Results of Proficiency Test
Total Bisphenol A in Polymers
June 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

1 INTRODUCTION

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 can be found in coatings on thermal printing paper. 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, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of total Bisphenol A in polymers every year. During the annual proficiency testing program 2018/2019, it was decided to continue the proficiency test for the analysis of total Bisphenol A in polymers.

In this interlaboratory study 60 laboratories in 19 different countries registered for participation. See appendix 3 for the number of participants per country. In this report, the results of the 2019 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 organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send two different samples. The first sample was a Polycarbonate (PC) granulate sample of 3 grams and labelled #19547. The second sample was a Polyvinylchloride (PVC) granulate sample of 3 grams and labelled #19548.
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/IEC17043:2010. 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 June 2018 (iis-protocol, version 3.5). 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

The first batch was a colorless Polycarbonate (PC) granulate artificially fortified to be positive on Bisphenol A. The second batch were brown Polyvinylchloride (PVC) blocks artificially fortified to be positive on Bisphenol A. From both batches 100 plastic bags with approximately 3 grams granulate each were filled and labelled #19547 and #19548 respectively. The homogeneity of subsamples #19547 and #19548 was checked by determination of Bisphenol A (BPA) content on respectively 8 and 7 stratified randomly selected samples of each batch.

	total BPA in mg/kg sample #19547	total BPA in mg/kg sample #19548
Sample-1	347	4007
Sample-2	338	3872
Sample-3	329	3827
Sample-4	352	3832
Sample-5	334	3986
Sample-6	361	3880
Sample-7	365	3990
Sample-8	359	--

Table 1: homogeneity test results of subsamples #19547 and #19548

From the above test results, the repeatabilities were calculated and compared with 0.3 times the estimated reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2.

	total BPA in mg/kg sample #19547	total BPA in mg/kg sample #19548
r (observed)	37	219
reference test method	EN14372:04	EN14372:04
0.3 x R (ref. test method)	39	443

Table 2: evaluation of the repeatabilities of subsamples #19547 and #19548

Both calculated repeatabilities are in agreement with 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples #19574 and #19548 was assumed.

To each of the participating laboratories one plastic bag with 3 grams of a PC granulate labelled #19547 and one plastic bag with 3 grams of a PVC granualte labelled #19548 were sent on May 8, 2019.

2.5 ANALYSES

The participants were requested to determine the total Bisphenol A content on both samples #19547 and #19548. It was also requested to report if the laboratory was accredited for the requested determined components and to report some analytical details.

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 to 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 reanalyzes). 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 June 2018 (iis-protocol, version 3.5).

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:

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

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. Four participants reported test results after the final reporting date and one other participant did not report any test results at all. In total 59 participants reported 117 numerical test results. Observed were 14 outlying test results, which is 10.7%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Both original data sets proved to have a normal Gaussian distribituion.

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.

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, due to the lack of a suitable test method with precision data for the determination of total BPA in polymers.
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 #19547

BPA: The determination of total Bisphenol A in the Polycarbonate (PC) sample was problematic at the level of 347 mg/kg. Eight 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 #19548

BPA: The determination of total Bisphenol A in the Polyvinylchloride (PVC) sample was problematic at the level of 2862 mg/kg. Six 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.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the estimated target reproducibility 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 EN14372:04 are presented in the next tables.

Component	unit	n	average	2.8 * sd	R (lit)
Bisphenol A (total)	mg/kg	50	346.8	176.1	131.1

Table 3: overview of results for sample #19547

Component	unit	n	average	2.8 * sd	R (lit)
Bisphenol A (total)	mg/kg	53	2862	1414	1082

Table 4: overview of results for sample #19548

Without further statistical calculations, it can be concluded that there is not a good compliance of the group of participating laboratories with the reference test method. See also the discussion in paragraphs 4.1 and 5.

4.3 COMPARISON OF THE PROFICIENCY TEST OF JUNE 2019 WITH PREVIOUS PTS

	June 2019	May 2018	May 2017	May 2016	April 2015
Number of reporting labs	59	69	55	53	53
Number of results reported	117	133	108	105	104
Number of statistical outliers	14	9	8	3	6
Percentage outliers	10.7%	6.3%	6.9%	2.8%	5.5%

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.

Conc. in mg/kg	June 2019	May 2018	May 2017	May 2016	2015-2014	EN14372
<1000	18%	21%	n.e.	30%	54%	13.5%
1000 – 2500	n.e.	n.e.	14%	24%	23-34%	13.5%
>2500	18%	11% *)	12% *)	n.e.	21%	13.5%

Table 6: development of uncertainties in BPA in polymers determinations 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 iis19P04 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 iis19P04 is larger compared to the previous PT's but is more in line with the other uncertainties. Possibly the papermatrix is easier to extract BPA from.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

From the reported test methods, it appeared that almost all participants used for the determination of total BPA an in house test method (50 laboratories = 85%). Some participants did use test method EN14372, this method is for the determination of the migration of BPA.

For this proficiency 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%).
- About 50% of the participants did use the samples #19547 and #19548 as received and about 40% of the participants further cut or further grinded the samples #19547 #19548 prior to analyses.
- The final estimated particle sizes of samples #19547 and #19548 most often used was smaller than 3x3mm.
- About 81% of the participants used a sample intake between 0.5 and 1.0 grams and about 16% 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 47% of the participants used Dichloromethane and 33% of the participants used Tetrahydrofurane (THF) as solvent.
- Almost all participants did use an extraction time between 30 and 60 min.
- About 60% of the participants reported to have used an extraction temperature of 40°C and about 35% mentioned to have used an extraction temperature of 60/70°C.

When evaluating the above details some differences in the execution of the test was found between these test conditions, but the differences are not significant (see appendix 1).

5 DISCUSSION

In this proficiency test for the determination of total BPA in polymers two different sample matrices were used: Polycarbonate granulate and Polyvinylchloride blocks.
For both samples #19547 and #19548 not a good compliance of the reproducibility was observed for the group of participating laboratories with the reproducibilities as estimated from the reference test method of EN14372:2004 (R (lit)). See also the discussion in paragraph 4.1.

Therefore, for both samples the influence of the extraction solvent and the influence of the particle size was further investigated (see in appendix 1). When the test results of the participants were evaluated separately, it appears that for sample #19547 the use of THF gave a lower mean and a larger varaition, compared against the use of Dichloromethane (DCM). The use of a different particle size gave no differences. For sample #19548, the differences between the use of different particle sizes and the use of different solvents are not large.

6 CONCLUSION

It can be concluded that the group of participants have problems with the determination of total BPA in the polycarbonate and polyvinylchloride samples in this proficiency test.
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.

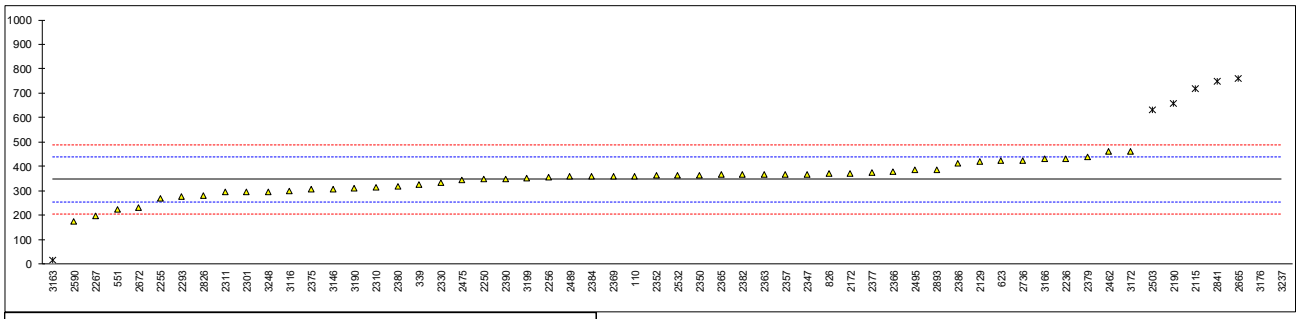
APPENDIX 1

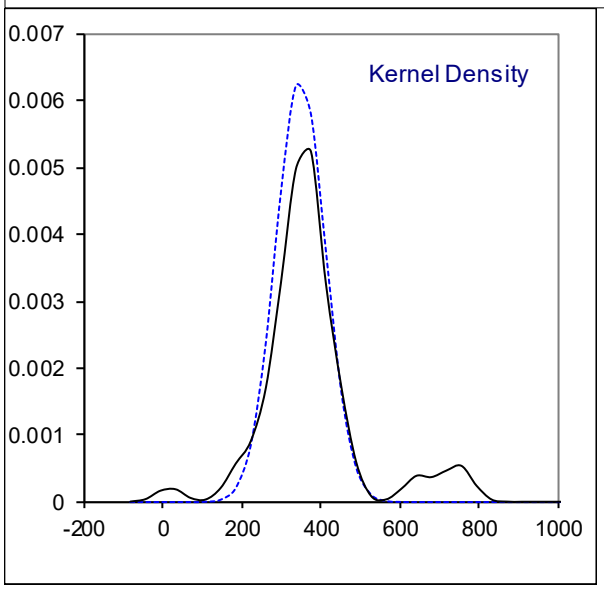
Determination of Total Bisphenol A (BPA) in polycarbonate sample #19547; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	360.82		0.30	
339	In house	326.8		-0.43	
362		-----		-----	
551	In house	225.08	C	-2.60	First reported 2.25
623	In house	422.89		1.63	
826	In house	369.3		0.48	
2115	EN14372	720	C,R(0.01)	7.97	First reported 116.37
2129	D7574	420.5		1.57	
2172	In house	370.20		0.50	
2190	In house	658.8	R(0.01)	6.66	
2236	In house	433.079		1.84	
2237		-----		-----	
2250	In house	346.92		0.00	
2255	In house	270.2		-1.64	
2256	In house	356.2		0.20	
2267	In house	197.8	C	-3.18	First reported 107.8
2293	EN14372	274.923		-1.53	
2301	In house	294.0		-1.13	
2310	In house	314		-0.70	
2311	In house	293.941		-1.13	
2330	In house	332.637		-0.30	
2347	In house	368		0.45	
2350	In house	364.50		0.38	
2352	JETRO-2D2	361.7		0.32	
2357	In house	367.6		0.44	
2363	In house	367		0.43	
2365	JETRO	365.41		0.40	
2366	In house	379.8		0.71	
2369	In house	360		0.28	
2375	In house	305		-0.89	
2377	In house	374.45		0.59	
2379	In house	439.254		1.98	
2380	In house	318.23		-0.61	
2382	In house	366.0		0.41	
2384	In house	359.629		0.27	
2386	In house	412.9		1.41	
2390	INH-239	347.48		0.01	
2462	EPA3550C/8321B	460		2.42	
2475	In house	344.902		-0.04	
2489	In house	359		0.26	
2495	In house	385.56		0.83	
2503	In house	630	R(0.01)	6.05	
2532	In house	363		0.35	
2590	In house	175.969		-3.65	
2665	In house	760.1	R(0.01)	8.83	Reported also 92366.1
2672	In house	230.89		-2.48	
2736	In house	424.87		1.67	
2826	In house	280		-1.43	
2841	In house	750.61	C,R(0.01)	8.63	First reported 0.0007506
2893	In house	387.0		0.86	
3116	In house	297.5		-1.05	
3146	In house	306		-0.87	
3163	In house	15	R(0.01)	-7.09	
3166	In house	432.9		1.84	
3172	EN14372	463		2.48	
3176	In house	2551	C,R(0.01)	47.08	First reported 2127.5
3190	In house	311.88		-0.75	
3199	In house	353.35		0.14	
3237	EN14372	9536	C,R(0.01)	196.29	First reported 5104
3248	In house	297		-1.06	

normality	OK	
n	50	
outliers	8	
mean (n)	346.781	
st.dev. (n)	62.8901	RSD = 18%
R(calc.)	176.092	
st.dev.(EN14372:04)	46.8155	
R(EN14372:04)	131.083	

	Only as received		Only further cut/grinded		Only DCM		Only THF	
normality	OK		suspect		OK		OK	
n	27		20		24		14	
outliers	3		4		0		3	
mean (n)	342.606		340.911		348.146		319.004	
st.dev. (n)	62.8070	RSD=18%	57.9267	RSD=17%	55.8566	RSD=16%	70.7148	RSD=22%
R(calc.)	175.860		162.195		156.399		198.001	
st.dev.(EN14372:04)	46.2518		46.0230		46.9997		43.0655	
R(EN14372:04)	129.505		128.864		131.599		120.584	

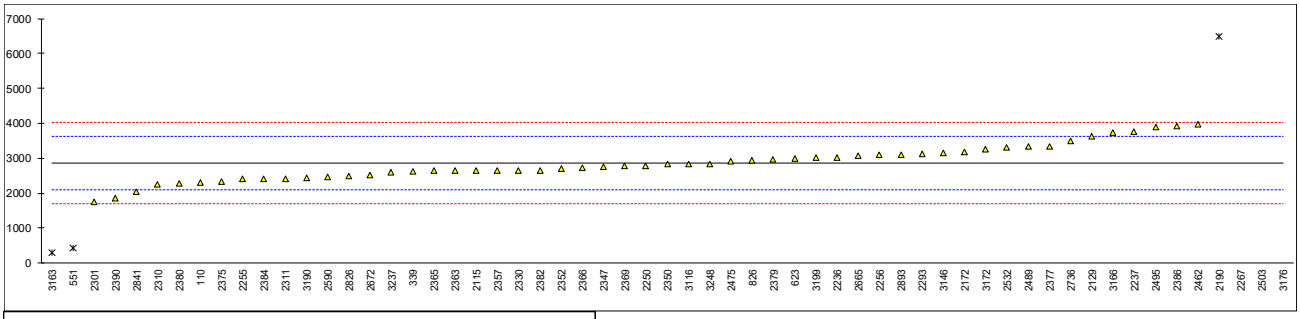


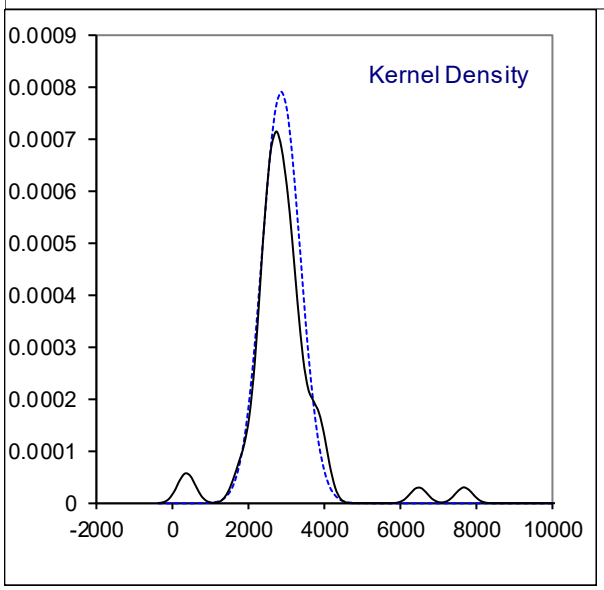


Determination of Total Bisphenol A (BPA) in a PVC sample #19548; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	2306.79		-1.44	
339	In house	2624.6		-0.61	
362		-----		-----	
551	In house	434.24	C,R(0.01)	-6.28	First reported 12.03
623	In house	3005.99		0.37	
826	In house	2952.2		0.23	
2115	EN14372	2650	C	-0.55	First reported 369.42
2129	D7574	3635		2.00	
2172	In house	3187.5		0.84	
2190	In house	6486.4	R(0.01)	9.38	
2236	In house	3024.533		0.42	
2237	In house	3762		2.33	
2250	In house	2786.6		-0.19	
2255	In house	2410		-1.17	
2256	In house	3096.5		0.61	
2267	In house	7682.46	C,R(0.01)	12.48	First repored 4540.33
2293	EN14372	3137.286		0.71	
2301	In house	1740.00		-2.90	
2310	In house	2250		-1.58	
2311	In house	2419.42		-1.14	
2330	In house	2653.599		-0.54	
2347	In house	2763		-0.26	
2350	In house	2834.17		-0.07	
2352	JETRO-2D2	2712.7		-0.39	
2357	In house	2650.3		-0.55	
2363	In house	2650		-0.55	
2365	JETRO	2646.39		-0.56	
2366	In house	2730.1		-0.34	
2369	In house	2775		-0.22	
2375	In house	2320		-1.40	
2377	In house	3349.40		1.26	
2379		2965.92		0.27	
2380	In house	2269.56		-1.53	
2382	In house	2660.0		-0.52	
2384	In house	2416.419		-1.15	
2386	In house	3918		2.73	
2390	INH-239	1848.24		-2.62	
2462	EPA3550C/8321B	3980		2.89	
2475	In house	2916.027		0.14	
2489	In house	3334		1.22	
2495	In house	3890.64		2.66	
2503	In house	17770	R(0.01)	38.59	
2532	In house	3299		1.13	
2590	In house	2476.947		-1.00	
2665	In house	3081.1		0.57	
2672	In house	2521.6		-0.88	
2736	In house	3503.69		1.66	
2826	In house	2500		-0.94	
2841	In house	2039.40	C	-2.13	First reported 0.00204
2893	In house	3105		0.63	
3116	In house	2837.9		-0.06	
3146	In house	3154		0.76	
3163	In house	293	R(0.01)	-6.65	
3166	In house	3740.3		2.27	
3172	EN14372	3249		1.00	
3176	In house	34970	C,R(0.01)	83.11	First reported 20546
3190	In house	2439.02		-1.09	
3199	In house	3014.67		0.40	
3237	EN14372	2600		-0.68	
3248	In house	2840		-0.06	
	normality	OK			
	n	53			
	outliers	6			
	mean (n)	2861.764			
	st.dev. (n)	504.8789	RSD = 18%		
	R(calc.)	1413.661			
	st.dev.(EN14372:04)	386.3382			
	R(EN14372:04)	1081.747			

	Only as received		Only further cut/grinded		Only DCM		Only THF	
normality	OK		OK		suspect		suspect	
n	29		20		23		14	
outliers	1		5		1		3	
mean (n)	2904.336		2764.471		2746.037		2908.036	
st.dev. (n)	491.7423	RSD=17%	445.2454	RSD=16%	465.5359	RSD=17%	477.4338	RSD=16%
R(calc.)	1376.879		1246.687		1303.501		1336.815	
st.dev.(EN14372:04)	392.0854		373.2036		370.7150		392.5849	
R(EN14372:04)	1097.839		1044.970		1038.002		1099.238	





APPENDIX 2
Analytical details as reported by the participating laboratories

Lab	ISO17025 accr.	sample grinded or cut	final particle size	sample intake (g)	extraction solvent	extraction time (min)	extraction temp (°C)
110	Yes	Further Cut		0.5			
339	No	Used as received	3 x 2 mm	0.5	Toluene/DCM (50/50)	30	70
362	---	---					
551	No	Further Cut	-	0.5	DCM	30	40
623	Yes	Further Cut	2 x 2mm	0.5	DCM	30	40
826	No	Used as received			DCM	30	40
2115	Yes	Used as received	0.5 cm	0.5	THF/Hexane	60	60
2129	Yes	Used as received	not determined	0,5	THF	30	60
2172	Yes	Used as received	3 x 3mm	0.5	#19547 DCM #19548 THF	60	40
2190	No	Further Cut	$2mm^2$	1	ACN	1440	23
2236	Yes	Used as received	2 x 2mm	0.5	Chloroform: Methanol (2:1)	60	70
2237	Yes	Further Grinded	>1mm	0,25	Toluene	60	room temp
2250	Yes	Used as received	Used as received	2,5	THF	30	40
2255	Yes	Further Cut		0.3	THF/ACN	30	70
2256	Yes	Further Cut	2 x 2mm	1.0	THF	60	60
2267	No	Further Grinded		0.1	THF/Ethanol,	120	60
2293	No	Used as received		0.5	THF/ACN (5 / 10)	70	
2301	Yes	Used as received	5 x 5 mm	1	THF or DCM	60	60 or 40
2310	Yes	Used as received	5 x 5mm	1	DCM	30	40
2311	Yes	Further Cut	<2mm	1	DCM and Acetone	30	40
2330	Yes	Used as received		0.50	20 ml of DCM, 100 ml of Acetone, 5 ml of ACN and 5 ml of 50% ACN with milliporewater	30	40±2
2347	Yes	Further Cut					
2350	No	Used as received	2 x 2mm	0.5g	MEC	30	40
2352	Yes	Further Cut	2 x 2 x 2mm	1	DCM	30	40
2357	Yes	Used as received	2 x 2mm	1	DCM	30	40
2363	Yes	Further Cut	2 x 2mm	1	DCM	30	40
2365	Yes	Further Cut	< 2 x 2mm	1	DCM	30	40
2366	No	Further Cut	2mm*2mm*2mm	0.5	DCM	30	40
2369	Yes	Further Cut	2mm*2mm*2mm	0.5	DCM	30	40
2375	Yes	Further Cut	2 x 2mm	0,5	DCM	30	40
2377	Yes	Used as received	2 x 2mm	1.0	DCM	30	40
2379	Yes	Used as received	3 x 3mm	0.5	DCM	30	40
2380	Yes	Used as received	As Received	0.5	DCM	30	40
2382	No	Further Cut	2 x 2mm	1.000	DCM	30	40
2384	Yes	Further Grinded	<500um	0.5	DCM	30	40
2386	Yes	Used as received	3 x 3mm	0,2 (#19547) 0,03 (#19548)	DCM	30	40
2390	---	---					
2462	---	---					
2475	No	Used as received		1	Chloroform: Methanol (2:1)	60	70
2489	Yes	Further Cut	2 x 2mm	0.5	THF/ACN	35	70
2495	Yes	Used as received		0.5	Chloroform: Methanol (2:1)	60	70
2503	Yes	Further Cut		0.10	THF:n-Hexane	90	70
2532	Yes	Further Cut	Powder / 2mm	0.5	THF:ACN:H2O (1:2:3)	60	70
2590	No	Used as received	3 x 3mm	0.5	THF	60	40
2665	---	---					
2672	Yes	Used as received		0,5	DCM/MeOH 90:10 (v/v)	60	60
2736	No	Further Cut	2 x 2 mm		DCM	1 hr	
2826	Yes	Used as received			DCM	30	40
2841	No	Further Grinded	< 220µm	0.5	Methanol	4300 min	60
2893	Yes	Used as received		0.5	THF/ACN (1:2)	30	25
3116	Yes	Used as received	5 x 5 mm	1			
3146	No	Used as received		0,5	Chloroform: Methanol (2:1)	60	70
3163	No	Further Cut	0.5mm	0.0005	--	--	--
3166	Yes	Used as received		0.5	Methylene Chloride	--	--
3172	---	---					
3176	No	Used as received	-	1	THF/ACN	60	40
3190	Yes	Further Cut	2 x 2mm	1.0	Chloroform: Methanol (2:1)	60	70
3199	No	Used as received	3 x 3mm.	1.0 / 0.5	PC = DCM PVC = THF	60	40
3237	Yes	Used as received		0,1	#19547: DCM/ACN/Acetone #19548: DCM/ACN	30	40
3248	Yes	Used as received		1	THF/MeOH	30	40

APPENDIX 3

Number of participating laboratories per country

2 labs in	BANGLADESH
1 lab in	BRAZIL
1 lab in	BULGARIA
1 lab in	CAMBODIA
3 labs in	FRANCE
8 labs in	GERMANY
1 lab in	GUATEMALA
4 labs in	HONG KONG
4 labs in	INDIA
2 labs in	INDONESIA
5 labs in	ITALY
2 labs in	KOREA
1 lab in	MALAYSIA
12 labs in	P.R. of CHINA
1 lab in	PAKISTAN
1 lab in	THAILAND
2 labs in	THE NETHERLANDS
3 labs in	TURKEY
6 labs in	U.S.A.

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, June 2018
2 ASTM E178:02
3 ASTM E1301:03
4 ISO 5725:86
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9 IP 367:84
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11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
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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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