Results of Proficiency Test
total Bisphenol A in Polymers
May 2016

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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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.

Bisphenol A is classified in Directive 2009/48/EC under Regulation (EC) No 1272/2008 as toxic. In the absence of any specific requirements, bisphenol A can be contained in toys in concentrations equal to or smaller than the relevant concentration established for the classification of mixtures containing it as CMRs, namely 5 % as from 20 July 2013 and 3 % as from 1 June 2015 respectively. It cannot be excluded that that concentration may lead to increased exposure to bisphenol A, compared to the migration limit of 0.1 mg/l for bisphenol A set by European standards EN 71-9:2005+A1:2007, EN 71-10:2005 and EN 71-11:2005.

The determination of Bisphenol A in plastics is known to give problems with the comparability of laboratory results. However, no appropriate Bisphenol A reference materials are yet available. As an alternative, participation in a proficiency test may enable laboratories to check their performance. Therefore, a proficiency test (laboratory-evaluating interlaboratory study) for the determination of Bisphenol A in plastics was organized by the Institute for Interlaboratory Studies in April 2014. This PT was continued in the 2015 and 2016 PT program.

In this proficiency test 56 laboratories in 20 different countries have registered for participation. See appendix 3 for the number of participating laboratories per country.
In this report the results of the 2016 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 in Spijkenisse, the Netherlands, was the organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO17025 accredited laboratory.
It was decided to send two different plastic samples. The first sample, a PP granulate, was especially prepared by a Chinese factory by addition of Bisphenol A to PP and subsequent homogenization and extrusion. The second sample, a PE granulate, was especially prepared by a Chinese factory by addition of Bisphenol A to PE and subsequent homogenization and extrusion. The participants were requested to report rounded and unrounded test results and also some details of the sample preparation and the test procedure. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC 17043:2010. This ensures strict adherence to protocols for

sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on a regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol can be downloaded from the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

Two different materials, one PP and one PE, both artificially fortified to be positive on Bisphenol A (with respective approx. 1800 mg/kg and 900 mg/kg), were selected. Both materials were divided over plastic bags, approx. 3 grams for each sample.
The homogeneity of the subsamples was checked by determination of Bisphenol A (BPA) content on 8 stratified randomly selected subsamples.

	total BPA in mg/kg		total BPA in mg/kg
Sample #16565-1	1690	Sample #16566-1	771
Sample #16565-2	1712	Sample #16566-2	814
Sample #16565-3	1688	Sample #16566-3	802
Sample #16565-4	1634	Sample #16566-4	813
Sample #16565-5	1631	Sample #16566-5	820
Sample #16565-6	1659	Sample #16566-6	782
Sample #16565-7	1732	Sample #16566-7	793
Sample #16565-8	1651	Sample #16566-8	795

Table 1: homogeneity test results of the subsamples #16565 and #16566

From the above test results the repeatabilities were calculated. Comparison of the repeatabilities with 0.3 times the estimated reproducibility of EN14372:04 in agreement with the procedure of ISO 13528, Annex B2 was not possible, because EN14372:04 does not mention a reproducibility, but only a repeatability. Therefore the comparison was made with the repeatability of EN14372:04.

	total BPA in mg/kg	total BPA in mg/kg
r (observed) #16565	103	--
r (observed) #16566	--	47
reference test method	EN14372:04	EN14372:04
r (ref. test method)	211	101

Table 2: evaluation of repeatabilities of BPA contents of the subsamples #16565 and #16566

For both samples #16565 and #16566, the observed repeatability of the 8 test results of the homogeneity study is smaller than the repeatability of the reference test method and therefore the homogeneity of subsamples #16565 and #16566 was assumed.

To each of the participating laboratories, one sample of approx. 3 grams PP granulate, labelled #16565 and one sample of approx. 3 grams PE granulate, labelled #16566, were sent on April 20, 2016.

2.5 ANALYSES

The participants were requested to determine and report the total Bisphenol A content on both samples #16565 and #16566 applying the analysis procedure that is routinely used in the laboratory.
To get comparable test results, a detailed report form, on which the analytes and the units were prescribed as well as the reference test method and a letter of instructions were prepared. Both were made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. A form to confirm receipt of the sample and a letter of instructions were added to the sample package.

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 reanalysis). 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.
A list of abbreviations used in the tables can be found in appendix 3.

3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3) 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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

According to ISO 5725 the original test results per determination were submitted subsequently to Dixon's, Grubbs' and Rosner 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 General ESD 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 General ESD 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. When the uncertainty passed the evaluation no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.
Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to visualise 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 under 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 individual participating laboratories the z-scores were calculated. In order to be able to have an objective evaluation of the performance of the individual participants, it was decided to evaluate this performance against the literature requirements. Therefore the z-scores were calculated using a target standard deviation. This target standard deviation was calculated from the literature reproducibility by division with 2.8.

The $z_{(target)}$-scores were calculated according to:

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

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

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.

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:

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

4 EVALUATION

In this interlaboratory study no problems were encountered during the execution.

Three participants did not report any test results for unknown reasons. Finally, 53 laboratories reported 105 numerical test results. Observed were 3 statistically outlying test results, which is 2.8% of all numerical test results. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER SAMPLE

In this section the results are discussed per sample.

Due to the lack of a suitable test method with precision data for the determination of total BPA in polymers, it was decided to use the requirements from the standardised method EN14372:04, “Child use and care articles, Cutlery and feeding utensils, Safety requirements and tests” for evaluation of the results of this interlaboratory study.
Regretfully, only a relative within-laboratory standard deviation RSDr is given in EN14372:04. Multiplication of RSDr by 2.8 gives the repeatability. Multiplication of the repeatability by 3 gives a good estimate of the target reproducibility. For comparison the estimated reproducibility calculated using the Horwitz equation is also given.
A number of laboratories did report migration test results instead of total BPA test results, using test methods EN14372, EN14350 -2 or CEN/TS13130-13. These test results were excluded from the statistical evaluations.

Sample #16565

BPA: The determination of total Bisphenol A in this PP sample was problematic at the level of 1400 mg/kg. No statistical outliers were detected, but 28 test results were excluded from the statistical evaluation, because the samples were not cut or grinded prior to the extraction step and 3 other test results were excluded for being migration results. The influence of the cutting/grinding was large for this sample, in general resulting in a significantly higher BPA concentration than for the determination on the sample as received. See also the discussion in chapter 6.
The calculated reproducibility after rejection of the 31 suspect test results is not in agreement with the estimated reproducibility of EN14372:04.

Sample #16566

BPA: The determination of total Bisphenol A in this PE sample was problematic at the level of 490 mg/kg. Three statistical outliers were detected, all three being migration test results. The influence of the cutting/grinding was small for this sample, and therefore no test results were excluded from the statistical evaluation as was done for sample #16566.
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 reproducibilities as found for the group of participating laboratories and the estimated reproducibilities of EN14372:2004 (R_{target}) in the next tables:

Parameter	Unit	n	Average	2.8 * sd	R (target)
Bisphenol A (total)	mg/kg	21	1423	962	538

Table 3: overview of results for sample #16565

Parameter	Unit	n	Average	2.8 * sd	R (target)
Bisphenol A (total)	mg/kg	50	489	410	185

Table 4: overview of results for sample #16566

5 COMPARISON OF THE PROFICIENCY TEST OF MAY 2016 WITH THE PREVIOUS PTS

	May 2016	April 2015	April 2014
Number of reporting labs	53	53	60
Number of results reported	105	104	120
Number of statistical outliers	3	6	6
Percentage outliers	2.8%	5.5%	4.8%

Table 5: Comparison with previous proficiency tests

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

Parameter	Conc. in mg/kg	May 2016	April 2015	April 2014	Est. EN14372
BPA	<1000	30%	54%	n.e.	13.5%
BPA	1000 – 2500	24%	23%	34%	13.5%
BPA	>2500	n.e.	n.e.	21%	13.5%

Table 6: Development of relative uncertainties over the years

The uncertainty in the test result of BPA for concentrations < 1000 mg/kg in the 2016 PT iis16P05 has improved in comparison with the previous PT, but it is still not in line with the uncertainty requirements of the method (see table 4).

6 DISCUSSION

No standard test method for the determination of total BPA in polymers has been published. Therefore it is no wonder that most often “in house” (46 laboratories = 87%) was mentioned as test method used. Regretfully a number of laboratories did report migration test results instead of total BPA test results. These migration test results were excluded from the statistical evaluations.
Laboratory 2665 used Xylene to dissolve the samples completely and thus it did avoid the difficulties of the extraction step. The test results of this laboratory are both high as to be expected (resp. 1816 and 746 mg/kg). These test results are probably most close to the true

total BPA contents of the samples. Two other laboratories used Thermal Desorption. The test results of these laboratories 2521 and 3163 are both high for sample #16565, but not high for sample #16566.
Only 21 laboratories did cut or grind the samples prior to the extraction step, while 27 other labratories did use the samples as received, which is the main reason that a number of the latter laboratories reported a low BPA concentration for sample #16565.
From the analytical details in appendix 2, it can be noticed that several different extraction techniques and solvents were used. In the previous report iis14P04 of 2014, it was observed that the calculated reproducibility for one sample was smaller (and the consensus value was higher) when only the reported results were evaluated of the laboratories that used Ultrasonic as release technique. This year the majority of the participants (42 = 79%) used Ultrasonic as release technique. However, the extraction solvents used vary over a large range.

As in previous years the influence of several analysis details on the test results were investigated. When for both samples the data sets for results from Ultrasonic extraction, from the use of dichloromethane (DCM) as extraction and from the use of the mixture chloroform/methanol (2:1) as extraction solvent are compared, it is clear that the use of one defined solvent mixture as extraction solvent has by far the largest influence on the dispersion of the test results, see tables 7 and 8. It would be a large improvement when a standard test method would be published prescribing the standard test conditions so that all laboratories would use the same extraction solvent (mixture).
It should be noted that from the 12 laboratories that used the mixture chloroform/methanol (2:1) as extraction solvent, only 4 laboratories did cut or grind the samples and 8 did use the samples as received. This number is too small to see the expected improvement when both factors (cutting/griding and use of the same extraction mixture) are done by the group of laboratories.

subset of test results	n	average in mg/kg	sd in mg/kg	RSD%
BPA (only ultrasonic)	41	1089	483	44%
BPA (only dichloromethane)	16	1207	553	46%
BPA (only chloroform/MeOH=2:1)	11	1247	285	23%
BPA (cut + chloroform/MeOH=2:1)	4	1430	225	16%

Table 7: overview of separate evaluation for sample #16565

subset of test results	n	Average	sd in mg/kg	RSD%
BPA (only ultrasonic)	42	492	148	30%
BPA (only dichloromethane)	16	462	100	22%
BPA (only chloroform/MeOH=2:1)	12	578	101	17%
BPA (cut + chloroform/MeOH=2:1)	4	510	122	24%

Table 8: overview of separate evaluation for sample #16566

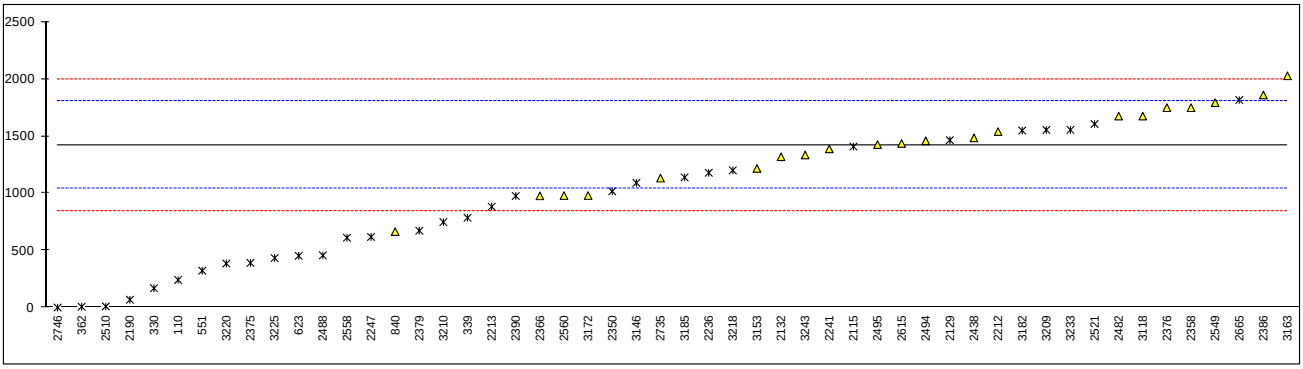
It can be concluded that the observed spread in this interlaboratory study may not be caused by just one critical point in the analysis. Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.

APPENDIX 1

Determination of Total Bisphenol A (BPA) on sample #16565; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	243.48	C, ex	-6.14	first reported 299.10
330	In house	171	ex	-6.52	
339	In house	786.47	C, ex	-3.31	first reported 87.6
362	CEN/TS13130-13	9.30	ex	-7.36	test result is not for total BPA, but for migration of BPA
551	In house	324.18	C, ex	-5.72	first reported 306.71
623	In house	454.15	C, ex	-5.04	first reported 362.75
840	In house	665		-3.94	
2115	In house	1408.77	ex	-0.07	
2129	In house	1465	ex	0.22	
2132	In house	1320		-0.53	
2190	DGGCCRF	70.33	ex	-7.04	Method used for Law 2012-1442
2212	In house	1540		0.61	
2213	Health Can. C36	884	ex	-2.80	
2216		-----		-----	
2236	In house	1179.89	ex	-1.26	
2241	In house	1388.88		-0.18	
2247	In house	619.13	ex	-4.18	
2293		-----		-----	
2295		-----		-----	
2350	In house	1017.8	ex	-2.11	
2358	In house	1748.81		1.70	
2366	In house	978		-2.31	
2375	In house	392.64	ex	-5.36	
2376	In house	1748.81		1.70	
2379	EN14372	673.59	ex	-3.90	
2386	In house	1860		2.28	
2390	JETRO-2009	977.68	ex	-2.32	
2438	In house	1484.84		0.32	
2475		-----		-----	
2482	In house	1675		1.31	
2488	In house	457.74	ex	-5.02	
2494	In house	1458.99		0.19	
2495	In house	1426.05		0.02	
2510	EN14372	11.88	C, ex	-7.35	first reported 1188.19, is not total BPA, but for migration of BPA
2521	In house	1606.5	C, ex	0.96	first reported EN14372 migration result 3.99
2549	In house	1792.315		1.93	
2558	In house	612	ex	-4.22	
2560	In house	981		-2.30	
2615	EPA3550C	1436.14		0.07	
2665	In house	1816	ex	2.05	
2735	In house	1133.36		-1.51	
2746	EN14350 -2	1.838	ex	-7.40	test result is in µg/ml; is not for total BPA, but for migration of BPA
3118	In house	1675.58		1.32	
3146	In house	1091.93	ex	-1.72	
3153	In house	1218		-1.07	
3163	In house	2027.034		3.15	
3172	In house	981		-2.30	
3182	In house	1548.9	ex	0.66	
3185	In house	1139.96	ex	-1.47	
3209	In house	1554.2	ex	0.69	
3210	In house	748.66	ex	-3.51	
3218	In house	1201.42	ex	-1.15	
3220	In house	387	C, ex	-5.39	first reported 257.9
3225	In house	434.855	ex	-5.14	
3233	In house	1554.31	ex	0.69	
3243	In house	1335		-0.46	

			All test results:	"As received" test results:
	normality	OK	OK	OK
	n	21	50	27
	outliers	0 + 31 excl.	2	0
	mean (n)	1422.56	1117.67	829.36
	st.dev. (n)	343.479	508.055	521.469
	R(calc.)	961.74	1422.55	1460.11
	R(EN14372:04)	537.73	422.48	313.50
Comp.	R(Horwitz)	213.68	174.09	135.12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2000
1500
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0
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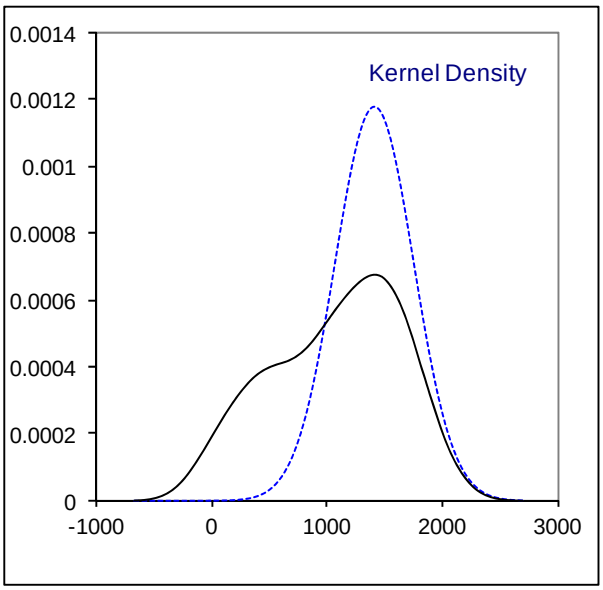
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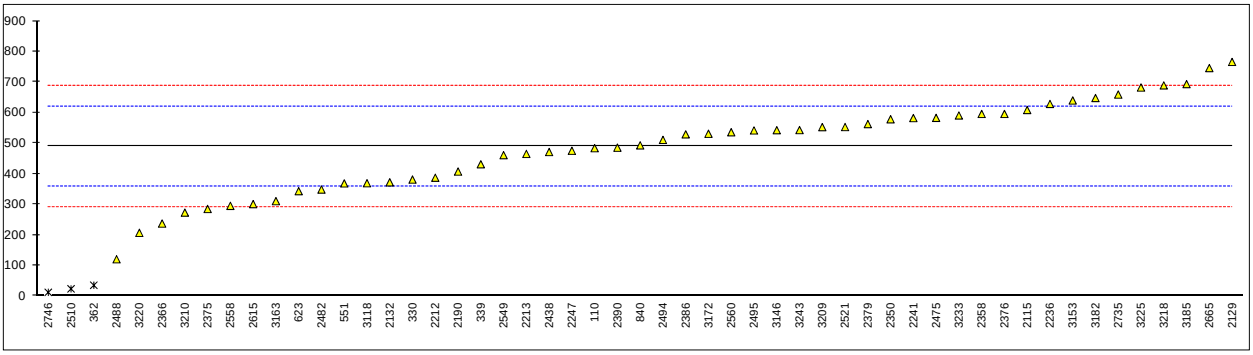
0.0012
0.001
0.0008
0.0006
0.0004
0.0002
0
-1000
0
1000
2000
3000

Determination of Total Bisphenol A (BPA) on sample #16566; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	483.81		-0.08	
330	In house	381		-1.64	
339	In house	431		-0.88	
362	CEN/TS13130-13	35.10	R(0.05)	-6.88	test result is not for total BPA, but for migration of BPA
551	In house	368.39		-1.83	
623	In house	343.06		-2.21	
840	In house	493		0.06	
2115	In house	608.63		1.81	
2129	In house	766		4.19	
2132	In house	372		-1.77	
2190	DGGCCRF	407.2		-1.24	Method used for Law 2012-1442
2212	In house	387		-1.55	
2213	Health Can. C36	465		-0.37	
2216		-----		-----	
2236	In house	628.59		2.11	
2241	In house	582.37		1.41	
2247	In house	475.23		-0.21	
2293		-----		-----	
2295		-----		-----	
2350	In house	578.4		1.35	
2358	In house	595.73		1.61	
2366	In house	237		-3.82	
2375	In house	284.69		-3.10	
2376	In house	595.73		1.61	
2379	EN14372	562.63		1.11	
2386	In house	529		0.60	
2390	JETRO-2009	485.41		-0.06	
2438	In house	471.17		-0.27	
2475	In house	582.97		1.42	
2482	In house	348.4		-2.13	
2488	In house	120.11		-5.59	
2494	In house	510.88		0.33	
2495	In house	541.77		0.80	
2510	EN14372	23.20	C,R(0.05)	27.72	first reported 2320.1, is not total BPA, but for migration of BPA
2521	In house	552.9	C	0.96	first reported EN14372 migration result 2.2
2549	In house	461.035		-0.43	
2558	In house	295		-2.94	
2560	In house	536		0.71	
2615	EPA3550C	300.76		-2.85	
2665	In house	746		3.89	
2735	In house	659.23		2.57	
2746	EN14350 -2	11.900	R(0.05)	-7.23	test result is in µg/ml; is not for total BPA, but for migration of BPA
3118	In house	368.89		-1.82	
3146	In house	542.70		0.81	
3153	In house	640		2.28	
3163	In house	310.451		-2.71	
3172	In house	531		0.63	
3182	In house	647.9		2.40	
3185	In house	693.55		3.09	
3209	In house	552.75		0.96	
3210	In house	272.75		-3.28	
3218	In house	689.32		3.03	
3220	In house	206.6		-4.28	
3225	In house	682.340		2.92	
3233	In house	590.84		1.54	
3243	In house	543		0.81	

					"cut & grinded" results:	"As received" test results:
	normality	OK			OK	OK
	n	50			21	28
	outliers	3			1	1
	mean (n)	489.18			476.88	480.79
	st.dev. (n)	146.414			118.78	189.019
	R(calc.)	409.96			332.580	529.25
	R(EN14372:04)	184.91			180.26	181.74
Comp.	R(Horwitz)	86.29			84.44	85.03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700
600
500
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300
200
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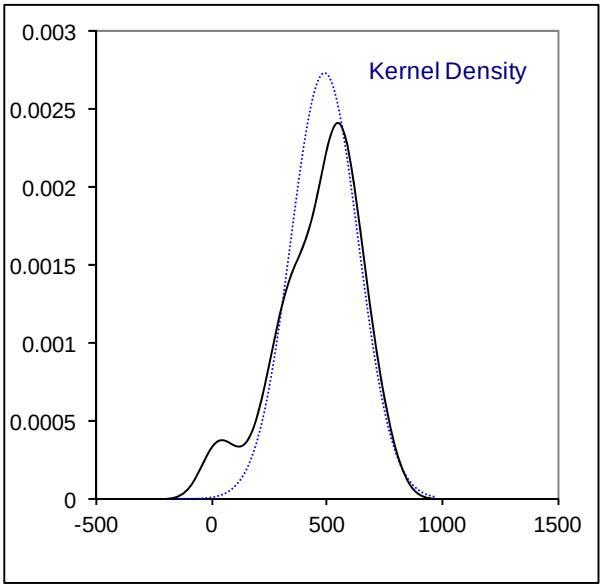
0
2746 2510 362 2488 3220 2366 3210 2375 2558 2615 3163 623 2482 551 3118 2132 330 2212 2190 339 2549 2213 2438 2247 110 2390 840 2494 2386 3172 2560 2495 3146 3243 3209 2521 2379 2350 2241 2475 3233 2358 2376 2115 2236 3153 3182 2735 3225 3218 3185 2665 2129


Kernel Density
0.003
0.0025
0.002
0.0015
0.001
0.0005
0
-500
0
500
1000
1500

APPENDIX 2

Method information as reported by the participating laboratories

Lab	sample grinded or cut	final particle size	extraction technique used	extraction solvent used	analysis technique
110	Used as received	5mm x 5mm	Ultrasonic	DCM	
330	Used as received		Ultrasonic		
339	Used as received		Ultrasonic	DCM	
362	Used as received		Migration	destilled water	
551	Used as received		Ultrasonic	DCM	
623	Cut (?)	5mm x 5mm(!)	Ultrasonic	DCM	
840	Cut		Ultrasonic	DCM/ACETONE	
2115	Used as received	0.5 cm	Ultrasonic	DCM/Methanol	
2129	Used as received		Ultrasonic	toluene, 60 °C / 30 min	
2132	Cut	< 2 mm	Ultrasonic	DCM	
2190	Used as received		static, only heating	Acetonitrile	
2212	Cut	2mm	Ultrasonic	DCM	
2213	yes		---	DCM/Methanol	
2216	---		---		
2236	Used as received	3 mm x 3 mm	Ultrasonic	Chloroform:Methanol 2:1	
2241	Cut	1mm*1mm	Ultrasonic	DCM	
2247	Used as received	3mm X 3mm	Ultrasonic	Methanol + Chloroform	LC/MS-MS
2293	---		---		
2295	---		---		
2350	Used as received		Ultrasonic	DCM / Acetonitrile	
2358	Grinded	Powder	Ultrasonic	DCM	
2366	Cut	2*2mm	Ultrasonic	DCM and acetone	
2375	Used as received		Ultrasonic		
2376	Grinded	Powder	Ultrasonic	DCM	
2379	Used as received		Ultrasonic	DCM	
2386	Grinded	< 0,5 mm	Ultrasonic	DCM	LC/MS-MS
2390	Used as received	L 5-7 mm, D 3-4 mm	Ultrasonic	DCM	
2438	Cut	2 x 2 mm	Soxhlet	Chloroform:Methanol 2:1	
2475	Used as received		Ultrasonic	Chloroform:Methanol 2:1	
2482	Cut	< 1 mm	Ultrasonic	DCM	GC/MSD
2488	Cut (?)	5mm x 5mm(!)	Ultrasonic	THF	
2494	Cut	2 mm	Ultrasonic	THF	
2495	Grinded	1mm - 2mm	Ultrasonic	Chloroform:Methanol 2:1	
2510	Cut	2mm x 2mm x 1mm	Migration	water (24 hrs, 40°C)	
2521	Used as received		Thermal Desorption		
2549	Cut	3mm X 3mm	Ultrasonic	DCM	
2558	Used as received		Soxhlet	Acetone/Hexane 50/50	GC/MS
2560	Cut	1mm*1mm	Ultrasonic	THF / Methanol	
2615	Grinded	1mm*1mm	Ultrasonic	DCM	
2665	Used as received		Total dissolution	xylene	
2735	Cut	2mm	Ultrasonic	Chloroform:Methanol 2:1	
2746	Used as received		Migration	3% acetic acid in water	
3118	Cut	<2mm	Ultrasonic	Chloroform:Methanol 2:1	
3146	Used as received		Ultrasonic	Chloroform:Methanol 2:1	
3153	Used as received	2mm x 3mm	Ultrasonic	Chloroform:Methanol 2:1	
3163	Cut	0.3mg	Thermal Desorption		TD-GC/MS
3172	Cut	1 mm	Ultrasonic	THF	
3182	Used as received	2-3 mm	Ultrasonic	Chloroform+methanol	
3185	Used as received	2mm*2mm	Ultrasonic	Chloroform:Methanol 2:1	
3209	---		---		
3210	Used as received		Ultrasonic	THF / Acetonitrile	LC/MS-MS
3218	Used as received		Ultrasonic	Chloroform:Methanol 2:1	
3220	Used as received		Ultrasonic	Methanol:THF 10:1	
3225	Used as received	5mm x 5mm	Ultrasonic	THF	
3233	Used as received		Ultrasonic	THF / Acetonitrile	
3243	Cut		Ultrasonic	DCM	GC/MS

APPENDIX 3

Number of participating laboratories per country

1 lab in	BANGLADESH
2 labs in	BRAZIL
1 lab in	BULGARIA
6 labs in	FRANCE
7 labs in	GERMANY
1 lab in	GUATEMALA
6 labs in	HONG KONG
4 labs in	INDIA
3 labs in	INDONESIA
1 lab in	IRELAND
1 lab in	ISRAEL
3 labs in	ITALY
1 lab in	KOREA
7 labs in	P.R. of CHINA
1 lab in	PAKISTAN
2 labs in	THAILAND
1 lab in	THE NETHERLANDS
4 labs in	TURKEY
3 labs in	U.S.A.
1 lab in	VIETNAM

APPENDIX 4

Abbreviations:

C	= final result after checking of first reported suspect 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
n.a.	= not applicable
n.d.	= not detected
fr	= first reported result

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, April 2014
2 ASTM E178-02
3 ASTM E1301-03
4 ISO 5725:1986
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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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18 https://chemicalwatch.com/44942/bpa-poised-for-classification-as-category-1-reprotoxin
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