Results of Proficiency Test
Total Bisphenol A in Polymers
May 2018

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. G.A. Oosterlaken-Buijs
Correctors: ing. A.S. Noordman-de Neef & ing. R.J. Starink

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1 INTRODUCTION

Bisphenol A is classified in Directive 2009/48/EC under Regulation (EC) No 1272/2008 as toxic. Bisphenol A (BPA) is a chemical that is mainly used in combination with other chemicals to manufacture plastics and resins. For example, BPA is used in polycarbonate, a high performance transparent, rigid plastic. Polycarbonate is used to make food containers, such as returnable beverage bottles, infant feeding (baby) bottles, tableware (plates and mugs) and storage containers. Residues of BPA are also present in epoxy resins used to make protective coatings and linings for food and beverage cans. BPA can migrate in small amounts into food and beverages stored in materials containing the substance.
Bisphenol A is a chemical that also often can be found in coatings on thermal printing paper. The surface of the paper is coated with a solid-state mixture of a dye and a reactant acid (Bisphenol A). The Bisphenol A can transfer readily to the skin in small amounts, especially when the skin is dry and free of grease. On 12 December 2016, the Official Journal of the European Union published Regulation (EU) 2016/2235 to include BPA restriction in Annex XVII to Regulation (EC) No 1907/2006 (REACH Regulation). The new restriction sets forth a threshold limit of 0.02 % (by weight) for Bisphenol A present in thermal paper after 2 January 2020.

Since 2014, a proficiency test for the analysis of Bisphenol A in polymers is organised every year by the Institute for Interlaboratory Studies (iis). During the annual proficiency testing program 2017/2018, it was decided to continue the proficiency test for the analysis of total Bisphenol A in polymers.
In this interlaboratory study 72 laboratories in 21 different countries registered for participation. See appendix 3 for the number of participants per country. In this report, the results of the 2018 proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory.
It was decided to send two different samples. The first sample (3 grams Polycarbonate (PC) granulate, labelled #18565) was especially prepared by a third party by addition of Bisphenol A to PC and subsequent homogenization and extrusion. The second sample (3 grams, labelled #18566) was a piece of thermal printing paper, positive on BPA.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC 17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on a regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

Two different materials, one batch of Polycarbonate (PC) granulate artificially fortified to be positive on Bisphenol A and one batch of thermal printing paper were selected. Both materials were divided over plastic bags, approx. 3 grams for each sample and labelled #18565 and #18566 respectively. The thermal printing paper was wrapped in Aluminium foil to avoid influence of light and heat.
The homogeneity of subsamples #18565 and #18566 was checked by determination of Bisphenol A (BPA) content on 8 stratified randomly selected samples of each batch.

	total BPA in mg/kg sample #18565	total BPA in mg/kg sample #18566
Sample -1	862	10244
Sample -2	920	10068
Sample -3	946	10188
Sample -4	864	10044
Sample -5	911	10512
Sample -6	932	10380
Sample -7	904	10180
Sample -8	844	10400

Table 1: homogeneity test results of subsamples #18565 and #18566

From the above test results, the repeatabilities were calculated and compared with 0.3 times the repeatability of EN14372:04 in agreement with the procedure of ISO 13528, Annex B2. Regretfully, EN14372:04 does not mention a reproducibility. Therefore, the comparison was made with the repeatability of EN14372:04.

	total BPA in mg/kg sample #18565	total BPA in mg/kg sample #18566
r (observed)	103	464
reference test method	EN14372:04	EN14372:04
r (ref. test method)	113	1292

Table 2: evaluation of repeatabilities of subsamples #18565 and #18566

For both samples #18565 and #18566, the observed repeatability is in agreement with the repeatability of the reference test method. Therefore, homogeneity of the subsamples #18565 and #18566 was assumed.

To each of the participating laboratories a set of samples (1 sample labelled #18565 containing approx. 3 grams PC granulate and 1 sample labelled #18566 containing approx. 3 grams thermal printing paper (wrapped in aluminium foil), were sent on April 18, 2018.

2.5 ANALYSES

The participants were requested to determine the total Bisphenol A content on both samples #18565 and #18566. It was also requested to report if the laboratory was accredited for the requested determined components. Also, some method details were requested to be reported.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report ‘less than’ results which are above the detection limit, because such results can not be used for meaningful statistical evaluations.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form, the reporting units are given and when applicable also the reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers. Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment.

Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyses). Additional or corrected test results are used for data analysis and the original reported test results placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report ‘'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ of March 2017 (iis-protocol, version 3.4).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. Results reported as ‘<…’ or ‘>…” were not used in the statistical evaluation. First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either ‘unknown’, ‘OK’, ‘suspect’ or ‘not OK’. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO 5725 the original test results per determination were submitted to Dixon’s and/or Grubbs' and/or Rosner’s outlier tests. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ test and by R(0.01) for the Rosner’s test. Stragglers are marked by D(0.05) for the Dixon’s test, by G(0.05) or DG(0.05) for the Grubbs’ test and by R(0.05) for the Rosner’s test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1 was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are on the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test results is fit-for-use.

The z-scores were calculated in according to:

$z_{(target)}$ = (test result – average of proficiency test) / target standard deviation

The $z_{(target)}$ scores are listed in the result tables of appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare. The usual interpretation of z-scores is as follows:

$\lvert z\rvert < 1$	good
$1 < \lvert z\rvert < 2$	satisfactory
$2 < \lvert z\rvert < 3$	questionable
$3 < \lvert z\rvert$	unsatisfactory

4 EVALUATION

During the execution of this proficiency test no problems occurred, although one participant reported test results after the final reporting date and three other participants did not report any test results at all. In total 69 participants reported 133 numerical test results. Observed were 9 outlying test results, which is 6.3%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal Gaussian distribituion. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER SAMPLE

In this section, the reported test results are discussed per sample.
The test methods, which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These methods are also in the table together with the original data. The abbreviations, used in these tables, are listed in appendix 4.

Due to the lack of a suitable test method with precision data for the determination of total BPA in polymers, it was decided to use the requirements from the standardised method EN14372:04, “Child use and care articles, Cutlery and feeding utensils, Safety requirements and tests” for evaluation of the results of this interlaboratory study.
Regretfully, only a relative within-laboratory standard deviation RSDr is given in EN14372:04. Multiplication of RSDr by 2.8 gives the relative repeatability. Multiplication of the repeatability by 3 gives a good estimate of the relative target reproducibility.

Sample #18565

BPA: The determination of total Bisphenol A in the Polycarbonat (PC) sample was problematic at the level of 929 mg/kg. Five statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility of EN14372:04. See also the discussion in paragraph 5.

Sample #18566

BPA: The determination of total Bisphenol A in this thermal printing paper sample was not problematic. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility of EN14372:04.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibilities as estimated from the reference test method of EN14372:2004 (R_{lit}) and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average result, the calculated reproducibility (2.8 * standard deviation) and the estimated target reproducibility of EN14372:2004 are presented in the next tables.

Component	unit	n	average	2.8 * sd	R (lit)
Bisphenol A (total)	mg/kg	62	929	534	351

Table 3: overview of results for sample #18565

Component	unit	n	average	2.8 * sd	R (lit)
Bisphenol A (total)	mg/kg	62	9665	3059	3653

Table 4: overview of results for sample #18566

Without further statistical calculations, it can be concluded that for sample #18565 (Polycarbonate (PC) granulate) there is not a good compliance of the group of participating laboratories with the relevant standard. However, for sample #18566 (thermal printing paper) there is a good compliance of the group of participating laboratories with the relevant standard. See also the discussion in paragraphs 4.1 and 5.

4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2018 WITH PREVIOUS PTS

	May 2018	May 2017	May 2016	April 2015	April 2014
Number of reporting labs	69	55	53	53	60
Number of results reported	133	108	105	104	120
Number of statistical outliers	9	8	3	6	6
Percentage outliers	6.3%	6.9%	2.8%	5.5%	4.8%

Table 5: comparison with previous proficiency tests

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency test was compared, expressed as relative standard deviation (RDS) of the PTs, see next table.

Component	Conc. in mg/kg	May 2018	May 2017	May 2016	April 2015	April 2014	EN14372
BPA	<1000	21%	n.e.	30%	54%	n.e.	13.5%
BPA	1000 – 2500	n.e.	14%	24%	23%	34%	13.5%
BPA	>2500	11% *)	12% *)	n.e.	n.e.	21%	13.5%

Table 6: development of relative uncertainties over the years

*) sample matrix: thermal printing paper

At a BPA concentration <1000 mg/kg the uncertainty in the test result of BPA in this PT iis18P04 has improved in comparison with the previous PTs. At a BPA concentration >2500 mg/kg the uncertainty in the test result of BPA in PT iis18P04 is in line with the PT of 2017 and is even in line with the uncertainty requirements of the target method.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

From the reported test methods it appeared that a large majority participants used for the determination of total BPA an in house test method (62 laboratories = 90%). Some participants did use test method EN14372, this method is for the determination of the migration of BPA.

For this Profiency Test some analytical details were requested (see appendix 2). Based on the answers given by the participants the following can be summarized:
Fourty-three of the participants answered to be ISO/IEC17025 accredited for the determination of total BPA in polymers (= 62%).
Almost all participants did use sample #18565 as received and did further cut sample #18566 prior to analyses.
A few participants adapted the analytical parameters which were used to the material of the sample (Polycarbonate or thermal print paper).
The final estimated particle sizes of sample #18566 most often used was between 2mmx2mm and 5mm x 5mm.
Almost all participants used a sample intake between 0.5 and 1.0 grams (about 70%), about 20% of the reporting participants mentioned to have used <0.5 grams. Which is remarkable as in the letter of instruction it is requested not to use less than 0.5 grams per determination.
The solvent (mixture) to release the BPA from the samples differs. About 40% of the participants used Dichloromethane and a Chloroform/Methanol mixture was used by about 15% of the participants. Also, other extraction solvents were used (for example Dichloromethane mixture, Tetrahydrofuran or Acetonitril). A few participants used an extraction solvent based on water, most likely the used method was based on migration instead of a total BPA determination.
Almost all participants did use an extraction time between 30 and 60 min.
About 50% of the participants reported to have used an extraction temperature of 40°C. Extraction temperatures like room temperature, 30 °C or 70 °C were also mentioned.

5 DISCUSSION

In this proficiency test for the determination of total BPA in polymers two different sample matrices were used (Polycarbonate granulate and thermal printing paper).
For sample #18566 (thermal printing paper) a good compliance of the reproducibility was observed for the group of participating laboratories with the reproducibility as estimated from the reference test method of EN14372:2004 (R (lit)). However, for sample #18565 (Polycarbonate (PC) granulate) the observed reproducibility was much larger compared to EN14372:2004. See also the discussion in paragraph 4.1.

Therefore, for sample #18565 the influence of the extraction solvent was further investigated. When the test results of the participants which used an extraction solvent of Chloroform/Methanol were evaluated separately, the calculated reproducibility is in agreement with the estimated reproducibility of EN14372:04. See below table.

Subset of test results	unit	n	average	2.8 * sd	R (lit)
Dichloromethane	mg/kg	27	925	406	350
Chloroform/Methanol	mg/kg	12	932	168	352

Table 7: overview of separate evaluation for sample #18565

Five statistical outliers were observed for sample #18565. The sample intake of the participants with an outlier was either < 0.5 grams or unknown. One should be aware that when a sample batch of samples containing 3 grams of material is tested for homogeneity, this is based on the use of a significant amount of the sample. When only a part of the sample is used, homogeneity of such a small sample might not meet the requirements and that will have an effect on the obtained and reported test result. It is strongly advised to use a sample size of at least 0.5 grams.

6 CONCLUSION

It can be concluded that the group of participants have no problems with the determination of total BPA in the thermal printing paper sample in this PT. However, one participant reported a possible false negative test result. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility of EN14372:04.

The group of participants have problems with the determination of total BPA in the Polycarbonate sample of this PT. The extraction solvent appeared to have influence on the reproducibility.
Participants are strongly advised to read the PT instructions carefully and to use a sample size that is at least 0.5 grams.

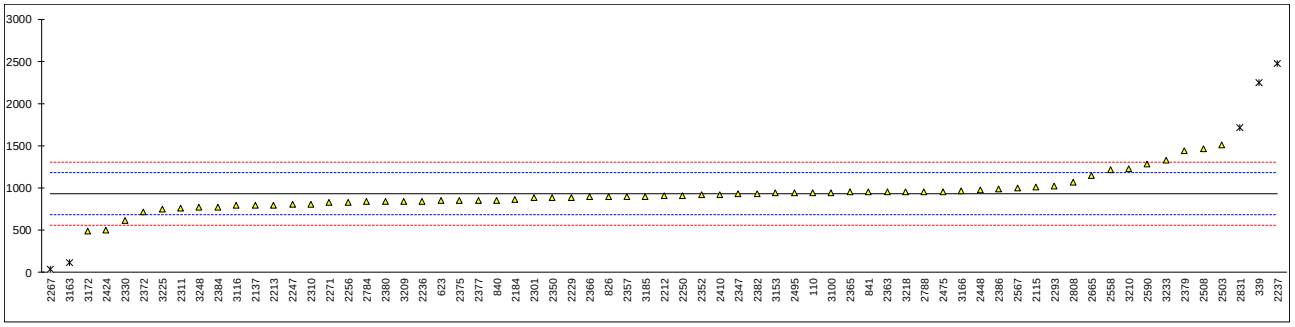
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.
Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

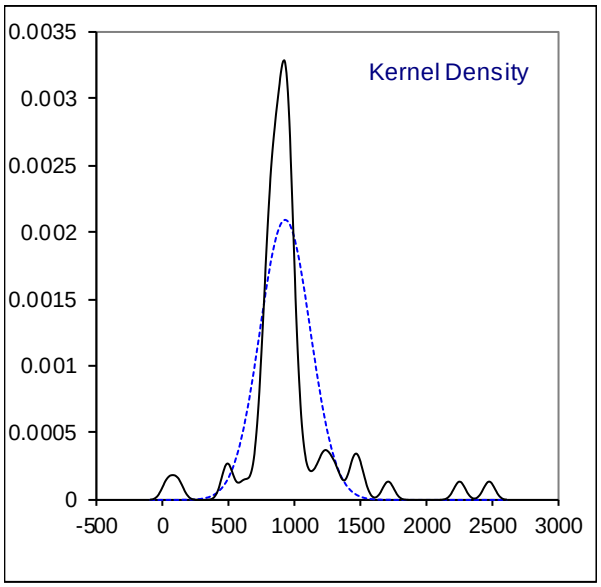
APPENDIX 1

Determination of Total Bisphenol A (BPA) in sample #18565; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	945.755		0.13	
330		-----		-----	
339	In house	2251.3	R(0.01)	10.54	
362	EN14372	<0.150		<-7.41	possible false negative test result?
623	In house	848.65		-0.64	
826	In house	897.978		-0.25	
840	In house	857		-0.58	
841	In house	950.289		0.17	
2115	In house	1012.8		0.67	
2137	KS M1997	800.72		-1.02	
2184	In house	869.16		-0.48	
2212	In house	908.4		-0.17	
2213	EN14372	801		-1.02	
2229	In house	891.584	C	-0.30	first reported 2909.330
2236	In house	844.96		-0.67	
2237	In house	2475.33	R(0.01)	12.33	
2247	In house	801.44		-1.02	
2250	In house	913.76		-0.12	
2255		-----		-----	
2256	In house	828.89		-0.80	
2267	In house	38.36	R(0.01)	-7.10	
2271	In house	826.8		-0.82	
2293	In house	1,018.64		0.71	
2301	In house	887.05		-0.34	
2310	In house	809		-0.96	
2311	In house	762.7		-1.33	
2330	In house	618.656		-2.48	
2347	In house	934		0.04	
2350	In house	888.36		-0.33	
2352	JTPC D2.II	919.2		-0.08	
2357	In house	902.0		-0.22	
2363	In house	951		0.17	
2365	In house	949.8		0.16	
2366	In house	895.2		-0.27	
2372	In house	720.8		-1.66	
2375	In house	850.3		-0.63	
2377	In house	851.597		-0.62	
2379	JETRO 2009	1441.5064		4.08	
2380	In house	838.31		-0.72	
2382	In house	936.8		0.06	
2384	In house	777.245		-1.21	
2386	In house	991		0.49	
2410	In house	925		-0.03	
2424	In house	498		-3.44	
2448	JETRO	979.75		0.40	
2475	In house	957.815		0.23	
2493	In house	<0.030	C	<-7.41	first reported 1499, possible false negative test result?
2495	In house	943.82		0.12	
2503	In house	1504		4.58	
2508	In house	1462	C	4.25	first reported 5118
2558	In house	1215.652		2.28	
2567	In house	998		0.55	
2590	In house	1278.539		2.79	
2665	In house	1144		1.71	
2784	In house	835.66		-0.75	
2788	In house	955.249		0.21	
2808	In house	1062.3	C	1.06	first reported 1959.2
2831	In house	1710	C, R(0.01)	6.23	first reported 1410
3100	In house	946.8		0.14	
3116	In house	796		-1.06	
3146		-----		-----	
3153	In house	939		0.08	
3163		115	C, R(0.01)	-6.49	first reported 15
3166	In house	967.6		0.31	
3172	In house	484.9		-3.54	
3185	In house	902.72		-0.21	
3209	In house	840.12		-0.71	
3210	In house	1225.851		2.37	
3218	In house	952.000		0.18	
3225	In house	749.7		-1.43	
3233	In house	1324.500		3.15	
3248	In house	775		-1.23	

		DCM	Chloroform/MeOH
normality	not OK	not OK	suspect
n	62	27	12
outliers	5	0	0
mean (n)	929.13	924.90	932.12
st.dev. (n)	190.843	145.063	59.853
R(calc.)	534.36	406.18	167.59
st.dev.(EN14372:04)	125.433	124.861	125.836
R(EN14372:04)	351.21	349.61	352.34

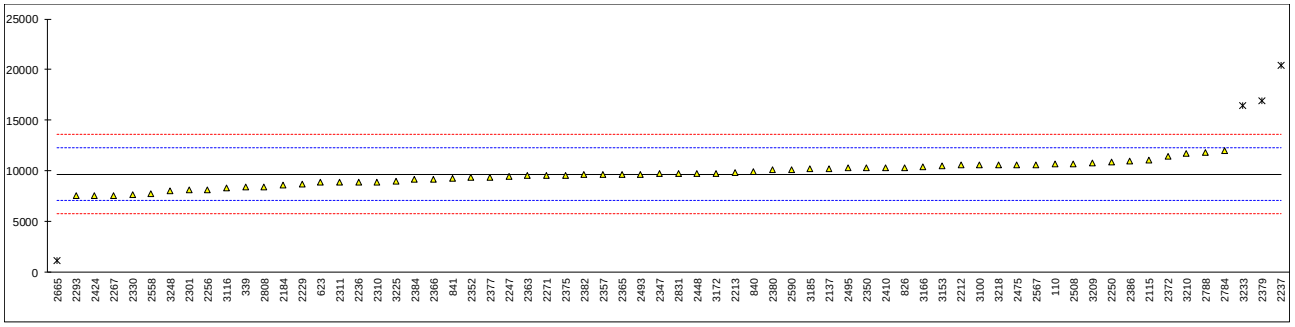


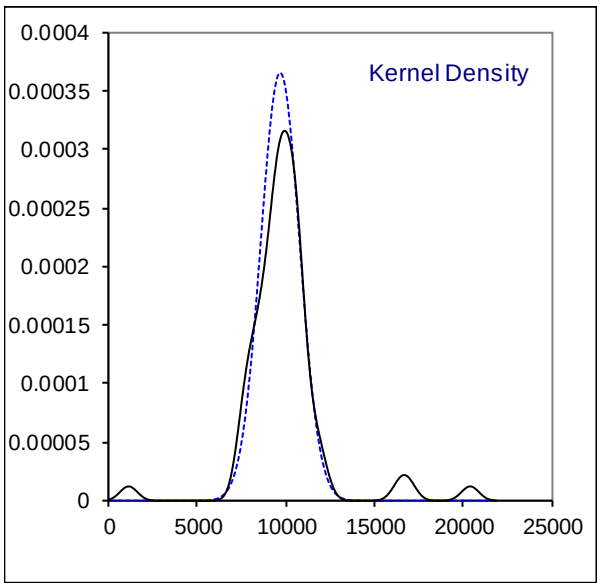


Determination of Total Bisphenol A (BPA) in sample #18566; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	10656.279		0.76	
330		-----		-----	
339	In house	8389.2		-0.98	
362	EN14372	<0.150		<-7.41	possible false negative test result?
623	In house	8850.21		-0.62	
826	In house	10327.056		0.51	
840	In house	9890		0.17	
841	In house	9276.150		-0.30	
2115	In house	11048.5		1.06	
2137	KS M1997	10263.63		0.46	
2184		8572.95		-0.84	
2212	In house	10560		0.69	
2213	EN14372	9805		0.11	
2229	In house	8734.856	C	-0.71	first reported 17704.653
2236	In house	8888.65		-0.59	
2237	In house	20380.00	R(0.01)	8.21	
2247	In house	9451.93		-0.16	
2250	In house	10879		0.93	
2255		-----		-----	
2256	In house	8178.74		-1.14	
2267	In house	7604.4		-1.58	
2271	In house	9559.6		-0.08	
2293	In house	7,591.02		-1.59	
2301	In house	8146.23		-1.16	
2310	In house	8920		-0.57	
2311	In house	8852.8		-0.62	
2330	In house	7665.845		-1.53	
2347	In house	9701		0.03	
2350	In house	10299.11		0.49	
2352	JTPC D2.II	9386.2		-0.21	
2357	In house	9665.6		0.00	
2363	In house	9550		-0.09	
2365	In house	9666.8		0.00	
2366	In house	9170.0		-0.38	
2372		11478.8		1.39	
2375	In house	9590		-0.06	
2377	In house	9403.805		-0.20	
2379	JETRO 2009	16890.6246	R(0.01)	5.54	
2380	In house	10077.67		0.32	
2382	In house	9638.9		-0.02	
2384	In house	9167.381		-0.38	
2386	In house	10943		0.98	
2410	In house	10300		0.49	
2424	In house	7603		-1.58	
2448	In house	9756.64		0.07	
2475	In house	10586.685		0.71	
2493	In house	9685		0.02	
2495	In house	10277.84		0.47	
2503		-----		-----	
2508	In house	10718		0.81	
2558	In house	7768.511		-1.45	
2567	In house	10610		0.72	
2590	In house	10154.211	C	0.38	first reported 21781.643
2665	In house	1118	C, R(0.01)	-6.55	first reported 4298
2784	In house	12048.87		1.83	
2788	In house	11819.691		1.65	
2808	In house	8446.4	C	-0.93	first reported 14634.7
2831	In house	9710		0.03	
3100	In house	10577		0.70	
3116	In house	8310		-1.04	
3146		-----		-----	
3153	In house	10470		0.62	
3163		-----		-----	
3166	In house	10408		0.57	
3172	In house	9761.9		0.07	
3185	In house	10203.43		0.41	
3209	In house	10800.20		0.87	
3210	In house	11725.75	C	1.58	first reported 17725.751
3218	In house	10583.900		0.70	
3225	In house	9002.77		-0.51	
3233	In house	16431.093	R(0.01)	5.19	
3248	In house	8038		-1.25	

normality	OK
n	62
outliers	4
mean (n)	9664.78
st.dev. (n)	1092.444
R(calc.)	3058.84
st.dev.(EN14372:04)	1304.745
R(EN14372:04)	3653.29





APPENDIX 2
Method information as reported by the participating laboratories

Lab	ISO17025 accr. for BPA	sample grinded or cut	final particle size	sample intake (g)	extraction solvent	extraction time (min)	extraction temp (°C)
110	Yes	#18565 as received, #18566 further cut	2mm x 2mm	0.5 g	Dichloromethane	30	40
330	---	---					
339	---	---					
362	No	---					
623	Yes	Further Cut	2 mm x 2 mm	0.5 g	Dichloromethane (DCM)	30	40
826	No	Further Cut	2mm*2mm		DCM	30	40
840	Yes	Further Cut	2x2mm	0.5g	DCM	30	40
841	No	Further Cut	2 mm x 2 mm	0.5	Dichloromethane, Acetone, Acetonitrile, H2O	30	40
2115	No	Used as received		0.5 g	Acetonitrile	60	40
2137	Yes	Further Cut		0.5	Dichloromethane	60	30
2184	No	Used as received	NA	NA	acetonitrile	1440 (60 more or less)	room temp
2212	Yes	Further Cut	2mm	0.2	Dichloromethane	60	40
2213	Yes	---					
2229	Yes	Further Cut	5mm*5mm	0.1g	dichloromethane	30	40
2236	Yes	Further Cut	3 mm x 3 mm	0.5 grams	2:1 Chloroform:Methanol	60	70
2237	Yes	Used as received		0,1	toluene	60	room temp
2247	Yes	#18565 as received, #18566 Further Cut	#18566 3mmx3mm	0.5014	Chloroform:Methanol (2:1)	60.0	70.0
2250	Yes	#18565 as received, #18566 further cut		0.25	Tetrahydrofuran	30	40
2255	---	---					
2256	Yes	Further Cut	5mm*5mm	1gram	DCM/THF	60	40
2267	---	---					
2271	Yes	#18565 as received, #18566 Further Cut	2mmx2mm	0.5g	dichloromethane/methanol	60	room temp
2293	No	Used as received	2 x 2 mm	1 g	chloroform:methanol (2:1) 10 mL	60	70
2301	Yes	Further Cut	2 mm x 2mm	1	DCM (#18565), THF (#18566)	60	40
2310	No	#18565 as received, #18566 further cut	5mm*5mm	1g	Dichloromethane	30	40
2311	Yes	Further Cut	<5mm x 5mm	1	Dichloromethane and Acetone	30	40
2330	Yes (No: thermal print paper)	#18565 as received, #18566 further cut	#18566 2 x 2 mm	0.5 g (for both samples)	Dichloromethane: Acetone(20:100) (#18565), Acetone(2:10) (#18566)	30 (For both samples)	40 ± 2 °C (for both samples)
2347	Yes	Further Cut	2mm*2mm*2mm	1.0g	dichlormethane	30	40
2350	No	Further Cut	2mm	0.5 g	50% ACN	30	40
2352	Yes	Used as received	2*2*3mm	0.5g	Dichloromethane	30	40
2357	Yes	Further Cut	2mm*2mm	1.0	dichloromethane	30	40
2363	Yes	Further Cut	1mm*1mm	0.5g	DCM	30	40
2365	Yes	Further Cut	2mm*2mm	0.5g	Dichloromethane	30	40
2366	No	#18565: as received, #18566: further cut	18566 5mm*5mm	0.5g	18565: dichloromethane 18566: acetonitrile	18565: 30min, 18566: 24h	18565: 40 18566: 20
2372	Yes	Further Cut	2mm	0.5	DCM	30	40
2375	Yes	Further Cut	2x2mm	0.5	dichloromethane	30	40
2377	Yes		2mm x 2mm	~0.5g	Dichloromethane	30	40
2379	No	Further Cut	#18565 2x2 mm #18566 5x5 mm	0.5 g	Dichloromethan : DCM	30	40
2380	Yes	#18565: as received, #18566: further cut	#18566 2X2 mm	#18565 0.5011 g #18566, 0.5012 g	Dichloromethane	30	40
2382	No	Further Cut	2*2cm	1g	#18565:DCM #18566:ACN	#18565:30min #18566:24h	#18565:40 #18566:23
2384	Yes	Further Grinded		0.5	Acetone	30 minutes	40 ± 2
2386	Yes	Used as received		200mg (#18565) 20mg (#18566)	Dichlormethane	30	40
2410	No	Further Grinded	NA	-	Chloroform (2): Methanol (1)	1 hr	70
2424	No	Used as received	0.5 mm diameter	0.5	Acetone / THF	1 hour	25
2448	Yes (No: thermal print paper)	#18565 as received, #18566 further cut	#18566 is 0.2mm	0.1 g	#18565 Dichloromethane: Acetone #18566: deionized water	#18565 until as solution #18566: 120 min	#18565 room temp #18566: 40 °C
2475	No	Used as received		0.1	chloroform/methanol (2/1)	60	70

Lab	ISO17025 accr. for BPA	sample grinded or cut	final particle size	sample intake (g)	extraction solvent	extraction time (min)	extraction temp (°C)
2493	Yes	Further Cut	2 mm	0,5	5ml distilled H2O (#18565) toluene/water (#18566)	60 min (#18565) overnight/180 min (#18566)	35 °C (#18565) room temp (#18566)
2495	Yes	Used as received		0.1	Chloroform:methanol 2:1	60	70
2503	Yes	Further Cut		0.05	THF	90	70
2508	No	Used as received	5mm	0.3	THF/Hexan	60	60
2558	Yes	#18565 as received, #18566 further cut	0,5 mm x 0,2 mm	0,5 g/ 2 mL (Acetone/Hexane)	Acetone / Hexane (1:1), meaurements done in a 1:5-dilution with dichloromethane	heating time: 90 min; extraction time: 60 min	180
2567	Yes	Further Cut	2mmx2mm	0.5 gm	Chloroform:methanol 2:1	60	70
2590	No	#18565 as received, #18566 further cut	5mm x 5mm	1 g	#18565 DCM, #18566 THF	60	40
2665	Yes	#18565 as received, #18566 further cut	#18566 pieces of 1 cm²	0,5	Tetrahydrofurane	30	room temp
2784	No	#18565: as received, #18566: further cut	#18566: 2x 10 mm	#18565 0.5 g #18566 0.1 g	acetonitrile	30	room temp
2788	No	Further Cut	<3x3 mm	1 g	chloroform:methanol, 3:1	2 hrs	70
2808	No	#18565 as received, #18566 further cut	#18566 2mmx2mm	#18565 (0.1 g) / #18566 (0.5 g)	Dichloromethane	1440	room temp
2831	No	#18565: as received, #18566: further cut	#18566 around 5 mm2	0.14	ACN/DCM 10:90 v/v%	30	40
3100	Yes	Further Cut	2 mm * 2 mm	1.000g	trichloromethane:methanol = 2:1	60	70
3116	Yes	#18565 as received, #18566 further cut	#18566: 5mm x 5mm	0.5	#18565 DCM #18566 THF	60	40
3146	---	---					
3153	No	#18565 as received, #18566 further cut	3mm x 3mm	0.5g	Chloroform / Methanol mixture	60	70
3163	No	Further Cut	1mm	0.2	toluene	60	60
3166	Yes	Further Cut		0.5g	DCM	60	ambient
3172	Yes	Further Cut	1mm	0.5	THF:Dichloromethane	60	25
3185	Yes	Further Cut	2mm*2mm	1g	chloroform : methanol (2:1)	60	70
3209	Yes	Further Cut	2*2mm	0.1	DCM	60	40
3210	No	Used as received		0.5	THF/ACN for #18565 Methanol for #18566	60	60
3218	Yes	#18565 as received, #18566 further cut	#18566 5mmx5mm	0.5g	Chroform:methanol=2:1	60	70
3225	Yes	Further Cut	2mm x 2mm	0.5	DCM:MeOH	60	23
3233	No	#18565 as received, #18566 further cut	#18565 3mm x 2mm, #18566 5mm x 5mm	0.25g for #18565 0.5g for #18566	dicholoromethane+acetone +acetonitrile for #18565, acetonitrile for #18566	60min for #18565, 1440min (24h) for #18566	40°C for #18565, 23°C for #18566
3248	Yes	#18565: as received #18566:Further Cut	#18566 5mm x 5mm	1g	THF, MeOH	1 hour	room temp

APPENDIX 3

Number of participating laboratories per country

2 labs in	BANGLADESH
1 lab in	BULGARIA
2 labs in	CAMBODIA
6 labs in	FRANCE
7 labs in	GERMANY
1 lab in	GUATEMALA
7 labs in	HONG KONG
1 lab in	HUNGARY
4 labs in	INDIA
2 labs in	INDONESIA
4 labs in	ITALY
4 labs in	KOREA
1 lab in	MALAYSIA
14 labs in	P.R. of CHINA
1 lab in	SPAIN
1 lab in	TAIWAN R.O.C.
2 labs in	THAILAND
3 labs in	THE NETHERLANDS
1 lab in	TURKEY
6 labs in	U.S.A.
2 labs in	VIETNAM

APPENDIX 4

Abbreviations:

C = final test result after checking of first reported suspect test result
D(0.01) = outlier in Dixon's outlier test
D(0.05) = straggler in Dixon's outlier test
G(0.01) = outlier in Grubbs' outlier test
G(0.05) = straggler in Grubbs' outlier test
DG(0.01) = outlier in Double Grubbs' outlier test
DG(0.05) = straggler in Double Grubbs' outlier test
R(0.01) = outlier in Rosner's outlier test
R(0.05) = straggler in Rosner's outlier test
E = probably an error in calculations
W = test result withdrawn on request of participant
ex = test result excluded from statistical evaluation
n.a. = not applicable
n.d. = not detected
n.e. = not evaluated
fr. = first reported

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, March 2017
2 ASTM E178:02
3 ASTM E1301:03
4 ISO 5725:86
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7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
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9 IP 367:84
10 DIN 38402 T41/42
11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
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14 Analytical Methods Committee Technical brief, No 4 January 2001
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16 ISO 13528:15, Statistical methods for use in proficiency testing by interlaboratory comparison
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18 https://chemicalwatch.com/44942/bpa-poised-for-classification-as-category-1-reprotoxin
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