Results of Proficiency Test
Migration of BPA: EN71-10/11
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Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Toy safety is the practice of ensuring that toys, especially those made for children, are safe, usually through the application of set safety standards. In many countries, toys must be able to pass safety tests in order to be sold. Many regions model their safety standards on the EU's EN71 standard. In Europe, toys must meet the criteria set by the 2009 EC Toy Safety Directive (Council Directive 2009/48/EC).

Migration of BPA is described in EN 71-9 (Requirements), EN 71-10 (Sample Preparation and extraction) and EN 71-11 (Methods of Analysis). The maximum specific limit, as described in EN 71-9 is 0.1 mg/L aqueous substrate (or simulant). Recently, the European Union has further restricted this limit, when it comes to toys. EU directive 2017/898 of 24 May 2017 amending Appendix C to Annex II to Directive 2009/48/EC as regards bisphenol A describes a maximum specific migration limit of 0.04 mg/L aqueous substrate (or simulant). This should be implemented from November 26, 2018 in its member states.

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, the Institute for Interlaboratory Studies has started a proficiency test (laboratory-evaluating interlaboratory study) for migration of Bisphenol A (EN71-10/11) during the annual proficiency testing program 2017/2018.
In this interlaboratory study 45 laboratories in 15 different countries registered for participation. See appendix 3 for the number of participants per country. In this report, the results of the 2017 proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory.
It was decided to send two different samples. The first sample was a Polypropylene (PP) plate of approx. 10 by 10 cm, labelled #17647 and the second sample was a piece of thermal printing paper of approx. 18 by 5.5 cm, wrapped in aluminium foil and labelled #17648. 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 March 2017 (iis-protocol, version 3.4). 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 batch of Polypropylene (PP) plates, artificially fortified to be positive on Bisphenol A and one batch of thermal printing paper were selected. The thermal printing paper was wrapped in Aluminium foil to avoid influence of light and heat.

The homogeneity of the subsamples of #17647 was checked by determination of total BPA content on 7 stratified randomly selected subsamples. The homogeneity of the subsamples of #17648 was checked by determination of total BPA content on 8 stratified randomly selected subsamples.

	BPA (total) in mg/kg #17647	BPA (total) in mg/kg #17648
Sample 1	2.38	15040
Sample 2	2.51	15635
Sample 3	2.47	15030
Sample 4	2.31	15770
Sample 5	2.59	16050
Sample 6	2.36	15775
Sample 7	2.41	15700
Sample 8	--	15330

Table 1: homogeneity test results of the subsamples #17647 and #17648

From the above test results the repeatabilities were calculated and compared with the repeatability of a target test method in agreement with the procedure of ISO 13528, Annex B2 in the next table.

	BPA (total) in mg/kg #17647	BPA (total) in mg/kg #17648
r (observed)	0.27	1035
reference method	EN14372:04	EN14372:04
r (ref. test method)	0.31	1958

Table 2: evaluation of repeatabilities of total BPA contents of the subsamples #17647 and #17648

For both samples #17647 and #17648, the observed repeatability is smaller than the repeatability of the target test method. Therefore, the homogeneity of subsamples #17647 and #17648 was assumed.
To each of the participating laboratories, one sample, labelled #17647 and one sample, labelled #17648, were sent on November 15, 2017.

2.5 ANALYSES

The participants were requested to determine and report the Bisphenol A by migration on both samples #17647 and #17648 applying the analysis procedure that is routinely used in the laboratory. Also some analytical details were requested to be reported.

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

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

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment.
Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no 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.

3.1 STATISTICS

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

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded 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.

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

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. 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 on the X-axis.
The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the

selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

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

3.3 Z-SCORES

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

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

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

The z-scores were calculated in according to:

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

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

Absolute values for z<2 are very common and absolute values for z>3 are very rare. 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 interlaboratory study, some problems were encountered. Three participants decided not to report any test results and none of the reporting participants reported test results after the final reporting date. Finally, the 42 reporting laboratories reported 66 numerical results for both determinations. In the reported test results 4 statistical outliers were observed, which is 5.7%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section, the results are discussed per sample and per component.
The test methods used by the various laboratories are in the table together with the original data.
The abbreviation used in these tables are either explained in the table or listed in appendix 3.

Method EN 71-11 does mention precision data, but only at a low level of 0.03 mg BPA/L aqueous migrate. Therefore, the calculated reproducibility was compared against the reproducibility estimated from the Horwitz equation.

Sample #17647

BPA: Although this sample is positive for BPA, the migration of BPA in water was very low. All participants, except one, reported a test result of <0.1 mg/L. Therefore, no significant conclusions were drawn.

Sample #17648

BPA: The determination of Bisphenol A in this thermal printing paper sample was problematic. A binormal distribution was found on the reported test results (see page 12). When this was investigated by requesting additional information from the participants, it became clear that some participants treated the paper as a one-sided surface, while others treated it as two-sided (see also paragraph 5 Discussion). Therefore, it was decided to evaluate all reported test results both as ‘one-sided surface’ and as ‘two-sided surface’. When adjusted for one-sided or two-sided surface, the determination of BPA was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the suspect data is in good agreement with the estimated reproducibility using the Horwitz equation.

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 the Horwitz equaition in the next tables:

Parameter	Unit	n	Average	2.8 * sd	R (target)
Bisphenol A	mg/kg	23	(0.04)	0.10	(0.05)

Table 3: overview of results for sample #17647

Parameter	Unit	n	Average	2.8 * sd	R (target)
Bisphenol A (1-sided surface)	mg/kg	23	7.23	1.68	2.40
Bisphenol A (2-sided surface)	mg/kg	23	3.61	0.84	1.33

Table 4: overview of results for sample #17648

Without further statistical calculations, it could be concluded that for migration of BPA there is a good compliance of the group of participating laboratories with the relevant standard. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON INTERLABORATORY RESULT

The variation observed in this proficiency test is smaller than the estimated requirements based on the Horwitz equation.

Parameter	February 2017	Est. Horwitz
BPA	8.3%	12-13%

Table 5: Comparison of uncertainties in iis proficiency tests st

4.4 EVALUATION OF THE ANALYTICAL DETAILS

In this PT also some analytical details were asked (see appendix 2) to use for further statistical analyses (see appendix 2). The majority (73%) of the participants is ISO/IEC 17025 accredited for the determination of BPA in toys.
The test methods EN71-10 and EN71-11 describe the extraction and analysis of Organic Chemical Compounds, including the determination of migration BPA, when 10 cm^2 of a toy or toy material gets into contact with 100 ml water (simulating saliva of a child) for 1 hour at 20°C.

Almost all participants used EN 71-11 as test method, only four used an in-house method. Five participants used a different surface area than 10 cm^2 and/or a different simulant volume than 100 mL. Almost all participants used a temperature of 20°C, a rotation speed of 60 rpm and a migration time of 60 minutes.

Because the migration value is determined as mg/mL aqueous substrate (or simulant), the most important factors will be the surface area used and the amount of simulant volume. Not all laboratories used 10 cm^2 and 100 mL, but took larger surface area's or simulant volumes.

5 DISCUSSION

What was observed in this PT is that laboratories used a certain piece of sample that has 10 cm^2 as surface area, but regarded this as one-sided (10 cm^2 on one side, not taking into account the other side), while others regarded this as two-sided (5 cm^2 on each side, taking into account both sides). It is no surprise that laboratories using the first approach, will find double the amount of BPA in the same volume of simulant as the second approach. This is probably why a binormal distribution was found in the evaluation of the original results.

Unfortunately, the method EN 71-10 does not describe if one or both sides should be used in the calculation of the contact surface. It only states to take 10 cm^2 and 100 mL. Other migration tests on for example food contact materials, like EN1186-1 and EN13130-1 do mention single surface and double surface. In short, samples thicker than 0.5 mm are considered to release on both sides, while thinner samples are considered to release on one side. However, as EN71-10 does not mention this difference, one may feel that always the actual surface should be used (= two-sided).

Additional analytical questions were requested by e-mail during the evaluation of the results. The most important finding was that most participants used different sized pieces of the sample (see appendix 2) for testing. Since the test result is measured directly in the aqueous migrate, the release is highly dependent on the surface area that is exposed. Therefore, the choice of sample size is important. For sample #17648 (paper sample), the dimensions used by the participants varied in length from 2 to 10 cm and in width from 1 to 5 cm. With this information from the participant, iis could calculate which surface was used to expose 10 cm^2: one-sided or two-sided. Based on this, it was decided to do two statistical evaluations: one as if the sample was tested one-sided and one as if the sample was tested two-sided. Only the test results from participants, for which the dimensions of the tested sample were known, were used for calculating the consensus value. The results of these participants were converted from one-sided to two-sided or vice versa, when needed, but only z-scores were calculated for the original reported results. The test results of participants that did not reply with the dimensions of the sample were excluded from the statistical evaluation and the determination of the assigned value. Some laboratories used a deviating contact surface/volume simulant ratio, the resulting test results were also excluded from the determination of the assigned value. For all excluded test results, z-scores were calculated.

The amount of BPA in sample #17647 was low, however, it is close to the new maximum limit, which will be implemented on November 26th according to EU directive 2017/898. When taking this new limit, seven participants reported numerical results higher than 0.04 mg/L simulant and would have found the sample positive for BPA. Another seven participants reported a “smaller than” result (like <0.1 mg/L), which are inconclusive about the sample being positive or negative when compared to the new limit. When looking at sample #17648, all but two laboratories would find the sample positive for BPA when compared to the new limit.

6 CONCLUSION

In this PT, it was found that the dimension of the piece of sample that was used for testing is important. In the next PT, these sample dimensions will be requested.

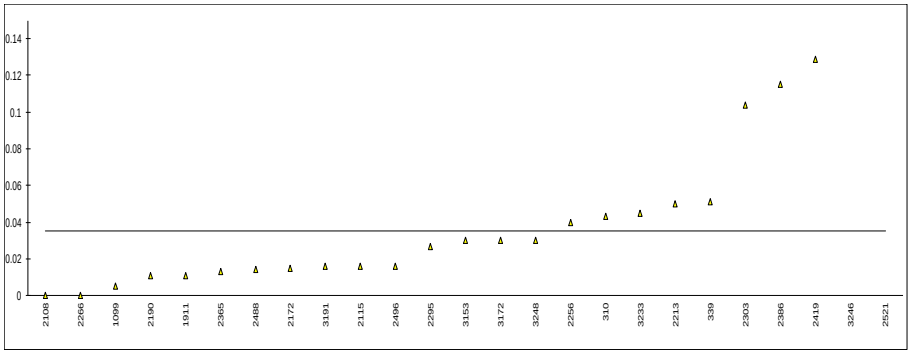
Although it can be concluded that the group of participants have no problems with the determination of BPA in these samples, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.
Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

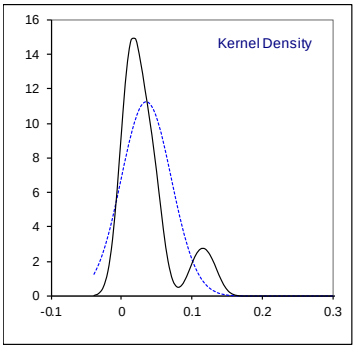
APPENDIX 1

Determination of Migration of BPA on sample #17647; results in mg/L

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
310	In house	0.043		-----	
339	EN71-11	0.051		-----	
551	EN71-11	N.D.		-----	
840	EN71-11	nd		-----	
1099	EN71-11	0.0051		-----	
1911	In house	0.01107		-----	
2108	EN71-11	0		-----	
2115	EN71-11	0.016		-----	
2132	EN71-11	<0.03		-----	
2172	EN71-11	0.0149		-----	
2182	EN71-11	<0.02		-----	
2190	EN71-11	0.0106		-----	
2213	EN71-11	0.05		-----	
2241	EN71-11	<0.02		-----	
2256	EN71-11	0.04		-----	
2266	In house	0		-----	
2295	EN71-11	0.027		-----	
2303	EN71-11	0.104		-----	
2363	EN71-11	<0.1		-----	
2365	EN71-11	0.0132		-----	
2366	EN71-11	<0.1		-----	
2376	EN71-11	<0.05		-----	
2386	EN71-11	0.1154		-----	
2419	EN71-11	0.129		-----	
2488	EN71-11	0.014	C	-----	first reported: 13.74
2492		-----		-----	
2495		-----		-----	
2496	EN71-11	0.0161		-----	
2521	In house	1.6	R(0.01)	-----	
2549	EN71-11	0.04	C	-----	first reported: not detected
2560	EN71-11	<0.03		-----	
2741	EN71-11	< 0.01		-----	
3146	EN71-11	< 0,1		-----	
3153	EN71-11	0.030		-----	
3172	EN71-11	0.03		-----	
3185	EN71-11	<0.05		-----	
3190		-----		-----	
3191	EN71-11	0.0159		-----	
3209	EN71-11	<0.01		-----	
3233	EN71-11	0.0447		-----	
3238	EN71-11	<0.01		-----	
3243	EN71-11	<0,02		-----	
3246	EN71-11	0.7656	C,R(0.01)	-----	first reported: 1.5312
3248	EN71-11	0.03		-----	

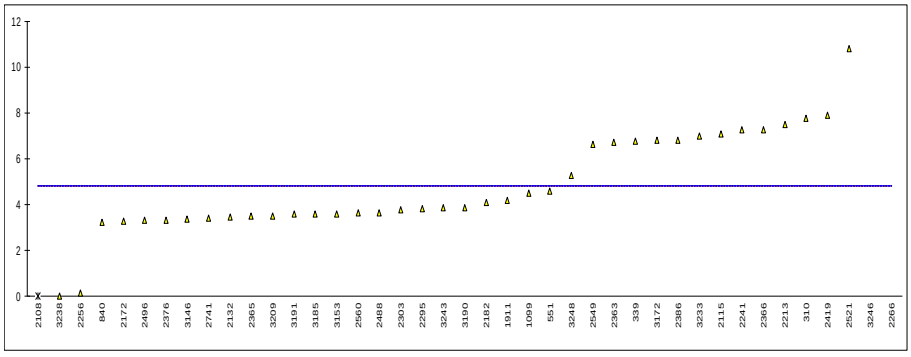
normality	not OK
n	23
outliers	2
mean (n)	(0.0353)
st.dev. (n)	0.03553
R(calc.)	0.0995
st.dev.(Horwitz)	(0.01705)
R(Horwitz)	(0.0477)

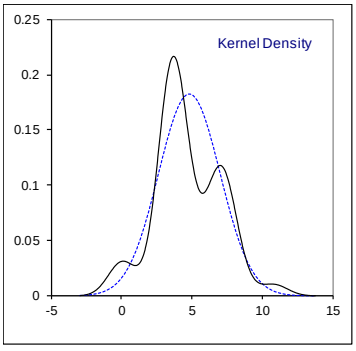




Determination of Migration of BPA on sample #17648; all reported results in mg/L

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
310	In house	7.771		-----	
339	EN71-11	6.76		-----	
551	EN71-11	4.594		-----	
840	EN71-11	3.22		-----	
1099	EN71-11	4.486		-----	
1911	In house	4.2022		-----	
2108	EN71-11	0	ex	-----	excluded for zero is not a real value
2115	EN71-11	7.09		-----	
2132	EN71-11	3.45		-----	
2172	EN71-11	3.295		-----	
2182	EN71-11	4.101		-----	
2190		-----		-----	
2213	EN71-11	7.5		-----	
2241	EN71-11	7.24		-----	
2256	EN71-11	0.13	C	-----	first reported 0.09
2266	In house	1642.40	R(0.01)	-----	
2295	EN71-11	3.8		-----	
2303	EN71-11	3.786		-----	
2363	EN71-11	6.7	C	-----	first reported 3.34
2365	EN71-11	3.52		-----	
2366	EN71-11	7.26	C	-----	first reported: 3.633
2376	EN71-11	3.326		-----	
2386	EN71-11	6.8063		-----	
2419	EN71-11	7.897		-----	
2488	EN71-11	3.651	C	-----	first reported 3651.46
2492		-----		-----	
2495		-----		-----	
2496	EN71-11	3.32		-----	
2521	In house	10.8		-----	
2549	EN71-11	6.65	C	-----	first reported 0.01
2560	EN71-11	3.64		-----	
2741	EN71-11	3.400		-----	
3146	EN71-11	3.344		-----	
3153	EN71-11	3.603		-----	
3172	EN71-11	6.80		-----	
3185	EN71-11	3.59		-----	
3190	EN71-11	3.87		-----	
3191	EN71-11	3.57		-----	
3209	EN71-11	3.52		-----	
3233	EN71-11	6.9692		-----	
3238	EN71-11	0.016		-----	
3243	EN71-11	3.85		-----	
3246	EN71-11	379.1	C,R(0.01)	-----	First reported 758.231
3248	EN71-11	5.25		-----	

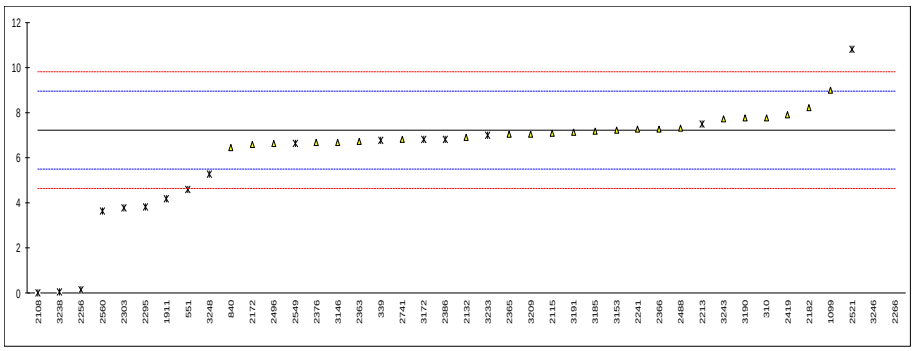


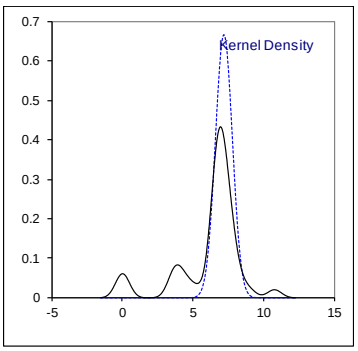


Determination of Migration of BPA on sample #17648; (1-sided surface) results in mg/L

lab	method	Value	mark	z(targ)	remarks
110		-----		-----	
310	In house	7.771		0.64	
339	EN71-11	6.76	ex1	-0.54	
551	EN71-11	4.594	ex1	-3.07	
840	EN71-11	6.44	C	-----	reported 2-sided; converted by iis to 1-sided
1099	EN71-11	8.972	C	-----	reported 2-sided; converted by iis to 1-sided
1911	In house	4.2022	R(0.01)	-3.52	
2108	EN71-11	0	ex3	-8.42	
2115	EN71-11	7.09		-0.16	
2132	EN71-11	6.90	C	-----	reported 2-sided; converted by iis to 1-sided
2172	EN71-11	6.590	C	-----	reported 2-sided; converted by iis to 1-sided
2182	EN71-11	8.202	C	-----	reported 2-sided; converted by iis to 1-sided
2190		-----		-----	
2213	EN71-11	7.5	ex1	0.32	
2241	EN71-11	7.24		0.02	
2256	EN71-11	0.13	ex1	-8.27	
2266	In house	1642.40	ex2	1904.76	
2295	EN71-11	3.8	ex2	-3.99	
2303	EN71-11	3.786	ex1	-4.01	
2363	EN71-11	6.7		-0.61	
2365	EN71-11	7.04	C	-----	reported 2-sided; converted by iis to 1-sided
2366	EN71-11	7.26		0.04	
2376	EN71-11	6.652	C	-----	reported 2-sided; converted by iis to 1-sided
2386	EN71-11	6.8063	ex1	-0.49	
2419	EN71-11	7.897		0.78	
2488	EN71-11	7.302	C	-----	reported 2-sided; converted by iis to 1-sided
2492		-----		-----	
2495		-----		-----	
2496	EN71-11	6.64	C	-----	reported 2-sided; converted by iis to 1-sided
2521	In house	10.8	ex2	4.16	
2549	EN71-11	6.65	ex2	-0.67	
2560	EN71-11	3.64	ex1	-4.18	
2741	EN71-11	6.800	C	-----	reported 2-sided; converted by iis to 1-sided
3146	EN71-11	6.688	C	-----	reported 2-sided; converted by iis to 1-sided
3153	EN71-11	7.206	C	-----	reported 2-sided; converted by iis to 1-sided
3172	EN71-11	6.80	ex1	-0.50	
3185	EN71-11	7.18	C	-----	reported 2-sided; converted by iis to 1-sided
3190	EN71-11	7.74	C	-----	reported 2-sided; converted by iis to 1-sided
3191	EN71-11	7.14	C	-----	reported 2-sided; converted by iis to 1-sided
3209	EN71-11	7.04	C	-----	reported 2-sided; converted by iis to 1-sided
3233	EN71-11	6.9692	ex1	-0.30	
3238	EN71-11	0.032	C,R(0.01)	-----	reported 2-sided; converted by iis to 1-sided
3243	EN71-11	7.70	C	-----	reported 2-sided; converted by iis to 1-sided
3246	EN71-11	379.1	ex2	433.18	
3248	EN71-11	5.25	ex1	-2.30	

normality	not OK	ex1 = unknown of 1-sided or 2-sided contact surface was used
n	23	ex2 = used deviating ratio contact surface : volume
outliers	2 (+16ex)	ex3 = zero is no real test result
mean (n)	7.226	
st.dev. (n)	0.5989	
R(calc.)	1.677	
st.dev.(Horwitz)	0.8585	
R(Horwitz)	2.404	Compare R(EN71-11) = 0.910

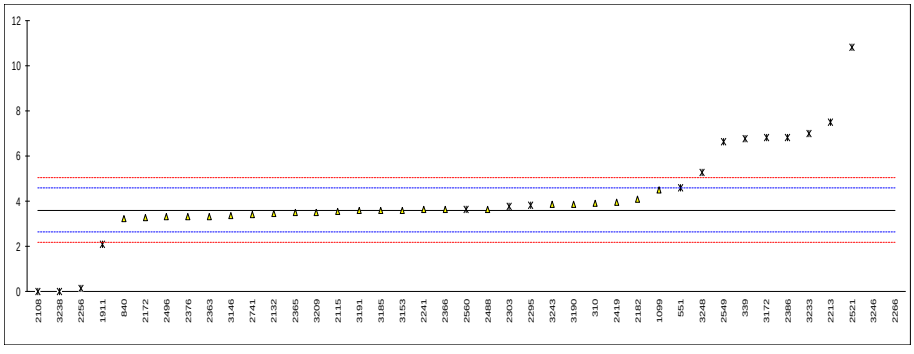


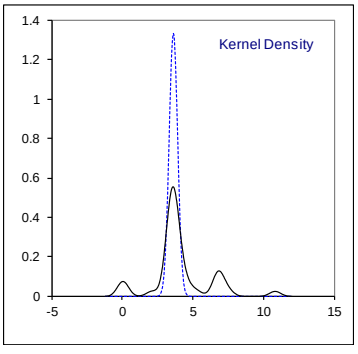


Determination of Migration of BPA on sample #17648; (2-sided surface) results in mg/L

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
310	In house	3.886	C	-----	reported 1-sided; converted by iis to 2-sided
339	EN71-11	6.76	ex1	6.61	
551	EN71-11	4.594	ex1	2.06	
840	EN71-11	3.22		-0.82	
1099	EN71-11	4.486		1.83	
1911	In house	2.1011	C,R(0.01)	-----	reported 1-sided; converted by iis to 2-sided
2108	EN71-11	0	ex3	-7.58	
2115	EN71-11	3.55	C	-----	reported 1-sided; converted by iis to 2-sided
2132	EN71-11	3.45		-0.34	
2172	EN71-11	3.295		-0.67	
2182	EN71-11	4.101		1.03	
2190		-----		-----	
2213	EN71-11	7.5	ex1	8.16	
2241	EN71-11	3.62	C	-----	reported 1-sided; converted by iis to 2-sided
2256	EN71-11	0.13	ex1	-7.31	
2266	In house	1642.40	ex2	3440.05	
2295	EN71-11	3.8	ex2	0.39	
2303	EN71-11	3.786	ex1	0.36	
2363	EN71-11	3.34	C	-----	reported 1-sided; converted by iis to 2-sided
2365	EN71-11	3.52		-0.19	
2366	EN71-11	3.63	C	-----	reported 1-sided; converted by iis to 2-sided
2376	EN71-11	3.326		-0.60	
2386	EN71-11	6.8063	ex1	6.70	
2419	EN71-11	3.949	C	-----	reported 1-sided; converted by iis to 2-sided
2488	EN71-11	3.651		0.08	
2492		-----		-----	
2495		-----		-----	
2496	EN71-11	3.32		-0.61	
2521	In house	10.8	ex2	15.09	
2549	EN71-11	6.65	ex2	6.38	
2560	EN71-11	3.64	ex1	0.06	
2741	EN71-11	3.400		-0.45	
3146	EN71-11	3.344		-0.56	
3153	EN71-11	3.603		-0.02	
3172	EN71-11	6.80	ex1	6.69	
3185	EN71-11	3.59		-0.05	
3190	EN71-11	3.87		0.54	
3191	EN71-11	3.57		-0.09	
3209	EN71-11	3.52		-0.19	
3233	EN71-11	6.9692	ex1	7.05	
3238	EN71-11	0.016	R(0.01)	-7.55	
3243	EN71-11	3.85		0.50	
3246	EN71-11	379.1	ex2	788.20	
3248	EN71-11	5.25	ex1	3.44	

normality	not OK	ex1 = unknown of 1-sided or 2-sided contact surface was used
n	23	ex2 = used deviating ratio contact surface : volume
outliers	2 (+16ex)	ex3 = zero is no real test result
mean (n)	3.612	
st.dev. (n)	0.2999	
R(calc.)	0.840	
st.dev.(Horwitz)	0.4764	
R(Horwitz)	1.334	Compare R(EN71-11) = 0.455





APPENDIX 2 Details reported for #17648 (thermal paper sample)

labnrs	ISO/IEC 17025 accredited?	Surf. area reported (cm^2)	Volume simulant (mL)	Temp. simulant (°C)	Rotation speed (rpm)	Time (min.)	Sample #17648			
							Length (cm)	Width (cm)	One-sided surface (cm^2)	Two-sided surface (cm^2)
110	---	-----	-----	-----	-----	-----	-----	-----	-----	-----
310	No	10.0	100	20.0	60	60	10	1	10	20
339	No	10	100	20	-----	60	-----	-----	-----	-----
551	Yes	10	100	20	60	60	-----	-----	-----	-----
840	No	10	100	22	60	60	5	1	5	10
1099	Yes	10.0	100	20.0	80	60	2.5	2	5	10
1911	No	10.23	100	20.5	57	60	4.992	2.049	10.23	20.46
2108	Yes	10	100	20	60	60	5	2	10	20
2115	No	10	100	20	60	60	5	2	10	20
2132	No	9.89	100	21	60	60	2.5	1.98	4.95	9.9
2172	---	10	100	20	60	60	5	1	5	10
2182	Yes	10	100	20	60	-----	2.5	2	5	10
2190	No	-----	-----	-----	-----	-----	-----	-----	-----	-----
2213	Yes	-----	-----	-----	-----	-----	-----	-----	-----	-----
2241	Yes	10	100	20	60	60	5	2	10	20
2256	Yes	10	100	22	60	60	-----	-----	-----	-----
2266	Yes	***100***	100	20	-----	60	-----	-----	-----	-----
2295	Yes	***4***	100	22	60	60	2	2	4	8
2303	No	10	100	20	60	60	-----	-----	-----	-----
2363	Yes	10	100	20	60	60	2	5	10	20
2365	Yes	10	100	21	60	60	5	1	5	10
2366	Yes	***20***	***200***	20	60	60	5	2	10	20
2376	Yes	10	100	21	60	60	2.5	2	5	10
2386	Yes	***104***	***1000***	20	60	60	-----	-----	-----	-----
2419	Yes	10	100	20	44	60	5	2	10	20
2488	Yes	10	100	20	60	60	2.5	2	5	10
2492	---	-----	-----	-----	-----	-----	-----	-----	-----	-----
2495	---	-----	-----	-----	-----	-----	-----	-----	-----	-----
2496	No	10	100	20.0	60	60	5	2	10	20
2521	Yes	-----	-----	-----	-----	-----	cut in small particles		-----	-----
2549	Yes	***215***	100	22	60	60	18.5	5.8	107.3	214.6
2560	Yes	***30.5046***	100	21	60	60	-----	-----	-----	-----
2741	Yes	9.992	100	20	60	60	2.497	2.001	5.00	9.99
3146	Yes	9.65	100	20	60	60	-----	-----	4.825	9.65
3153	Yes	10	100	20	60	60	2.5	2	5	10
3172	Yes	-----	-----	-----	-----	-----	-----	-----	-----	-----
3185	Yes	10	100	20	60	60	2.5	2	5	10
3190	Yes	-----	-----	-----	-----	-----	5	1	5	10
3191	Yes	10	100	20	60	60	5	1	5	10
3209	Yes	10	100	20.2	200.5	60	3.3	1.5	4.95	9.9
3233	No	10	100	20	60	60	-----	-----	5	10
3238	No	10	100	20	60	60	5	2	10	20
3243	Yes	10	100	***23***	125	60	5	1	5	10
3246	Yes	***200***	100	20	60	60	18	6	108	216
3248	Yes	10	100	***25***	-----	-----	-----	-----	-----	-----

APPENDIX 3

Number of participating laboratories per country

1 lab in	BANGLADESH
1 lab in	BRAZIL
1 lab in	BULGARIA
5 labs in	FRANCE
4 labs in	GERMANY
6 labs in	HONG KONG
2 labs in	INDIA
3 labs in	ITALY
11 labs in	P.R. of CHINA
2 labs in	POLAND
1 lab in	THE NETHERLANDS
3 labs in	TURKEY
1 lab in	U.S.A.
1 lab in	UNITED KINGDOM
3 labs in	VIETNAM

APPENDIX 4

Abbreviations:

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

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, March 2017
2 ASTM E178:02
3 ASTM E1301:03
4 ISO 5725:86
5 ISO 5725, parts 1-6, 1994
6 Directive 2014/81/EU amending Appendix C of Annex II to Directive 2009/48/EC of the European Parliament and of the Council on the safety of toys, as regards bisphenol A
7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
9 IP 367:84
10 DIN 38402 T41/42
11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
12 J.N. Miller, Analyst, 118, 455, (1993)
13 ASTM F963:"standard consumer safety specification on toy safety"
14 Analytical Methods Committee Technical brief, No4 January 2001.
15 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry 2002, Analyst 2002, 127 pages 1359-1364
16 ISO 13528:15, Statistical methods for use in proficiency testing by interlaboratory comparison
17 R.G. Visser, Reliability of proficiency test results for metals and phthalates in plastics, Accred Qual Assur, 14:29-34 (2009)
18 https://chemicalwatch.com/44942/bpa-poised-for-classification-as-category-1-reprotoxin
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20 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, T*echnometrics*, 25(2), pp. 165-172, (1983)