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

EURL-PT-POP_2401-BM

FOOD

Report

PBDEs and HBCDDs

(Report Version 1.0)

24 March 2025

EURL for halogenated POPs in Feed and Food
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Summary

Test sample	**FOOD:** Bovine Meat [2401-BM]
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 food business operators
Statistical evaluation	ISO 13528:2022 [1], IUPAC Protocol [2]
Report of final results	24 March 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, HBCDDs** and **PFAS** in **Bovine Meat** was organized by the EURL for halogenated POPs in Feed and Food to be performed between February and April 2024. The objective was to assess analytical performance of laboratories and interlaboratory comparability of results from analyses of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in one sample of **Bovine Meat**.

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

First results were discussed by representatives of European Commission, NRLs and the EURL at the EURL/NRL workshop in May 2024 in Freiburg, Germany.

1.1. Samples and coding

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

Bovine Meat	**Sample no. 2401-BM-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 wet weight (w. w.)** 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

129 laboratories registered for this proficiency test. 28 laboratories reported results for PBDEs and 23 for HBCDDs; one lab 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	27	17/16
	Other Countries	5	1/-
Official Laboratories	European Union	69	5/5
	Other European Countries	4	1/1
	Africa	-	-
	Americas	1	1
	Asia	-	-
	Oceania	-	-
Commercial Laboratories	European Union	18	3/1
	Other European Countries	2	-
	Africa	-	-
	Americas	1	-
	Asia	2	-
	Oceania	-	-
	Total	**129**	**28/23**

2.1. Number of reported results

Table 2: Reported results for PBDEs and moisture content for bovine meat (2401-BM)

Reported results (2401-BM)	All laboratories	NRLs
BDE-28 / -47 / -99 / -100 / -153 / -154 / -183	28	17
BDE-49	24	14
BDE-209	21	11
Sum of 8 PBDEs (without BDE-209) (ub) / (lb)	27	16
Sum of 9 PBDEs (with BDE-209) (ub) / (lb)	20	10
Fat content	28	17

Table 3: Reported results for HBCDDs for bovine meat (2401-BM)

Reported results (2401-BM)	All laboratories	NRLs
α-HBCDD / β-HBCDD / γ-HBCDD	21	14
Sum of α-, β-, γ-HBCDD (ub)	18	12
Sum of α-, β-, γ-HBCDD (lb)	13	8
Total HBCDD (using GC methods)	2	2

2.2. Accreditation

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

Bovine meat	PBDEs	HBCDDs
Accreditation	21	7
No accreditation	10	12

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 in bovine meat (2401-BM)

Detection methods	PBDEs	HBCDDs
GC-HRMS	**18**	**1**
GC-MS/MS	**9**	**-**
GC-LRMS	**1**	**1**
LC-MS/MS	**-**	**18**
LC-HRMS	**-**	**3**

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 2401-BM were analyzed in duplicate for PBDEs. The test for sufficient homogeneity was performed for the individual congeners and sum parameters. The test materials showed sufficient homogeneity for PBDEs for this proficiency test. 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 PBDEs for this proficiency test. Homogeneity and stability can be concluded also for HBCDDs due to similar physico-chemical properties 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 (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:

- (+/–)-α- HBCDD; (+/–)-β – HBCDD; (+/–)-μ – HBCDD
- Total HBCDD (using GC-methods)
- HBCDD sum parameters
- BDE 28; BDE 49; BDE 209
- Sum of PBDE including BDE-209 ub and lb

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 28 participating laboratories were accredited according to ISO/IEC 17025 for PBDEs. After eliminating outliers, 15 to 16 results contributed to the calculation of the assigned values from the ISO/IEC 17025 group. No significant differences (5 %) between the assigned values calculated for both data sets for PBDEs were observed. (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 wet weight		%
Sum of PBDE without BDE-209 (ub)	0.218	0.205	4
Sum of PBDE without BDE-209 (lb)	0.214	0.207	5

4.1. PBDEs – individual congeners and sum parameter

The assigned values for the test sample 2401-BM 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 and 2).

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

Bovine meat (2401-BM)	Assigned value µg/kg (wet weight)
BDE-47	0.0529
BDE-99	0.0670
BDE-100	0.0143
BDE-153	0.0159
BDE-154	0.00733
BDE-183	0.0547
Sum of 8 PBDEs (without BDE-209) (ub)	0.218
Sum of 8 PBDEs (without BDE-209) (lb)	0.214

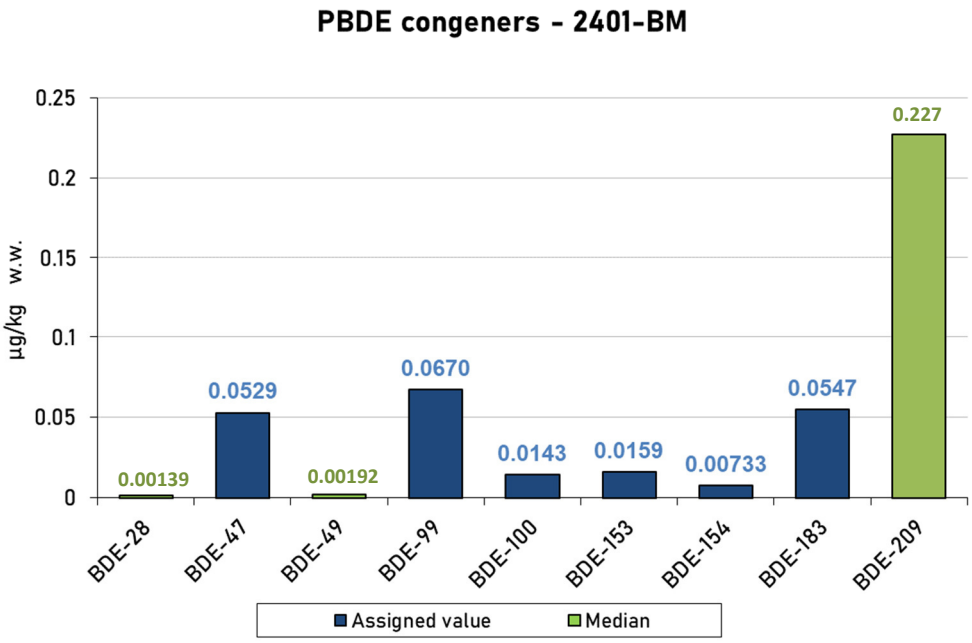

Figure 1: Assigned values (blue) and median values (green) for PBDE individual congeners for bovine meat (2401-BM) [µg/kg wet weight]

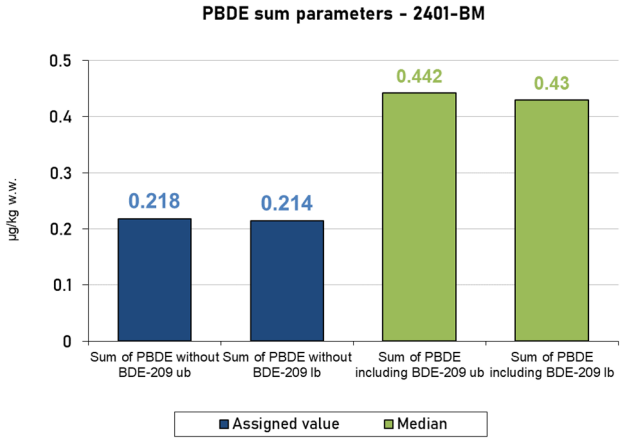

Figure 2: Assigned values (blue) and median values (green) for PBDE sum parameters for bovine meat (2401-BM) [µg/kg wet weight]

4.2. HBCDDs – individual stereoisomers and sum parameter

No assigned values could be calculated for any of the HBCDD parameters in this test sample 24301-BM taking into account the calculation criteria described above (tabular summary of median values see annex 1).

4.3. Lipid content

For the lipid content an assigned value of 6.54 % for the test sample 2401-BM was calculated as a consensus of the participants' results, taking into account the calculation criteria described above (tabular summary see annex 1).

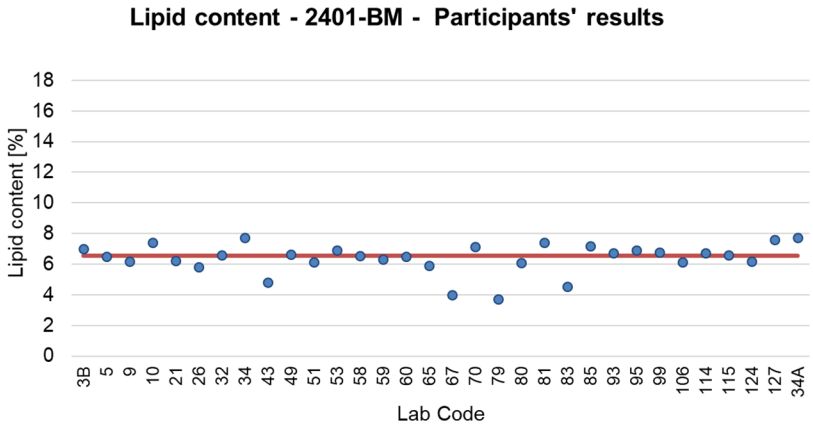

Figure 3: Participant's results (blue dots) compared to the assigned value (red line) of the lipid content in % for Bovine Meat (2401-BM)

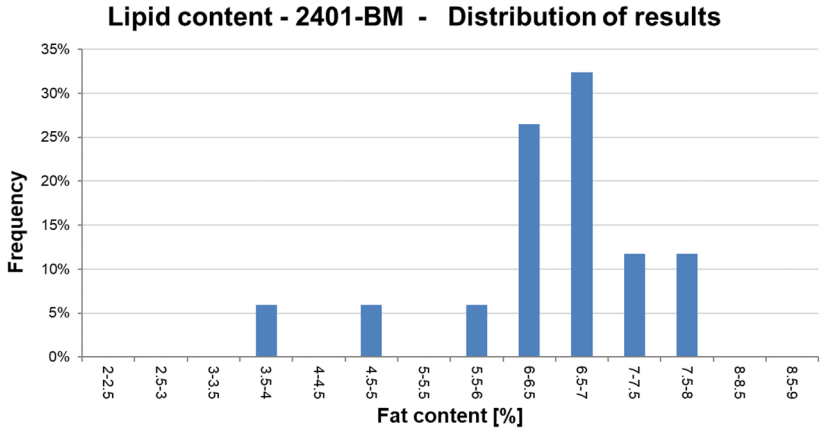

Figure 4: Frequency of reported values for the lipid content in % for Bovine Meat (2401-BM)

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 8). 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 9; [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 8). 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 9).

Table 8: 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 ≤ 0.01

Table 9: 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 value for BDE-154 (0.00733µg/kg) was in the range between the targeted and recommended LOQs for the test sample bovine meat (2401-BM). For the calculation of the BDE-28 assigned value, 24 out of 28 results were above the LOQs of the laboratories, showing that most participating laboratories are able to reliably achieve the recommended LOQ of 0.01 µg/kg product for feed. No assigned values could be calculated for BDE-28, -49 and -209. Calculated median values, for BDE-28 (0.00139 µg/kg product) and BDE-49 (0.00192 µg/kg product) were at the targeted LOQ. For BDE-209 the calculated median value from 21 participants' reported values was 0.227 µg/kg product which is around 20 times higher than the recommended LOQ (Figure 4).

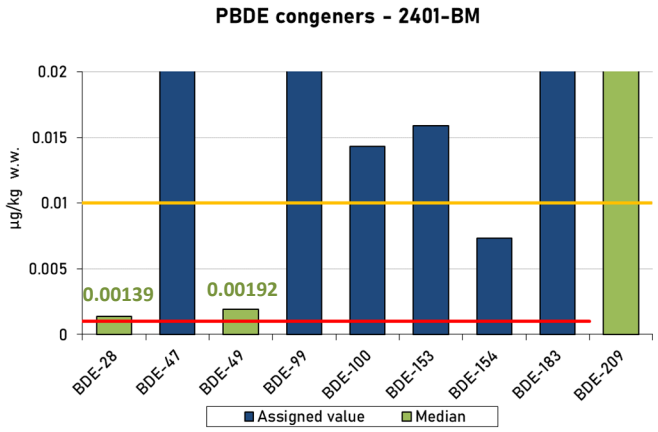

Figure 4: Comparison of assigned values for PBDE congeners with recommended LOQs (yellow line at 0.01 µg/kg wet weight and red line at 0.001 µg/kg wet weight) in bovine meat (2401-BM)

HBCDDs:

For α-, β-, γ-HBCDD and sum parameters no assigned values could be calculated, because less than 2/3 of all 21 reported results were above the LOQs (see calculation criteria section 4). Therefore, the median values were taken for comparison with the recommended LOQs. Median values for individual stereoisomers were 0.010 µg/kg product, which is at the recommended LOQ (Figure 5).

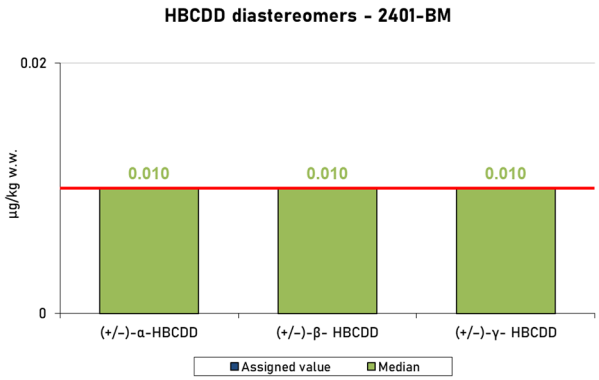

Figure 5: Comparison of assigned values for HBCDD stereoisomers with recommended LOQs (red line at 0.01 µg/kg wet weight) in bovine meat (2401-BM)

5. Scoring of results – Z-scores

5.1. Z-scores calculation

Criteria for successful participation of laboratories were based on the evaluation of the results of individual congeners and sum parameters. For evaluation of results of physico-chemical methods the z-scores were calculated according to the following formula:

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

x: *participant's result*

x_a: *assigned value*

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

For individual PBDE congeners, individual HBCDD diastereomers and PBDE and HBCDD sum parameters, the standard deviation for proficiency assessment σ_{prel} 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:

$|z\text{-score}| \leq 2$ *satisfactory performance*

$2 < |z\text{-score}| < 3$ *questionable performance (warning signal)*

$|z\text{-score}| \geq 3$ *unsatisfactory performance (action signal)*

5.2. PBDEs - Participants' z-scores

Table 10: Distribution of participants' z-scores for PBDEs for bovine meat (2401-BM)

Percentage of participants' results	|z-score| ≤ 2	2 < |z-score| < 3	|z-score| ≥ 3
BDE-47	75%	7%	18%
BDE-99	75%	7%	18%
BDE-100	74%	7%	19%
BDE-153	71%	4%	25%
BDE-154	71%	-	29%
BDE-183	71%	7%	22%
Sum of 8 PBDEs without BDE-209 (ub)	74%	7%	19%
Sum of 8 PBDE including BDE-209 (lb)	74%	7%	19%

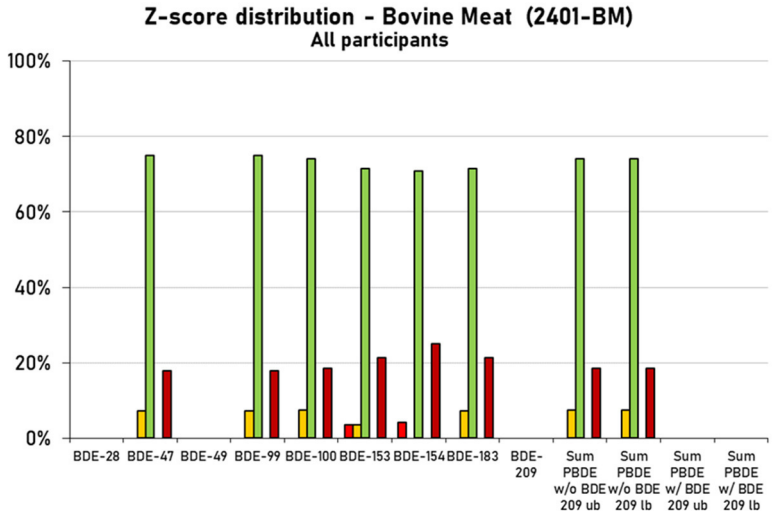

Figure 7: Distribution of participants' z-scores and NRLs only for PBDE congeners / sum parameters for bovine meat (2401-BM) [Green bars: -2 ≤ z-score ≤ 2, orange bars: -3 < z-score < -2, 2 < z-score < 3, red bars: z-score ≤ -3, z-score ≥ 3]

6. Participants' feedback

A questionnaire for feedback from participants of this EURL proficiency test was available as online survey between 24 April 2024 and 31 May 2024. The survey was anonymous, but participants could also give their laboratory name. The identity of the laboratories is kept confidential. The survey included several questions related to different topics (participants' information, organization of the proficiency test, PT test samples and evaluation of results and summary of data) and a possibility to include comments and further suggestions. In total, 10 laboratories (13 % of all PT participants) replied to this survey.

Table 11: Participating laboratories in the feedback survey

Type of laboratory	Answers
National Reference Laboratory (NRL)	5
Official Laboratory (OFL)	3
Commercial laboratory	1
Other (e.g. research and development)	1
No Answer	0

General aspects

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 

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?	

Additional comments:

- The amount of sample received was not enough for BIOSSAY and CONFIMATORY METHOD. We had subscribed for both participation. *EURL comment: if you need more material please let us know (the amount of one portion of reference material can be found in the announcement) you can add this as a comment during registration*
- Very suitible matrix, would be good to investigate and see if something could be done to achieve assigned value for BDE-209. Maybe spike PT with just that to make it easier to analyze? or if it is hard to press the LOQ to the level it needs to be, maybe allow a higher LOQ? *EURL POPs: BDE-209 was spiked 20-times higher than the recommended LOQ of 0.01µg/kg w.w.; unfortunately, no assigned value could be calculated*
- HBCDD: Sample was below the limit of quantification. *EURL POPs: The aim was to spike HBCDD at the recommended LOQ of 0.01µg/kg w.w.; unfortunately, no assigned value could be calculated at that low level*

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 Bovine Meat (2401-BM) 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

Bovine Meat – 2401-BM	
1	Assigned and median values –PBDEs, HBCDDs and lipid content
2	Participants' results – Tables – PBDEs, HBCDDs and lipid content
3	Participants' z-scores – Tables – PBDEs and lipid content
4	Participants' z-scores – Charts – PBDEs
5	Homogeneity and stability test – PBDEs
6	Overview participants' methods – Weighed sample, internal and recovery standards and comments
7	Overview participants' methods – Extractions, clean-up and detection
8	Overview participants' methods – Measurement uncertainty and Limit of Quantification

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EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food
24 March 2025

Annex 1: Assigned values of PBDEs and lipid content

Test sample - Bovine Meat (2401-BM)

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

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Bovine Meat (2401-BM)
PBDE - Assigned values

Analyte		Result µg/kg wet weight	**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						0.00139
BDE-47	2,2′,4,4′-tetrabromodiphenyl ether		0.0529	0.0095	0.0025	22	0.0559
BDE-49	2,2′,4,5′-tetrabromodiphenyl ether						0.00192
BDE-99	2,2′,4,4′,5-pentabromodiphenyl ether		0.0670	0.0097	0.0027	21	0.0702
BDE-100	2,2′,4,4′,6-pentabromodiphenyl ether		0.0143	0.0023	0.00061	22	0.0153
BDE-153	2,2′,4,4′,5,5′-hexabromodiphenyl ether		0.0159	0.0024	0.00068	20	0.0166
BDE-154	2,2′,4,4′,5,6′-hexabromodiphenyl ether		0.00733	0.0017	0.00047	20	0.0079
BDE-183	2,2′,3,4,4′,5′,6-heptabromodiphenyl ether		0.0547	0.011	0.0029	21	0.0603
BDE-209	2,2′,3,3′,4,4′,5,5′,6,6′-decabromodiphenyl ether						0.227
Sum of 8 PBDE	without BDE-209 (ub)		0.218	0.031	0.0086	20	0.231
Sum of 8 PBDE	without BDE-209 (lb)		0.214	0.028	0.0078	20	0.216
Sum of 9 PBDE	including BDE-209 (ub)						0.442
Sum of 9 PBDE	including BDE-209 (lb)						0.430

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Bovine Meat (2401-BM)
HBCDD - Assigned values

Analyte		Result µg/kg wet weight	**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)						0.0100
(+/–)-β- HBCDD	(1,2,5,6,9,10-hexabromo-(1R,2S,5R,6R,9R,10S)-rel-cyclododecane)						0.0100
(+/–)-γ- HBCDD	(1,2,5,6,9,10-hexabromo-(1R,2R,5R,6S,9S,10R)-rel-cyclododecane)						0.0100
Sum of α-, β-, γ-HBCDD	(ub)						0.0300
Sum of α-, β-, γ-HBCDD	(lb)						0
Total HBCDD	(using GC-methods)						0.00451

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Bovine Meat 2024 [EURL-PT-POP_2401-BM]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Bovine Meat (2401-BM)
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		6.54	0.731	0.16	32	6.58

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Bovine Meat 2024 [EURL-PT-POP_2401-BM]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food
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Annex 2: Participants' results of PBDEs, HBCDDs and lipid content

Test sample - Bovine Meat (2401-BM)

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

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Bovine Meat 2024 [EURL-PT-POP_2401-BM]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Bovine Meat (2401-BM)
PBDE - Results

LC	Sample	Result µg/kg wet weight	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)
3B	2401-BM		< 0.00173	0.0335	< 0.00173	0.0458	0.0094	0.0122	< 0.00518	0.0387	0.227	0.148	0.14	0.376	0.367
5	2401-BM		0.000588	0.0524	0.00123	0.0676	0.0142	0.0167	0.00755	0.0404	< 0.197	0.201	0.201	0.398	0.201
9	2401-BM		0.00192	0.0638	0.00236	0.073	0.0161	0.0289	0.012	0.0873	0.365	0.285	0.285	0.65	0.65
10	2401-BM		0.00057	0.041	0.00116	0.0534	0.0108	0.0123	0.00525	0.0423		0.167	0.167		
21	2401-BM		< 0.00133	0.0489	< 0.00133	0.0646	0.0169	0.0158	0.00743	0.0529	0.188	0.209	0.206	0.397	0.394
26	2401-BM		< 0.001	0.05	0.001	0.083	0.014	0.022	0.008	0.062		0.242	0.241		
32	2401-BM		0.00088	0.0587	0.0022	0.0714	0.016	0.0176	0.0097	0.0621		0.24	0.24		
34	2401-BM		0.00471	0.646	0.0161	0.887	0.21	0.206	0.0905	0.731	2.12	2.79	2.79	4.92	4.92
43	2401-BM														
49	2401-BM		< 0.019	0.85	0.015	1.2	0.22	0.27	0.11	0.94		3.6	3.6		
51	2401-BM		0.000609	0.053	0.00153	0.0607	0.0136	0.0146	0.007	0.052	0.177	0.203	0.203	0.38	0.38
53	2401-BM		< 0.005	0.029	< 0.005	0.03	0.008	0.008	< 0.005	0.026	0.082	0.115	0.1	0.197	0.182
58	2401-BM		0.0112	0.889	0.0208	1.07	0.224	0.262	0.122	0.919	3.72	3.52	3.52	7.23	7.23
59	2401-BM		0.000406	0.0486	0.000731	0.0644	0.0144	0.0141	0.00687	0.0516	0.268	0.201	0.201	0.469	0.469
60	2401-BM		< 0.0006	0.058	0.00166	0.0774	0.0161	0.0152	0.00675	0.0562	0.198	0.232	0.231	0.429	0.429
65	2401-BM		< 0.00181	0.0453	< 0.00181	0.0628	0.0129	0.016	0.00696	0.0529	0.254	0.201	0.197	0.454	0.451
67	2401-BM		< 0.01	0.056	< 0.01	0.068	0.0155	0.0146	< 0.01	0.0620	0.239	0.246	0.216	0.49	0.46
70	2401-BM		< 0.000358	0.0601	0.00146	0.0699	0.0166	0.0151	0.00621	0.0449		0.215	0.214		
79	2401-BM		< 0.001	0.026	< 0.001	0.0312	0.0065	0.0055	0.0028	0.0226	0.086	0.0965	0.0945	0.183	0.181
80	2401-BM		< 0.00037	0.0631		0.0472	0.0104	0.0171	0.00919	0.0484		0.199	0.198		
81	2401-BM		0.00144	0.0627	0.0021	0.0777	0.0175	0.017	0.00832	0.0608	0.121	0.248	0.248	0.369	0.369
83	2401-BM														
85	2401-BM		< 0.0013	0.0577	0.00184	0.0705	0.0153	0.0167	0.00867	0.0643	0.247	0.236	0.235	0.483	0.482
93	2401-BM		< 0.002	0.036	< 0.002	0.049	0.013	0.01	0.005	0.037	0.115	0.154	0.15	0.269	0.265
95	2401-BM		0.000582	0.0545		0.0722	0.014	0.0165	0.00743	0.0636	0.201	0.229	0.229	0.43	0.43
99	2401-BM		< 0.0036	0.0558		0.0715	0.0156	0.0161	0.00785	0.0605		0.231	0.227		
106	2401-BM		< 0.002	0.066	< 0.007	0.067	< 0.014	0.019	< 0.012	0.06	< 0.6	0.247	0.212	0.847	0.212
114	2401-BM		< 0.0005	0.0512		0.0747	0.0152	0.0196	0.0085	0.0624	0.219				
115	2401-BM														
124	2401-BM														
127	2401-BM		0.0187	0.947	0.0288	1.07	0.634	0.218	0.0986	0.872	4.28	3.89	3.89	8.17	8.17
34A	2401-BM		0.00716	0.654	0.0204	0.881	0.216	0.212	0.0968	0.84	2.2	2.93	2.93	5.13	5.13
34*	2401-BM		0.000367	0.0504	0.00126	0.0692	0.0164	0.0161	0.00706	0.057	0.166	0.218	0.218	0.383	0.383
34A*	2401-BM		0.000558	0.051	0.00159	0.0687	0.0168	0.0165	0.00755	0.0655	0.172	0.228	0.228	0.4	0.4
49*	2401-BM		< 0.0013	0.056	0.0013	0.071	0.015	0.018	0.0075	0.062		0.23	0.23		
127*	2401-BM		< 0.01	0.072	< 0.01	0.0817	0.0482	< 0.02	< 0.02	0.0663	< 1	0.296	0.296	0.621	0.621

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Bovine Meat (2401-BM)
HBCDD - Results

LC	Sample	Result µg/kg wet weight	(+/–)-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)
3B	2401-BM		< 0.00575	< 0.00575	< 0.00575				
5	2401-BM								
9	2401-BM								
10	2401-BM		< 0.01	< 0.01	< 0.01		0.03		
21	2401-BM		< 0.01	< 0.01	< 0.01		0.03		
26	2401-BM								
32	2401-BM								
34	2401-BM		0.0018	< 0.001	< 0.001		0.0038	0.0018	
43	2401-BM		< 0.2	< 0.02	< 0.04		0.26	0	
49	2401-BM		< 0.1	< 0.1	< 0.1		0.3		
51	2401-BM		< 0.01	< 0.01	< 0.01		0.03		
53	2401-BM		< 0.01	< 0.01	< 0.01		0.03	0	
58	2401-BM		< 0.006	< 0.005	< 0.005		0.016	0	
59	2401-BM								< 0.00312
60	2401-BM		0.0025	0.002	0.002		0.0065	0.0025	
65	2401-BM		< 0.006	< 0.006	< 0.006				
67	2401-BM		0.005	0.003	0.002		0.010	0.010	
70	2401-BM								
79	2401-BM								
80	2401-BM								
81	2401-BM		< 0.01	< 0.01	< 0.01		0.03	0	
83	2401-BM		< 0.03	< 0.03	< 0.03		0.09		
85	2401-BM		< 0.1	< 0.1	< 0.1		0.3	0.00001	
93	2401-BM		< 0.045	< 0.03	< 0.092		0.167	0	
95	2401-BM		< 0.0025	< 0.0035	< 0.00175		0.00683	0	
99	2401-BM		< 0.02	< 0.02	< 0.02				
106	2401-BM								
114	2401-BM								0.0059
115	2401-BM		0.011	< 0.006	0.079		0.096	0.09	
124	2401-BM		< 0.2	< 0.05	< 0.05		0.3	0	
127	2401-BM								
34A	2401-BM		0.0018	< 0.001	< 0.001		0.0038	0.0018	
34*	2401-BM		0.0018	< 0.001	< 0.001		0.0038	0.0018	
34A*	2401-BM		0.0018	< 0.001	< 0.001		0.0038	0.0018	
49*	2401-BM		< 0.1	< 0.1	< 0.1		0.3		
127*	2401-BM								

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Bovine Meat (2401-BM)
Lipid content - Results

LC	Sample	Result %	Lipid content PBDE	Lipid content HBCDD	Lipid content Mean
3B	2401-BM		7.0	7.0	7.0
5	2401-BM		6.5		6.5
9	2401-BM		6.2		6.2
10	2401-BM		7.4	7.4	7.4
21	2401-BM		6.2	6.2	6.2
26	2401-BM		5.8		5.8
32	2401-BM		6.6		6.6
34	2401-BM		7.8	7.8	7.8
43	2401-BM			4.8	4.8
49	2401-BM		6.7	6.6	6.7
51	2401-BM		5.7	6.6	6.2
53	2401-BM		6.9	6.9	6.9
58	2401-BM		6.7	6.4	6.6
59	2401-BM		6.3	6.3	6.3
60	2401-BM		6.5	6.5	6.5
65	2401-BM		5.9	5.9	5.9
67	2401-BM		4.0	4.0	4.0
70	2401-BM		7.2		7.2
79	2401-BM		3.7		3.7
80	2401-BM		6.1		6.1
81	2401-BM		7.4	7.4	7.4
83	2401-BM			4.5	4.5
85	2401-BM		7.2	7.2	7.2
93	2401-BM		6.7	6.7	6.7
95	2401-BM		6.9	6.9	6.9
99	2401-BM		6.8	6.8	6.8
106	2401-BM		6.1		6.1
114	2401-BM		6.7	6.7	6.7
115	2401-BM			6.6	6.59
124	2401-BM			6.2	6.2
127	2401-BM		7.6		7.6
34A	2401-BM		7.8	7.8	7.8
34*	2401-BM		7.8	7.8	
34A*	2401-BM		7.8	7.8	
49*	2401-BM		6.7	6.6	6.7
127*	2401-BM		7.6		7.6

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Bovine Meat 2024 [EURL-PT-POP_2401-BM]
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Annex 3: Participants' z-scores of PBDEs and lipid content - Tables

Test sample - Bovine Meat (2401-BM)

Z-scores of sum parameters and individual results

Calculation of z-score on basis of assigned value

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

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

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

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Bovine Meat 2024 [EURL-PT-POP_2401-BM]
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Bovine Meat (2401-BM)
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)
3B	2401-BM			-1.8		-1.6	-1.7	-1.2		-1.5			-1.6	-1.7		
5	2401-BM			0.0		0.0	0.0	0.3	0.2	-1.3			-0.4	-0.3		
9	2401-BM			1.0		0.4	0.6	4.1	3.2	3.0			1.5	1.7		
10	2401-BM			-1.1		-1.0	-1.2	-1.1	-1.4	-1.1			-1.2	-1.1		
21	2401-BM			-0.4		-0.2	0.9	0.0	0.1	-0.2			-0.2	-0.2		
26	2401-BM			-0.3		1.2	-0.1	1.9	0.5	0.7			0.6	0.6		
32	2401-BM			0.5		0.3	0.6	0.5	1.6	0.7			0.5	0.6		
34	2401-BM			56.1		61.2	68.4	59.8	56.7	61.8			59.0	60.2		
43	2401-BM															
49	2401-BM			75.3		84.6	71.9	79.9	70.0	80.9			77.6	79.1		
51	2401-BM			0.0		-0.5	-0.2	-0.4	-0.2	-0.2			-0.3	-0.3		
53	2401-BM			-2.3		-2.8	-2.2	-2.5		-2.6			-2.4	-2.7		
58	2401-BM			79.0		74.9	73.3	77.4	78.2	79.0			75.7	77.2		
59	2401-BM			-0.4		-0.2	0.0	-0.6	-0.3	-0.3			-0.4	-0.3		
60	2401-BM			0.5		0.8	0.6	-0.2	-0.4	0.1			0.3	0.4		
65	2401-BM			-0.7		-0.3	-0.5	0.0	-0.3	-0.2			-0.4	-0.4		
67	2401-BM			0.3		0.1	0.4	-0.4		0.7			0.6	0.0		
70	2401-BM			0.7		0.2	0.8	-0.3	-0.8	-0.9			-0.1	0.0		
79	2401-BM			-2.5		-2.7	-2.7	-3.3	-3.1	-2.9			-2.8	-2.8		
80	2401-BM			1.0		-1.5	-1.4	0.4	1.3	-0.6			-0.4	-0.4		
81	2401-BM			0.9		0.8	1.1	0.3	0.7	0.6			0.7	0.8		
83	2401-BM															
85	2401-BM			0.5		0.3	0.3	0.3	0.9	0.9			0.4	0.5		
93	2401-BM			-1.6		-1.3	-0.5	-1.9	-1.6	-1.6			-1.5	-1.5		
95	2401-BM			0.2		0.4	-0.1	0.2	0.1	0.8			0.3	0.4		
99	2401-BM			0.3		0.3	0.5	0.1	0.4	0.5			0.3	0.3		
106	2401-BM			1.2		0.0		1.0		0.5			0.7	0.0		
114	2401-BM			-0.2		0.6	0.3	1.2	0.8	0.7						
115	2401-BM															
124	2401-BM															
127	2401-BM			84.5		74.9	216.7	63.6	62.3	74.7			84.2	85.9		
34A	2401-BM			56.8		60.7	70.5	61.7	61.0	71.8			62.2	63.5		
34*	2401-BM			-0.2		0.2	0.7	0.1	-0.2	0.2			0.0	0.1		
34A*	2401-BM			-0.2		0.1	0.9	0.2	0.2	1.0			0.2	0.3		
49*	2401-BM			0.3		0.3	0.2	0.7	0.1	0.7			0.3	0.4		
127*	2401-BM			1.8		1.1	11.9			1.1			1.8	1.9		

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Bovine Meat 2024 [EURL-PT-POP_2401-BM]
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Bovine Meat (2401-BM)
Lipid content - Results

LC	Sample	Z-score [σ_p = 10 %]	Lipid content	Lipid content	Lipid content
			PBDE	HBCDD	Mean
3B	2401-BM		0.7	0.7	0.7
5	2401-BM		-0.1		-0.1
9	2401-BM		-0.5		-0.5
10	2401-BM		1.3	1.3	1.3
21	2401-BM		-0.5	-0.5	-0.5
26	2401-BM		-1.1		-1.1
32	2401-BM		0.1		0.1
34	2401-BM		1.9	1.9	1.9
43	2401-BM			-2.7	-2.7
49	2401-BM		0.2	0.1	0.2
51	2401-BM		-1.3	0.1	-0.6
53	2401-BM		0.6	0.6	0.6
58	2401-BM		0.3	-0.2	0.0
59	2401-BM		-0.4	-0.4	-0.4
60	2401-BM		-0.1	-0.1	-0.1
65	2401-BM		-1.0	-1.0	-1.0
67	2401-BM		-3.9	-3.9	-3.9
70	2401-BM		0.9		0.9
79	2401-BM		-4.3		-4.3
80	2401-BM		-0.7		-0.7
81	2401-BM		1.3	1.3	1.3
83	2401-BM			-3.1	-3.1
85	2401-BM		1.0	1.0	1.0
93	2401-BM		0.3	0.3	0.3
95	2401-BM		0.6	0.6	0.6
99	2401-BM		0.4	0.4	0.4
106	2401-BM		-0.6		-0.6
114	2401-BM		0.3	0.3	0.3
115	2401-BM			0.1	0.1
124	2401-BM			-0.5	-0.5
127	2401-BM		1.6		1.6
34A	2401-BM		1.9	1.9	1.9
34*	2401-BM		1.9	1.9	
34A*	2401-BM		1.9	1.9	
49*	2401-BM		0.2	0.1	0.2
127*	2401-BM		1.6		1.6

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Bovine Meat 2024 [EURL-PT-POP_2401-BM]
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Annex 4: Participants' z-scores of PBDEs - Charts

Test sample - Bovine Meat (2401-BM)

Z-scores of sum parameters and individual results

Calculation of z-score on basis of assigned value

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

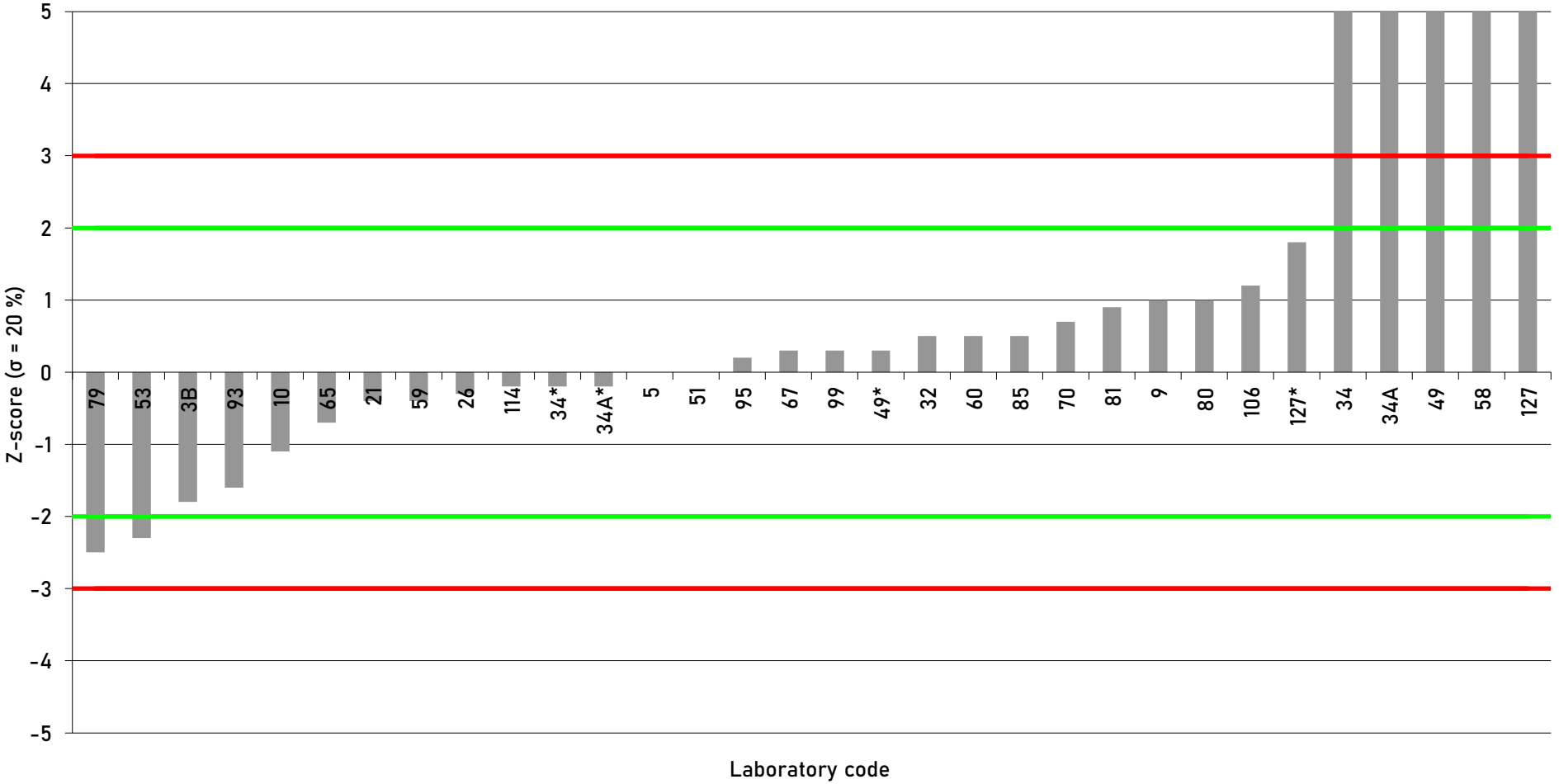
x_a: assigned value
x: participant's result
σ_p: fitness-for-purpose-based standard deviation for proficiency assessment
20%: Evaluated individual PBDE congeners and HBCDD diastereomers and sum

± 2 z-scores:
± 3 z-scores:

Bovine Meat (2401-BM)

BDE-47

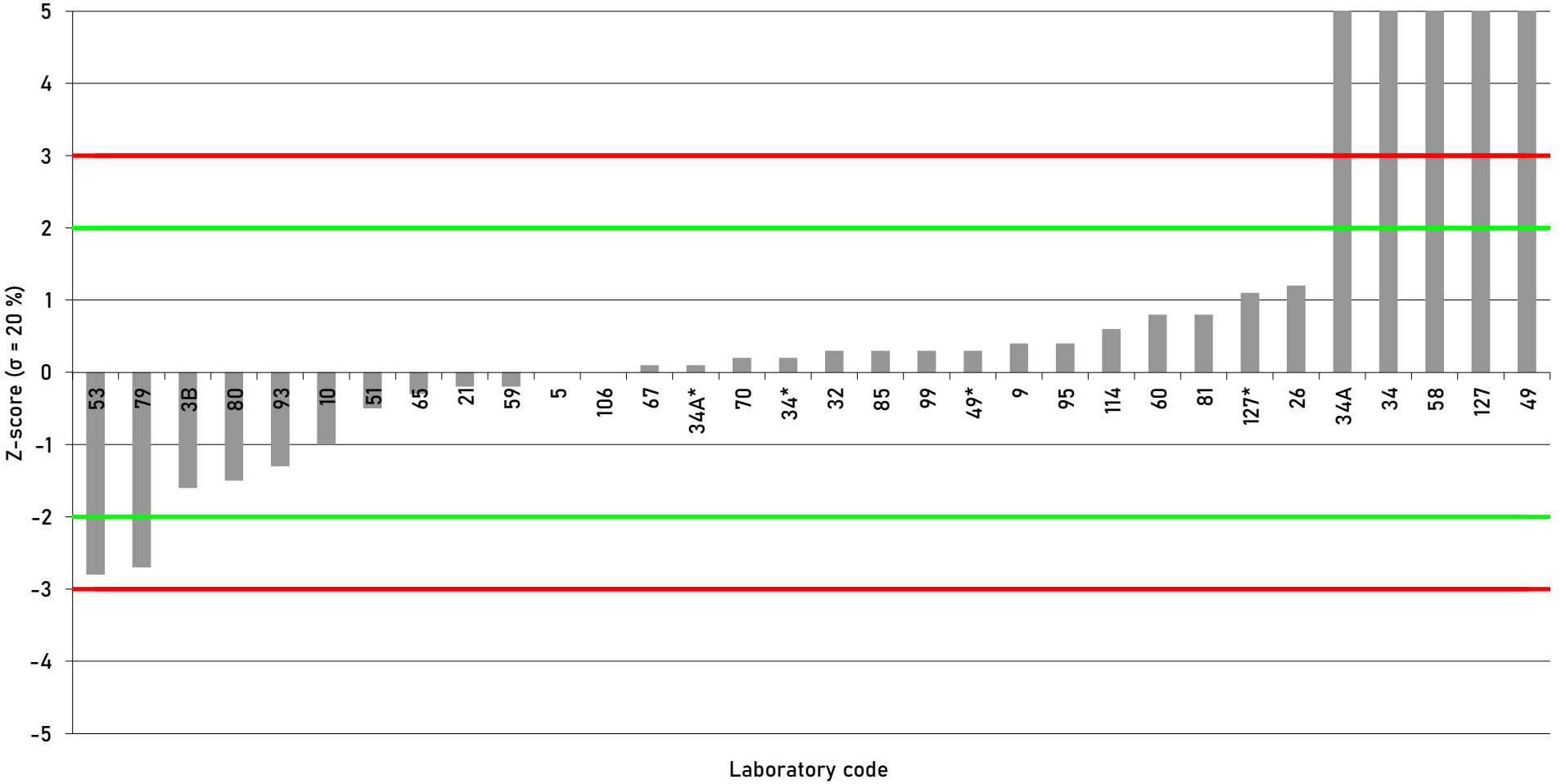
Assigned value: 0.0529 µg/kg wet weight



Bovine Meat (2401-BM)

BDE-99

Assigned value: 0.067 µg/kg wet weight



Bovine Meat (2401-BM)

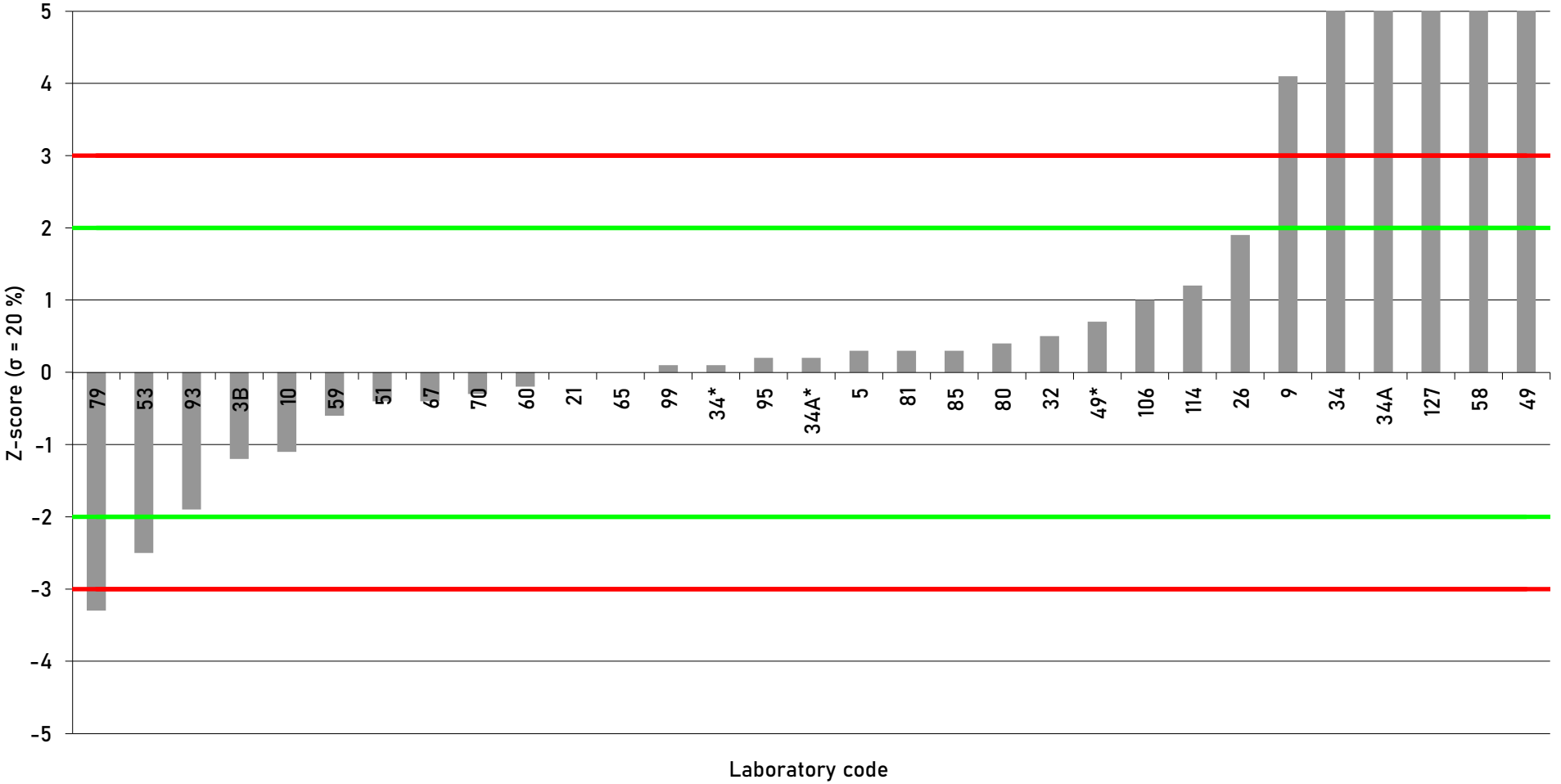
BDE-100

Assigned value: 0.0143 µg/kg wet weight

Bovine Meat (2401-BM)

BDE-153

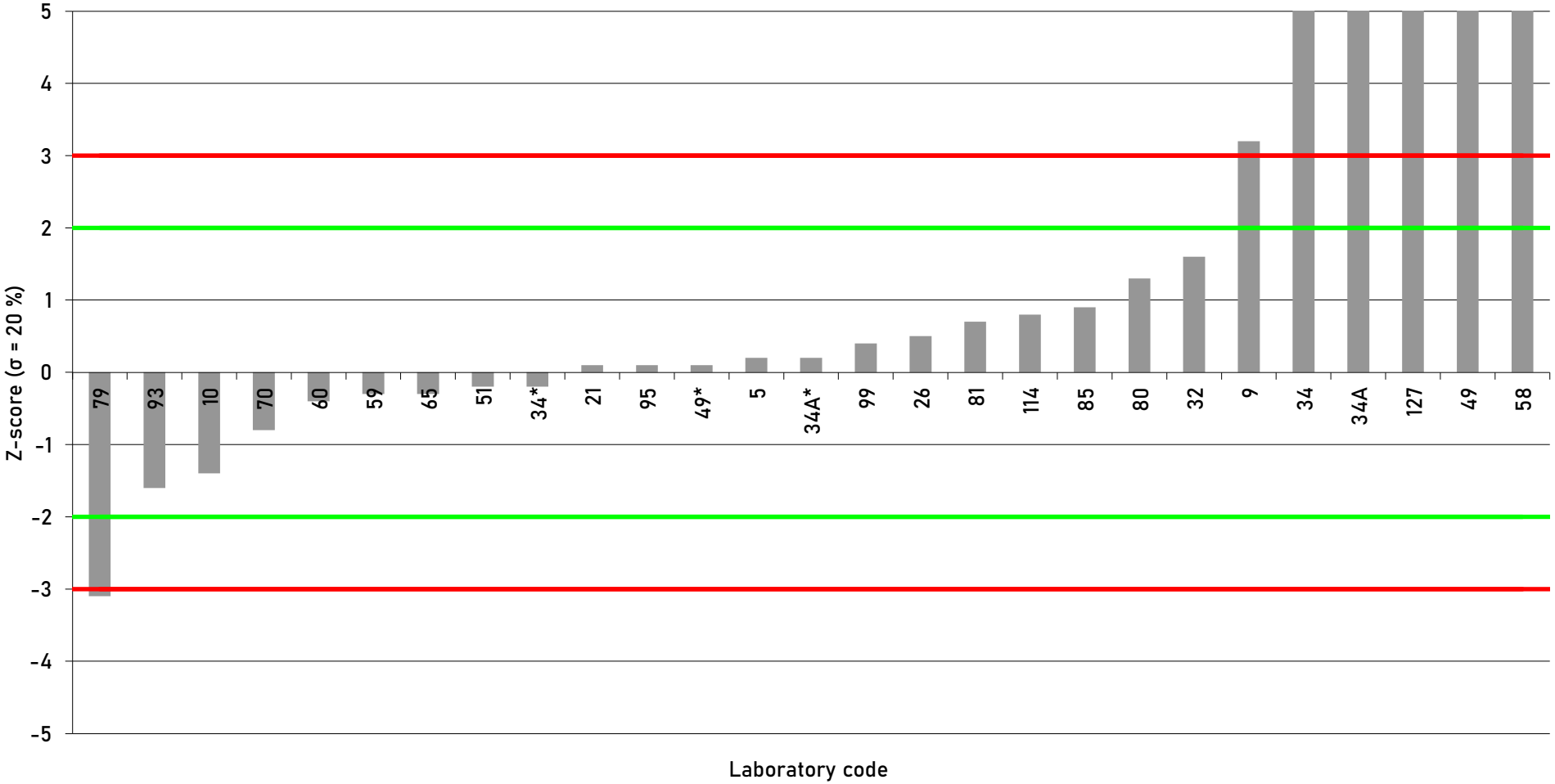
Assigned value: 0.0159 µg/kg wet weight



Bovine Meat (2401-BM)

BDE-154

Assigned value: 0.00733 µg/kg wet weight



Bovine Meat (2401-BM)

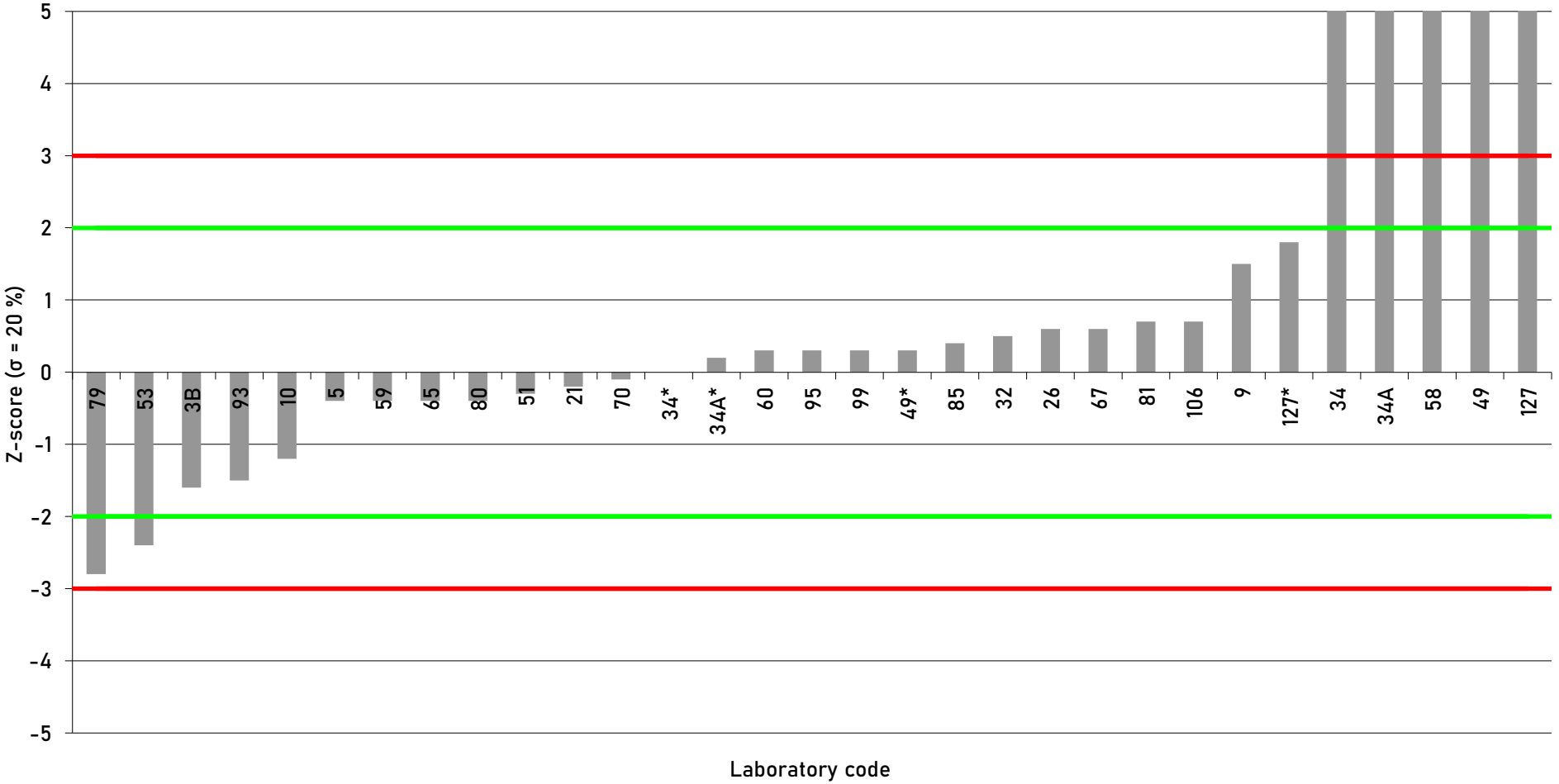
BDE-183

Assigned value: 0.0547 µg/kg wet weight

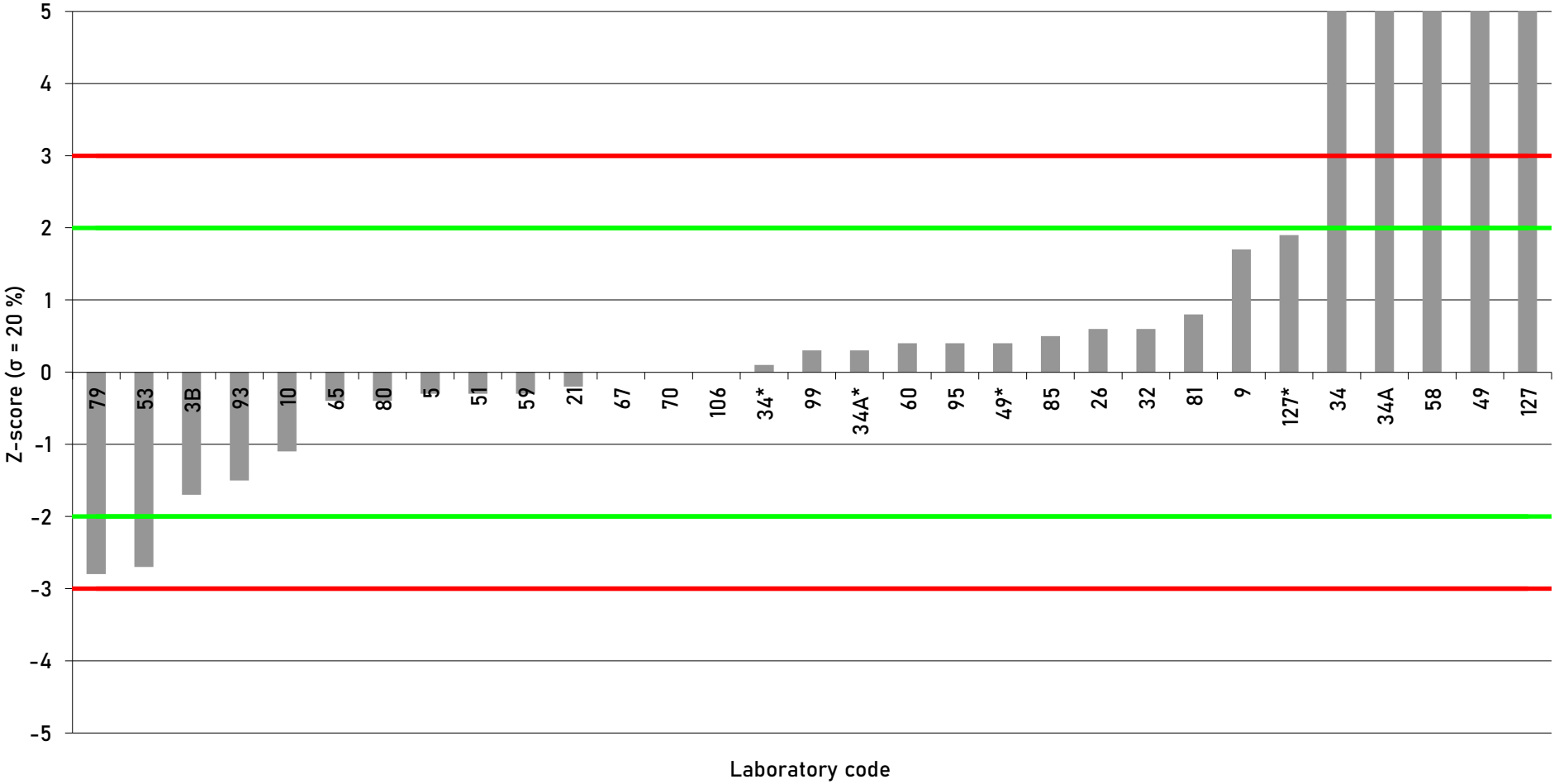
Bovine Meat (2401-BM)

Sum of PBDE without BDE-209 ub

Assigned value: 0.218 µg/kg wet weight



Bovine Meat (2401-BM)
Sum of PBDE without BDE-209 lb
Assigned value: 0.214 µg/kg wet weight



EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Bovine Meat 2024 [EURL-PT-POP_2401-BM]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food
24 March 2025

Annex 5: Test for sufficient homogeneity and stability for PBDEs

Test sample - Bovine Meat (2401-BM)

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Bovine Meat 2024 [EURL-PT-POP_2401-BM]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Bovine Meat (2401-BM)
PBDE - Homogeneity test - Data

Analyte	Result µg/kg wet weight	**Mean** (n = 10, duplicate analysis)	**Median** (n = 10, duplicate analysis)	**Relative standard deviation** [%]
Sum of PBDE without BDE-209 ub		0.185	0.175	9%
Sum of PBDE including BDE-209 ub		0.380	0.363	15%
BDE-28		0.000469	0.000473	10%
BDE-47		0.0451	0.0430	8%
BDE-49		0.00106	0.00106	7%
BDE-99		0.0610	0.0584	10%
BDE-100		0.0135	0.0131	9%
BDE-153		0.0129	0.0121	10%
BDE-154		0.00662	0.00658	10%
BDE-183		0.0440	0.0416	10%
BDE-209		0.210	0.193	26%

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Bovine Meat 2024 [EURL-PT-POP_2401-BM]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Bovine Meat (2401-BM)
Selected PBDE congeners - Homogeneity test - Data

Sample	Replicate	Result µg/kg wet weight	**BDE-47**
65	1		0.0509
	2		0.0498
129	1		0.0422
	2		0.0487
131	1		0.0435
	2		0.0423
160	1		0.0424
	2		0.0417
232	1		0.0409
	2		0.0426
238	1		0.0413
	2		0.0486
254	1		0.0475
	2		0.0493
272	1		0.0400
	2		0.0502
275	1		0.0426
	2		0.0424
277	1		0.0482
	2		0.0466
Cochran's C-test			
C			0.489
$C_{critical}$ (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 $\bar{x}$			0.0451
Standard deviation of sample averages s_x			0.0029
Wthin-sample standard deviation s_w			0.0033
Between-sample standard deviation s_s			0.0018
Standard deviation for proficiency assessment σ_{PT}			0.0090
s_s / σ_{PT}			0.200
Test for homogeneity ($s_s \leq 0.3\ \sigma_{PT}$)			passed

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFASs in Bovine Meat 2024 [EURL-PT-POP_2401-BM]
EURL for Halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Bovine Meat (2401-BM)
Selected congeners - Stability test - Data

Sample	Replicate	Result µg/kg wet weight	**BDE-47**
7	1		0.0421
	2		0.0431
82	1		0.0454
	2		0.0456
210	1		0.0472
	2		0.0507
Stability test			
General average (stability test) $\bar{y}$			0.046
General average (homogeneity test) $\bar{x}$			0.045
Standard deviation for proficiency assessment σPT			0.0090
$\lvert \bar{y} - \bar{x} \rvert$			0.00059
Test for stability ($\lvert \bar{y} - \bar{x} \rvert \leq 0.3$ σPT)			passed

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Bovine Meat 2024 [EURL-PT-POP_2401-BM]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food
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Annex 6: Overview participants' methods – Weighed sample, internal and recovery standards and comments

Test sample - Bovine Meat (2401-BM)

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Bovine Meat 2024 [EURL-PT-POP_2401-BM]

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

Bovine Meat (2401-BM)

Physico-chemical Methods PBDEs - Internal standards

LC	Sample	Weighed sample [g]	Use of isotope-labelled internal standards for … PBDE congeners (yes/no)
5	2401-BM	100	yes
9	2401-BM	11	YES
10	2401-BM	15.0	yes
21	2401-BM	94.2	yes
26	2401-BM	30	yes
32	2401-BM	61.7	yes (except for BDE-49)
34	2401-BM	4.0	YES
43	2401-BM		
49	2401-BM	1.2	yes
51	2401-BM	20	yes
53	2401-BM	15.0	yes
58	2401-BM	23.0	
59	2401-BM	25	yes
60	2401-BM	10	yes
65	2401-BM	5.0	yes
67	2401-BM	20.0	yes
70	2401-BM	0.415	Yes
79	2401-BM	10	yes
80	2401-BM	12.0	yes
81	2401-BM	15.0	yes
83	2401-BM		
85	2401-BM	20.6	
93	2401-BM	5.0	yes
95	2401-BM	32.5	yes
99	2401-BM	20	yes
106	2401-BM	40	yes
114	2401-BM	5.5	only for BDE-209
115	2401-BM		
124	2401-BM		
127	2401-BM	47.98	yes
003B	2401-BM	5g	yes
34A	2401-BM	4.0	YES

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Bovine Meat 2024 [EURL-PT-POP_2401-BM]

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

Bovine Meat (2401-BM)

Methods PBDEs - Internal Standards

LC	Sample		BDE 28	BDE 47	BDE 49	BDE 99	BDE 100	BDE 153	BDE 154	BDE 183	BDE 209
5	2401-BM		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
9	2401-BM		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
10	2401-BM										
21	2401-BM		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
26	2401-BM		13C-BDE 153	13C-BDE 153	13C-BDE 153	13C-BDE 153	13C-BDE 153	13C-BDE 153	13C-BDE 153	13C-BDE 153	
32	2401-BM		BDE-28	BDE-47	BDE-47	BDE-99	BDE-100	BDE-153	BDE-154	BDE-183	
34	2401-BM		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
43	2401-BM										
49	2401-BM		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
51	2401-BM		MBDE-28	MBDE-47	MBDE-47	MBDE-99	MBDE-100	MBDE-153	MBDE-154	MBDE-183	MBDE-209
53	2401-BM		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
58	2401-BM		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
59	2401-BM		BDE-28	BDE-47	BDE47	BDE-99	BDE-100	BDE-153	BDE-154	BDE-183	BDE-209
60	2401-BM		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
65	2401-BM		13C12-TriBDE (BDE-´28)	13C12-TetraBDE (BDE′,47)	13C12-TetraBDE (BDE′,47)	13C12-PentaBDE (BDE-´99)	13C12-PentaBDE (BDE-´99)	13C12-HexaBDE (BDE-´153)	13C12-HexaBDE (BDE-´153)	13C12-HeptaBDE (BDE-´183)	13C12-DecaBDE (BDE-´209)
67	2401-BM		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
70	2401-BM		13C-PBDE 28	13C-PBDE 47	13C-PBDE 47	13C-PBDE 99	13C-PBDE 99	13C-PBDE 153	13C-PBDE 154	13C-PBDE 183	
79	2401-BM		13C12-2,4,4'-TriBDE	13C12-2,2',4,4'-TetraBDE	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-DecaBDE
80	2401-BM		C13-BDE-28	C13-BDE-47		C13-BDE-99	C13-BDE-100	C13-BDE-153	C13-BDE-154	C13-BDE-183	C13-BDE-209
81	2401-BM		BDE-28	BDE-47	BDE-47	BDE-99	BDE-100	BDE-153	BDE-154	BDE-183	BDE-209
83	2401-BM										
85	2401-BM		13C-BDE-28	13C-BDE-47	13C-BDE-47	13C-BDE-99	13C-BDE-100	13C-BDE-153	13C-BDE-154	13C-BDE-183	13C-BDE-209
93	2401-BM		13C-BDE28	13C-BDE47	13C-BDE47	13C-BDE99	13C-BDE100	13C-BDE153	13C-BDE154	13C-BDE183	13C-BDE209
95	2401-BM		13C BDE 28	13C BDE 47		13C BDE 99	13C BDE 100	13C BDE 153	13C BDE 154	13C BDE 183	13C BDE 209
99	2401-BM		13C12-PBDE 28	13C12-PBDE 47	-	13C12-PBDE 99	13C12-PBDE 100	13C12-PBDE 153	13C12-PBDE 154	13C12-PBDE 183	-
106	2401-BM		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
114	2401-BM		13C-BDE-155	13C-BDE-155	13C-BDE-155	13C-BDE-155	13C-BDE-155	13C-BDE-155	13C-BDE-155	13C-BDE-155	13C-BDE-209
115	2401-BM										
124	2401-BM										
127	2401-BM		13C12 BDE-28	13C12 BDE-47	13C12 BDE-47	13C12 BDE-99	13C12 BDE-99	13C12 BDE-153	13C12 BDE-154	13C12 BDE-183	13C12 BDE-209
003B	2401-BM		13C12-TriBDE (BDE-´28)	13C12-TetraBDE (BDE′,47)	13C12-TetraBDE (BDE′,47)	13C12-PentaBDE (BDE-´99)	13C12-PentaBDE (BDE-´99)	13C12-HexaBDE (BDE-´153)	13C12-HexaBDE (BDE-´153)	13C12-HeptaBDE (BDE-´183)	13C12-DecaBDE (BDE-´209)
34A	2401-BM		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

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Bovine Meat 2024 [EURL-PT-POP_2401-BM]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Bovine Meat (2401-BM)
Methods PBDEs - Recovery Standards

LC	Sample		BDE 28	BDE 47	BDE 49	BDE 99	BDE 100	BDE 153	BDE 154	BDE 183	BDE 209
5	2401-BM		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
9	2401-BM		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
10	2401-BM										
21	2401-BM		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
26	2401-BM		13C-PCB 194	13C-PCB 194	13C-PCB 194	13C-PCB 194	13C-PCB 194	13C-PCB 194	13C-PCB 194	13C-PCB 194	
32	2401-BM		BDE-77	BDE-77	BDE-77	BDE-77	BDE-77	BDE-77	BDE-77	BDE-77	
34	2401-BM		13C BDE-77	13C BDE-77	13C BDE-77	13C BDE-77	13C BDE-77	13C BDE-138	13C BDE-138	13C BDE-138	
43	2401-BM										
49	2401-BM		13C-BDE 118	13C-BDE 118	13C-BDE 118	13C-BDE 118	13C-BDE 118	13C-BDE 118	13C-BDE 118	13C-BDE 118	13C-BDE 118
51	2401-BM		MBDE-79	MBDE-79	MBDE-79	MBDE-139	MBDE-139	MBDE-139	MBDE-139	MBDE-139	MBDE-206
53	2401-BM		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
58	2401-BM		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
59	2401-BM		PCB-138	PCB-138	PCB-138	PCB-138	PCB-138	PCB-138	PCB-138	PCB-138	PCB-138
60	2401-BM		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
65	2401-BM		13C12-HexaBDE (BDE-´138)	13C12-HexaBDE (BDE-´138)	13C12-HexaBDE (BDE-´138)	13C12-HexaBDE (BDE-´138)	13C12-HexaBDE (BDE-´138)	13C12-HexaBDE (BDE-´138)	13C12-HexaBDE (BDE-´138)	13C12-HexaBDE (BDE-´138)	13C12-HexaBDE (BDE-´138)
67	2401-BM							13C BDE-139			
70	2401-BM		13C-PCB 80								
79	2401-BM										
80	2401-BM		C13-BDE-139	C13-BDE-139		C13-BDE-139	C13-BDE-139	C13-BDE-139	C13-BDE-139	C13-BDE-139	C13-BDE-139
81	2401-BM		BDE-79	BDE-79	BDE-79	BDE-79	BDE-79	BDE-138	BDE-138	BDE-138	BDE-206
83	2401-BM										
85	2401-BM		13C-BDE-118	13C-BDE-118	13C-BDE-118	13C-BDE-118	13C-BDE-118	13C-BDE-118	13C-BDE-118	13C-BDE-118	13C-BDE-208
93	2401-BM		13C-BDE126	13C-BDE126	13C-BDE126	13C-BDE126	13C-BDE126	13C-BDE126	13C-BDE126	13C-BDE126	13C-BDE126
95	2401-BM		13C BDE 77	13C BDE 77		13C BDE 77	13C BDE 77	13C BDE 138	13C BDE 138	13C BDE 138	13C BDE 138
99	2401-BM		13C12-PCB 52	13C12-PCB 52	-	13C12-PCB 138	13C12-PCB 138	13C12-PCB 138	13C12-PCB 138	13C12-PCB 138	-
106	2401-BM		13C12 BDE-138	13C12 BDE-138	13C12 BDE-138	13C12 BDE-138	13C12 BDE-138	13C12 BDE-138	13C12 BDE-138	13C12 BDE-138	13C12 BDE-138
114	2401-BM										
115	2401-BM										
124	2401-BM										
127	2401-BM		13C12 BDE-126	13C12 BDE-126	13C12 BDE-126	13C12 BDE-126	13C12 BDE-126	13C12 BDE-126	13C12 BDE-126	13C12 BDE-126	13C12 BDE-126
003B	2401-BM		13C12-HexaBDE (BDE-´138)	13C12-HexaBDE (BDE-´138)	13C12-HexaBDE (BDE-´138)	13C12-HexaBDE (BDE-´138)	13C12-HexaBDE (BDE-´138)	13C12-HexaBDE (BDE-´138)	13C12-HexaBDE (BDE-´138)	13C12-HexaBDE (BDE-´138)	13C12-HexaBDE (BDE-´138)
34A	2401-BM		13C BDE-77	13C BDE-77	13C BDE-77	13C BDE-77	13C BDE-77	13C BDE-138	13C BDE-138	13C BDE-138	

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Bovine Meat 2024 [EURL-PT-POP_2401-BM]

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

Bovine Meat (2401-BM)

Methods PBDEs - Comments

LC	Sample		BDE 28	BDE 47	BDE 49	BDE 99	BDE 100	BDE 153	BDE 154	BDE 183	BDE 209
5	2401-BM										
9	2401-BM										
10	2401-BM										
21	2401-BM										
26	2401-BM										
32	2401-BM										
34	2401-BM										
43	2401-BM										
49	2401-BM										
51	2401-BM										
53	2401-BM		MU estimated at C<0.100 µg/kg	MU estimated at C<0.100 µg/kg	MU estimated at C<0.100 µg/kg	MU estimated at C<0.100 µg/kg	MU estimated at C<0.100 µg/kg	MU estimated at C<0.100 µg/kg	MU estimated at C<0.100 µg/kg	MU estimated at C<0.100 µg/kg	MU estimated at C<0.100 µg/kg
58	2401-BM										
59	2401-BM										
60	2401-BM										
65	2401-BM										
67	2401-BM										
70	2401-BM										
79	2401-BM										
80	2401-BM										Too high background
81	2401-BM										
83	2401-BM										
85	2401-BM										
93	2401-BM										
95	2401-BM										
99	2401-BM				not analysed						not analysed
106	2401-BM										
114	2401-BM				not analaysed						
115	2401-BM										
124	2401-BM										
127	2401-BM										
003B	2401-BM										
34A	2401-BM										

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Bovine Meat 2024 [EURL-PT-POP_2401-BM]

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

Bovine Meat (2401-BM)

Physico-chemical Methods HBCDDs - Internal standards

LC	Sample	Weighed sample [g]	Use of isotope-labelled internal standards for … HBCDD diastereomers (yes/no)
5	2401-BM		
9	2401-BM		
10	2401-BM	2.5	Yes
21	2401-BM	94.2	yes
26	2401-BM		
32	2401-BM		
34	2401-BM	4	YES
43	2401-BM	10	yes
49	2401-BM	1	yes
51	2401-BM	10	yes
53	2401-BM	15	yes
58	2401-BM	18	yes
59	2401-BM	25	yes
60	2401-BM	7	yes
65	2401-BM	5	yes
67	2401-BM	20	yes
70	2401-BM		
79	2401-BM		
80	2401-BM		
81	2401-BM	15	yes
83	2401-BM	10.0075	Yes
85	2401-BM	20.6	yes
93	2401-BM	10	yes
95	2401-BM	18.2	yes
99	2401-BM	5	yes
106	2401-BM		
114	2401-BM	5.5	no
115	2401-BM	4	yes
124	2401-BM	15	yes
127	2401-BM		
003B	2401-BM	5	yes
034A	2401-BM	4	YES

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Bovine Meat 2024 [EURL-PT-POP_2401-BM]

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

Bovine Meat (2401-BM)

Methods HBCDDs - Internal Standards

LC	Sample		(+/–)-α-HBCDD	(+/–)-β- HBCDD	(+/–)-γ- HBCDD
5	2401-BM				
9	2401-BM				
10	2401-BM		13C12-α-HBCDD	13C12-β-HBCDD	13C12-γ-HBCDD
21	2401-BM		13C-α-HBCD	13C-β-HBCD	13C-γ-HBCD
26	2401-BM				
32	2401-BM				
34	2401-BM		13C-α-HBCDD	13C-β-HBCDD	13C-γ-HBCDD
43	2401-BM		alpha- HBCDD (13C)	beta- HBCDD (13C)	gamma- HBCDD (13C)
49	2401-BM		alpha-HBCDD-C13	beta-HBCDD-C13	gamma-HBCDD-C13
51	2401-BM		13C12-alpha-HBCDD	13C12-beta-HBCDD	13C12-gamma-HBCDD
53	2401-BM		alfa-HBCD-13C12	gamma-HBCD-13C12	gamma-HBCD-13C12
58	2401-BM		13C alphaHBCDD	13C betaHBCDD	13C gammaHBCDD
59	2401-BM				
60	2401-BM		alpha-13C-HBCDD	beta-13C-HBCDD	gamma-13C-HBCDD
65	2401-BM		13C12-alpha-HBCD	13C12-beta-HBCD	13C12-gamma-HBCD
67	2401-BM		HBCDD 13C12	HBCDD 13C12	HBCDD 13C12
70	2401-BM				
79	2401-BM				
80	2401-BM				
81	2401-BM		alpha-HBCDD	beta-HBCDD	gamma-HBCDD
83	2401-BM		13C A-HBCD	13C B-HBCD	13C Y-HBCD
85	2401-BM		13C -α- HBCDD	13C -β- HBCDD	13C -γ- HBCDD
93	2401-BM		13C-a-HBCD	13C-B-HBCD	13C-j-HBCD
95	2401-BM		13C alpha HBCDD	13C gamma HBCDD	13C gamma HBCDD
99	2401-BM		alfa-HBCDD (13C12)	beta-HBCDD (13C12)	gamma-HBCDD (13C12)
106	2401-BM				
114	2401-BM		13C-BDE-155	13C-BDE-155	13C-BDE-155
115	2401-BM		13C12-a-HBCDD	13C12-a-HBCDD	13C12-a-HBCDD
124	2401-BM		13C-alpha-HBCDD	13C-beta-HBCDD	13C-gamma-HBCDD
127	2401-BM				
003B	2401-BM		13C12-alpha-HBCD	13C12-beta-HBCD	13C12-gamma-HBCD
34A	2401-BM		13C-α-HBCDD	13C-β-HBCDD	13C-γ-HBCDD

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Bovine Meat 2024 [EURL-PT-POP_2401-BM]

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

Bovine Meat (2401-BM)

Methods HBCDDs - Recovery Standards

LC	Sample		(+/–)-α-HBCDD	(+/–)-β- HBCDD	(+/–)-γ- HBCDD
5	2401-BM				
9	2401-BM				
10	2401-BM		N/A	N/A	N/A
21	2401-BM		13C-δ-HBCD	13C-δ-HBCD	13C-δ-HBCD
26	2401-BM				
32	2401-BM				
34	2401-BM		d18-β-HBCDD	d18-β-HBCDD	d18-β-HBCDD
43	2401-BM		D- beta-HBCDD	D- beta-HBCDD	D- beta-HBCDD
49	2401-BM		alpha-HBCDD-d18	beta-HBCDD-d18	gamma-HBCDD-d18
51	2401-BM		alpha-HBCDD-d18	beta-HBCDD-d18	gamma-HBCDD-d18
53	2401-BM		beta-HBCD-13C12	beta-HBCD-13C12	beta-HBCD-13C12
58	2401-BM		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
59	2401-BM				
60	2401-BM				
65	2401-BM		d18-beta-HBCD	d18-beta-HBCD	d18-beta-HBCD
67	2401-BM				DBHBCD
70	2401-BM				
79	2401-BM				
80	2401-BM				
81	2401-BM		D18-beta-HBCDD	D18-beta-HBCDD	D18-beta-HBCDD
83	2401-BM		D18-B-HBCD	D18-B-HBCD	D18-B-HBCD
85	2401-BM		d18 -α- HBCDD	d18 -β- HBCDD	d18 -γ- HBCDD
93	2401-BM		none	none	none
95	2401-BM				
99	2401-BM				
106	2401-BM				
114	2401-BM				
115	2401-BM				
124	2401-BM		-	-	-
127	2401-BM				
003B	2401-BM		d18-beta-HBCD	d18-beta-HBCD	d18-beta-HBCD
34A	2401-BM		d18-β-HBCDD	d18-β-HBCDD	d18-β-HBCDD

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Bovine Meat 2024 [EURL-PT-POP_2401-BM]

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

Bovine Meat (2401-BM)

Methods HBCDDs - Comments

LC	Sample		(+/–)-α-HBCDD	(+/–)-β- HBCDD	(+/–)-γ- HBCDD
5	2401-BM				
9	2401-BM				
10	2401-BM				
21	2401-BM				
26	2401-BM				
32	2401-BM				
34	2401-BM				
43	2401-BM				
49	2401-BM				
51	2401-BM				
53	2401-BM		MU estimated at C<0.020 µg/kg	MU estimated at C<0.020 µg/kg	MU estimated at C<0.020 µg/kg
58	2401-BM				
59	2401-BM				
60	2401-BM				
65	2401-BM				
67	2401-BM				
70	2401-BM				
79	2401-BM				
80	2401-BM		Not measured	Not measured	Not measured
81	2401-BM				
83	2401-BM		<LOQ	<LOQ	<LOQ
85	2401-BM				
93	2401-BM				
95	2401-BM				
99	2401-BM				
106	2401-BM				
114	2401-BM				
115	2401-BM				
124	2401-BM				
127	2401-BM		not determined	not determined	not determined
003B	2401-BM				
34A	2401-BM				

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24 March 2025

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

Test sample - Bovine Meat (2401-BM)

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Bovine Meat 2024 [EURL-PT-POP_2401-BM]
EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Bovine Meat (2401-BM)
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]
5	2401-BM	Freeze-drying	Twisselman	Toluene / Cyclohexan (50/50)	6		
9	2401-BM	Freeze Drying	SOXHLET	HEXANE/DCM (1/1)	24H		
10	2401-BM		Soxhlet	DCM/Hexane (1/1)	12h	boiling	
21	2401-BM	homogenize	shaking with solvent	hexane/acetone 50/50	0.1 for each step (4 consecutive extractions)	22	
26	2401-BM	drying	Soxhlet	n-Hexane	2	70	
32	2401-BM	sample mixed with sodium sulphate before extraction	Soxhlet	DCM:Hexane (50:50)	24 h		
34	2401-BM	drying	PLE	Toluene/Acetone			
43	2401-BM						
49	2401-BM	none	Soxhlet (water separator)	Toluene	8		
51	2401-BM	mixing with natriumsulfate	cold extraction	DCM/Cyclohexane 1/1	roughly 2-3 hours	ambient	ambient
53	2401-BM	no	QuEChERs like-extraction	Ethyl acetate	no	no	no
58	2401-BM	freeze-dried	Accelerated Solvent Extraction (ASE) - ASE 350	dichloromethane/ n-hexane (50/50)	0.35	100	10
59	2401-BM	Homogenisation, isolation of fat + lipid content (%)	Soxhlet, ASE	Soxhlet: hexane, ASE: hexane:DCM (4:1,v/v)	Soxhlet: 8 h, ASE: 30 min	ASE: 120 °C	ASE: 10 MPa
60	2401-BM		cold extraction	DCM/Cyclohexane		RT	
65	2401-BM	homogenization, drying with polyacrylamide	cold extraction	hexane		room temperature	
67	2401-BM	Na2SO4	Soxhlet	Hex/DCM=1/1	16	/	/
70	2401-BM	Mixing with sodium sulfate	Soxhlet	DCM	18	boiling	
79	2401-BM		ASE	Toluene/Cyclohexane (50/50)	0.3	120	10
80	2401-BM		ASE	pentane/acetone 88/12	0,5	80	1500
81	2401-BM	Mixed with sodium sulphate	ASE	Hexane/Acetone (70:30)	1	125	10.3
83	2401-BM						
85	2401-BM	drying	ASE	toluene (85%) / ethanol (15%)	20 min	120	10
93	2401-BM		LL Extraction/Adding silica/H2SO4 40% and then extraction with 10 ml hexane (2x). Evaporated to dryness.	Ethylacetate/Hexane	over night	Roomtemp	
95	2401-BM	freeze drying, mixing with sodium sulphate	PSE	DCM:n-hexane (1:1)	3 cycles per 2 minutes	100°C	10
99	2401-BM	thorough homogenization, drying with anhydrous Sodium Sulfate	Soxhlet (ultrasonic bath, agitate)	cyclohexane:2-propanol 2:1	21	109	0.1
106	2401-BM	-	Soxhlet	hexane-dichloromethane 1:1	20h	boiling point of solvent mixture	-
114	2401-BM	no	manually liuid-liquid extraction	n-hexane:acetone 1/1			
115	2401-BM						
124	2401-BM						
127	2401-BM	Drying with 150g sodium sulfate	Soxhlet	Toluene	24h		
003B	2401-BM	homogenization, drying with polyacrylamide	cold extraction	hexane		room temperature	
034A	2401-BM	drying	PLE	Toluene/Acetone			

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

Bovine Meat (2401-BM)
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
5	2401-BM		no	yes	no	yes	no	acidic treatment	50
9	2401-BM		NO	YES	NO	YES	NO	NO	20
10	2401-BM		Yes	Yes	Yes	No	Yes	Sufuric acid treatment of the final extract	50
21	2401-BM		no	yes	no	yes	no		25
26	2401-BM		no	yes	no	yes	yes		200
32	2401-BM		no	yes	no			basic set of "power-prep system" columns	40
34	2401-BM		YES	YES	YES	NO	YES		50
43	2401-BM								
49	2401-BM		no	yes	no	yes	yes	Silica/AgNO3	100
51	2401-BM		no	yes	no	yes	yes	AgNO3 (MIURA)	50
53	2401-BM		yes	yes	no	no	no	no	250
58	2401-BM		no	yes	yes	yes	no	no	100
59	2401-BM		no	yes	yes	yes	yes	PowerPrep FMS columns (basic-neutral silica, alumina, carbon)	20
60	2401-BM		no	yes	no	yes	yes		100
65	2401-BM		no	yes	no	yes	no		100
67	2401-BM		yes	yes	no	no	yes		20-30 ul
70	2401-BM		No	Yes	No	Yes	No	-	50
79	2401-BM		yes	yes	no	yes	no		50
80	2401-BM		no	yes	no	yes	yes	Silver nitrate column	1000
81	2401-BM		no	yes	no	yes	no	acid hydrolysis with sulphuric acid	40
83	2401-BM								
85	2401-BM		no	yes	no	yes	yes		500
93	2401-BM		no	yes	no	no	no	no	250 µl
95	2401-BM		no	yes	no	no	yes		50
99	2401-BM		no	yes	no	yes	yes	basic silica, silver nitrate silica	30
106	2401-BM		no	yes	no	no	no	-	250
114	2401-BM		no	no	no	no	no	sulfuric acid treatment, silica gel column	100
115	2401-BM								
124	2401-BM								
127	2401-BM			yes	no	no	no		50
003B	2401-BM		no	yes	no	yes	no		100
034A	2401-BM		YES	YES	YES	NO	YES		50

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Bovine Meat 2024 [EURL-PT-POP_2401-BM]

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

Bovine Meat (2401-BM)

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
5	2401-BM		splitless	2.0	DB-5HT	HRMS (Autospec Ultima Waters)
9	2401-BM		SPLITLESS	1	DB-5ms 40m x 0,18 mm x 0,18 um	HRMS
10	2401-BM		Splitless	1	RTX-1614	Autospec Premier HRMS
21	2401-BM		splitless	1	Rtx-1614	HRMS
26	2401-BM		MMI	2	DB-XLB	MS/MS
32	2401-BM		Splitless	1	DB-5MS (30 m, 0.25 mm id, 0,25 mm film)	HRMS (Mat-95 XP)
34	2401-BM		Splitless	2	HT8PCB	GC-HRMS
43	2401-BM					
49	2401-BM		PTV	1	ZB - Semi Volatiles, 20m, 0.18 mm, 0.18µm	MS/MS
51	2401-BM		large volume	4	Rtx-1614 (15m x 0,25 mm x 0,1 µm) + 1,5m retention gap	MS/MS
53	2401-BM		PTV	10	DB5HT 15 m x 0.25 mm; 0.1 um	MS/MS
58	2401-BM		PTV Splitless	1	15 m RTX 1614	HRMS
59	2401-BM		splitless	2	DB-5ht (15m x 0,25mm x 0,10um)	HRMS
60	2401-BM		splitless	2	DB 5 MS	HRMS
65	2401-BM		pulsed splitless	2	Diphenyl-/dimethylpolysiloxan (5%/95%)	MS/MS
67	2401-BM		splitless MMI	5	ZB Semivolatiles (20m, 0,18/0,18)	MS/MS
70	2401-BM		Splitless	1.5	DB-5MS	HRMS, DFS
79	2401-BM		PTV	5	5% diphenyl, 95% dimethyl polysiloxane (RTX-1614)	MS/MS
80	2401-BM		PTV	1	DB-5MS	HRMS
81	2401-BM		PTV	2	Rtx-1614 30m x 0.25mm x 0.1µm	HRMS
83	2401-BM					
85	2401-BM		PTV	5	DB5-MS (5%-phenyl)-methylpolysiloxane	MS/MS
93	2401-BM		PTV	10	Rtx-CIPesticides	GC-HRMS
95	2401-BM		pulsed splitless	2	RTX-1614	HRMS
99	2401-BM		pulsed splitless	1	DB-5MS (60m x 0,25mm x 0,10 µm)	HRMS (R>10000)
106	2401-BM		splitless	2	Rxi-5 Sil MS	HRMS
114	2401-BM		NCI	6	DB-5MS, 15m x 0,25 mmx 0,10 um	LRMS
115	2401-BM					
124	2401-BM					
127	2401-BM		PTV	1	15m x 0.25mm x0.25um Film RXi-5ms	HRMS Q-Exactive Orbitrap at R=60000
003B	2401-BM		pulsed splitless	2	Diphenyl-/dimethylpolysiloxan (5%/95%)	MS/MS
034A	2401-BM		Splitless	2	HT8PCB	GC-HRMS

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

Bovine Meat (2401-BM)
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]
5	2401-BM						
9	2401-BM						
10	2401-BM	Drying using sodium sulfate	Ultrasound assisted extraction	DCM:Hex (1:1, v/v)	30 min	Room temp.	N/A
21	2401-BM	homogenize	shaking with solvent	hexane/acetone 50/50	0.1 for each step (4 consecutive extractions)	22	
26	2401-BM						
32	2401-BM						
34	2401-BM	drying	PLE	Toluene/Acetone			
43	2401-BM	freeze-drying	Ultra turrax	Hexane dichloromethane 50/50	0.03	20	ambiante
49	2401-BM		Soxhlet	Toulene	12	unknown	ambient pressure
51	2401-BM	mixing with natriumsulfate	cold extraction	DCM/Hexan 1/1	roughly 2-3 hours	ambient	ambient
53	2401-BM		QuEChERS-like	Ethyl Acetate			
58	2401-BM	Freeze drying.	ASE 350	dichloromethane/hexane (50/50)	0.35	100	10
59	2401-BM	Homogenisation, isolation of fat + lipid content (%)	Soxhlet, ASE	Soxhlet: hexane, ASE: hexane:DCM (4:1,v/v)	Soxhlet: 8 h, ASE: 30 min	ASE: 120 °C	ASE: 10 MPa
60	2401-BM		Cold Extraction	DCM/Cyclohexane			
65	2401-BM	homogenization	Quechers	acetonitrile/water (1/1)	10 min	room temperature	
67	2401-BM	Na2SO4	Soxhlet	Hex/DCM=1/1	16	/	/
70	2401-BM						
79	2401-BM						
80	2401-BM						
81	2401-BM	Mixed with sodium sulphate	ASE	Hexane/Acetone (70:30)	1	125	10.3
83	2401-BM	None	ASE	Hexane:Acetone 50:50	0.5	100	10.3
85	2401-BM	drying	ASE	toluene (85%) / ethanol (15%)	20 min	120	10
93	2401-BM	Sodium Sulfate	ASE	Hexane/Acetone 1:1	30 minutes	100	1500 psi
95	2401-BM	freeze drying, mixing with sodium sulphate	PSE	DCM:n-hexane (1:1)	3 cycles per 2 minutes	100°C	10
99	2401-BM	drying	shaking with dichlormethan : acetone 2:1 for 90 min	100 ml of dichlormethan : acetone 2:1	90 min	ambient	0.10
106	2401-BM						
114	2401-BM	no	manually liquid-liquid extraction	n-hexane:acetone 1/1			
115	2401-BM		Soxleth	Acetone/Hexane (1/1)	6		
124	2401-BM	freeze drying	Twisselmann	Cyclohexan / Toluol (1:1, v/v)	6	boiling point	
127	2401-BM						
003B	2401-BM	homogenization	Quechers	acetonitrile/water (1/1)	10 min	room temperature	
034A	2401-BM	drying	PLE	Toluene/Acetone			

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Bovine Meat 2024 [EURL-PT-POP_2401-BM]

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

Bovine Meat (2401-BM)

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	2401-BM								
9	2401-BM								
10	2401-BM		No	Yes (both)	Yes	No	No	Direct H2SO4 treatment	50
21	2401-BM		no	yes	no	no	no		1000
26	2401-BM								
32	2401-BM								
34	2401-BM		YES	YES	NO	NO	NO	liquid liquid extraction	50
43	2401-BM		No	Yes	No	No	No	No	200
49	2401-BM		no	yes	yes	no	no	no	500
51	2401-BM		no	yes	no	no	no	no	400
53	2401-BM		yes	no	no	no	no	Extrelut NT-3 column, acidic for H2SO4 tandem Si 1g/6 mL column	250
58	2401-BM		no	yes	no	no	no		50
59	2401-BM		no	yes	no	no	no	no	20
60	2401-BM		yes	no	no	no	no	silicagel column	250
65	2401-BM		no	yes	no	no	no		1000
67	2401-BM		yes	yes	no	no	yes		20-30 ul
70	2401-BM								
79	2401-BM								
80	2401-BM								
81	2401-BM		no	yes	no	no	no	acid hydrolysis with sulphuric acid	200
83	2401-BM		No	Yes	Yes	No	No	None	100
85	2401-BM		no	yes	no	no	no	no	500
93	2401-BM		no	yes	no	no	no		500 µl
95	2401-BM		no	yes	no	no	no	no	50
99	2401-BM		yes	yes	no	no	no		500
106	2401-BM								
114	2401-BM		no	no	no	no	no	sulfuric acid treatment, silica gel column	100 ul
115	2401-BM		no	no	no	no	no	Conc. sulphuricacid	200
124	2401-BM		no	yes	yes	no	no	no	100
127	2401-BM								
003B	2401-BM		no	yes	no	no	no		1000
034A	2401-BM		YES	YES	NO	NO	NO	liquid liquid extraction	50

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Bovine Meat 2024 [EURL-PT-POP_2401-BM]

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

Bovine Meat (2401-BM)

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
5	2401-BM				
9	2401-BM				
10	2401-BM	-	5	C18-RP-HPLC	HRMS
21	2401-BM	LC		Acquity BEH C18 150 mm	MS/MS
26	2401-BM				
32	2401-BM				
34	2401-BM		15	Hypersil Gold	MS/MS
43	2401-BM	UPLC	5	Eclipse Plus C18, 2,1 x 100mm	MS/MS
49	2401-BM	direct injection	5, 10, 20, 40	reversed phase C18 (Eclipse XDB-C18 5µm 4,6x150mm)	MS/MS
51	2401-BM		15	C18	MS/MS
53	2401-BM		20 µL	KINETEX 2.6um XB-C18 100A (100 x 2.1 mm) (PHENOMENEX)	LC-MS/MS
58	2401-BM		10	Hypersil Gold C18, 100x2,1mm, 1,9µm	MS/MS
59	2401-BM	splitless	3	DB-5ht (15m x 0,25mm x 0,10um)	HRMS
60	2401-BM		10	PR 18	LC-MS/MS
65	2401-BM	standard	10	C18	MS/MS
67	2401-BM	LC	70	Luna PFP	MS/MS
70	2401-BM				
79	2401-BM				
80	2401-BM				
81	2401-BM		5	BEH C18 (50 mm x 2.1 mm; 1.7 µm)	HRMS
83	2401-BM		10	C18	HRMS (hybrid quadrupole/orbitrap)
85	2401-BM	ESI negative	5 µl	C18 column	MS/MS
93	2401-BM		10	C18 (Waters symmetry), 150 mm x 2.1 mm x 3.5 µm	LCMSMS
95	2401-BM	LC	5	C-18	MS/MS
99	2401-BM	normal	10	C18	MS/MS
106	2401-BM				
114	2401-BM	NCI	6	DB-5MS, 15 m x 0,25 mm id, 0,10 um	LRMS
115	2401-BM	HPLC injection	5	Acquity BEH C18 (1.7 µm, 2.1*100 mm)	MS/MS
124	2401-BM	HPLC	2	C18	MS/MS
127	2401-BM				
003B	2401-BM	standard	10	C18	MS/MS
034A	2401-BM		15	Hypersil Gold	MS/MS

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24 March 2025

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

Test sample - Bovine Meat (2401-BM)

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Bovine Meat 2024 [EURL-PT-POP_2401-BM]
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Bovine Meat (2401-BM)
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
5	2401-BM			
9	2401-BM			
10	2401-BM		Established from quality control charts (2*RSD)	
21	2401-BM			
26	2401-BM		estimated with validation data	
32	2401-BM			
34	2401-BM	S/N > 3		
43	2401-BM			
49	2401-BM	blank + 3s		
51	2401-BM		top-down approach as discribed in the "Guidance document on measurment uncertainty for laboratories performing PCDD/F and PCB analysis"	
53	2401-BM	Samples spiked at LOQ level (5 pg/g; 100ppg for BDE 209); for BDE 47 LOQ (15 pg/g) was estimated from; procedural blanks run within the sample batch	Nordtest	
58	2401-BM	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.	
59	2401-BM	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	
60	2401-BM	S/N like dioxin	QC-samples, PT results	
65	2401-BM			
67	2401-BM			
70	2401-BM			
79	2401-BM	Estimation by sample	by method validation	
80	2401-BM			
81	2401-BM	The LOQs were estimated using the approach "Congener-based LOQs from S/N Calculations".This methodology is reported in the Guidance Document on the Estimation of LOD and LOQ for Measurements in the Field of Contaminants in Feed and Food.	The MU was estimated using the "semi-empirical approach" based on the ISO Guide to the expression of Uncertainty in Measurement applied to analytical chemistry by EURACHEM/CITAC. This approach is reported in the Guidance Document on Measurement Uncertanty for Laboratories performing PCDD/F and PCB Analysis using Isotope Dilution Mass Spectrometry	
83	2401-BM			
85	2401-BM			
93	2401-BM	0.002 - 0.080 µg/kg; according to NEN 7777	according to NEN 7777 expanded measurement uncertainty	
95	2401-BM	TargetLynx calculation sample and compound specific for S/N 10:1 (Peak to Peak)	expanded RSD for 10 repeated reference material anlayses	
99	2401-BM			
106	2401-BM			
114	2401-BM	standarddeviation of the blanks multiplied with a factor of 6	MU was calculated by the root of the sum of the measurement uncertainty for reproducibility and recovery including bias. Expanded uncertainty is then calculated by multiplying MU with a factor of 2	We are not accreditated for meat. The method used is the one for fish. Recoveries are the ones for fish.
115	2401-BM			
124	2401-BM			
127	2401-BM	S/N =5	Estimated measurement uncertainty from several individual sample analyses with similar matrices	
003B	2401-BM			
034A	2401-BM	S/N > 3		

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Bovine Meat (2401-BM)

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
5	2401-BM										0.19730843
9	2401-BM		0.00007	0.00003	0.00006	0.00011	0.00008	0.00014	0.00008	0.00017	0.00621
10	2401-BM		0.000088	0.000164	0.000255	0.000496	0.000461	0.000402	0.000284	0.000340	
21	2401-BM		0.00133		0.00133						
26	2401-BM		0.001								
32	2401-BM		0.00001	0.00001	0.00001	0.00002	0.00002	0.00002	0.00003	0.00018	
34	2401-BM										
43	2401-BM										
49	2401-BM		0.019	0.35	0.011	0.12	0.029	0.007	0.008	0.019	
51	2401-BM										
53	2401-BM		0.005		0.005				0.005		
58	2401-BM		0.0033	0.0055	0.0074	0.0136	0.009	0.0038	0.0044	0.0107	0.0373
59	2401-BM		0.000151	0.0000248	0.0000283	0.000369	0.000301	0.000833	0.000378	0.000131	0.00137
60	2401-BM		0.0006								
65	2401-BM		0.00181	0.00181	0.00181	0.00363	0.00363	0.00544	0.00544	0.00907	0.182
67	2401-BM		0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
70	2401-BM		0.000358	0.000358	0.000358	0.000358	0.000358	0.000358	0.000358	0.000358	
79	2401-BM		0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.01
80	2401-BM		0.00037								
81	2401-BM		0.001	0.02	0.001	0.02	0.01	0.008	0.005	0.01	0.06
83	2401-BM										
85	2401-BM		0.0013	0.0014	0.0009	0.0011	0.0013	0.0009	0.0011	0.001	0.0221
93	2401-BM		0.002	0.002	0.002	0.002	0.002	0.002	0.002	0.002	0.08
95	2401-BM		0.00007	0.000291		0.000282	0.00042	0.000306	0.000284	0.00051	0.00594
99	2401-BM		0.00360								
106	2401-BM		0.002		0.007		0.014		0.012		0.6
114	2401-BM		0.0005								
115	2401-BM										
124	2401-BM										
127	2401-BM		0.01	0.01	0.01	0.01	0.02	0.02	0.02	0.02	1
003B	2401-BM		0.00173	0.00173	0.00173	0.00345	0.00345	0.00518	0.00518	0.00864	0.17271
34A	2401-BM										

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Bovine Meat (2401-BM)

Methods PBDEs - Measurement Uncertainty [%]

LC	Sample	BDE 28	BDE 47	BDE 49	BDE 99	BDE 100	BDE 153	BDE 154	BDE 183	BDE 209
5	2401-BM	30	30	60	30	30	30	30	30	30
9	2401-BM									
10	2401-BM									
21	2401-BM									
26	2401-BM	40	40	40	40	40	40	40	40	
32	2401-BM									
34	2401-BM									
43	2401-BM									
49	2401-BM	30	30	30	30	30	30	30	30	40
51	2401-BM	48	18	25	25	21	23	30	20	42
53	2401-BM	59	59	59	59	59	59	59	59	59
58	2401-BM	28.8	21.3	28	15.3	13	27.6	26.6	22.7	29.3
59	2401-BM	67.9	20.3	20.3	17.5	19.4	20.2	23.8	9.68	31.4
60	2401-BM	50	25	30	25	25	25	25	25	35
65	2401-BM	25	25	25	25	25	25	25	30	40
67	2401-BM	35	30	45	30	30	30	30	30	45
70	2401-BM	10	10	10	10	10	10	10	10	
79	2401-BM	17	18	29	11	19	23	23	22	20
80	2401-BM	30	30		30	30	30	30	30	30
81	2401-BM	28	41	34	34	27	27	28	36	40
83	2401-BM									
85	2401-BM	70	30	70	30	30	30	50	50	50
93	2401-BM	25	25	25	25	25	30	25	25	50
95	2401-BM	15.3	12.9		12	17.2	18.9	16	21.2	16.5
99	2401-BM	30	30	-	30	30	30	30	30	-
106	2401-BM	30	30	30	30	30	30	30	30	30
114	2401-BM	35	25		40	40	40	40	30	50
115	2401-BM									
124	2401-BM									
127	2401-BM	20	20	20	20	20	20	20	20	40
003B	2401-BM	25	25	25	25	25	25	25	30	40
34A	2401-BM									

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Bovine Meat (2401-BM)
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
5	2401-BM			
9	2401-BM			
10	2401-BM	Empirically determined as the lowest calibration point producing signal above S/N 10	Established from quality control charts (2*RSD)	
21	2401-BM			
26	2401-BM			
32	2401-BM			
34	2401-BM			
43	2401-BM	lowest validated concentration	from validation data	
49	2401-BM	calculated on base of standard with S/N 10:1 multiplied with the worst recovery rate	estimated value based on the RSD of multiple determination	Method according to Bichon et.al. 2018
51	2401-BM	ten point equidistant calibration in Matrix at the expected LOQ using DIN 32645		For lipid determination 4-5 g sample were used lipid content was determined with weibull-stold method (hydrolyse with 4M HCL, washing neutral, drying and soxlethextraction with Hexan) HBCDD analysis were done from 2 different samplecontainers. All results were <LOQ
53	2401-BM	Samples spiked at LOQ level (10 pg/g) within the batch	From validation data	
58	2401-BM	LOD and LOQ were estimated based on analysis of 10 blank spiked samples	MU were estimated based on precision and truenes from fortyfication experiment	
59	2401-BM	LOQ = 3 x LOD; LOD evaluated as 3 times of noise		
60	2401-BM	calibration curve, lowest standard solution	QC-samples, PT results	
65	2401-BM			
67	2401-BM			
70	2401-BM			
79	2401-BM			
80	2401-BM			
81	2401-BM	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	
83	2401-BM	Lowest validated level		
85	2401-BM	LOQ = 8 x SD where, SD is the standard deviation of the lowest successfully validated level of HBCDD	U = k x uc where, Coverage factor k =2 is used, corresponding to a level of confidence of ~95% and, uc is combined standard uncertainty, calculated from the combination of the relative uncertainty components describing the random variations and the bias contribution	
93	2401-BM	according to validation.	according to NEN 7777	
95	2401-BM			
99	2401-BM			
106	2401-BM			
114	2401-BM	standarddeviation of the blanks multiplied with a factor of 6	MU was calculated by the root of the sum of the measurement uncertainty for reproducibility and recovery including bias. Expanded uncertainty is then calculated by multiplying MU with a factor of 2	
115	2401-BM	From analysis of the tissue sample LOQ estimated as 10*S/N of calculated concentration	Combined intralaboratory reproducibility and method bias	
124	2401-BM			
127	2401-BM			
003B	2401-BM			
034A	2401-BM			

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Bovine Meat (2401-BM)

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

LC	Sample		(+/–)-α-HBCDD	(+/–)-β- HBCDD	(+/–)-γ- HBCDD	Total HBCDD (using GC-methods)
5	2401-BM					
9	2401-BM					
10	2401-BM		0.01	0.01	0.01	
21	2401-BM		0.01	0.01	0.01	
26	2401-BM					
32	2401-BM					
34	2401-BM			0.001	0.001	
43	2401-BM		0.2	0.02	0.04	
49	2401-BM		0.1	0.1	0.1	
51	2401-BM		0.01	0.01	0.01	
53	2401-BM		0.01	0.01	0.01	
58	2401-BM		0.006	0.005	0.005	
59	2401-BM					
60	2401-BM			0.002	0.002	
65	2401-BM		0.006	0.006	0.006	
67	2401-BM		0.002	0.002	0.002	
70	2401-BM					
79	2401-BM					
80	2401-BM					
81	2401-BM		0.01	0.01	0.01	
83	2401-BM		0.03	0.03	0.03	
85	2401-BM		0.1	0.1	0.1	
93	2401-BM		0.045	0.03	0.092	
95	2401-BM		0.0025	0.0035	0.00175	
99	2401-BM		0.02	0.02	0.02	
106	2401-BM					
114	2401-BM					0.0059
115	2401-BM		0.007	0.006	0.005	
124	2401-BM		0.2	0.05	0.05	
127	2401-BM					
003B	2401-BM		0.00575	0.00575	0.00575	
34A	2401-BM			0.001	0.001	

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Bovine Meat (2401-BM)

Methods HBCDDs - Measurement Uncertainty [%]

LC	Sample		(+/–)-α-HBCDD	(+/–)-β- HBCDD	(+/–)-γ- HBCDD
5	2401-BM				
9	2401-BM				
10	2401-BM		21	21	21
21	2401-BM				
26	2401-BM				
32	2401-BM				
34	2401-BM				
43	2401-BM		50	50	50
49	2401-BM		30	30	30
51	2401-BM				
53	2401-BM		49	49	49
58	2401-BM		39	32	42
59	2401-BM				
60	2401-BM		25	30	30
65	2401-BM		30	50	30
67	2401-BM		20	20	20
70	2401-BM				
79	2401-BM				
80	2401-BM				
81	2401-BM		27	30	32
83	2401-BM		30	30	30
85	2401-BM		30	30	30
93	2401-BM		25	41	50
95	2401-BM		9	17	5
99	2401-BM		23	18	16
106	2401-BM				
114	2401-BM		40	40	40
115	2401-BM		0.19		0.22
124	2401-BM				
127	2401-BM				
003B	2401-BM		30	50	30
34A	2401-BM				