Results of Proficiency Test
Total Bisphenol A in Polymers
May 2021

Organized 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. The Bisphenol A can transfer readily to the skin in small amounts, especially when the skin is dry and free of grease.

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 2020/2021 it was decided to continue the proficiency test for the analysis of Total Bisphenol A in polymers.

In this interlaboratory study 57 laboratories in 18 different countries registered for participation. See appendix 3 for the number of participants per country. In this report the results of the Total Bisphenol A in Polymers 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, both positive on BPA. The first sample was orange Polypropylene (PP) granulates of 3 grams labelled #21600. The second sample was transparent Polycarbonate (PC) granulates of 3 grams labelled #21601.
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 organization 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 selected batch for the first sample was orange Polypropylene (PP) granulates artificially fortified to be positive on Bisphenol A. From this batch 100 small plastic bags were filled with approximately 3 grams each and labelled #21600.
The homogeneity of the subsamples was checked by the determination of Total BPA with an in-house test method on 8 stratified randomly selected subsamples.

	Total BPA in mg/kg
Sample #21600-1	1522
Sample #21600-2	1537
Sample #21600-3	1481
Sample #21600-4	1481
Sample #21600-5	1508
Sample #21600-6	1581
Sample #21600-7	1603
Sample #21600-8	1537

Table 1: homogeneity test results of subsamples #21600

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2, in the next table.

	Total BPA in mg/kg
r (observed)	122.3
reference test method	EN14372:04
0.3 x R (reference test method)	173.6

Table 2: evaluation of the repeatability of subsamples #21600

The calculated repeatability is in agreement with 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

The selected batch for the second sample was transparent Polycarbonate (PC) granulates artificially fortified to be positive on Bisphenol A. From this batch 100 small plastic bags were filled with approximately 3 grams each and labelled #21601. The batch for sample #21601 was used in a previous proficiency test on BPA in polymers (as sample #17565 in iis17P04). Therefore, homogeneity of the subsamples was assumed

To each of the participating laboratories one sample labelled #21600 and one sample labelled #21601 was sent on May 5, 2021.

2.5 ANALYZES

The participants were requested to determine the Total Bisphenol A content on both samples #21600 and #21601. It was also requested to report if the laboratory was accredited for the requested component 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 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 test results cannot 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 as well as the reference test methods (when applicable) 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 appendices 1 and 2 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. The original test results are placed under 'Remarks' in the result tables in appendix 1 and 2. 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 organization 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 test results. Test 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.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. 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 the averages and the 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 test 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. This 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

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 proficienct test (PT) against the literature requirements, e.g. EN reproducibilities, 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, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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 according to:

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

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

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

	|z|	< 1	good
1 <	|z|	< 2	satisfactory
2 <	|z|	< 3	questionable
3 <	|z|		unsatisfactory

4 Evaluation

In this proficiency test no problems were encountered with the dispatch of the samples. Five participants reported test results after the final reporting date and four other participants did not report any test results.
In total 53 participants reported 106 numerical test results. Observed were 5 outlying test results, which is 4.7%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

One of the two data sets was found to have no normal Gaussian distribution. This one is referred to as “suspect”. The statistical evaluation of this data set 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 test methods are also in the tables together with the original data. The abbreviations, used in these tables, are explained in appendix 4.

No official test method exists for the determination of the total content of BPA in polymers. It was therefore decided to use the requirements from the test 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 #21600

Total BPA: The determination of Total Bisphenol A in the Polypropylene (PP) sample was very problematic. One statistical outlier was observed and three other test results were excluded. The calculated reproducibility after rejection of the suspect data was very large. Therefore, no z-scores are calculated. See also the discussion in paragraph 5.

Sample #21601

Total BPA: The determination of Total Bisphenol A in the Polycarbonate (PC) sample was not problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the estimated reproducibility of EN14372:04.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test method (in casu EN test method) are presented in the next tables.

Component	unit	n	average	2.8 * sd	R(lit)
Total Bisphenol A	mg/kg	49	911	838	(344)

Table 3: reproducibilities of BPA determination on sample #21600

Component	unit	n	average	2.8 * sd	R(lit)
Total Bisphenol A	mg/kg	49	2127	795	804

Table 4: reproducibilities of BPA determination on sample #21601

Without further statistical calculations, it can be concluded that for sample #21600 (Polypropylene (PP) granulates) there is not a good compliance of the group of participants with the reference test method. For sample #21601 (Polycarbonate (PC) granulates) there is a good compliance of the group of participants with the reference test method. See also the discussion in paragraphs 4.1 and 5.

4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2021 WITH PREVIOUS PTS

	May 2021	June 2020	June 2019	May 2018	May 2017
Number of reporting laboratories	53	56	59	69	55
Number of test results	106	110	117	133	108
Number of statistical outliers	5	4	14	9	8
Percentage of statistical outliers	4.7%	3.6%	10.7%	6.3%	6.9%

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 (RSD) of the PTs, see next table.

Matrix	May 2021	June 2020	June 2019	May 2018	2014 - 2017	EN14372
Polycarbonate (PC)	13%	16%	18%	21%	14%	14%
Polyethylene (PE)	n.e.	n.e.	n.e.	n.e.	24%	14%
Polypropylene (PP)	33%	40%	n.e.	n.e.	34 - 54%	14%
Polyvinylchloride (PVC)	n.e.	n.e.	18%	n.e.	21 - 23%	14%
Thermal paper (TP)	n.e.	n.e.	n.e.	11%	12%	14%

Table 6: development of uncertainties in BPA in polymers determinations over the years

The uncertainty of BPA in PC did improve compared to previous years. The determination of BPA in PP improved but is still high for the group of participants compared to the reference test method EN14372.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

It appeared that almost all participants used an in-house test method for the determination of Total BPA (46 laboratories = 88%).

It is requested whether a participant is accreditated for the determination of Total BPA and some analytical details. The reported details are given in appendix 2. Based on the answers given by the participants the following can be summarized:

- Thirty-four participants answered to be ISO/IEC17025 accredited for the determination of Total BPA in polymers (= 68%).
- About 46% of the participants did use the samples as received and about 54% of the participants further cut or further grinded the samples prior to analyses.

- About 64% of the participants used a sample intake between 0.5 and 1.0 grams and about 34% of the reporting participants mentioned to have used <0.5 grams.
- The solvent (mixture) to release the BPA from the samples differs. About 46% of the participants used Dichloromethane and 44% of the participants used Tetrahydrofurane (THF) as solvent. The effect of solvent is further investigated and reported in appendix 1, see also discussion in paragraph 5.
- Almost all participants did use an extraction time between 30 and 60 min.
- About 38% of the participants reported to have used an extraction temperature of 40°C and about 52% mentioned to have used an extraction temperature of 60-70°C.

5 DISCUSSION

In this proficiency test the determination of Total BPA in polymers two different sample matrices were used: Polypropylene (PP #21600) and Polycarbonate (PC #21601). For the PP sample the observed reproducibility was much larger compared to the reproducibility as estimated from the reference test method of EN14372:04. For the PC sample a good compliance of the reproducibility was observed compared to test method EN14372:04.
It occurs that releasing BPA from Polypropylene is far more difficult than from Polycarbonate resulting in a higher relative standard deviation (RSD) of 33% compared to an RSD of 13% from the PC matrix for the same group of participants. The different solvents may have an influene on the extraction of BPA from the Polypropylene, but this influence is less profound on the extraction from Polycarbonate. When compared in the Polypropylene sample the group that used DCM as solvent found a lower average than the group that used THF (786 vs 1003 mg/kg), see appendix 1 for PP and PC. The RSD of the THF group is smaller than for DCM (23% vs 42%) but is still large compared to target of 14% of EN14372:04.
Therefore, it was decided not to use one of the two groups for assigned values and to calculate no z-scores.

Sample #21601 was used earlier as sample #17565 in the PT iis17P04 (2017). In table 7 a comparison is given over the two proficiency tests.

	Sample #21601				Sample #17565			
	unit	n	average	R(calc)	unit	n	average	R(calc)
Total BPA	mg/kg	49	2127	795	mg/kg	48	2124	841

Table 7: comparison of sample #21601 with #17565

It is observed that the average level of Total BPA in the 2021 PT is quite good in line with the 2017PT and the observed reproducibility R(calc) for BPA has slightly improved in 2021. This is not uncommon. Each time that a laboratory participates in a PT it has the opportunity to learn from the evaluation of the results and improve the analysis.

6 CONCLUSION

For the analysis of Total BPA from polymers a sound test method which prescribe the analysis of Total BPA from different polymers in detail is desirable, especially for other polymers than Polycarbonate. Also, the choice of solvent may play a role in the determination other polymers than in Polycarbonate.

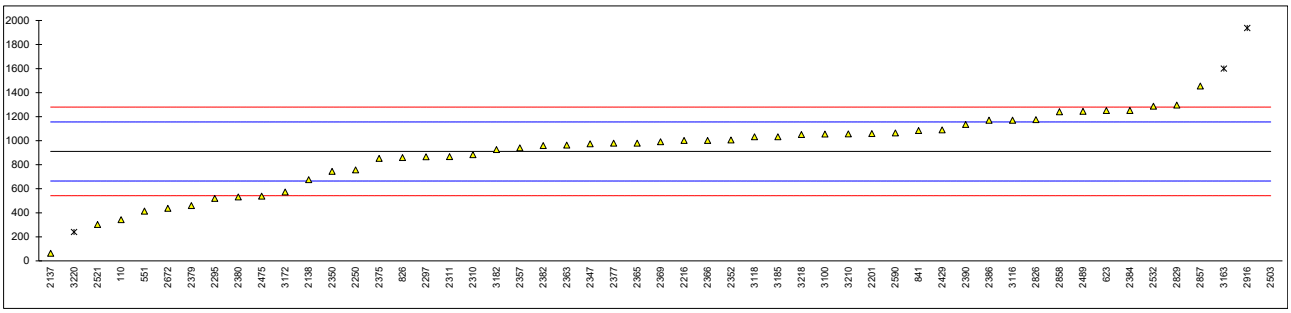
It can be concluded that the group of participants have problems with the determination of Total BPA in the Polypropylene sample in this proficiency test. Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, 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 Polypropylene sample #21600; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	342.4		-----	
339		-----		-----	
551	JETRO	414.69	C	-----	First reported 244.97
623	In house	1250.670		-----	
826	In house	860.2		-----	
841	In house	1085.1		-----	
2137	KS M1997	63.35		-----	
2138	In house	674.9		-----	
2201	In house	1059.3		-----	
2216	In house	1002		-----	
2236		-----		-----	
2250	In house	757		-----	
2295	In house	520		-----	
2297	ASTM D 7574 MOD	866		-----	
2310	In house	884		-----	
2311	In house	867.08		-----	
2347	In house	974		-----	
2350	In house	744.68		-----	
2352	JETRO	1005.6		-----	
2357	In house	941.6		-----	
2363	In house	963		-----	
2365	In house	980.5		-----	
2366	In house	1002.1		-----	
2369	In house	992		-----	
2372		-----		-----	
2375	In house	853		-----	
2377	In house	980		-----	
2379	JETRO	460.43	C	-----	First reported 382.66
2380	In house	532.3		-----	
2382	In house	960.4		-----	
2384	In house	1251.1307		-----	
2386	In house	1170	C	-----	First reported 354.9
2390	In house	1135.49		-----	
2429	In house	1089		-----	
2475		539.35		-----	
2489	In house	1245		-----	
2503	In house	3507	R(0.01)	-----	
2521	In house	303	C	-----	First reported 1820
2532	In house	1287		-----	
2590	In house	1064.961		-----	
2644		-----		-----	
2672	In house	437.9	C	-----	First reported 334.3
2826	In house	1175		-----	
2829	In house	1296.49		-----	
2857	EPA3550C/8321B	1455		-----	
2858	In house	1240.88		-----	
2916	In house	1937	ex	-----	Test result excluded as outlier in BPA in Polycarbonate
3100	In house	1055.2		-----	
3116	In house	1170.5		-----	
3118	In house	1032.8567		-----	
3163	In house	1600	ex	-----	Test result excluded as outlier in BPA in Polycarbonate
3172	In house	573		-----	
3182	In house	926.46		-----	
3185	In house	1033.44		-----	
3210	In house	1056.92		-----	
3218	In house	1051.96		-----	
3220	In house	240.3	ex	-----	Test result excluded as outlier in BPA in Polycarbonate

			Only DCM	Only THF
	normality	OK	OK	OK
	n	49	23	20
	outliers	1 + 3ex	0	1 + 1 ex
	mean (n)	910.752	785.707	1003.255
	st.dev. (n)	299.1090 RSD = 33%	327.4630 RSD = 42%	235.1706 RSD = 23%
	R(calc.)	837.505	916.897	658.478
	st.dev.(EN14372:04)	(122.9515)	(106.0704)	(135.4395)
	R(EN14372:04)	(344.264)	(296.997)	(379.231)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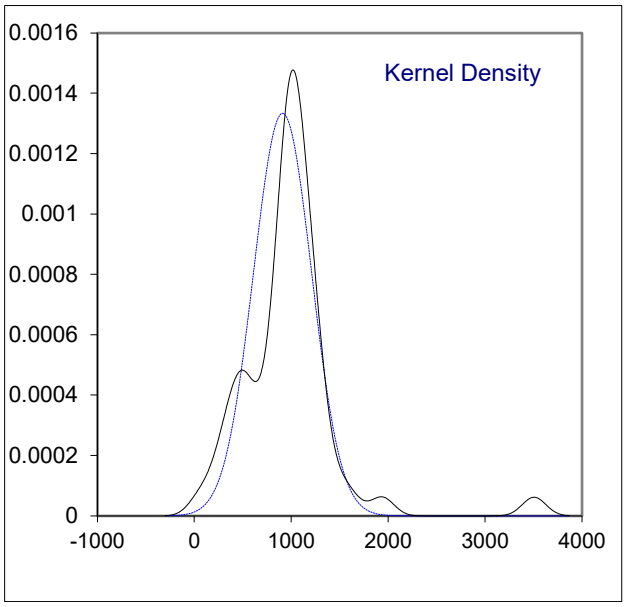
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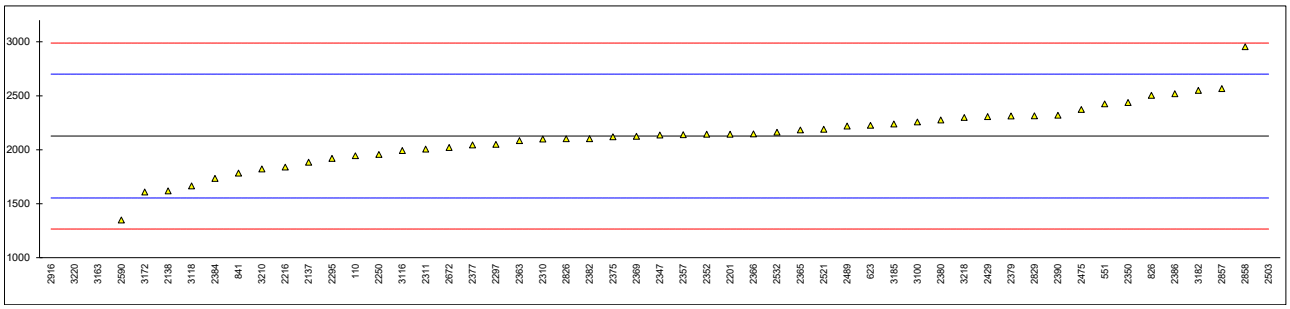
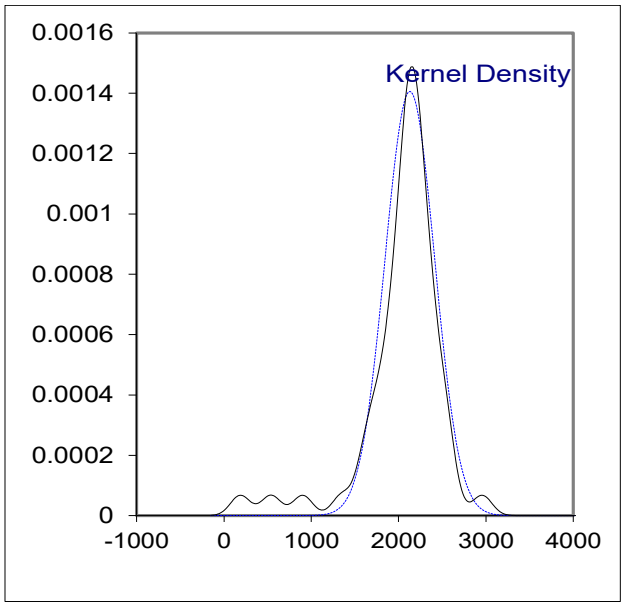
0.0014
0.0012
0.001
0.0008
0.0006
0.0004
0.0002
0
-1000
0
1000
2000
3000
4000

Determination of Total Bisphenol A (BPA) in a Polycarbonate sample #21601; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
110	In house	1945.2		-0.63	
339		-----		-----	
551	JETRO	2425.64		1.04	
623	In house	2226.540		0.35	
826	In house	2504.7		1.31	
841	In house	1784.2		-1.20	
2137	KS M1997	1885.66		-0.84	
2138	In house	1618.5	C	-1.77	First reported 1119.6
2201	In house	2145.6		0.06	
2216	In house	1840		-1.00	
2236		-----		-----	
2250	In house	1957		-0.59	
2295	In house	1920		-0.72	
2297	ASTM D 7574 MOD	2050		-0.27	
2310	In house	2100		-0.10	
2311	In house	2006.27		-0.42	
2347	In house	2137		0.03	
2350	In house	2437.91		1.08	
2352	JETRO	2144.5		0.06	
2357	In house	2140.3		0.04	
2363	In house	2085		-0.15	
2365	In house	2183.4		0.19	
2366	In house	2147.7		0.07	
2369	In house	2125		-0.01	
2372		-----		-----	
2375	In house	2120		-0.03	
2377	In house	2045		-0.29	
2379	JETRO	2313.341		0.65	
2380	In house	2276.0		0.52	
2382	In house	2103.5		-0.08	
2384	In house	1734.4173		-1.37	
2386	In house	2519.56		1.37	
2390	In house	2320.10		0.67	
2429	In house	2307		0.63	
2475		2373.49		0.86	
2489	In house	2220		0.32	
2503	In house	6457	R(0.01)	15.07	
2521	In house	2190		0.22	
2532	In house	2163		0.12	
2590	In house	1349.079		-2.71	
2644		-----		-----	
2672	In house	2021	C	-0.37	First reported 1203
2826	In house	2102		-0.09	
2829	In house	2314.63		0.65	
2857	EPA3550C/8321B	2567		1.53	
2858	In house	2955.06		2.88	
2916	In house	186	R(0.01)	-6.76	
3100	In house	2258.4		0.46	
3116	In house	1992.5		-0.47	
3118	In house	1666.1207		-1.61	
3163		900	R(0.01)	-4.27	
3172	In house	1609		-1.81	
3182	In house	2551.65		1.48	
3185	In house	2240.03		0.39	
3210	In house	1822.80		-1.06	
3218	In house	2299.49		0.60	
3220	In house	537.7	R(0.01)	-5.54	
					Only DCM / Only THF
	normality	suspect			not OK / suspect
	n	49			24 / 19
	outliers	4			0 / 2
	mean (n)	2127.435			2116.020 / 2165.827
	st.dev. (n)	283.8453	RSD = 13%		260.581 RSD = 12% / 308.4572 RSD = 14%
	R(calc.)	794.767			729.626 / 863.680
	st.dev.(EN14372:04)	287.2037			285.6627 / 292.3866
	R(EN14372:04)	804.170			799.856 / 818.683


3000
2500
2000
1500
1000
2916 3220 3163 2590 3172 2138 3118 2384 841 3210 2216 2137 2295 110 2250 3116 2311 2672 2377 2297 2363 2310 2826 2382 2375 2369 2347 2357 2352 2201 2366 2532 2365 2521 2489 623 3185 3100 2380 3218 2429 2379 2829 2390 2475 551 2350 826 2386 3182 2857 2858 2503


Kernel Density
0.0016
0.0014
0.0012
0.001
0.0008
0.0006
0.0004
0.0002
0
-1000
0
1000
2000
3000
4000

APPENDIX 2 Analytical details as reported by the participating laboratories

Lab	ISO17025 accr.	sample used as received or grinded/cut	sample intake (g)	extraction solvent	extraction time (min)	extraction temp (°C)
110	Yes	Further cut	1	dichloromethane	30	38
339	---	---				
551	No	Used as received	0.5g	DCM/Acetone	30 min	40°C
623	Yes	Further cut	0.5	THF	60	60
826	No	Further cut	0.5	DCM	60	40
841	Yes	Further cut	0.6 grams	Tetrahydrofuran	60 minutes	60°C
2137	Yes	Used as received	1	MeOH, DCM	40	25
2138	No	Used as received	0.1	THF/MeOH	60	60
2201	Yes	Further cut	0.5g	TFH and ACN	30 min	70°C
2216	Yes	Further grinded	1.38	Methylene Chloride/Methanol	60	Ambient
2236	---	---				
2250	Yes	Used as received	0,3 g	THF	60	60
2295	Yes	Further cut	0.2 grams	THF	60	25
2297	Yes	Used as received	1g	THF+Methanol	60	40
2310	Yes	Used as received	1	THF	60	60
2311	Yes	Further cut	0.5	Dichloromethane and Acetone	30	40
2347	Yes	Further cut	0.25g	Dichloromethane	30min±2min	40℃±2℃
2350	Yes	Used as received	0.5 g	DCM	30 min	40 °C
2352	Yes	Further cut	1g	dichloromethane	30min	40°C
2357	---	---				
2363	Yes	Further cut	0.5g	DCM	30min	40
2365	Yes	Further cut	1.0g	Dichloromethane	30min	40℃
2366	No	Further cut	0.5g	DCM	30min	40℃
2369	No	Further cut	0.2g			
2372	---	---				
2375	Yes	Further cut	0.5 gram	Dichloromethane	30 min	40 °C
2377	---	---				
2379	Yes	Used as received	0.2 grams	DCM	30 minute	40°C
2380	Yes	Used as received	0.5 g	Dichloromethane	30 Minutes	40 °C
2382	Yes	Further cut	1.000g±0.010g	DCM (Dichloromethane)	30min	40±2℃
2384	Yes	Further grinded	0.5g	Dichloromethane	180	40
2386	Yes	Further grinded	0,01	Dichlormethane	30	40
2390	No	Further cut	0.5 gram/0.5 gram	Dichloromethane (DCM)	30 minute	30 °C
2429	Yes	Used as received	0.5001g	THF	30 minutes	70°C
2475	No	Used as received	0.1g	THF	30 min	70°C
2489	Yes	Further cut	0.5060g/0.5001g	THF/ACN/WATER	30	70°C
2503	No	Used as received	0.05	THF / n-Hexane	90	70
2521	Yes	Used as received	0.1 gr	Dichloromethane, acetone	60 minutes	Room temperature
2532	Yes	Further cut	0.5 grams	THF,ACN,Water	30 minutes	70°C
2590	No	Further cut	0.5g	DCM for Polycarbonate, THF for other material	60 min	60°C
2644	---	---				
2672	Yes	Used as received	0,1	Dichlormethane/Methanol 90/10 (v/v)	60	60
2826	Yes	Used as received	0.5g	Dichloromethane + methanol	30 mins	40°C
2829	No	Further cut	0.2	THF/Exane	60	60
2857	No	#21600-Further grinded #21601- Further cut	#21600- 0.2481 g #21601- 0.2541 g	Acetone	120 minutes	60°C
2858	Yes	Used as received	0.3 gm	Tetrahydrofuran (THF)	60 min	60
2916	No	Further grinded	0,5	n-Hexane	360	69
3100	Yes	Used as received	0.3g	Tetrahydrofuran,Acetonitrile	30minutes	70℃
3116	No	Used as received	1g	Method for other materials: Tetrahydrofuran / Methanol, 1:1 MeOH / water Method for PC: Dichloromethane / Acetone, 1:1 MeOH / water	60 minutes	For other materials: 60°C; for PC: 40°C
3118	Yes	Further cut	0.5 gram	THF:ACN:Water (1:2:3)	35 minute	70°C
3163	No	Further cut	0.0005	-	-	-
3172	---	---				
3182	Yes	Used as received	#21600 = 0.1 g #21601 = 0.05 g	Tetrahydrofuran : Acetonitrile : water (1:2:3)	30 minutes	70°C
3185	Yes	Used as received	0.5g	Tetrahydrofuran	30minutes	70℃
3210	No	Used as received	1 gram	Toluene	60 min	60°C
3218	Yes	Used as received	0.5g	Tetrahydrofuran	30min	70°C
3220	No	Used as received	0.5g	Methanol, THF	60 min	60°C

APPENDIX 3

Number of participants per country

2 labs in BANGLADESH
1 lab in BRAZIL
3 labs in FRANCE
4 labs in GERMANY
3 labs in HONG KONG
5 labs in INDIA
2 labs in INDONESIA
4 labs in ITALY
1 lab in MALAYSIA
14 labs in P.R. of CHINA
1 lab in PAKISTAN
4 labs in SOUTH KOREA
2 labs in TAIWAN
2 labs in THAILAND
1 lab in THE NETHERLANDS
3 labs in TURKEY
4 labs in U.S.A.
1 lab 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	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
SDS	= Safety Data Sheet

Literature

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