Institute for
Interlaboratory Studies

Results of Proficiency Test
Total Bisphenol A in Polymers
June 2023

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis23P64

September 2023

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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 determination of Total Bisphenol A in Polymers every year. During the annual proficiency testing program 2022/2023 it was decided to continue the proficiency test for the determination of Total Bisphenol A in Polymers.

In this interlaboratory study 72 laboratories in 21 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 polymer samples of 3 grams each labelled #23615 and #23616 respectively.
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

For the first sample a batch of brown Polyvinylchloride (PVC) blocks was selected which was artificially fortified with Bisphenol A by a third party. After homogenization 75 small plastic bags were filled with approximately 3 grams each and labelled #23615.
The batch for sample #23615 was used in a previous proficiency test on Total Bisphenol A in Polymers as sample #19548 in iis19P04. Therefore, homogeneity of the subsamples was assumed.

For the second sample a batch of transparent Polycarbonate (PC) granulates was selected which artificially fortified with Bisphenol A by a third party. After homogenization 75 small plastic bags were filled with approximately 3 grams each and labelled #23616.
The batch for sample #23616 was used in a previous proficiency test on Total Bisphenol A in Polymers as sample #19547 in iis19P04. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one polymer sample labelled #23615 and one polymer sample labelled #23616 was sent on May 24, 2023.

2.5 ANALYZES

The participants were requested to determine the Total Bisphenol A content on both samples #23615 and #23616. 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’ test 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 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 test results are 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 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 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 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 proficiency test (PT) against the literature requirements (derived from e.g. ISO or ASTM test methods), 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 result is fit-for-use.

The z-scores were calculated according to:

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

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 two other participants did not report any test results.
In total 70 participants reported 138 numerical test results. Observed were 6 outlying test results, which is 4.3%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Both data sets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section the reported test results are discussed per sample and per component. 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 in appendix 1. 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. For the evaluation of the test results of this interlaboratory study 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”. 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 #23615

Total BPA: The determination of Total Bisphenol A in the PVC sample was problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility of EN14372:04.

sample #23616

Total BPA: The determination of Total Bisphenol A in the Polycarbonate sample was problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not 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 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 reference methods are presented in the next tables.

Component	unit	n	average	2.8 * sd	R(lit)
Total Bisphenol A	mg/kg	66	2932	1252	1108

Table 1: reproducibility of BPA determination on sample #23615

Component	unit	n	average	2.8 * sd	R(lit)
Total Bisphenol A	mg/kg	66	321	158	121

Table 2: reproducibility of BPA determination on sample #23616

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF JUNE 2023 WITH PREVIOUS PTS

	June 2023	June 2022	May 2021	June 2020	June 2019
Number of reporting laboratories	70	57	53	56	59
Number of test results	138	112	106	110	117
Number of statistical outliers	6	10	5	4	14
Percentage of statistical outliers	4.3%	8.9%	4.7%	3.6%	10.7%

Table 3: 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 to uncertainties observed in PTs over the years, expressed as relative standard deviation (RSD) of the PTs, per matrix see next table.

Matrix	June 2023	June 2022	May 2021	June 2020	2014 - 2019	EN14372
Polycarbonate (PC)	18%	15%	13%	16%	14 - 21%	14%
Polyethylene (PE)	---	20%	---	---	24%	14%
Polypropylene (PP)	---	---	33%	40%	34 - 54%	14%
Polyvinylchloride (PVC)	15%	---	---	---	18% - 23%	14%

Table 4: development of the uncertainties over the years

The uncertainties observed in this PT are comparable to the uncertainties observed in previous PTs.

Sample #23615 was used in a previous PT as sample #19548 in iis19P04. The average and calculated reproducibility for the component in the 2023 PT are in line with the corresponding parameters in the 2019 PT.

Component	unit	sample #23615			sample #19548		
		n	average	R(calc)	n	average	R(calc)
Total Bisphenol A	mg/kg	66	2932	1252	53	2862	1414

Table 5: comparison of sample #23615 with #19548

Sample #23616 was used in a previous PT as sample #19547 in iis19P04. The average and calculated reproducibility for the component in the 2023 PT are in line with the corresponding parameters in the 2019 PT.

Component	unit	sample #23616			sample #19547		
		n	average	R(calc)	n	average	R(calc)
Total Bisphenol A	mg/kg	66	321	158	50	347	176

Table 6: comparison of sample #23616 with #19547

4.4 EVALUATION OF THE ANALYTICAL DETAILS

It appeared that 87% of the participants used an in-house test method for the determination of Total BPA. For this PT some analytical details were requested, the reported details are given in appendix 2. Based on the answers given by the participants the following can be summarized:

- 67% answered to be ISO/IEC17025 accredited for the determination of Total BPA in polymers.
- 49% used the samples as received, 45% further cut and 6% further grinded the samples prior to analyses.
- 78% used a sample intake between 0.5 and 1.0 grams and 22% mentioned to have used less than 0.5 grams.
- 58% used (a mix of) Tetrahydrofurane (THF) as solvent to release the BPA from the samples while 40% used (a mix of) Dichloromethane.
- Almost all participants did use an extraction time between 30 and 60 min.
- 57% mentioned to have used an extraction temperature of 60-70 °C, 35% used an extraction temperature of 40°C and the others used either 70 °C or room temperature.

In this PT the effect of the reported analytical details of sample size and release solvent was not significant on the averages or variation between reported test results.

5 DISCUSSION

In this proficiency test the determination of Total BPA in polymers was performed on two different sample matrices: Polyvinylchloride (PVC) (#23615) and Polycarbonate (#23616). For both samples the observed reproducibility was somewhat larger compared to the reproducibility as estimated from the reference test method of EN14372:04, but in line with previous findings for this type of polymers.

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.

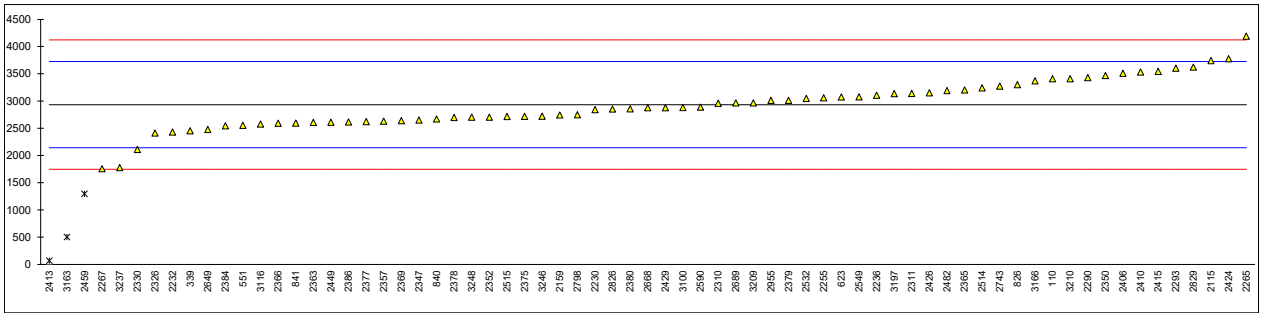
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

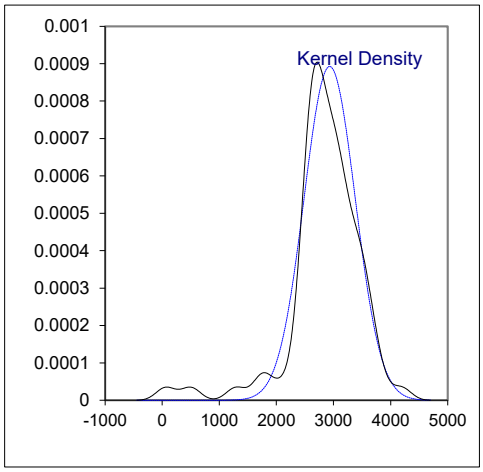
APPENDIX 1

Determination of Total Bisphenol A (BPA) in a PVC sample #23615; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	3407.5		1.20	
339	In house	2452.8		-1.21	
551	In house	2551.5373		-0.96	
623	In house	3072.10		0.35	
826	In house	3298.58		0.93	
840	In house	2669		-0.67	
841	In house	2593.2		-0.86	
2115	In house	3741		2.04	
2137		-----		-----	
2159	In house	2740.40		-0.48	
2230	In house	2839		-0.24	
2232	In house	2430		-1.27	
2236	In house	3101.9		0.43	
2255	EN14372	3057.0		0.31	
2265	In house	4188.6		3.17	
2267		1754.39		-2.98	
2290	In house	3425.9		1.25	
2293	EN14372	3600		1.69	
2310	In house	2956		0.06	
2311	In house	3134.57		0.51	
2326		2410.29		-1.32	
2330	JETRO2009	2107.61		-2.08	
2347	In house	2649.75		-0.71	
2350	In house	3465.14		1.35	
2352	JETRO2009	2701.92		-0.58	
2357	In house	2627.91		-0.77	
2363	In house	2606.5		-0.82	
2365	In house	3201.71		0.68	
2366	In house	2589		-0.87	
2369	In house	2637		-0.75	
2375	In house	2716		-0.55	
2377	In house	2620.14		-0.79	
2378	In house	2695.27		-0.60	
2379	JETRO2009	3011.28		0.20	
2380	In house	2857.0		-0.19	
2384	In house	2542.79		-0.98	
2386	In house	2613		-0.81	
2406	In house	3505		1.45	
2410	In house	3530		1.51	
2413	In house	64.78	R(0.01)	-7.24	
2415	In house	3542.1		1.54	
2424	In house	3774		2.13	
2426	ISO11936	3149.32	C	0.55	first reported as BPA in the Polycarbonate sample #23616
2429	In house	2876.0		-0.14	
2449	EN14372	2607		-0.82	
2459	ISO/DIS11936	1294.5	R(0.05)	-4.14	
2482	In house	3190		0.65	
2514	In house	3239.41		0.78	
2515	In house	2713.00		-0.55	
2532	In house	3047		0.29	
2549	In house	3075.841		0.36	
2590	In house	2885.652		-0.12	
2649	In house	2474.48		-1.16	
2668	In house	2874.21		-0.15	
2689	In house	2963.22		0.08	
2743	In house	3272.50		0.86	
2798	In house	2747		-0.47	
2826	EN14372	2849		-0.21	
2829	In house	3619.4		1.74	
2955	In house	3011.2		0.20	
2977		-----		-----	
3100	In house	2877.46		-0.14	
3116	In house	2576		-0.90	
3118		-----		-----	
3163	In house	501	R(0.01)	-6.14	
3166	In house	3370.7895		1.11	
3197	In house	3134.5		0.51	
3209	In house	2964.7		0.08	
3210	In house	3407.57		1.20	
3237	In house	1775.00	C	-2.92	first reported 331.21
3246	In house	2720.75		-0.53	
3248	In house	2700		-0.59	

normality	OK	
n	66	
outliers	3	
mean (n)	2932.392	
st.dev. (n)	446.9936	RSD=15%
R(calc.)	1251.582	
st.dev.(EN14372:04)	395.8729	
R(EN14372:04)	1108.444	

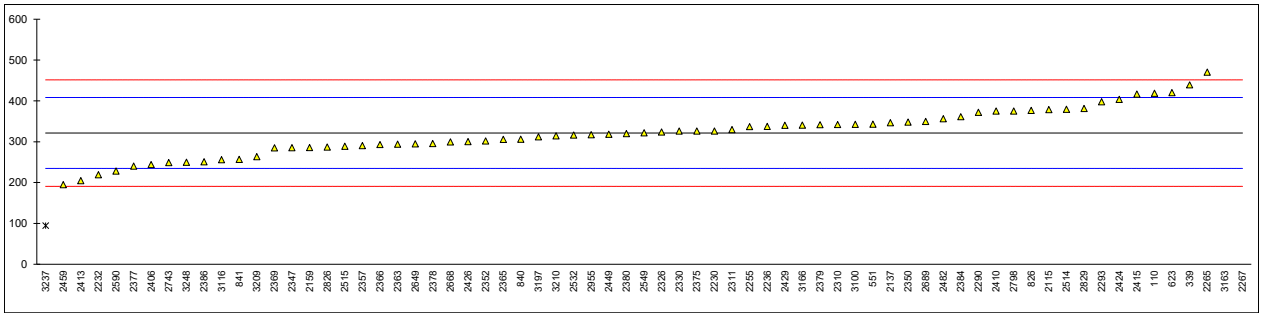


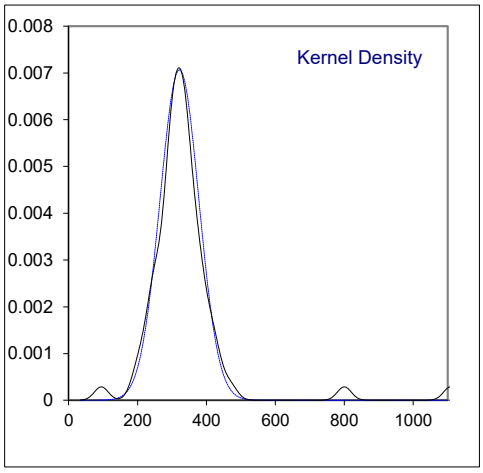


Determination of Total Bisphenol A (BPA) in a Polycarbonate sample #23616; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	418.33		2.23	
339	In house	439.097		2.71	
551	In house	342.9862		0.50	
623	In house	420.41		2.28	
826	In house	376.95		1.28	
840	In house	306		-0.35	
841	In house	256.9		-1.49	
2115	In house	378.7		1.32	
2137	KS M1997	346.2		0.57	
2159	In house	285.70		-0.82	
2230	In house	326		0.11	
2232	In house	219		-2.36	
2236	In house	337.5		0.37	
2255	EN14372	337.0		0.36	
2265	In house	470.3		3.43	
2267		1108.3	R(0.01)	18.14	
2290	In house	372.1		1.17	
2293	EN14372	397.97		1.77	
2310	In house	342		0.48	
2311	In house	329.56		0.19	
2326		323.99		0.06	
2330	JETRO2009	325.87		0.10	
2347	In house	285.54		-0.83	
2350	In house	348.02		0.61	
2352	JETRO2009	301.70		-0.45	
2357	In house	290.43		-0.71	
2363	In house	294.2		-0.63	
2365	In house	305.53		-0.37	
2366	In house	293		-0.65	
2369	In house	285		-0.84	
2375	In house	326		0.11	
2377	In house	240.35		-1.87	
2378	In house	295.37		-0.60	
2379	JETRO2009	341.58		0.47	
2380	In house	320.0		-0.03	
2384	In house	361.09		0.92	
2386	In house	250.83		-1.63	
2406	In house	244		-1.78	
2410	In house	375		1.24	
2413	In house	204.51		-2.69	
2415	In house	416.7		2.20	
2424	In house	403.7		1.90	
2426	ISO11936	300.21	C	-0.49	first reported as BPA in the PVC sample #23615
2429	In house	340.3		0.44	
2449	EN14372	318.52		-0.07	
2459	ISO/DIS11936	194.6		-2.92	
2482	In house	356		0.80	
2514	In house	379.27		1.33	
2515	In house	288.96		-0.75	
2532	In house	316.3		-0.12	
2549	In house	321.73		0.01	
2590	In house	228.182		-2.15	
2649	In house	294.9561		-0.61	
2668	In house	299.61		-0.50	
2689	In house	349.44		0.65	
2743	In house	248.80		-1.67	
2798	In house	375		1.24	
2826	EN14372	287		-0.79	
2829	In house	381.6		1.39	
2955	In house	317.1		-0.10	
2977		-----		-----	
3100	In house	342.57		0.49	
3116	In house	256		-1.51	
3118		-----		-----	
3163	In house	800	R(0.01)	11.03	
3166	In house	340.4975		0.44	
3197	In house	311.7		-0.22	
3209	In house	263.4		-1.34	
3210	In house	314.22		-0.17	
3237	In house	94.60	C,R(0.05)	-5.23	first reported 53.22
3246		-----		-----	
3248	In house	250		-1.65	

normality	OK	
n	66	
outliers	3	
mean (n)	321.380	
st.dev. (n)	56.4622	RSD=18%
R(calc.)	158.094	
st.dev.(EN14372:04)	43.3863	
R(EN14372:04)	121.482	





APPENDIX 2 Analytical details

lab	ISO17025 Accr.	sample used as received or grinded/cut	sample intake (g)	extraction solvent	extraction time (minutes)	extraction temperature (°C)
110	Yes	Used as received	1 g	Dichloromethane for extraction and Acetone for precipitation	30 minutes	40°C
339	Yes	Used as received	0.5	Dichlorométhane	30	70
551	No	Further cut	0.5g	DCM/Acetone	---	---
623	Yes	Further cut	1	THF Methanol	60	60
826	No	Further grinded	0.1 g	DCM	60 minutes	40 °C
840	Yes	Further cut	0.5	THF	60	60
841	Yes	Further cut	1g	THF	60 min	60 °C
2115	No	Used as received	0.4 g	THF	60 min	60 °C
2137	Yes	Used as received	1 g	DCM	60 MIN	---
2159	Yes	Further cut	1 gr	THF: Methanol	60 minutes	60°C
2230	Yes	Used as received	0.5g	thf	60	60
2232	Yes	Used as received	1g	PVC - THF Polycarbonate - DCM	PVC - 60min Polycarbonate - until dissolve	PVC - 60C Polycarbonate - room temperature
2236	Yes	Used as received	0.5100 and 0.5004	Chloroform:Methanol (2:1)	60	70
2255	Yes	Further cut	0.5	THF+ACN	60	60
2265	No	Used as received	0,5g	THF	30	70
2267	---	---	---	---	---	---
2290	Yes	---	---	---	---	---
2293	Yes	Further grinded	0.5 GRAMS	THF	60 MINUTES	60°C
2310	Yes	Used as received	1	THF:Methanol	60	60
2311	Yes	Further cut	0.2	THF/Methanol	60	60
2326	Yes	Further cut	1 gm	Tetrahydrofurane	60 min	60 C
2330	Yes	Used as received	0.5 g	DCM	30 min	40 C
2347	No	Further cut	1g	ACN:H2o=1:1	30mins	40℃
2350	Yes	Further cut	0.5g	DCM	30min	40°C
2352	Yes	Further cut	1g	Dichloromethane	30min	40℃
2357	---	---	---	---	---	---
2363	Yes	Further cut	0.5g	DCM	30min	40
2365	Yes	Further cut	0.1g	Dichloromethane	30min	40℃
2366	No	Further cut	0.5	DCM	30	40
2369	---	---	---	---	---	---
2375	Yes	Further cut	0,5 gram	DCM	30±1 dk	40±2 °C
2377	Yes	Used as received	1.0	DCM	30	40
2378	No	Used as received	0.5g	THF	60min	60°C
2379	Yes	Used as received	0.2 g	DCM	30 minutes	40 C
2380	Yes	Used as received	1.0 g	Tetrahydrofuran (THF)	60 Minute	60 °C
2384	Yes	Further grinded	0.5g	Dichloromethane	30 min	40 oC
2386	Yes	Used as received	0.5 g	Dichlormethane	30 min	40°C
2406	No	Used as received	#23615: 0.4 gram #23616: 0.2 gram	#23615: THF #23616: DCM	#23615: 30 minutes #23616: 60 minutes	#23615: 60C #23616: 40C
2410	Yes	Used as received	0.1 g	THF+ACN	60 minutes	(60 ± 2) ℃
2413	No	Further cut	Sample 23615 - 0.5012 grams Sample 23616 - 0.4994 grams	Tetrahydrofuran/Acetonitrile	30 minutes for both	40 C for Tetrahydrofuran for 30 mimutes followed by Room Temperature for 30 minutes for Acetonitrile
2415	Yes	Further cut	1 grams	THF/ACN	60 minutes	60 °C
2424	No	Used as received	0.25	Tetrahydrafurane, then diluted with H20 to a final 1:1 ratio	60	60
2426	Yes	Used as received	0.5g	THF:ACN:H2O	60 min	60 °C
2429	Yes	Used as received	0.5002g	THF	30 minutes	70℃
2449	No	Further cut	1.0 gram	DCM , Methanol and THF	60	40 C
2459	No	Used as received	1 grams	THF & n-Hexane	60 minutes	60 °C
2482	No	Used as received	0,1	DCM:MeOH 90:10	60	Room Temp
2514	Yes	Further cut	23615=0.1881 23616=0.3138	THF/ACN/water 1:2:3	60	60
2515	Yes	Used as received	1.0007g	Tetrahydrofuran (THF)	60 minutes	40°C
2532	Yes	Further cut	0.5 grams	THF:ACN: Water [1:2:3]	65 minutes	60 °C
2549	Yes	Further cut	0.5 gram	Dichloromethane:Methanol	60 minutes	26 deg C
2590	No	Used as received	0.5g	THF - ACN	60 min	50°C
2649	No	Further cut	0.5 gram	Tetrahydrofuran + methanol	sonicate 60 min	60
2668	Yes	Used as received	0.5	DCM, MeOH	60 min	room temp

lab	ISO17025 Accr.	sample used as received or grinded/cut	sample intake (g)	extraction solvent	extraction time (minutes)	extraction temperature (°C)
2689	Yes	Further cut	0.1g	tetrahydrofuran and dichloromethane	60 mins	40°C
2743	No	Used as received	0.5	THF	60	60
2798	No	Further cut	0.5	THF/MEOH	60min	60
2826	Yes	Used as received	0.5 g	Dichloromethane (DCM)	30 minutes	40°C
2829	No	Further cut	0.2	THF/n-hexane	60	60
2955	Yes	Further cut	0.5	THF+ ACN+H2O	60	60
2977	---	---	---	---	---	---
3100	Yes	Used as received	0.5g	THF	60min	60℃
3116	No	Used as received	1	For PC material, use dichloromethane followed by acetone For non-PC material, use tetrahydrofuran followed by methanol	60	For PC material, 40 deg.C For non-PC material, 60 deg.C
3118	---	---	---	---	---	---
3163	No	Further cut	0.05	ethanol	120	90
3166	Yes	Further grinded	0.500	Methylene chloride	15	20
3197	Yes	Further cut	0,5g	THF/ACN	30 min (1.process) 5 min (2.process)	70C
3209	Yes	Further cut	0.1g	DCM	60min	room temperature
3210	No	Used as received	0.5 gram	Toluene	60 minutes	60 °C
3237	No	Used as received	0,5	MeOH: THF	30	40
3246	Yes	---	---	---	---	---
3248	Yes	Used as received	1g	20	60 minutes	60

APPENDIX 3

Number of participants per country

5 labs in BANGLADESH

1 lab in BRAZIL

1 lab in CAMBODIA

2 labs in FRANCE

3 labs in GERMANY

1 lab in GUATEMALA

5 labs in HONG KONG

5 labs in INDIA

2 labs in INDONESIA

5 labs in ITALY

4 labs in KOREA, Republic of

1 lab in MALAYSIA

13 labs in P.R. of CHINA

4 labs in PAKISTAN

1 lab in SINGAPORE

1 lab in TAIWAN

1 lab in THAILAND

2 labs in THE NETHERLANDS

4 labs in TURKEY

5 labs in U.S.A.

6 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	= 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

Literature

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