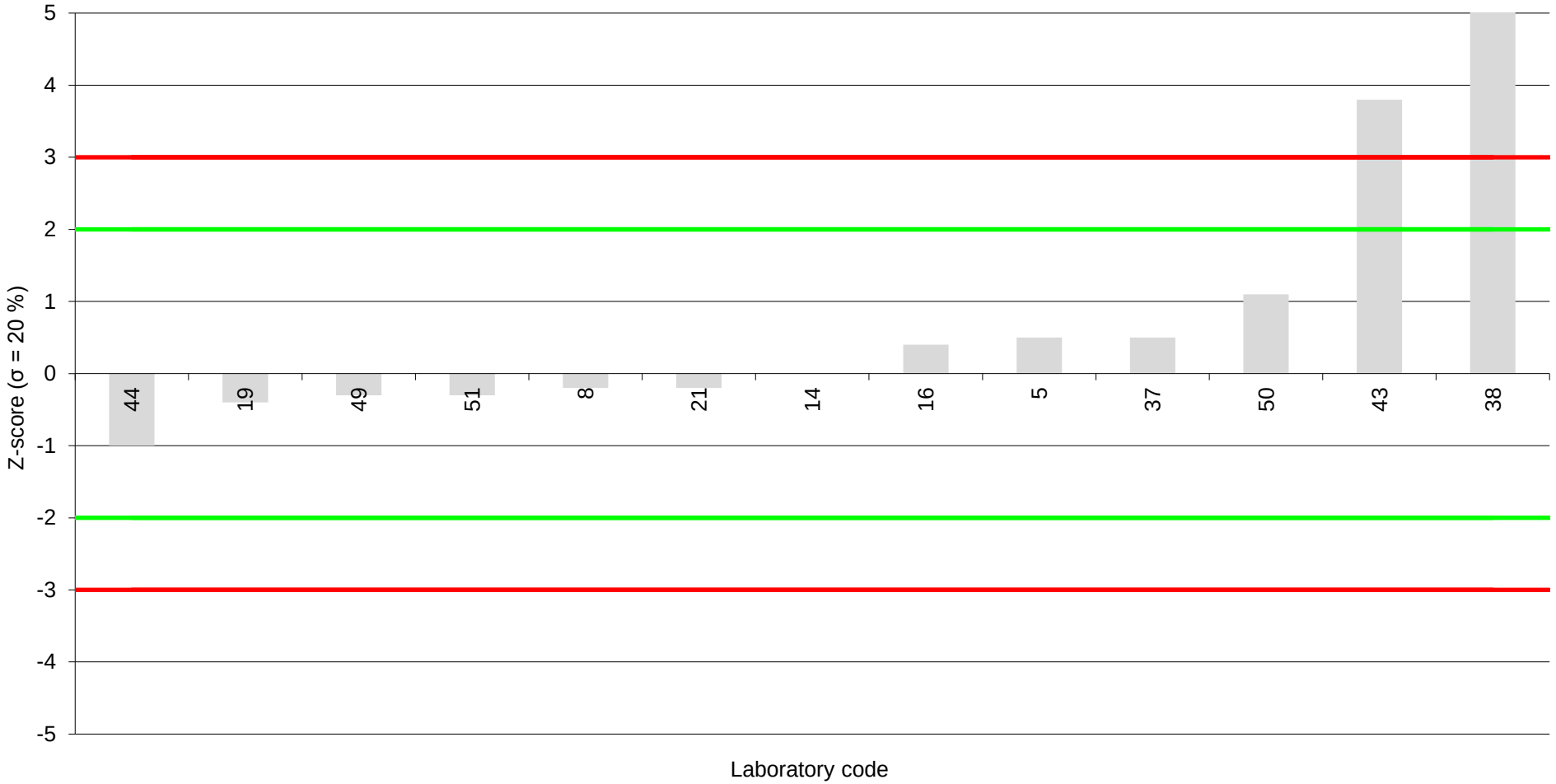
Assigned value: 0.0983 µg/kg product



Powdered Infant Milk A (2403-PIM-A)

Sum of α-, β-, γ-HBCDD lb

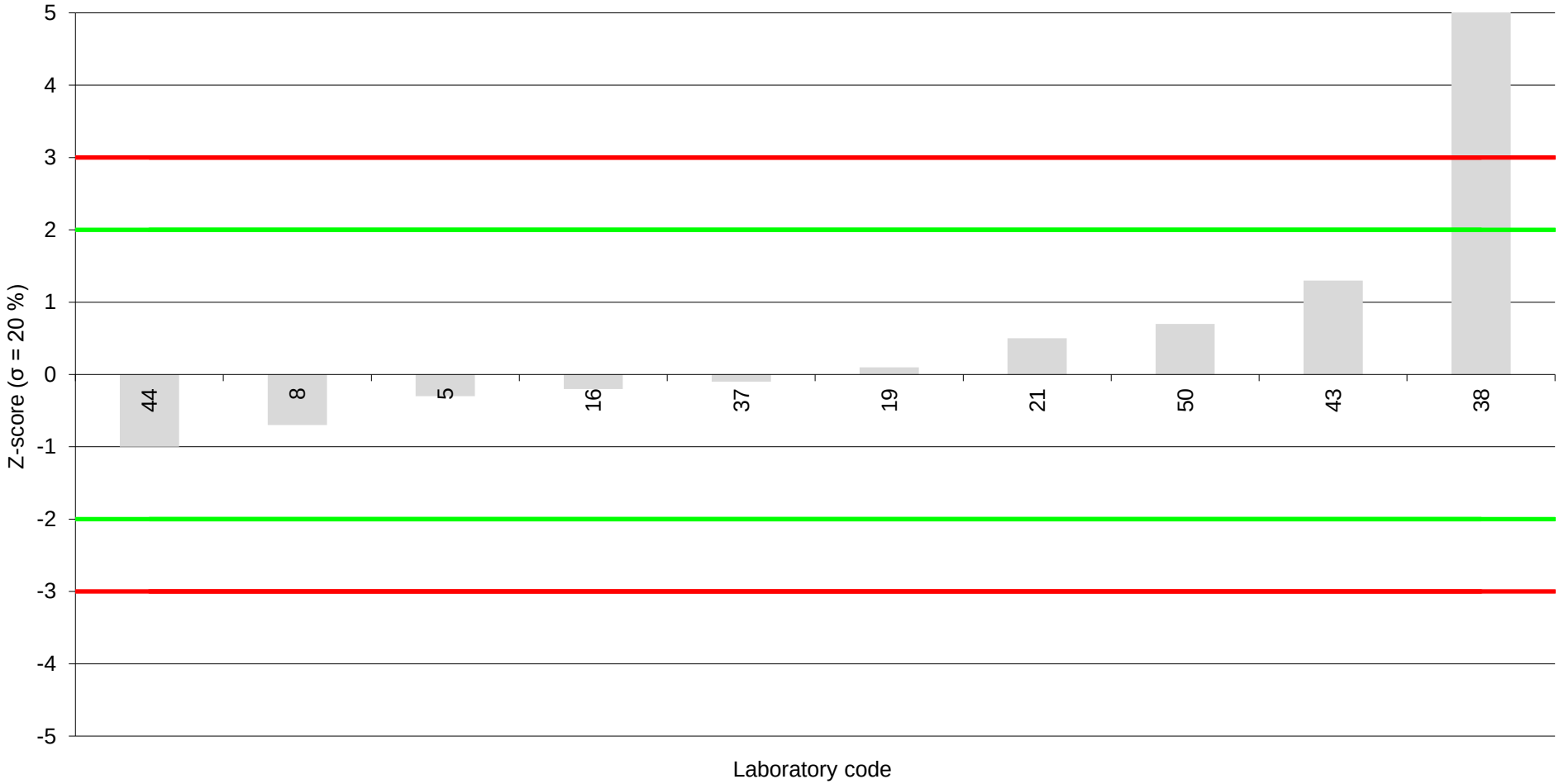
Assigned value: 0.0844 µg/kg product



Powdered Infant Milk A (2403-PIM-A)

(+/–)-α-HBCDD

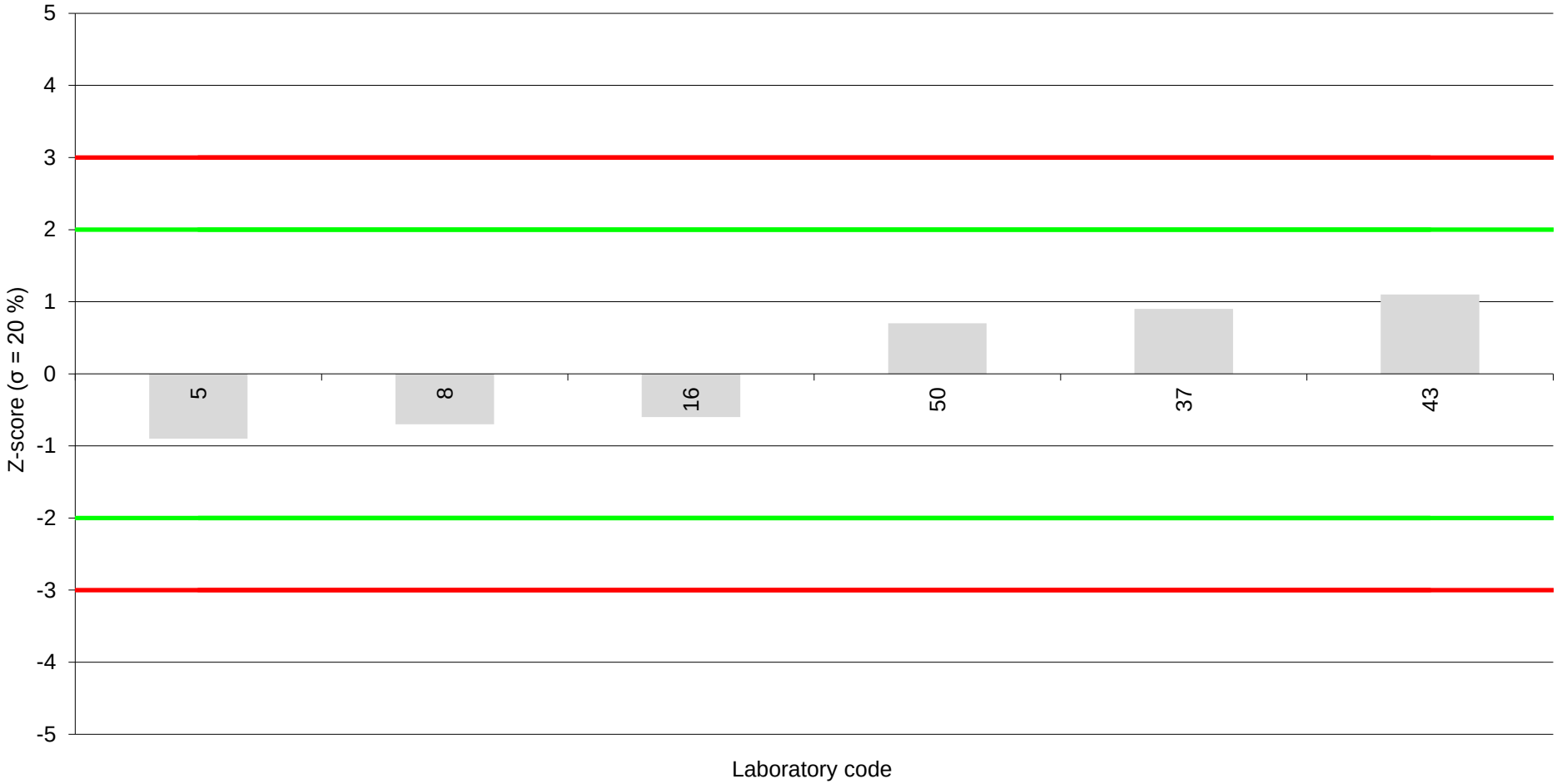
Assigned value: 0.314 µg/kg fat



Powdered Infant Milk A (2403-PIM-A)

(+/–)-β- HBCDD

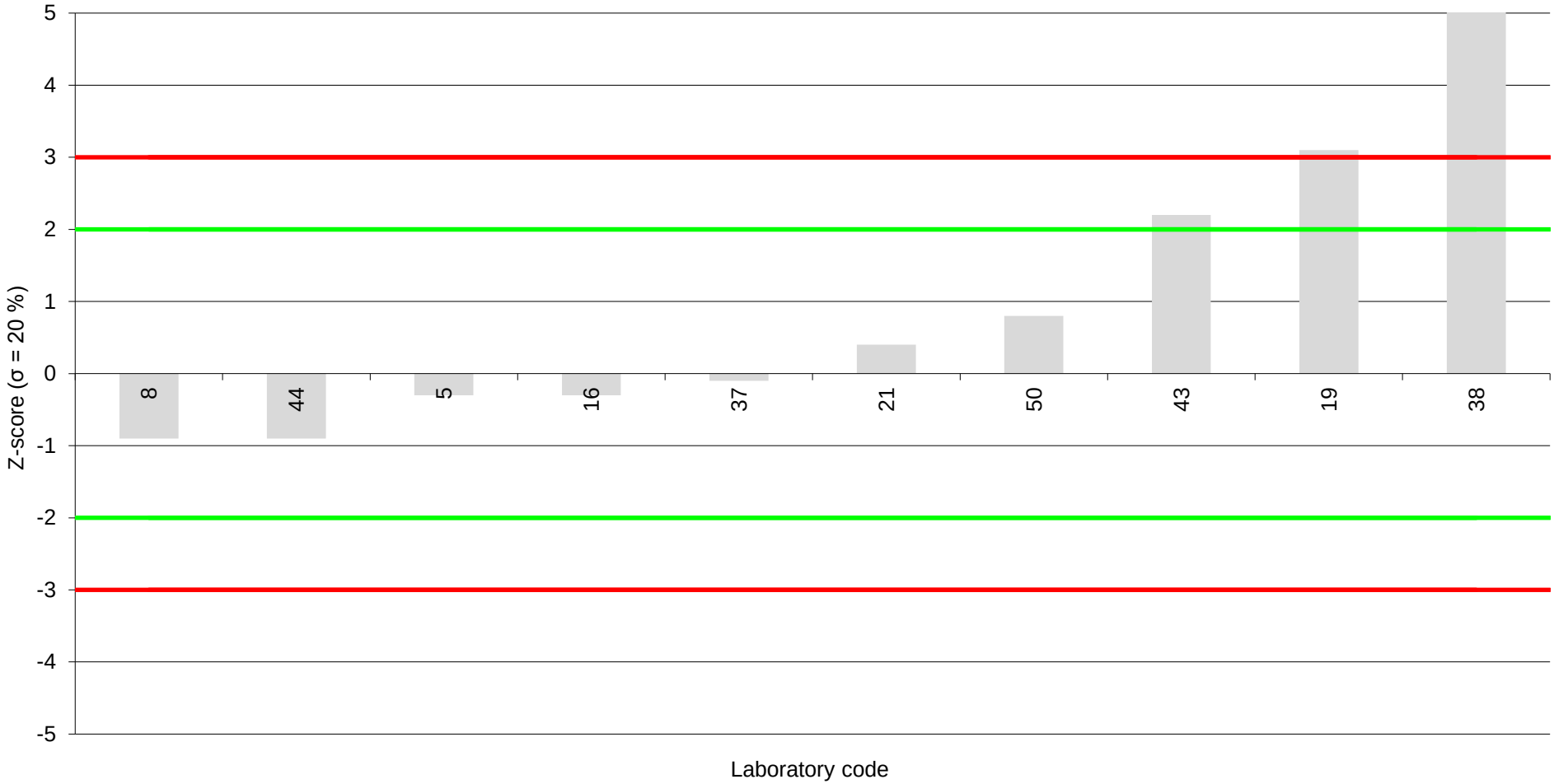
Assigned value: 0.0421 µg/kg fat



Powdered Infant Milk A (2403-PIM-A)

Sum of α-, β-, γ-HBCDD ub

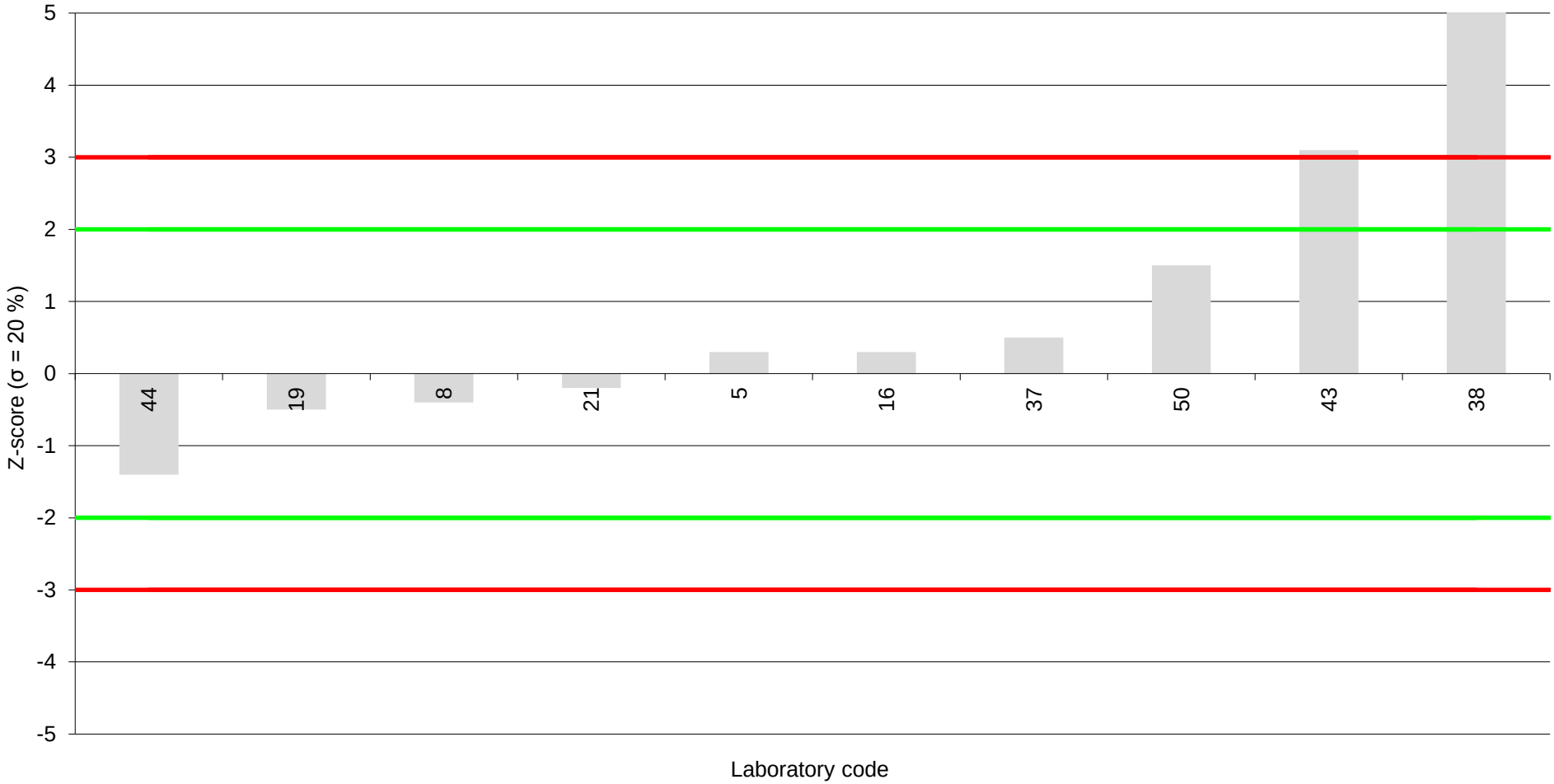
Assigned value: 0.396 µg/kg fat



Powdered Infant Milk A (2403-PIM-A)

Sum of α-, β-, γ-HBCDD lb

Assigned value: 0.354 µg/kg fat



EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]

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

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Annex 5: Test for sufficient homogeneity for PBDEs

Test sample - Powdered Infant Milk A (2403-PIM-A)

EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]
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Powdered Infant Milk A (2403-PIM-A)
PBDE - Homogeneity test - Data

Analyte	Result µg/kg product	**Mean** (n = 10, duplicate analysis)	**Median** (n = 10, duplicate analysis)	**Relative standard deviation** [%]
Sum of PBDE without BDE-209 ub				
Sum of PBDE including BDE-209 ub				
BDE-28		0.00111	0.00112	17%
BDE-47		0.0921	0.0918	4%
BDE-49		0.00247	0.00237	17%
BDE-99		0.126	0.124	6%
BDE-100		0.0250	0.0253	8%
BDE-153		0.0275	0.0272	6%
BDE-154		0.0134	0.0136	7%
BDE-183		0.100	0.100	8%
BDE-209		0.238	0.238	12%

EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]
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Powdered Infant Milk A (2403-PIM-A)
Selected PBDE congeners - Homogeneity test - Data

Sample	Replicate	Result µg/kg product	**BDE-99**
13	1		0.119
	2		0.126
27	1		0.133
	2		0.128
37	1		0.133
	2		0.120
39	1		0.130
	2		0.146
47	1		0.121
	2		0.124
49	1		0.124
	2		0.110
58	1		0.136
	2		0.132
77	1		0.119
	2		0.120
78	1		0.124
	2		0.134
128	1		0.124
	2		0.124
Cochran's C-test			
C			0.324
$C_{critical}$ (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 $\bar{x}$			0.126
Standard deviation of sample averages s_x			0.0066
Wthin-sample standard deviation s_w			0.0063
Between-sample standard deviation s_s			0.0049
Standard deviation for proficiency assessment σ_{PT}			0.0253
s_s / σ_{PT}			0.200
Test for homogeneity ($s_s \leq 0.3\ \sigma_{PT}$)			passed

EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]

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

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Annex 6a: Participants' results of PBDEs and HBCDDs on product basis

Test sample - Powdered Infant Milk B (2403-PIM-B)

EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]

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Powdered Infant Milk B (2403-PIM-B)

PBDE - Results

LC	Sample	Result µg/kg product	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** including BDE-209 (lb)
1	2403-PIM-B		< 0.0007	< 0.005	< 0.0007	< 0.002	< 0.0008	< 0.001	< 0.0007	< 0.001		0.012			
4	2403-PIM-B		< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.1				
5	2403-PIM-B		< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	0.003	0.008		0.011	0.003
6	2403-PIM-B		0.000219	0.00148	0.000137	< 0.000893	< 0.000673	< 0.000926	< 0.000792	0.00029	0.0145	0.00494	0.00213	0.0194	0.0166
9	2403-PIM-B		< 0.00103	< 0.0188	< 0.00054	< 0.00369	< 0.00245	< 0.000632	< 0.00126	< 0.00422	< 0.066	0.033		0.099	
13	2403-PIM-B		< 1000	< 1000	< 1000	< 1000	< 1000	< 1000	< 1000	< 1000					
14	2403-PIM-B		< 0.002	< 0.002	< 0.002	< 0.00399	< 0.00399	< 0.00599	< 0.00599	< 0.00998	< 0.2	0.0359		0.236	
15	2403-PIM-B		0.0029	0.056	< 0.00098	0.033	0.01	0.0022	0.0015	0.0054	< 0.12	0.11	0.11	0.23	0.11
16	2403-PIM-B														
20	2403-PIM-B		< 0.0025	< 0.02		< 0.014	< 0.003	< 0.0076	< 0.0025	< 0.0025	0.021				
21	2403-PIM-B		0.00496	0.204	0.00556	0.0526	0.0188	0.00163	0.0026	< 0.000432	0.0961	0.291	0.29	0.387	0.386
23	2403-PIM-B		0.0009	0.0144	0.0002	0.0066	0.0014	0.0009	0.0005	0.0039		0.029	0.029		
36	2403-PIM-B		0.000558	0.0134	0.000917	0.00842	0.0028	0.0416	0.0183	0.101	0.341	0.187	0.187	0.529	0.529
37	2403-PIM-B		< 0.006	< 0.019	< 0.01	< 0.0156	< 0.008	< 0.008	< 0.01	< 0.016	< 0.2	0.094	0.06	0.3	0.3
38	2403-PIM-B		< 0.084	< 1	< 0.16	< 1.5	< 0.5	< 0.14	< 0.24	< 0.4	14.2	4.02		18.2	14.2
41	2403-PIM-B		< 0.0002	0.0008	< 0.0001	0.0008		< 0.0001	0.0004	< 0.0005	< 0.002	0.0029	0.002	0.0049	0.002
44	2403-PIM-B		< 0.0036	< 0.00748		< 0.00462	< 0.00581	< 0.00473	< 0.00489	< 0.00549					
49	2403-PIM-B		< 0.005	< 0.015	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.1	0.05		0.15	
50	2403-PIM-B		0.000084	< 0.000052	< 0.000017	< 0.000024	< 0.000021	0.00019	< 0.00019	0.00058	< 0.003	0.00116	0.00085	0.00416	0.00085
53	2403-PIM-B		< 0.00146	0.0124	< 0.00146	0.0071	< 0.00146	0.00169	< 0.00146	0.00236		0.0294	0.0236		
58	2403-PIM-B		< 0.0005	< 0.0005	< 0.0005	< 0.002	< 0.001	< 0.001	< 0.001	< 0.001	0.015	0.0075		0.0225	0.015
44A	2403-PIM-B		< 0.0036	< 0.00748		< 0.00462	< 0.00581	< 0.00473	< 0.00489	< 0.00549					
49A	2403-PIM-B		< 0.005	< 0.015	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.1	0.05		0.15	

EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]

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

Powdered Infant Milk B (2403-PIM-B)

HBCDD - Results

LC	Sample	Result µg/kg product	**(+/–)-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)
5	2403-PIM-B								
6	2403-PIM-B								0.0058
8	2403-PIM-B		0.014	< 0.005	< 0.005		0.024	0.014	
13	2403-PIM-B		< 1000	< 1000	< 1000				
14	2403-PIM-B		< 0.00595	< 0.00595	< 0.00595		0.0179		
16	2403-PIM-B		< 0.01	< 0.01	< 0.01		0.03		
19	2403-PIM-B								
20	2403-PIM-B								0.0052
21	2403-PIM-B		< 0.01	< 0.01	< 0.01		0.03		
37	2403-PIM-B		< 0.005	< 0.005	< 0.005		0.015		
38	2403-PIM-B		< 0.2	< 0.2	< 0.2		0.6		
43	2403-PIM-B		< 0.0126	< 0.0026	0.0097		0.025	0.0097	
44	2403-PIM-B		< 0.01	< 0.01	< 0.01		0.03		
49	2403-PIM-B		< 0.01	< 0.01	< 0.01		0.03		
50	2403-PIM-B		0.003	< 0.001	0.002		0.005	0.005	
51	2403-PIM-B		< 0.03	< 0.03	< 0.03		0.09		

EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]

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

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Annex 6b: Participants' results of PBDEs and HBCDDs on fat basis

Test sample - Powdered Infant Milk B (2403-PIM-B)

EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]

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

Powdered Infant Milk B (2403-PIM-B)

PBDE - Results

LC	Sample	Result µg/kg fat	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** including BDE-209 (lb)
1	2403-PIM-B		< 0.003	< 0.02	< 0.003	< 0.007	< 0.003	< 0.005	< 0.003	< 0.005		0.049			
4	2403-PIM-B														
5	2403-PIM-B		< 0.004	< 0.004	< 0.004	< 0.004	< 0.004	< 0.004	< 0.004	< 0.004	0.011	0.032		0.043	0.011
6	2403-PIM-B		0.000832	0.00528	0.000482	< 0.00639	< 0.00371	< 0.00593	< 0.00442	0.00172	0.11	0.0288	0.00832	0.139	0.118
9	2403-PIM-B		< 0.00746	< 0.137	< 0.00391	< 0.0267	< 0.0178	< 0.00458	< 0.00913	< 0.0305	< 0.478	0.237		0.715	
13	2403-PIM-B														
14	2403-PIM-B														
15	2403-PIM-B														
16	2403-PIM-B														
20	2403-PIM-B														
21	2403-PIM-B		0.0212	0.872	0.0238	0.224	0.08	0.00694	0.0111	< 0.00184	0.409	1.24	1.24	1.65	1.65
23	2403-PIM-B		0.0039	0.0636	0.0008	0.0291	0.006	0.004	0.0023	0.0173		0.127	0.127		
36	2403-PIM-B		0.00208	0.0501	0.00342	0.0314	0.0105	0.155	0.0682	0.378	1.27	0.699	0.699	1.97	1.97
37	2403-PIM-B		< 0.021	< 0.074	< 0.039	< 0.06	< 0.031	< 0.03	< 0.037	< 0.06	< 0.77	0.354	0.228	1.19	1.19
38	2403-PIM-B		< 0.61	< 8	< 1.1	< 10	< 3	< 1	< 1.7	< 3	104	28.4		132	104
41	2403-PIM-B		< 0.0007	0.0035	< 0.0005	< 0.0005	0.0037	< 0.0005	0.0015	< 0.002	< 0.01	0.0129	0.0087	0.0229	0.0087
44	2403-PIM-B		< 0.036	< 0.0748		< 0.0462	< 0.0581	< 0.0473	< 0.0489	< 0.0549					
49	2403-PIM-B														
50	2403-PIM-B		0.00037	< 0.00023	< 0.00007	< 0.0001	< 0.00009	0.00083	< 0.00083	0.00278	< 0.0132	0.0053	0.00398	0.0185	0.00398
53	2403-PIM-B		< 0.005	0.0425	< 0.005	0.0243	< 0.005	0.00578	< 0.005	0.00807		0.101	0.0807		
58	2403-PIM-B														
44A	2403-PIM-B		< 0.036	< 0.0748		< 0.0462	< 0.0581	< 0.0473	< 0.0489	< 0.0549					
49A	2403-PIM-B														

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

Powdered Infant Milk B (2403-PIM-B)

HBCDD - Results

LC	Sample	Result µg/kg fat	**(+/–)-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)
5	2403-PIM-B								
6	2403-PIM-B								0.0258
8	2403-PIM-B		0.056	< 0.02	< 0.02		0.096	0.056	
13	2403-PIM-B								
14	2403-PIM-B								
16	2403-PIM-B		< 0.01	< 0.01	< 0.01		0.03		
19	2403-PIM-B								
20	2403-PIM-B								
21	2403-PIM-B		< 0.043	< 0.043	< 0.043		0.129		
37	2403-PIM-B		< 0.02	< 0.02	< 0.02		0.06		
38	2403-PIM-B		< 1	< 1	< 1		3		
43	2403-PIM-B		< 0.048	< 0.01	0.037		0.095	0.037	
44	2403-PIM-B		< 0.037	< 0.037	< 0.037		0.111		
49	2403-PIM-B								
50	2403-PIM-B		0.013	< 0.003	0.009		0.025	0.023	
51	2403-PIM-B								

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

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Annex 7: Participants' methods for PBDEs and HBCDDs

Test sample - Powdered Infant Milk (2403-PIM)

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

Powdered Infant Milk (2403-PIM)
Methods PBDEs - General, internal standards

LC	Sample		**Accreditation** according to ISO/IEC 17025	**Weighed sample** [g]	**Subtraction of** procedural blank		**Use of isotope-labelled internal standards for …** PBDE congeners (yes/no)
1	2403-PIM		Yes	50.18	no		yes (15)
4	2403-PIM		yes	1.2	no		yes
5	2403-PIM		yes	10.0	yes		yes
6	2403-PIM		yes	20.0	yes		yes
9	2403-PIM		yes	5	yes		yes
13	2403-PIM		no	1g			no
14	2403-PIM		yes	5.0			yes
15	2403-PIM		yes	7.5	yes		yes
16	2403-PIM		no	8	no		yes
20	2403-PIM		yes	2	yes		BDE-209
21	2403-PIM		yes	20.0	no*		yes
23	2403-PIM		no	29.6	no		yes (except for BDE-49)
36	2403-PIM		NO	5	no		yes
37	2403-PIM		no	5	no		yes
38	2403-PIM		yes	20.1	no		yes
41	2403-PIM		yes	10.0	no		yes
44	2403-PIM		yes	5	yes		yes
49	2403-PIM		yes	2	yes		yes
50	2403-PIM		YES	5.0	yes		yes
53	2403-PIM		Yes	1.5	no		yes
58	2403-PIM		no	15	no		no

EURL Interlaboratory Study on the Determination of Brominated Contaminants, PCNs, CPs and PFAS in Powdered Infant Milk / Formula 2024 [EURL-ILS-BCF_2403-PIM]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Powdered Infant Milk (2403-PIM)
Methods PBDEs - Internal Standards

LC	Sample		BDE 28	BDE 47	BDE 49	BDE 99	BDE 100	BDE 153	BDE 154	BDE 183	BDE 209
1	2403-PIM		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	
4	2403-PIM		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
5	2403-PIM		13C BDE28	13C BDE47	13C BDE47	13C BDE99	13C BDE100	13C BDE153	13C BDE154	13C BDE183	13C BDE209
6	2403-PIM		BDE-28	BDE-47	BDE47	BDE-99	BDE-100	BDE-153	BDE-154	BDE-183	BDE-209
9	2403-PIM		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
13	2403-PIM		none	none	none	none	none	none	none	none	
14	2403-PIM		13C12-TriBDE 28	13C12-TetraBDE 47	13C12-TetraBDE 47	13C12-PentaBDE 99	13C12-PentaBDE 99	13C12-HexaBDE 153	13C12-HexaBDE 153	13C12-HeptaBDE 183	13C12-DecaBDE 209
15	2403-PIM		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
16	2403-PIM		MBDE-28	MBDE-47	MBDE-47	MBDE-99	MBDE-100	MBDE-153	MBDE-154	MBDE-183	MBDE-209
20	2403-PIM										
21	2403-PIM		BDE-28	BDE-47	BDE-47	BDE-99	BDE-100	BDE-153	BDE-154	BDE-183	BDE-209
23	2403-PIM		BDE-28	BDE-47	BDE-47	BDE-99	BDE-100	BDE-153	BDE-154	BDE-183	
36	2403-PIM		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	2403-PIM		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
38	2403-PIM		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
41	2403-PIM		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
44	2403-PIM		13C12-PBDE 28	13C12-PBDE 47	-	13C12-PBDE 99	13C12-PBDE 100	13C12-PBDE 153	13C12-PBDE 154	13C12-PBDE 183	-
49	2403-PIM		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
50	2403-PIM		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
53	2403-PIM		13C-BDE-28	13C-BDE-47	13C-BDE-47	13C-BDE-99	13C-BDE-99	13C-BDE-153	13C-BDE-154	13C-BDE-183	
58	2403-PIM										

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Powdered Infant Milk (2403-PIM)
Methods PBDEs - Recovery Standards

LC	Sample		BDE 28	BDE 47	BDE 49	BDE 99	BDE 100	BDE 153	BDE 154	BDE 183	BDE 209
1	2403-PIM		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	
4	2403-PIM										
5	2403-PIM		13C BDE 153	13C BDE 153	13C BDE 153	13C BDE 153	13C BDE 153	13C BDE 153	13C BDE 153	13C BDE 153	13C BDE 153
6	2403-PIM		PCB-138	PCB-138	PCB-138	PCB-138	PCB-138	PCB-138	PCB-138	PCB-138	PCB-138
9	2403-PIM		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
13	2403-PIM		PCB209	PCB209	PCB209	PCB209	PCB209	PCB209	PCB209	PCB209	
14	2403-PIM		13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138	13C12-HexaBDE138
15	2403-PIM		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
16	2403-PIM		MBDE-79	MBDE-79		MBDE-139	MBDE-139	MBDE-139	MBDE-139	MBDE-139	MBDE-206
20	2403-PIM										
21	2403-PIM		BDE-79	BDE-79	BDE-79	BDE-79	BDE-79	BDE-138	BDE-138	BDE-138	BDE-206
23	2403-PIM		BDE-77	BDE-77	BDE-77	BDE-77	BDE-77	BDE-138	BDE-138	BDE-138	
36	2403-PIM		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
37	2403-PIM		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
38	2403-PIM		13C12 BDE 79	13C12 BDE 79	13C12 BDE 79	13C12 BDE 79	13C12 BDE 139	13C12 BDE 139	13C12 BDE 139	13C12 BDE 180	13C12 BDE 206
41	2403-PIM		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
44	2403-PIM		13C12-PCB 52	13C12-PCB 52	-	13C12-PCB 138	13C12-PCB 138	13C12-PCB 138	13C12-PCB 138	13C12-PCB 138	-
49	2403-PIM		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
50	2403-PIM		13C BDE-77	13C BDE-77	13C BDE-77	13C BDE-77	13C BDE-77	13C BDE-138	13C BDE-138	13C BDE-138	
53	2403-PIM		13C-PCB-80	13C-PCB-80	13C-PCB-80	13C-PCB-80	13C-PCB-80	13C-PCB-80	13C-PCB-80	13C-PCB-80	
58	2403-PIM										

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Powdered Infant Milk (2403-PIM)
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]
1	2403-PIM	No sample pre-treatment, used directly from container supplied.	Digest in precleaned concentrated hydrochloric acid, tumble with solvent mixture and decant top layer	Extracted with 3 x 80mL hexane/dichloromethane (3:1 vol/vol) added to acid mixture	16 hours initially, followed by 1 hour, then 0.5 hours for successive extractions.	Ambient	ambient
4	2403-PIM	Homogenisation	Hydrochloric acid digestion in DigiPREP thermoreactor	Liquid/liquid partition with hexane	0,5	80°C	Atmospheric pressure
5	2403-PIM		soxhlet	hex/dcm	6		
6	2403-PIM	Homogenisation, reconstitution in water only for LLE method (not for ASE), isolation of fat, lipid content (%)	LLE, ASE	LLE: hexane, DEE, ethanol, ASE: hexane:iso-propanol (3:2,v/v)	LLE: 30 min, ASE: 30 min	ASE: 120 °C	ASE: 10 MPa
9	2403-PIM	Reconstitution in water	Röse-Gottlieb (liquid-liquid)	ethanol/ammonia/ether/n-hexane	0.5	25	1
13	2403-PIM		shaking	Cylohexan/Ethylacetat 1:1	0,5h	RT	
14	2403-PIM	homogenization, drying with polyacrylamide	cold extraction	hexane		room temperature	
15	2403-PIM	reconstitution in hot water, spiking with internal standard, freeze-drying	Soxhlet with water-separator, LLE of Toluene 3x with water after Soxhlet, drying with Na2SO4 before further sample preparation	Toluene	approx 16 h		
16	2403-PIM		ASE	Ethanol/Toluol (7/3)	1 h	100°C	10
20	2403-PIM	dissolved the powder in Milli-Q water	Liquid-liquid extraction manual	Hexane/acetone 1/1			
21	2403-PIM	reconstitution of liquid milk by adding water to the powder	solvent partitioning (liquid-liquid extraction)	ethanol/diethylether/petroleum ether (1/2/2)			
23	2403-PIM	the extraction of the milk powder samples was performed by the EURL-PE-02.02 (Fast Extraction of Sampels by Ultra Turrax Dispenser)	Ultra Turrax	1) 100 mL 2- propanol 2), 120 mL Cyclohexane, 3) Ultra Turrax 40 sec, 4) 90 mL water bidestilated 5) repeated extraction x 2, 6) Cyclohexane 80 mL, 7) Ultra turrax 30 sec			
36	2403-PIM	NO	SOXHLET	TOLUENE/ETHANOL (3:7)	24H		
37	2403-PIM		Accelerated Solvent Extraction (ASE) - ASE 350	Dichloromethane:n-hexane:methanol (25:60:15)	0.3	100.00	10
38	2403-PIM	none	Soxhlet	DCM	16	40.00	ambient
41	2403-PIM	reconstitution in water 10g/200 mL	liquid/liquid	n-Pentane/diethyether			
44	2403-PIM	thorough homogenization, powder reconstitution with MQ water (1:9), acid hydrolysis (sodium oxalate,ethanol)	(ultrasonic bath, agitate), L-L cold extraction (2 fold)	diethylether, n-hexane	2	20	0.1
49	2403-PIM	no	QuEChERs like-extraction	Ethyl acetate	no	no	no
50	2403-PIM	drying	Reconstituted with water then liquid/liquid extraction	Hexane/diethylether/ethanol			
53	2403-PIM		Soxhlet	Toluene/EtOH 70/30	18 h		
58	2403-PIM	no	solution in water / Triton X100 heat for 1 hour 95°C	2-Butanone / Ethylacetate	1	30°C	

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Powdered Infant Milk (2403-PIM)
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]: PBDEs
1	2403-PIM		No	Yes	No	Yes	Yes	Sample extract partitioned with concentrated sulfuric acid to remove bulk organic material residues	10µL
4	2403-PIM		no	yes	no	no	no	no	50
5	2403-PIM		yes	yes	yes				30
6	2403-PIM		no	yes	no	no	yes	modified carbon column	20
9	2403-PIM		no	yes	no	no	no	no	100
13	2403-PIM		yes	no	no	no	no		1ml
14	2403-PIM		no	yes	no	yes	no		100
15	2403-PIM		no	yes	no	yes	yes	Silica/AgNO3	100
16	2403-PIM		no	yes	no	yes	yes	MIURA	50
20	2403-PIM		no	no	no	no	no	Sulfuric acid distribution and silica column	100
21	2403-PIM		no	yes	no	yes	no	acid hydrolysis with sulphuric acid	40
23	2403-PIM		no	yes	no			basic set of "power-prep system" columns	40
36	2403-PIM		NO	YES	NO	YES	NO	NO	20
37	2403-PIM		no	yes	yes	yes	no	no	100
38	2403-PIM		No	Yes	No	Yes	Yes	No	45
41	2403-PIM		no	yes	no	yes	yes		100
44	2403-PIM		no	yes	no	yes	yes	basic silica, silver nitrate silica	20
49	2403-PIM		yes	no	no	no	no	Extrelut NT-3 column, acidic for H2SO4 tandem Si 1g/6 mL column	250
50	2403-PIM		YES	YES	YES	NO	YES		50
53	2403-PIM		No	Yes	No	Yes	No		50
58	2403-PIM		no	yes	no	yes	no	no	20

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Powdered Infant Milk (2403-PIM)
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
1	2403-PIM		Splitless	1µL	5%-Phenyl-Arylene-95% DimethylPolysiloxane (ZB5-MS) column 30m×0.1mm×0.1µm	Thermo DFS HRMS (>10,000 Mass resolution)
4	2403-PIM		pulsed splitless	2	TG-PBDE, 15 m x 0,25 mm, 0,10 µm (Thermo Scientific)	MS/MS
5	2403-PIM		splitless	2	db5 ms	ms/ms
6	2403-PIM		split-splitless	2	DB-5ht	HRMS
9	2403-PIM		PTV	5	RTX-1614 (15m)	HRMS
13	2403-PIM		PTV	2	DB-5MS	MS
14	2403-PIM		pulsed splitless	2	Diphenyl-/dimethylpolysiloxan (5%/95%)	MS/MS
15	2403-PIM		PTV	1	ZB-Semivolatile 20m, 0.18mm, 0.18µm	MS/MS
16	2403-PIM		large volume	4	Rtx-1614 (15m x 0,25 mm x 0,1 µm) + 2m retention gap (uncoated)	MS/MS
20	2403-PIM		pulsed splitless	6	DB-5MS	LRMS
21	2403-PIM		PTV	2	Rtx-1614 30m x 0.25mm x 0.1µm	HRMS
23	2403-PIM		Splitless	1	DB-5MS (30 m, 0.25 mm id, 0,25 mm film)	HRMS (Mat-95 XP)
36	2403-PIM		SPLITLESS	1	DB-5ms 40m x 0,18 mm x 0,18 um	HRMS
37	2403-PIM		PTV Splitless	1	15 m RTX 1614	HRMS
38	2403-PIM		Programmed	2	DB5	HRMS
41	2403-PIM		splitless	2	DB5 MS	HRMS
44	2403-PIM		pulsed splitless	1	DB-5MS (60m x 0,25mm x 0,10 µm)	HRMS (R>10000)
49	2403-PIM		PTV	5	DB5HT 15 m x 0.25 mm; 0.1 um	MS/MS
50	2403-PIM		Splitless	2	DB5MS	GC-HRMS
53	2403-PIM		Splitless	1.5	DB-5MS	HRMS, DFS
58	2403-PIM		splitt/splitless	1	DB-5MS	MS/MS

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Powdered Infant Milk (2403-PIM)
Physico-chemical Methods HBCDDs - Internal standards

LC	Sample		**Accreditation** according to ISO/IEC 17025	**Weighed sample** [g]	**Subtraction of** procedural blank		**Use of isotope-labelled internal standards for …** HBCDD diastereomers (yes/no)
5	2403-PIM		no	10	yes		yes
6	2403-PIM		no	20	yes		yes
8	2403-PIM		YES	3.5	yes		yes
13	2403-PIM		no	1g			no
14	2403-PIM		yes	5			yes
16	2403-PIM		no	10	no		yes
19	2403-PIM		no	5	no		yes
20	2403-PIM		yes	2	yes		no
21	2403-PIM		no	20	no		yes
37	2403-PIM		no	10	no		yes
38	2403-PIM		No	1.28	no		yes
43	2403-PIM		no	1.34	no		yes
44	2403-PIM		yes	5	no		yes
49	2403-PIM		yes	2	yes		yes
50	2403-PIM		YES	5.51	no		yes
51	2403-PIM		Yes	10			yes

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Powdered Infant Milk (2403-PIM)
Methods HBCDDs - Internal Standards

LC	Sample		(+/–)-α-HBCDD	(+/–)-β- HBCDD	(+/–)-γ- HBCDD
5	2403-PIM				
6	2403-PIM				
8	2403-PIM		13C12-α-HBCDD	13C12-β-HBCDD	13C12-γ-HBCDD
13	2403-PIM		none	none	none
14	2403-PIM		13C12-α-HBCD	13C12-β-HBCD	13C12-γ-HBCD
16	2403-PIM		13C12-alpha-HBCDD	13C12-beta-HBCDD	13C12-gamma-HBCDD
19	2403-PIM		alpha- HBCDD (13C)	beta- HBCDD (13C)	gamma- HBCDD (13C)
20	2403-PIM				
21	2403-PIM		alpha-HBCDD	beta-HBCDD	gamma-HBCDD
37	2403-PIM		13C alphaHBCDD	13C betaHBCDD	13C gammaHBCDD
38	2403-PIM		alpha-HBCDD-13C12	alpha-HBCDD-13C12	alpha-HBCDD-13C12
43	2403-PIM		13C-alpha-HBCD	13C-beta-HBCD	13C-gamma-HBCD
44	2403-PIM				
49	2403-PIM		alfa-HBCD-13C12	gamma-HBCD-13C12	gamma-HBCD-13C12
50	2403-PIM		13C-α-HBCDD	13C-β-HBCDD	13C-γ-HBCDD
51	2403-PIM		13C-A-HBCD	13C-B-HBCD	13-C-Y-HBCD

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Powdered Infant Milk (2403-PIM)
Methods HBCDDs - Recovery Standards

LC	Sample		(+/–)-α-HBCDD	(+/–)-β- HBCDD	(+/–)-γ- HBCDD
5	2403-PIM				
6	2403-PIM				
8	2403-PIM		N/A	N/A	N/A
13	2403-PIM		PCB209	PCB209	PCB209
14	2403-PIM		d18-β-HBCD	d18-β-HBCD	d18-β-HBCD
16	2403-PIM		alpha-HBCDD-d18	beta-HBCDD-d18	beta-HBCDD-d18
19	2403-PIM		D- beta-HBCDD	D- beta-HBCDD	D- beta-HBCDD
20	2403-PIM				
21	2403-PIM		D18-beta-HBCDD	D18-beta-HBCDD	D18-beta-HBCDD
37	2403-PIM		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
38	2403-PIM				
43	2403-PIM		none	none	none
44	2403-PIM				
49	2403-PIM		beta-HBCD-13C12	beta-HBCD-13C12	beta-HBCD-13C12
50	2403-PIM		d18-β-HBCDD	d18-β-HBCDD	d18-β-HBCDD
51	2403-PIM		D18-B-HBCD	D18-B-HBCD	D18-B-HBCD

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Powdered Infant Milk (2403-PIM)
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]
5	2403-PIM							
6	2403-PIM		Homogenisation, reconstitution in water only for LLE method (not for ASE), isolation of fat, lipid content (%)	LLE, ASE	LLE: hexane, DEE, ethanol, ASE: hexane:iso-propanol (3:2,v/v)	LLE: 30 min, ASE: 30 min	ASE: 120 °C	ASE: 10 MPa
8	2403-PIM		NA	Ultrasound + Mechanical Shaking	DCM:N-Hex (1:1, v/v)	1h	Room temp.	NA
13	2403-PIM			shaking	Cylohexan/Ethylacetat 1:1	0,5h	RT	
14	2403-PIM		homogenization	Quechers	acetonitrile/water (1/1)	10 min	room temperature	
16	2403-PIM		slurry with Aceton/Water (4/1) and than mix with sodiumfulfate	hot extraction (Randall)	Hexane/Dichlormethane/Ethanol (5/2/1)	2	95	ambient
19	2403-PIM		no		Hexane/Dichloromethane 50/50	0.33	20	Ambiant
20	2403-PIM		dissolved in water	manual liquid-liquid extraction	hexane/acetone 1/1			
21	2403-PIM		reconstitution of liquid milk by adding wate	liquid-liquid partitioning process	ethanol/diethylether/petroleum ether (1/2/2)			
37	2403-PIM			ASE 350	dichloromethane/hexane/methanol (25/60/15)	0.35	100	10
38	2403-PIM		none	Soxhlet	DCM	16	40	ambient
43	2403-PIM		treatment as milk (1.34 g powder + 9 g water)	LLE	PE - acetone			
44	2403-PIM		drying	shaking with dichlormethan : acetone 2:1 for 90 min	100 ml of dichlormethan : acetone 2:1	90 min	ambient	0.1
49	2403-PIM			QuEChERS-like	Ethyl Acetate			
50	2403-PIM		Reconstitution in 50 mL water		Liquide/Liquide extraction with hexane			
51	2403-PIM		None	ASE	50:50 Acetone:Hexane	0.5	100	10.3

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Powdered Infant Milk (2403-PIM)
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
5	2403-PIM								
6	2403-PIM		no	yes	no	no	no	no	20
8	2403-PIM		No	Yes	Yes	No	No	no	50
13	2403-PIM		yes	no	no	no	no	no	1ml
14	2403-PIM		no	yes	no	no	no		1000
16	2403-PIM		no	yes	no	no	no	no	400
19	2403-PIM		no	yes	no	no	no		200
20	2403-PIM		no	no	no	no	no	sulfuric acid distribution and silica column	100
21	2403-PIM		no	yes	no	no	no	acid hydrolysis with sulphuric acid	100
37	2403-PIM		no	yes	no	no	no		50
38	2403-PIM		No	Yes	No	No	No	No	500
43	2403-PIM		no	yes	no	no	no	no	500
44	2403-PIM		yes	yes	no	no	no		500
49	2403-PIM		yes	no	no	no	no	Extrelut NT-3 column, acidic for H2SO4 tandem Si 1g/6 mL column	250
50	2403-PIM		YES	YES	NO	NO	NO	liquid liquid extraction	50
51	2403-PIM		No	Yes	Yes	No	No	None	100

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Powdered Infant Milk (2403-PIM)
Physico-chemical Methods HBCDDs - Chromatographic separation and detection method

LC	Sample		Chromatographic separation and detection method Injection	Injected volume [µl]	Chromatographic separation: Stationary phase	Detector
5	2403-PIM					
6	2403-PIM		split-splitless	2 and 3	DB-5ht	HRMS
8	2403-PIM		Liquid injection	5	LC - C18	HRMS (Orbitrap Q-Executive)
13	2403-PIM		PTV	2	DB-5MS	MS
14	2403-PIM		standard	10	C18	MS/MS
16	2403-PIM			15	C18	MS/MS
19	2403-PIM		UPLC	5	Eclipse Plus C18, 2,1 x 100mm	MS/MS
20	2403-PIM		pulsed splitless	6	DB-5MS	LRMS
21	2403-PIM			5	BEH C18 (50 mm x 2.1 mm; 1.7 µm)	HRMS
37	2403-PIM			10	Hypersil Gold C18, 100x2,1mm, 1,9µm	MS/MS
38	2403-PIM		Autosyringe	20	ACQUITY UPLC BEH C18 1.7 um	LCMSMS
43	2403-PIM		LC-injection	10	C18	LC-MS/MS
44	2403-PIM		normal	10	C18	MS/MS
49	2403-PIM			20 µL	KINETEX 2.6um XB-C18 100A (100 x 2.1 mm) (PHENOMENEX)	LC-MS/MS
50	2403-PIM			15	Hypersil Gold	MS/MS
51	2403-PIM			10	C18	HRMS (Hybrid Quadrupole/Orbitrap)