European Union Reference Laboratory
for Halogenated POPs in Feed and Food

State Institute for Chemical and Veterinary Analysis of Food, Freiburg, Germany

Chemisches und Veterinäruntersuchungsamt Freiburg
PO Box 100462 ◆ D-79123 Freiburg ◆ Germany

EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in Baby Food 2021

EURL-PT-DPB_2101-BF

FOOD

Report
PCDD/Fs and PCBs

(Version 1.1)

26 November 2021

This report on the EURL Proficiency Test on the Determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in baby food 2021 [EURL-PT-DPB-2101-BF] organized by the EURL for halogenated persistent organic pollutants (POPs) in Feed and Food is only available as pdf-version. The forwarding and reproduction of this report is permitted only as entire document, including 7 annexes.

Office Building
Bissierstraße 5
79114 Freiburg

Phone/Fax
Phone: +49-761-8855-500
Fax: +49-761-8855-100

E-Mail
EURL: eurl-pops@cvuafr.bwl.de

Internet
www.eurl-dioxin-freiburg.eu

Summary

Test sample (food)	Baby food – 2101-BF
Analytes of interest	Mandatory for NRLs: - **PCDD/Fs** (17 2,3,7,8-substituted PCDD/Fs) - **PCBs** (12 DL-PCBs, 6 NDL-PCBs)
Methods	PCDD/Fs, DL-PCBs: GC-HRMS, GC-MS/MS and alternative methods; Bioanalytical screening methods Indicator PCBs: Any kind of method
Participants	NRLs, OFLs, other official laboratories, commercial laboratories performing the analysis of samples taken by food business operators
Statistical evaluation	ISO 13528:2020, IUPAC Protocol, positive scoring system
Report	30 July 2021 (Version 1.0) 26 November 2021 (Version 1.1): **Correction of z-scores for lipid content**

1. Structure of the PT, test material and analytes

This proficiency test (PT) on the determination of PCDD/Fs, PCBs, PBDEs and HBCDDs in baby food was organized by the EURL for halogenated persistent organic pollutants (POPs) in feed and food to be performed between February and April 2021. The objective was to assess analytical performance of laboratories and the interlaboratory comparability of results from analyses of PCDD/Fs, PCBs, PBDEs and HBCDDs in one sample of baby food.

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 2021. 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 will allow the extension of the data basis for calculation of assigned values and evaluation of results.

This PT was also open to other official laboratories and commercial laboratories analysing samples taken by food business operators in the performance of official control or self-control in order to check the comparability of results not only within the EURL/NRL/OFL network, but also with official and private laboratories.

The evaluated results were discussed by representatives of EU Commission, NRLs and the EURL at the COM/EURL/NRL workshop in May 2021.

1.1 Samples and coding

The baby food test sample was prepared of regular market food (pumpkin, pork, vegetable oil). The test sample was fortified with the analytes of interest using PCDD/F standards and technical mixtures of PCBs, PBDEs and HBCDDs.

Baby food	**Sample no. 2101-BF-xxx**

Each participant received about 100 g of the test sample.

1.2 Analytes of interest

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

- 17 2,3,7,8-substituted PCDD/Fs
- WHO-PCDD/F-TEQ (using WHO_{2005}-TEF)
- 12 dioxin-like PCBs
- WHO-PCB-TEQ (using WHO_{2005}-TEF)
- WHO-PCDD/F-PCB-TEQ (using WHO_{2005}-TEF)
- Six indicator PCBs: PCB 28, 52, 101, 138, 153, 180
- Sum of six indicator PCBs: Sum of PCB 28, 52, 101, 138, 153, 180
- PCDD/F-PCB-BEQ, PCDD/F-BEQ and/or PCB-BEQ
  (using bioanalytical screening methods)

1.3 Methods

One or more of the following detection methods could be applied:

- GC-HRMS-, GC-MS/MS-methods or other alternative methods for PCDD/Fs and dioxin-like PCBs
- Bioanalytical screening methods for PCDD/Fs and dioxin-like PCBs
- Any kind of method for indicator PCBs

1.4 Coding of laboratories and confidentiality

The laboratory code of the participating laboratories will be kept confidential and will not be revealed to other participants.

For NRLs, the “Protocol for management of underperformance in comparative testing and/or lack of collaboration of National Reference Laboratories (NRLs) with Community reference laboratories (CRLs) activities” will be observed. The confidentiality of NRLs will be kept according to this protocol.

The identity of OFLs will be kept confidential, unless a Member State initiated a co-operation between the NRL, OFLs and the EURL.

1.5 Results of PCDD/Fs and PCBs determined by physico-chemical methods

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,
- report upper, middle and lower bound results for WHO-PCDD/F-PCB-TEQ, WHO-PCDD/F-TEQ, WHO-PCB-TEQ and sum of six indicator PCBs,
- report if sample exceeds respective EU maximum or action levels for WHO-PCDD/F-PCB-TEQ, WHO-PCDD/F-TEQ and/or WHO-PCB-TEQ or the maximum level for the sum of six indicator PCBs beyond reasonable doubt taking into account the measurement uncertainty,
- report the measurement uncertainty, applied for checking of compliance, for WHO-PCDD/F-PCB-TEQ, WHO-PCDD/F-TEQ, WHO-PCB-TEQ and the sum of six indicator PCBs,
- 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 pg/g wet weight for PCDD/Fs and dioxin-like PCBs, in ng/g wet weight for indicator PCBs. TEQ-based results had to be calculated using the WHO-TEFs of 2005 [3].

1.6 Results of PCDD/Fs and PCBs determined by bioanalytical screening methods

Laboratories should

- use their own reference standards,
- report if the samples are suspected to be non-compliant with EU legal limits and confirmation is required
- report PCDD/F and/or PCB results in BEQ, if applicable,
- report the reporting limit, maximum / action level, which the evaluation is based on, and the bioassay cut-off, if applicable,
- give method information and
- give information about the accreditation of the laboratory according to ISO/IEC 17025.

Results had to be reported in pg BEQ/g wet weight for PCDD/Fs and dioxin-like PCBs.

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

102 laboratories registered for this proficiency test and 94 laboratories reported results.

Table 1: Participating laboratories

Participating laboratories	Region	No. of participants
National Reference Laboratories	European Union	28
	Other Countries	3
Official Laboratories	European Union	41
	Other European Countries	1
	Africa	0
	Americas	0
	Asia	0
	Oceania	1
Commercial Laboratories	European Union	12
	Other European Countries	1
	Africa	0
	Americas	3
	Asia	4
	Oceania	0
	Total	**94**

2.1 Number of reported results

Table 2: Reported results for PCDD/F and PCB sum parameters and lipid content

Reported results	WHO-PCDD/F-PCB-TEQ	WHO-PCDD/F-TEQ	WHO-PCB-TEQ	Sum of six indicator PCBs	PCDD/F-PCB-BEQ [Bioanalytical screening methods]	Lipid content
All laboratories	75	75	77	84	6	63
NRLs	21	21	22	27	3	23

2.2 Accreditation

Table 3: Reported accreditation according to ISO/IEC 17025 by participants for PCDD/Fs and PCBs

Baby Food (2101-BF)	PCDD/Fs, PCBs [Physico-chemical methods]	PCDD/Fs, PCBs [Bioanalytical screening methods]
yes	82	4
no	5	2

3. Detection methods

The following detection methods were applied:

- GC-HRMS-, GC-MS/MS-, GC-LRMS-methods for PCDD/Fs and non- ortho PCBs
- GC-HRMS-, GC-MS/MS-, GC-LRMS-, GC-ECD-methods for mono-ortho-PCBs and indicator PCBs
- Bioanalytical screening methods for PCDD/Fs and dioxin-like PCBs

Table 4: Overview of physico-chemical detection methods for PCDD/Fs and PCBs applied by participants

Detection methods	PCDD/Fs	non-ortho-PCBs	mono-ortho-PCBs	Indicator PCBs
HRMS	60	61	57	45
HRMS/MS/MS	2	2	2	2
MS/MS	10	10	11	20
LRMS	1	1	1	7
ECD	-	-	-	5

4. Homogeneity and stability of the test material

The test for sufficient homogeneity was performed according to ISO 13528:2020 [2] and the International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [1].

Therefore, 10 portions of the test samples 2101-BF were analyzed in duplicate for PCDD/Fs and PCBs. The test for sufficient homogeneity was performed for the sum parameters WHO-PCDD/F-PCB-TEQ, WHO-PCDD/F-TEQ, WHO-PCB-TEQ, the sum of six indicator PCBs and individual congeners. The test material was sufficiently homogeneous for this proficiency test.

The stability check of the analytes of interest applying room temperature storage was performed according to ISO 13528:2020 [2]. The test material showed sufficient stability for this proficiency test.

5. Determination of the assigned value

Statistical evaluation of the PT results is performed by the EURL for halogenated POPs in feed and food according to ISO 13528:2020 [2] and the International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [1].

The determination of the assigned value was performed according [1] by estimating of the assigned value as the consensus of participants' results (using only results of physico-chemical methods). The Huber robust mean was taken as assigned value after excluding extreme outliers (outside the range of ± 50 % of the median of all reported results) and examination of the distribution of the remaining results using histogram and kernel density estimation, if necessary.

The assigned value was calculated for WHO-PCDD/F-PCB-TEQ, WHO-PCDD/F-TEQ, WHO-PCB-TEQ, the sum of six indicator PCBs and individual PCDD/F and PCB congeners (including limits of quantification (LOQs)), if possible. Additionally the median of all values is calculated.

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

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

- the lower bound WHO-PCDD/F-TEQ
- 2,3,7,8-TCDD, 1,2,3,7,8-PeCDF, 1,2,3,7,8,9-HxCDF, 1,2,3,4,7,8,9-HpCDF
- PCB 123 and PCB 169

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 these submitted results. Additionally the results of all participants reporting results and the results of participants having accreditation according ISO/IEC 17025 were compared for PCDD/F and PCB sum parameters. No significant differences between the assigned values calculated for both data sets were observed (Table 5).

Table 5: Comparison of assigned values for all participants and participants with reported accreditation according to ISO/IEC 17025 for PCDD/F and PCB sum parameters in one sample of baby food 2101-BF

Sum parameters	Assigned value All participants	Assigned value ISO/IEC 17025 accreditation	Deviation
	pg/g, ng/g wet weight	pg/g, ng/g wet weight	%
WHO-PCDD/F-PCB-TEQ ub rep	0.104	0.103	< 1
WHO-PCDD/F-TEQ ub rep	0.0508	0.0507	< 1
WHO-PCB-TEQ ub rep	0.0509	0.0502	1
Sum Indicator PCBs ub rep	0.533	0.534	< 1

5.1 PCDD/Fs and PCBs – Sum parameters

The assigned values for the test sample 2101-BF were calculated as consensus of participants' results for the PCDD/F and PCB sum parameters, taking into account the calculation criteria described above.

Table 6: Assigned values for physico-chemical methods for PCDD/Fs and PCBs (rounded to three significant figures)

Test sample	WHO-PCDD/F-PCB-TEQ upper bound	WHO-PCDD/F-TEQ upper bound	WHO-PCB-TEQ upper bound	Sum Indicator PCBs upper bound
	pg/g wet weight	pg/g wet weight	pg/g wet weight	ng/g wet weight
Baby Food (2101-BF)	0.104	0.0508	0.0509	0.533

Table 7: Assigned values for PCDD/Fs and DL-PCBs for comparison with BEQ results of bioanalytical screening methods (rounded to two significant figures)

Test sample	WHO-PCDD/F-PCB-TEQ upper bound	WHO-PCDD/F-TEQ upper bound	WHO-PCB-TEQ upper bound
	pg/g wet weight	pg/g wet weight	pg/g wet weight
Baby Food (2101-BF)	0.10	0.051	0.051

5.2 PCDD/Fs and PCBs – Individual congeners

The assigned values for the test sample 2101-BF for individual congeners were calculated as a consensus of the participants' results, taking into account the calculation criteria described above (Figure 1; tabular summary see annex 1).

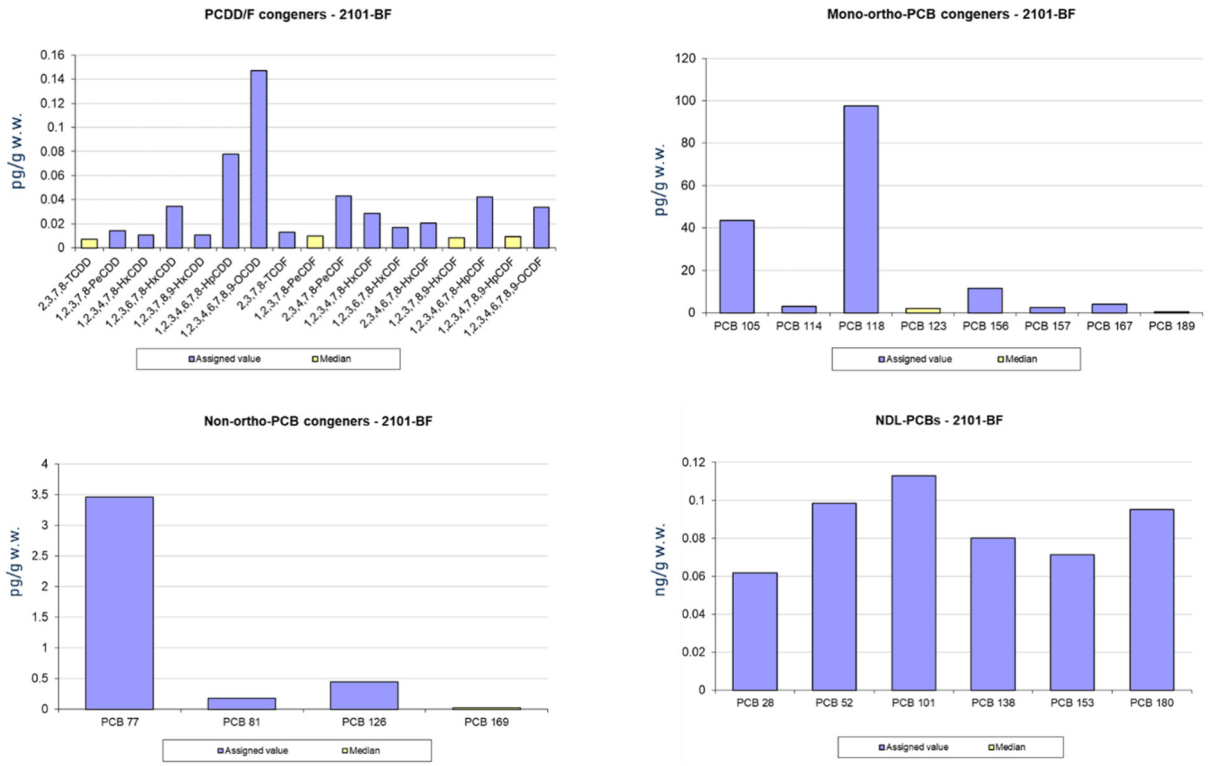

Figure 1: Assigned values (blue) and median values (yellow) for PCDD/F and PCB congeners for baby food (2101-BF) [pg/g or ng/g w. w]

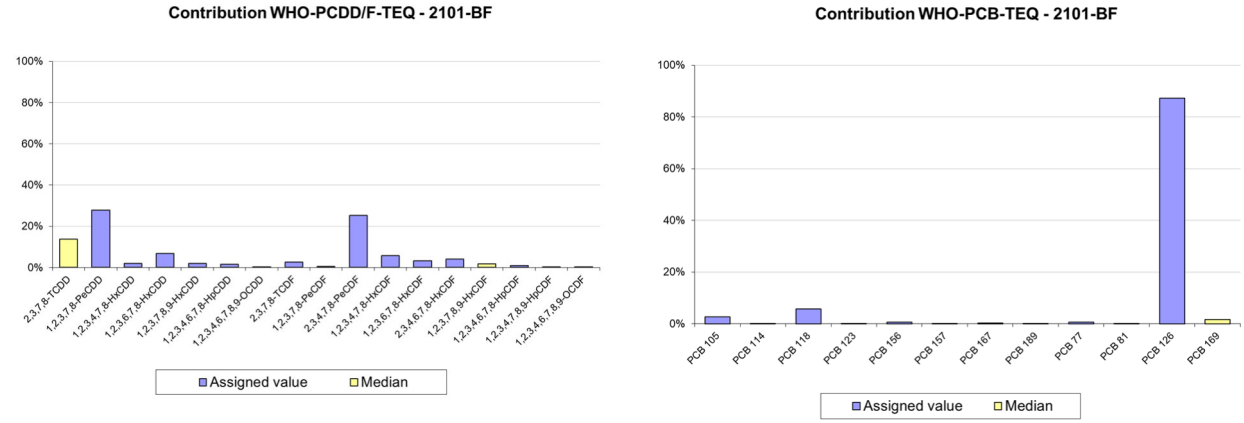

Figure 2: Contributions in % to WHO-PCDD/F-TEQ and WHO-PCB-TEQ for PCDD/F and PCB assigned (blue) and median (yellow) values for baby food (2101-BF)

5.3 Lipid content

For the lipid content an assigned value of 11.6 % for the test sample 2101-BF was calculated as a consensus of the participants' results, taking into account the calculation criteria described above.

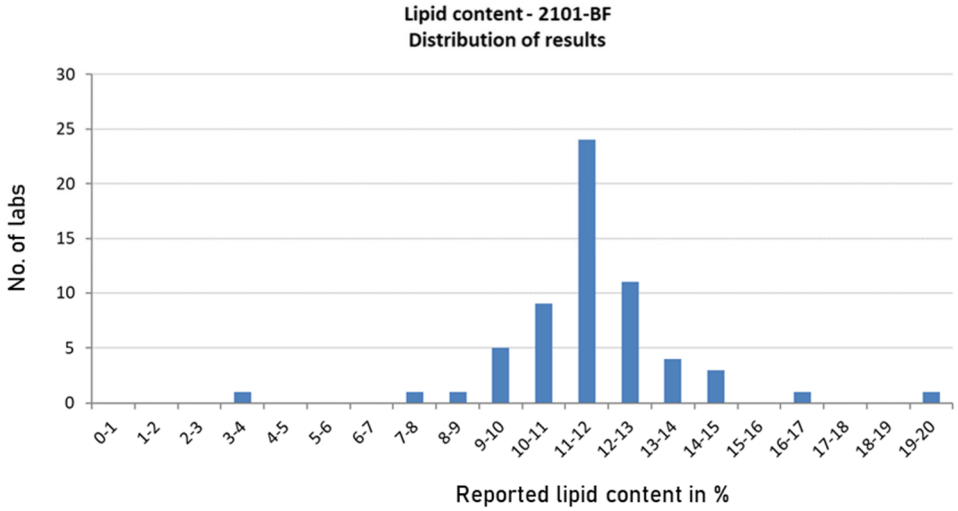

Figure 3: Distribution of participant's results of the lipid content in % for baby food (2101-BF)

5.4 Comparison of assigned values with legal limits

Maximum levels for food are defined in Commission Regulation (EC) No 1881/2006 of 19 December 2006 setting maximum levels for certain contaminants in foodstuff. Presently, no action levels are defined for PCDD/Fs and PCBs in infant food.

Table 8: Maximum levels for baby food (Annex: Selection 5 "Dioxins and PCBs No 5.13" of Commission Regulation (EC) No 1881/2006)

Foods for infants and young children (*)		**Maximum level**	**Action level**
WHO-PCDD/F-PCB-TEQ	pg/g wet weight	0.2	-
WHO-PCDD/F-TEQ	pg/g wet weight	0.1	-
WHO-PCB-TEQ	pg/g wet weight	-	-
Sum of PCB28, PCB52, PCB101, PCB138, PCB153 and PCB180	ng/g wet weight	1.0	-

(*) The maximum level refers to the products ready to use (marketed as such or after reconstitution as instructed by the manufacturer)

For the baby food test sample 2101-BF the assigned values for the sum parameters WHO-PCDD/F-PCB-TEQ, WHO-PCDD/F-TEQ and the sum of indicator PCBs were one-half of the respective maximum levels (Figure 4).

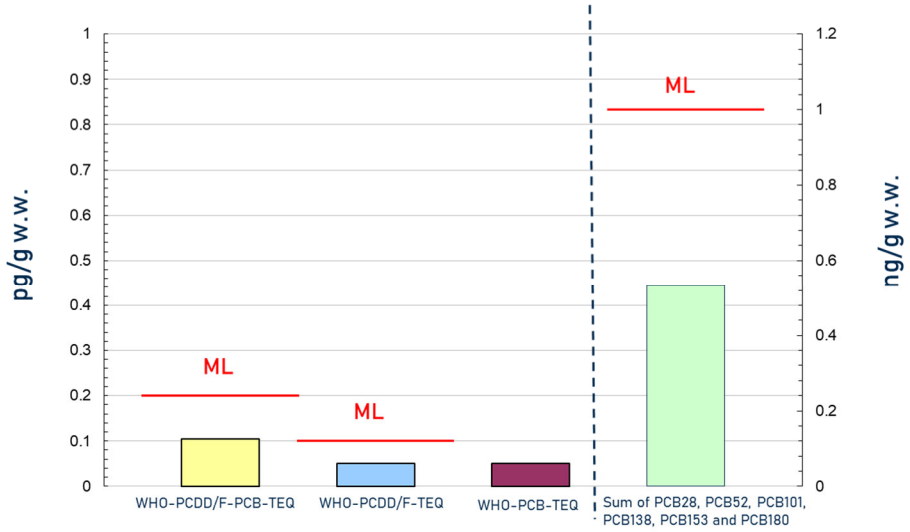

Figure 4: Comparison of the assigned values for sum parameters for baby food (2101-BF) with maximum levels (red lines) [pg/g and ng/g wet weight]

6. Evaluation of results

6.1 Participants' results for physico-chemical methods

6.1.1 Z-score calculation

The criteria for successful participation of laboratories using physico-chemical methods are based on the evaluation of the results of the sum parameters WHO-PCDD/F-TEQ, WHO-PCB-TEQ, WHO-PCDD/F-PCB-TEQ and the sum of six indicator PCBs and evaluated individual congeners. The criteria apply to sum parameter concentrations in the range (about 0.5 to 4 times) of the level of interest (maximum or action level).

For the evaluation of the results of physico-chemical methods, the z-scores are calculated according to the following formula:

$$z = \frac{(x - x_a)}{\sigma_p}$$

x: participant's result

x_a: assigned value

σ_p: fitness-for-purpose-based standard deviation for proficiency assessment

For WHO-PCDD/F-TEQ, WHO-PCB-TEQ and WHO-PCDD/F-PCB-TEQ the standard deviation for proficiency assessment σ_p is defined as 10 %, for the sum of six indicator PCBs (PCB 28, 52, 101, 138, 153, 180) as 15 % and for evaluated individual PCDD/F, PCB congeners as 20 %.

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

Interpretation of z-scores:

$\|z-score\| \leq 2$	*Satisfactory performance*
$2 < \|z-score\| < 3$	*Questionable performance (warning signal)*
$\|z-score\| \geq 3$	*Unsatisfactory performance (action signal)*

6.1.2 PCDD/Fs and PCBs - Participants' z-scores

The concentrations of the sum parameters for the test samples 2101-BF were in the range (about 0.5 to 4 times) of the respective maximum levels (tabular summaries of participants' results and z-scores see annex 2 and 3).

Table 9: Distribution of participants' z-scores for sum parameters

Baby Food (2101-BF)	WHO-PCDD/F-PCB-TEQ	WHO-PCDD/F-TEQ	WHO-PCB-TEQ	Sum of six indicator PCBs
|z-score| ≤ 2	80 %	70 %	82 %	87 %
2 <|z-score| < 3	7 %	9 %	8 %	2 %
|z-score| ≥ 3	13 %	21 %	10 %	11 %

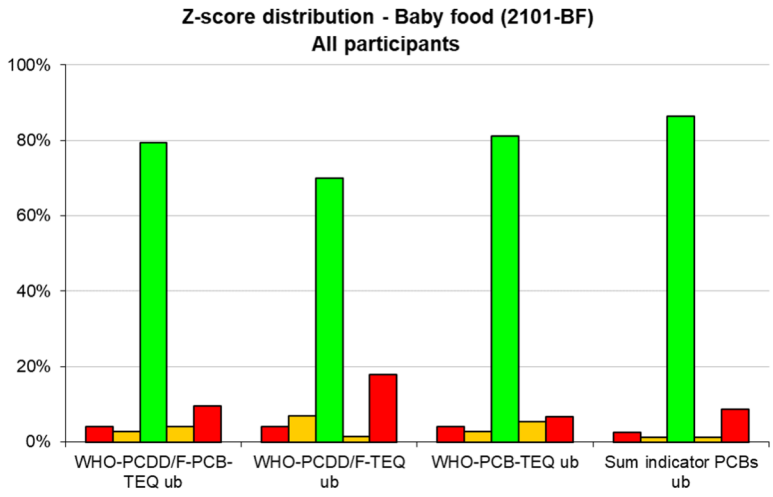

Figure 5: Distribution of participants' z-scores for sum parameters for baby food (2101-BF)
[Green bars: -2 ≤ z-score ≤ 2, yellow bars: -3 < z-score < -2, 2 < z-score < 3, red bars: z-score ≤ -3, z-score ≥ 3]

6.1.3 Comparison of reported and calculated sum parameters

In addition to the calculation of the sum parameters for reported individual PCDD/F and PCB congener values, the calculated sum parameters for PCDD/Fs and PCBs by the EURL were compared with the ones reported by each participant. As the reported sum parameters are decisive to compare the results with the legal limits, an incorrect calculation might lead to a wrong assessment of a sample. In case of a significant deviation of the reported sum parameter value from the (EURL) calculated one (deviation > 10 %) the laboratory has therefore not successfully participated in the PT according to the positive scoring system (see 6.1.5).

Table 10: Difference between reported and calculated sum parameters for PCDD/Fs and PCBs for baby food (2101-BF) given in percentage of participants' results

Baby Food (2101-BF)	WHO-PCDD/F-PCB-TEQ	WHO-PCDD/F-TEQ	WHO-PCB-TEQ	Sum of six indicator PCBs
Deviation ≤ 10 %	99%	96%	97%	96%
Deviation > 10 %	1%	4%	3%	4%

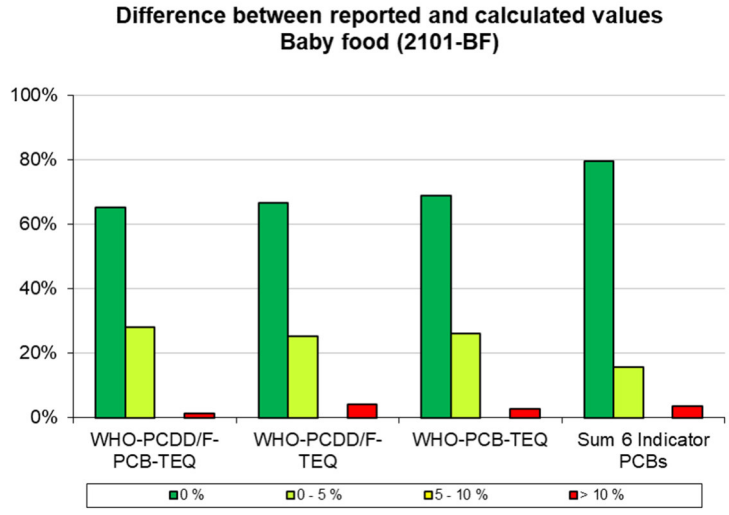

Figure 6: Difference between upper and lower bound calculation for baby food (2101-BF) given in percentage of participants' results [Dark green bars: 0 – 10 %, light green bars: 10 – 20 %, yellow bars: 20 – 50 %, red bars: > 50 %]

6.1.4 Difference between upper and lower bound calculation

According to Commission Regulation (EU) No 2017/644 the difference between upper bound level and lower bound level shall not exceed 20 % for confirmation of exceedance of maximum level or in case of need of action levels for PCDD/Fs and DL-PCBs. For indicator PCBs the difference between upper bound and lower bound levels for the sum of

six indicator PCBs shall be ≤ 20 % at the level of interest. Participants with a larger deviation should review their analytical methods, especially with regard to sensitivity and limit of quantification.

For the test samples 2101-BF the assigned values for all sum parameters were below the respective maximum levels.

Table 11: Difference between upper and lower bound calculation for baby food (2101-BF) given in percentage of participants' results

Baby Food (2101-BF)	WHO-PCDD/F-PCB-TEQ	WHO-PCDD/F-TEQ	WHO-PCB-TEQ	Sum of six indicator PCBs
0 – 10 %*	68%	65%	93%	94%
10 – 20 %*	13%	8%	3%	1%
20 – 50 %*	11%	12%	0%	2%
> 50 %*	8%	15%	4%	2%

* Difference between upper and lower bound calculation

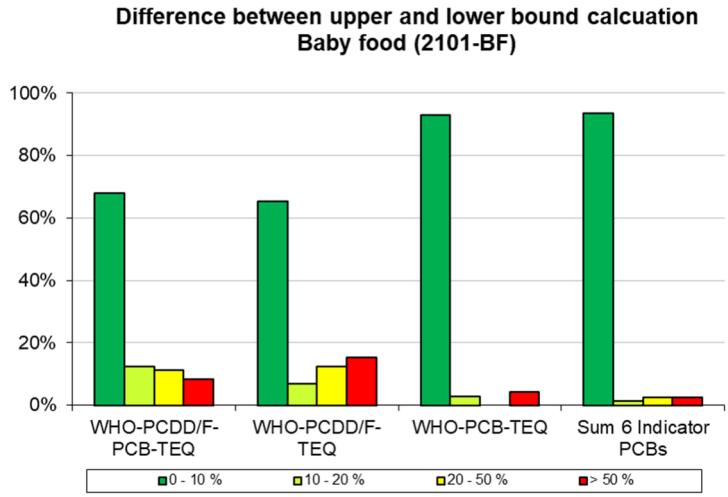

Figure 7: Difference between upper and lower bound calculation for baby food (2101-BF) given in percentage of participants' results [Dark green bars: 0 – 10 %, light green bars: 10 – 20 %, yellow bars: 20 – 50 %, red bars: > 50 %]

6.1.5 Positive scoring system

The "positive scoring system" gives one assessment for each PT sample covering all relevant PCDD/F and PCB sum parameters and congeners.

The total score for the positive scoring system was calculated according to the following general principles:

- Calculation of z-scores for sum parameters and evaluated individual congeners
- Calculation of the positive scores according to the following table:

Table 12: Positive scores allocated to z-scores achieved for all parameters

Positive scoring system	**I z-score I ≤ 2**	**2 < I z-score I < 3**	**I z-score I ≥ 3**
Individual congeners	**Positive score**	**Positive score**	**Positive score**
Contribution to sum parameter* > 10 %	12	6	0
Contribution to sum parameter* 3 – 10 %	8	4	0
Contribution to sum parameter* < 3 %	6	3	0
Not evaluated congeners	0	0	0

*separately for the respective sum parameters WHO-PCDD/F-TEQ, WHO-PCB-TEQ and the sum of six indicator PCBs

- Calculation of maximum achievable scores (I z-score I ≤ 2) for PCDD/F and DL-PCB and indicator PCB congeners separately:

$$Maximum\ Score = \sum_{i=1}^{n} Max.Score_{(>10\%)i} + \sum_{i=1}^{m} Max.Score_{(3-10\%)i} + \sum_{i=1}^{p} Max.Score_{(<3\ \%)i}$$

- Calculation of the participant's scores for PCDD/F and DL-PCB and indicator PCB congeners separately:

$$Participant's\ Score = \sum_{i=1}^{n} Score_{(>10\%)i} + \sum_{i=1}^{m} Score_{(3-10\%)i} + \sum_{i=1}^{p} Score_{(<3\ \%)i}$$

- Calculation of achieved scoring percentage for each participant:

$$Participant's\ Scoring\ Percentage = \frac{Participant's\ score}{Maximum\ score} \cdot 100$$

- Criteria for successful participation:

Sum parameters:	≤ 1 parameter with | z-score | > 2, no parameter with | z-score | ≥ 3
PCDD/F congeners:	≥ 75 % of maximum score
DL-PCB congeners:	≥ 75 % of maximum score
Indicator PCB congeners:	≥ 75 % of maximum score
Difference between reported and calculated results for sum parameters	≤ 10 %

The assessment based on the positive scoring system was performed for the PT test sample baby food 2101-BF. A laboratory participated successfully in an EURL PT for PCDD/Fs and PCBs, if all above mentioned criteria for the reported analytes are met.

Table 13: Successful participation rate according to positive scoring system for baby food (2101-BF)

Scoring system	**Successful participation**				
Percentage of participants' results	**yes**	**no**	**Sum parameters**	**Individual congeners**	**Calculation of sum parameters**
2101-BF	59%	41%	68%	73%	27%

6.2 Participants' results for bioanalytical screening methods

According to Commission Regulation (EU) No 2017/644, "a screening method in principle classifies a sample as compliant or suspected to be non-compliant. For this, the calculated BEQ level is compared to the cut-off value […]. Samples below the cut-off value are declared compliant, samples equal or above the cut-off value as suspected to be non-compliant, requiring analysis by a confirmatory method".

Therefore, the main criterion for evaluation of results from bioanalytical screening methods is their ability to reliably identify compliant samples and samples suspected to be non-compliant with established legal limits.

For further evaluation of the performance of bioanalytical screening methods, bioassay-scores are applied: The reported BEQ-values derived from bioanalytical screening methods are compared with the WHO-TEQ assigned values calculated on basis of the results

of physical-chemical methods for the concentration range of 0.5 to 2 times the level of interest.

Because bioanalytical screening methods focus mainly on distinguishing between compliant and potentially non-compliant samples, a direct comparison of bioassay-scores and z-scores is not possible. However, bioassay scores may serve as a tool to assess method performance within the scope of external quality control measures of the respective laboratory.

Bioassay-scores are calculated according to the following formula:

$$\boldsymbol{bioassay - score} = \frac{(\boldsymbol{x} - \boldsymbol{x_{a)}}}{\boldsymbol{\sigma_{bioassay}}}$$

x_a: *assigned value (physical-chemical methods)*

x: *participant's result (BEQ from bioanalytical screening method)*

$\sigma_{bioassay}$: *bioassay target deviation*

For PCDD/F-BEQ, PCB-BEQ and PCDD/F-PCB-BEQ the bioassay target deviation $\sigma_{Bioassay}$ is defined as 20 %.

6.2.1 Assessment of analytical results

As a consequence of the comparison of the assigned values with legal limits, the assessment of the analytical results using bioanalytical screening methods should read "compliant with the maximum level for WHO-PCDD/F-PCB-TEQ and WHO-PCDD/F-TEQ" for the test sample 2101-BF.

Six laboratories reported results using CALUX bioassay for Total-BEQ and hereof two also for PCDD/F-BEQ and/or PCB-BEQ.

Table 12: Participants' assessment of analytical results using bioanalytical screening methods for 2101-BF

Laboratories' assessment of analytical results	**WHO-PCDD/F-PCB-TEQ** Maximum level	**WHO-PCDD/F-TEQ** Maximum level
Suspected to be non-compliant	2	2
Compliant	4	1

6.2.2 Participants' bioassay-scores

Concentrations for WHO-PCDD/F-PCB-TEQ and WHO-PCDD/F-TEQ in the test sample 2101-BF are in the range (about 0.5 to 2 times) of the respective maximum levels.

Table 14: Distribution of participants' bioassay-scores for BEQ parameters for baby food (2101-BF)

Percentage of participants' results	PCDD/F-PCB-BEQ	PCDD/F-BEQ	PCB-BEQ
|bioassay-score| ≤ 2	33 %	-	50 %
2 < |bioassay-score| < 3	-	-	-
|bioassay-score| ≥ 3	67 %	100 %	50 %

7. Participants' feedback

A questionnaire for feedback from participants of this EURL proficiency test was available as online survey between 10 May 2021 and 11 June 2021. The survey was anonymous, but participants could also give their laboratory name. The identity of the laboratories is kept confidential. The survey included seven questions related to different topics (participants' information, organization of the proficiency test, PT test samples and evaluation of results and summary of data) and a possibility to include comments and further suggestions.

In total, 29 laboratories (31 % of all participants) participated in this survey.

7.1 Overview of questions and answers of participants

Participants' information (more than one answer possible):

National Reference Laboratory (NRL)	Official Laboratory (OFL)	Commercial laboratory	Other
14	11	4	1

Rating of organization of the PT from no at all (1/5) to full satisfaction (5/5) by **29** laboratories:

	No opinion	1/5	2/5	3/5	4/5	5/5
Announcement	-	-	-	3	5	23
Instructions	-	-	1	1	4	23
Sample shipment	-	-	-	1	5	23
Reporting of results	-	-	-	1	8	20

Was all information provided in an understandable way	-	-	1	-	5	23
Was the time frame acceptable	-	-	1	1	6	21
Was the handling of EU survey as webtool mangeable	1	2	-	2	10	14

Rating of chosen PT test sample from no at all (1/5) to full satisfaction (5/5) by **29** laboratories:

	No opinion	**1/5**	**2/5**	**3/5**	**4/5**	**5/5**
Selection of matrix	-	1	3	4	2	19
Adequate level of contamination	-	1	3	4	10	11

Rating of evaluation of results and summary of data from no at all (1/5) to full satisfaction (5/5) by **29** laboratories:

	No opinion	**1/5**	**2/5**	**3/5**	**4/5**	**5/5**
Preliminary report	-	-	-	1	9	19
Evaluation of results and report clear and comprehensible	-	-	-	3	5	23
Met the PT your expectations	-	-	-	6	7	16

7.2 Comments and suggestions

Comments referred to chosen matrix and range of concentration. A more frequent matrix, higher concentrations and more discussion about levels of PCDD/Fs including LOQs would be helpful. It would be desirable if there were more opportunities to discuss some methodological aspects within the EURL/NRL network. Feedback on the experiences with the EUsurvey webtool was positive.

8. 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).

9. Results of participants

An overview of the PCDD/F and PCB results for the PT test sample baby food (2101-BF) and the evaluation of the results are given in the following annexes 1 – 7. Laboratories are coded according to the laboratory codes sent after registration.

10. References

[1] 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.

[2] ISO 13528:2020, Statistical methods for use in proficiency testing by interlaboratory comparisons, International Organization for Standardization

[3] M. van den Berg et al., The 2005 World Health Organization Re-evaluation of Human and Mammalian Toxic Equivalency Factors for Dioxins and Dioxin-like Compounds. Toxicological Sciences 93(2), 223-241 (2006)

11. Annex

(Please double click on the pdf-icons to open the annexes.)

Baby Food – 2101-BF		
1	Assigned values – PCDD/F, PCB	
2	Participants' results – Tables – PCDD/F, PCB	
3	Participants' z-scores / bioassay-scores – Tables – PCDD/F, PCB	
4	Participants' z-scores – Charts – PCDD/F, PCB	
5	Scoring system – PCDD/F, PCB	
6	Homogeneity and stability test – PCDD/F, PCB	
7	Participants' methods – PCDD/F, PCB	

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

Phone: +49 761 8855 500
E-Mail: eurl-pops@cvuafr.bwl.de