Bundesinstitut für Risikobewertung

Interlaboratory comparison exercise on the determination of bisphenol A (BPA), bisphenol S (BPS) and aluminium (Al) from cold and/or hot water extracts of recycled paper/cardboard FCM

Part A: Determination of BPA and BPS

Report on the interlaboratory comparison exercise NRL-DE-FCM-01/2020 of the German National Reference Laboratory (NRL) for Food Contact Materials

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1 Summary

The Interlaboratory comparison (ILC) exercise NRL-DE-FCM-01/2020 was organized by the German National Reference Laboratory for Food Contact Materials (NRL-FCM) established within the Unit Product Analytics of the Department of Chemicals and Product Safety at the German Federal Institute for Risk Assessment (BfR). The ILC aimed at assessing the analytical capabilities of Official Control Laboratories (OCLs) and NRLs in the determination of bisphenol A (BPA), bisphenol S (BPS) and aluminium (Al) from cold and/or hot water extracts (CWE; HWE) of recycled paper/cardboard FCM as well as in the determination of BPA, BPS and Al concentrations in provided solutions. The determination of BPA was mandatory whereas the determination of BPS was optional. Hence, not all laboratories submitted quantitative results for BPS. This part (A) discusses only the determination of BPA and BPS.

Recently the BfR recommendation regarding paper and cardboard FCM has been revised [1]. The limit values for the migration of BPA and Al into foodstuff were lowered to 0.05 and 1 mg per kg food or food simulant, respectively. This ILC was organized to assess if the analytical methods of the participating laboratories are capable to quantitatively detect BPA in the concentration range required by the amended recommendation.

The participating laboratories were asked to carry out cold and hot water extracts according to DIN EN 645:1994 [2] and DIN EN 647:1994 [3], respectively. Together with the extracts, two additionally provided solutions, which were mixtures of either CWE or HWE solutions, were to be analyzed for BPA and BPS.

In total, seventeen laboratories from eight EU Member States took part in this ILC. All laboratories reported results for BPA. One laboratory reported two results for BPA, which were measured with different detectors. Eleven laboratories reported quantitative results for BPS.

For the determination of BPA and BPS each participant received three samples and two already prepared extract solutions. The homogeneity of the samples and solutions as well as the stability of the solutions were evaluated by the German NRL-FCM beforehand.

The assigned values for BPA were derived as a robust average of the single results reported by the participants. Because of the small number of submitted BPS results, the estimation of assigned values from the submitted results according to the Q/Hampel method [4, 5] was not reasonable. Therefore, the extract solutions provided by the participants were analyzed for BPS at the German NRL-FCM and the results were used to determine the corresponding assigned values.

The statistical evaluation of the ILC was done using z, z', and ζ scores in accordance with ISO 13528:2015 [5]. Based on expert judgment, relative standard deviations for proficiency assessment (σ_{pt}) were set to 20 % of the assigned values for extracts and to 15 % for the provided solutions.

A very good performance was observed for the analysis of BPA in both, solutions and extracts. All participating laboratories performed satisfactorily (according to the z score) for the analysis of BPA in the provided solutions. For BPA analyses in extracts, more than 89 % of the laboratories achieved acceptable z scores. Acceptable ζ scores were obtained by more than 67 % of the laboratories.

Most laboratories performed well for the analysis of BPS in both hot and cold water extracts, with more than 70 % obtaining acceptable z scores. However, the evaluation of ζ scores revealed that some laboratories had trouble to obtain results covering the assigned value within their reported measurement uncertainties. Up to 64 % of ζ scores were either questionable or unacceptable. Additionally, the evaluation of the reported results revealed that the analytical

methods used for the determination of BPS should be improved in some of the participating laboratories.

In general, it could be clearly demonstrated that the cold and hot water extracts according to DIN EN 645:1994 [2] and DIN EN 647:1994 [3] as well as the analytical methods of all participating laboratories work well for the determination of BPA regarding the new limit value for migration of 0.05 mg/kg.

2 Introduction

The inter-laboratory comparison (ILC) exercise on the determination of bisphenol A (BPA), bisphenol S (BPS) and aluminum (Al) from cold and/or hot water extracts of commercially available recycled paper/cardboard food contact materials was organized by the German National Reference Laboratory for Food Contact Materials (NRL-FCM) established within the Unit Product Analytics of the Department of Chemicals and Product Safety at the German Federal Institute for Risk Assessment (BfR). The determination of BPA was mandatory whereas the determination of BPS and Al was optional. Cold and/or hot water extracts should be prepared according to DIN EN 645:1994 [2] and DIN EN 647:1994 [3], respectively. Additionally, the concentrations of BPA and BPS in two provided solutions were to be determined.

In the BfR recommendation XXXVI. regarding paper and cardboard FCM [1] the limit value for the migration of BPA into foodstuff has been lowered to 0.05 mg/kg. Hence, a major aspect of this ILC was to assess if the analytical methods of the Official Control Laboratories (OCLs) are already capable to quantify BPA at concentrations at or below the revised limit value for the migration into food.

For the determination of BPA and BPS each participant received:

- Sample 1: cardboard dish for hot and cold water extract
- Sample 2: pizza box for hot and cold water extract
- Sample 3: paper tissue for cold water extract
- Solution 1: aqueous solution containing BPA and BPS
- Solution 2: aqueous solution containing BPA and BPS

The solutions were prepared in the laboratories of the German NRL-FCM. Solution 1 was a mixture of 24 cold water extracts (CWE) from sample 1 and 2 (12 each) and solution 2 was a mixture of 24 hot water extracts (HWE) from sample 1 and 2 (12 each, 5 times diluted with Milli-Q water). The individual extracts were used to determine the homogeneity of samples 1 and 2 beforehand.

For an additional study, the participants were asked to send undiluted aliquots (~15 ml) of each of the respective extracts to the German NRL-FCM. These solutions were subsequently analyzed on a single analytical instrument, allowing the comparison of the extracts without the influence of the laboratory bias and precision differences.

This proficiency test was open to OCLs and NRLs. The seventeen laboratories listed here are kindly acknowledged for their participation in the ILC exercise. The laboratory codes were allocated randomly to the participants and do not correspond to the alphabetical order shown here.

Table 1: Participating laboratories

Organization	Country
Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit (LGL) (OCL)	Germany
Bundesinstitut für Risikobewertung (BfR) (NRL)	Germany
Centro Nacional Alimentacion (CNA) – Agencia Española de Seguridad Alimentaria y Nutrición (AESAN) (NRL)	Spain
Chemisches und Veterinäruntersuchungsamt (CVUA) Stuttgart (OCL)	Germany
Chemisches und Veterinäruntersuchungsamt Münsterland-Emscher-Lippe (CVUA-MEL) (OCL)	Germany
Escola Superior de Biotecnologia, Universidade Católica Portuguesa (NRL)	Portugal
General Chemical State Laboratory, 2nd Chemical Service of Athens Food Contact Materials Laboratory (NRL)	Greece
Health Board Central Chemistry Laboratory (NRL)	Estonia
Landesamt für Verbraucherschutz (LAV) Sachsen-Anhalt (OCL)	Germany
Landesbetrieb Hessisches Landeslabor (LHL) (OCL)	Germany
Landeslabor (LL) Schleswig-Holstein (OCL)	Germany
Landesuntersuchungsamt (LUA) Rheinland-Pfalz (OCL)	Germany
Landesuntersuchungsanstalt für das Gesundheits- und Veterinärwesen (LUA) Sachsen (OCL)	Germany
National Institute of Public Health (SZU) (NRL)	Czech Republic
National Laboratory of Health, Environment and Food (NRL)	Slovenia
The Public Analysts Laboratory Sir Patrick Dun's Hospital (NRL)	Ireland
Zentrales Institut des Sanitätsdienstes der Bundeswehr München (UA Bundeswehr) (OCL)	Germany

This report summarizes the outcome of the ILC exercise for the determination of BPA and BPS; results for AI have been described in part B of the report.

3 Scope

As stated in Regulation (EU) 2017/625 [6] one of the core duties of NRLs is to organize ILCs and proficiency tests for OCLs. The present ILC aims to assess the analytical capabilities of OCLs and NRLs on the analysis of cold and hot water extracts of paper/cardboard FCM. The participants were asked to carry out cold and/or hot water extracts of recycled paper/cardboard FCM and to analyze provided solutions containing BPA and BPS.
This ILC is identified as “NRL-DE-FCM-01/2020”.

4 Set up of the exercise

4.1 Time frame of the ILC

The ILC NRL-DE-FCM-01/2020 was announced on January 29, 2020. Registration was open until February 21, 2020. Samples were sent to the participants on February 28, 2020 and the deadline for reporting the results was set to April 09, 2020. This deadline was extended due to the COVID-19 lockdown until May 10, 2020. The last results were received on May 26, 2020, but two laboratories asked for a further extension of the deadline until the end of June. Unfortunately, due to COVID-19 restrictions they did not submit any results. One laboratory submitted results on the determination of BPS after the preliminary report had been sent to the participants.

4.2 Quality assurance

The German NRL-FCM is accredited according to: ISO/IEC 17025 [7] (certificate number: D-PL-18583-02). The reported results were evaluated following the relevant administrative and logistic procedures.

4.3 Confidentiality

The procedures used for the organization of this ILC guarantee that the identity of the participants and the information provided by them is treated confidentially. The participants in this ILC were assigned with a random and unique laboratory code used throughout this report.

4.4 Distribution

Each participant received:

- Sample 1 (cardboard dish)
- Sample 2 (pizza box)
- Sample 3 (paper tissue)
- 2 solutions (solutions no1 and no2)
- NRL_DE_FCM_01_2020_Confirmation of receipt.pdf
- NRL_DE_FCM_01_2020_Instructions.pdf
- NRL_DE_FCM_01_2020_Questionnaire_Results.xlsx

4.5 Instructions to participants

Detailed instructions to the participants were given in the “NRL_DE_FCM_01_2020_Instructions.pdf” (see 12.1 Instructions).

Participants were asked to check and report whether the test items were undamaged after transport using the “NRL_DE_FCM_01_2020_Confirmation of receipt.pdf” form.

The questionnaire form is divided into three sheets: “General”, “C&HWE” (cold and hot water extracts), and “Results”. The sheet “General” contains questions about the analytical methods. The detailed information about the experimental procedure is requested in the sheet “C&HWE”. In the sheet “Results” the single results should be reported together with the corresponding measurement uncertainty (MU) and the coverage factor (*k*).

Participants were asked to determine the concentrations of BPA and BPS in cold and/or hot water extracts of recycled paper/cardboard FCM according to DIN EN 645:1994 [2] and

DIN EN 647:1994 [3], respectively, as well as in the provided solutions. Additionally, participants were asked to provide aliquots of each of the respective extracts (~15 ml).

5 Test items

5.1 Preparation

5.1.1 Recycled paper/cardboard FCM

Commercially available cardboard dishes, pizza boxes and paper tissues were cut in 1 cm^2 pieces according to DIN 645 and 647 [2, 3], stored in a wide neck barrel and mixed by manual shaking. The samples (samples 1 and 2: 70 g; sample 3: 10 g) were prepared from these individual mixtures, wrapped in aluminum foil and sent to the participants.

5.1.2 Solutions

Solution 1 was a mixture of 24 cold water extracts from samples 1 and 2 (12 each) and solution 2 was a mixture of 24 hot water extracts from samples 1 and 2 (12 each, diluted 5-fold with Milli-Q water). 20 ml of each solution were filled into glass vials and sent to the participants.

5.2 Homogeneity and stability

Investigations for the homogeneity and stability studies and the statistical treatment of data were performed by the German NRL-FCM. The homogeneity assessment of the samples and solutions was performed after the preparation of the test items and before distribution to the participants. Results were evaluated according to ISO 13528:2015 [5]. The test items (samples) and solutions were proved to be adequately homogeneous (see 12.2 Homogeneity and stability of the samples and solutions).

The stability of the solutions was tested by the German NRL-FCM over a period of 158 days, which was longer than the timeframe of the ILC. The solutions were stable according to ISO 13528:2015 [5] (see 12.2 Homogeneity and stability of the samples and solutions). Because BPA and BPS are compounds with negligible volatilities and their stability in solution was confirmed, samples 1–3 were considered to be stable by expert judgement.

6 Assigned values and standard uncertanties

For the analysis of BPA the assigned value x_{pt} was derived as a robust average (according to the Q/Hampel method [4, 5]) of the single results reported by the participants. The standard uncertainty of the assigned value was estimated as:

$$u(x_{pt}) = 1.25 \frac{s^*}{\sqrt{p}} \qquad \text{Equation 1}$$

where s^* is the robust standard deviation of mean values of the results reported by the participants (according to the Q/Hampel method [4, 5]) and p is the number of participants. The values of σ_{pt} were set to 20 % for the extraction experiments and to 15 % for the analysis of solutions by perception of experts.

Table 2 presents the relevant BPA parameters needed for scoring.

Table 2: Assigned ranges related to the determination of BPA in extracts and solutions.

Test	x_{pt} ± $U(x_{pt})$* [µg L^{-1}]	σ_{pt} [µg L^{-1}]	[% of x_{pt}]	$u(x_{pt})/\sigma_{pt}$
Cold water extract (CWE)				
Sample 1	23.63 ± 1.83	4.73	20	0.19
Sample 2	79.36 ± 6.37	15.87	20	0.20
Sample 3	49.70 ± 2.88	9.94	20	0.15
Hot water extract (HWE)				
Sample 1	120.69 ± 10.13	24.14	20	0.21
Sample 2	258.31 ± 32.22	51.66	20	0.31
Solutions				
Solution 1	44.12 ± 2.32	6.62	15	0.18
Solution 2	41.78 ± 1.82	6.27	15	0.15

* $U(x_{pt})$ is the expanded uncertainty at a given coverage factor (k = 2)

Because of the small number of submitted BPS results, the estimation of x_{pt} and $u(x_{pt})$ values from the submitted results was not reasonable. Therefore, the extract solutions provided by the participants were analyzed for BPS at the German NRL-FCM and the results were used for the assessment of x_{pt} (as a robust average according to the Q/Hampel method [4, 5]) and $u(x_{pt})$ (according to Equation 1). The assigned value for BPS and its standard uncertainty for solutions 1 and 2 were derived as a robust average (according to the Q/Hampel method [4, 5]) of the single results reported by the participants. The standard uncertainty of the assigned value was estimated according to Equation 1. The relevant BPS parameters are summarized in Table 3.

Table 3: Assigned ranges related to the determination of BPS in extracts and solutions.

Test	x_{pt} ± $U(x_{pt})$* [µg L^{-1}]			σ_{pt} [µg L^{-1}]	σ_{pt} [% of x_{pt}]	$u(x_{pt})/\sigma_{pt}$
Cold water extract (CWE)						
Sample 1	11.25	±	0.72	2.25	20	0.16
Sample 2	53.21	±	2.40	10.64	20	0.11
Sample 3	8.30	±	0.50	1.66	20	0.15
Hot water extract (HWE)						
Sample 1	21.51	±	1.75	4.30	20	0.20
Sample 2	72.81	±	4.58	14.56	20	0.16
Solutions						
Solution 1	30.18	±	4.44	4.53	15	0.49
Solution 2	9.36	±	3.03	1.40	15	1.08

* $U(x_{pt})$ is the expanded uncertainty at a given coverage factor (k = 2).

Since the results from LC-001 were received after the Preliminary Report was sent to the participants, these values were not included in the estimation of x_{pt}, σ_{pt}, *and* $u(x_{pt})$.

7 Evaluation

7.1 Scores and evaluation criteria

The individual laboratory performance was expressed in terms of *z* and *ζ* scores according to ISO 13528:2015 [5]. The *z* and *ζ* scores for the proficiency test results x_i were calculated as follows:

$$z_i = \frac{x_i - x_{pt}}{\sigma_{pt}} \qquad \text{Equation 2}$$

$$\zeta_i = \frac{x_i - x_{pt}}{\sqrt{u^2(x_i) + u^2(x_{pt})}} \qquad \text{Equation 3}$$

z' scores were used instead of *z* scores, when the proportion $u(x_{pt})/\sigma_{pt}$ was significantly higher than 0.3, which was the case for BPS in solutions 1 and 2.

$$z_i' = \frac{x_i - x_{pt}}{\sqrt{\sigma_{pt}^2 + u^2(x_{pt})}} \qquad \text{Equation 4}$$

where:

x_i	mean value, calculated from single values reported by the participant *i*
x_{pt}	assigned value
σ_{pt}	standard deviation for proficiency test assessment
$u(x_i)$	standard uncertainty of mean value from participant *i*
$u(x_{pt})$	standard uncertainty of the assigned value

The interpretation of the *z*, *z'* and *ζ* performance scores is done according to ISO 13528:2015 [5]:

$\lvert z_i \rvert \leq 2.00$	acceptable performance (green in Annex 12.3 Results of the ILC)
$2.00 < \lvert z_i \rvert < 3.00$	questionable performance (yellow in Annex 12.3 Results of the ILC)
$\lvert z_i \rvert \geq 3.00$	unacceptable performance (red in Annex 12.3 Results of the ILC)

The *z* score demonstrates the deviation between the participants' mean and assigned values in terms of the standard deviation for proficiency test assessment (σ_{pt}). The *z'* score is used instead of the *z* score if $u(x_{pt}) > 0.3\ \sigma_{pt}$. The *ζ* score is a modified *z* score that includes uncertainties of the participants' results and the assigned value. It can be used in addition to the *z* score in order to evaluate whether the participants' results are close to the assigned value within their reported uncertainty.

The standard measurement uncertainty of the laboratory $u(x_i)$ was calculated by dividing the reported expanded measurement uncertainty $U(x_i)$ by the reported coverage factor *k*.
In order to verify how reasonable the measurement uncertainty of the laboratory is, an additional assessment was performed for each $u(x_i)$. For this reason, a relative standard uncertainty of the mean value from participant "*i*" was calculated.

$$u(x_i)_{\%} = 100\% \left(\frac{u(x_i)}{x_i} \right) \qquad \text{Equation 5}$$

The values of $u(x_i)_{\%}$ were divided into three groups:

a: $u_{min\,\%} \leq u(x_i)_{\%} \leq u_{max\,\%}$ reasonable estimation of $u(x_i)_{\%}$

b: $u(x_i)_{\%} < u_{min\,\%}$ underestimation of $u(x_i)_{\%}$

c: $u(x_i)_{\%} > u_{max\,\%}$ overestimation of $u(x_i)_{\%}$

where:

$u_{min\,\%} = u(x_{pt})_{\%} = 100\% \left(\frac{u(x_{pt})}{x_{pt}}\right)$ is the minimum of the accepted relative standard uncertainty

$u_{max\,\%} = \sigma_{pt\,\%} = 100\% \left(\frac{\sigma_{pt}}{x_{pt}}\right)$ is the maximum of the accepted relative standard uncertainty

If $u(x_i)_{\%}$ is in the range between a minimum and a maximum of the allowed uncertainty (case "a") the laboratory standard uncertainty may be reasonably estimated.

If $u(x_i)_{\%}$ is smaller than $u_{min\%} = u(x_{pt})_{\%}$ (case "b") the laboratory standard uncertainty may be underestimated. However, the following should be taken into account. Because the values of $u(x_{pt})$ were derived from the robust standard deviation of the single results reported by the participants, these values include contributions from (in)homogeneity, transport, and (in)stability. Therefore, a relative standard uncertainty $u(x_i)_{\%}$ smaller than $u(x_{pt})_{\%}$ is possible and plausible if these contributions are significant.

If $u(x_i)_{\%}$ is larger than $u_{max\%} = \sigma_{pt\%}$ (case "c") the laboratory standard uncertainty may be overestimated. However, if $u(x_i)_{\%} > \sigma_{pt\%}$ but x_i agrees with x_{pt} within their respective expanded measurement uncertainties, then the measurement uncertainty is properly assessed. In this case, however, the usefulness of the corresponding z score for the performance evaluation may be questionable.

7.2 General observations

Seventeen laboratories from eight EU Member States participated in this ILC. All laboratories reported results for BPA. One laboratory reported two results for BPA which were measured with different detectors (labeled with 'a' and 'b'). Eleven laboratories reported results for BPS. Three laboratories did not report measurement uncertainties for BPA results.

Most laboratories used liquid chromatography (LC) coupled with fluorescence detectors (FLD) or tandem mass spectrometers (MS/MS) for the quantification of BPA. For the analysis of BPS either LC coupled with MS/MS or diode array detectors (DAD) was used. One laboratory used gas chromatography (GC) coupled with mass spectrometry (MS) for the analysis of BPA and BPS (see Table 4).

Table 4: Analytical techniques used in this ILC for the analysis of BPA and BPS

Technique	BPA	BPS
	No. of labs	No. of labs
LC-FLD	9	-
LC-MS/MS	6	6
LC-DAD	2	4
GC-MS	1	1

No obvious differences in the reported results were observed when different filter types (glass fiber or other) were used for the filtration of the cold and hot water extract solutions. The same

is true for the amount of water, which had to be added to fill the volumetric flask up to the mark, and whether or not the temperature of the HWE solution was controlled and adjusted during this process.

7.3 Laboratory results and scorings

7.3.1 Performance

A graphical overview of the individual laboratory performance expressed by *z*, *z'* and *ζ* scores is given in Figure 1 and Figure 2.

Figure 1: Overview of the laboratory performance according to *z* and *ζ* scores for the analysis of BPA. 18 laboratories reported results (*z* scores) but only 15 laboratories reported MUs (*ζ* scores). The numbers in the bars correspond to the number of laboratories assigned with the respective scoring.
CWE_S1: cold water extract of sample 1; CWE_S2: cold water extract of sample 2; CWE_S3: cold water extract of sample 3; HWE_S1: hot water extract of sample 1; HWE_S2: hot water extract of sample 2; S_1: solution 1; S_2: solution 2.

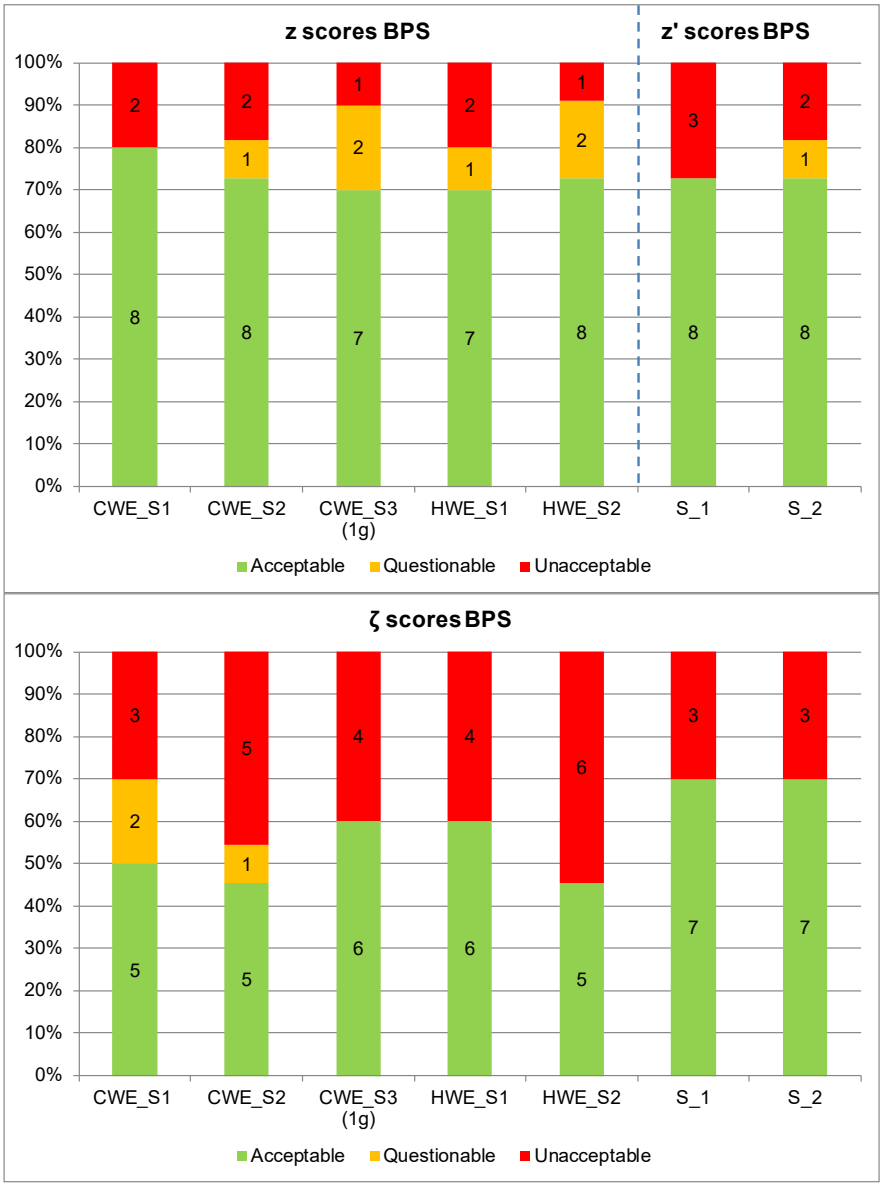

Figure 2: Overview of the laboratory performance according to *z*, *z'* and *ζ* scores for the analysis of BPS. The numbers in the bars correspond to the number of laboratories assigned with the respective scoring. CWE_S1: cold water extract of sample 1; CWE_S2: cold water extract of sample 2; CWE_S3: cold water extract of sample 3; HWE_S1: hot water extract of sample 1; HWE_S2: hot water extract of sample 2; S_1: solution 1; S_2: solution 2.

z and z' scores

100 % acceptable *z* scores were obtained for BPA in solutions 1 and 2. The *z* scores for the analysis of BPA in the cold water extracts were acceptable for 89–94 %, questionable for 6–11 % and unacceptable for 6 % of the participating laboratories. For the hot water extracts 94–100 % acceptable *z* scores were obtained.

The *z* scores for the analysis of BPS in the cold water extracts were in an acceptable range for 70–80 % of the laboratories. 9–20 % of the laboratories obtained questionable *z* scores whereas 10–20 % obtained unacceptable *z* scores. For the hot water extracts 70–73 % of the laboratories obtained acceptable *z* scores, 10–18 % were questionable and 9–20 % were unacceptable.

Since the proportion $u(x_{pt})/\sigma_{pt}$ was significantly higher than 0.3 for the BPS results of solutions 1 and 2, *z'* scores instead of *z* scores were calculated. 80 % of the laboratories received

acceptable *z'* scores, up to 10 % were questionable and 20–30 % were unacceptable *z'* scores.
As evident from the BPS results (Figure 2), some laboratories need to improve their analytical methods regarding this analyte. This can be assumed because the performance of the laboratories for the analysis of BPS in solutions 1 and 2 (extraction step is not included) was not significantly different from that for the analysis of BPS in the extracts.

ζ scores
73–80 % of the laboratories obtained acceptable *ζ* scores for the analysis of BPA in solutions 1 and 2. 13 % of the calculated *ζ* scores were found to be questionable and 13–20 % were unacceptable. Similar performances were observed for the results reported for the analysis of BPA in the cold water extracts of samples 1–3. 73–80 % of the calculated *ζ* scores were acceptable, 13–20 % were questionable and 7–20 % were unacceptable. 67–87 % of the *ζ* scores for the analysis of BPA in the hot water extracts were acceptable, 27 % were questionable and 7–13 % were unacceptable. 40 % of the laboratories reported all results (7 of 7) resulting in acceptable *ζ* scores. However, 27 % of the participants reported 3 (of 7) or more results with questionable and/or unacceptable *ζ* scores.

70 % of the *ζ* scores for the analysis of BPS in solutions 1 and 2 were acceptable and 30 % were unacceptable. For the analysis of BPS in the extracts the calculated *ζ* scores were even worse. Only 45–60 % of the *ζ* scores were acceptable for the cold water extracts, whereas 9–20 % were found to be questionable and 30–45 % were unacceptable. For the analysis of BPS in the hot water extracts 45–60 % of the *ζ* scores were acceptable and 40–55 % were unacceptable. 36 % of the laboratories reported results leading to acceptable *ζ* scores and 64 % of the laboratories obtained questionable and/or unacceptable *ζ* scores. A small majority of the laboratories (55 %) reported 3 (of 7) or more results resulting in questionable and/or unacceptable *ζ* scores. Two participants obtained unacceptable *ζ* scores for all results.

Taking the obtained *ζ* scores into consideration, some laboratory results were not close to the assigned value within their stated measurement uncertainty. Consequently, the determination of the measurement uncertainties should be checked and readjusted by these laboratories. It seems that a further harmonization for the determination of the MU – not only for BPS – is indispensable.

7.3.2 Measurement uncertainties (MU)

According to the questionnaire, the majority of the participants (88 %) usually provide uncertainty statements to their customers. Unfortunately, three laboratories did not report MUs for their results in this ILC, although they usually do so for their customers (according to the questionnaire). Most laboratories reported comparable relative MUs ($U(x_i)_{\%}$) for the extraction experiments and for the analysis of the solutions (see Table 5). Actually, due to the additional uncertainty from the extraction process itself, $U(x_i)_{\%}$ for the extraction experiments should be higher than that for the analysis of a solution.

Table 5: Calculated relative MUs $U(x_i)_{\%} = 100\% \left(\frac{u(x_i)}{x_i}\right)$. The values of $U(x_i)_{\%}$ are rounded to the nearest hundredths.

$U(x_i)_{\%}$							
BPA							
Lab. code	CWE_S1	CWE_S2	CWE_S3	HWE_S1	HWE_S2	Sol. 1	Sol. 2
LC-001	13	13	13	13	13	13	13
LC-003	12	12	12	12	12	12	12
LC-004	2	5	3	5	3	3	3
LC-005	14	17	17	19	16	6	6
LC-006	35	12	25	12	8	24	25
LC-007	19	19	19	19	19	19	19
LC-009	30	30	30	30	30	30	30
LC-010	24	24	24	24	24	24	24
LC-013	20	20	20	20	20	5	5
LC-014	20	20	20	20	20	20	20
LC-015	38	7	13	11	12	0*	3
LC-016	18	17	23	25	25	10	10
LC-017	25	25	25	25	25	25	25
LC-018	28	5	6	6	13	4	1
LC-019 a	30	29	30	29	29	29	30
BPS							
LC-001	17	17	17	17	17	17	17
LC-005	15	17	18	16	15	6	5
LC-006	-	36	-	-	36	-	-
LC-007	20	20	20	20	20	20	20
LC-008	4	5	6	5	5	5	4
LC-009	35	35	35	35	35	35	35
LC-010	9	9	9	9	9	9	9
LC-014	-	20	-	31	20	22	48
LC-015	31	12	8	12	7	2	3
LC-016	30	29	48	23	14	15	15
LC-019 a	33	33	33	32	32	32	33

* 0.499

Figure 3 depicts the evaluation of the reported measurement uncertainties for each experiment and laboratory. A detailed description of the evaluation criteria (case “a”, “b” and “c”) is given in section 7.1 Scores and evaluation criteria. In general, for the analysis of the extracts, the estimation of the measurement uncertainties was satisfactorily performed by most laboratories. For these experiments the fraction of reasonably estimated MUs was higher than 70 % for the analysis of both BPA and BPS. High fraction (>73 %) of reasonably estimated MUs was also reported for the analysis of BPA in the solutions. Due to the high value of $u(x_{pt})/\sigma_{pt}$, the evaluation of the reported measurement uncertainties for the analysis of BPS in solutions 1 and 2 was not reasonable.

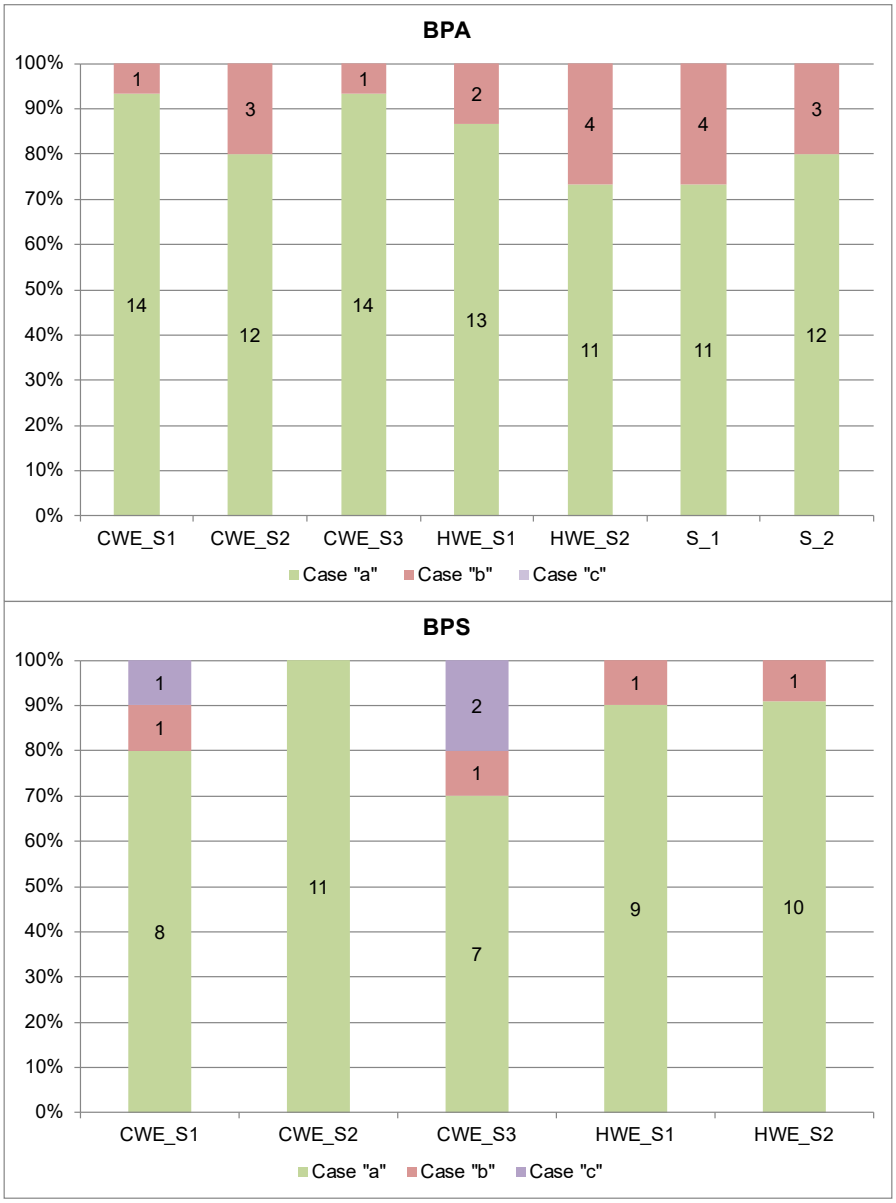

Figure 3: Evaluation of the reported measurement uncertainties for the analysis of BPA and BPS. The numbers in the bars correspond to the number of laboratories assigned to the respective cases.
Case "a": $u_{min\,\%} \leq u(x_i)_{\%} \leq u_{max\,\%}$;
Case "b": $u(x_i)_{\%} < u_{min\,\%}$;
Case "c": $u(x_i)_{\%} > u_{max\,\%}$;
CWE_S1: cold water extract of sample 1; CWE_S2: cold water extract of sample 2; CWE_S3: cold water extract of sample 3; HWE_S1: hot water extract of sample 1; HWE_S2: hot water extract of sample 2; S_1: solution 1; S_2: solution 2.

7.4 Additional information extracted from the questionnaire

The following information could be found in the questionnaire. All questions and answers are listed in 12.4 Results of the questionnaire.

General
All participating laboratories have a quality management system according to ISO 17025 [7]. Five laboratories used accredited analytical methods for the analysis of BPA and BPS and five validated methods, seven laboratories neither validated nor accredited their methods.

The analytical methods are used for less than one year in nine laboratories, and five of them do not usually use this analytical method. Only one laboratory uses it regularly (251–1000 times per year). In the remaining laboratories the method is used between 1–50 times per year. Quality control is performed with certified reference materials in seven out of seventeen laboratories.

The estimation of the measurement uncertainty is done by an in house validation in twelve laboratories, whereas one laboratory uses Nordtest [8] and three laboratories use other estimation models.

Cold and hot water extracts
Glass-fiber filters (size C) were used by eight laboratories for the filtration of both the cold and the hot water extracts, while four laboratories used glass-fiber filters with other sizes. Three laboratories used glass frits, one laboratory used folded cellulose filters, one laboratory did not filtered and one laboratory did not specify their filtration equipment.

Eight (CWE) or nine (HWE) laboratories needed 11–50 ml water, six laboratories needed more than 50 ml and one (HWE) or two (CWE) needed only 0–10 ml to fill the volumetric flask up to the mark. For the hot water extracts twelve laboratories controlled the solution temperature (23 ± 2 °C) before filling up the volumetric flasks. Four laboratories did not control the temperature. The extracts were warmed up by only two laboratories before taking a sample for analysis.

8 Conclusion

The overall performance, expressed in *z* scores, for the analysis of BPA was very good. All laboratories received acceptable *z* scores for the analysis of solutions 1 and 2 as well as for the hot water extract of sample 1. For the other extracts more than 89 % of the participating laboratories were assigned with acceptable *z* scores, with less than 11 % receiving questionable or unacceptable *z* scores. The performance, expressed in ζ scores, was also good. More than 67 % achieved acceptable ζ scores for the analysis of extracts and solutions, with 33 % receiving questionable or unacceptable ζ scores.

The evaluation of the BPS results indicated that some laboratories had difficulties with the analysis of this analyte. First, the overall performance was not as good as for BPA, with up to 30 % of the laboratories obtaining either questionable or unacceptable *z* scores. Second, for solutions 1 and 2 the proportion $u(x_{pt})/\sigma_{pt}$ was significantly higher than 0.3. This may be attributed to the small number of submitted BPS results and the occurrence of outliers. Since outliers were not considered for the evaluation, the number of available values for the statistical analysis was very limited. Therefore, *z'* scores had to be calculated instead of the conventional *z* scores. 80 % of the laboratories received acceptable *z'* scores, up to 10 % were questionable and 20–30 % were unacceptable *z'* scores. Additionally, the evaluation of ζ scores revealed that some laboratories had trouble to obtain results close to the assigned value within their reported measurement uncertainties. Up to 55 % of the ζ scores were either questionable or unacceptable. However, it has to be considered that at the time of the ILC, seven laboratories did not have an accredited or validated method available for the determination of BPS, and five out of these seven did not use their method routinely.

Most laboratories correctly estimated the measurement uncertainties for the analysis of BPA and BPS in the extracts as well as for BPA in the provided solutions. Contrarily, u_i *est.* could not be determined for BPS in the solutions because of the high $u(x_{pt})_{\%}$ values. Most laboratories reported comparable relative measurement uncertainties $U(x_i)_{\%}$ for both the analysis of extracts and the analysis of the provided solutions. However, due to the additional uncertainty from the extraction step, $U(x_i)_{\%}$ for the extraction experiments (including the analysis of the solution) is higher than that for the analysis of a solution only.

Consequently, the results of this ILC revealed that all participating laboratories have well working analytical methods for the quantification of BPA. It is worth highlighting that these analytical methods cover BPA concentrations that correspond to the newly recommended limit value of 0.05 mg/kg food/food simulant and below. Furthermore, the ILC clearly confirmed that the cold and hot water extracts work very well. For some laboratories, the analytical methods used to quantify BPS still require further optimization. However, the comparison of results from the analyses of extracts and provided solutions indicate that this is rather an issue with the instrumental analysis than with the extraction procedure, pointing to the suitability of the cold and hot water extracts for the quantification of BPS migration. This suitability for the determination of BPS is also confirmed by the additional analysis of the provided aliquots, which yielded only small reproducibility standard deviations of 8–12 % (compared to 33–60 % in the ILC). Details on this will be published elsewhere.

9 References

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12 Annex

12.1 Instructions

Please perform cold (DIN EN 645) and hot water extracts (DIN EN 647) as specified below. Analyze the three additionally provided solutions together with the extracts. Please determine bisphenol A (BPA) and bisphenol S (BPS) for solutions no 1 and no 2 provided in glass vials and aluminium (Al) for solution no 3 provided in the plastic tube.
Solution no 1 and no 2 are aqueous, solution no 3 is aqueous with 1 % HNO_3.

For added value of this entire study, we would appreciate if you could send us an aliquot (~15 ml) of each of the respective extracts. In consequence, we will examine all incoming solutions in one sequence with our LC-MS/MS. With a growing dataset we expect to improve the data basis for the estimation of the measurement uncertainty for the estimation of BPA and BPS from cold and hot water extracts. To perform the same for Al we would welcome ~5 ml of the cold water extracts of sample no 1 in plastic tubes (sealed with Parafilm), please filtrate (syringe filter max 0.45 µm) and stabilize the extracts beforehand (according to DIN 12498).

Before starting the experiments please read the Questionnaire carefully so that you can answer all questions.

1. **Cold water extract of the commercial cardboard sample no 1 (cardboard dish) and no 2 (pizza box) according to DIN EN 645**

Please perform the cold water extracts according to DIN EN 645 in triplicate and perform a filtration, do **not** only decant the extract. We suggest using a glass-fibre filter (size C) instead of a glass frit. Please give the volume you used to wash and estimate (see questionnaire) the added volume (ml water) to finally fill the volumetric flask. Determine the BPA and BPS mass fractions in all extracts. For sample no 1 determine the Al mass fraction, additionally. Previous to the Al measurement we ask you to filtrate the extracts further by a syringe filter 0.2 µm. It is necessary to perform this filtration before stabilizing the solution (e. g. acidification according to DIN 12498).

2. **Hot water extracts of the commercial cardboard samples no 1 and 2 (cardboard dish and pizza box) according to DIN EN 647**

Please perform the hot water extracts according to DIN EN 647 in triplicate and perform a filtration, do **not** only decant the extract. We suggest using a glass-fibre filter instead of a glass frit. Please give the volume you used to wash and estimate (see questionnaire) how much ml water you had to add to finally fill the volumetric flask. Determine BPA and BPS mass fractions in the extracts.

3. **Cold water extracts of the commercial paper sample no 3 (paper tissue) according to a modified DIN EN 645 (cold water extract) method**

Please perform the cold water extracts according to DIN EN 645 in triplicate, in contrast to DIN EN 645 use only **1 g** of sample 3! Perform a filtration, do **not** only decant the extract. We suggest to use a glass-fibre filter (size C) instead of a glass frit. Please give the volume you used to wash and estimate (see questionnaire) how much ml water you had to add to finally fill the volumetric flask. Determine BPA and BPS mass fractions in the extracts and **do not** correct the value to 10 g.

12.2 Homogeneity and stability of the samples and solutions

12.2.1 Homogeneity assessment for sample 1

Table 6: Results of the homogeneity assessment for hot water extracts of sample 1 (5 times diluted with Milli-Q water). The analysis was performed in duplicate. The values are reported in [µg L^{-1}]. The results are evaluated according to ISO 13528:2015 (B.2.3) [5] using the expanded criterion ($\sqrt{c}$) to consider the actual sampling error and repeatability.

	BPA		BPS	
	Rep. 1	Rep. 2	Rep. 1	Rep. 2
1	31.35	25.20	4.03	4.02
2	24.91	22.88	4.08	3.55
3	23.78	24.50	3.77	4.24
4	26.47	24.07	4.47	4.15
5	23.56	26.02	4.16	4.34
6	28.42	24.07	4.13	3.85
7	26.52	23.53	4.17	3.85
8	28.09	27.79	3.70	3.89
9	27.61	29.57	4.20	4.31
10	30.46	26.87	4.02	4.03
11	28.53	22.95	3.85	4.22
12	25.26	23.52	3.63	4.20
Mean	26.08		4.04	
$s_{\bar{x}}$	1.81		0.16	
s_w	2.36		0.24	
s_s	0.72		0.00	
σ_{pt} (20 % of Mean)	5.22		0.81	
σ_{allow}	1.56		0.24	
F1	1.79		1.79	
F2	0.86		0.86	
σ^2_{allow}	2.45		0.06	
c	9.15		0.15	
$\sqrt{c}$	3.03		0.39	
$s_s \leq \sqrt{c}$	passed		passed	
Assessment	Homogenous		Homogenous	

Where:

$s_{\bar{x}}$	standard deviation of sample averages,
s_w	within-sample standard deviation,
s_s	estimate of between-sample standard deviation,
σ_{pt}	standard deviation for proficiency assessment,
σ_{allow}	$\sigma_{allow}=0.3\ \sigma_{pt}$; criterion of sufficient homogeneity,
F1, F2	factors for use in testing for sufficient homogeneity,
c	$c=F1\sigma^2_{allow}+F2s^2_w$; is used to expand the criterion to allow for the actual sampling error and repeatability.

12.2.2 Homogeneity assessment for sample 2

Table 7: Results of the homogeneity assessment for hot water extracts of sample 2 (5 times diluted with Milli-Q water). The analysis was performed in duplicate. The values are reported in [µg L^{-1}]. The results are evaluated according to ISO 13528:2015 (B.2.3) [5] using the expanded criterion ($\sqrt{c}$) to consider the actual sampling error and repeatability.

	BPA		BPS	
	Rep. 1	Rep. 2	Rep. 1	Rep. 2
1	41.97	43.93	11.95	15.47
2	56.52	55.10	12.82	16.30
3	59.80	49.29	12.84	17.27
4	57.24	51.70	14.39	20.53
5	66.33	48.85	12.58	15.82
6	53.58	56.86	16.09	16.75
7	57.01	56.01	15.23	18.58
8	61.11	54.65	15.93	18.44
9	50.07	46.80	14.83	17.67
10	62.35	62.01	15.99	16.81
11	50.96	54.68	14.44	16.12
12	58.74	52.31	19.02	16.98
Mean	54.49		15.95	
$s_{\bar{x}}$	4.86		1.37	
s_w	4.88		2.29	
s_s	3.43		0.00	
σ_{pt} (20 % of Mean)	10.90		3.19	
σ_{allow}	3.27		0.96	
F1	1.79		1.79	
F2	0.86		0.86	
σ^2_{allow}	10.69		0.92	
c	39.63		6.15	
$\sqrt{c}$	6.30		2.48	
$s_s \leq \sqrt{c}$	passed		passed	
Assessment	Homogenous		Homogenous	

Where:

$s_{\bar{x}}$	standard deviation of sample averages,
s_w	within-sample standard deviation,
s_s	estimate of between-sample standard deviation,
σ_{pt}	standard deviation for proficiency assessment,
σ_{allow}	$\sigma_{allow}=0.3\ \sigma_{pt}$; criterion of sufficient homogeneity,
F1, F2	factors for use in testing for sufficient homogeneity,
c	$c=F1\sigma^2_{allow}+F2s^2_w$; is used to expand the criterion to allow for the actual sampling error and repeatability.

12.2.3 Homogeneity assessment for sample 3

Table 8: Results of the homogeneity assessment for cold water extracts of sample 3. The analysis was performed in duplicate. The values are reported in [µg L^{-1}]. The results are evaluated according to ISO 13528:2015 (B.2.3) [5] using the expanded criterion ($\sqrt{c}$) to consider the actual sampling error and repeatability.

	BPA		BPS	
	Rep. 1	Rep. 2	Rep. 1	Rep. 2
1	39.53	45.47	6.47	8.3
2	45.45	49.73	6.39	7.42
3	46.01	39.86	7.1	8.74
4	48.88	49.53	8.14	8.01
5	43.42	46.43	7.74	7.28
6	48.95	43.17	7.79	6.88
7	42.31	43.66	6.34	7.12
8	46.63	47.1	6.74	7.09
9	46.51	43.78	6.83	7.58
10	45.96	47.75	6.99	7.13
11	44.14	46.14	7.25	6.11
12	48.39	53.27	6.04	7.38
Mean	45.92		7.20	
$s_{\bar{x}}$	2.54		0.46	
s_w	2.70		0.72	
s_s	1.67		0.00	
σ_{pt} (20 % of Mean)	9.18		1.44	
σ_{allow}	2.76		0.43	
F1	1.79		1.79	
F2	0.86		0.86	
σ^2_{allow}	7.59		0.19	
c	19.85		0.78	
$\sqrt{c}$	4.46		0.89	
$s_s \leq \sqrt{c}$	passed		passed	
Assessment	Homogenous		Homogenous	

Where:

$s_{\bar{x}}$	standard deviation of sample averages,
s_w	within-sample standard deviation,
s_s	estimate of between-sample standard deviation,
σ_{pt}	standard deviation for proficiency assessment,
σ_{allow}	$\sigma_{allow}=0.3\ \sigma_{pt}$; criterion of sufficient homogeneity,
F1, F2	factors for use in testing for sufficient homogeneity,
c	$c=F1\sigma^2_{allow}+F2s^2_w$; is used to expand the criterion to allow for the actual sampling error and repeatability.

12.2.4 Homogeneity assessment for solution 1

Table 9: Results of the homogeneity assessment for solution 1 (mixture of cold water extracts from samples 1 and 2). The analysis was performed in duplicate. The values are reported in [µg L^{-1}]. Results are evaluated according to ISO 13528:2015 [5].

	BPA		BPS	
	Rep. 1	Rep. 2	Rep. 1	Rep. 2
1	28.85	36.28	22.59	24.37
2	30.97	34.1	21.86	21.82
3	32.76	34.01	24.62	22.16
4	31.78	35.25	23.54	23.63
5	31.47	35.78	24.24	23.86
6	30.9	34.89	23.4	22.94
7	29.41	38.27	25.64	24.82
8	31.6	27.43	26.18	22.4
9	31.97	29.11	26.26	23.31
10	32.68	29.57	25.31	22.74
11	36.88	32.62	27.12	24.1
Mean	32.57		23.95	
$s_{\bar{x}}$	1.57		1.05	
s_w	3.34		1.49	
s_s	0.00		0.00	
σ_{pt} (15 % of Mean)	4.89		3.59	
σ_{allow}	1.47		1.08	
$s_s \leq \sigma_{allow}$	passed		passed	
Assessment	Homogenous		Homogenous	

Where: $s_{\bar{x}}$ standard deviation of sample averages,

s_w within-sample standard deviation,

s_s estimate of between-sample standard deviation,

σ_{pt} standard deviation for proficiency assessment,

σ_{allow} $\sigma_{allow}=0.3\ \sigma_{pt}$; criterion of sufficient homogeneity.

12.2.5 Homogeneity assessment for solution 2

Table 10: Results of the homogeneity assessment for solution 2 (mixture of hot water extracts from samples 1 and 2; 5 times diluted with Milli-Q water). The analysis was performed in duplicate. The values are reported in [µg L^{-1}]. Results are evaluated according to ISO 13528:2015 [5].

	BPA		BPS	
	Rep. 1	Rep. 2	Rep. 1	Rep. 2
1	33.66	34.02	8.02	7.07
2	30.69	33.74	7.84	7.53
3	31.77	32.12	8.26	7.17
4	33.14	34.46	7.45	6.83
5	31.92	34.21	7.51	6.75
6	31.54	32.68	7.38	7.17
7	30.43	34.2	7.87	6.68
8	31.47	30.13	7.83	7.02
9	35.03	32.43	7.8	6.67
10	33.64	31.96	8.32	6.7
11	35.6	33.08	8.02	7.11
Mean	32.81		7.41	
$s_{\bar{x}}$	1.06		0.21	
s_w	1.50		0.67	
s_s	0.00		0.00	
σ_{pt} (15 % of Mean)	4.92		1.11	
σ_{allow}	1.48		0.33	
$s_s \leq \sigma_{allow}$	passed		passed	
Assessment	Homogenous		Homogenous	

Where: $s_{\bar{x}}$ standard deviation of sample averages,

s_w within-sample standard deviation,

s_s estimate of between-sample standard deviation,

σ_{pt} standard deviation for proficiency assessment,

σ_{allow} $\sigma_{allow}=0.3\ \sigma_{pt}$; criterion of sufficient homogeneity.

12.2.6 Stability assessment for solution 1

Table 11: Results of the stability assessment of solution 1. The analysis was performed in duplicate (for d_0: BPA and BPS and for d_{158}: BPA) or in quadruplicate (d_{158}: BPS). The values are reported in [µg L^{-1}].

	BPA	BPS
Bottle ID	Mean	Mean
d_0	44.29	30.43
d_{158}	43.91	29.90
$\lvert d_0-d_{158}\rvert$	0.37	0.53
σ_{pt}	6.62	4.53
0,3 σ_{pt}	1.98	1.36
$\lvert d_0-d_{158}\rvert \leq 0.3\ \sigma_{pt}$	Passed	Passed
Assessment	Stable	Stable

Where: d_0 analysis in the beginning of the stability study,

d_{158} analysis in the end of the stability study,

σ_{pt} standard deviation for proficiency assessment.

12.2.7 Stability assessment for solution 2

Table 12: Results of the stability assessment of solution 2. The analysis was performed in duplicate (for d_0: BPA and BPS) or in quadruplicate (d_{158}: BPA and BPS). The values are reported in [µg L^{-1}].

	BPA	BPS
Bottle ID	Mean	Mean
d_0	38.77	10.36
d_{158}	38.20	10.72
$\lvert d_0-d_{158}\rvert$	0.57	0.36
σ_{pt}	6.27	1.40
0.3 σ_{pt}	1.88	0.42
$\lvert d_0-d_{158}\rvert \leq 0.3\ \sigma_{pt}$	Passed	Passed
Assessment	Stable	Stable

Where: d_0 analysis in the beginning of the stability study,

d_{158} analysis in the end of the stability study,

σ_{pt} standard deviation for proficiency assessment.

12.3 Results of the ILC

12.3.1 Results for the determination of BPA in cold water extract of sample 1

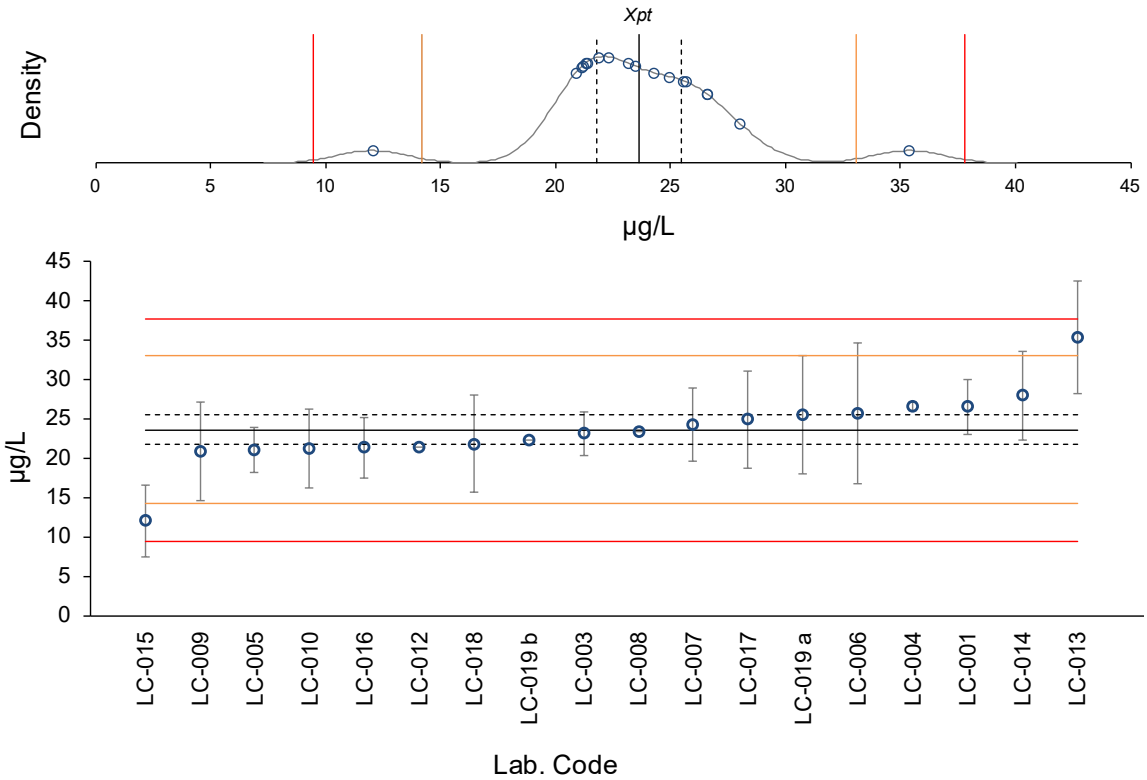

Figure 4: Measurement result range reported by the participants for the determination of BPA in cold water extract of sample 1. Circles and bars represent the reported results [x_i] with the corresponding expanded uncertainties [$U(x_i)$]; orange and red lines represent *z* scores = 2 and 3, respectively; solid and dotted black lines represent the assigned value [x_{pt}] and its expanded uncertainty [$U(x_{pt})$].

Table 13: Results for the determination of BPA in cold water extract of sample 1.
Assigned range: x_{pt} = 23.629 ± 1.830 µg L^{-1}; σ_{pt} = 4.726 µg L^{-1}; x_i and $U(x_i)$ values are reported in µg L^{-1}.

Lab. Code	x_i	$U(x_i)$	k	*z* score	ζ score	$u(x_i)$ est. §
LC-001	26.600	3.500	2	0.63	1.50	a
LC-003	23.133	2.767	2	-0.10	-0.30	a
LC-004	26.573	0.557	2	0.62	3.08	b
LC-005	21.090	2.910	2	-0.54	-1.48	a
LC-006	25.667	8.987	2	0.43	0.44	a
LC-007	24.272	4.612	2	0.14	0.26	a
LC-008	23.421	-	-	-0.04	-	-
LC-009	20.900	6.270	2	-0.58	-0.84	a
LC-010	21.200	5.033	2	-0.51	-0.91	a
LC-012	21.367	-	-	-0.48	-	-
LC-013	35.367	7.100	2	2.48	3.20	a
LC-014	28.000	5.678	2	0.92	1.47	a
LC-015	12.027	4.605	2	-2.46	-4.68	a
LC-016	21.333	3.794	2	-0.49	-1.09	a
LC-017	24.937	6.234	2	0.28	0.40	a
LC-018	21.863	6.184	1.96	-0.37	-0.54	a
LC-019 a	25.543	7.535	2	0.41	0.49	a
LC-019 b	22.297	-	-	-0.28	-	-

§ (a) Reasonable estimation of $u(x_i)$; (b) underestimation of $u(x_i)$; (c) overestimation of $u(x_i)$.

12.3.2 Results for the determination of BPA in cold water extract of sample 2

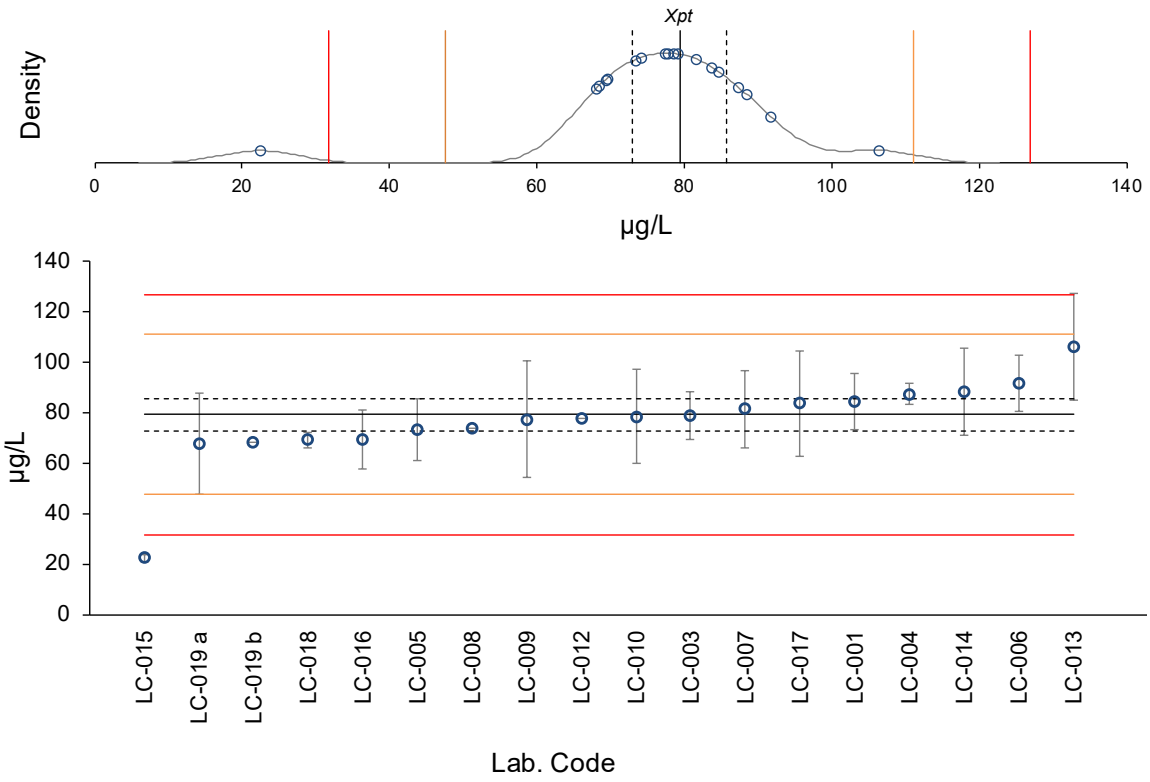

Figure 5: Measurement result range reported by the participants for the determination of BPA in cold water extract of sample 2. Circles and bars represent the reported results [x_i] with the corresponding expanded uncertainties [$U(x_i)$]; orange and red lines represent *z* scores = 2 and 3, respectively; solid and dotted black lines represent the assigned value [x_{pt}] and its expanded uncertainty [$U(x_{pt})$].

Table 14: Results for the determination of BPA in cold water extract of sample 2.
Assigned range: x_{pt} = 79.356 ± 6.368 µg L^{-1}; σ_{pt} = 15.871 µg L^{-1}; x_i and $U(x_i)$ values are reported in µg L^{-1}.

Lab. Code	x_i	$U(x_i)$	k	*z* score	ζ score	$u(x_i)$ *est.* §
LC-001	84.533	11.200	2	0.33	0.80	a
LC-003	78.967	9.466	2	-0.02	-0.07	a
LC-004	87.330	4.103	2	0.50	2.11	b
LC-005	73.285	12.239	2	-0.38	-0.88	a
LC-006	91.667	11.006	2	0.78	1.94	a
LC-007	81.502	15.485	2	0.14	0.26	a
LC-008	74.107	-	-	-0.33	-	-
LC-009	77.400	23.220	2	-0.12	-0.16	a
LC-010	78.500	18.667	2	-0.05	-0.09	a
LC-012	77.667	-	-	-0.11	-	-
LC-013	106.333	21.267	2	1.70	2.43	a
LC-014	88.333	17.312	2	0.57	0.97	a
LC-015	22.400	1.471	2	-3.59	-17.43	b
LC-016	69.467	11.610	2	-0.62	-1.49	a
LC-017	83.692	20.923	2	0.27	0.40	a
LC-018	69.233	3.140	1.96	-0.64	-2.84	b
LC-019 a	67.986	20.056	2	-0.72	-1.08	a
LC-019 b	68.450	-	-	-0.69	-	-

§ (a) Reasonable estimation of $u(x_i)$; (b) underestimation of $u(x_i)$; (c) overestimation of $u(x_i)$.

12.3.3 Results for the determination of BPA in cold water extract of sample 3

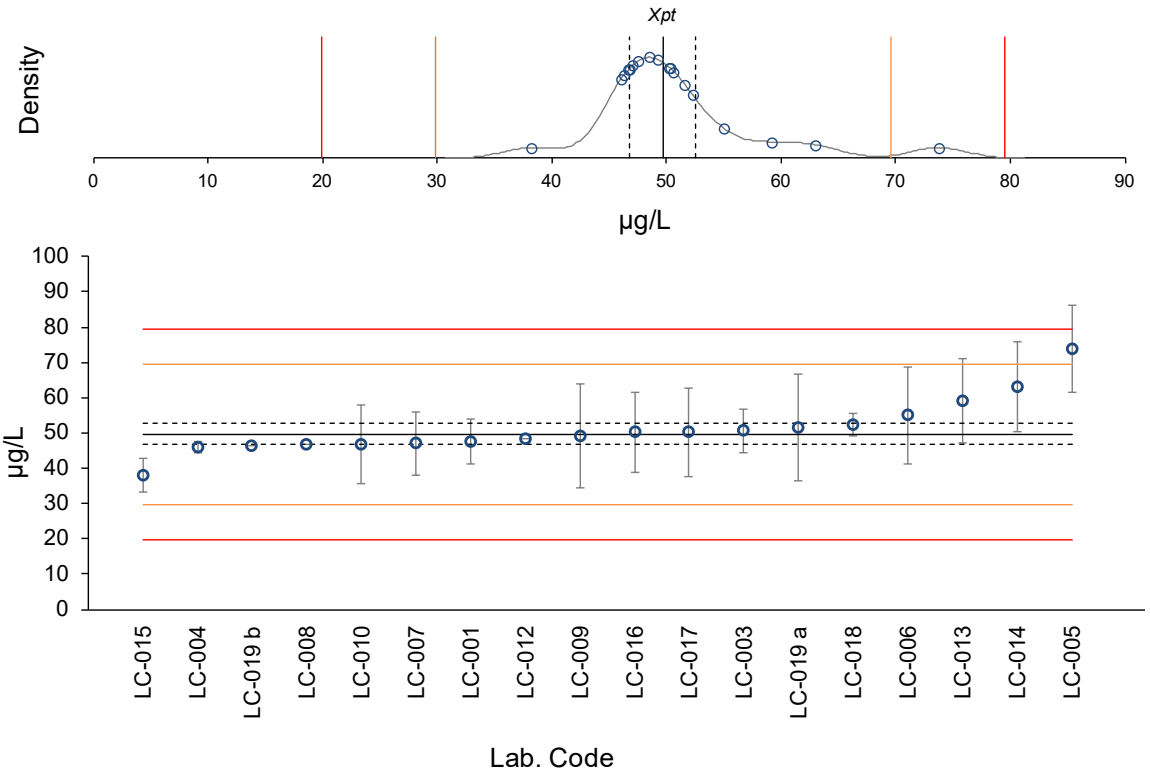

Figure 6: Measurement result range reported by the participants for the determination of BPA in cold water extract of sample 3. Circles and bars represent the reported results [x_i] with the corresponding expanded uncertainties [$U(x_i)$]; orange and red lines represent *z* scores = 2 and 3, respectively; solid and dotted black lines represent the assigned value [x_{pt}] and its expanded uncertainty [$U(x_{pt})$].

Table 15: Results for the determination of BPA in cold water extract of sample 3.
Assigned range: x_{pt} = 49.703 ± 2.883 µg L^{-1}; σ_{pt} = 9.941 µg L^{-1}; x_i and $U(x_i)$ values are reported in µg L^{-1}.

Lab. Code	x_i	$U(x_i)$	k	*z* score	ζ score	$u(x_i)$ *est.* §
LC-001	47.567	6.300	2	-0.21	-0.62	a
LC-003	50.633	6.067	2	0.09	0.28	a
LC-004	46.087	1.563	2	-0.36	-2.21	b
LC-005	73.771	12.320	2	2.42	3.80	a
LC-006	55.000	13.666	2	0.53	0.76	a
LC-007	47.031	8.936	2	-0.27	-0.57	a
LC-008	46.669	-	-	-0.31	-	-
LC-009	49.167	14.750	2	-0.05	-0.07	a
LC-010	46.733	11.133	2	-0.30	-0.52	a
LC-012	48.533	-	-	-0.12	-	-
LC-013	59.100	11.867	2	0.95	1.54	a
LC-014	63.000	12.686	2	1.34	2.04	a
LC-015	38.180	4.813	2	-1.16	-4.11	a
LC-016	50.233	11.336	2	0.05	0.09	a
LC-017	50.280	12.570	2	0.06	0.09	a
LC-018	52.270	3.122	1.96	0.26	1.19	a
LC-019 a	51.573	15.214	2	0.19	0.24	a
LC-019 b	46.306	-	-	-0.34	-	-

§ (a) Reasonable estimation of $u(x_i)$; (b) underestimation of $u(x_i)$; (c) overestimation of $u(x_i)$.

12.3.4 Results for the determination of BPA in hot water extract of sample 1

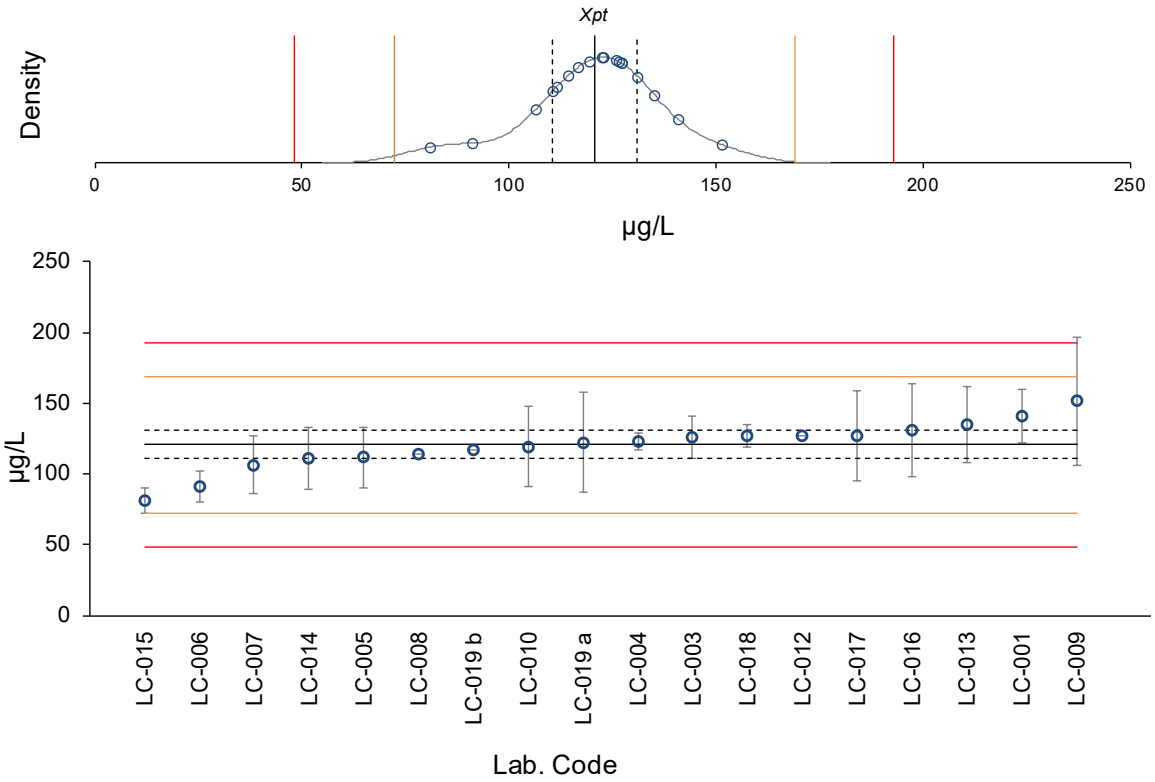

Figure 7: Measurement result range reported by the participants for the determination of BPA in hot water extract of sample 1. Circles and bars represent the reported results [x_i] with the corresponding expanded uncertainties [$U(x_i)$]; orange and red lines represent *z* scores = 2 and 3, respectively; solid and dotted black lines represent the assigned value [x_{pt}] and its expanded uncertainty [$U(x_{pt})$].

Table 16: Results for the determination of BPA in hot water extract of sample 1.
Assigned range: x_{pt} = 120.690 ± 10.131 µg L^{-1}; σ_{pt} = 24.138 µg L^{-1}; x_i and $U(x_i)$ values are reported in µg L^{-1}.

Lab. Code	x_i	$U(x_i)$	k	*z* score	ζ score	$u(x_i)$ *est.* §
LC-001	140.867	18.733	2	0.84	1.89	a
LC-003	126.000	15.004	2	0.22	0.59	a
LC-004	122.817	5.933	2	0.09	0.36	b
LC-005	111.578	21.534	2	-0.38	-0.77	a
LC-006	91.000	11.006	2	-1.23	-3.97	a
LC-007	106.454	20.226	2	-0.59	-1.26	a
LC-008	114.175	-	-	-0.27	-	-
LC-009	151.433	45.430	2	1.27	1.32	a
LC-010	119.333	28.400	2	-0.06	-0.09	a
LC-012	127.100	-	-	0.27	-	-
LC-013	135.000	27.000	2	0.59	0.99	a
LC-014	110.667	22.007	2	-0.42	-0.83	a
LC-015	80.947	8.721	2	-1.65	-5.95	a
LC-016	131.000	33.096	2	0.43	0.60	a
LC-017	127.148	31.787	2	0.27	0.39	a
LC-018	126.600	8.107	1.96	0.24	0.90	b
LC-019 a	122.305	35.224	2	0.07	0.09	a
LC-019 b	116.632	-	-	-0.17	-	-

§ (a) Reasonable estimation of $u(x_i)$; (b) underestimation of $u(x_i)$; (c) overestimation of $u(x_i)$.

12.3.5 Results for the determination of BPA in hot water extract of sample 2

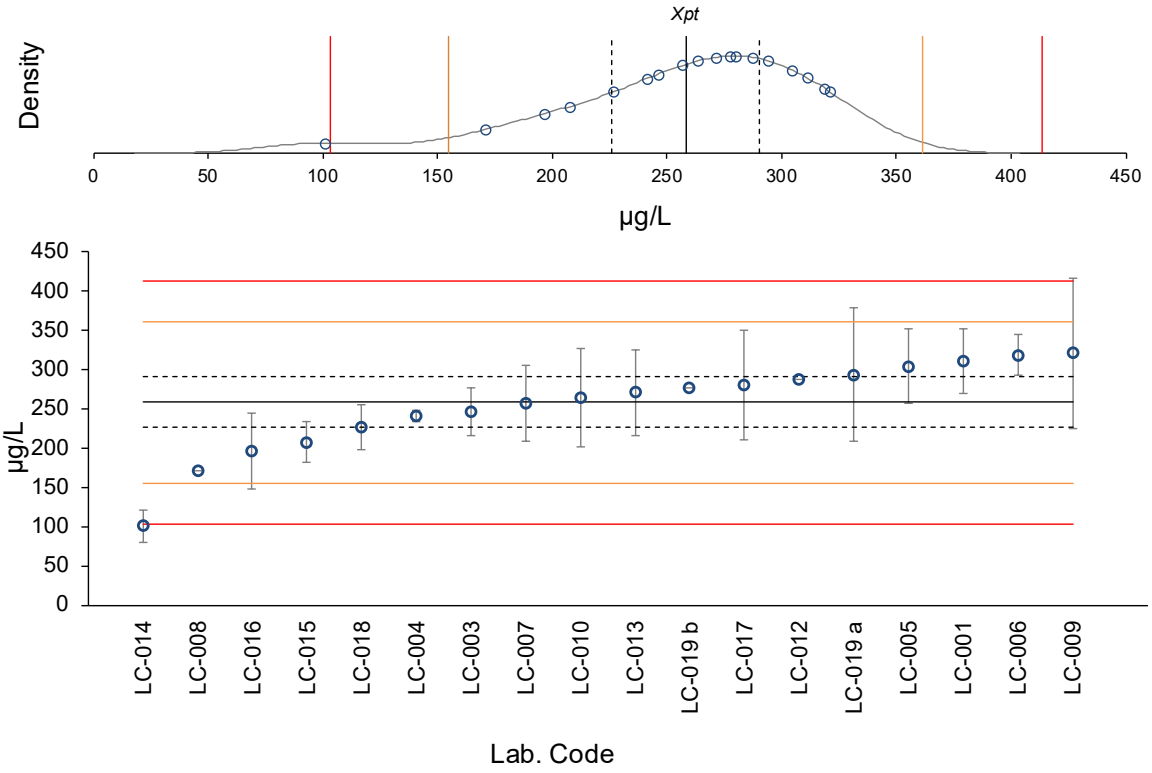

Figure 8: Measurement result range reported by the participants for the determination of BPA in hot water extract of sample 2. Circles and bars represent the reported results [x_i] with the corresponding expanded uncertainties [$U(x_i)$]; orange and red lines represent *z* scores = 2 and 3, respectively; solid and dotted black lines represent the assigned value [x_{pt}] and its expanded uncertainty [$U(x_{pt})$].

Table 17: Results for the determination of BPA in hot water extract of sample 2.
Assigned range: x_{pt} = 258.313 ± 32.221 µg L^{-1}; σ_{pt} = 51.663 µg L^{-1}; x_i and $U(x_i)$ values are reported in µg L^{-1}.

Lab. Code	x_i	$U(x_i)$	k	*z* score	ζ score	$u(x_i)$ est. §
LC-001	311.167	41.334	2	1.02	2.02	a
LC-003	246.333	29.662	2	-0.23	-0.55	b
LC-004	241.153	7.060	2	-0.33	-1.04	b
LC-005	304.470	47.497	2	0.89	1.61	a
LC-006	318.667	25.665	2	1.17	2.93	b
LC-007	256.740	48.781	2	-0.03	-0.05	a
LC-008	170.946	-	-	-1.69	-	-
LC-009	320.867	96.260	2	1.21	1.23	a
LC-010	263.533	62.734	2	0.10	0.15	a
LC-012	287.267	-	-	0.56	-	-
LC-013	271.000	54.200	2	0.25	0.40	a
LC-014	100.667	20.006	2	-3.05	-8.31	a
LC-015	207.717	25.640	2	-0.98	-2.46	b
LC-016	196.733	48.466	2	-1.19	-2.12	a
LC-017	279.908	69.977	2	0.42	0.56	a
LC-018	226.433	28.316	1.96	-0.62	-1.47	a
LC-019 a	293.750	84.600	2	0.69	0.78	a
LC-019 b	277.251	-	-	0.37	-	-

§ (a) Reasonable estimation of $u(x_i)$; (b) underestimation of $u(x_i)$; (c) overestimation of $u(x_i)$.

12.3.6 Results for the determination of BPA in solution 1

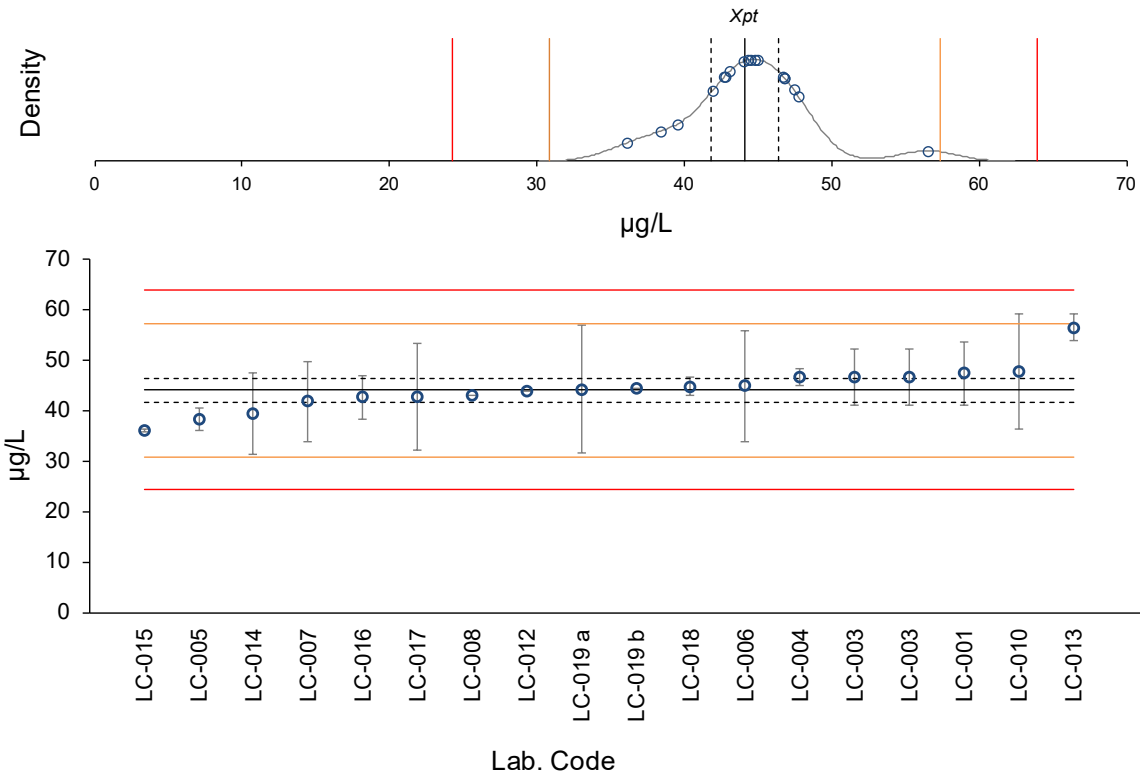

Figure 9: Measurement result range reported by the participants for the determination of BPA in solution 1. Circles and bars represent the reported results [x_i] with the corresponding expanded uncertainties [$U(x_i)$]; orange and red lines represent *z* scores = 2 and 3, respectively; solid and dotted black lines represent the assigned value [x_{pt}] and its expanded uncertainty [$U(x_{pt})$].

Table 18: Results for the determination of BPA in solution 1.
Assigned range: x_{pt} = 44.107 ± 2.316 µg L^{-1}; σ_{pt} = 6.616 µg L^{-1}; x_i and $U(x_i)$ values are reported in µg L^{-1}.

Lab. Code	x_i	$U(x_i)$	k	*z* score	ζ score	$u(x_i)$ est. §
LC-001	47.400	6.300	2	0.50	0.98	a
LC-003	46.800	5.600	2	0.41	0.89	a
LC-004	46.682	1.580	2	0.39	1.84	b
LC-005	38.400	2.227	2	-0.86	-3.55	a
LC-006	45.000	11.000	2	0.13	0.16	a
LC-007	41.896	7.960	2	-0.33	-0.53	a
LC-008	43.086	-	-	-0.15	-	-
LC-009	46.800	14.040	2	0.41	0.38	a
LC-010	47.700	11.400	2	0.54	0.62	a
LC-012	44.000	-	-	-0.02	-	-
LC-013	56.500	2.600	2	1.87	7.12	b
LC-014	39.500	8.000	2	-0.70	-1.11	a
LC-015	36.090	0.180	2	-1.21	-6.90	b
LC-016	42.700	4.270	2	-0.21	-0.58	a
LC-017	42.751	10.688	2	-0.20	-0.25	a
LC-018	44.800	1.770	1.96	0.10	0.47	b
LC-019 a	44.287	12.755	2	0.03	0.03	a
LC-019 b	44.511	-	-	0.06	-	-

§ (a) Reasonable estimation of $u(x_i)$; (b) underestimation of $u(x_i)$; (c) overestimation of $u(x_i)$.

12.3.7 Results for the determination of BPA in solution 2

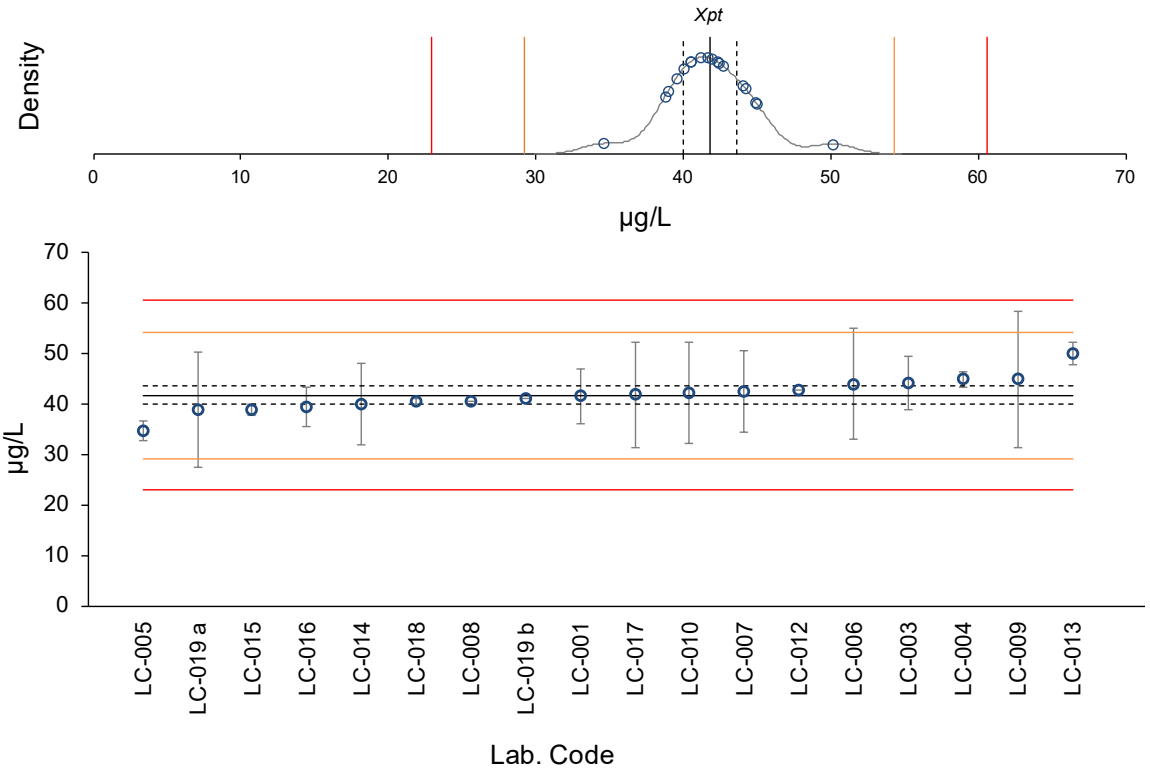

Figure 10: Measurement result range reported by the participants for the determination of BPA in solution 2. Circles and bars represent the reported results [x_i] with the corresponding expanded uncertainties [$U(x_i)$]; orange and red lines represent *z* scores = 2 and 3, respectively; solid and dotted black lines represent the assigned value [x_{pt}] and its expanded uncertainty [$U(x_{pt})$].

Table 19: Results for the determination of BPA in solution 2.
Assigned range: x_{pt} = 41.775 ± 1.817 µg L^{-1}; σ_{pt} = 6.266 µg L^{-1}; x_i and $U(x_i)$ values are reported in µg L^{-1}.

Lab. Code	x_i	$U(x_i)$	k	*z* score	ζ score	$u(x_i)$ est. [§]
LC-001	41.600	5.500	2	-0.03	-0.06	A
LC-003	44.200	5.300	2	0.39	0.87	a
LC-004	44.899	1.520	2	0.50	2.64	b
LC-005	34.600	1.938	2	-1.15	-5.40	a
LC-006	44.000	11.000	2	0.36	0.40	a
LC-007	42.398	8.056	2	0.10	0.15	a
LC-008	40.516	-	-	-0.20	-	-
LC-009	45.000	13.500	2	0.51	0.47	a
LC-010	42.300	10.100	2	0.08	0.10	a
LC-012	42.700	-	-	0.15	-	-
LC-013	50.100	2.300	2	1.33	5.68	a
LC-014	40.000	8.000	2	-0.28	-0.43	a
LC-015	39.000	1.110	2	-0.44	-2.61	b
LC-016	39.500	3.950	2	-0.36	-1.05	a
LC-017	41.901	10.475	2	0.02	0.02	a
LC-018	40.510	0.510	1.96	-0.20	-1.34	b
LC-019 a	38.769	11.437	2	-0.48	-0.52	a
LC-019 b	41.111	-	-	-0.11	-	-

[§] (a) Reasonable estimation of $u(x_i)$; (b) underestimation of $u(x_i)$; (c) overestimation of $u(x_i)$.

12.3.8 Results for the determination of BPS in cold water extract of sample 1

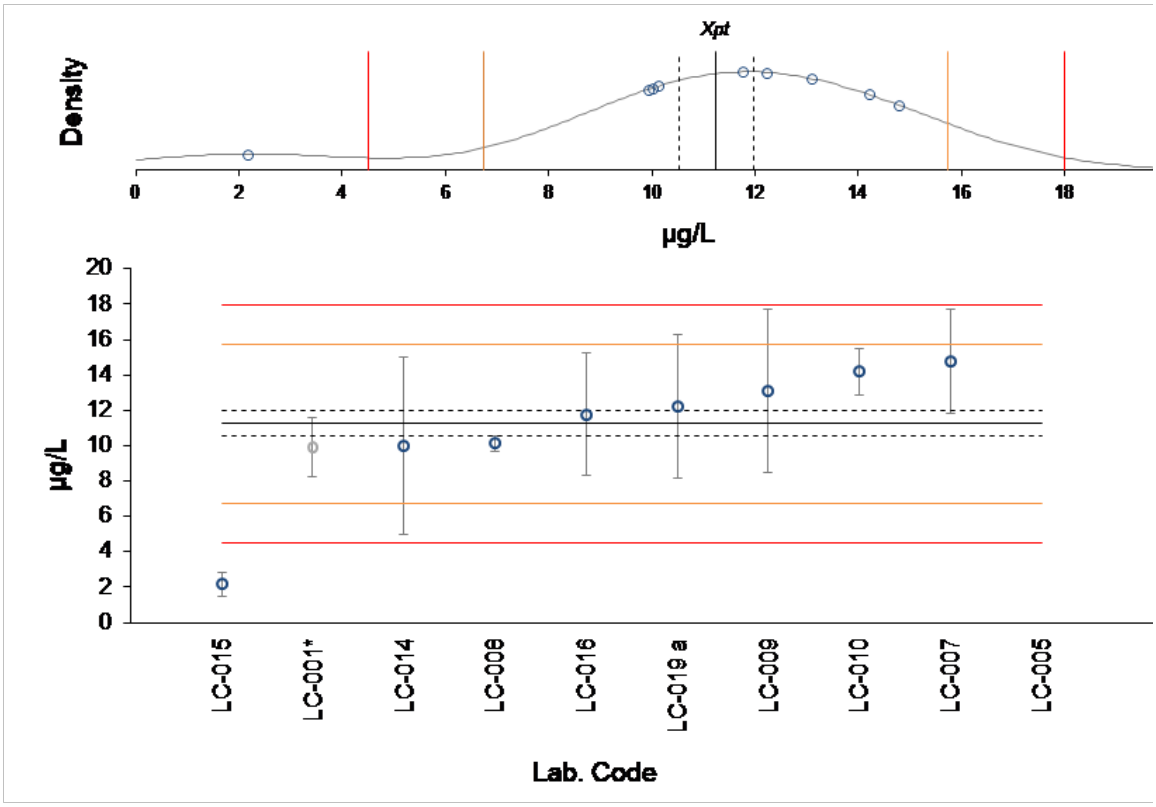

Figure 11: Measurement result range reported by the participants for the determination of BPS in cold water extract of sample 1. Circles and bars represent the reported results [x_i] with the corresponding expanded uncertainties [$U(x_i)$]; orange and red lines represent *z* scores = 2 and 3, respectively; solid and dotted black lines represent the assigned value [x_{pt}] and its expanded uncertainty [$U(x_{pt})$].
* The results were reported after the Preliminary Report was sent to the participants. These values were not included in the calculations of x_{pt} and σ_{pt}, this is indicated by the grey circle.

Table 20: Results for the determination of BPS in cold water extract of sample 1.
Assigned range: x_{pt} = 11.247 ± 0.722 µg L^{-1}; σ_{pt} = 2.249 µg L^{-1}; x_i and $U(x_i)$ values are reported in µg L^{-1}.

Lab. Code	x_i	$U(x_i)$	k	z score	ζ score	$u(x_i)$ est. §
LC-001*	9.933	1.667	2	-0.58	-1.45	a
LC-005	3021.989	461.357	2	1338.48	13.05	a
LC-007	14.773	2.955	2	1.57	2.32	a
LC-008	10.121	0.445	2	-0.50	-2.65	b
LC-009	13.100	4.585	2	0.82	0.80	a
LC-010	14.200	1.300	2	1.31	3.97	a
LC-014#	10.000	5.000	2	-0.55	-0.49	c
LC-015	2.166	0.680	2	-4.04	-18.30	a
LC-016	11.767	3.479	2	0.23	0.29	a
LC-019 a	12.226	4.034	2	0.44	0.48	a

* The results were reported after the Preliminary Report was sent to the participants. These values were not included in the calculations of x_{pt} and σ_{pt}.
# The values for x_i, $U(x_i)$ and k as well as the calculations of u_i *est.*, z and ζ scores are based on results reported for extract 1. For extracts 2 and 3 the reported x_i values are < 10 µg L^{-1} (LOQ).
§ (a) Reasonable estimation of $u(x_i)$; (b) underestimation of $u(x_i)$; (c) overestimation of $u(x_i)$.

12.3.9 Results for the determination of BPS in cold water extract of sample 2

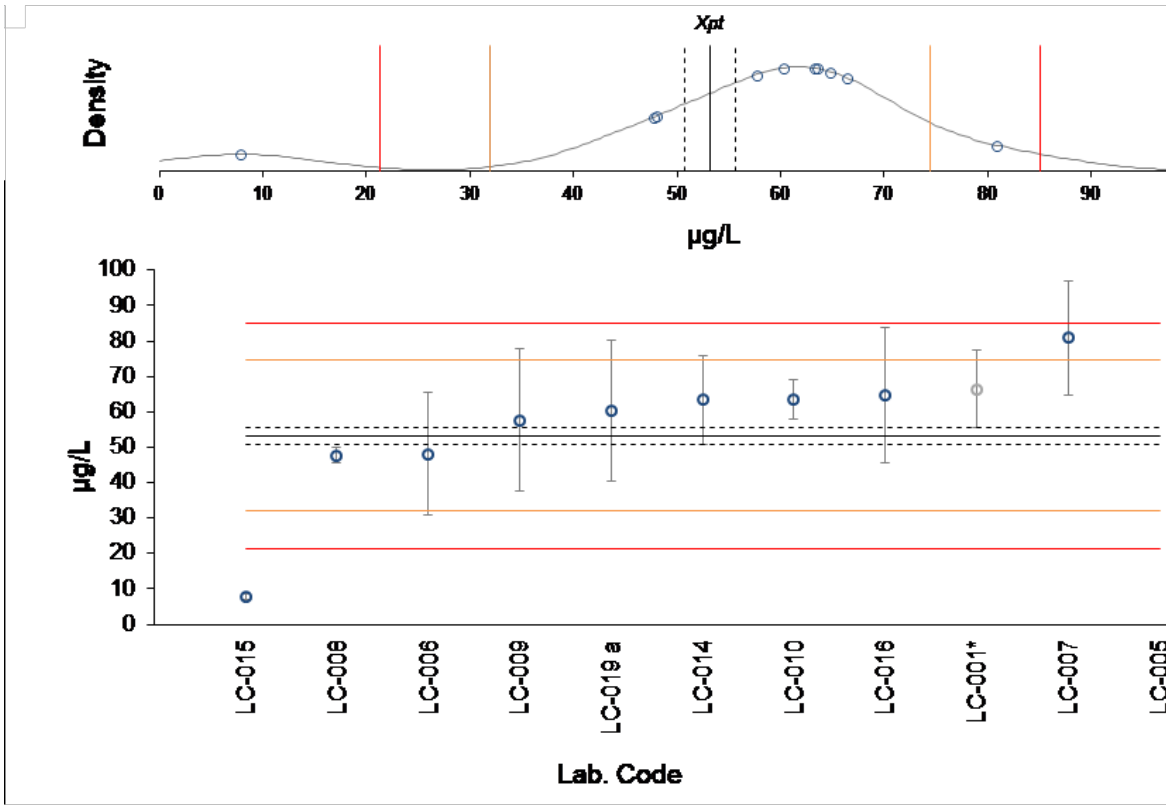

Figure 12: Measurement result range reported by the participants for the determination of BPS in cold water extract of sample 2. Circles and bars represent the reported results [x_i] with the corresponding expanded uncertainties [$U(x_i)$]; orange and red lines represent *z* scores = 2 and 3, respectively; solid and dotted black lines represent the assigned value [x_{pt}] and its expanded uncertainty [$U(x_{pt})$].
* The results were reported after the Preliminary Report was sent to the participants. These values were not included in the calculations of x_{pt} and σ_{pt}, this is indicated by the grey circle.

Table 21: Results for the determination of BPS in cold water extract of sample 2.
Assigned range: x_{pt} = 53.207 ± 2.400 µg L^{-1}; σ_{pt} = 10.641 µg L^{-1}; x_i and $U(x_i)$ values are reported in µg L^{-1}.

Lab. Code	x_i	$U(x_i)$	k	z score	ζ score	$u(x_i)$ est. §
LC-001*	66.400	11.033	2	1.24	2.34	a
LC-005	2547.703	445.848	2	234.42	11.19	a
LC-006	48.000	17.322	2	-0.49	-0.60	a
LC-007	80.835	16.167	2	2.60	3.38	a
LC-008	47.712	2.290	2	-0.52	-3.31	a
LC-009	57.667	20.183	2	0.42	0.44	a
LC-010	63.500	5.734	2	0.97	3.31	a
LC-014	63.333	12.664	2	0.95	1.57	a
LC-015	7.780	0.964	2	-4.27	-35.13	a
LC-016	64.833	19.041	2	1.09	1.21	a
LC-019 a	60.296	19.898	2	0.67	0.71	a

* The results were reported after the Preliminary Report was sent to the participants. These values were not included in the calculations of x_{pt} and σ_{pt}.
§ (a) Reasonable estimation of $u(x_i)$; (b) underestimation of $u(x_i)$; (c) overestimation of $u(x_i)$.

12.3.10 Results for the determination of BPS in cold water extract of sample 3

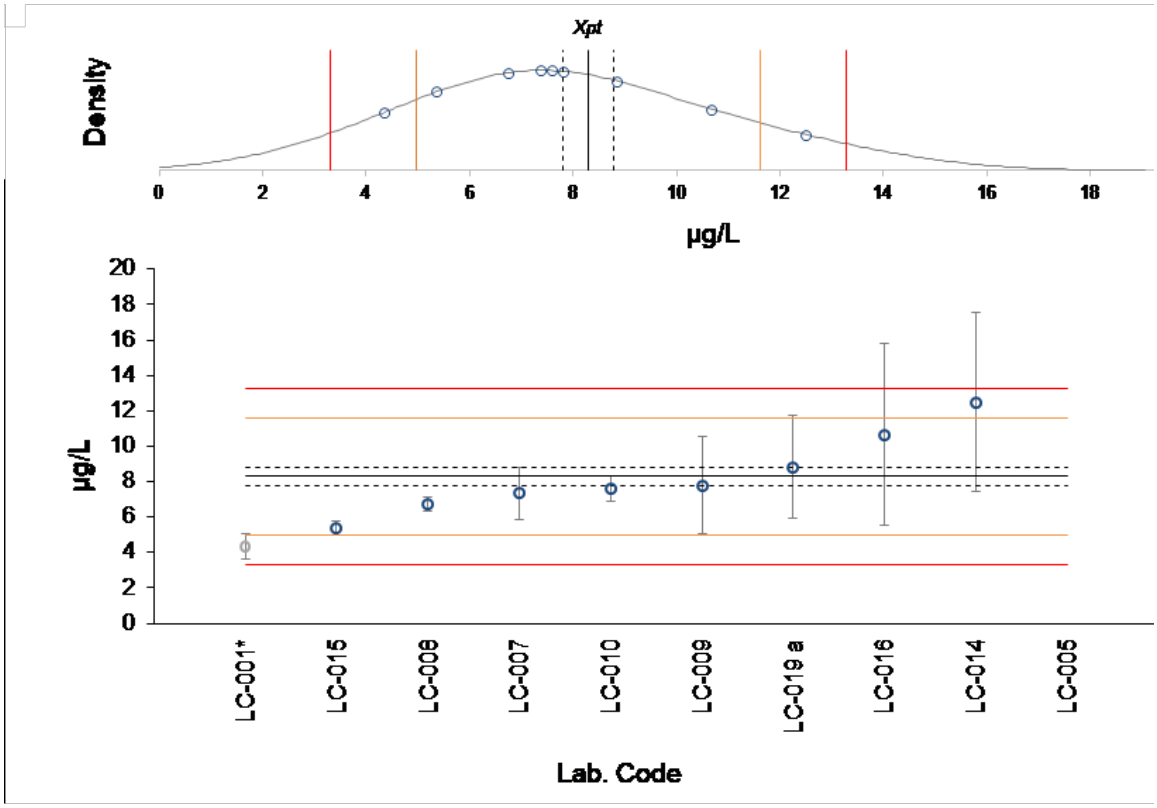

Figure 13: Measurement result range reported by the participants for the determination of BPS in cold water extract of sample 3. Circles and bars represent the reported results [x_i] with the corresponding expanded uncertainties [$U(x_i)$]; orange and red lines represent *z* scores = 2 and 3, respectively; solid and dotted black lines represent the assigned value [x_{pt}] and its expanded uncertainty [$U(x_{pt})$].
* The results were reported after the Preliminary Report was sent to the participants. These values were not included in the calculations of x_{pt} and σ_{pt}, this is indicated by the grey circle.

Table 22: Results for the determination of BPS in cold water extract of sample 3.
Assigned range: x_{pt} = 8.296 ± 0.500 µg L^{-1}; σ_{pt} = 1.659 µg L^{-1}; x_i and $U(x_i)$ values are reported in µg L^{-1}.

Lab. Code	x_i	$U(x_i)$	k	*z* score	ζ score	$u(x_i)$ est. §
LC-001*	4.333	0.733	2	-2.39	-8.93	a
LC-005	2545.659	445.490	2	1529.25	11.39	a
LC-007	7.359	1.472	2	-0.56	-1.21	a
LC-008	6.740	0.391	2	-0.94	-4.90	b
LC-009	7.800	2.730	2	-0.30	-0.36	a
LC-010	7.600	0.700	2	-0.42	-1.62	a
LC-014#	12.500	5.073	2	2.53	1.65	c
LC-015	5.363	0.410	2	-1.77	-9.07	a
LC-016	10.657	5.153	2	1.42	0.91	c
LC-019 a	8.841	2.918	2	0.33	0.37	a

* The results were reported after the Preliminary Report was sent to the participants. These values were not included in the calculations of x_{pt} and σ_{pt}.
# The values for xi, U(xi) and k as well as the calculations of ui est., z and ζ scores are based on results reported for extracts 1 and 2. For extract 3 the reported xi value is < 10 µg L-1 (LOQ).
§ (a) Reasonable estimation of $u(x_i)$; (b) underestimation of $u(x_i)$; (c) overestimation of $u(x_i)$.

12.3.11 Results for the determination of BPS in hot water extract of sample 1

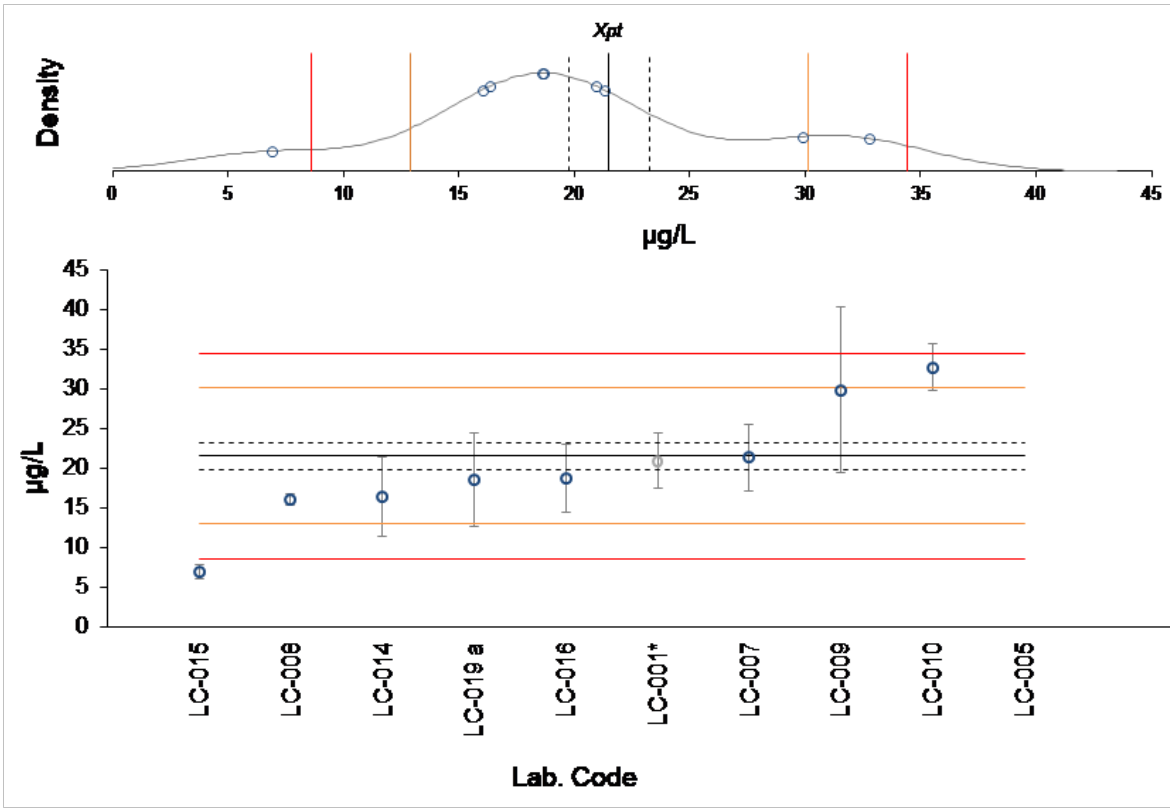

Figure 14: Measurement result range reported by the participants for the determination of BPS in hot water extract of sample 1. Circles and bars represent the reported results [x_i] with the corresponding expanded uncertainties [$U(x_i)$]; orange and red lines represent *z* scores = 2 and 3, respectively; solid and dotted black lines represent the assigned value [x_{pt}] and its expanded uncertainty [$U(x_{pt})$].
* The results were reported after the Preliminary Report was sent to the participants. These values were not included in the calculations of x_{pt} and σ_{pt}, this is indicated by the grey circle.

Table 23: Results for the determination of BPS in hot water extract of sample 1.
Assigned range: x_{pt} = 21.513 ± 1.754 µg L^{-1}; σ_{pt} = 4.303 µg L^{-1}; x_i and $U(x_i)$ values are reported in µg L^{-1}.

Lab. Code	x_i	$U(x_i)$	k	*z* score	ζ score	$u(x_i)$ *est.* §
LC-001*	20.933	3.467	2	-0.13	-0.30	a
LC-005	4331.705	701.736	2	1001.75	12.28	a
LC-007	21.331	4.266	2	-0.04	-0.08	a
LC-008	16.025	0.769	2	-1.28	-5.73	b
LC-009	29.900	10.465	2	1.95	1.58	a
LC-010	32.767	2.969	2	2.62	6.53	a
LC-014	16.333	5.004	2	-1.20	-1.95	a
LC-015	6.900	0.842	2	-3.40	-15.02	a
LC-016	18.667	4.275	2	-0.66	-1.23	a
LC-019 a	18.594	5.931	2	-0.68	-0.94	a

* The results were reported after the Preliminary Report was sent to the participants. These values were not included in the calculations of x_{pt} and σ_{pt}.
§ (a) Reasonable estimation of $u(x_i)$; (b) underestimation of $u(x_i)$; (c) overestimation of $u(x_i)$.

12.3.12 Results for the determination of BPS in hot water extract of sample 2

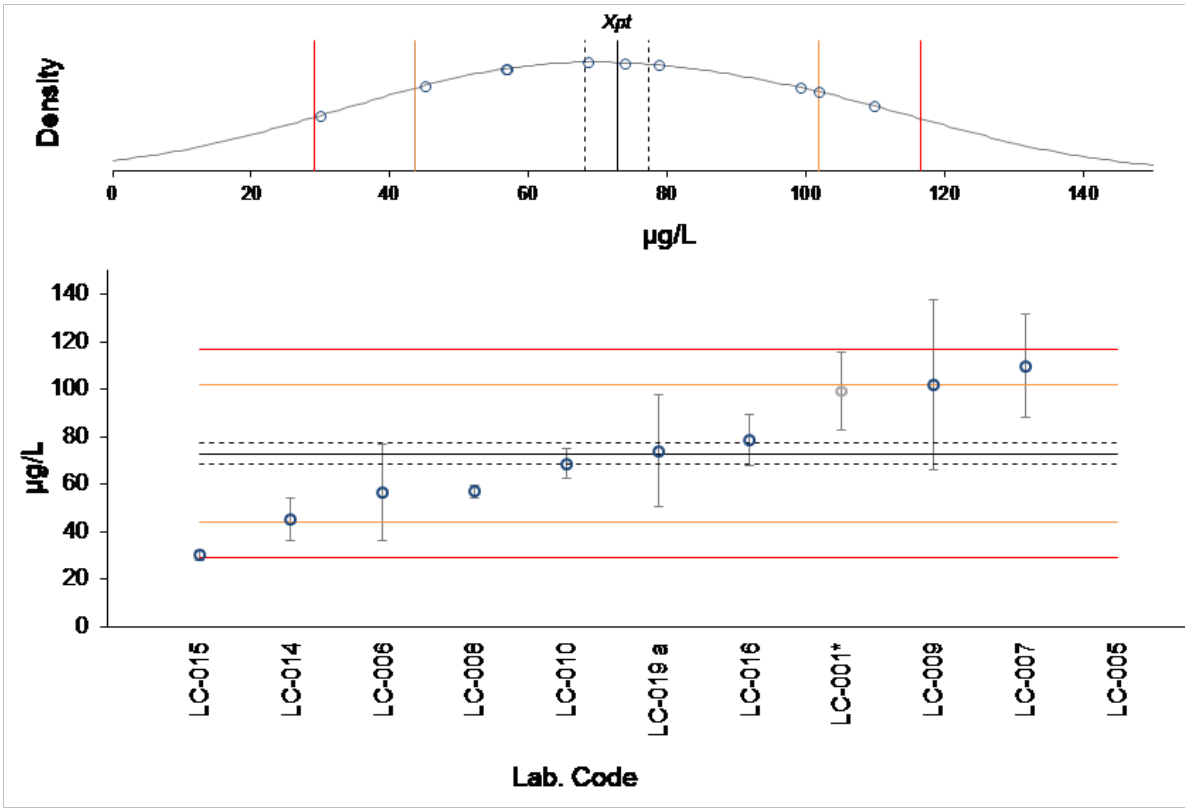

Figure 15: Measurement result range reported by the participants for the determination of BPS in hot water extract of sample 2. Circles and bars represent the reported results [x_i] with the corresponding expanded uncertainties [$U(x_i)$]; orange and red lines represent *z* scores = 2 and 3, respectively; solid and dotted black lines represent the assigned value [x_{pt}] and its expanded uncertainty [$U(x_{pt})$].
* The results were reported after the Preliminary Report was sent to the participants. These values were not included in the calculations of x_{pt} and σ_{pt}, this is indicated by the grey circle.

Table 24: Results for the determination of BPS in hot water extract of sample 2.
Assigned range: x_{pt} = 72.811 ± 4.578 µg L^{-1}; σ_{pt} = 14.562 µg L^{-1}; x_i and $U(x_i)$ values are reported in µg L^{-1}.

Lab. Code	x_i	$U(x_i)$	k	z score	ζ score	$u(x_i)$ est. §
LC-001*	99.233	16.534	2	1.81	3.08	a
LC-005	3683.976	563.648	2	247.98	12.81	a
LC-006	56.667	20.328	2	-1.11	-1.55	a
LC-007	109.866	21.973	2	2.54	3.30	a
LC-008	56.995	2.736	2	-1.09	-5.93	b
LC-009	101.967	35.688	2	2.00	1.62	a
LC-010	68.600	6.167	2	-0.29	-1.10	a
LC-014	45.000	9.009	2	-1.91	-5.50	a
LC-015	29.930	1.961	2	-2.94	-17.22	a
LC-016	78.767	10.773	2	0.41	1.02	a
LC-019 a	73.822	23.549	2	0.07	0.08	a

* The results were reported after the Preliminary Report was sent to the participants. These values were not included in the calculations of x_{pt} and σ_{pt}.
§ (a) Reasonable estimation of $u(x_i)$; (b) underestimation of $u(x_i)$; (c) overestimation of $u(x_i)$.

12.3.13 Results for the determination of BPS in solution 1

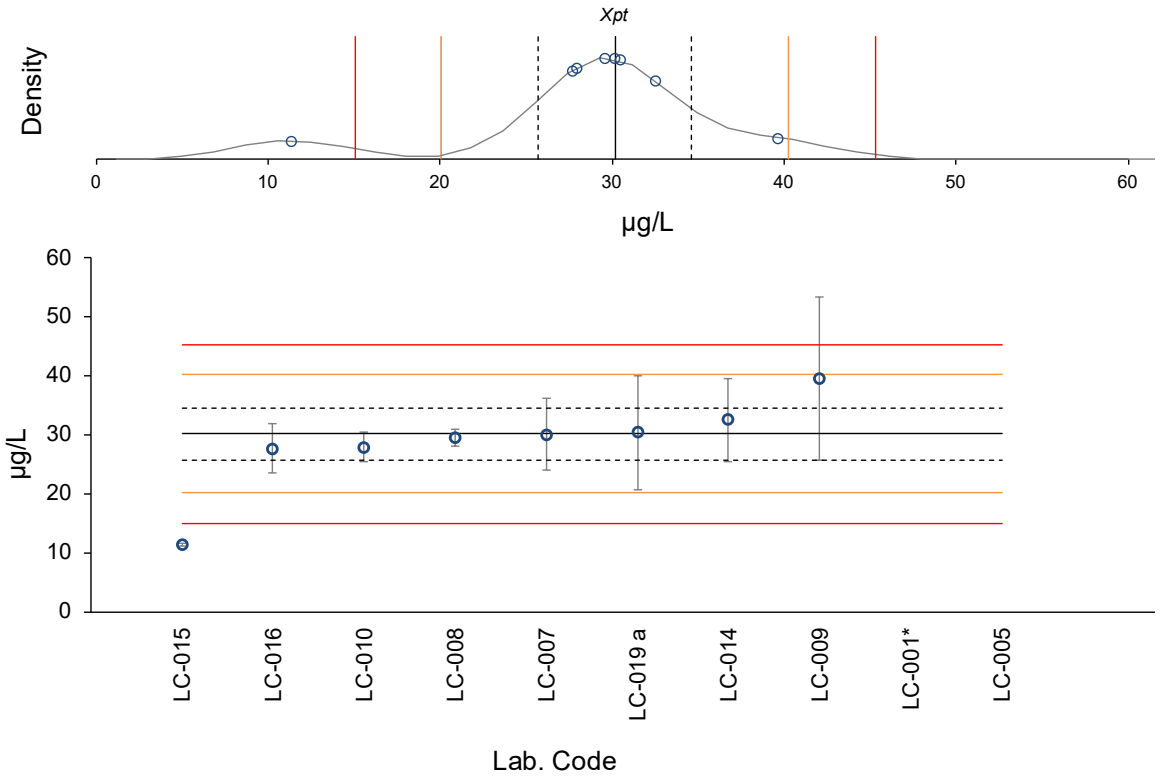

Figure 16: Measurement result range reported by the participants for the determination of BPS in solution 1. Circles and bars represent the reported results [x_i] with the corresponding expanded uncertainties [$U(x_i)$]; orange and red lines represent *z* scores = 2 and 3, respectively; solid and dotted black lines represent the assigned value [x_{pt}] and its expanded uncertainty [$U(x_{pt})$].

* The results were reported after the Preliminary Report was sent to the participants. These values were not included in the calculations of x_{pt} and σ_{pt}.

Table 25: Results for the determination of BPS in solution 1.
Assigned range: x_{pt} = 30.175 ± 4.438 µg L^{-1}; σ_{pt} = 4.526 µg L^{-1}; x_i and $U(x_i)$ values are reported in µg L^{-1}.

Lab. Code	x_i	$U(x_i)$	k	z' score #	ζ score	$u(x_i)$ *est.* §,**
LC-001*	363.700	60.600	2	66.16	10.98	a
LC-005	1229.100	68.829	2	237.84	34.77	
LC-007	30.098	6.020	2	-0.02	-0.02	a
LC-008	29.562	1.419	2	-0.12	-0.26	
LC-009	39.600	13.860	2	1.87	1.30	c
LC-010	27.900	2.500	2	-0.45	-0.89	
LC-014	32.500	7.000	2	0.46	0.56	a
LC-015	11.340	0.220	2	-3.74	-8.48	
LC-016	27.700	4.160	2	-0.49	-0.81	a
LC-019 a	30.431	9.707	2	0.05	0.05	c

* The results were reported after the Preliminary Report was sent to the participants. These values were not included in the calculations of x_{pt} and σ_{pt}.

z' score was used instead of *z* score, because the proportion $u(x_{pt})/\sigma_{pt}$ was found to be significantly higher than 0.3.

** Case (b) for u_i *est.* is not reasonable due to the high value of $u(x_{pt})$.

§ (a) Reasonable estimation of $u(x_i)$; (b) underestimation of $u(x_i)$; (c) overestimation of $u(x_i)$.

12.3.14 Results for the determination of BPS in solution 2

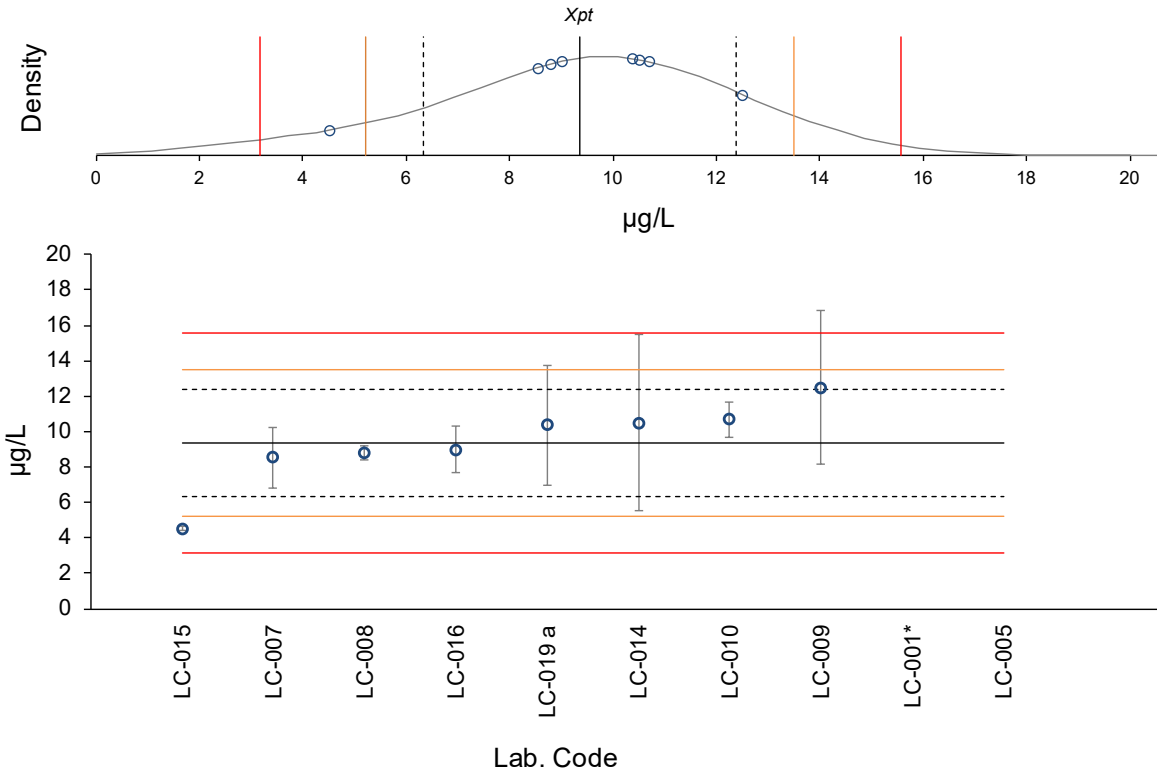

Figure 17: Measurement result range reported by the participants for the determination of BPS in solution 2. Circles and bars represent the reported results [x_i] with the corresponding expanded uncertainties [$U(x_i)$]; orange and red lines represent *z* scores = 2 and 3, respectively; solid and dotted black lines represent the assigned value [x_{pt}] and its expanded uncertainty [$U(x_{pt})$].

* The results were reported after the Preliminary Report was sent to the participants. These values were not included in the calculations of x_{pt} and σ_{pt}.

Table 26: Results for the determination of BPS in solution 2.
Assigned range: x_{pt} = 9.361 ± 3.03 µg L^{-1}; σ_{pt} = 1.404 µg L^{-1}; x_i and $U(x_i)$ values are reported in µg L^{-1}.

Lab. Code	x_i	$U(x_i)$	k	z' score [#]	ζ score	$u(x_i)$ *est.* [§,**]
LC-001*	99.000	16.500	2	43.39	10.69	
LC-005	742.400	38.605	2	354.87	37.86	
LC-007	8.529	1.706	2	-0.40	-0.48	
LC-008	8.781	0.386	2	-0.28	-0.38	
LC-009	12.500	4.375	2	1.52	1.18	c
LC-010	10.700	1.000	2	0.65	0.84	
LC-014	10.500	5.000	2	0.55	0.39	c
LC-015	4.520	0.120	2	-2.34	-3.19	
LC-016	9.000	1.340	2	-0.17	-0.22	
LC-019 a	10.358	3.418	2	0.48	0.44	c

* The results were reported after the Preliminary Report was sent to the participants. These values were not included in the calculations of x_{pt} and σ_{pt}.

[#] z' score was used instead of *z* score, because the proportion $u(x_{pt})/\sigma_{pt}$ was found to be significantly higher than 0.3.

** Case (b) for u_i *est.* is not reasonable due to the high value of $u(x_{pt})$.

[§] (a) Reasonable estimation of $u(x_i)$; (b) underestimation of $u(x_i)$; (c) overestimation of $u(x_i)$.

12.4 Results of the questionnaire

12.4.1 General Information

Table 27: General Information

Lab Code	1. Please identify yourself. You are …	2. Does your laboratory have a quality management system?	if YES, based on which standard?	3. Do you usually provide an uncertainty statement to your customer?
LC-001	NRL	Yes	ISO 17025	Yes
LC-003	NRL	Yes	ISO 17025	Yes
LC-004	NRL	Yes	ISO 17025	No
LC-005	NRL	Yes	ISO 17025	Yes
LC-006	NRL	Yes	ISO 17025	Yes
LC-007	NRL	Yes	ISO 17025	Yes
LC-008	NRL	Yes	ISO 17025	Yes
LC-009	OCL	Yes	ISO 17025	Yes
LC-010	OCL	Yes	ISO 17025	Yes
LC-012	OCL	Yes	ISO 17025	Yes
LC-013	OCL	Yes	ISO 17025	Yes
LC-014	OCL	Yes	ISO 17025	Yes
LC-015	OCL	Yes	ISO 17025	Yes
LC-016	OCL	Yes	ISO 17025	Yes
LC-017	OCL	Yes	ISO 17025	No
LC-018	OCL	Yes	ISO 17025	Yes
LC-019 a	NRL	Yes	ISO 17025	Yes
LC-019 b	NRL	Yes	ISO 17025	Yes

12.4.2 Analytical method (BPA, BPS)

Table 28: Information on the used analytical methods (Part 1)

Lab Code	1. Which analytical technique was used for the analysis of BPA and BPS?	If other specify here	2. Is this method validated/accredited for the following conditions?	Describe shortly the way of the method validation
LC-001	LC-FLD	LC-MS/MS for BPS	Not validated/accredited	
LC-003	LC-FLD	note: BPS was not analyzed	Accredited method	BPA: validation of linearity of calibration function, LOD, LOQ, repeatability, trueness, measurement uncertainty; method is validated and accredited for determination of BPA in deionized water, 3 % acetic acid, 50 % ethanol; extraction part (from paper)/migration part (from plastic articles, coatings ...) is not accredited
LC-004	LC-FLD		Not validated/accredited	
LC-005	LC-DAD		Not validated/accredited	

Continuation Table 29: Information on the used analytical methods (Part 1)

Lab Code	1. Which analytical technique was used for the analysis of BPA and BPS?	If other specify here	2. Is this method validated/accredited for the following conditions?	Describe shortly the way of the method validation
LC-006	LC-FLD	for BPS it was used LC-DAD	Validated Method	The determination step of the method is validated by replicate injections of standard solutions (and the analysis of PT sample solutions for BPA). The determination of BPA in food simulants is included in the laboratory accreditation scope.
LC-007	LC-MS/MS		Validated Method	Linearity, LOQ, repeatability, uncertainty according to Bratinova S, Raffael B, Simoneau C (2009)
LC-008	LC-MS/MS		Not validated/accredited	We have accreditation for BPA (LC/FLD) and BPS (LC-MS/MS) in foods, food simulants, but not specifically for P&B extracts.
LC-009	Other	GC-MS	Accredited method	validated only for the analysis of BPA (not BPS): specificity, limit of detection, measurement uncertainty, recovery
LC-010	Other	HPLC-FLD/DAD (FLD for BPA, DAD for BPS)	Not validated/accredited	not validated for this matrix
LC-012	Other	LC DAD/FLD	Not validated/accredited	validation not ready
LC-013	LC-MS/MS		Accredited method	Determination of linearity, reproducibility, precision, recovery and LOD/LOQ
LC-014	Other	LC-DAD + LC FLD	Validated Method	The method is validated for Bisphenol A regarding reproducibility, trueness, linearity, LOD and LOQ.
LC-015	LC-MS/MS		Accredited method	BPA/BPS: LOD: 0.35/0.098 µg/L, LOQ: see No.5, recovery: 103.1 %/---, uncertainty: 0.53 µg/L at 55.65 µg/L/-- (P = 95 %), linearity: R2 0.9999/0.9999 repeatability: 1.8 µg/L at 55.65 µg/L
LC-016	LC-MS/MS		Validated Method	precision data obtained from six-fold sample at two levels each; accuracy data obtained from recovery experiments with six-fold sample without matrix but conducting extraction process;
LC-017	LC-FLD		Accredited method	Determination of LOD/LOQ according to DIN 32645, calculation of expanded uncertainty U_c from random deviation (U_{rw}) and systematic method deviation and laboratory deviation (U_{bias})

Continuation Table 30: Information on the used analytical methods (Part 1)

Lab Code	1. Which analytical technique was used for the analysis of BPA and BPS?	If other specify here	2. Is this method validated/accredited for the following conditions?	Describe shortly the way of the method validation
LC-018	LC-DAD		Not validated/accredited	
LC-019 a	LC-MS/MS		Validated Method	
LC-019 b	LC-FLD		Validated Method	

Table 31: Information on the used analytical methods (Part 2)

Lab Code	3. How long and frequently is this method used in your laboratory?		4. Do you use certified reference materials for quality control?	5. Please provide LOQs:		6. Please enter the method for the estimation of the measurement uncertainty	if other specify here	Is the uncertainty of the extraction-step included in the estimation of measurement uncertainty?
	year(s)	/year		BPA [µg L^{-1}]	BPS [µg L^{-1}]			
LC-001	<1	Never	No	1	1	In house validation		No
LC-003	>5	1–50	No	5	na	In house validation		No
LC-004	2-5	1–50	No	1.3	150	Other	By the measurement of replicates (precision)	Yes
LC-005	<1	Never	Yes	6	40	In house validation		Yes
LC-006	>5	1–50	Yes	15	25	In house validation		No
LC-007	>5	1–50	No	3	3	In house validation		No
LC-008	2-5	1–50	No	2	4	In house validation	from other aqueous matrix for BPS	No
LC-009	<1	1–50	No	7.5	1.8	In house validation		Yes
LC-010	<1	Never	No	5	5	Other	estimation based on an existing method for the analysis of bisphenols in paper-extracts (95 % EtOH)	Yes
LC-012	<1	Never	No	5		Please Select		Please Select
LC-013	<1	1–50	No	3.1	-	In house validation		Yes

Continuation Table 32: Information on the used analytical methods (Part 2)

Lab Code	3. How long and frequently is this method used in your laboratory?		4. Do you use certified reference materials for quality control?	5. Please provide LOQs:		6. Please enter the method for the estimation of the measurement uncertainty	if other specify here	Is the uncertainty of the extraction-step included in the estimation of measurement uncertainty?
	year(s)	/year		BPA [µg L^{-1}]	BPS [µg L^{-1}]			
LC-014	>5	251–1000	No	10	10	In house validation		Yes
LC-015	>5	1–50	Yes	1.325	0.348	In house validation		No
LC-016	<1	1–50	Yes	0.6	0.3	In house validation		Yes
LC-017	<1	1–50	Yes	5	na	In house validation		Yes
LC-018	<1	Never	Yes	28		Other		Yes
LC-019 a	1–2	1–50	Yes	7	2	NORDTEST		No
LC-019 b	2–5	Please Select	Yes	3.4		NORDTEST		No

na – not analyzed

Table 33: Information on the used analytical methods (Part 3)

Lab Code	7. Did you test a blank sample?	if YES specify here	8. Did you subtract these blank values?	9. Did you apply any special treatment to the samples provided?	if YES specify here
LC-001	Yes	Both solutions used for the hot and cold extractions were analyzed – both solutions contained no BPA	No	No	
LC-003	Yes	as blank we used deionized water, treated according to the same procedure as samples	No	No	
LC-004	Yes	Procedural blank. EN645/647 without samples but with all	No	No	
LC-005	Yes	Blank sample distilled water	No	No	
LC-006	Yes	Ultrapure water filtered through glass fiber filter (grade C)	No	No	
LC-007	Yes	demineralized water same as used for preparation of the extracts filtered in the same way as the extracts	Yes	No	
LC-008	Yes	WATER	No	No	
LC-009	Yes	napkin from fresh fiber; results for BPS were negative; BPA showed a blank value of 1,80 µg/L, which was also detectable in a solvent blank sample	No	Yes	continuous shaking (at very low rpm) of the extracts during hot water extraction

Continuation Table 34: Information on the used analytical methods (Part 3)

Lab Code	7. Did you test a blank sample?	if YES specify here	8. Did you subtract these blank values?	9. Did you apply any special treatment to the samples provided?	if YES specify here
LC-010	Yes	Water	No	Yes	Hot water extracts were stored in a temperature-controlled oven (instead of a water bath)
LC-012	Yes	Water used for HWE/CWE. There was no signal at RT of BPA.	No	No	
LC-013	Yes	complete sample preparation without addition of test material	No	No	
LC-014	Yes	Wasser	No	No	
LC-015	No	0	No	No	
LC-016	Yes	extraction and filtration procedure run without matrix	Yes	No	
LC-017	Yes	Matrix blank, carried along with the sample preparation	No	No	
LC-018	Yes	Water as used for the water extracts without any further matrix, treated equal to the samples	Yes	No	
LC-019 a	Yes	Solvent blank	No	No	
LC-019 b	Yes	Solvent blank	No	No	

Table 35: Information on the used analytical methods (Part 4)

Lab Code	Did you encounter any problems with the sample analysis?	if YES specify here
LC-001	Yes	Due to Covid-19 difficulties and closure of the laboratory it was not possible to analyze the samples for BPS or Al within the given timeframe
LC-003	No	Note: We planned to introduce BPS method in our laboratory.
LC-004	Yes	For BPS, LC-UV method used was not able to detect this substance in samples
LC-005	No	
LC-006	Yes	High measurement background
LC-007	No	
LC-008	Please Select	
LC-009	Yes	all samples were packed in aluminum foil, although sample 1 was to be analyzed for aluminum content --- could this represent a possible error source?
LC-010	Yes	The quantification of BPS by HPLC-DAD was strongly impaired by matrix-peaks in the chromatogram
LC-012	No	
LC-013	No	
LC-014	Yes	Co-elution occur because of the large number of compounds, which can influence the response of the compounds during detection and quantification.
LC-015	Yes	Solution 3 in HNO_3
LC-016	No	
LC-017	No	
LC-018	No	
LC-019 a	Please Select	
LC-019 b	Please Select	

12.4.3 Cold water extract

Table 36: Information on the cold water extract

Lab Code	1. Did you use a glass-fiber filter for the filtration of the extract-solution?	if NO specify here	2. How much water did you add to fill the volumetric flask up to the mark?	3. Did you acidify the extract solution before Al analysis?
LC-001	glass-fiber filter (size C)		more than 50 ml	Yes
LC-003	Yes, glass-fiber filter (size C)		more than 50 ml	Yes
LC-004	glass-fiber filter (size C)		11–50 ml	No
LC-005	Yes, glass-fiber filter (size C)		more than 50 ml	Yes
LC-006	Yes, glass-fiber filter (size C)		11–50 ml	Please Select
LC-007	No (Specify)	Samples were filtered through a glass frit, porosity S2 (glass-fiber filters were not available)	11–50 ml	Yes
LC-008	Yes, glass-fiber filter (other size)		more than 50 ml	Yes
LC-009	No (Specify)	filtration through glass frit	11–50 ml	Yes
LC-010	No (Specify)	folded filters (Sartorius; Cellulose, 185 mm; grade 1288; and before HPLC-Analysis a syringe filter PHENEX PTFE 0,45 µm)	0–10 ml	Yes
LC-012	Please Select		Please Select	Yes
LC-013	Yes, glass-fiber filter (other size)		0–10 ml	Yes
LC-014	Yes, glass-fiber filter (other size)		11–50 ml	Yes
LC-015	Yes, glass-fiber filter (size C)		11–50 ml	Yes
LC-016	Yes		11–50 ml	Yes
LC-017	Yes, glass-fiber filter (size C)		more than 50 ml	Yes
LC-018	Yes, glass-fiber filter (other size)	Glass frit as in the standard	more than 50 ml	Yes
LC-019 a	Yes, glass-fiber filter (size C)		11–50 ml	Yes
LC-019 b	Yes, glass-fiber filter (size C)		11–50 ml	Please Select

12.4.4 Hot water extract

Table 37: Information on the hot water extract (Part 1)

Lab Code	1. Did you use a glass-fiber filter for the filtration of the extract-solution?	if NO specify here	2. How much water did you add to fill the volumetric flask up to the mark?
LC-001	glass-fiber filter (size C)		more than 50 ml
LC-003	Yes, glass-fiber filter (size C)		more than 50 ml
LC-004	glass-fiber filter (size C)		11–50 ml
LC-005	Yes, glass-fiber filter (size C)		more than 50 ml
LC-006	Yes, glass-fiber filter (size C)		11–50 ml
LC-007	No (Specify)	Samples were filtered through a glass frit, porosity S2 (glass-fiber filters were not available)	11–50 ml
LC-008	Yes, glass-fiber filter (other size)		more than 50 ml
LC-009	No (Specify)	filtration through glass frit	11–50 ml

Continuation Table 38: Information on the hot water extract (Part 1)

Lab Code	1. Did you use a glass-fiber filter for the filtration of the extract-solution?	if NO specify here	2. How much water did you add to fill the volumetric flask up to the mark?
LC-010	No (Specify)	same as cold water extract	0–10 ml
LC-012	Please Select		Please Select
LC-013	Yes, glass-fiber filter (other size)		11–50 ml
LC-014	Yes, glass-fiber filter (other size)		11–50 ml
LC-015	Yes, glass-fiber filter (size C)		11–50 ml
LC-016	No (Specify)	just using a Büchner funnel	11–50 ml
LC-017	Yes, glass-fiber filter (size C)		more than 50 ml
LC-018	Yes, glass-fiber filter (other size)		more than 50 ml
LC-019 a	Yes, glass-fiber filter (size C)		11–50 ml
LC-019 b	Yes, glass-fiber filter (size C)		11–50 ml

Table 39: Information on the hot water extract (Part 2)

Lab Code	3. Did you control the solution temperature before filling the volumetric flask up to the mark?	specify here	4. Was the extract warmed up before taking a sample for analysis?
LC-001	Yes	The solutions were made to volume after they had cooled to room temperature	Yes up to 80 ± 2 °C (Temperature was controlled)
LC-003	Please Select	room temperature (22 °C to 25 °C)	No
LC-004	23 ± 2 °C		No
LC-005	Yes, 23 ± 2 °C		Yes up to 80 ± 2 °C (Temperature was controlled)
LC-006	Yes, 23 ± 2 °C		No
LC-007	No	Samples were let to cool down to lab temperature overnight.	No
LC-008	No	The extracts were let to cool down to ambient T before filling up to the mark	No
LC-009	No	extracts were left standing for 3–4 hours at room temperature	No
LC-010	Yes, 23 ± 2 °C		No
LC-012	Please Select		Please Select
LC-013	No		No
LC-014	Yes, 23 ± 2 °C		No
LC-015	Yes, 23 ± 2 °C		No
LC-016	23 ± 2°C		No
LC-017	Yes, 23 ± 2 °C		No
LC-018	Yes, 23 ± 2 °C		No
LC-019 a	Yes, 23 ± 2 °C		No
LC-019 b	Yes, 23 ± 2 °C		No