

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in May 2025

EURL-PT-POP_2502-HY

FEED

Final Report PBDEs and HBCDDs (Report Version 1.0)

21 May 2026



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Summary

| | |
|--|--|
| Test sample | FEED: Hay [2502-HY] |
| Analytes of interest Mandatory for NRLs: | PBDEs (BDE-28, -47, -49, -99, -100, -153, -154, -183, -209) HBCDDs (α -HBCDD, β -HBCDD, γ -HBCDD or total HBCDD) |
| Methods | Any kind of method |
| Participants | NRLs, OFLs, other official laboratories, commercial laboratories performing the analysis of samples taken by feed business operators |
| Statistical evaluation | ISO 13528:2022 [1], IUPAC Protocol [2] |
| Report of preliminary results | 21 May 2026 (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 **Hay** was organized by the EURL for halogenated POPs in Feed and Food to be performed between August and November 2025. The objective is to assess analytical performance of laboratories and interlaboratory comparability of results from analyses of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in one sample of **Hay**.

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 2025. NRLs were invited to encourage the participation of Official Laboratories (OFLs) from their member states as part of their duties following Article 101 of regulation (EU) 2017/625 of the European Parliament and of the Council of 15 March 2017. Furthermore, participation of OFLs allowed the extension of the data basis for calculation of assigned values and evaluation of results. **Other official laboratories** and **commercial laboratories** performing the analysis of samples taken by feed business operators were invited to participate in this proficiency test.

The evaluated results were discussed by representatives of European Commission, NRLs and the EURL at the EURL/NRL workshop on 26 November 2025.

1.1. Samples and coding

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

| | |
|------------|-------------------------------|
| Hay | Sample no. 2502-HY-xxx |
|------------|-------------------------------|

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

1.2. Analytes of interest

Participants were requested to determine the following parameters:

Polybrominated diphenyl ethers (PBDEs)

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

Hexabromocyclododecanes (HBCDDs)

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

1.3. Methods

All kinds of detection and quantification methods could be applied.

1.4. Coding of laboratories and confidentiality

The identity of participating laboratories will be kept confidential.

For NRLs of EU member states, the suggested “protocol for management of underperformance in comparative testing or lack of collaboration of National Reference Laboratories (NRLs)” will be followed. The confidentiality of NRLs will be kept according to this protocol.

For OFLs of EU member states cooperating with NRL, the respective NRLs will inform the EURL for halogenated POPs about the participating OFLs and will receive the respective laboratory codes, invoices for participation fee and certificates of participation of the OFLs.

1.5. Results of PBDEs and HBCDDs

Laboratories should:

- use their own reference standards for identification and quantification,
- report results for each analyte,
- report the limit of quantification (LOQ), at least for each non-quantified analyte,
- give method information and
- give information about the accreditation of the laboratory according to ISO/IEC 17025 (*for metrological traceability of consensus values of participants used as assigned values*).

Results had to be reported in **µg/kg, relative to a feed with a moisture content of 12 %**, for PBDEs and HBCDDs.

2. Participating laboratories

This proficiency test was open for participation of:

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

92 laboratories registered for this proficiency test. For PBDE and HBCDD, 26 and 18 sets of results were reported, respectively. Two laboratories laboratory submitted second sets of data for PBDE and one for HBCDD.

Table 1: Participating laboratories

| Participating laboratories | Region | No. all participants | No. participants PBDE/HBCDD |
|--|--------------------------|----------------------|-----------------------------|
| National Reference Laboratories | European Union | 24 | 16/15 |
| | Other Countries | 2 | - |
| Official Laboratories | European Union | 53 | 5/7 |
| | Other European Countries | 1 | - |
| | Africa | - | - |
| | Americas | 2 | - |
| | Asia | - | - |
| | Oceania | - | - |
| Commercial Laboratories | European Union | 9 | 3/2 |
| | Other European Countries | - | - |
| | Africa | - | - |
| | Americas | 1 | - |
| | Asia | - | - |
| | Oceania | - | - |
| | Total | 92 | 24/24 |

2.1. Number of reported results

Table 2: Reported results for PBDEs and moisture content for Hay (2502-HY)

| Reported results (2502-HY) | All laboratories |
|---------------------------------------|------------------|
| BDE-28, 47, 99, 100, 153, 154, 183 | 26 |
| BDE-49, 209 | 25 / 22 |
| Sum of 8 PBDEs (without BDE-209) (ub) | 25 |
| Sum of 8 PBDEs (without BDE-209) (lb) | 25 |
| Sum of 9 PBDEs (with BDE-209) (ub) | 22 |
| Sum of 9 PBDEs (with BDE-209) (lb) | 22 |
| Moisture content | 23 |

Table 3: Reported results for HBCDDs for Hay (2502-HY)

| Reported results (2502-HY) | All laboratories |
|--|------------------|
| α -, β -, γ -HBCDD | 18 |
| Sum of α -, β -, γ -HBCDD (ub) | 18 |
| Sum of α -, β -, γ -HBCDD (lb) | 18 |
| Total HBCDD (using GC methods) | - |
| Moisture content | 17 |

2.2. Accreditation

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

| Hay | PBDEs | HBCDDs |
|------------------|-------|--------|
| Accreditation | 17 | 9 |
| No accreditation | 5 | 7 |



2.3. Detection methods

The following detection methods were applied:

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

Table 5: Overview of chromatographic separation and detection methods for the determination of PBDEs and HBCDDs for Hay (2502-HY)

| Detection methods | PBDEs | HBCDDs |
|-------------------|-------|--------|
| GC-HRMS | 15 | - |
| GC-MS/MS | 9 | 2 |
| GC-NCI-MS | - | - |
| LC-MS/MS | - | 13 |
| LC-HRMS | - | 1 |

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 2502-HY were analyzed in duplicate for PBDEs. The test for sufficient homogeneity was performed for individual congeners and sum parameters. The test materials showed sufficient homogeneity and stability for PBDEs for this proficiency test. Homogeneity and stability can be concluded also for HBCDDs, due to similar physico-chemical properties.

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 $\pm 50\%$ of the median of all reported results) and examination of the distribution of the remaining results using histogram and Kernel density estimation, if necessary.



Assigned values were calculated for individual PBDE congeners, sum of 8 (without BDE-209) and sum of 9 (with BDE-209) PBDEs, for individual HBCDD diastereomers, sum of α -, β - and γ -HBCDD and total HBCDD (including limits of quantification (LOQs)), if possible. Additionally, the median of all values was calculated.

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

Due to high variation of participants' results in the range of the respective LOQ or too few results, no assigned values could be calculated for:

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

Since there are no traceable reference values available, the assigned values in this PT were calculated based on the Huber robust mean of the participants' results. Therefore, the assigned values are only traceable to the results of the participants. Additionally, the results of all participants reporting results and the results of participants having accreditation according to ISO/IEC 17025 were compared for PBDE sum parameters. 21 of 29 participating laboratories were accredited according to ISO/IEC 17025 for PBDEs. No significant differences (0 to 3 %) between the assigned values calculated for both data sets for PBDEs were observed. For HBCDDs only 9 out of 29 participating laboratories were accredited according to ISO/IEC 17025. Therefore, no assigned values for HBCDDs could be calculated for this group of participants (Table 6).

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

| Sum parameters | Assigned value | Assigned value | Deviation |
|---|---|-----------------------------|-----------|
| | All participants | ISO/IEC 17025 accreditation | |
| | $\mu\text{g/kg product}$ (12 % moisture content) | | % |
| Sum of PBDE without BDE-209 (ub) | 0.625 | 0.627 | <1 |
| Sum of PBDE without BDE-209 (lb) | 0.625 | 0.626 | <1 |
| Sum of PBDE including BDE-209 | 8.01 | -* | |
| γ -HBCDD | 0.907 | -* | |
| Sum of α -, β -, γ -HBCDD | 0.988 | -* | |

*too few results from accredited laboratories to calculate an reliable assigned value for comparison

4.1. PBDEs – individual congeners and sum parameter

The assigned values for the test sample 2502-HY 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)

| Hay (2502-HY) | Assigned value µg/kg product (12 % moisture content) |
|---|---|
| BDE-28 | 0.0105 |
| BDE-47 | 0.210 |
| BDE-49 | 0.00664 |
| BDE-99 | 0.241 |
| BDE-100 | 0.0520 |
| BDE-153 | 0.0322 |
| BDE-154 | 0.0220 |
| BDE-183 | 0.0530 |
| BDE-209 | 7.36 |
| Sum of PBDE without BDE-209 ub | 0.625 |
| Sum of PBDE without BDE-209 lb | 0.625 |
| Sum of PBDE including BDE-209 ub | 8.01 |
| Sum of PBDE including BDE-209 lb | 8.01 |

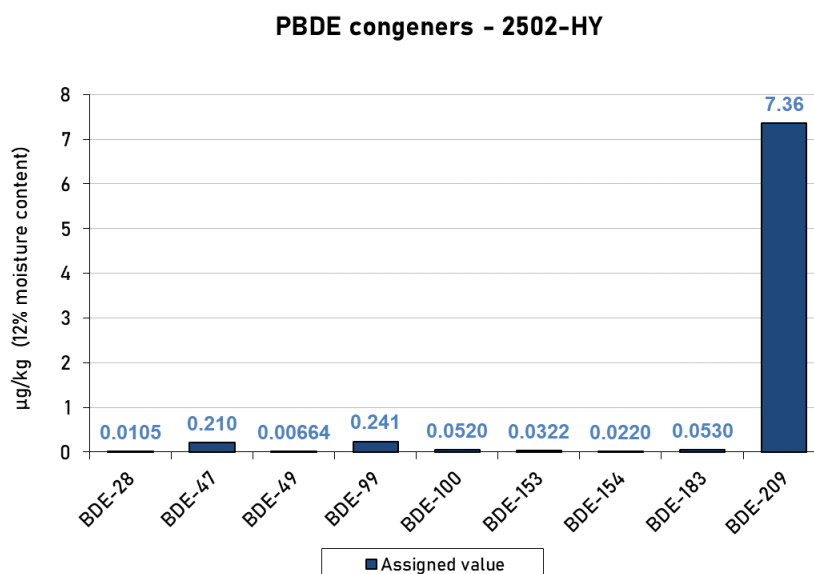


Figure 1: Assigned values (blue) for PBDE individual congeners for Hay (2502-HY) [µg/kg product (12% moisture content)]

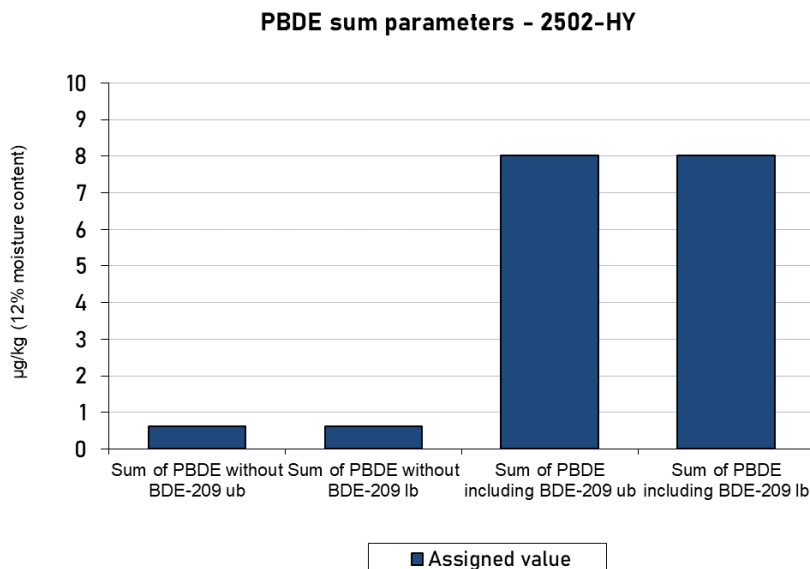


Figure 2: Assigned values (blue) for PBDE sum parameters for Hay (2502-HY) [$\mu\text{g}/\text{kg}$ product (12% moisture content)] y-axis scaled for better overview

4.2. HBCDDs – individual stereoisomers and sum parameter

The assigned values for the test sample 2502-HY were calculated as consensus of participants' results for individual HBCDDs and sum parameters, taking into account the calculation criteria described above (Table 8; tabular summary see annex 7; Figure 3 and 4).

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

| Hay (2502-HY) | Assigned value $\mu\text{g}/\text{kg}$ product (12 % moisture content) |
|--|--|
| (+/-)- α -HBCDD | 0.107 |
| (+/-)- γ -HBCDD | 0.969 |
| Sum of α -, β -, γ -HBCDD (ub) | 1.080 |
| Sum of α -, β -, γ -HBCDD (lb) | 1.07 |

HBCDD diastereomers - 2502-HY

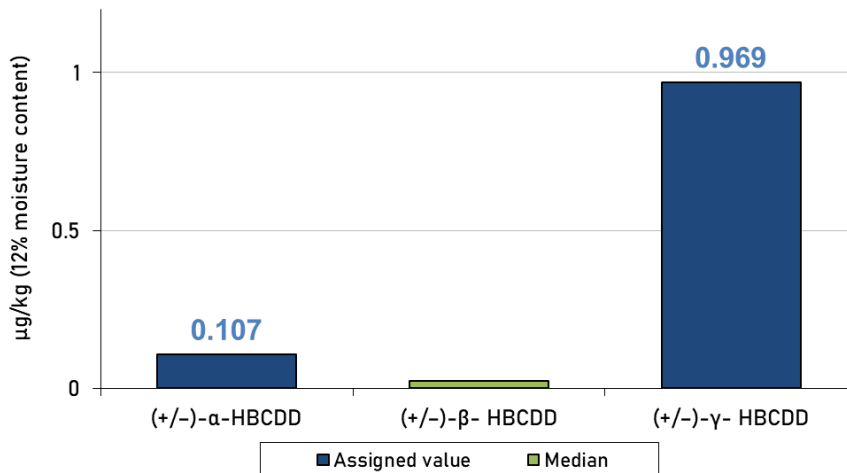


Figure 3: Assigned values (blue) and median values (green) for HBCDD individual congeners for Hay (2502-HY) [µg/kg product (12% moisture content)]

HBCDD sum parameters - 2502-HY

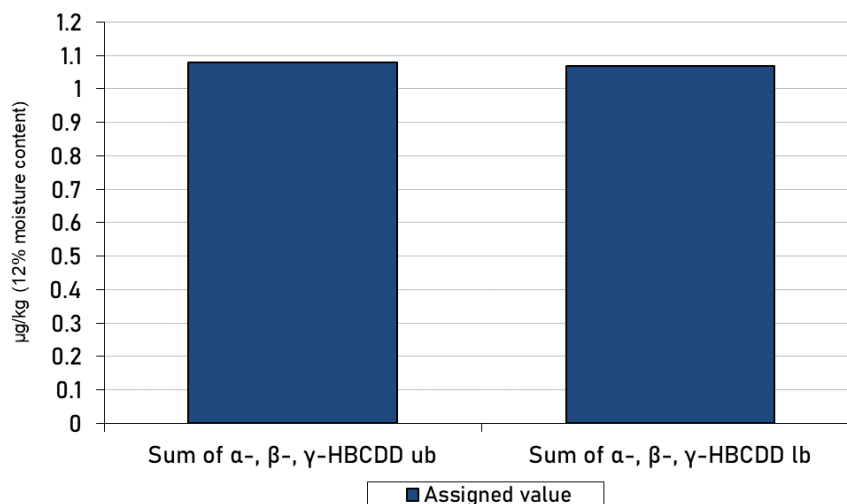


Figure 4: Assigned values (blue) and median values (green) for HBCDD individual congeners and sum parameters for Hay (2502-HY) [µg/kg product (12% moisture content)]

4.3. Moisture content

For the moisture content an assigned value of **8.34 %** for the test sample 2502-HY was calculated as a consensus of the participants' results, taking into account the calculation criteria described above. Participants' results are listed as tabular summary see annex 2, 8 and displayed graphically in figure 5 and 6.

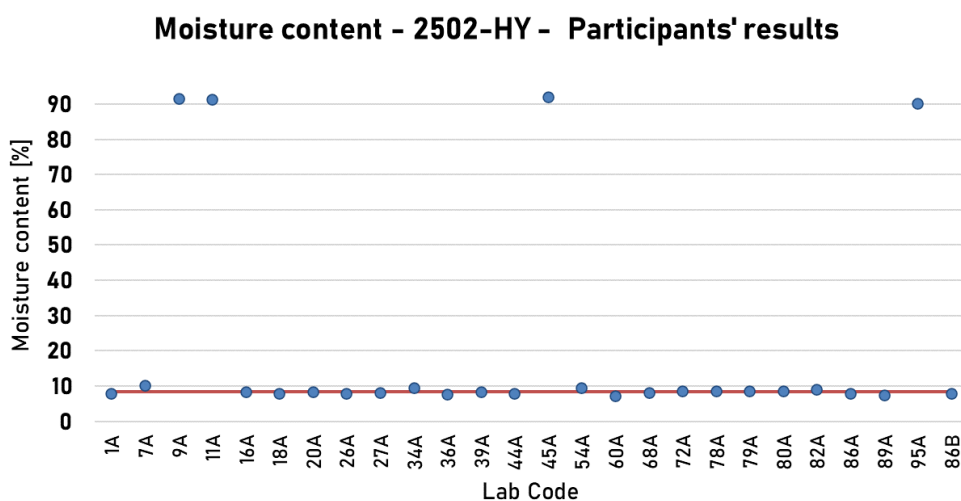


Figure 5: Participant's results of the moisture content in % for Hay (2502-HY)

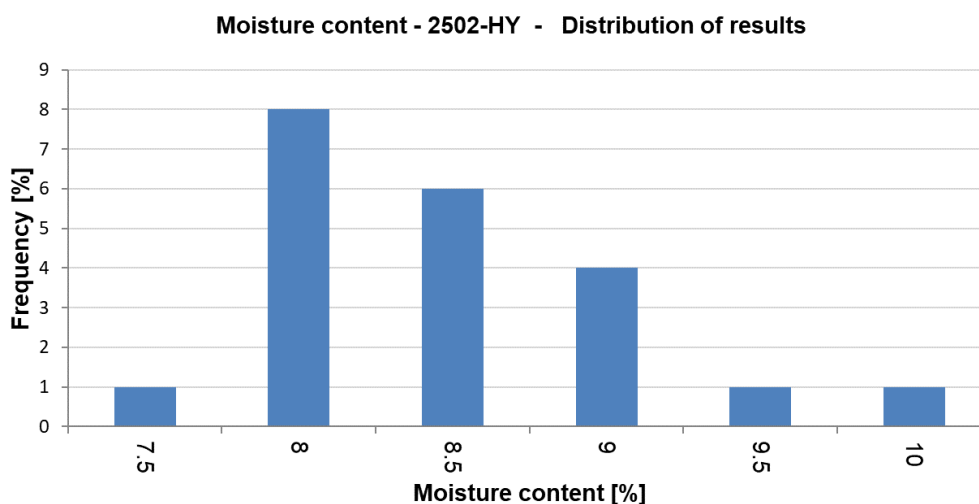


Figure 6: Frequency distribution of the participant's reported moisture contents in % for Hay (2502-HY)



4.4. Comparison of assigned values with recommended LOQs

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

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

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

PBDEs:

Assigned values for BDE-28 (0.0105 µg/kg) and BDE-154 (0.0220 µg/kg) were in the range of the recommended LOQ (Table 9) and could be determined successfully by most of the laboratories. The BDE-49 concentration in this hay sample was below the recommended LOQ of 0.01 µg/kg (12% moisture content) with a calculated assigned value of 0.00664 µg/kg. It is the only BDE congener from this study without its own internal ¹³C-labelled standards, but could be determined by most of the laboratories very successfully in this study.

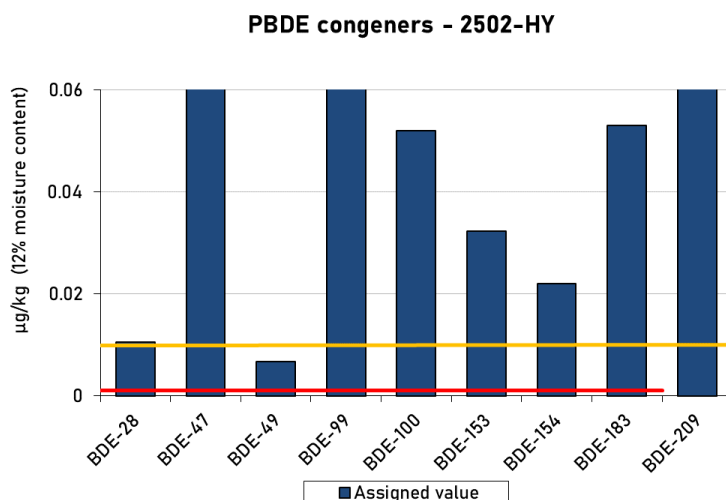


Figure 7: Comparison of assigned values for PBDE congeners with recommended LOQs (yellow line at 0.01 µg/kg product and red line at 0.001 µg/kg product) in Hay (2502-HY)

HBCDDs:

For alpha- and gamma-HBCDD assigned values could be calculated which are 10 to 100-times above the recommended LOQ in this hay sample. For β-HBCDD no assigned value could be calculated because less than two-thirds of all reported results were above the LOQs (see calculation criteria section 4). Therefore, the median value was taken for comparison with the recommended LOQ. The calculated median value for β-HBCDD is 0.0250 µg/kg and in the range of the recommended LOQ of 0.01 µg/kg product, showing that not all participating laboratories could reliably report results for β-HBCDDs in Hay in this low concentration range and reported as results values below their method LOQs.

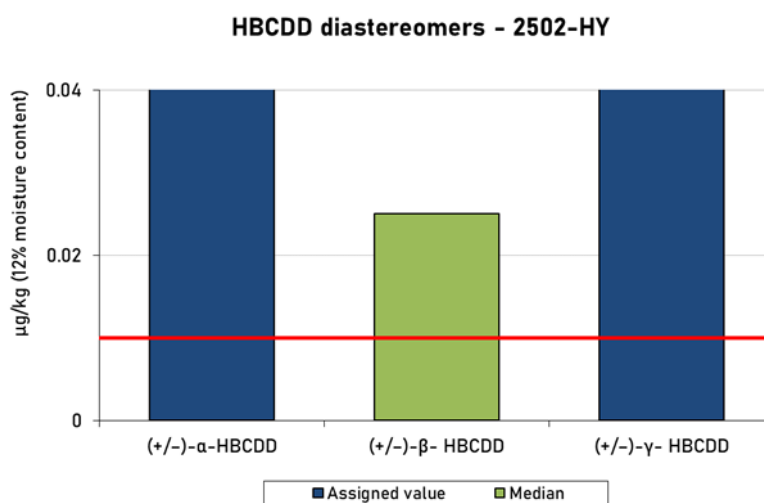


Figure 8: Comparison of assigned values for HBCDD stereoisomers with recommended LOQs (red line at 0.01 µg/kg product) in Hay (2502-HY)

5. Scoring of results – Z-scores

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

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

x : participant's result

x_a : assigned value

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

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

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

Interpretation of z-scores:

| | |
|----------------------------|---|
| $ z\text{-score} \leq 2$ | <i>satisfactory performance</i> |
| $2 < z\text{-score} < 3$ | <i>questionable performance (warning signal)</i> |
| $ z\text{-score} \geq 3$ | <i>unsatisfactory performance (action signal)</i> |

5.1. PBDEs - Participants' z-scores

Table 10: Distribution of participants' z-scores for individual PBDE congeners for Hay (2502-HY)

| Percentage of participants' results | $ z\text{-score} \leq 2$ | $2 < z\text{-score} < 3$ | $ z\text{-score} \geq 3$ |
|-------------------------------------|---------------------------|----------------------------|---------------------------|
| BDE-28 | 96% | - | 4% |
| BDE-47 | 96% | - | 4% |
| BDE-49 | 82% | 9% | 9% |
| BDE-99 | 92% | 8% | - |
| BDE-100 | 92% | 8% | - |
| BDE-153 | 96% | - | 4% |
| BDE-154 | 96% | 4% | - |
| BDE-183 | 96% | - | 4% |
| BDE-209 | 73% | 23% | 5% |

Table 11: Distribution of participants' z-scores for PBDE sum parameters for Hay (2502-HY)

| Percentage of participants' results | $ z\text{-score} \leq 2$ | $2 < z\text{-score} < 3$ | $ z\text{-score} \geq 3$ |
|-------------------------------------|---------------------------|----------------------------|---------------------------|
| Sum of PBDE without BDE-209 ub | 92% | 8% | - |
| Sum of PBDE without BDE-209 lb | 92% | 8% | - |
| Sum of PBDE including BDE-209 ub | 82% | 18% | - |
| Sum of PBDE including BDE-209 lb | 82% | 18% | - |

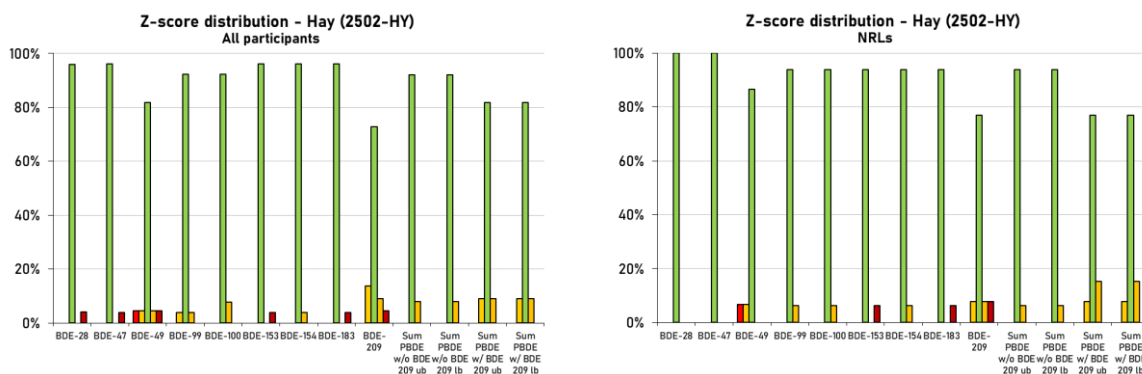


Figure 9: Distribution of participants' z-scores and NRLs only for PBDE congeners / sum parameters for Hay (2502-HY) [Green bars: $-2 \leq z\text{-score} \leq 2$, orange bars: $-3 < z\text{-score} < -2$, $2 < z\text{-score} < 3$, red bars: $z\text{-score} \leq -3$, $z\text{-score} \geq 3$]

5.2. HBCDDs - Participants' z-scores

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

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

Table 12: Distribution of participants' z-scores for HBCDD for Hay (2502-HY)

| Percentage of participants' results | $ z\text{-score} \leq 2$ | $2 < z\text{-score} < 3$ | $ z\text{-score} \geq 3$ |
|--|---------------------------|----------------------------|---------------------------|
| (+/-)- α -HBCDD | 94% | - | 6% |
| (+/-)- γ -HBCDD | 83% | 6% | 11% |
| Sum of α -, β -, γ -HBCDD (ub) | 78% | 6% | 16% |
| Sum of α -, β -, γ -HBCDD (lb) | 78% | 6% | 16% |

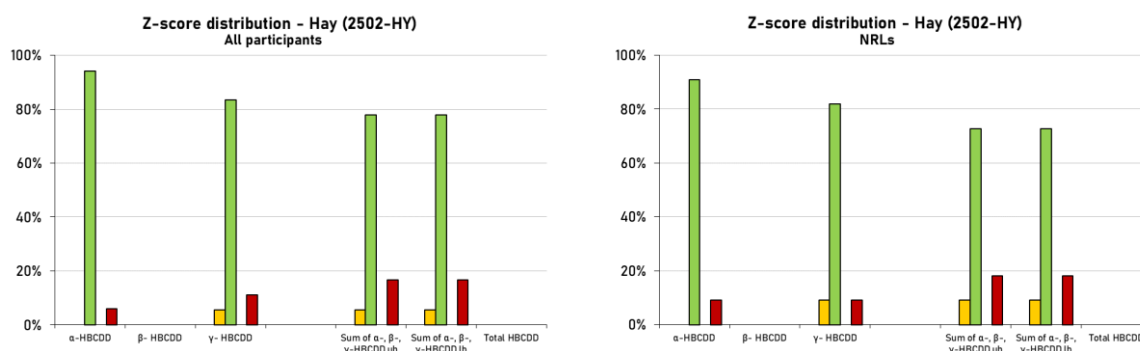


Figure 10: Distribution of participants' z-scores and NRLs only for HBCDD stereoisomers / sum parameters for Hay (2502-HY) [Green bars: $-2 \leq z\text{-score} \leq 2$, orange bars: $-3 < z\text{-score} < -2$, $2 < z\text{-score} < 3$, red bars: $z\text{-score} \leq -3$, $z\text{-score} \geq 3$]

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 sample Hay (2502-HY) are 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.

9. Annex

| Hay – 2502-HY | |
|---------------|---|
| Annex 1 | Assigned values – PBDEs |
| Annex 2 | Participants' results – Tables – PBDEs |
| Annex 3 | Participants' z-scores – Tables – PBDEs |
| Annex 4 | Participants' z-scores – Charts – PBDEs |
| Annex 5 | Test for sufficient homogeneity and stability – PBDEs |
| Annex 6 | Participants' methods – PBDEs |
| Annex 7 | Assigned values – HBCDDs |
| Annex 8 | Participants' results – Tables – HBCDDs |
| Annex 9 | Participants' z-scores – Tables – HBCDDs |
| Annex 10 | Participants' z-scores – Charts – HBCDDs |
| Annex 11 | Participants' methods – HBCDDs |



EURL for halogenated POPs in Feed and Food
c/o State Institute for Chemical and Veterinary Analysis of Food Freiburg

A handwritten signature in black ink, appearing to read 'Alex Schächtele', is written over a white background.

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EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Hay 2025 [EURL-PT-POP_2502-HY]

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

21 May 2026

Annex 1: Assigned values of PBDEs

Test sample - Hay (2502-HY)

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 exclusion of extreme outliers



EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Hay 2025 [EURL-PT-POP_2502-HY]

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

Hay (2502-HY)

PBDE - Assigned values

| Analyte | | Result µg/kg (12% moisture content) | Assigned value [outliers removed] | Robust standard deviation [outliers removed] | Standard uncertainty [outliers removed] | No. of results contributing to assigned value | Median [all values] |
|---------------|--|---|--------------------------------------|--|---|---|------------------------|
| Sum of 8 PBDE | without BDE-209 (ub) | | 0.625 | 0.0787 | 0.021 | 23 | 0.630 |
| Sum of 8 PBDE | without BDE-209 (lb) | | 0.625 | 0.0810 | 0.021 | 23 | 0.630 |
| Sum of 9 PBDE | including BDE-209 (ub) | | 8.01 | 1.89 | 0.57 | 17 | 7.85 |
| Sum of 9 PBDE | including BDE-209 (lb) | | 8.01 | 1.89 | 0.57 | 17 | 7.85 |
| BDE-28 | 2,2',4-tribromodiphenyl ether | | 0.0105 | 0.00144 | 0.00038 | 22 | 0.0108 |
| BDE-47 | 2,2',4,4'-tetrabromodiphenyl ether | | 0.210 | 0.0246 | 0.0064 | 23 | 0.212 |
| BDE-49 | 2,2',4,5'-tetrabromodiphenyl ether | | 0.00664 | 0.00127 | 0.00035 | 20 | 0.00672 |
| BDE-99 | 2,2',4,4',5-pentabromodiphenyl ether | | 0.241 | 0.0250 | 0.0065 | 23 | 0.240 |
| BDE-100 | 2,2',4,4',6-pentabromodiphenyl ether | | 0.0520 | 0.00337 | 0.00090 | 22 | 0.0522 |
| BDE-153 | 2,2',4,4',5,5'-hexabromodiphenyl ether | | 0.0322 | 0.00343 | 0.00089 | 23 | 0.0325 |
| BDE-154 | 2,2',4,4',5,6'-hexabromodiphenyl ether | | 0.0220 | 0.00229 | 0.00060 | 23 | 0.0220 |
| BDE-183 | 2,2',3,4,4',5',6'-heptabromodiphenyl ether | | 0.0528 | 0.00683 | 0.0018 | 23 | 0.0517 |
| BDE-209 | 2,2',3,3',4,4',5,5',6,6'-decabromodiphenyl ether | | 7.36 | 1.87 | 0.57 | 17 | 7.18 |



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Hay (2502-HY)

Moisture content (PBDE) - Assigned value

| Analyte | Result % | Assigned value [outliers removed] | Robust standard deviation [outliers removed] | Standard uncertainty [outliers removed] | No. of results contributing to assigned value | Median [all values] |
|------------------|----------|--------------------------------------|---|--|---|------------------------|
| Moisture content | | 8.34 | 0.629 | 0.17 | 21 | 8.47 |



EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Hay 2025 [EURL-PT-POP_2502-HY]

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Annex 2: Participants' results of PBDEs

Test sample - Hay (2502-HY)

Hay (2502-HY)

PBDE - Results

| LC | Data set | Sample | Result µg/kg 12% moisture content | 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) | 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 |
|----|----------|---------|---|--|--|--|--|--|--|--|---|--|---|---|---|---|
| 1 | | 2502-HY | | | | | | | | | | | | | | |
| 2 | | 2502-HY | | | | | | | | | | | | | | |
| 3 | | 2502-HY | | | | | | | | | | | | | | |
| 4 | | 2502-HY | | | | | | | | | | | | | | |
| 5 | | 2502-HY | | | | | | | | | | | | | | |
| 6 | | 2502-HY | | | | | | | | | | | | | | |
| 7 | A | 2502-HY | | 0.652 | 0.652 | 7.46 | 7.46 | 0.011 | 0.204 | 0.006 | 0.275 | 0.054 | 0.03 | 0.02 | 0.051 | 6.81 |
| 8 | | 2502-HY | | | | | | | | | | | | | | |
| 9 | A | 2502-HY | | 0.622 | 0.622 | 7.42 | 7.42 | 0.0128 | 0.204 | 0.00634 | 0.234 | 0.0545 | 0.0331 | 0.0215 | 0.0557 | 6.8 |
| 10 | | 2502-HY | | | | | | | | | | | | | | |
| 11 | A | 2502-HY | | 0.625 | 0.625 | 8.2 | 8.2 | 0.0116 | 0.192 | 0.00396 | 0.249 | 0.0557 | 0.0308 | 0.0222 | 0.0601 | 7.57 |
| 12 | | 2502-HY | | | | | | | | | | | | | | |
| 13 | | 2502-HY | | | | | | | | | | | | | | |
| 14 | | 2502-HY | | | | | | | | | | | | | | |
| 15 | A | 2502-HY | | 0.416 | 0.416 | 7.49 | 7.49 | 0.01 | 0.209 | 0.007 | 0.239 | 0.052 | 0.035 | 0.023 | 0.051 | 6.87 |
| 16 | A | 2502-HY | | 0.641 | 0.641 | 10.9 | 10.9 | 0.01 | 0.231 | 0.00672 | 0.234 | 0.0519 | 0.033 | 0.0231 | 0.0508 | 10.3 |
| 17 | | 2502-HY | | | | | | | | | | | | | | |
| 18 | A | 2502-HY | | 0.64 | 0.64 | 7.5 | 7.5 | 0.011 | 0.23 | 0.0056 | 0.24 | 0.049 | 0.032 | 0.021 | 0.05 | 6.89 |
| 19 | | 2502-HY | | | | | | | | | | | | | | |
| 20 | | 2502-HY | | | | | | | | | | | | | | |
| 21 | | 2502-HY | | | | | | | | | | | | | | |
| 22 | | 2502-HY | | | | | | | | | | | | | | |
| 23 | | 2502-HY | | | | | | | | | | | | | | |
| 24 | | 2502-HY | | | | | | | | | | | | | | |
| 25 | | 2502-HY | | | | | | | | | | | | | | |
| 26 | A | 2502-HY | | 0.587 | 0.587 | 5.86 | 5.86 | 0.00892 | 0.201 | 0.00631 | 0.222 | 0.05 | 0.0303 | 0.0195 | 0.0486 | 5.27 |
| 27 | A | 2502-HY | | 0.595 | 0.595 | | | 0.00986 | 0.195 | 0.00745 | 0.23 | 0.0512 | 0.0283 | 0.0232 | 0.0499 | |
| 28 | | 2502-HY | | | | | | | | | | | | | | |
| 29 | | 2502-HY | | | | | | | | | | | | | | |
| 30 | | 2502-HY | | | | | | | | | | | | | | |
| 31 | | 2502-HY | | | | | | | | | | | | | | |
| 32 | | 2502-HY | | | | | | | | | | | | | | |
| 33 | | 2502-HY | | | | | | | | | | | | | | |
| 34 | A | 2502-HY | | 0.889 | 0.889 | 8.36 | 8.36 | 0.0125 | 0.293 | 0.00786 | 0.368 | 0.0605 | 0.0431 | 0.0294 | 0.0744 | 7.47 |
| 35 | | 2502-HY | | | | | | | | | | | | | | |
| 36 | | 2502-HY | | | | | | | | | | | | | | |
| 37 | | 2502-HY | | | | | | | | | | | | | | |
| 38 | | 2502-HY | | | | | | | | | | | | | | |
| 39 | A | 2502-HY | | 0.675 | 0.675 | 10 | 10 | 0.0106 | 0.221 | 0.00589 | 0.274 | 0.0524 | 0.0346 | 0.0218 | 0.0542 | 9.37 |
| 40 | | 2502-HY | | | | | | | | | | | | | | |
| 41 | | 2502-HY | | | | | | | | | | | | | | |
| 42 | | 2502-HY | | | | | | | | | | | | | | |
| 43 | | 2502-HY | | | | | | | | | | | | | | |
| 44 | A | 2502-HY | | 0.619 | 0.619 | 8.75 | 8.75 | 0.0105 | 0.218 | 0.00722 | 0.226 | 0.0531 | 0.03 | 0.0211 | 0.0526 | 8.13 |
| 45 | A | 2502-HY | | 0.63 | 0.63 | 7.2 | 7.2 | 0.0097 | 0.205 | 0.0081 | 0.24 | 0.0547 | 0.0321 | 0.0212 | 0.0597 | 6.57 |
| 46 | | 2502-HY | | | | | | | | | | | | | | |
| 47 | | 2502-HY | | | | | | | | | | | | | | |
| 48 | | 2502-HY | | | | | | | | | | | | | | |
| 49 | | 2502-HY | | | | | | | | | | | | | | |
| 50 | | 2502-HY | | | | | | | | | | | | | | |
| 51 | | 2502-HY | | | | | | | | | | | | | | |
| 52 | | 2502-HY | | | | | | | | | | | | | | |
| 53 | | 2502-HY | | | | | | | | | | | | | | |
| 54 | A | 2502-HY | | 0.772 | 0.772 | 12 | 12 | 0.011 | 0.232 | 0.00739 | 0.257 | 0.0536 | 0.0634 | 0.0344 | 0.113 | 11.2 |
| 55 | | 2502-HY | | | | | | | | | | | | | | |
| 56 | | 2502-HY | | | | | | | | | | | | | | |
| 57 | | 2502-HY | | | | | | | | | | | | | | |
| 58 | | 2502-HY | | | | | | | | | | | | | | |
| 59 | | 2502-HY | | | | | | | | | | | | | | |
| 60 | A | 2502-HY | | 0.672 | 0.672 | 9.83 | 9.83 | 0.0109 | 0.227 | 0.00678 | 0.263 | 0.0557 | 0.0343 | 0.0234 | 0.05 | 9.16 |
| 61 | | 2502-HY | | | | | | | | | | | | | | |
| 62 | | 2502-HY | | | | | | | | | | | | | | |
| 63 | | 2502-HY | | | | | | | | | | | | | | |
| 64 | A | 2502-HY | | 0.581 | 0.581 | | | 0.00919 | 0.185 | 0.00348 | 0.218 | 0.0823 | 0.0287 | 0.0192 | 0.0345 | |
| 65 | | 2502-HY | | | | | | | | | | | | | | |
| 66 | | 2502-HY | | | | | | | | | | | | | | |
| 67 | | 2502-HY | | | | | | | | | | | | | | |
| 68 | A | 2502-HY | | 0.55 | 0.54 | 12.7 | 12.7 | 0.0093 | 0.158 | <0.01 | 0.22 | 0.0482 | 0.0314 | 0.0201 | 0.0524 | 12.1 |
| 69 | | 2502-HY | | | | | | | | | | | | | | |

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Hay (2502-HY)

PBDE - Results

| LC | Data set | Sample | Result µg/kg 12% moisture content | 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) | 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 |
|------------------------|----------|---------|---|--|--|--|--|---|---|---|--|---|--|--|--|--|
| 70 | | 2502-HY | | | | | | | | | | | | | | |
| 71 | | 2502-HY | | | | | | | | | | | | | | |
| 72 | A | 2502-HY | | | | | | 0.0109 | 0.235 | | 0.282 | 0.0559 | 0.0343 | 0.0244 | 0.0583 | |
| 73 | | 2502-HY | | | | | | | | | | | | | | |
| 74 | | 2502-HY | | | | | | | | | | | | | | |
| 75 | | 2502-HY | | | | | | | | | | | | | | |
| 76 | | 2502-HY | | | | | | | | | | | | | | |
| 77 | | 2502-HY | | | | | | | | | | | | | | |
| 78 | A | 2502-HY | | 0.645 | 0.645 | 9.7 | 9.7 | 0.0104 | 0.216 | 0.0061 | 0.248 | 0.052 | 0.0328 | 0.0226 | 0.0577 | 9.05 |
| 79 | A | 2502-HY | | 0.476 | 0.476 | 3.52 | 3.52 | 0.0078 | 0.157 | 0.0057 | 0.188 | 0.039 | 0.0239 | 0.0168 | 0.0378 | 3.05 |
| 80 | A | 2502-HY | | 0.69 | 0.69 | 7.5 | 7.5 | 0.012 | 0.24 | 0.0085 | 0.26 | 0.051 | 0.04 | 0.026 | 0.051 | 6.8 |
| 81 | | 2502-HY | | | | | | | | | | | | | | |
| 82 | A | 2502-HY | | 0.57 | 0.57 | | | 0.0085 | 0.199 | 0.0015 | 0.217 | 0.0481 | 0.0312 | 0.0198 | 0.0487 | |
| 83 | | 2502-HY | | | | | | | | | | | | | | |
| 84 | | 2502-HY | | | | | | | | | | | | | | |
| 85 | | 2502-HY | | | | | | | | | | | | | | |
| 86 | A | 2502-HY | | 0.409 | 0.369 | 4.68 | 4.64 | <0.02 | 0.124 | <0.02 | 0.128 | 0.036 | 0.025 | 0.023 | 0.034 | 4.27 |
| 87 | | 2502-HY | | | | | | | | | | | | | | |
| 88 | | 2502-HY | | | | | | | | | | | | | | |
| 89 | A | 2502-HY | | 0.907 | 0.907 | 4.75 | 4.75 | 0.0261 | 0.38 | 0.0157 | 0.266 | 0.0811 | 0.0373 | 0.0273 | 0.0738 | 3.84 |
| 90 | | 2502-HY | | | | | | | | | | | | | | |
| 91 | | 2502-HY | | | | | | | | | | | | | | |
| 92 | | 2502-HY | | | | | | | | | | | | | | |
| 93 | | 2502-HY | | | | | | | | | | | | | | |
| 94 | | 2502-HY | | | | | | | | | | | | | | |
| 95 | A | 2502-HY | | 0.637 | 0.637 | 9.67 | 9.67 | 0.0127 | 0.214 | 0.00584 | 0.24 | 0.0509 | 0.0331 | 0.0215 | 0.0587 | 9.03 |
| 96 | | 2502-HY | | | | | | | | | | | | | | |
| 97 | | 2502-HY | | | | | | | | | | | | | | |
| Additional Sets | | | | | | | | | | | | | | | | |
| 16 | B | 2502-HY | | 0.666 | 0.666 | 11.1 | 11.1 | 0.013 | 0.229 | 0.00986 | 0.246 | 0.0561 | 0.0334 | 0.0229 | 0.0554 | 10.4 |
| 86 | B | 2502-HY | | 0.588 | 0.548 | 7.34 | 7.3 | <0.02 | 0.173 | <0.02 | 0.201 | 0.054 | 0.036 | 0.029 | 0.055 | 6.75 |

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Hay (2502-HY)
 Moisture content (PBDE) - Results

| LC | Data set | Sample | Result % | Moisture content PBDE |
|----|----------|---------|----------|-----------------------|
| 1 | | 2502-HY | | |
| 2 | | 2502-HY | | |
| 3 | | 2502-HY | | |
| 4 | | 2502-HY | | |
| 5 | | 2502-HY | | |
| 6 | | 2502-HY | | |
| 7 | A | 2502-HY | | 10.2 |
| 8 | | 2502-HY | | |
| 9 | A | 2502-HY | | 91.6 |
| 10 | | 2502-HY | | |
| 11 | A | 2502-HY | | 91.3 |
| 12 | | 2502-HY | | |
| 13 | | 2502-HY | | |
| 14 | | 2502-HY | | |
| 15 | A | 2502-HY | | |
| 16 | A | 2502-HY | | 8.28 |
| 17 | | 2502-HY | | |
| 18 | A | 2502-HY | | 7.98 |
| 19 | | 2502-HY | | |
| 20 | | 2502-HY | | |
| 21 | | 2502-HY | | |
| 22 | | 2502-HY | | |
| 23 | | 2502-HY | | |
| 24 | | 2502-HY | | |
| 25 | | 2502-HY | | |
| 26 | A | 2502-HY | | 7.9 |
| 27 | A | 2502-HY | | 8.21 |
| 28 | | 2502-HY | | |
| 29 | | 2502-HY | | |
| 30 | | 2502-HY | | |
| 31 | | 2502-HY | | |
| 32 | | 2502-HY | | |
| 33 | | 2502-HY | | |
| 34 | A | 2502-HY | | 9.6 |
| 35 | | 2502-HY | | |
| 36 | | 2502-HY | | |
| 37 | | 2502-HY | | |
| 38 | | 2502-HY | | |
| 39 | A | 2502-HY | | 8.41 |
| 40 | | 2502-HY | | |
| 41 | | 2502-HY | | |
| 42 | | 2502-HY | | |
| 43 | | 2502-HY | | |
| 44 | A | 2502-HY | | 7.67 |
| 45 | A | 2502-HY | | 92 |
| 46 | | 2502-HY | | |
| 47 | | 2502-HY | | |
| 48 | | 2502-HY | | |
| 49 | | 2502-HY | | |
| 50 | | 2502-HY | | |
| 51 | | 2502-HY | | |
| 52 | | 2502-HY | | |
| 53 | | 2502-HY | | |
| 54 | A | 2502-HY | | 9.5 |
| 55 | | 2502-HY | | |
| 56 | | 2502-HY | | |
| 57 | | 2502-HY | | |

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Hay (2502-HY)
 Moisture content (PBDE) - Results

| LC | Data set | Sample | Result % | Moisture content PBDE |
|------------------------|----------|---------|----------|-----------------------|
| 58 | | 2502-HY | | |
| 59 | | 2502-HY | | |
| 60 | A | 2502-HY | | 7.34 |
| 61 | | 2502-HY | | |
| 62 | | 2502-HY | | |
| 63 | | 2502-HY | | |
| 64 | A | 2502-HY | | |
| 65 | | 2502-HY | | |
| 66 | | 2502-HY | | |
| 67 | | 2502-HY | | |
| 68 | A | 2502-HY | | 8.25 |
| 69 | | 2502-HY | | |
| 70 | | 2502-HY | | |
| 71 | | 2502-HY | | |
| 72 | A | 2502-HY | | 8.6 |
| 73 | | 2502-HY | | |
| 74 | | 2502-HY | | |
| 75 | | 2502-HY | | |
| 76 | | 2502-HY | | |
| 77 | | 2502-HY | | |
| 78 | A | 2502-HY | | 8.65 |
| 79 | A | 2502-HY | | 8.5 |
| 80 | A | 2502-HY | | 8.61 |
| 81 | | 2502-HY | | |
| 82 | A | 2502-HY | | 9 |
| 83 | | 2502-HY | | |
| 84 | | 2502-HY | | |
| 85 | | 2502-HY | | |
| 86 | A | 2502-HY | | 8 |
| 87 | | 2502-HY | | |
| 88 | | 2502-HY | | |
| 89 | A | 2502-HY | | 7.52 |
| 90 | | 2502-HY | | |
| 91 | | 2502-HY | | |
| 92 | | 2502-HY | | |
| 93 | | 2502-HY | | |
| 94 | | 2502-HY | | |
| 95 | A | 2502-HY | | 90.2 |
| 96 | | 2502-HY | | |
| 97 | | 2502-HY | | |
| Additional Sets | | | | |
| 16 | B | 2502-HY | | 8.28 |
| 86 | B | 2502-HY | | 8 |



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

Test sample - Hay (2502-HY)

Z-scores of sum parameters and individual results

Calculation of z-score on basis of assigned value

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

x_a : assigned value

x : participant's result

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

20%: Evaluated individual PBDE congeners and sum parameters

Hay (2502-HY)
 PBDE- Z-scores

| LC | Data set | Sample | Z-score [σ _p = 20 %] | 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) | 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 |
|----|----------|---------|------------------------------------|--|--|--|--|------------------------------------|--|--|--|---|--|---|---|---|---|
| 1 | | 2502-HY | | | | | | | | | | | | | | | |
| 2 | | 2502-HY | | | | | | | | | | | | | | | |
| 3 | | 2502-HY | | | | | | | | | | | | | | | |
| 4 | | 2502-HY | | | | | | | | | | | | | | | |
| 5 | | 2502-HY | | | | | | | | | | | | | | | |
| 6 | | 2502-HY | | | | | | | | | | | | | | | |
| 7 | A | 2502-HY | | 0.2 | 0.2 | -0.3 | -0.3 | | 0.2 | -0.1 | -0.5 | 0.7 | 0.2 | -0.3 | -0.5 | -0.2 | -0.4 |
| 8 | | 2502-HY | | | | | | | | | | | | | | | |
| 9 | A | 2502-HY | | 0.0 | 0.0 | -0.4 | -0.4 | | 1.1 | -0.1 | -0.2 | -0.1 | 0.2 | 0.1 | -0.1 | 0.3 | -0.4 |
| 10 | | 2502-HY | | | | | | | | | | | | | | | |
| 11 | A | 2502-HY | | 0.0 | 0.0 | 0.1 | 0.1 | | 0.5 | -0.4 | -2.0 | 0.2 | 0.4 | -0.2 | 0.0 | 0.7 | 0.1 |
| 12 | | 2502-HY | | | | | | | | | | | | | | | |
| 13 | | 2502-HY | | | | | | | | | | | | | | | |
| 14 | | 2502-HY | | | | | | | | | | | | | | | |
| 15 | A | 2502-HY | | -1.7 | -1.7 | -0.3 | -0.3 | | -0.2 | 0.0 | 0.3 | 0.0 | 0.0 | 0.4 | 0.2 | -0.2 | -0.3 |
| 16 | A | 2502-HY | | 0.1 | 0.1 | 1.8 | 1.8 | | -0.2 | 0.5 | 0.1 | -0.1 | 0.0 | 0.1 | 0.3 | -0.2 | 2.0 |
| 17 | | 2502-HY | | | | | | | | | | | | | | | |
| 18 | A | 2502-HY | | 0.1 | 0.1 | -0.3 | -0.3 | | 0.2 | 0.5 | -0.8 | 0.0 | -0.3 | 0.0 | -0.2 | -0.3 | -0.3 |
| 19 | | 2502-HY | | | | | | | | | | | | | | | |
| 20 | | 2502-HY | | | | | | | | | | | | | | | |
| 21 | | 2502-HY | | | | | | | | | | | | | | | |
| 22 | | 2502-HY | | | | | | | | | | | | | | | |
| 23 | | 2502-HY | | | | | | | | | | | | | | | |
| 24 | | 2502-HY | | | | | | | | | | | | | | | |
| 25 | | 2502-HY | | | | | | | | | | | | | | | |
| 26 | A | 2502-HY | | -0.3 | -0.3 | -1.3 | -1.3 | | -0.8 | -0.2 | -0.2 | -0.4 | -0.2 | -0.3 | -0.6 | -0.4 | -1.4 |
| 27 | A | 2502-HY | | -0.2 | -0.2 | | | | -0.3 | -0.4 | 0.6 | -0.2 | -0.1 | -0.6 | 0.3 | -0.3 | |
| 28 | | 2502-HY | | | | | | | | | | | | | | | |
| 29 | | 2502-HY | | | | | | | | | | | | | | | |
| 30 | | 2502-HY | | | | | | | | | | | | | | | |
| 31 | | 2502-HY | | | | | | | | | | | | | | | |
| 32 | | 2502-HY | | | | | | | | | | | | | | | |
| 33 | | 2502-HY | | | | | | | | | | | | | | | |
| 34 | A | 2502-HY | | 2.1 | 2.1 | 0.2 | 0.2 | | 1.0 | 2.0 | 0.9 | 2.6 | 0.8 | 1.7 | 1.7 | 2.0 | 0.1 |
| 35 | | 2502-HY | | | | | | | | | | | | | | | |
| 36 | | 2502-HY | | | | | | | | | | | | | | | |
| 37 | | 2502-HY | | | | | | | | | | | | | | | |
| 38 | | 2502-HY | | | | | | | | | | | | | | | |
| 39 | A | 2502-HY | | 0.4 | 0.4 | 1.2 | 1.2 | | 0.0 | 0.3 | -0.6 | 0.7 | 0.0 | 0.4 | 0.0 | 0.1 | 1.4 |
| 40 | | 2502-HY | | | | | | | | | | | | | | | |
| 41 | | 2502-HY | | | | | | | | | | | | | | | |
| 42 | | 2502-HY | | | | | | | | | | | | | | | |
| 43 | | 2502-HY | | | | | | | | | | | | | | | |
| 44 | A | 2502-HY | | 0.0 | 0.0 | 0.5 | 0.5 | | 0.0 | 0.2 | 0.4 | -0.3 | 0.1 | -0.3 | -0.2 | 0.0 | 0.5 |
| 45 | A | 2502-HY | | 0.0 | 0.0 | -0.5 | -0.5 | | -0.4 | -0.1 | 1.1 | 0.0 | 0.3 | 0.0 | -0.2 | 0.7 | -0.5 |
| 46 | | 2502-HY | | | | | | | | | | | | | | | |
| 47 | | 2502-HY | | | | | | | | | | | | | | | |
| 48 | | 2502-HY | | | | | | | | | | | | | | | |
| 49 | | 2502-HY | | | | | | | | | | | | | | | |
| 50 | | 2502-HY | | | | | | | | | | | | | | | |
| 51 | | 2502-HY | | | | | | | | | | | | | | | |
| 52 | | 2502-HY | | | | | | | | | | | | | | | |
| 53 | | 2502-HY | | | | | | | | | | | | | | | |
| 54 | A | 2502-HY | | 1.2 | 1.2 | 2.5 | 2.5 | | 0.2 | 0.5 | 0.6 | 0.3 | 0.2 | 4.8 | 2.8 | 5.7 | 2.6 |
| 55 | | 2502-HY | | | | | | | | | | | | | | | |
| 56 | | 2502-HY | | | | | | | | | | | | | | | |
| 57 | | 2502-HY | | | | | | | | | | | | | | | |
| 58 | | 2502-HY | | | | | | | | | | | | | | | |
| 59 | | 2502-HY | | | | | | | | | | | | | | | |
| 60 | A | 2502-HY | | 0.4 | 0.4 | 1.1 | 1.1 | | 0.2 | 0.4 | 0.1 | 0.5 | 0.4 | 0.3 | 0.3 | -0.3 | 1.2 |
| 61 | | 2502-HY | | | | | | | | | | | | | | | |
| 62 | | 2502-HY | | | | | | | | | | | | | | | |
| 63 | | 2502-HY | | | | | | | | | | | | | | | |
| 64 | A | 2502-HY | | -0.4 | -0.4 | | | | -0.6 | -0.6 | -2.4 | -0.5 | 2.9 | -0.5 | -0.6 | -1.7 | |
| 65 | | 2502-HY | | | | | | | | | | | | | | | |
| 66 | | 2502-HY | | | | | | | | | | | | | | | |
| 67 | | 2502-HY | | | | | | | | | | | | | | | |
| 68 | A | 2502-HY | | -0.6 | -0.7 | 2.9 | 2.9 | | -0.6 | -1.2 | | -0.4 | -0.4 | -0.1 | -0.4 | 0.0 | 3.2 |
| 69 | | 2502-HY | | | | | | | | | | | | | | | |
| 70 | | 2502-HY | | | | | | | | | | | | | | | |
| 71 | | 2502-HY | | | | | | | | | | | | | | | |
| 72 | A | 2502-HY | | | | | | | 0.2 | 0.6 | | 0.9 | 0.4 | 0.3 | 0.5 | 0.5 | |
| 73 | | 2502-HY | | | | | | | | | | | | | | | |
| 74 | | 2502-HY | | | | | | | | | | | | | | | |

Hay (2502-HY)
 PBDE- Z-scores

| LC | Data set | Sample | Z-score [σ _p = 20 %] | 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) | 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 |
|------------------------|----------|---------|------------------------------------|--|--|--|--|------------------------------------|--|--|--|---|--|---|---|---|---|
| 75 | | 2502-HY | | | | | | | | | | | | | | | |
| 76 | | 2502-HY | | | | | | | | | | | | | | | |
| 77 | | 2502-HY | | | | | | | | | | | | | | | |
| 78 | A | 2502-HY | | 0.2 | 0.2 | 1.1 | 1.1 | | 0.0 | 0.1 | -0.4 | 0.1 | 0.0 | 0.1 | 0.1 | 0.5 | 1.1 |
| 79 | A | 2502-HY | | -1.2 | -1.2 | -2.8 | -2.8 | | -1.3 | -1.3 | -0.7 | -1.1 | -1.3 | -1.3 | -1.2 | -1.4 | -2.9 |
| 80 | A | 2502-HY | | 0.5 | 0.5 | -0.3 | -0.3 | | 0.7 | 0.7 | 1.4 | 0.4 | -0.1 | 1.2 | 0.9 | -0.2 | -0.4 |
| 81 | | 2502-HY | | | | | | | | | | | | | | | |
| 82 | A | 2502-HY | | -0.4 | -0.4 | | | | -1.0 | -0.3 | -3.9 | -0.5 | -0.4 | -0.2 | -0.5 | -0.4 | |
| 83 | | 2502-HY | | | | | | | | | | | | | | | |
| 84 | | 2502-HY | | | | | | | | | | | | | | | |
| 85 | | 2502-HY | | | | | | | | | | | | | | | |
| 86 | A | 2502-HY | | -1.7 | -2.0 | -2.1 | -2.1 | | | -2.0 | | -2.3 | -1.5 | -1.1 | 0.2 | -1.8 | -2.1 |
| 87 | | 2502-HY | | | | | | | | | | | | | | | |
| 88 | | 2502-HY | | | | | | | | | | | | | | | |
| 89 | A | 2502-HY | | 2.3 | 2.3 | -2.0 | -2.0 | | 7.4 | 4.0 | 6.8 | 0.5 | 2.8 | 0.8 | 1.2 | 2.0 | -2.4 |
| 90 | | 2502-HY | | | | | | | | | | | | | | | |
| 91 | | 2502-HY | | | | | | | | | | | | | | | |
| 92 | | 2502-HY | | | | | | | | | | | | | | | |
| 93 | | 2502-HY | | | | | | | | | | | | | | | |
| 94 | | 2502-HY | | | | | | | | | | | | | | | |
| 95 | A | 2502-HY | | 0.1 | 0.1 | 1.0 | 1.0 | | 1.0 | 0.1 | -0.6 | 0.0 | -0.1 | 0.1 | -0.1 | 0.6 | 1.1 |
| 96 | | 2502-HY | | | | | | | | | | | | | | | |
| 97 | | 2502-HY | | | | | | | | | | | | | | | |
| Additional Sets | | | | | | | | | | | | | | | | | |
| 16 | B | 2502-HY | | 0.3 | 0.3 | 1.9 | 1.9 | | 1.2 | 0.5 | 2.4 | 0.1 | 0.4 | 0.2 | 0.2 | 0.2 | 2.1 |
| 86 | B | 2502-HY | | -0.3 | -0.6 | -0.4 | -0.4 | | | -0.9 | | -0.8 | 0.2 | 0.6 | 1.6 | 0.2 | -0.4 |



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Hay (2502-HY)

Moisture content (PBDE) - Z-scores

| LC | Data set | Sample | Z-score [$\sigma_p = 10\%$] | Moisture content PBDE |
|----|----------|---------|----------------------------------|--------------------------|
| 1 | | 2502-HY | | |
| 2 | | 2502-HY | | |
| 3 | | 2502-HY | | |
| 4 | | 2502-HY | | |
| 5 | | 2502-HY | | |
| 6 | | 2502-HY | | |
| 7 | A | 2502-HY | | 2.2 |
| 8 | | 2502-HY | | |
| 9 | A | 2502-HY | | 99.8 |
| 10 | | 2502-HY | | |
| 11 | A | 2502-HY | | 99.5 |
| 12 | | 2502-HY | | |
| 13 | | 2502-HY | | |
| 14 | | 2502-HY | | |
| 15 | A | 2502-HY | | |
| 16 | A | 2502-HY | | -0.1 |
| 17 | | 2502-HY | | |
| 18 | A | 2502-HY | | -0.4 |
| 19 | | 2502-HY | | |
| 20 | | 2502-HY | | |
| 21 | | 2502-HY | | |
| 22 | | 2502-HY | | |
| 23 | | 2502-HY | | |
| 24 | | 2502-HY | | |
| 25 | | 2502-HY | | |
| 26 | A | 2502-HY | | -0.5 |
| 27 | A | 2502-HY | | -0.2 |
| 28 | | 2502-HY | | |
| 29 | | 2502-HY | | |
| 30 | | 2502-HY | | |
| 31 | | 2502-HY | | |
| 32 | | 2502-HY | | |
| 33 | | 2502-HY | | |
| 34 | A | 2502-HY | | 1.5 |
| 35 | | 2502-HY | | |
| 36 | | 2502-HY | | |
| 37 | | 2502-HY | | |
| 38 | | 2502-HY | | |
| 39 | A | 2502-HY | | 0.1 |
| 40 | | 2502-HY | | |
| 41 | | 2502-HY | | |
| 42 | | 2502-HY | | |
| 43 | | 2502-HY | | |
| 44 | A | 2502-HY | | -0.8 |
| 45 | A | 2502-HY | | 100.3 |
| 46 | | 2502-HY | | |
| 47 | | 2502-HY | | |
| 48 | | 2502-HY | | |
| 49 | | 2502-HY | | |
| 50 | | 2502-HY | | |
| 51 | | 2502-HY | | |
| 52 | | 2502-HY | | |
| 53 | | 2502-HY | | |
| 54 | A | 2502-HY | | 1.4 |
| 55 | | 2502-HY | | |
| 56 | | 2502-HY | | |
| 57 | | 2502-HY | | |

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Hay (2502-HY)

Moisture content (PBDE) - Z-scores

| LC | Data set | Sample | Z-score [$\sigma_p = 10\%$] | Moisture content PBDE |
|------------------------|----------|---------|----------------------------------|--------------------------|
| 58 | | 2502-HY | | |
| 59 | | 2502-HY | | |
| 60 | A | 2502-HY | | -1.2 |
| 61 | | 2502-HY | | |
| 62 | | 2502-HY | | |
| 63 | | 2502-HY | | |
| 64 | A | 2502-HY | | |
| 65 | | 2502-HY | | |
| 66 | | 2502-HY | | |
| 67 | | 2502-HY | | |
| 68 | A | 2502-HY | | -0.1 |
| 69 | | 2502-HY | | |
| 70 | | 2502-HY | | |
| 71 | | 2502-HY | | |
| 72 | A | 2502-HY | | 0.3 |
| 73 | | 2502-HY | | |
| 74 | | 2502-HY | | |
| 75 | | 2502-HY | | |
| 76 | | 2502-HY | | |
| 77 | | 2502-HY | | |
| 78 | A | 2502-HY | | 0.4 |
| 79 | A | 2502-HY | | 0.2 |
| 80 | A | 2502-HY | | 0.3 |
| 81 | | 2502-HY | | |
| 82 | A | 2502-HY | | 0.8 |
| 83 | | 2502-HY | | |
| 84 | | 2502-HY | | |
| 85 | | 2502-HY | | |
| 86 | A | 2502-HY | | -0.4 |
| 87 | | 2502-HY | | |
| 88 | | 2502-HY | | |
| 89 | A | 2502-HY | | -1.0 |
| 90 | | 2502-HY | | |
| 91 | | 2502-HY | | |
| 92 | | 2502-HY | | |
| 93 | | 2502-HY | | |
| 94 | | 2502-HY | | |
| 95 | A | 2502-HY | | 98.2 |
| 96 | | 2502-HY | | |
| 97 | | 2502-HY | | |
| Additional Sets | | | | |
| 16 | B | 2502-HY | | -0.1 |
| 86 | B | 2502-HY | | -0.4 |



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

Test sample - Hay (2502-HY)

Z-scores of sum parameters and individual results

Calculation of z-score on basis of assigned value


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


x_a : assigned value

x : participant's result

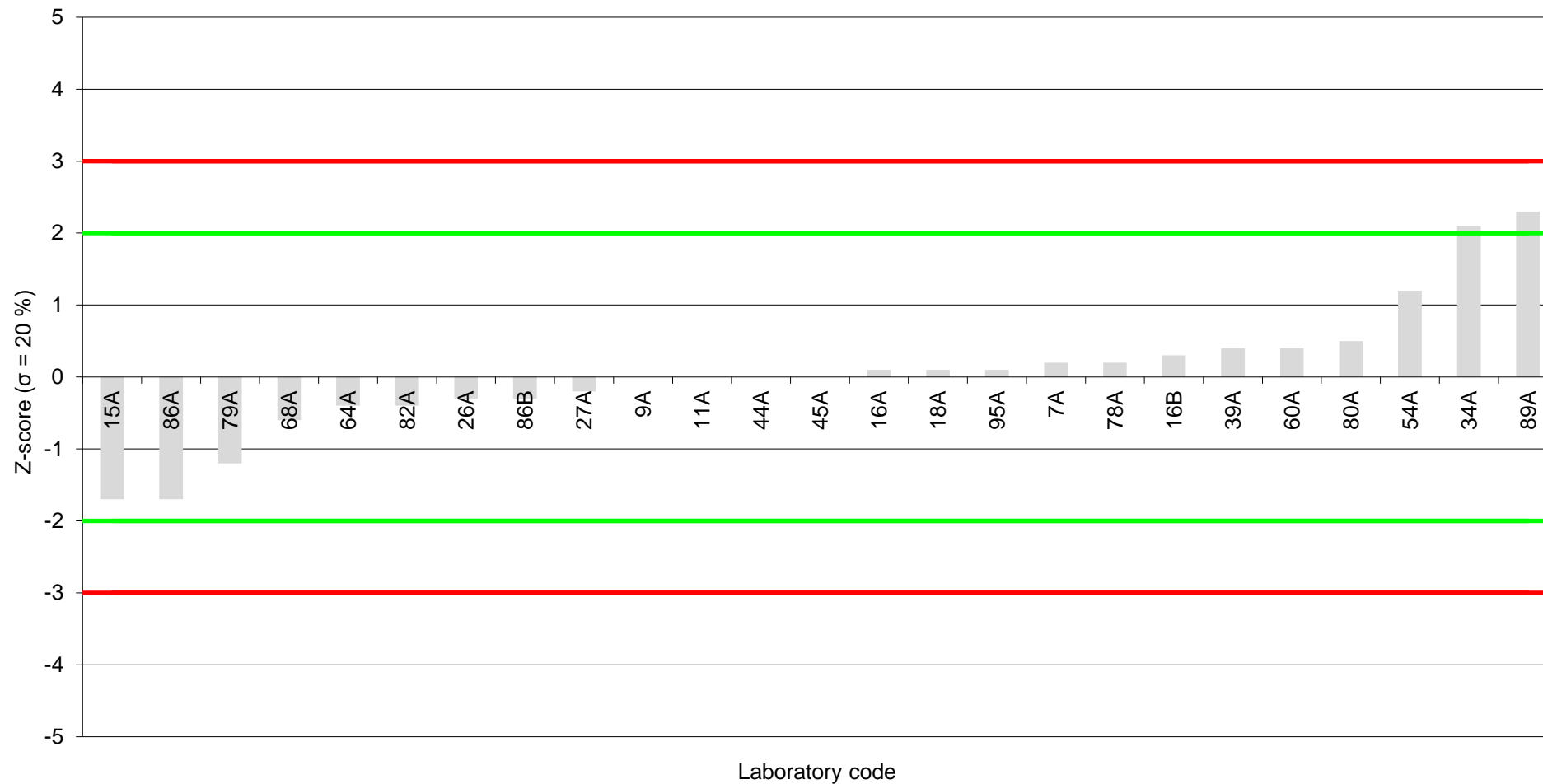
σ_{prel} : fitness-for-purpose-based standard deviation for proficiency assessment

20%: Evaluated individual PBDE congeners and sum parameters

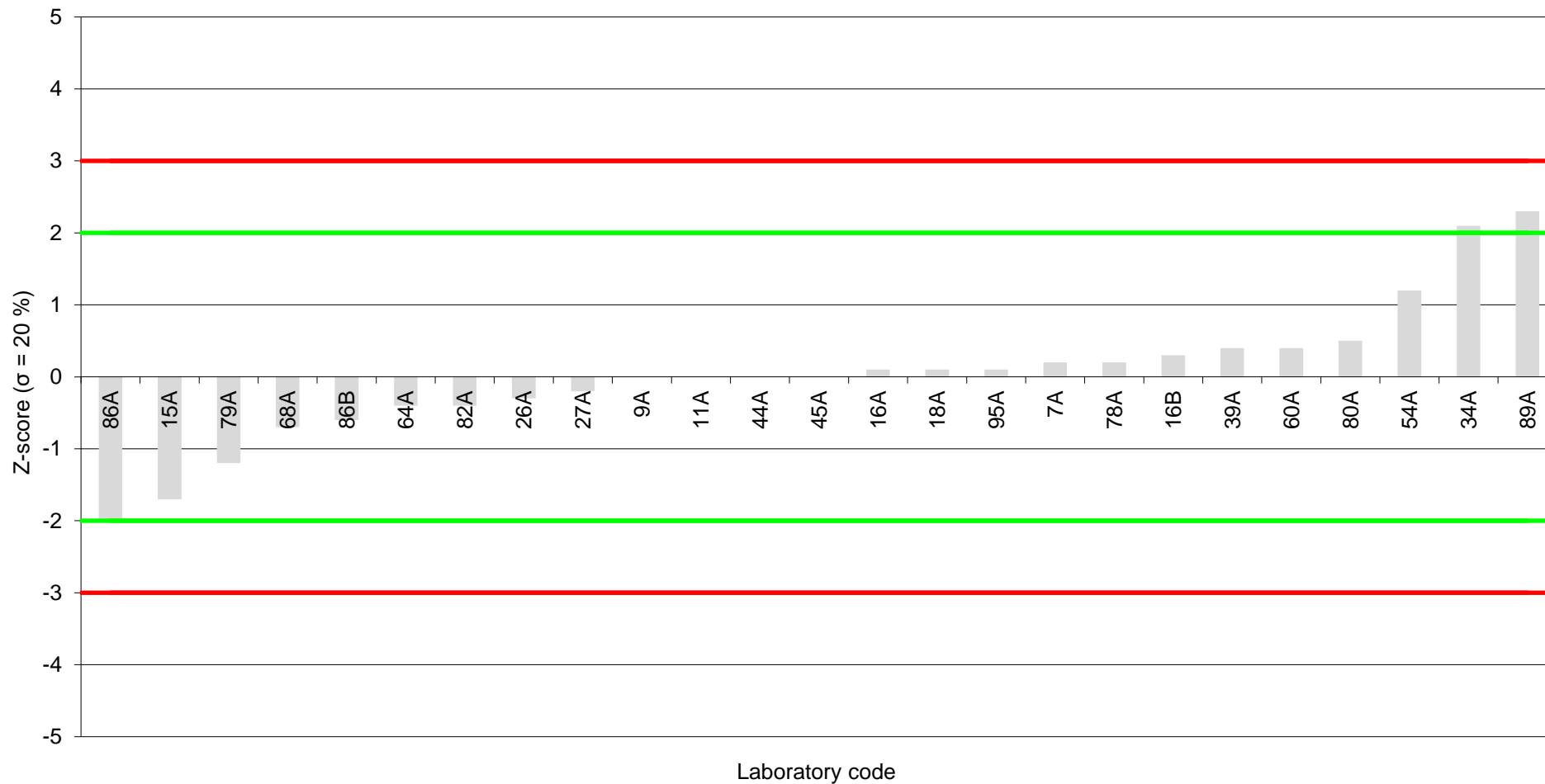
± 2 z-scores: 

± 3 z-scores: 

Hay (2502-HY)
Sum of 8 PBDE without BDE-209 ub
Assigned value: 0.625 µg/kg product (12 % moisture content)



Hay (2502-HY)
Sum of 8 PBDE without BDE-209 lb
Assigned value: 0.625 µg/kg product (12 % moisture content)



Hay (2502-HY)
Sum of 9 PBDE including BDE-209 ub
Assigned value: 8.01 µg/kg product (12 % moisture content)



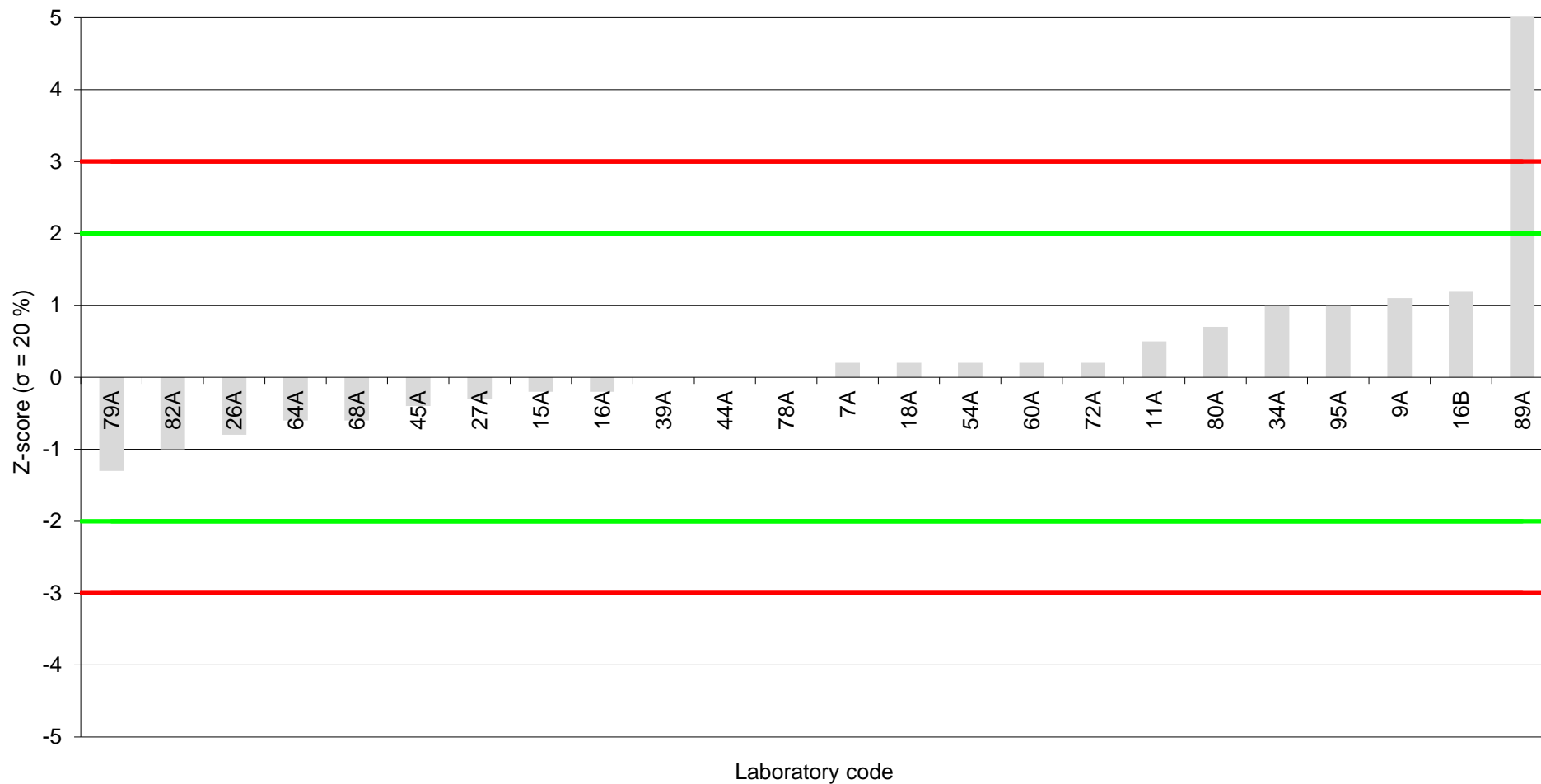
Hay (2502-HY)
Sum of 9 PBDE including BDE-209 lb
Assigned value: 8.01 µg/kg product (12 % moisture content)



Hay (2502-HY)

BDE-28

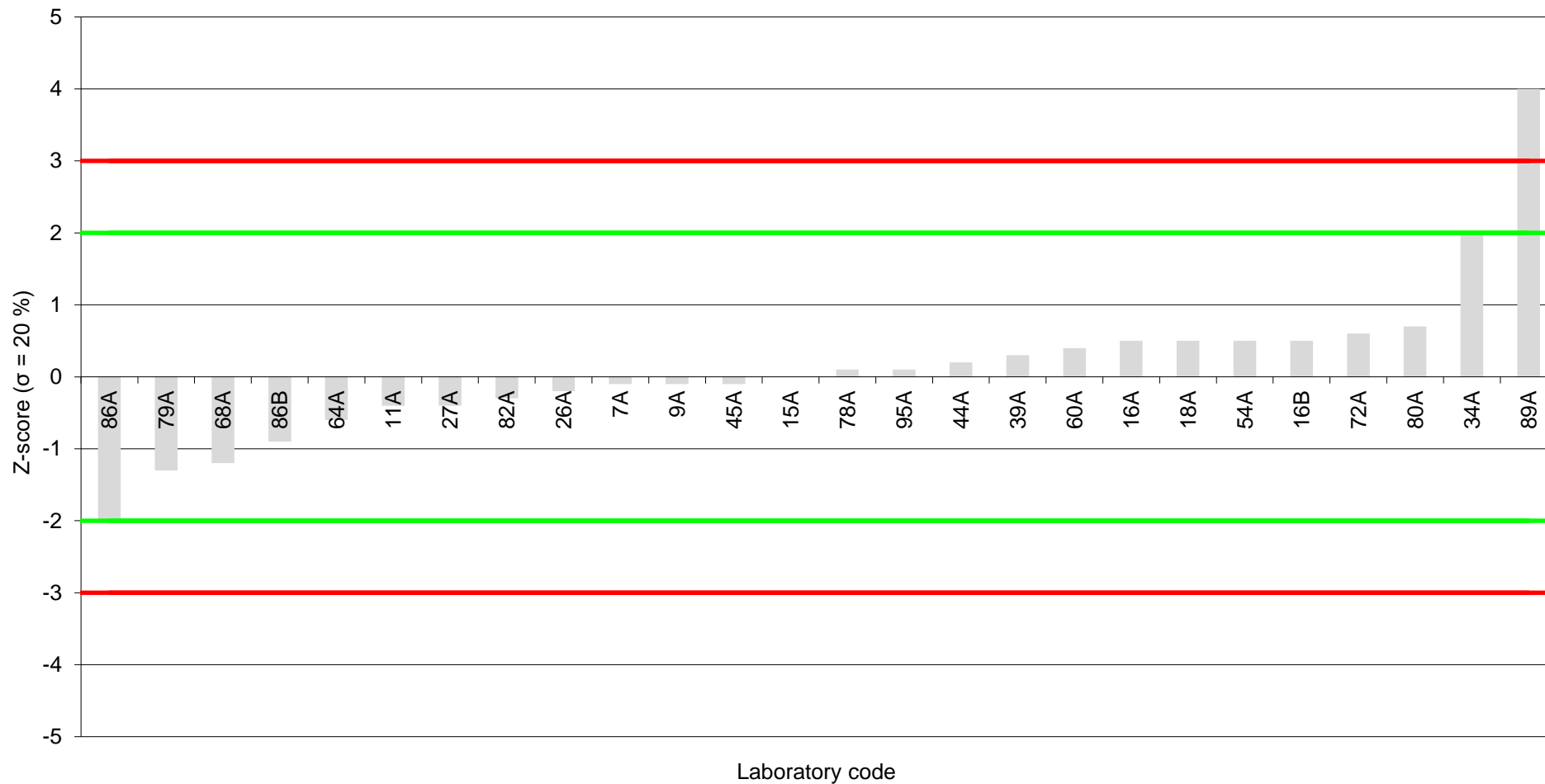
Assigned value: 0.0105 µg/kg product (12 % moisture content)



Hay (2502-HY)

BDE-47

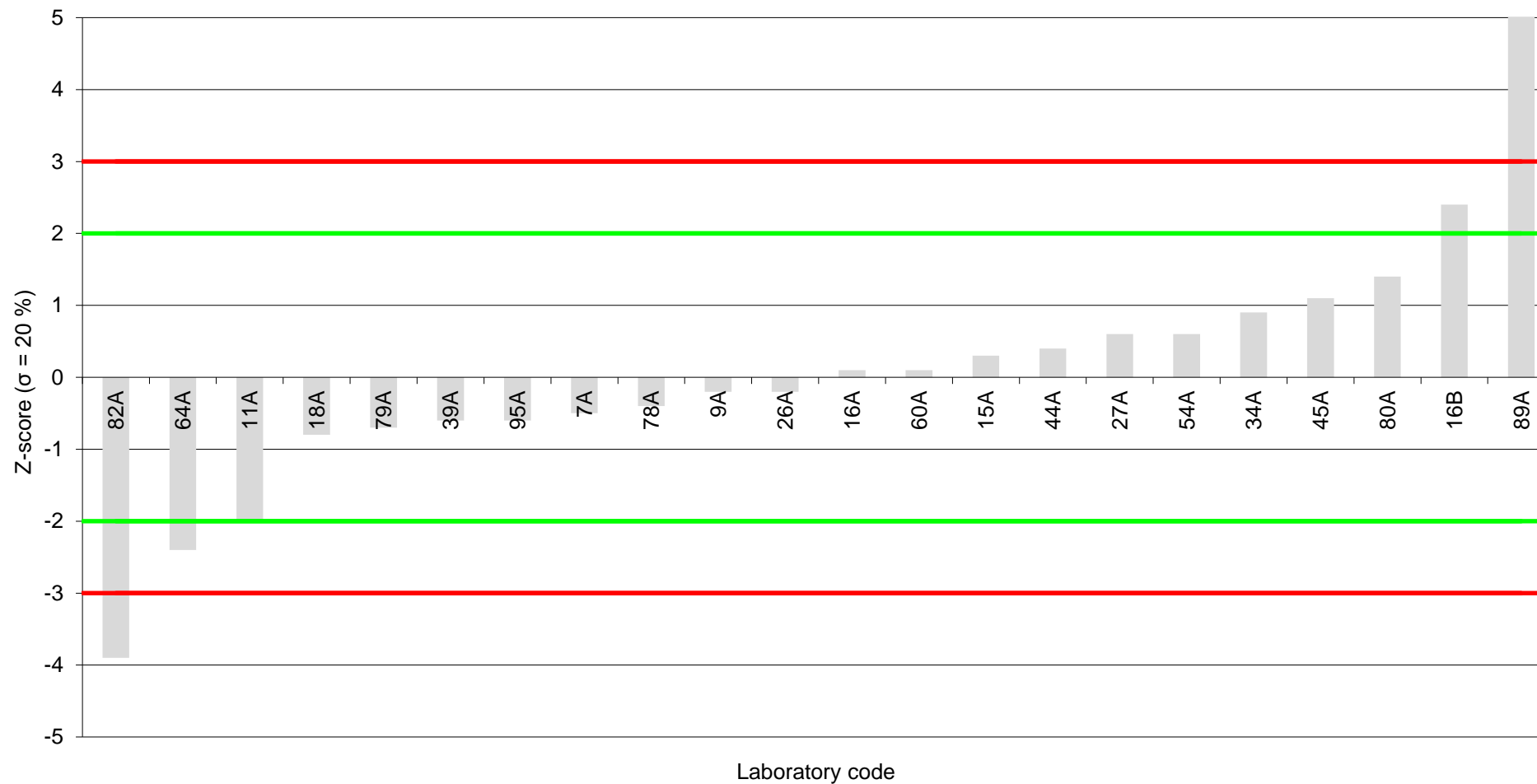
Assigned value: 0.21 µg/kg product (12 % moisture content)



Hay (2502-HY)

BDE-49

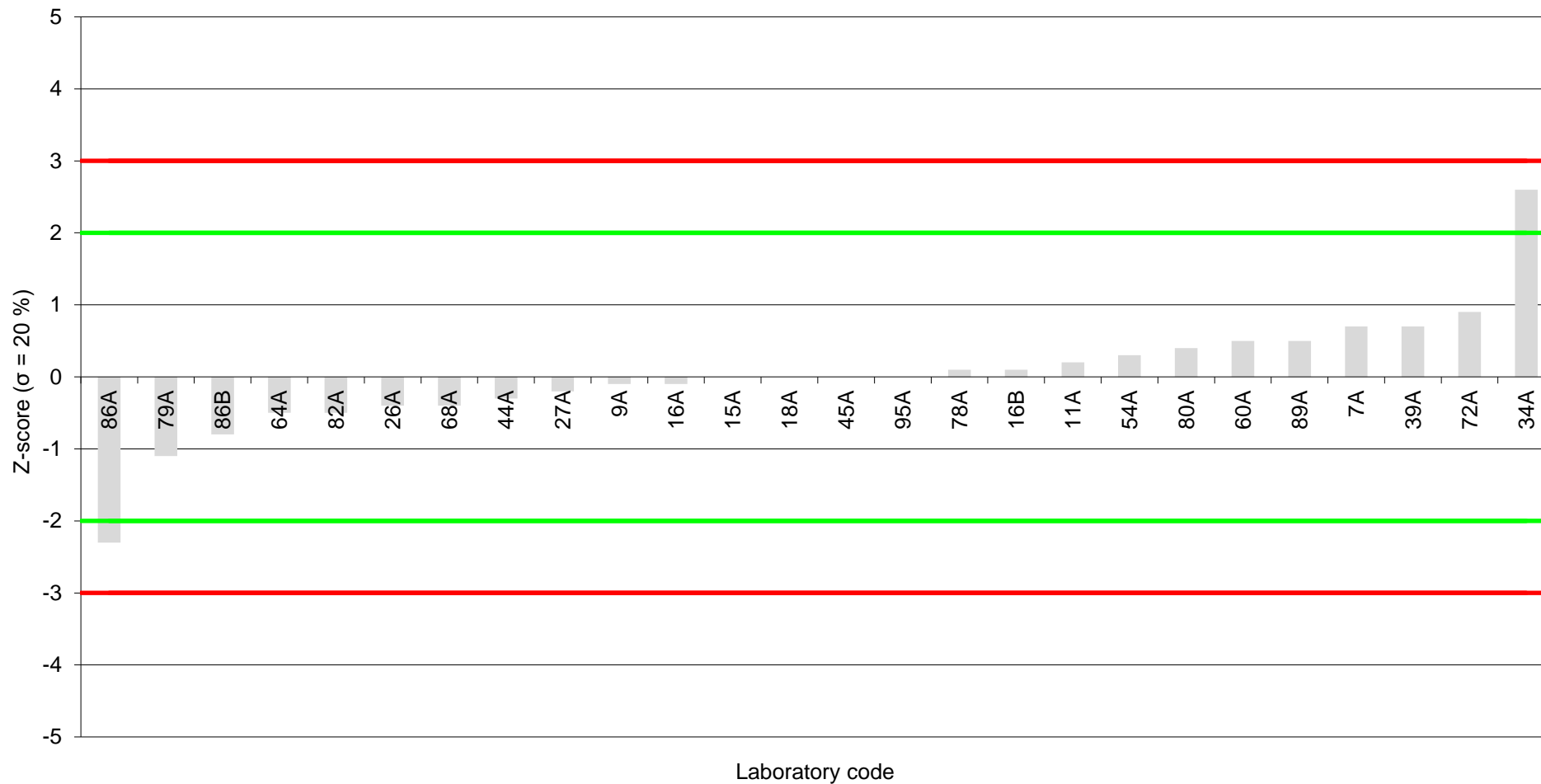
Assigned value: 0.00664 µg/kg product (12 % moisture content)



Hay (2502-HY)

BDE-99

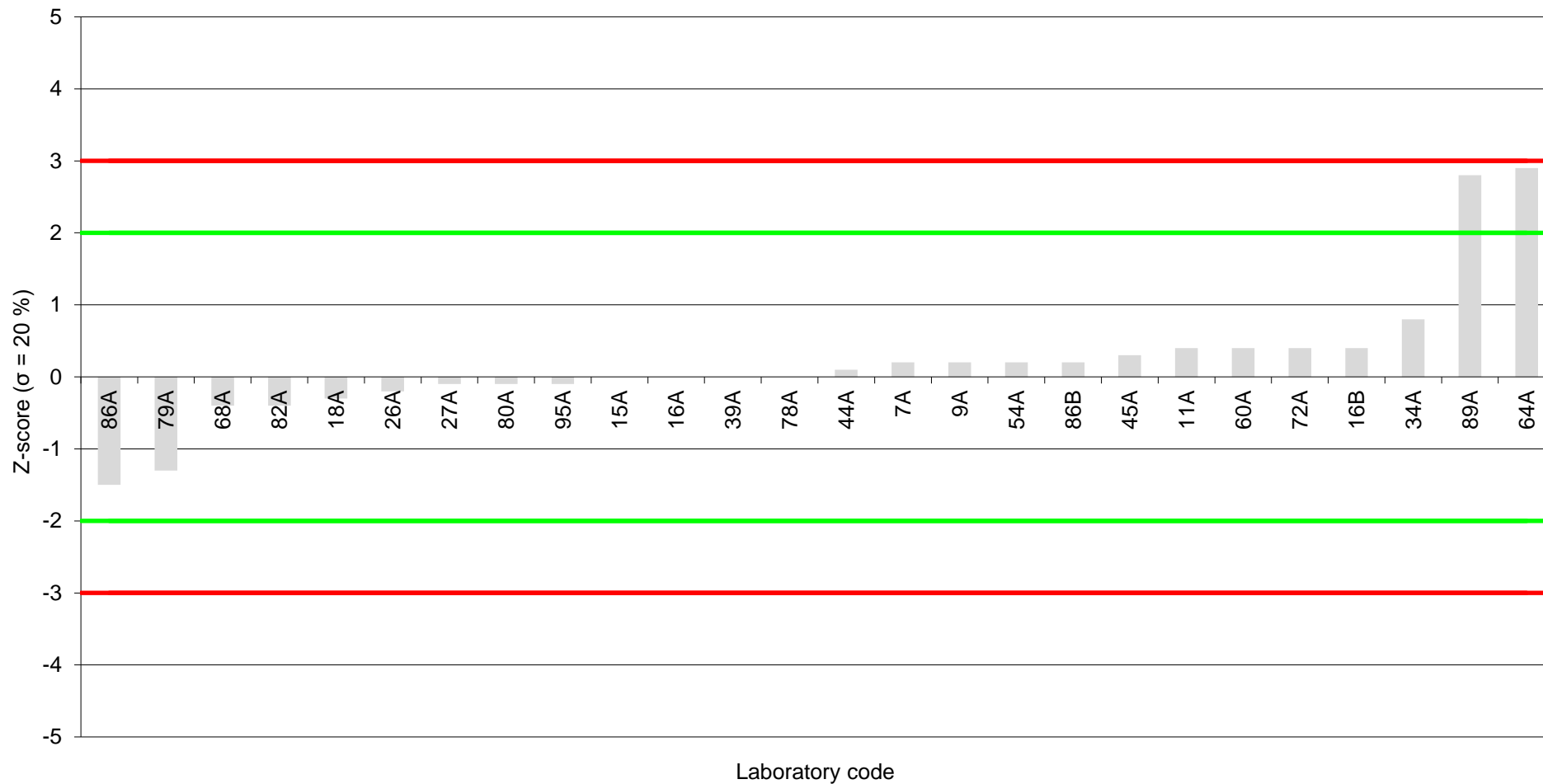
Assigned value: 0.241 µg/kg product (12 % moisture content)



Hay (2502-HY)

BDE-100

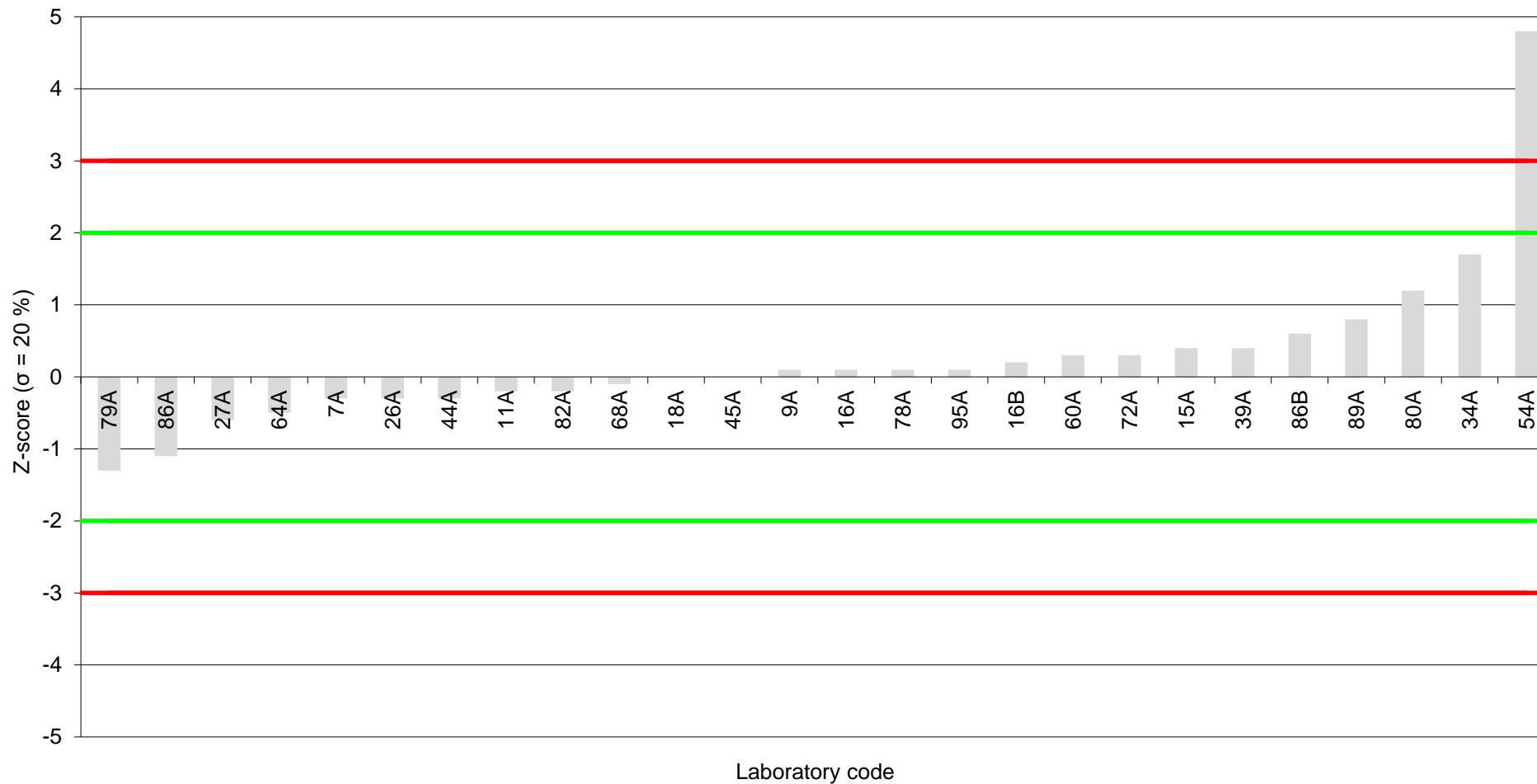
Assigned value: 0.052 µg/kg product (12 % moisture content)



Hay (2502-HY)

BDE-153

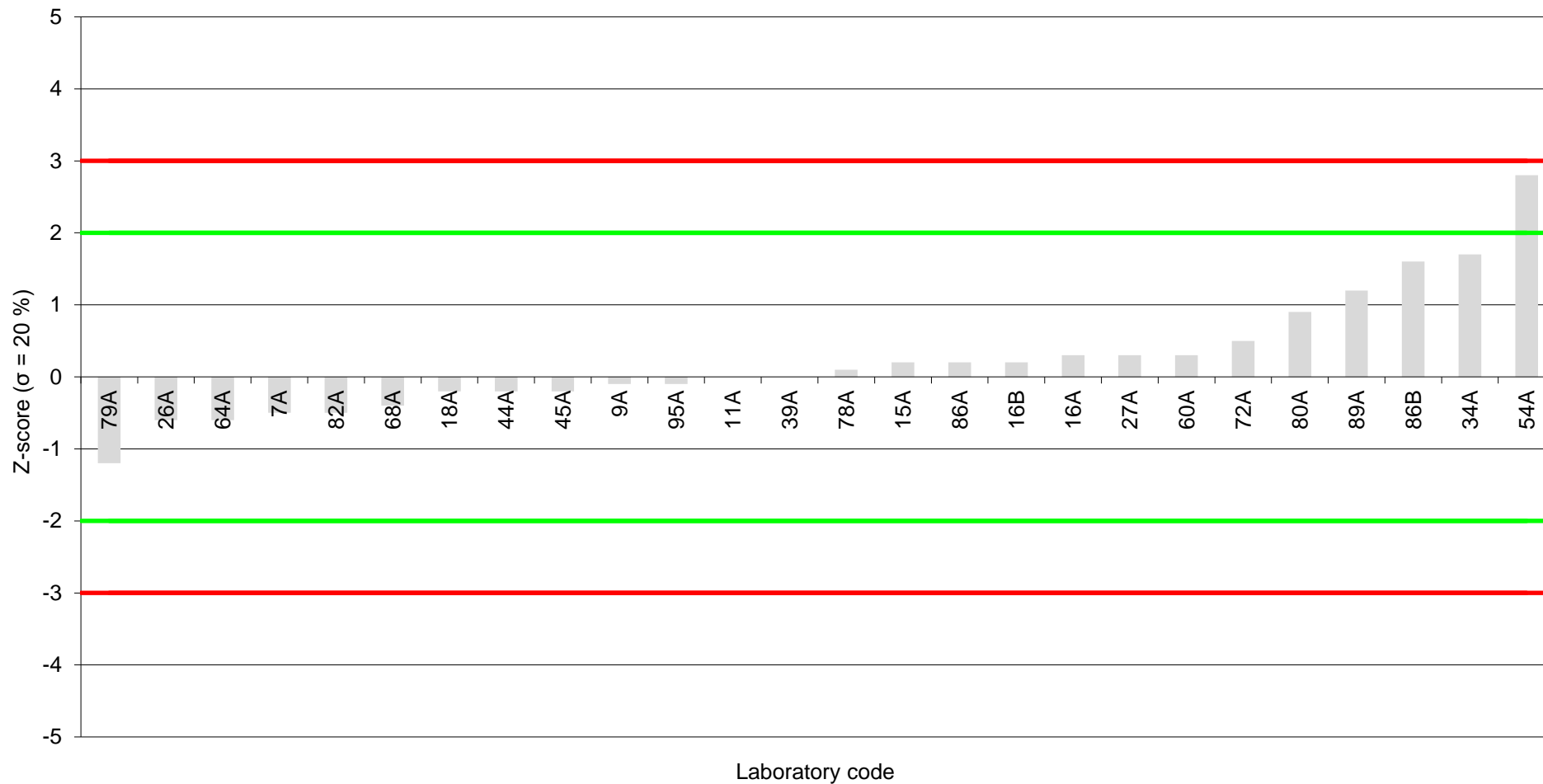
Assigned value: 0.0322 µg/kg product (12 % moisture content)



Hay (2502-HY)

BDE-154

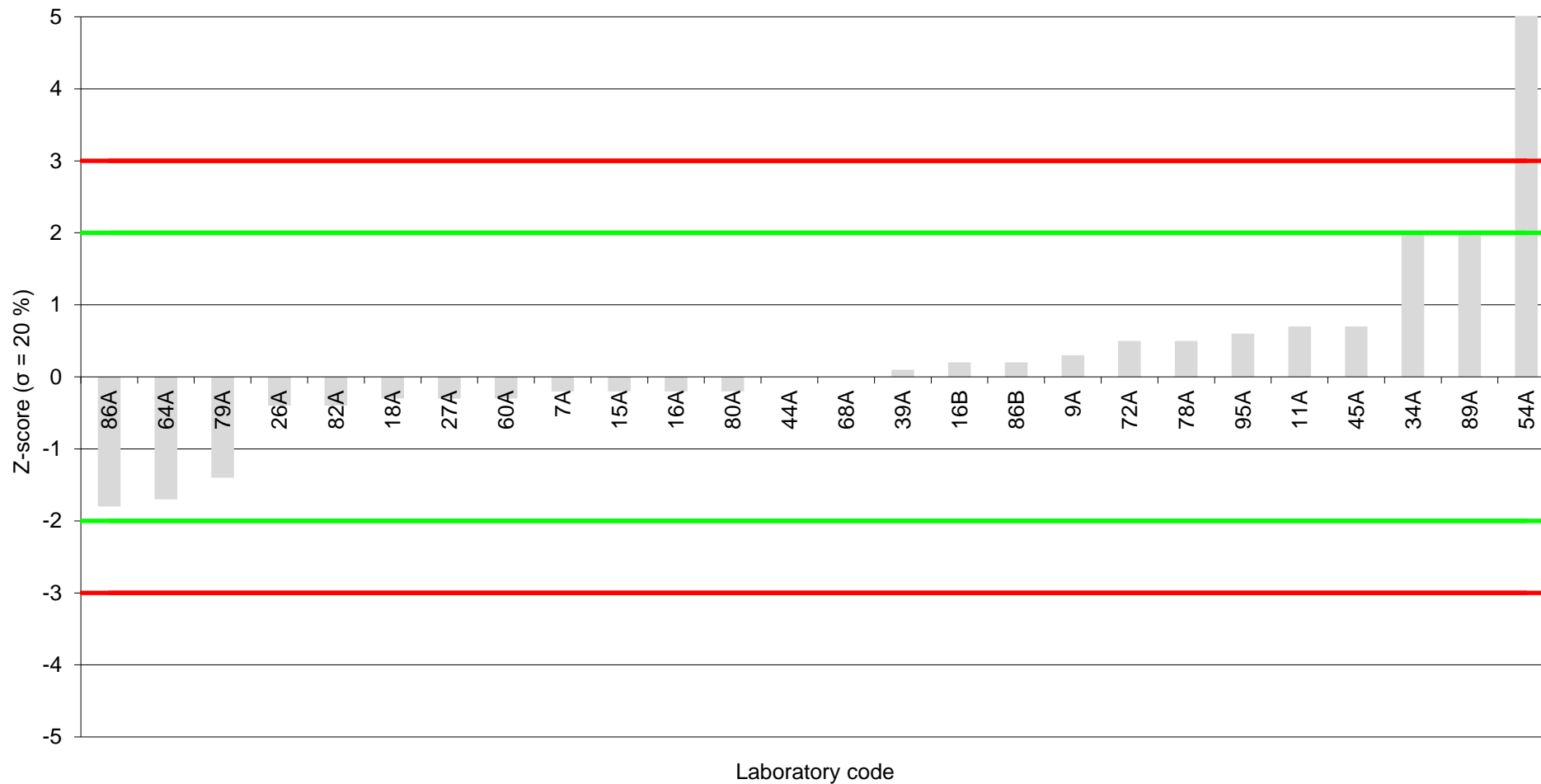
Assigned value: 0.022 µg/kg product (12 % moisture content)



Hay (2502-HY)

BDE-183

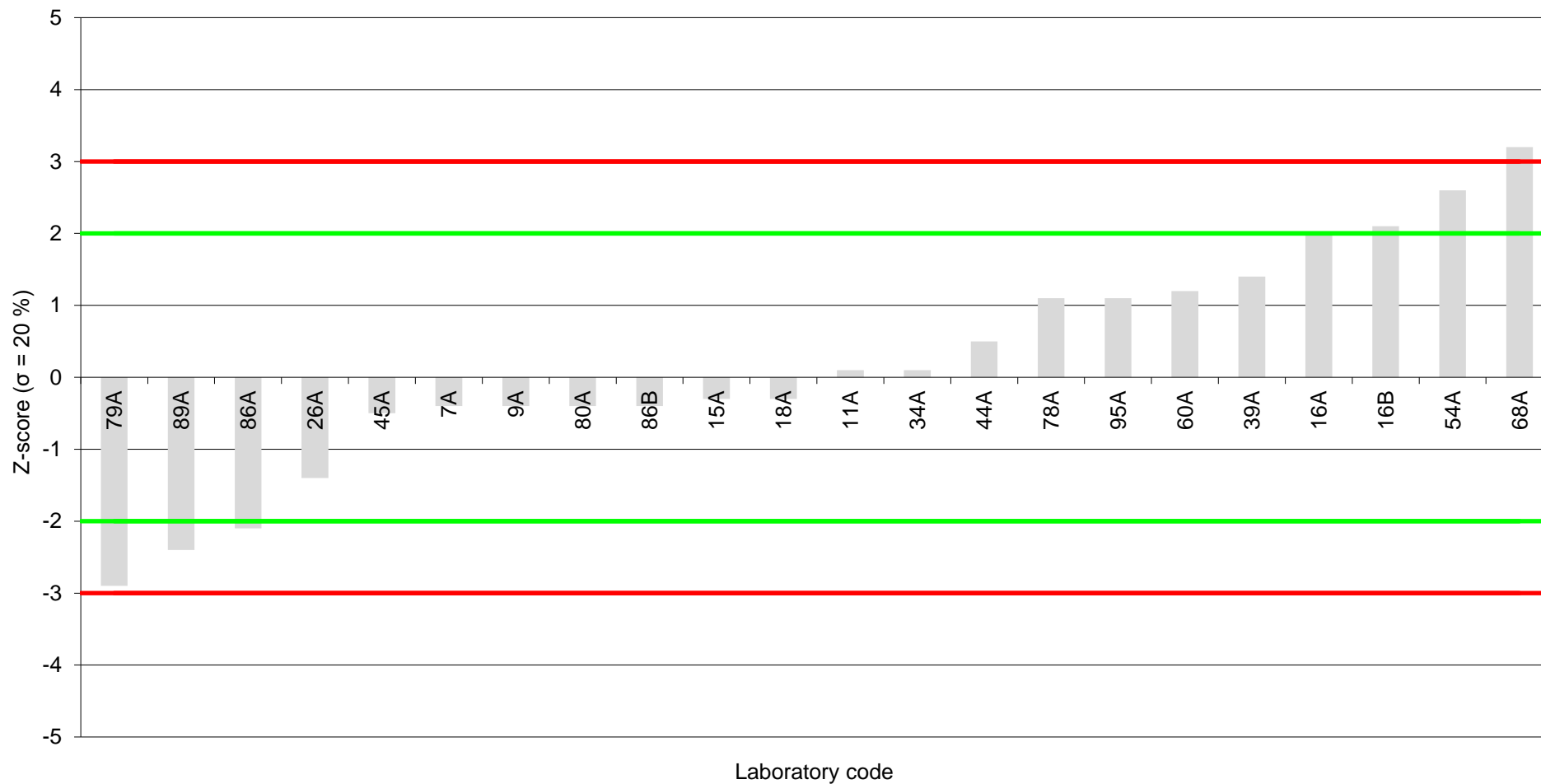
Assigned value: 0.0528 µg/kg product (12 % moisture content)



Hay (2502-HY)

BDE-209

Assigned value: 7.36 µg/kg product (12 % moisture content)





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Annex 5: Test for sufficient homogeneity and stability for PBDEs

Test sample - Hay (2502-HY)



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

Hay (2502-HY)

PBDE - Homogeneity test - Data

| Analyte | Result µg/kg 12% moisture content | Mean (n = 10, duplicate analysis) | Median (n = 10, duplicate analysis) | Relative standard deviation [%] |
|--|---|--------------------------------------|--|------------------------------------|
| Sum of 8 PBDE - without BDE-209 (lb) | | 0.578 | 0.574 | 3% |
| Sum of 8 PBDE - without BDE-209 (ub) | | 0.578 | 0.574 | 3% |
| Sum of 9 PBDE - including BDE-209 (lb) | | 8.07 | 7.92 | 11% |
| Sum of 9 PBDE - including BDE-209 (ub) | | 8.07 | 7.92 | 11% |
| BDE-28 | | 0.00951 | 0.00953 | 1% |
| BDE-47 | | 0.176 | 0.177 | 4% |
| BDE-49 | | 0.0119 | 0.0113 | 10% |
| BDE-99 | | 0.231 | 0.229 | 4% |
| BDE-100 | | 0.0500 | 0.0494 | 3% |
| BDE-153 | | 0.0320 | 0.0317 | 4% |
| BDE-154 | | 0.0217 | 0.0215 | 4% |
| BDE-183 | | 0.0457 | 0.0448 | 9% |
| BDE-209 | | 7.50 | 7.34 | 12% |

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Hay 2025 [EURL-PT-POP_2502-HY]

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

Hay (2502-HY)

Selected PBDE congener - Homogeneity test - Data

| Sample | Replicate | Result µg/kg 12% moisture content | BDE-153 |
|---|-----------|---|-------------------------------------|
| 6 | 1 | | 0.0323 |
| | 2 | | 0.0297 |
| 9 | 1 | | 0.0349 |
| | 2 | | 0.0319 |
| 69 | 1 | | 0.0311 |
| | 2 | | 0.0310 |
| 71 | 1 | | 0.0315 |
| | 2 | | 0.0315 |
| 85 | 1 | | 0.0309 |
| | 2 | | 0.0324 |
| 111 | 1 | | 0.0304 |
| | 2 | | 0.0320 |
| 136 | 1 | | 0.0314 |
| | 2 | | 0.0344 |
| 152 | 1 | | 0.0321 |
| | 2 | | 0.0333 |
| 181 | 1 | | 0.0310 |
| | 2 | | 0.0316 |
| 185 | 1 | | 0.0333 |
| | 2 | | 0.0332 |
| Cochran's C-test | | | |
| C | | | 0.305 |
| 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} | | | 0.0320 |
| Standard deviation of sample averages s_x | | | 0.0010 |
| Within-sample standard deviation s_w | | | 0.0012 |
| Between-sample standard deviation s_s | | | 0.000374 |
| Standard deviation for proficiency assessment σ_{PT} | | | 0.0064 |
| s_s / σ_{PT} | | | 0.100 |
| Test for homogeneity ($s_s \leq 0.3 \sigma_{PT}$) | | | passed |



EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Hay 2025 [EURL-PT-POP_2502-HY]

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

Hay (2502-HY)

Selected PBDE congener - Stability test - Data

| Sample | Replicate | Result µg/kg 12% moisture content | BDE-153 | |
|---|-----------|---|---------|--------|
| 33 | 1 | | 0.0313 | |
| | 2 | | 0.0312 | |
| 150 | 1 | | 0.0315 | |
| | 2 | | 0.0337 | |
| 85 | 1 | | 0.0246 | |
| | 2 | | 0.0329 | |
| Stability test | | | | |
| General average (stability test) \bar{y} | | | | 0.0309 |
| General average (homogeneity test) \bar{x} | | | | 0.0320 |
| Standard deviation for proficiency assessment σ_{PT} | | | | 0.0064 |
| $ \bar{y} - \bar{x} $ | | | 0.00113 | |
| Test for stability ($ \bar{y} - \bar{x} \leq 0.3 \sigma_{PT}$) | | | passed | |



EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Hay 2025 [EURL-PT-POP_2502-HY]

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

21 May 2026

Annex 6: Participants' methods for PBDEs

Test sample - Hay (2502-HY)

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Hay 2025 [EURL-PT-P]
 EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Hay (2502-HY)

Methods PBDEs - Accreditation, weighed sample, standards

| LC | Data set | Sample | Accreditation according to ISO/IEC 17025 | Weighed sample [g] | Use of isotope-labelled internal standards for PBDE congeners (yes/no) |
|----|----------|---------|--|--------------------|--|
| 1 | | 2502-HY | | | |
| 2 | | 2502-HY | | | |
| 3 | | 2502-HY | | | |
| 4 | | 2502-HY | | | |
| 5 | | 2502-HY | | | |
| 6 | | 2502-HY | | | |
| 7 | A | 2502-HY | yes | 10 | yes |
| 8 | | 2502-HY | | | |
| 9 | A | 2502-HY | yes | 5 | yes |
| 10 | | 2502-HY | | | |
| 11 | A | 2502-HY | yes | 10 | yes |
| 12 | | 2502-HY | | | |
| 13 | | 2502-HY | | | |
| 14 | | 2502-HY | | | |
| 15 | | 2502-HY | | | |
| 16 | A | 2502-HY | yes | 10 | yes |
| 17 | | 2502-HY | | | |
| 18 | A | 2502-HY | yes | 10 | yes |
| 19 | | 2502-HY | | | |
| 20 | | 2502-HY | | | |
| 21 | | 2502-HY | | | |
| 22 | | 2502-HY | | | |
| 23 | | 2502-HY | | | |
| 24 | | 2502-HY | | | |
| 25 | | 2502-HY | | | |
| 26 | A | 2502-HY | yes | 3 | yes |
| 27 | A | 2502-HY | yes | 20 | yes |
| 28 | | 2502-HY | | | |
| 29 | | 2502-HY | | | |
| 30 | | 2502-HY | | | |
| 31 | | 2502-HY | | | |
| 32 | | 2502-HY | | | |
| 33 | | 2502-HY | | | |
| 34 | A | 2502-HY | yes | 20 | yes |
| 35 | | 2502-HY | | | |
| 36 | | 2502-HY | | | |
| 37 | | 2502-HY | | | |
| 38 | | 2502-HY | | | |
| 39 | A | 2502-HY | yes | 25.8739 | yes |
| 40 | | 2502-HY | | | |
| 41 | | 2502-HY | | | |
| 42 | | 2502-HY | | | |
| 43 | | 2502-HY | | | |
| 44 | A | 2502-HY | no | 10 | yes |
| 45 | A | 2502-HY | yes | 5.05 | yes |
| 46 | | 2502-HY | | | |
| 47 | | 2502-HY | | | |
| 48 | | 2502-HY | | | |
| 49 | | 2502-HY | | | |
| 50 | | 2502-HY | | | |
| 51 | | 2502-HY | | | |
| 52 | | 2502-HY | | | |
| 53 | | 2502-HY | | | |
| 54 | A | 2502-HY | no | 6 | yes |
| 55 | | 2502-HY | | | |
| 56 | | 2502-HY | | | |
| 57 | | 2502-HY | | | |
| 58 | | 2502-HY | | | |

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Hay 2025 [EURL-PT-P]
 EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Food

Hay (2502-HY)

Methods PBDEs - Accreditation, weighed sample, standards

| LC | Data set | Sample | Accreditation according to ISO/IEC 17025 | Weighed sample [g] | Use of isotope-labelled internal standards for PBDE congeners (yes/no) |
|------------------------|----------|---------|--|--------------------|--|
| 59 | | 2502-HY | | | |
| 60 | A | 2502-HY | yes | 36 | yes |
| 61 | | 2502-HY | | | |
| 62 | | 2502-HY | | | |
| 63 | | 2502-HY | | | |
| 64 | | 2502-HY | | | |
| 65 | | 2502-HY | | | |
| 66 | | 2502-HY | | | |
| 67 | | 2502-HY | | | |
| 68 | A | 2502-HY | | 10 | yes |
| 69 | | 2502-HY | | | |
| 70 | | 2502-HY | | | |
| 71 | | 2502-HY | | | |
| 72 | A | 2502-HY | no | 10.0589 | yes |
| 73 | | 2502-HY | | | |
| 74 | | 2502-HY | | | |
| 75 | | 2502-HY | | | |
| 76 | | 2502-HY | | | |
| 77 | | 2502-HY | | | |
| 78 | A | 2502-HY | yes | 10 | yes |
| 79 | A | 2502-HY | no | 2.668 | yes |
| 80 | A | 2502-HY | yes | 5 | yes |
| 81 | | 2502-HY | | | |
| 82 | A | 2502-HY | no | 31.97 | yes |
| 83 | | 2502-HY | | | |
| 84 | | 2502-HY | | | |
| 85 | | 2502-HY | | | |
| 86 | A | 2502-HY | yes | 5 | yes |
| 87 | | 2502-HY | | | |
| 88 | | 2502-HY | | | |
| 89 | A | 2502-HY | yes | 29.737 | yes |
| 90 | | 2502-HY | | | |
| 91 | | 2502-HY | | | |
| 92 | | 2502-HY | | | |
| 93 | | 2502-HY | | | |
| 94 | | 2502-HY | | | |
| 95 | A | 2502-HY | yes | 5 | yes |
| 96 | | 2502-HY | | | |
| 97 | | 2502-HY | | | |
| Additional Sets | | | | | |
| 16 | B | 2502-HY | no | 10 | yes |
| 86 | B | 2502-HY | no | 5 | yes |

| LC | Data set | Sample | BDE 28 | BDE 47 | BDE 49 | BDE 99 | BDE 100 | BDE 153 | BDE 154 | BDE 183 | BDE 209 | BDE 28 | BDE 47 | BDE 49 | BDE 99 | BDE 100 | BDE 153 | BDE 154 | BDE 183 | BDE 209 | | |
|----|----------|---------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|
| | | | Internal standard | Internal standard | Internal standard | Internal standard | Internal standard | Internal standard | Internal standard | Internal standard | Internal standard | Internal standard | Internal standard | Recovery standard | Recovery standard | Recovery standard | Recovery standard | Recovery standard | Recovery standard | Recovery standard | Recovery standard | Recovery standard |
| 1 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 2 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 3 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 4 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 5 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 6 | A | 2502-HY | 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 | 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 | 13C12-BDE-138 | |
| 7 | A | 2502-HY | 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 | 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 | 13C12-BDE-138 | |
| 8 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 9 | A | 2502-HY | 13C-TriBDE28 | 13C-TetraBDE47 | 13C-TetraBDE47 | 13C-PentaBDE99 | 13C-PentaBDE99 | 13C-HexaBDE153 | 13C-HexaBDE153 | 13C-OctaBDE183 | 13C-DecaBDE209 | 13C-HexaBDE138 | 13C-HexaBDE138 | 13C-HexaBDE138 | 13C-HexaBDE138 | 13C-HexaBDE138 | 13C-HexaBDE138 | 13C-HexaBDE138 | 13C-HexaBDE138 | 13C-HexaBDE138 | 13C-HexaBDE138 | |
| 10 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 11 | A | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 12 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 13 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 14 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 15 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 16 | A | 2502-HY | 13C12-PBDE-28 | 13C12-PBDE-47 | 13C12-PBDE-47 | 13C12-PBDE-99 | 13C12-PBDE-100 | 13C12-PBDE-153 | 13C12-PBDE-154 | 13C12-PBDE-183 | 13C12-PBDE-209 | 13C12-PBDE-138 | 13C12-PBDE-138 | 13C12-PBDE-138 | 13C12-PBDE-138 | 13C12-PBDE-138 | 13C12-PBDE-138 | 13C12-PBDE-138 | 13C12-PBDE-138 | 13C12-PBDE-138 | 13C12-PBDE-138 | |
| 17 | | 2502-HY | BDE-28 | BDE-47 | BDE-47 | BDE-99 | BDE-100 | BDE-153 | BDE-154 | BDE-183 | BDE-209 | BDE-79 | BDE-79 | BDE-79 | BDE-79 | BDE-79 | BDE-138 | BDE-138 | BDE-138 | BDE-138 | BDE-206 | |
| 18 | A | 2502-HY | 13C-BDE28 | 13C-BDE47 | 13C-BDE47 | 13C-BDE99 | 13C-BDE100 | 13C-BDE153 | 13C-BDE154 | 13C-BDE183 | 13C-BDE209 | 13C-BDE118 | 13C-BDE118 | 13C-BDE118 | 13C-BDE118 | 13C-BDE118 | 13C-BDE118 | 13C-BDE118 | 13C-BDE118 | 13C-BDE118 | 13C-BDE208 | |
| 19 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 20 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 21 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 22 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 23 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 24 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 25 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 26 | A | 2502-HY | 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 | 13C BDE-77 | 13C BDE-77 | 13C BDE-77 | 13C BDE-77 | 13C BDE-77 | 13C BDE-138 | 13C BDE-138 | 13C BDE-138 | 13C BDE-138 | 13C BDE-206 | |
| 27 | A | 2502-HY | 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-PCB 52 | 13C12-PCB 52 | 13C12-PCB 52 | 13C12-PCB 138 | 13C12-PCB 138 | 13C12-PCB 138 | 13C12-PCB 138 | 13C12-PCB 138 | 13C12-PCB 138 | - | |
| 28 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 29 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 30 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 31 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 32 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 33 | A | 2502-HY | BDE-28 | BDE-47 | BDE-47 | BDE-99 | BDE-100 | BDE-153 | BDE-154 | BDE-183 | BDE-209 | PCB-138 | PCB-138 | PCB-138 | PCB-138 | PCB-138 | PCB-138 | PCB-138 | PCB-138 | PCB-138 | PCB-138 | |
| 34 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 35 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 36 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 37 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 38 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 39 | A | 2502-HY | 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 | 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-138 | 13C-BDE-206 | |
| 40 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 41 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 42 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 43 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 44 | A | 2502-HY | MBDE-28 | MBDE-47 | MBDE-47 | MBDE-99 | MBDE-100 | MBDE-153 | MBDE-154 | MBDE-183 | MBDE-209 | MBDE-79 | MBDE-79 | MBDE-79 | MBDE-139 | MBDE-139 | MBDE-139 | MBDE-139 | MBDE-139 | MBDE-139 | MBDE-206 | |
| 45 | A | 2502-HY | 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 | 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 | 13C12 BDE-138 | |
| 46 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 47 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 48 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 49 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 50 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 51 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 52 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 53 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 54 | A | 2502-HY | 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 | 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-138 | 13C-BDE-206 | |
| 55 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 56 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 57 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 58 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 59 | A | 2502-HY | 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 | 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-126 | 13C-BDE-207 | |
| 60 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 61 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 62 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 63 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 64 | | 2502-HY | 13C-BDE-28 | 13C-BDE-47 | 13C-BDE-47 | 13C-BDE-99 | 13C-BDE-99 | 13C-BDE-153 | 13C-BDE-154 | 13C-BDE-183 | | 13C-PCB-80 | 13C-PCB-80 | 13C-PCB-80 | 13C-PCB-80 | 13C-PCB-80 | 13C-PCB-80 | 13C-PCB-80 | 13C-PCB-80 | 13C-PCB-80 | 13C-PCB-80 | |
| 65 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 66 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 67 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 68 | A | 2502-HY | 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 | 13c bde 139 | 13c bde 139 | 13c bde 139 | 13c bde 139 | 13c bde 139 | 13c bde 139 | 13c bde 139 | 13c bde 139 | 13c bde 139 | 13c bde 139 | |
| 69 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 70 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 71 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 72 | A | 2502-HY | BDE-28 | BDE-47 | | BDE-99 | BDE-100 | BDE-153 | BDE-154 | BDE-183 | | PCB-52 | PCB-52 | | PCB-52 | PCB-52 | PCB-138 | PCB-138 | PCB-138 | PCB-138 | | |
| 73 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 74 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 75 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 76 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 77 | | 2502-HY | | | | | | | | | | | | | | | | | | | | |
| 78 | A | 2502-HY | 13C12-BDE-28 | 13C12-BDE-49 | 13C12-BDE-47 | 13C12-BDE-99 | 13C12-BDE-100 | 13C12-BDE-153 | 13C12-BDE-154 | 13C12-BDE-183 | 13C12-BDE-209 | 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 | 13C12-BDE-139 | |
| 79 | A | 2502-HY | 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 | 90 | 101 | 101 | 97 | 86 | 61 | 7 | | | | |

Hay (2502-HY)
 Methods PBDEs - Extraction

| LC | Data set | Sample | Extraction | Sample preparation/pre-treatment | Extraction technique | Extraction solvent | Extraction time [h] | Extraction temperature [°C] | Extraction pressure [MPa] |
|----|----------|---------|------------|--|---|---|---------------------------|----------------------------------|--|
| 1 | | 2502-HY | | | | | | | |
| 2 | | 2502-HY | | | | | | | |
| 3 | | 2502-HY | | | | | | | |
| 4 | | 2502-HY | | | | | | | |
| 5 | | 2502-HY | | | | | | | |
| 6 | | 2502-HY | | | | | | | |
| 7 | A | 2502-HY | | - | Pressurized liquid extraction | (1) Toluene - (2) Toluene:Ethanol 90:10 | 0.25 | 100 | 10.3 |
| 8 | | 2502-HY | | | | | | | |
| 9 | A | 2502-HY | | homogenization | Pressurized liquid extraction | | | | |
| 10 | | 2502-HY | | | | | | | |
| 11 | A | 2502-HY | | | Soxhlet | toluene/acetone 7/3 | 6 | | |
| 12 | | 2502-HY | | | | | | | |
| 13 | | 2502-HY | | | | | | | |
| 14 | | 2502-HY | | | | | | | |
| 15 | | 2502-HY | | | | | | | |
| 16 | A | 2502-HY | | | Pressurized liquid extraction | Hexane/Acetone (70:30) | 1 | 125 | 10.3 |
| 17 | | 2502-HY | | | | | | | |
| 18 | A | 2502-HY | | | Soxhlet | toluene/ethanol 70/30 | 12 | bp | ambient |
| 19 | | 2502-HY | | | | | | | |
| 20 | | 2502-HY | | | | | | | |
| 21 | | 2502-HY | | | | | | | |
| 22 | | 2502-HY | | | | | | | |
| 23 | | 2502-HY | | | | | | | |
| 24 | | 2502-HY | | | | | | | |
| 25 | | 2502-HY | | | | | | | |
| 26 | A | 2502-HY | | drying | Pressurized liquid extraction | Toluene / Acetone (70 / 30) | 1 | 120 | 10 |
| 27 | A | 2502-HY | | thorough homogenization | Soxhlet | toluene:methanol 2:1 | 21 | 109 | 0,1 |
| 28 | | 2502-HY | | | | | | | |
| 29 | | 2502-HY | | | | | | | |
| 30 | | 2502-HY | | | | | | | |
| 31 | | 2502-HY | | | | | | | |
| 32 | | 2502-HY | | | | | | | |
| 33 | | 2502-HY | | | | | | | |
| 34 | A | 2502-HY | | homogenization; drying to determine moisture content | Pressurized liquid extraction , Soxhlet | ASE: toluene, toluene:EtOH (9:1, v/v); Soxhlet: toluene | ASE: 40 min; Soxhlet: 8 h | ASE: 100 °C | ASE: 10,34 MPa; Soxhlet: atm. pressure |
| 35 | | 2502-HY | | | | | | | |
| 36 | | 2502-HY | | | | | | | |
| 37 | | 2502-HY | | | | | | | |
| 38 | | 2502-HY | | | | | | | |
| 39 | A | 2502-HY | | Homogenisation | Soxhlet | toluene/ethanol 50/50 | 24 | 120 | 0.1013 |
| 40 | | 2502-HY | | | | | | | |
| 41 | | 2502-HY | | | | | | | |
| 42 | | 2502-HY | | | | | | | |
| 43 | | 2502-HY | | | | | | | |
| 44 | A | 2502-HY | | - | Pressurized liquid extraction | 1) Toluol (cycles: 1) 2) Toluol/Ethanol (9:1) (cycles: 2) | 1 h | 100 °C | 10 MPa |
| 45 | A | 2502-HY | | homogenisation | Soxhlet | toluene/acetone | 16 | | |
| 46 | | 2502-HY | | | | | | | |
| 47 | | 2502-HY | | | | | | | |
| 48 | | 2502-HY | | | | | | | |
| 49 | | 2502-HY | | | | | | | |
| 50 | | 2502-HY | | | | | | | |
| 51 | | 2502-HY | | | | | | | |
| 52 | | 2502-HY | | | | | | | |
| 53 | | 2502-HY | | | | | | | |
| 54 | A | 2502-HY | | | Soxhlet | Hexane/Dichloromethane (1/1) | 24 | | |
| 55 | | 2502-HY | | | | | | | |
| 56 | | 2502-HY | | | | | | | |
| 57 | | 2502-HY | | | | | | | |
| 58 | | 2502-HY | | | | | | | |
| 59 | | 2502-HY | | | | | | | |
| 60 | A | 2502-HY | | | Soxhlet | Toluene / Ethanol (30/70) | 12 | boiling point of solvent mixture | |
| 61 | | 2502-HY | | | | | | | |
| 62 | | 2502-HY | | | | | | | |
| 63 | | 2502-HY | | | | | | | |
| 64 | | 2502-HY | | | | | | | |
| 65 | | 2502-HY | | | | | | | |
| 66 | | 2502-HY | | | | | | | |
| 67 | | 2502-HY | | | | | | | |
| 68 | A | 2502-HY | | | Soxhlet | DCM:hex 1:1 | 6 | 80 | room |
| 69 | | 2502-HY | | | | | | | |
| 70 | | 2502-HY | | | | | | | |
| 71 | | 2502-HY | | | | | | | |
| 72 | A | 2502-HY | | hydromatrix added | ASE | Hexane/Acetona (5:1) | 20 min | 100 °C | 1500 psi |
| 73 | | 2502-HY | | | | | | | |

Hay (2502-HY)
 Methods PBDEs - Extraction

| LC | Data set | Sample | Extraction | Sample preparation/pre-treatment | Extraction technique | Extraction solvent | Extraction time [h] | Extraction temperature [°C] | Extraction pressure [MPa] |
|------------------------|----------|---------|------------|--|-------------------------------|-------------------------------|---------------------|-----------------------------|---------------------------|
| 74 | | 2502-HY | | | | | | | |
| 75 | | 2502-HY | | | | | | | |
| 76 | | 2502-HY | | | | | | | |
| 77 | | 2502-HY | | | | | | | |
| 78 | A | 2502-HY | | no | Pressurized liquid extraction | toluene /acetone (70/30, v/v) | 0,5 | 100 | 10 |
| 79 | A | 2502-HY | | | Pressurized liquid extraction | hexane/acetone 1:1 | 0,5 | 100 | 10.3 |
| 80 | A | 2502-HY | | | Soxhlet | Toluene | 12 | | |
| 81 | | 2502-HY | | | | | | | |
| 82 | A | 2502-HY | | drying the sample in a vacuum oven, then, mixed with sodium sulphate before extraction | Twisselmann | n-hexane:DCM (1:1) | 24 | | |
| 83 | | 2502-HY | | | | | | | |
| 84 | | 2502-HY | | | | | | | |
| 85 | | 2502-HY | | | | | | | |
| 86 | A | 2502-HY | | | QuEChERS like-extraction | Ethyl acetate | | | |
| 87 | | 2502-HY | | | | | | | |
| 88 | | 2502-HY | | | | | | | |
| 89 | A | 2502-HY | | - | Pressurized liquid extraction | Toluene | 1 | 150 | 13 |
| 90 | | 2502-HY | | | | | | | |
| 91 | | 2502-HY | | | | | | | |
| 92 | | 2502-HY | | | | | | | |
| 93 | | 2502-HY | | | | | | | |
| 94 | | 2502-HY | | | | | | | |
| 95 | A | 2502-HY | | homogenization | Pressurized liquid extraction | | | | |
| 96 | | 2502-HY | | | | | | | |
| 97 | | 2502-HY | | | | | | | |
| Additional Sets | | | | | | | | | |
| 16 | B | 2502-HY | | | Pressurized liquid extraction | Hexane/Acetone (70:30) | 1 | 125 | 10.3 |
| 86 | B | 2502-HY | | | QuEChERS like-extraction | Ethyl acetate | | | |

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Hay (2502-HY)

Methods PBDEs - Clean-up

| LC | Data set | Sample | Clean-up | | | | | | Other columns / clean-up method | Final volume [µl]: PBDE |
|----|----------|---------|-------------------|-----------------------------|-----------------|----------------|---------------|------------------------|--|-------------------------|
| | | | Gelchromatography | Silica/sulfuric acid column | Florisil column | Alumina column | Carbon column | Fully automated system | | |
| 1 | | 2502-HY | | | | | | | | |
| 2 | | 2502-HY | | | | | | | | |
| 3 | | 2502-HY | | | | | | | | |
| 4 | | 2502-HY | | | | | | | | |
| 5 | | 2502-HY | | | | | | | | |
| 6 | | 2502-HY | | | | | | | | |
| 7 | A | 2502-HY | no | no | no | no | no | DexTech | no | 100 |
| 8 | | 2502-HY | | | | | | | | |
| 9 | A | 2502-HY | no | yes | no | no | no | no | no | 100 |
| 10 | | 2502-HY | | | | | | | | |
| 11 | A | 2502-HY | no | yes | no | yes | yes | DexTech | no | 100 |
| 12 | | 2502-HY | | | | | | | | |
| 13 | | 2502-HY | | | | | | | | |
| 14 | | 2502-HY | | | | | | | | |
| 15 | | 2502-HY | | | | | | | | |
| 16 | A | 2502-HY | no | yes | no | yes | yes | no | acid hydrolysis with sulphuric acid | 60 |
| 17 | | 2502-HY | | | | | | | | |
| 18 | A | 2502-HY | no | yes | no | yes | yes | no | | 500 |
| 19 | | 2502-HY | | | | | | | | |
| 20 | | 2502-HY | | | | | | | | |
| 21 | | 2502-HY | | | | | | | | |
| 22 | | 2502-HY | | | | | | | | |
| 23 | | 2502-HY | | | | | | | | |
| 24 | | 2502-HY | | | | | | | | |
| 25 | | 2502-HY | | | | | | | | |
| 26 | A | 2502-HY | no | no | no | no | no | Miura | no | 20 |
| 27 | A | 2502-HY | no | yes | no | yes | yes | no | basic silica, silver nitrate silica | 50 |
| 28 | | 2502-HY | | | | | | | | |
| 29 | | 2502-HY | | | | | | | | |
| 30 | | 2502-HY | | | | | | | | |
| 31 | | 2502-HY | | | | | | | | |
| 32 | | 2502-HY | | | | | | | | |
| 33 | | 2502-HY | | | | | | | | |
| 34 | A | 2502-HY | no | yes | yes | yes | yes | yes | Power-Prep FMS with PBDE Free disp. columns; Supelco Supelclean TM Sulfoxide SPE 6 g/ 20 ml (removal of waxes) | 20 |
| 35 | | 2502-HY | | | | | | | | |
| 36 | | 2502-HY | | | | | | | | |
| 37 | | 2502-HY | | | | | | | | |
| 38 | | 2502-HY | | | | | | | | |
| 39 | A | 2502-HY | no | yes | no | yes | no | no | | 25 |
| 40 | | 2502-HY | | | | | | | | |
| 41 | | 2502-HY | | | | | | | | |
| 42 | | 2502-HY | | | | | | | | |
| 43 | | 2502-HY | | | | | | | | |
| 44 | A | 2502-HY | no | no | no | no | no | Miura | no | 50 |
| 45 | A | 2502-HY | no | no | no | yes | no | no | | 20 |
| 46 | | 2502-HY | | | | | | | | |
| 47 | | 2502-HY | | | | | | | | |
| 48 | | 2502-HY | | | | | | | | |
| 49 | | 2502-HY | | | | | | | | |
| 50 | | 2502-HY | | | | | | | | |
| 51 | | 2502-HY | | | | | | | | |
| 52 | | 2502-HY | | | | | | | | |
| 53 | | 2502-HY | | | | | | | | |
| 54 | A | 2502-HY | no | yes | no | yes | no | no | | 20 |
| 55 | | 2502-HY | | | | | | | | |
| 56 | | 2502-HY | | | | | | | | |
| 57 | | 2502-HY | | | | | | | | |
| 58 | | 2502-HY | | | | | | | | |
| 59 | | 2502-HY | | | | | | | | |
| 60 | A | 2502-HY | no | yes | no | yes | no | no | acidic treatment | 50 |
| 61 | | 2502-HY | | | | | | | | |
| 62 | | 2502-HY | | | | | | | | |
| 63 | | 2502-HY | | | | | | | | |
| 64 | | 2502-HY | | | | | | | | |
| 65 | | 2502-HY | | | | | | | | |
| 66 | | 2502-HY | | | | | | | | |
| 67 | | 2502-HY | | | | | | | | |
| 68 | A | 2502-HY | yes | yes | no | no | yes | yes | | 20 |
| 69 | | 2502-HY | | | | | | | | |
| 70 | | 2502-HY | | | | | | | | |
| 71 | | 2502-HY | | | | | | | | |
| 72 | A | 2502-HY | no | no | no | no | no | Miura | no | 20 |
| 73 | | 2502-HY | | | | | | | | |

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Hay (2502-HY)

Methods PBDEs - Clean-up

| LC | Data set | Sample | Clean-up | | | | | | | Other columns / clean-up method | Final volume [µl]: PBDE |
|------------------------|----------|---------|-------------------|-----------------------------|-----------------|----------------|---------------|------------------------|-----------------------|--|-------------------------|
| | | | Gelchromatography | Silica/sulfuric acid column | Florisil column | Alumina column | Carbon column | Fully automated system | Semi-automated system | | |
| 74 | | 2502-HY | | | | | | | | | |
| 75 | | 2502-HY | | | | | | | | | |
| 76 | | 2502-HY | | | | | | | | | |
| 77 | | 2502-HY | | | | | | | | | |
| 78 | A | 2502-HY | no | yes | yes | yes | no | | no | | 50 |
| 79 | A | 2502-HY | no | yes | no | no | no | | no | | 2500 |
| 80 | A | 2502-HY | no | yes | no | yes | yes | Miura | no | Silica/AgNO3 | 100 |
| 81 | | 2502-HY | | | | | | | | | |
| 82 | A | 2502-HY | no | yes | no | no | no | | no | basic set of "power-prep system" columns | 40 |
| 83 | | 2502-HY | | | | | | | | | |
| 84 | | 2502-HY | | | | | | | | | |
| 85 | | 2502-HY | | | | | | | | | |
| 86 | A | 2502-HY | no | yes | no | no | no | | no | d-SPE on Agilent EMR dispersive kit | 250 |
| 87 | | 2502-HY | | | | | | | | | |
| 88 | | 2502-HY | | | | | | | | | |
| 89 | A | 2502-HY | no | yes | no | yes | yes | DexTech | no | | 100 |
| 90 | | 2502-HY | | | | | | | | | |
| 91 | | 2502-HY | | | | | | | | | |
| 92 | | 2502-HY | | | | | | | | | |
| 93 | | 2502-HY | | | | | | | | | |
| 94 | | 2502-HY | | | | | | | | | |
| 95 | A | 2502-HY | no | yes | no | no | no | | no | | 100 |
| 96 | | 2502-HY | | | | | | | | | |
| 97 | | 2502-HY | | | | | | | | | |
| Additional Sets | | | | | | | | | | | |
| 16 | B | 2502-HY | no | yes | no | yes | yes | | no | acid hydrolysis with sulphuric acid | 60 |
| 86 | B | 2502-HY | no | yes | no | no | no | | no | d-SPE on Agilent EMR dispersive kit | 250 |

| LC | Data set | Sample | Chromatographic separation and detection method | | | Detector | Limit of quantification Determination of LOQ | Measurement uncertainty Estimation |
|----|----------|---------|---|-------------------------|---|----------|--|---|
| | | | GC injection | Injected volume [µl] | GC separation: Stationary phase | | | |
| 1 | | 2502-HY | | | | | | |
| 2 | | 2502-HY | | | | | | |
| 3 | | 2502-HY | | | | | | |
| 4 | | 2502-HY | | | | | | |
| 5 | | 2502-HY | | | | | | |
| 6 | | 2502-HY | | | | | | |
| 7 | A | 2502-HY | PTV | 4 | RTX-1614 | HRMS | LOQs were based on the blank level contamination and on the signal to noise (for compounds not present in the blank) | MU was calculated according to the dioxin guideline based on replicates of PTs samples. A single MU have been calculated for all the matrices using the same extraction, purification and GC-MS method |
| 8 | | 2502-HY | | | | | | |
| 9 | A | 2502-HY | Splitless | 2 | | MS/MS | 2 times higher than blanks and S/N ratio over 10 | Proficiency testing and spiked samples |
| 10 | | 2502-HY | | | | | | |
| 11 | A | 2502-HY | Splitless | 2 | DB5 MS | HRMS | | |
| 12 | | 2502-HY | | | | | | |
| 13 | | 2502-HY | | | | | | |
| 14 | | 2502-HY | | | | | | |
| 15 | | 2502-HY | | | | | | |
| 16 | A | 2502-HY | PTV | 2 | Rtx-1614 30m x 0.25mm x 0.1µm□ | HRMS | 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 Uncertainty for Laboratories performing PCDD/F and PCB Analysis using Isotope Dilution Mass Spectrometry |
| 17 | | 2502-HY | | | | | | |
| 18 | A | 2502-HY | Splitless | 5 | (5%-phenyl)-methylpolysiloxane | MS/MS | | |
| 19 | | 2502-HY | | | | | | |
| 20 | | 2502-HY | | | | | | |
| 21 | | 2502-HY | | | | | | |
| 22 | | 2502-HY | | | | | | |
| 23 | | 2502-HY | | | | | | |
| 24 | | 2502-HY | | | | | | |
| 25 | | 2502-HY | | | | | | |
| 26 | A | 2502-HY | Splitless | 2 | DB5MS | HRMS | LOD = LOQ (S/N =3) | GUM |
| 27 | A | 2502-HY | pulsed splitless | 1 | DB-5MS (60m x 0,25mm x 0,10 µm) | HRMS | estimated from procedural blanks | based on validation - calculation from RSD and bias |
| 28 | | 2502-HY | | | | | | |
| 29 | | 2502-HY | | | | | | |
| 30 | | 2502-HY | | | | | | |
| 31 | | 2502-HY | | | | | | |
| 32 | | 2502-HY | | | | | | |
| 33 | | 2502-HY | | | | | | |
| 34 | A | 2502-HY | Splitless | 2 | DB-5HT | HRMS | 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. uncertainty of weighing, volume measurements, etc.), Uc = combined uncertainty is summed uncertainty of type A and B according the law of propagation of measurement uncertainty. |
| 35 | | 2502-HY | | | | | | |
| 36 | | 2502-HY | | | | | | |
| 37 | | 2502-HY | | | | | | |
| 38 | | 2502-HY | | | | | | |
| 39 | A | 2502-HY | Splitless | 1 | Rtx-1614 | HRMS | | |
| 40 | | 2502-HY | | | | | | |
| 41 | | 2502-HY | | | | | | |
| 42 | | 2502-HY | | | | | | |
| 43 | | 2502-HY | | | | | | |
| 44 | A | 2502-HY | PTV | 4 | Rtx-1614 (15m x 0,25 mm x 0,1 µm) + 2m retention gap (uncoated) | MS/MS | | |
| 45 | A | 2502-HY | PTV | 2 | 30 m DB-5 MS 0,25 mm 0,25 µm | HRMS | LOQ = 3 * Limit of Detection (LOD), LOD determined using blank samples | nach DIN ISO 11352:2013-03 Chapter. 8.2, Abs. 8.2.2 |
| 46 | | 2502-HY | | | | | | |
| 47 | | 2502-HY | | | | | | |
| 48 | | 2502-HY | | | | | | |
| 49 | | 2502-HY | | | | | | |
| 50 | | 2502-HY | | | | | | |
| 51 | | 2502-HY | | | | | | |
| 52 | | 2502-HY | | | | | | |

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Hay (2502-HY)
 Methods PBDEs - Detection, LOQ, measurement uncertainty

| LC | Data set | Sample | Chromatographic separation and detection method | | | Detector | Limit of quantification Determination of LOQ | Measurement uncertainty Estimation |
|------------------------|----------|---------|---|-------------------------|---|----------|---|---------------------------------------|
| | | | GC injection | Injected volume [µl] | GC separation: Stationary phase | | | |
| 53 | | 2502-HY | | | | | | |
| 54 | A | 2502-HY | Splitless | 1 | DB5MS | HRMS | | |
| 55 | | 2502-HY | | | | | | |
| 56 | | 2502-HY | | | | | | |
| 57 | | 2502-HY | | | | | | |
| 58 | | 2502-HY | | | | | | |
| 59 | | 2502-HY | | | | | | |
| 60 | A | 2502-HY | Splitless | 2 | DB-5HT | HRMS | The LOQ was determined with fortified matrix. The measurement uncertainty was calculated according to NF ISO 11352. | |
| 61 | | 2502-HY | | | | | | |
| 62 | | 2502-HY | | | | | | |
| 63 | | 2502-HY | | | | | | |
| 64 | | 2502-HY | | | | | | |
| 65 | | 2502-HY | | | | | | |
| 66 | | 2502-HY | | | | | | |
| 67 | | 2502-HY | | | | | | |
| 68 | A | 2502-HY | PTV | 5 | phenomenex zebron ZB semi volatiles | MS/MS | 0.01 (lowest spiked level reaching requirements, mentioned in the EURL recommendation) taking into account uncertainty from bias and RSD under reproducibility conditions and rounding up | |
| 69 | | 2502-HY | | | | | | |
| 70 | | 2502-HY | | | | | | |
| 71 | | 2502-HY | | | | | | |
| 72 | A | 2502-HY | Splitless | 1 | DB5ms | HRMS | quantitation software calculation based on s/n Horwitz | |
| 73 | | 2502-HY | | | | | | |
| 74 | | 2502-HY | | | | | | |
| 75 | | 2502-HY | | | | | | |
| 76 | | 2502-HY | | | | | | |
| 77 | | 2502-HY | | | | | | |
| 78 | A | 2502-HY | PTV | 1.5 | RTX-1614 , DB-5-MS | HRMS | calculated from validation and verified with a blank sample validation | |
| 79 | A | 2502-HY | PTV | 100 | Rtx-CL Pesticides | HRMS | LOR = 0.004 µg/kg 12% moisture, determined by validation study 0.25 | |
| 80 | A | 2502-HY | PTV | 1 | ZB-Semivolatiles | MS/MS | Mean of Blank + 3s multiple analysis of reference material | |
| 81 | | 2502-HY | | | | | | |
| 82 | A | 2502-HY | Splitless | 1 | DB-5MS (30 m, 0.25 mm id, 0.25 mm film) | HRMS | LOQs were calculated using the "Quandesk" software | |
| 83 | | 2502-HY | | | | | | |
| 84 | | 2502-HY | | | | | | |
| 85 | | 2502-HY | | | | | | |
| 86 | A | 2502-HY | PTV | 10 | DB5HT 15 m x 0.25 mm; 0.1 µm | MS/MS | 0.020 µg/kg (0.100 µg/kg for BDE 209) determined by fortified samples Nordtest | |
| 87 | | 2502-HY | | | | | | |
| 88 | | 2502-HY | | | | | | |
| 89 | A | 2502-HY | Splitless | 1 | 5MS | MS/MS | Not calculated (No validation available) Estimated (No validation available) | |
| 90 | | 2502-HY | | | | | | |
| 91 | | 2502-HY | | | | | | |
| 92 | | 2502-HY | | | | | | |
| 93 | | 2502-HY | | | | | | |
| 94 | | 2502-HY | | | | | | |
| 95 | A | 2502-HY | Splitless | 2 | | MS/MS | 2 times higher than blanks and S/N ratio over 10 Proficiency testing and spiked samples | |
| 96 | | 2502-HY | | | | | | |
| 97 | | 2502-HY | | | | | | |
| Additional Sets | | | | | | | | |
| 16 | B | 2502-HY | | 5 | | HRMS | | |
| 86 | B | 2502-HY | PTV | 10 | DB5HT 15 m x 0.25 mm; 0.1 µm | MS/MS | 0.020 µg/kg (0.100 µg/kg for BDE 209) determined by fortified samples | |



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21 May 2026

Annex 7: Assigned values of HBCDDs

Test sample - Hay (2502-HY)

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 exclusion of extreme outliers



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

Hay (2502-HY)

HBCDD - Assigned values

| Analyte | Result µg/kg (12% moisture content) | Assigned value [outliers removed] | Robust standard deviation [outliers removed] | Standard uncertainty [outliers removed] | No. of results contributing to assigned value | Median [all values] |
|--|---|--------------------------------------|--|---|---|------------------------|
| Sum of α-, β-, γ-HBCDD (ub) | | 1.08 | 0.232 | 0.075 | 15 | 1.04 |
| Sum of α-, β-, γ-HBCDD (lb) | | 1.07 | 0.246 | 0.079 | 15 | 1.04 |
| (+/-)-α-HBCDD (1,2,5,6,9,10-hexabromo-(1R,2R,5S,6R,9R,10S)-rel-cyclododecane) | | 0.107 | 0.0145 | 0.0047 | 15 | 0.110 |
| (+/-)-β- HBCDD (1,2,5,6,9,10-hexabromo-(1R,2S,5R,6R,9R,10S)-rel-cyclododecane) | | | | | | 0.0250 |
| (+/-)-γ- HBCDD (1,2,5,6,9,10-hexabromo-(1R,2R,5R,6S,9S,10R)-rel-cyclododecane) | | 0.969 | 0.225 | 0.070 | 16 | 0.918 |
| Total HBCDD (using GC-methods) | | | | | | |



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Hay (2502-HY)

Moisture content (HBCDD) - Assigned value

| Analyte | Result % | Assigned value [outliers removed] | Robust standard deviation [outliers removed] | Standard uncertainty [outliers removed] | No. of results contributing to assigned value | Median [all values] |
|------------------|----------|--------------------------------------|---|--|---|------------------------|
| Moisture content | | 8.34 | 0.629 | 0.17 | 21 | |



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Annex 8: Participants' results of HBCDDs

Test sample - Hay (2502-HY)

Hay (2502-HY)
HBCDD - Results

| LC | Data set | Sample | Result µg/kg 12% moisture content | Sum of α-, β-, γ-HBCDD (ub) | Sum of α-, β-, γ-HBCDD (lb) | (+/-)-α-HBCDD | | (+/-)-β- HBCDD | | (+/-)-γ- HBCDD | | Total HBCDD (using GC-methods) |
|----|----------|---------|---|--------------------------------|--------------------------------|---|---|---|---|----------------|--|-----------------------------------|
| | | | | | | 1,2,5,6,9,10-hexabromo-(1R,2R,5S,6R,9R,10S)- rel-cyclododecane | 1,2,5,6,9,10-hexabromo-(1R,2S,5R,6R,9R,10S)- rel-cyclododecane | 1,2,5,6,9,10-hexabromo-(1R,2R,5R,6R,9R,10S)- rel-cyclododecane | 1,2,5,6,9,10-hexabromo-(1R,2R,5R,6S,9S,10R)- rel-cyclododecane | | | |
| 1 | A | 2502-HY | | 0.92 | 0.91 | 0.08 | | 0.01 | | 0.82 | | |
| 2 | | 2502-HY | | | | | | | | | | |
| 3 | | 2502-HY | | | | | | | | | | |
| 4 | | 2502-HY | | | | | | | | | | |
| 5 | | 2502-HY | | | | | | | | | | |
| 6 | | 2502-HY | | | | | | | | | | |
| 7 | | 2502-HY | | | | | | | | | | |
| 8 | | 2502-HY | | | | | | | | | | |
| 9 | A | 2502-HY | | 1.01 | 0.992 | 0.095 | | <0.02 | | 0.897 | | |
| 10 | | 2502-HY | | | | | | | | | | |
| 11 | A | 2502-HY | | | | | | | | | | |
| 12 | | 2502-HY | | | | | | | | | | |
| 13 | | 2502-HY | | | | | | | | | | |
| 14 | | 2502-HY | | | | | | | | | | |
| 15 | A | 2502-HY | | 0.824 | 0.79 | 0.078 | | <0.03 | | 0.712 | | |
| 16 | A | 2502-HY | | 1.33 | 1.33 | 0.112 | | 0.0165 | | 1.2 | | |
| 17 | | 2502-HY | | | | | | | | | | |
| 18 | A | 2502-HY | | 1.36 | 1.36 | 0.13 | | 0.027 | | 1.2 | | |
| 19 | | 2502-HY | | | | | | | | | | |
| 20 | A | 2502-HY | | 1.4 | 1.4 | 0.12 | | | | 1.26 | | |
| 22 | | 2502-HY | | | | | | | | | | |
| 23 | | 2502-HY | | | | | | | | | | |
| 24 | | 2502-HY | | | | | | | | | | |
| 25 | | 2502-HY | | | | | | | | | | |
| 26 | A | 2502-HY | | 1.02 | 1.02 | 0.113 | | 0.015 | | 0.894 | | |
| 27 | A | 2502-HY | | 0.919 | 0.869 | 0.104 | | <0.05 | | 0.765 | | |
| 28 | | 2502-HY | | | | | | | | | | |
| 29 | | 2502-HY | | | | | | | | | | |
| 30 | | 2502-HY | | | | | | | | | | |
| 31 | | 2502-HY | | | | | | | | | | |
| 32 | | 2502-HY | | | | | | | | | | |
| 33 | | 2502-HY | | | | | | | | | | |
| 34 | | 2502-HY | | | | | | | | | | |
| 35 | | 2502-HY | | | | | | | | | | |
| 36 | A | 2502-HY | | 3.29 | 3.01 | <0.191 | | <0.096 | | 3.01 | | |
| 37 | | 2502-HY | | | | | | | | | | |
| 38 | | 2502-HY | | | | | | | | | | |
| 39 | A | 2502-HY | | 1.15 | 1.13 | 0.14 | | <0.025 | | 0.989 | | |
| 40 | | 2502-HY | | | | | | | | | | |
| 41 | | 2502-HY | | | | | | | | | | |
| 42 | | 2502-HY | | | | | | | | | | |
| 43 | | 2502-HY | | | | | | | | | | |
| 44 | A | 2502-HY | | 1.04 | 1.04 | 0.104 | | 0.0153 | | 0.918 | | |
| 45 | | 2502-HY | | | | | | | | | | |
| 46 | | 2502-HY | | | | | | | | | | |
| 47 | | 2502-HY | | | | | | | | | | |
| 48 | | 2502-HY | | | | | | | | | | |
| 49 | | 2502-HY | | | | | | | | | | |
| 50 | | 2502-HY | | | | | | | | | | |
| 51 | | 2502-HY | | | | | | | | | | |
| 52 | | 2502-HY | | | | | | | | | | |
| 53 | | 2502-HY | | | | | | | | | | |
| 54 | | 2502-HY | | | | | | | | | | |
| 55 | | 2502-HY | | | | | | | | | | |
| 56 | | 2502-HY | | | | | | | | | | |
| 57 | | 2502-HY | | | | | | | | | | |
| 58 | | 2502-HY | | | | | | | | | | |
| 59 | | 2502-HY | | | | | | | | | | |
| 60 | | 2502-HY | | | | | | | | | | |
| 61 | | 2502-HY | | | | | | | | | | |
| 62 | | 2502-HY | | | | | | | | | | |
| 63 | | 2502-HY | | | | | | | | | | |
| 64 | | 2502-HY | | | | | | | | | | |
| 65 | | 2502-HY | | | | | | | | | | |
| 66 | | 2502-HY | | | | | | | | | | |
| 67 | | 2502-HY | | | | | | | | | | |

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Hay 2025 [EURL-PT-POP_2502-HY]

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

Hay (2502-HY)
HBCDD - Results

| LC | Data set | Sample | Result µg/kg 12% moisture content | Sum of α-, β-, γ-HBCDD (ub) | Sum of α-, β-, γ-HBCDD (lb) | HBCDD isomers | | | Total HBCDD (using GC-methods) |
|------------------------|----------|---------|---|--------------------------------|--------------------------------|--|---|---|-----------------------------------|
| | | | | | | (+/-)-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 | |
| 68 | A | 2502-HY | | 1.31 | 1.31 | 0.101 | 0.01 | 1.2 | |
| 69 | | 2502-HY | | | | | | | |
| 70 | | 2502-HY | | | | | | | |
| 71 | | 2502-HY | | | | | | | |
| 72 | | 2502-HY | | | | | | | |
| 73 | | 2502-HY | | | | | | | |
| 74 | | 2502-HY | | | | | | | |
| 75 | | 2502-HY | | | | | | | |
| 76 | | 2502-HY | | | | | | | |
| 77 | | 2502-HY | | | | | | | |
| 78 | A | 2502-HY | | 0.98 | 0.95 | 0.11 | <0.03 | 0.85 | |
| 79 | | 2502-HY | | | | | | | |
| 80 | | 2502-HY | | | | | | | |
| 81 | | 2502-HY | | | | | | | |
| 82 | | 2502-HY | | | | | | | |
| 83 | | 2502-HY | | | | | | | |
| 84 | | 2502-HY | | | | | | | |
| 85 | | 2502-HY | | | | | | | |
| 86 | A | 2502-HY | | 0.61 | 0.56 | 0.1 | <0.05 | 0.46 | |
| 87 | | 2502-HY | | | | | | | |
| 88 | | 2502-HY | | | | | | | |
| 89 | A | 2502-HY | | 2.1 | 2.1 | 0.53 | 0.37 | 1.19 | |
| 90 | | 2502-HY | | | | | | | |
| 91 | | 2502-HY | | | | | | | |
| 92 | | 2502-HY | | | | | | | |
| 93 | | 2502-HY | | | | | | | |
| 94 | | 2502-HY | | | | | | | |
| 95 | A | 2502-HY | | 1.01 | 0.989 | 0.107 | <0.02 | 0.882 | |
| 96 | | 2502-HY | | | | | | | |
| 97 | | 2502-HY | | | | | | | |
| Additional Sets | | | | | | | | | |
| 86 | B | 2502-HY | | 2.63 | 2.58 | 0.1 | <0.05 | 2.48 | |

Hay (2502-HY)
 Moisture content (HBCDD) - Results

| LC | Data set | Sample | Result % | Moisture content HBCDD |
|----|----------|---------|----------|------------------------|
| 1 | A | 2502-HY | | 7.9 |
| 2 | | 2502-HY | | |
| 3 | | 2502-HY | | |
| 4 | | 2502-HY | | |
| 5 | | 2502-HY | | |
| 6 | | 2502-HY | | |
| 7 | | 2502-HY | | |
| 8 | | 2502-HY | | |
| 9 | A | 2502-HY | | 91.6 |
| 10 | | 2502-HY | | |
| 11 | A | 2502-HY | | 91.3 |
| 12 | | 2502-HY | | |
| 13 | | 2502-HY | | |
| 14 | | 2502-HY | | |
| 15 | A | 2502-HY | | |
| 16 | A | 2502-HY | | 8.28 |
| 17 | | 2502-HY | | |
| 18 | A | 2502-HY | | 8 |
| 19 | | 2502-HY | | |
| 20 | A | 2502-HY | | 8.47 |
| | | 2502-HY | | |
| 22 | | 2502-HY | | |
| 23 | | 2502-HY | | |
| 24 | | 2502-HY | | |
| 25 | | 2502-HY | | |
| 26 | A | 2502-HY | | 7.9 |
| 27 | A | 2502-HY | | 8.21 |
| 28 | | 2502-HY | | |
| 29 | | 2502-HY | | |
| 30 | | 2502-HY | | |
| 31 | | 2502-HY | | |
| 32 | | 2502-HY | | |
| 33 | | 2502-HY | | |
| 34 | | 2502-HY | | |
| 35 | | 2502-HY | | |
| 36 | A | 2502-HY | | 7.7 |
| 37 | | 2502-HY | | |
| 38 | | 2502-HY | | |
| 39 | A | 2502-HY | | 8.41 |
| 40 | | 2502-HY | | |
| 41 | | 2502-HY | | |
| 42 | | 2502-HY | | |
| 43 | | 2502-HY | | |
| 44 | A | 2502-HY | | 8.3 |
| 45 | | 2502-HY | | |
| 46 | | 2502-HY | | |
| 47 | | 2502-HY | | |
| 48 | | 2502-HY | | |
| 49 | | 2502-HY | | |
| 50 | | 2502-HY | | |
| 51 | | 2502-HY | | |
| 52 | | 2502-HY | | |
| 53 | | 2502-HY | | |
| 54 | | 2502-HY | | |

Hay (2502-HY)
 Moisture content (HBCDD) - Results

| LC | Data set | Sample | Result % | Moisture content HBCDD |
|------------------------|----------|---------|----------|------------------------|
| 55 | | 2502-HY | | |
| 56 | | 2502-HY | | |
| 57 | | 2502-HY | | |
| 58 | | 2502-HY | | |
| 59 | | 2502-HY | | |
| 60 | | 2502-HY | | |
| 61 | | 2502-HY | | |
| 62 | | 2502-HY | | |
| 63 | | 2502-HY | | |
| 64 | | 2502-HY | | |
| 65 | | 2502-HY | | |
| 66 | | 2502-HY | | |
| 67 | | 2502-HY | | |
| 68 | A | 2502-HY | | 8.25 |
| 69 | | 2502-HY | | |
| 70 | | 2502-HY | | |
| 71 | | 2502-HY | | |
| 72 | | 2502-HY | | |
| 73 | | 2502-HY | | |
| 74 | | 2502-HY | | |
| 75 | | 2502-HY | | |
| 76 | | 2502-HY | | |
| 77 | | 2502-HY | | |
| 78 | A | 2502-HY | | 8.63 |
| 79 | | 2502-HY | | |
| 80 | | 2502-HY | | |
| 81 | | 2502-HY | | |
| 82 | | 2502-HY | | |
| 83 | | 2502-HY | | |
| 84 | | 2502-HY | | |
| 85 | | 2502-HY | | |
| 86 | A | 2502-HY | | 8 |
| 87 | | 2502-HY | | |
| 88 | | 2502-HY | | |
| 89 | A | 2502-HY | | 7.52 |
| 90 | | 2502-HY | | |
| 91 | | 2502-HY | | |
| 92 | | 2502-HY | | |
| 93 | | 2502-HY | | |
| 94 | | 2502-HY | | |
| 95 | A | 2502-HY | | 90.2 |
| 96 | | 2502-HY | | |
| 97 | | 2502-HY | | |
| Additional Sets | | | | |
| 86 | B | 2502-HY | | 8 |



EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Hay 2025 [EURL-PT-POP_2502-HY]

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

21 May 2026

Annex 9: Participants' z-scores of HBCDDs - Tables

Test sample - Hay (2502-HY)

Z-scores of sum parameters and individual results

Calculation of z-score on basis of assigned value

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

x_a : assigned value

x : participant's result

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

20%: Evaluated individual HBCDD diastereomers and sum parameters

Hay (2502-HY)
 HBCDD - Z-scores

| LC | Data set | Sample | Z-score [σ _p = 20 %] | Sum of α-, β-, γ-HBCDD (ub) | Sum of α-, β-, γ-HBCDD (lb) | (+/-)-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 | Total HBCDD* (using GC-methods) |
|----|----------|---------|------------------------------------|--------------------------------|--------------------------------|--|---|---|------------------------------------|
| 1 | A | 2502-HY | | -0.7 | -0.7 | -1.3 | | -0.8 | |
| 2 | | 2502-HY | | | | | | | |
| 3 | | 2502-HY | | | | | | | |
| 4 | | 2502-HY | | | | | | | |
| 5 | | 2502-HY | | | | | | | |
| 6 | | 2502-HY | | | | | | | |
| 7 | | 2502-HY | | | | | | | |
| 8 | | 2502-HY | | | | | | | |
| 9 | A | 2502-HY | | -0.3 | -0.4 | -0.6 | | -0.4 | |
| 10 | | 2502-HY | | | | | | | |
| 11 | A | 2502-HY | | | | | | | |
| 12 | | 2502-HY | | | | | | | |
| 13 | | 2502-HY | | | | | | | |
| 14 | | 2502-HY | | | | | | | |
| 15 | A | 2502-HY | | -1.2 | -1.3 | -1.4 | | -1.3 | |
| 16 | A | 2502-HY | | 1.2 | 1.2 | 0.2 | | 1.2 | |
| 17 | | 2502-HY | | | | | | | |
| 18 | A | 2502-HY | | 1.3 | 1.4 | 1.1 | | 1.2 | |
| 19 | | 2502-HY | | | | | | | |
| 20 | A | 2502-HY | | 1.5 | 1.5 | 0.6 | | 1.5 | |
| 21 | | 2502-HY | | | | | | | |
| 22 | | 2502-HY | | | | | | | |
| 23 | | 2502-HY | | | | | | | |
| 24 | | 2502-HY | | | | | | | |
| 25 | | 2502-HY | | | | | | | |
| 26 | A | 2502-HY | | -0.3 | -0.2 | 0.3 | | -0.4 | |
| 27 | A | 2502-HY | | -0.7 | -0.9 | -0.1 | | -1.1 | |
| 28 | | 2502-HY | | | | | | | |
| 29 | | 2502-HY | | | | | | | |
| 30 | | 2502-HY | | | | | | | |
| 31 | | 2502-HY | | | | | | | |
| 32 | | 2502-HY | | | | | | | |
| 33 | | 2502-HY | | | | | | | |
| 34 | | 2502-HY | | | | | | | |
| 35 | | 2502-HY | | | | | | | |
| 36 | A | 2502-HY | | 10.2 | 9.1 | | | 10.5 | |
| 37 | | 2502-HY | | | | | | | |
| 38 | | 2502-HY | | | | | | | |
| 39 | A | 2502-HY | | 0.3 | 0.3 | 1.5 | | 0.1 | |
| 40 | | 2502-HY | | | | | | | |
| 41 | | 2502-HY | | | | | | | |
| 42 | | 2502-HY | | | | | | | |
| 43 | | 2502-HY | | | | | | | |
| 44 | A | 2502-HY | | -0.2 | -0.1 | -0.1 | | -0.3 | |
| 45 | | 2502-HY | | | | | | | |
| 46 | | 2502-HY | | | | | | | |
| 47 | | 2502-HY | | | | | | | |
| 48 | | 2502-HY | | | | | | | |
| 49 | | 2502-HY | | | | | | | |
| 50 | | 2502-HY | | | | | | | |
| 51 | | 2502-HY | | | | | | | |
| 52 | | 2502-HY | | | | | | | |
| 53 | | 2502-HY | | | | | | | |
| 54 | | 2502-HY | | | | | | | |
| 55 | | 2502-HY | | | | | | | |
| 56 | | 2502-HY | | | | | | | |
| 57 | | 2502-HY | | | | | | | |

Hay (2502-HY)

HBCDD - Z-scores

| LC | Data set | Sample | Z-score [σ _p = 20 %] | Sum of α-, β-, γ-HBCDD (ub) | Sum of α-, β-, γ-HBCDD (lb) | (+/-)-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 | Total HBCDD* (using GC-methods) |
|------------------------|----------|---------|------------------------------------|--------------------------------|--------------------------------|--|---|---|------------------------------------|
| 58 | | 2502-HY | | | | | | | |
| 59 | | 2502-HY | | | | | | | |
| 60 | | 2502-HY | | | | | | | |
| 61 | | 2502-HY | | | | | | | |
| 62 | | 2502-HY | | | | | | | |
| 63 | | 2502-HY | | | | | | | |
| 64 | | 2502-HY | | | | | | | |
| 65 | | 2502-HY | | | | | | | |
| 66 | | 2502-HY | | | | | | | |
| 67 | | 2502-HY | | | | | | | |
| 68 | A | 2502-HY | | 1.1 | 1.1 | -0.3 | | 1.2 | |
| 69 | | 2502-HY | | | | | | | |
| 70 | | 2502-HY | | | | | | | |
| 71 | | 2502-HY | | | | | | | |
| 72 | | 2502-HY | | | | | | | |
| 73 | | 2502-HY | | | | | | | |
| 74 | | 2502-HY | | | | | | | |
| 75 | | 2502-HY | | | | | | | |
| 76 | | 2502-HY | | | | | | | |
| 77 | | 2502-HY | | | | | | | |
| 78 | A | 2502-HY | | -0.5 | -0.6 | 0.1 | | -0.6 | |
| 79 | | 2502-HY | | | | | | | |
| 80 | | 2502-HY | | | | | | | |
| 81 | | 2502-HY | | | | | | | |
| 82 | | 2502-HY | | | | | | | |
| 83 | | 2502-HY | | | | | | | |
| 84 | | 2502-HY | | | | | | | |
| 85 | | 2502-HY | | | | | | | |
| 86 | A | 2502-HY | | -2.2 | -2.4 | -0.3 | | -2.6 | |
| 87 | | 2502-HY | | | | | | | |
| 88 | | 2502-HY | | | | | | | |
| 89 | A | 2502-HY | | 4.7 | 4.8 | 19.8 | | 1.1 | |
| 90 | | 2502-HY | | | | | | | |
| 91 | | 2502-HY | | | | | | | |
| 92 | | 2502-HY | | | | | | | |
| 93 | | 2502-HY | | | | | | | |
| 94 | | 2502-HY | | | | | | | |
| 95 | A | 2502-HY | | -0.3 | -0.4 | 0.0 | | -0.4 | |
| 96 | | 2502-HY | | | | | | | |
| 97 | | 2502-HY | | | | | | | |
| Additional Sets | | | | | | | | | |
| 86 | B | 2502-HY | | 7.2 | 7.1 | -0.3 | | 7.8 | |

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs
 EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Fo

Hay (2502-HY)
 Moisture content (HBCDD) - Z-scores

| LC | Data set | Sample | Z-score [$\sigma_p = 10\%$] | Moisture content PBDE |
|----|----------|---------|----------------------------------|--------------------------|
| 1 | A | 2502-HY | | -0.5 |
| 2 | | 2502-HY | | |
| 3 | | 2502-HY | | |
| 4 | | 2502-HY | | |
| 5 | | 2502-HY | | |
| 6 | | 2502-HY | | |
| 7 | | 2502-HY | | |
| 8 | | 2502-HY | | |
| 9 | A | 2502-HY | | 99.8 |
| 10 | | 2502-HY | | |
| 11 | A | 2502-HY | | 99.5 |
| 12 | | 2502-HY | | |
| 13 | | 2502-HY | | |
| 14 | | 2502-HY | | |
| 15 | A | 2502-HY | | |
| 16 | A | 2502-HY | | -0.1 |
| 17 | | 2502-HY | | |
| 18 | A | 2502-HY | | -0.4 |
| 19 | | 2502-HY | | |
| 20 | A | 2502-HY | | 0.2 |
| 21 | | 2502-HY | | |
| 22 | | 2502-HY | | |
| 23 | | 2502-HY | | |
| 24 | | 2502-HY | | |
| 25 | | 2502-HY | | |
| 26 | A | 2502-HY | | -0.5 |
| 27 | A | 2502-HY | | -0.2 |
| 28 | | 2502-HY | | |
| 29 | | 2502-HY | | |
| 30 | | 2502-HY | | |
| 31 | | 2502-HY | | |
| 32 | | 2502-HY | | |
| 33 | | 2502-HY | | |
| 34 | | 2502-HY | | |
| 35 | | 2502-HY | | |
| 36 | A | 2502-HY | | -0.8 |
| 37 | | 2502-HY | | |
| 38 | | 2502-HY | | |
| 39 | A | 2502-HY | | 0.1 |
| 40 | | 2502-HY | | |
| 41 | | 2502-HY | | |
| 42 | | 2502-HY | | |
| 43 | | 2502-HY | | |
| 44 | A | 2502-HY | | 0.0 |
| 45 | | 2502-HY | | |
| 46 | | 2502-HY | | |
| 47 | | 2502-HY | | |
| 48 | | 2502-HY | | |
| 49 | | 2502-HY | | |
| 50 | | 2502-HY | | |
| 51 | | 2502-HY | | |
| 52 | | 2502-HY | | |
| 53 | | 2502-HY | | |
| 54 | | 2502-HY | | |
| 55 | | 2502-HY | | |
| 56 | | 2502-HY | | |
| 57 | | 2502-HY | | |

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs
 EURL for halogenated Persistent Organic Pollutants (POPs) in Feed and Fo

Hay (2502-HY)
 Moisture content (HBCDD) - Z-scores

| LC | Data set | Sample | Z-score [$\sigma_p = 10\%$] | Moisture content PBDE |
|------------------------|----------|---------|----------------------------------|--------------------------|
| 58 | | 2502-HY | | |
| 59 | | 2502-HY | | |
| 60 | | 2502-HY | | |
| 61 | | 2502-HY | | |
| 62 | | 2502-HY | | |
| 63 | | 2502-HY | | |
| 64 | | 2502-HY | | |
| 65 | | 2502-HY | | |
| 66 | | 2502-HY | | |
| 67 | | 2502-HY | | |
| 68 | A | 2502-HY | | -0.1 |
| 69 | | 2502-HY | | |
| 70 | | 2502-HY | | |
| 71 | | 2502-HY | | |
| 72 | | 2502-HY | | |
| 73 | | 2502-HY | | |
| 74 | | 2502-HY | | |
| 75 | | 2502-HY | | |
| 76 | | 2502-HY | | |
| 77 | | 2502-HY | | |
| 78 | A | 2502-HY | | 0.3 |
| 79 | | 2502-HY | | |
| 80 | | 2502-HY | | |
| 81 | | 2502-HY | | |
| 82 | | 2502-HY | | |
| 83 | | 2502-HY | | |
| 84 | | 2502-HY | | |
| 85 | | 2502-HY | | |
| 86 | A | 2502-HY | | -0.4 |
| 87 | | 2502-HY | | |
| 88 | | 2502-HY | | |
| 89 | A | 2502-HY | | -1.0 |
| 90 | | 2502-HY | | |
| 91 | | 2502-HY | | |
| 92 | | 2502-HY | | |
| 93 | | 2502-HY | | |
| 94 | | 2502-HY | | |
| 95 | A | 2502-HY | | 98.2 |
| 96 | | 2502-HY | | |
| 97 | | 2502-HY | | |
| Additional Sets | | | | |
| 86 | B | 2502-HY | | -0.4 |



EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Hay 2025 [EURL-PT-POP_2502-HY]

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Annex 10: Participants' z-scores of HBCDDs - Charts

Test sample - Hay (2502-HY)

Z-scores of sum parameters and individual results

Calculation of z-score on basis of assigned value


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


x_a : assigned value

x : participant's result

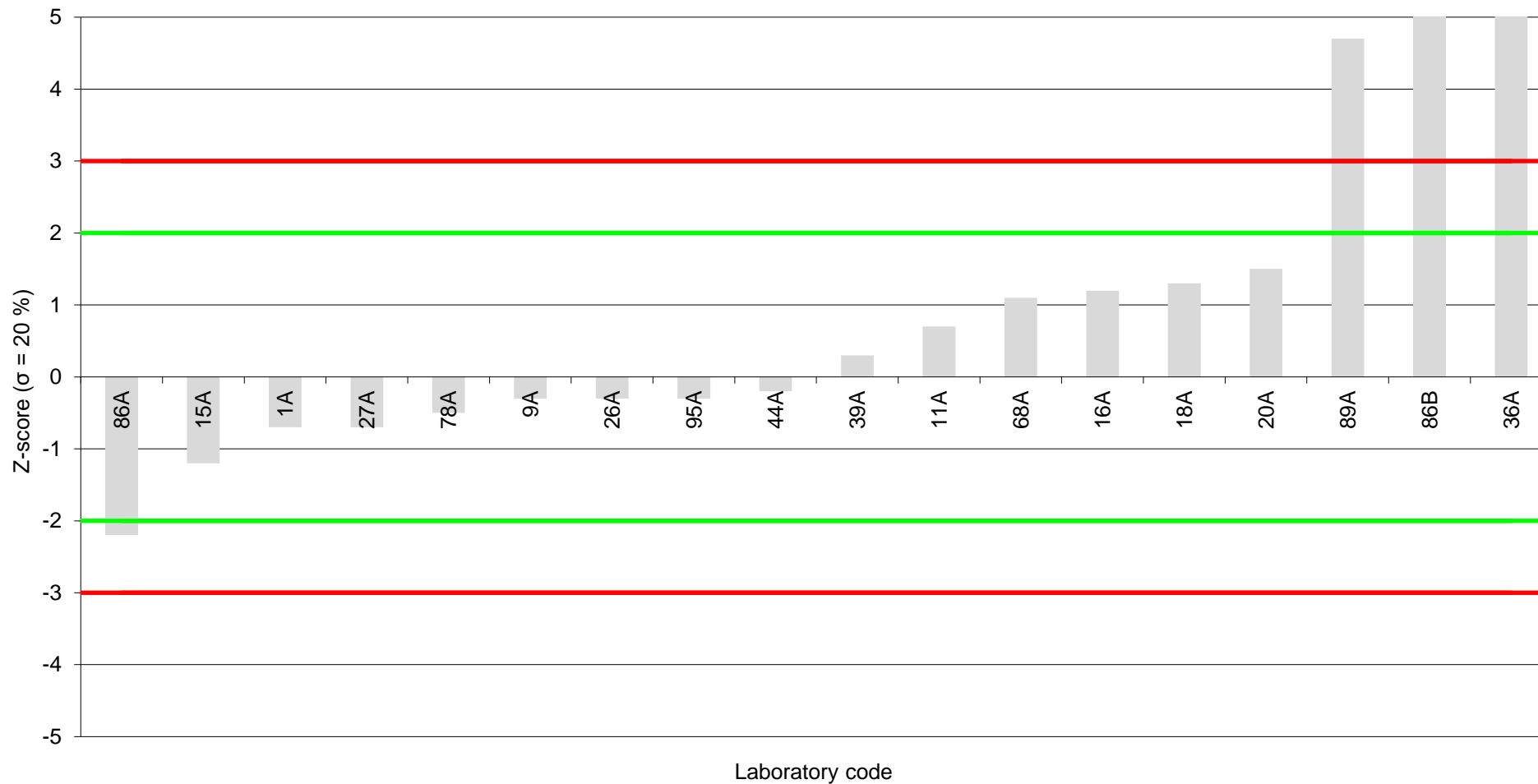
σ_{prel} : fitness-for-purpose-based standard deviation for proficiency assessment

20%: Evaluated individual HBCDD diastereomers and sum parameters

± 2 z-scores: 

± 3 z-scores: 

Hay (2502-HY)
Sum of α -, β -, γ -HBCDD ub
Assigned value: 1.08 $\mu\text{g}/\text{kg}$ product (12 % moisture content)



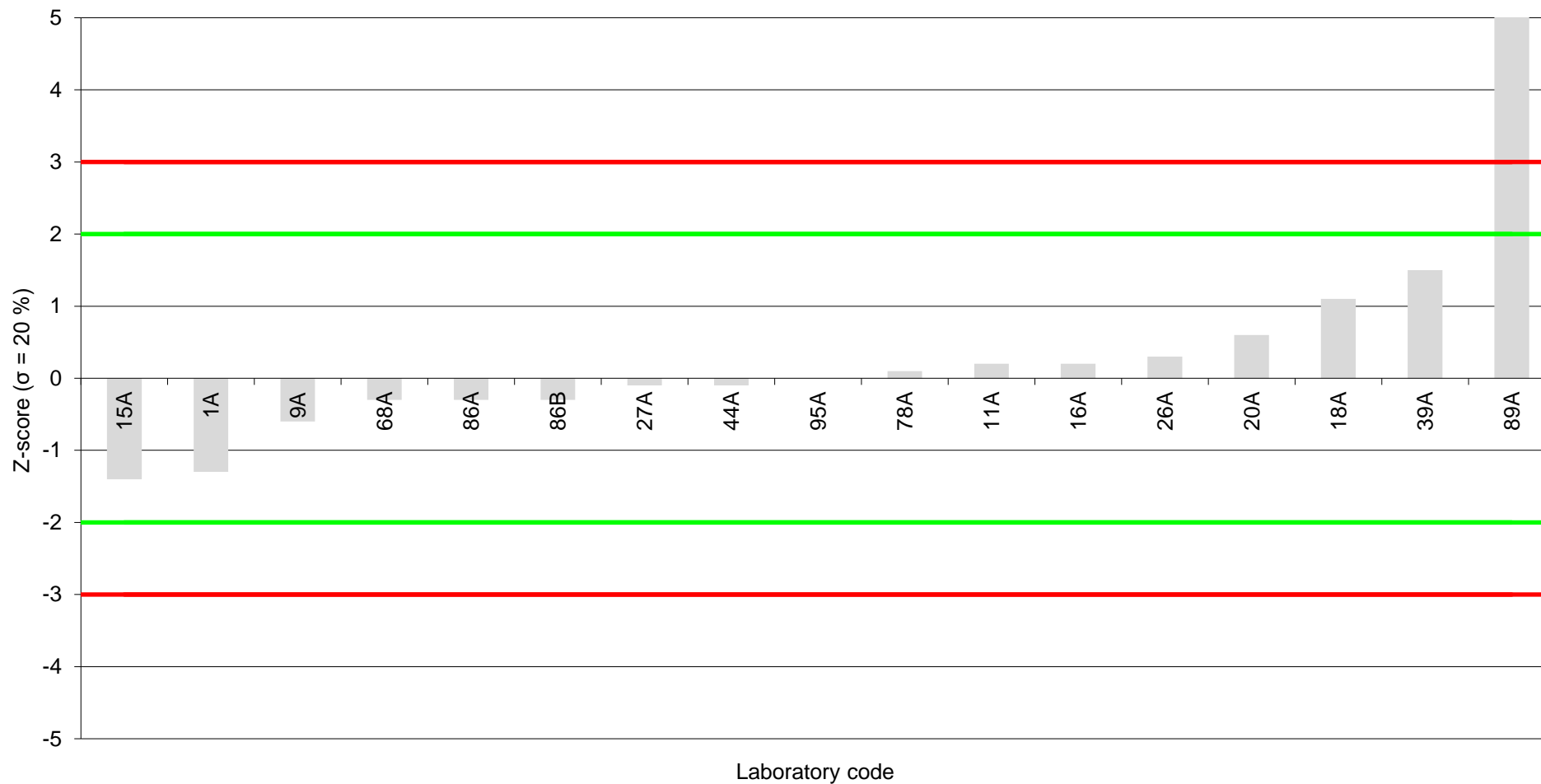
Hay (2502-HY)
Sum of α -, β -, γ -HBCDD lb
Assigned value: 1.07 $\mu\text{g}/\text{kg}$ product (12 % moisture content)



Hay (2502-HY)

(+/-)- α -HBCDD

Assigned value: 0.107 $\mu\text{g}/\text{kg}$ product (12 % moisture content)



Hay (2502-HY)

(+/-)- γ -HBCDD

Assigned value: 0.969 $\mu\text{g}/\text{kg}$ product (12 % moisture content)





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Annex 11: Participants' methods for HBCDDs

Test sample - Hay (2502-HY)

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Hay (2502-HY)

Methods HBCDDs - Accreditation, weighed sample, standards

| LC | Data set | Sample | Accreditation according to ISO/IEC 17025 | Weighed sample [g] | Use of isotope-labelled internal standards HBCDD diastereomers (yes/no) |
|----|----------|---------|--|--------------------|---|
| 1 | A | 2502-HY | yes | 9 | yes |
| 2 | | 2502-HY | | | |
| 3 | | 2502-HY | | | |
| 4 | | 2502-HY | | | |
| 5 | | 2502-HY | | | |
| 6 | | 2502-HY | | | |
| 7 | | 2502-HY | | | |
| 8 | | 2502-HY | | | |
| 9 | A | 2502-HY | | 5.18 | |
| 10 | | 2502-HY | | | |
| 11 | A | 2502-HY | yes | 10 | yes |
| 12 | | 2502-HY | | | |
| 13 | | 2502-HY | | | |
| 14 | | 2502-HY | | | |
| 15 | | 2502-HY | | | |
| 16 | A | 2502-HY | no | 10 | yes |
| 17 | | 2502-HY | | | |
| 18 | A | 2502-HY | no | 10 | yes |
| 19 | | 2502-HY | | | |
| 20 | A | 2502-HY | yes | 10 | yes |
| 21 | | 2502-HY | | | |
| 22 | | 2502-HY | | | |
| 23 | | 2502-HY | | | |
| 24 | | 2502-HY | | | |
| 25 | | 2502-HY | | | |
| 26 | A | 2502-HY | yes | 3 | yes |
| 27 | A | 2502-HY | yes | 5 | yes |
| 28 | | 2502-HY | | | |
| 29 | | 2502-HY | | | |
| 30 | | 2502-HY | | | |
| 31 | | 2502-HY | | | |
| 32 | | 2502-HY | | | |
| 33 | | 2502-HY | | | |
| 34 | | 2502-HY | | | |
| 35 | | 2502-HY | | | |
| 36 | A | 2502-HY | no | 2 | yes |
| 37 | | 2502-HY | | | |
| 38 | | 2502-HY | | | |
| 39 | A | 2502-HY | no | 25.8739 | yes |
| 40 | | 2502-HY | | | |
| 41 | | 2502-HY | | | |
| 42 | | 2502-HY | | | |
| 43 | | 2502-HY | | | |
| 44 | A | 2502-HY | no | 10 | yes |
| 45 | | 2502-HY | | | |
| 46 | | 2502-HY | | | |
| 47 | | 2502-HY | | | |
| 48 | | 2502-HY | | | |
| 49 | | 2502-HY | | | |
| 50 | | 2502-HY | | | |
| 51 | | 2502-HY | | | |
| 52 | | 2502-HY | | | |
| 53 | | 2502-HY | | | |
| 54 | | 2502-HY | | | |
| 55 | | 2502-HY | | | |
| 56 | | 2502-HY | | | |
| 57 | | 2502-HY | | | |

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Hay (2502-HY)

Methods HBCDDs - Accreditation, weighed sample, standards

| LC | Data set | Sample | Accreditation according to ISO/IEC 17025 | Weighed sample [g] | Use of isotope-labelled internal standards HBCDD diastereomers (yes/no) |
|------------------------|----------|---------|--|--------------------|---|
| 58 | | 2502-HY | | | |
| 59 | | 2502-HY | | | |
| 60 | | 2502-HY | | | |
| 61 | | 2502-HY | | | |
| 62 | | 2502-HY | | | |
| 63 | | 2502-HY | | | |
| 64 | | 2502-HY | | | |
| 65 | | 2502-HY | | | |
| 66 | | 2502-HY | | | |
| 67 | | 2502-HY | | | |
| 68 | A | 2502-HY | yes | 10 | yes |
| 69 | | 2502-HY | | | |
| 70 | | 2502-HY | | | |
| 71 | | 2502-HY | | | |
| 72 | | 2502-HY | | | |
| 73 | | 2502-HY | | | |
| 74 | | 2502-HY | | | |
| 75 | | 2502-HY | | | |
| 76 | | 2502-HY | | | |
| 77 | | 2502-HY | | | |
| 78 | A | 2502-HY | no | 9 | yes |
| 79 | | 2502-HY | | | |
| 80 | | 2502-HY | | | |
| 81 | | 2502-HY | | | |
| 82 | | 2502-HY | | | |
| 83 | | 2502-HY | | | |
| 84 | | 2502-HY | | | |
| 85 | | 2502-HY | | | |
| 86 | A | 2502-HY | yes | 5 | yes |
| 87 | | 2502-HY | | | |
| 88 | | 2502-HY | | | |
| 89 | A | 2502-HY | no | 2 | yes |
| 90 | | 2502-HY | | | |
| 91 | | 2502-HY | | | |
| 92 | | 2502-HY | | | |
| 93 | | 2502-HY | | | |
| 94 | | 2502-HY | | | |
| 95 | A | 2502-HY | yes | 5.03 | |
| 96 | | 2502-HY | | | |
| 97 | | 2502-HY | | | |
| Additional Sets | | | | | |
| 86 | B | 2502-HY | no | 5 | yes |

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Hay (2502-HY)

Methods HBCDDs - Internal and recovery standards

| LC | Data set | Sample | (+/-)- α -HBCDD Internal standard | (+/-)- β -HBCDD Internal standard | (+/-)- γ -HBCDD Internal standard | (+/-)- α -HBCDD Recovery standard | (+/-)- β -HBCDD Recovery standard | (+/-)- γ -HBCDD Recovery standard |
|----|----------|---------|---|--|---|---|--|---|
| 1 | A | 2502-HY | 13C-alpha-HBCDD | 13C-beta-HBCDD | 13C-gamma-HBCDD | D18-beta-HBCDD | D18-beta-HBCDD | D18-beta-HBCDD |
| 2 | | 2502-HY | | | | | | |
| 3 | | 2502-HY | | | | | | |
| 4 | | 2502-HY | | | | | | |
| 5 | | 2502-HY | | | | | | |
| 6 | | 2502-HY | | | | | | |
| 7 | | 2502-HY | | | | | | |
| 8 | | 2502-HY | | | | | | |
| 9 | A | 2502-HY | 13C12-alpha-HBCD | 13C12-beta-HBCD | 13C12-gamma-HBCD | d18-beta-HBCD | d18-beta-HBCD | d18-beta-HBCD |
| 10 | | 2502-HY | | | | | | |
| 11 | A | 2502-HY | | | | | | |
| 12 | | 2502-HY | | | | | | |
| 13 | | 2502-HY | | | | | | |
| 14 | | 2502-HY | | | | | | |
| 15 | | 2502-HY | 13C12- α -HBCDD | 13C12- β -HBCDD | 13C12- γ -HBCDD | NA | NA | NA |
| 16 | A | 2502-HY | α -HBCD | β -HBCD | γ -HBCD | D18- β -HBCD | D18- β -HBCD | D18- β -HBCD |
| 17 | | 2502-HY | | | | | | |
| 18 | A | 2502-HY | 13C α -HBCDD | 13C β -HBCDD | 13C γ -HBCDD | | | |
| 19 | | 2502-HY | | | | | | |
| 20 | A | 2502-HY | 13C-A-HBCD | 13C-B-HBCD | 13C-Y-HBCD | RS-D18-B-HBCD | RS-D18-B-HBCD | RS-D18-B-HBCD |
| 21 | | 2502-HY | | | | | | |
| 22 | | 2502-HY | | | | | | |
| 23 | | 2502-HY | | | | | | |
| 24 | | 2502-HY | | | | | | |
| 25 | | 2502-HY | | | | | | |
| 26 | A | 2502-HY | 13C- α -HBCDD | 13C- β -HBCDD | 13C- γ -HBCDD | d18- β -HBCDD | d18- β -HBCDD | d18- β -HBCDD |
| 27 | A | 2502-HY | (13C12)- α -HBCDD | (13C12)- β -HBCDD | (13C12)- γ -HBCDD | | | |
| 28 | | 2502-HY | | | | | | |
| 29 | | 2502-HY | | | | | | |
| 30 | | 2502-HY | | | | | | |
| 31 | | 2502-HY | | | | | | |
| 32 | | 2502-HY | | | | | | |
| 33 | | 2502-HY | | | | | | |
| 34 | | 2502-HY | | | | | | |
| 35 | | 2502-HY | | | | | | |
| 36 | A | 2502-HY | alpha-HBCDD (13C) | beta-HBCDD (13C) | gamma-HBCDD (13C) | | D18-beta-HBCDD | |
| 37 | | 2502-HY | | | | | | |
| 38 | | 2502-HY | | | | | | |
| 39 | A | 2502-HY | 13C- α -HBCD | 13C- β -HBCD | 13C- γ -HBCD | 13C- δ -HBCD | 13C- δ -HBCD | 13C- δ -HBCD |
| 40 | | 2502-HY | | | | | | |
| 41 | | 2502-HY | | | | | | |
| 42 | | 2502-HY | | | | | | |
| 43 | | 2502-HY | | | | | | |
| 44 | A | 2502-HY | 13C12-alpha-HBCDD | 13C12-beta-HBCDD | 13C12-gamma-HBCDD | alpha-HBCDD-d18 | beta-HBCDD-d18 | gamma-HBCDD-d18 |
| 45 | | 2502-HY | | | | | | |
| 46 | | 2502-HY | | | | | | |
| 47 | | 2502-HY | | | | | | |
| 48 | | 2502-HY | | | | | | |
| 49 | | 2502-HY | | | | | | |
| 50 | | 2502-HY | | | | | | |
| 51 | | 2502-HY | | | | | | |
| 52 | | 2502-HY | | | | | | |
| 53 | | 2502-HY | | | | | | |
| 54 | | 2502-HY | | | | | | |
| 55 | | 2502-HY | | | | | | |
| 56 | | 2502-HY | | | | | | |
| 57 | | 2502-HY | | | | | | |
| 58 | | 2502-HY | | | | | | |

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Hay (2502-HY)

Methods HBCDDs - Internal and recovery standards

| LC | Data set | Sample | (+/-)- α -HBCDD Internal standard | (+/-)- β -HBCDD Internal standard | (+/-)- γ -HBCDD Internal standard | (+/-)- α -HBCDD Recovery standard | (+/-)- β -HBCDD Recovery standard | (+/-)- γ -HBCDD Recovery standard |
|------------------------|----------|---------|---|--|---|---|---|---|
| 59 | | 2502-HY | | | | | | |
| 60 | | 2502-HY | | | | | | |
| 61 | | 2502-HY | | | | | | |
| 62 | | 2502-HY | | | | | | |
| 63 | | 2502-HY | | | | | | |
| 64 | | 2502-HY | | | | | | |
| 65 | | 2502-HY | | | | | | |
| 66 | | 2502-HY | | | | | | |
| 67 | | 2502-HY | | | | | | |
| 68 | A | 2502-HY | 13Calfa | 13Cbeta | 13Cgama | dbhbcdd | dbhbcdd | dbhbcdd |
| 69 | | 2502-HY | | | | | | |
| 70 | | 2502-HY | | | | | | |
| 71 | | 2502-HY | | | | | | |
| 72 | | 2502-HY | | | | | | |
| 73 | | 2502-HY | | | | | | |
| 74 | | 2502-HY | | | | | | |
| 75 | | 2502-HY | | | | | | |
| 76 | | 2502-HY | | | | | | |
| 77 | | 2502-HY | | | | | | |
| 78 | A | 2502-HY | 13C alphaHBCDD | 13C betaHBCDD | 13C gammaHBCDD | d18-racbeta-1,2,5,6,9,10 - Hexabromocyclododecane | d18-racbeta-1,2,5,6,9,10 - Hexabromocyclododecane | d18-racbeta-1,2,5,6,9,10 - Hexabromocyclododecane |
| 79 | | 2502-HY | | | | | | |
| 80 | | 2502-HY | | | | | | |
| 81 | | 2502-HY | | | | | | |
| 82 | | 2502-HY | | | | | | |
| 83 | | 2502-HY | | | | | | |
| 84 | | 2502-HY | | | | | | |
| 85 | | 2502-HY | | | | | | |
| 86 | A | 2502-HY | α -HBCD-13C12 | γ -HBCD-13C12 | γ -HBCD-13C12 | β -HBCD-13C12 | β -HBCD-13C12 | β -HBCD-13C12 |
| 87 | | 2502-HY | | | | | | |
| 88 | | 2502-HY | | | | | | |
| 89 | A | 2502-HY | alpha HBCD 13C12 | alpha HBCD 13C12 | gamma HBCD D18 | no | no | no |
| 90 | | 2502-HY | | | | | | |
| 91 | | 2502-HY | | | | | | |
| 92 | | 2502-HY | | | | | | |
| 93 | | 2502-HY | | | | | | |
| 94 | | 2502-HY | | | | | | |
| 95 | A | 2502-HY | 13C12-alpha-HBCD | 13C12-beta-HBCD | 13C12-gamma-HBCD | d18-gamma-HBCD | d18-gamma-HBCD | d18-gamma-HBCD |
| 96 | | 2502-HY | | | | | | |
| 97 | | 2502-HY | | | | | | |
| Additional Sets | | | | | | | | |
| 86 | B | 2502-HY | α -HBCDD-13C12 | γ -HBCDD-13C12 | γ -HBCDD-13C12 | β -HBCD-13C12 | β -HBCD-13C12 | β -HBCD-13C12 |

Hay (2502-HY)

Methods HBCDDs - Extraction

| LC | Data set | Sample | Extraction | | | | | |
|----|----------|---------|----------------------------------|-------------------------------|---|---------------------|-----------------------------|---------------------------|
| | | | Sample preparation/pre-treatment | Extraction technique | Extraction solvent | Extraction time [h] | Extraction temperature [°C] | Extraction pressure [MPa] |
| 1 | A | 2502-HY | | Twisselmann | Ethanol / Toluene (70:30, v/v) | 6 h | boiling point | |
| 2 | | 2502-HY | | | | | | |
| 3 | | 2502-HY | | | | | | |
| 4 | | 2502-HY | | | | | | |
| 5 | | 2502-HY | | | | | | |
| 6 | | 2502-HY | | | | | | |
| 7 | | 2502-HY | | | | | | |
| 8 | | 2502-HY | | | | | | |
| 9 | A | 2502-HY | | QuECHERS | | 1 | | |
| 10 | | 2502-HY | | | | | | |
| 11 | A | 2502-HY | | Soxhlet | toluene/acetone 7/3 | 6 | | |
| 12 | | 2502-HY | | | | | | |
| 13 | | 2502-HY | | | | | | |
| 14 | | 2502-HY | | | | | | |
| 15 | | 2502-HY | | | | | | |
| 16 | A | 2502-HY | | Pressurized liquid extraction | Hexane/Acetone (70:30) | 1 | 125 | 10.3 |
| 17 | | 2502-HY | | | | | | |
| 18 | A | 2502-HY | | Pressurized liquid extraction | toluene:ethanol | 0,25 | 120 | 100 |
| 19 | | 2502-HY | | | | | | |
| 20 | A | 2502-HY | | ASE | Acetone/Hexane 50/50 | 0.5 | 100 | 10.3 |
| 21 | | 2502-HY | | | | | | |
| 22 | | 2502-HY | | | | | | |
| 23 | | 2502-HY | | | | | | |
| 24 | | 2502-HY | | | | | | |
| 25 | | 2502-HY | | | | | | |
| 26 | A | 2502-HY | | Pressurized liquid extraction | Toluene / Acetone (70 / 30) | 1 | 120 | 10 |
| 27 | A | 2502-HY | | Cold extraction | shaking with dichlormethane : acetone 2:1 | 1.5 | ambient | ambient |
| 28 | | 2502-HY | | | | | | |
| 29 | | 2502-HY | | | | | | |
| 30 | | 2502-HY | | | | | | |
| 31 | | 2502-HY | | | | | | |
| 32 | | 2502-HY | | | | | | |
| 33 | | 2502-HY | | | | | | |
| 34 | | 2502-HY | | | | | | |
| 35 | | 2502-HY | | | | | | |
| 36 | A | 2502-HY | | Mechanical shaking | Hexane/Dichloromethane (50/50) | 0.33 | 20 | Ambiant |
| 37 | | 2502-HY | | | | | | |
| 38 | | 2502-HY | | | | | | |
| 39 | A | 2502-HY | | Soxhlet | toluene/ethanol 50/50 | 24 | 120 | 0.1013 |
| 40 | | 2502-HY | | | | | | |
| 41 | | 2502-HY | | | | | | |
| 42 | | 2502-HY | | | | | | |
| 43 | | 2502-HY | | | | | | |
| 44 | A | 2502-HY | | Randall | Hexane / Dichloromethan / Ethanol 5 / 2 / 1 | 1.5 | 95 | atmosphere |
| 45 | | 2502-HY | | | | | | |
| 46 | | 2502-HY | | | | | | |
| 47 | | 2502-HY | | | | | | |
| 48 | | 2502-HY | | | | | | |
| 49 | | 2502-HY | | | | | | |
| 50 | | 2502-HY | | | | | | |
| 51 | | 2502-HY | | | | | | |
| 52 | | 2502-HY | | | | | | |
| 53 | | 2502-HY | | | | | | |
| 54 | | 2502-HY | | | | | | |
| 55 | | 2502-HY | | | | | | |
| 56 | | 2502-HY | | | | | | |
| 57 | | 2502-HY | | | | | | |

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Hay (2502-HY)
 Methods HBCDDs - Extraction

| LC | Data set | Sample | Extraction | | | | | |
|------------------------|----------|---------|----------------------------------|-------------------------------|--------------------|---------------------|-----------------------------|---------------------------|
| | | | Sample preparation/pre-treatment | Extraction technique | Extraction solvent | Extraction time [h] | Extraction temperature [°C] | Extraction pressure [MPa] |
| 58 | | 2502-HY | | | | | | |
| 59 | | 2502-HY | | | | | | |
| 60 | | 2502-HY | | | | | | |
| 61 | | 2502-HY | | | | | | |
| 62 | | 2502-HY | | | | | | |
| 63 | | 2502-HY | | | | | | |
| 64 | | 2502-HY | | | | | | |
| 65 | | 2502-HY | | | | | | |
| 66 | | 2502-HY | | | | | | |
| 67 | | 2502-HY | | | | | | |
| 68 | A | 2502-HY | | Soxhlet | DCM:hex 1:1 | 6 | 80 | room |
| 69 | | 2502-HY | | | | | | |
| 70 | | 2502-HY | | | | | | |
| 71 | | 2502-HY | | | | | | |
| 72 | | 2502-HY | | | | | | |
| 73 | | 2502-HY | | | | | | |
| 74 | | 2502-HY | | | | | | |
| 75 | | 2502-HY | | | | | | |
| 76 | | 2502-HY | | | | | | |
| 77 | | 2502-HY | | | | | | |
| 78 | A | 2502-HY | | Pressurized liquid extraction | toluene/acetone | 0,3 | 100 | 10 |
| 79 | | 2502-HY | | | | | | |
| 80 | | 2502-HY | | | | | | |
| 81 | | 2502-HY | | | | | | |
| 82 | | 2502-HY | | | | | | |
| 83 | | 2502-HY | | | | | | |
| 84 | | 2502-HY | | | | | | |
| 85 | | 2502-HY | | | | | | |
| 86 | A | 2502-HY | | QuEChERS like-extraction | Ethyl acetate | | | |
| 87 | | 2502-HY | | | | | | |
| 88 | | 2502-HY | | | | | | |
| 89 | A | 2502-HY | | QuECHERS | Acetonitrile | 30 min | RT | |
| 90 | | 2502-HY | | | | | | |
| 91 | | 2502-HY | | | | | | |
| 92 | | 2502-HY | | | | | | |
| 93 | | 2502-HY | | | | | | |
| 94 | | 2502-HY | | | | | | |
| 95 | A | 2502-HY | | QuECHERS | | | | |
| 96 | | 2502-HY | | | | | | |
| 97 | | 2502-HY | | | | | | |
| Additional Sets | | | | | | | | |
| 86 | B | 2502-HY | | QuEChERS like-extraction | Ethyl acetate | | | |

Hay (2502-HY)
 Methods HBCDDs - Clean-up

| LC | Data set | Sample | Clean-up | | | | | | | | | | Any additional information (e.g. SPE column, dSPE) | Final volume [µl]: HBCDD |
|----|----------|---------|-------------------|-----------------------------|-----------------|----------------|---------------|------------------------|-----------------------|------------------------|-----------------------------------|---|--|--------------------------|
| | | | Gelchromatography | Silica/sulfuric acid column | Florisil column | Alumina column | Carbon column | Fully automated system | Semi-automated system | Solid Phase Extraction | Dispersive Solid Phase Extraction | Other columns / clean-up method (please specify) | | |
| 1 | A | 2502-HY | no | yes | yes | no | no | | no | no | no | | | 100 |
| 2 | | 2502-HY | | | | | | | | | | | | |
| 3 | | 2502-HY | | | | | | | | | | | | |
| 4 | | 2502-HY | | | | | | | | | | | | |
| 5 | | 2502-HY | | | | | | | | | | | | |
| 6 | | 2502-HY | | | | | | | | | | | | |
| 7 | | 2502-HY | | | | | | | | | | | | |
| 8 | | 2502-HY | | | | | | | | | | | | |
| 9 | A | 2502-HY | no | no | no | no | no | | no | no | no | | | 10 |
| 10 | | 2502-HY | | | | | | | | | | | | |
| 11 | A | 2502-HY | yes | no | no | no | no | | no | yes | no | silica gel column | Florisil SPE | 100 |
| 12 | | 2502-HY | | | | | | | | | | | | |
| 13 | | 2502-HY | | | | | | | | | | | | |
| 14 | | 2502-HY | | | | | | | | | | | | |
| 15 | | 2502-HY | | | | | | | | | | | | |
| 16 | A | 2502-HY | no | yes | no | no | no | | no | no | no | acid hydrolysis with sulphuric acid | | 100 |
| 17 | | 2502-HY | | | | | | | | | | | | |
| 18 | A | 2502-HY | no | yes | no | no | no | | no | no | no | | | 120 |
| 19 | | 2502-HY | | | | | | | | | | | | |
| 20 | A | 2502-HY | no | yes | yes | no | no | | no | no | no | | | 100 |
| 21 | | 2502-HY | | | | | | | | | | | | |
| 22 | | 2502-HY | | | | | | | | | | | | |
| 23 | | 2502-HY | | | | | | | | | | | | |
| 24 | | 2502-HY | | | | | | | | | | | | |
| 25 | | 2502-HY | | | | | | | | | | | | |
| 26 | A | 2502-HY | no | yes | no | no | no | | no | no | no | liquid / liquid extraction silica + Na2SO4 column | | 50 |
| 27 | A | 2502-HY | yes | no | no | no | no | | no | no | no | | | 500 |
| 28 | | 2502-HY | | | | | | | | | | | | |
| 29 | | 2502-HY | | | | | | | | | | | | |
| 30 | | 2502-HY | | | | | | | | | | | | |
| 31 | | 2502-HY | | | | | | | | | | | | |
| 32 | | 2502-HY | | | | | | | | | | | | |
| 33 | | 2502-HY | | | | | | | | | | | | |
| 34 | | 2502-HY | | | | | | | | | | | | |
| 35 | | 2502-HY | | | | | | | | | | | | |
| 36 | A | 2502-HY | no | yes | no | no | no | | no | no | no | | | 200 |
| 37 | | 2502-HY | | | | | | | | | | | | |
| 38 | | 2502-HY | | | | | | | | | | | | |
| 39 | A | 2502-HY | no | yes | no | no | no | | no | no | no | | | 1000 |
| 40 | | 2502-HY | | | | | | | | | | | | |
| 41 | | 2502-HY | | | | | | | | | | | | |
| 42 | | 2502-HY | | | | | | | | | | | | |
| 43 | | 2502-HY | | | | | | | | | | | | |
| 44 | A | 2502-HY | no | yes | no | no | no | | no | no | no | | | 400 |
| 45 | | 2502-HY | | | | | | | | | | | | |
| 46 | | 2502-HY | | | | | | | | | | | | |
| 47 | | 2502-HY | | | | | | | | | | | | |
| 48 | | 2502-HY | | | | | | | | | | | | |
| 49 | | 2502-HY | | | | | | | | | | | | |
| 50 | | 2502-HY | | | | | | | | | | | | |
| 51 | | 2502-HY | | | | | | | | | | | | |
| 52 | | 2502-HY | | | | | | | | | | | | |
| 53 | | 2502-HY | | | | | | | | | | | | |
| 54 | | 2502-HY | | | | | | | | | | | | |
| 55 | | 2502-HY | | | | | | | | | | | | |
| 56 | | 2502-HY | | | | | | | | | | | | |
| 57 | | 2502-HY | | | | | | | | | | | | |
| 58 | | 2502-HY | | | | | | | | | | | | |
| 59 | | 2502-HY | | | | | | | | | | | | |
| 60 | | 2502-HY | | | | | | | | | | | | |
| 61 | | 2502-HY | | | | | | | | | | | | |
| 62 | | 2502-HY | | | | | | | | | | | | |
| 63 | | 2502-HY | | | | | | | | | | | | |
| 64 | | 2502-HY | | | | | | | | | | | | |
| 65 | | 2502-HY | | | | | | | | | | | | |
| 66 | | 2502-HY | | | | | | | | | | | | |
| 67 | | 2502-HY | | | | | | | | | | | | |
| 68 | A | 2502-HY | yes | yes | no | no | yes | | yes | no | no | | | 200 |
| 69 | | 2502-HY | | | | | | | | | | | | |
| 70 | | 2502-HY | | | | | | | | | | | | |
| 71 | | 2502-HY | | | | | | | | | | | | |
| 72 | | 2502-HY | | | | | | | | | | | | |
| 73 | | 2502-HY | | | | | | | | | | | | |

Hay (2502-HY)
 Methods HBCDDs - Clean-up

| LC | Data set | Sample | Clean-up | | | | | | | | | | Any additional information (e.g. SPE column, dSPE) | Final volume [µl]: HBCDD | |
|------------------------|----------|---------|-------------------|-----------------------------|-----------------|----------------|---------------|------------------------|-----------------------|------------------------|-----------------------------------|--|--|--------------------------|------|
| | | | Gelchromatography | Silica/sulfuric acid column | Florisil column | Alumina column | Carbon column | Fully automated system | Semi-automated system | Solid Phase Extraction | Dispersive Solid Phase Extraction | Other columns / clean-up method (please specify) | | | |
| 74 | | 2502-HY | | | | | | | | | | | | | |
| 75 | | 2502-HY | | | | | | | | | | | | | |
| 76 | | 2502-HY | | | | | | | | | | | | | |
| 77 | | 2502-HY | | | | | | | | | | | | | |
| 78 | A | 2502-HY | no | yes | no | no | no | no | no | no | no | | | | 50 |
| 79 | | 2502-HY | | | | | | | | | | | | | |
| 80 | | 2502-HY | | | | | | | | | | | | | |
| 81 | | 2502-HY | | | | | | | | | | | | | |
| 82 | | 2502-HY | | | | | | | | | | | | | |
| 83 | | 2502-HY | | | | | | | | | | | | | |
| 84 | | 2502-HY | | | | | | | | | | | | | |
| 85 | | 2502-HY | | | | | | | | | | | | | |
| 86 | A | 2502-HY | no | yes | no | no | no | no | no | no | no | d-SPE on Agilent EMR dispersive kit | | | 250 |
| 87 | | 2502-HY | | | | | | | | | | | | | |
| 88 | | 2502-HY | | | | | | | | | | | | | |
| 89 | A | 2502-HY | no | no | no | no | no | no | no | no | no | liquid-liquid partition (water-cyclohexane) | | | 200 |
| 90 | | 2502-HY | | | | | | | | | | | | | |
| 91 | | 2502-HY | | | | | | | | | | | | | |
| 92 | | 2502-HY | | | | | | | | | | | | | |
| 93 | | 2502-HY | | | | | | | | | | | | | |
| 94 | | 2502-HY | | | | | | | | | | | | | |
| 95 | A | 2502-HY | no | yes | no | no | no | no | no | no | no | | | | 1000 |
| 96 | | 2502-HY | | | | | | | | | | | | | |
| 97 | | 2502-HY | | | | | | | | | | | | | |
| Additional Sets | | | | | | | | | | | | | | | |
| 86 | B | 2502-HY | no | yes | no | no | no | no | no | no | no | d-SPE on Agilent EMR dispersive kit | | | 250 |

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Hay 2025 [EURL-PT-POP_2502-HY]

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

Hay (2502-HY)

Methods HBCDDs - Detection, LOQ, measurement uncertainty

| LC | Data set | Sample | Chromatographic separation and detection method | | | | Detector | Limit of quantification Determination of LOQ | Measurement uncertainty Estimation |
|----|----------|---------|---|-----------|----------------------|--|----------|--|--|
| | | | Chromatographic separation | Injection | Injected volume [µl] | Chromatographic separation: Stationary phase | | | |
| 1 | A | 2502-HY | LC | | 2 | C18 | MS/MS | lowest validated level | expanded MU according to EURL |
| 2 | | 2502-HY | | | | | | | |
| 3 | | 2502-HY | | | | | | | |
| 4 | | 2502-HY | | | | | | | |
| 5 | | 2502-HY | | | | | | | |
| 6 | | 2502-HY | | | | | | | |
| 7 | | 2502-HY | | | | | | | |
| 8 | | 2502-HY | | | | | | | |
| 9 | A | 2502-HY | LC | 1.00 | | 2 times higher then Blanks and S/N over 10 | | no | no |
| 10 | | 2502-HY | | | | | | | |
| 11 | A | 2502-HY | LC | | | RP 18 | MS/MS | | |
| 12 | | 2502-HY | | | | | | | |
| 13 | | 2502-HY | | | | | | | |
| 14 | | 2502-HY | | | | | | | |
| 15 | | 2502-HY | | | | | | | |
| 16 | A | 2502-HY | LC | | 5 | BEH C18 (50x2.1 mm) | HRMS | 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 Uncertainty for Laboratories performing PCDD/F and PCB Analysis using Isotope Dilution Mass Spectrometry |
| 17 | | 2502-HY | | | | | | | |
| 18 | A | 2502-HY | LC | Direct | 8 | C18 | MS/MS | 0.020 ng/g | 40%. This is an estimation based on the methods performance with other matrix |
| 19 | | 2502-HY | | | | | | | |
| 20 | A | 2502-HY | LC | | 10 | C18 | HRMS | Lowest validated level | RSS |
| 21 | | 2502-HY | | | | | | | |
| 22 | | 2502-HY | | | | | | | |
| 23 | | 2502-HY | | | | | | | |
| 24 | | 2502-HY | | | | | | | |
| 25 | | 2502-HY | | | | | | | |
| 26 | A | 2502-HY | LC | | 3 | Hypersil Gold | MS/MS | S/N = 10 | |
| 27 | A | 2502-HY | LC | | 10 | C-18 | MS/MS | spiking of matrix with low amount of analytes | based on validation - calculation from RSD and bias |
| 28 | | 2502-HY | | | | | | | |
| 29 | | 2502-HY | | | | | | | |
| 30 | | 2502-HY | | | | | | | |
| 31 | | 2502-HY | | | | | | | |
| 32 | | 2502-HY | | | | | | | |
| 33 | | 2502-HY | | | | | | | |
| 34 | | 2502-HY | | | | | | | |
| 35 | | 2502-HY | | | | | | | |
| 36 | A | 2502-HY | LC | UPLC | 5 | Eclipse Plus C18, 2.1 x 100mm | MS/MS | Lowest point of validation | 40% - This is the uncertainty measurement we use for accredited food samples. We are not accredited for feed matrices. |
| 37 | | 2502-HY | | | | | | | |
| 38 | | 2502-HY | | | | | | | |
| 39 | A | 2502-HY | LC | | | Acquity BEH C18 150 mm | MS/MS | | |
| 40 | | 2502-HY | | | | | | | |
| 41 | | 2502-HY | | | | | | | |
| 42 | | 2502-HY | | | | | | | |
| 43 | | 2502-HY | | | | | | | |
| 44 | A | 2502-HY | LC | | 15 | C18 | MS/MS | ten point equidistant calibration in Matrix at the expected LOQ using DIN 32645 | |
| 45 | | 2502-HY | | | | | | | |
| 46 | | 2502-HY | | | | | | | |
| 47 | | 2502-HY | | | | | | | |
| 48 | | 2502-HY | | | | | | | |
| 49 | | 2502-HY | | | | | | | |
| 50 | | 2502-HY | | | | | | | |
| 51 | | 2502-HY | | | | | | | |
| 52 | | 2502-HY | | | | | | | |
| 53 | | 2502-HY | | | | | | | |
| 54 | | 2502-HY | | | | | | | |

EURL Proficiency Study on the Determination of PCDD/Fs, PCBs, PBDEs, HBCDDs and PFAS in Hay 2025 [EURL-PT-POP_2502-HY]

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

Hay (2502-HY)

Methods HBCDDs - Detection, LOQ, measurement uncertainty

| LC | Data set | Sample | Chromatographic separation and detection method | | | Detector | Limit of quantification Determination of LOQ | Measurement uncertainty Estimation | |
|------------------------|----------|---------|---|-------------|----------------------|---|---|--|--|
| | | | Chromatographic separation | Injection | Injected volume [µl] | | | | Chromatographic separation: Stationary phase |
| 55 | | 2502-HY | | | | | | | |
| 56 | | 2502-HY | | | | | | | |
| 57 | | 2502-HY | | | | | | | |
| 58 | | 2502-HY | | | | | | | |
| 59 | | 2502-HY | | | | | | | |
| 60 | | 2502-HY | | | | | | | |
| 61 | | 2502-HY | | | | | | | |
| 62 | | 2502-HY | | | | | | | |
| 63 | | 2502-HY | | | | | | | |
| 64 | | 2502-HY | | | | | | | |
| 65 | | 2502-HY | | | | | | | |
| 66 | | 2502-HY | | | | | | | |
| 67 | | 2502-HY | | | | | | | |
| 68 | A | 2502-HY | LC | | 70 | Luna php | MS/MS | 0.01 (lowest spiked level reaching requirements laid down in EURL recommendations) | taking into account uncertainty from bias and RSD under reproducibility conditions and rounding up |
| 69 | | 2502-HY | | | | | | | |
| 70 | | 2502-HY | | | | | | | |
| 71 | | 2502-HY | | | | | | | |
| 72 | | 2502-HY | | | | | | | |
| 73 | | 2502-HY | | | | | | | |
| 74 | | 2502-HY | | | | | | | |
| 75 | | 2502-HY | | | | | | | |
| 76 | | 2502-HY | | | | | | | |
| 77 | | 2502-HY | | | | | | | |
| 78 | A | 2502-HY | LC | | 10 | Hypersil Gold C18, 100x2,1mm, 1,9µm | MS/MS | LOD and LOQ were estimated based on analysis of 10 blank spiked samples | MU were estimated based on precision and truenes from fortyfication experiment |
| 79 | | 2502-HY | | | | | | | |
| 80 | | 2502-HY | | | | | | | |
| 81 | | 2502-HY | | | | | | | |
| 82 | | 2502-HY | | | | | | | |
| 83 | | 2502-HY | | | | | | | |
| 84 | | 2502-HY | | | | | | | |
| 85 | | 2502-HY | | | | | | | |
| 86 | A | 2502-HY | LC | | 20 | KINETEX 2.6um XB-C18 100A (100 x 2.1 mm) (PHENOMENEX) | MS/MS | 0.050 µg/kg determined by fortified samples | Nordtest |
| 87 | | 2502-HY | | | | | | | |
| 88 | | 2502-HY | | | | | | | |
| 89 | A | 2502-HY | LC | sample loop | 5 | cyclodextrine | MS/MS | 10>1 signal to noise at spiked samples | multiple measurement with reference material RSD x 2 |
| 90 | | 2502-HY | | | | | | | |
| 91 | | 2502-HY | | | | | | | |
| 92 | | 2502-HY | | | | | | | |
| 93 | | 2502-HY | | | | | | | |
| 94 | | 2502-HY | | | | | | | |
| 95 | A | 2502-HY | LC | | 10 | | MS/MS | 2 times higher than Blanks and S/N over 10 | profficiency testings and spiked samples |
| 96 | | 2502-HY | | | | | | | |
| 97 | | 2502-HY | | | | | | | |
| Additional Sets | | | | | | | | | |
| 86 | B | 2502-HY | LC | | 20 | KINETEX 2.6um XB-C18 100A (100 x 2.1 mm) (PHENOMENEX) | MS/MS | 0.050 µg/kg determined by fortified samples | Nordtest |