Results of Proficiency Test
Migration of BPA: EN71-10/11
December 2018

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. C.M. Nijssen-Wester
Correctors: ing. A.S. Noordman-de Neef & ing. R.J. Starink

Report: iis18V04

March 2019

CONTENTS

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 has been implemented from November 26, 2018 in its member states.
In 2017, 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 2018/2019, it was decided to continue this proficiency test.
In this interlaboratory study, 17 laboratories in 9 different countries registered for participation. See appendix 3 for the number of participants per country. In this report, the results of the 2018 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/IEC 17025 accredited laboratory.
It was decided to send two different samples. A piece of thermal printing paper of 3 grams labelled #18655 and a subsample consisted of two strips of Polyethylene (PE) labelled #18656. 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/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 organization 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 June 2018 (iis-protocol, version 3.5). This protocol is electronically available 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 PT samples were prepared; one sample of thermal printing paper of at least 10 by 10 cm and labelled #18655 and one sample of Polyethylene (PE) strips, approx. 12.5 by 1 cm each and labelled #18656. The PE strips are artificially fortified to be positive on Bisphenol A. The thermal printing paper was wrapped in Aluminum foil to avoid influence of light.

The homogeneity of the subsamples of #18655 was checked by determination of total BPA content by an in-house method on 10 stratified randomly selected subsamples. The homogeneity of the subsamples of #18656 was checked by determination of BPA according to test methods EN71-10/11 on 12 stratified randomly selected subsamples.

	BPA (total) in mg/kg #18655	BPA (migration) in mg/L #18656
Sample 1	6665.0	0.3332
Sample 2	6752.0	0.3251
Sample 3	7105.0	0.3407
Sample 4	6671.0	0.3062
Sample 5	6482.0	0.3514
Sample 6	7286.5	0.3144
Sample 7	7093.5	0.3290
Sample 8	6665.0	0.3133
Sample 9	7123.5	0.3169
Sample 10	6550.0	0.3667
Sample 11	--	0.3405
Sample 12	--	0.3470

Table 1: homogeneity test results of the subsamples #18655 and #18656

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

	BPA (total) in mg/kg #18655	BPA (migration) in mg/L #18656
r (observed)	794	0.050
reference method	EN14372:04	Horwitz
0.3 x R (reference method)	776	0.053

Table 2: evaluation of repeatabilities of total BPA contents of the subsamples #18655 and #18656

The calculated repeatabilities were in agreement with 0.3 times the reproducibility of the target test methods. Therefore, the homogeneity of subsamples #18655 and #18656 was assumed.

To each of the participating laboratories, one sample labelled #18655 containing thermal paper and one sample labelled #18656 containing two PE strips were sent on November 14, 2018.

2.5 ANALYSES

The participants were requested to determine and report the Bisphenol A by migration on both samples #18655 and #18656 applying the analysis procedure that is routinely used in the laboratory. The fixed sample conditions for this PT were: Simulant is Deionized Water, Exposure Temperature is 20°C, Exposure Time is 1 hour and Rotation Speed is 60 rounds per minute. Also accreditation status for the test and some analytical details were asked. It was advised to keep the thermal print paper (#18655) stored dark, dry and cool and packed until the start of the test. Also to not touch it with bare hands.

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 appropriate 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 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.
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. 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 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 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. 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 PT) / target standard deviation

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

Absolute values for z<2 are very common and absolute values for z>3 are very rare.
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 major problems were encountered with the dispatch. One participant decided not to report any test results and none of the reporting participants reported test results after the final reporting date. Finally, the 16 reporting laboratories reported 30 numerical test results for both determinations. In the reported test results 3 statistical outliers were observed, which is 9.1%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

All original data sets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER SAMPLE

In this section, the results are discussed per sample.
The test method, 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 table together with the original data. The abbreviation used in these tables are either explained in the table or listed in appendix 4.

Test 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.
Test method EN 71-10 does not describe whether the sample should be used one-sided or two-sided. Since the test method EN 71-10 does describe to put the sample in the bottle with simulant, so it is exposed on all sides, it is assumed the required surface area is also based on a two-sided exposure. Therefore, all test results were evaluated as a two-sided exposure. Where needed, the test results were recalculated as two-sided exposure, see for more discussion paragraph 5.

Sample #18655

BPA: This determination was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements estimated from the Horwitz equation.

Sample #18656

BPA: This determination was very problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements estimated from the Horwitz equation.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibilities as declared by the estimated target reproducibilities using the Horwitz equation and the reproducibilities found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the estimated target reproducibility are presented in the next tables:

Parameter	unit	n	average	2.8 * sd	R (target)
Bisphenol A (2-sided surface)	mg/L	15	1.78	1.69	0.73

Table 3: overview of results for sample #18655

Parameter	unit	n	average	2.8 * sd	R (target)
Bisphenol A (2-sided surface)	mg/L	12	1.04	1.14	0.46

Table 4: overview of results for sample #18656

Without further statistical calculations, it could be concluded that for migration of BPA there is not a good compliance of the group of participating laboratories with the reference method.

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2018 WITH PREVIOUS PT

The performance of the determinations of the proficiency test was compared, expressed as relative standard deviation (RSD) of the PTs, see table below.

Parameter	December 2018	December 2017	Est. Horwitz
BPA	34-39%	8.3%	12-13%

Table 5: development of uncertainties over the years

The uncertainties observed in this PT are larger than observed in the previous PT and larger than the estimated target reproducibility using the Horwitz equation.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

In this PT, also some analytical details were asked (see appendix 2). The majority (64%) of the participants is ISO/IEC 17025 accredited for this test. Furthermore, details were requested about the test conditions as described in EN71-10 and 11.
This will be further discussed in paragraph 5.

5 DISCUSSION

The test methods EN71-10 and EN71-11 describe the extraction and analysis of Organic Chemical Compounds, including the determination of migration of 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. The analytical details showed that almost all participants used 100 mL of simulant, 20°C as temperature, 60 minutes of time and a rotation speed of 60 rpm.

Further was observed 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 than the second approach.

Unfortunately, test 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 put it in 100 mL. Other migration tests on for example food contact materials, like EN1186-1 and EN13130-1 do mention single surface and double surface. These test methods describe that samples thicker than 0.5 mm are considered to release from both sides, while thinner samples are considered to release as being one side. However, as EN71-10 does not mention this difference, in this case one may conclude that the actual surface of both sides should be used (so-called two-sided approach).

It is observed that the dimensions in length and width of the samples that were used by the laboratories were all different. This would be no problem, if the end test result would be recalculated to 10 cm^2 for deviating surface areas. Allthough, test method EN71-10 does not describe what to do using a deviating surface area and recalculating.

The majority of the participants were contacted to clarify the exact surface of the sample that had been exposed to the simulant. The participant's details about the sample and e-mail answers (see appendix 2) showed that cutting a surface of 10 cm^2 from the sample can be done in many ways. Some participants cut a sample of 1 x 5 cm, used it two-sided, others used the same dimension, but one-sided. Some even used a much higher surface, like 100 cm^2, without taking this into account in the end test result.

It is remarkedly that so many participants chose a deviating surface area than described in test method EN71-10 and reported the migrated amount as if it was determined on a sample of 10 cm^2 in 100 mL. A total of seven participants corrected their test result because of this clarification and the test results from seven other participants were recalculated by iis to a surface of 10 cm^2 taking two sides into account. Not following test method EN71-10 with regards to cutting a surface area of precisely 10 cm^2 for testing may be one of the reasons why the variation of the test results was large in this PT.

6 CONCLUSION

All participants did find both samples to be positive on BPA (above the limit of EN71-9 (0.1 mg/L) as well as directive EU/2017/898 (0.04 mg/L).

In this PT, it was found that the dimension of the piece of sample that was used for testing is very relevant. In the next PT, these sample dimensions will be requested again. Next to this, it will be also mentioned in the set of conditions that the sample should be considered as two-sided.

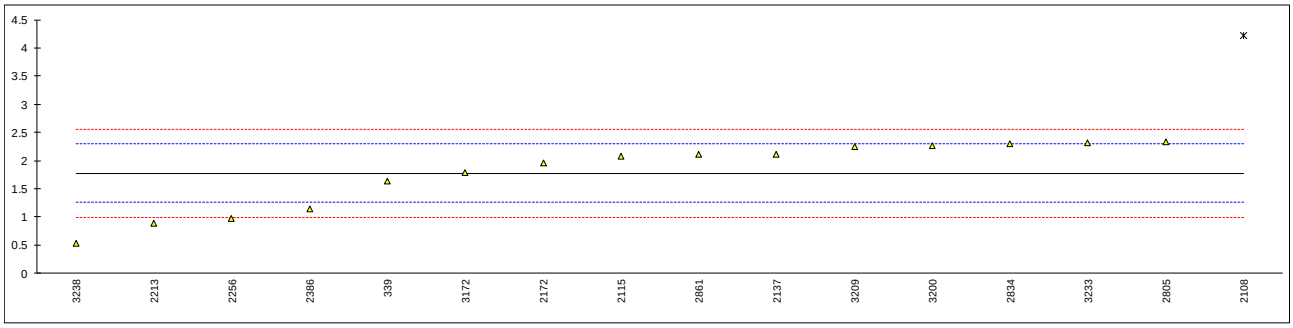
Although it can be concluded that the group of participants have problems with the determination of BPA in these samples, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

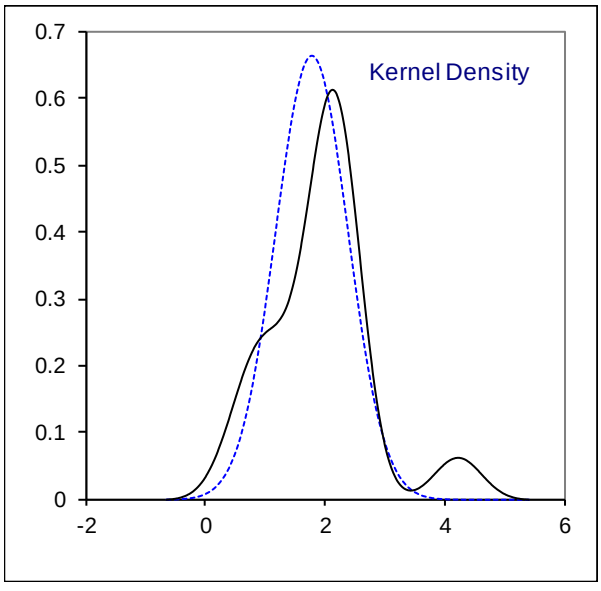
APPENDIX 1

Determination of Migration of BPA (Thermal Paper, two-sided, 10 cm^2) on sample #18655; results in mg/L

lab	method	original rep. result	corrected by lab	converted by iis*	value	mark	z(targ)	remarks
110		-----			-----		-----	
339	In house	3268.95	3.269	1.635	1.635	C	-0.55	lab correction µg/L > mg/L
2108	EN71-11	8.432	4.261		4.216	C,G(0.05)	9.35	lab correction one-sided>two-sided
2115	EN71-11	4.152		2.076	2.076	C	1.15	iis correction
2137	EN71-11	2.113			2.113		1.29	
2172	EN71-11	1.95			1.95		0.66	
2213	EN71-11	44.1499	4.41	0.88	0.88	C	-3.44	lab correction for typo error
2256	EN71-11	1.95	0.975		0.975	C	-3.08	lab correction one-sided>two-sided
2386	EN71-11	22.938	2.29	1.15	1.15	C	-2.42	lab correction from 100 -> 10 cm^2
2805	EN71-11	4.68		2.34	2.34	C	2.16	iis correction
2834	EN71-11	2.30			2.30		2.01	
2861	EN71-11	4.60636	2.30318	2.10817	2.10817	C	1.27	lab correction one-sided>two-sided
3172	EN71-11	3.57		1.79	1.79	C	0.03	iis correction
3200	EN71-11	2.26			2.26		1.85	
3209	EN71-11	2.241			2.241		1.78	
3233	EN71-11	2.31			2.31		2.04	
3238	EN71-11	0.41	0.537		0.537	C	-4.76	lab correction
	normality				OK			
	n				15			
	outliers				1			
	mean (n)				1.777			
	st.dev. (n)				0.6018			
	R(calc.)				1.685			
	st.dev.(Horwitz)				0.2607			
	R(Horwitz)				0.730			Compare R(EN71-11) = 0.224

*) iis converted test result to a total surface of 10 cm^2 (two-sided)



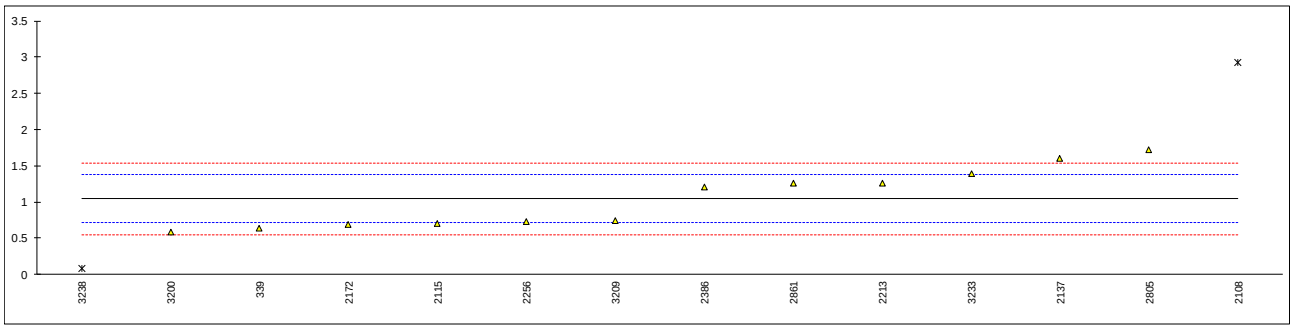


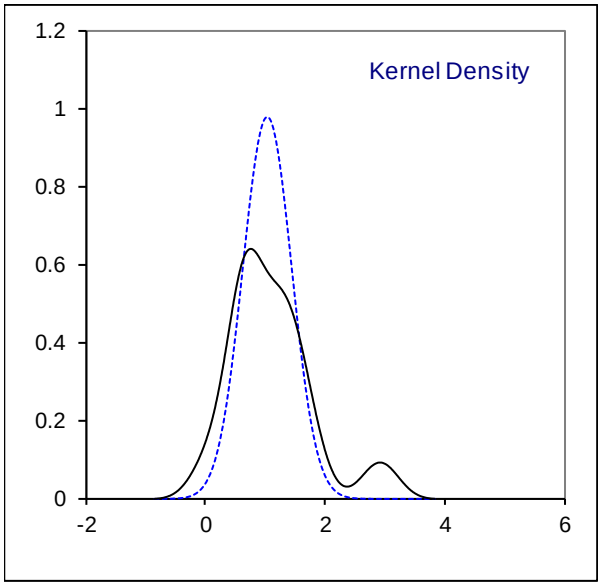
Determination of Migration of BPA (PE strips, two-sided, 10 cm^2) on sample #18656; results in mg/L

lab	method	original rep. result	corrected by lab	converted by iis*	value	mark	z(targ)	remarks
110		-----			-----		-----	
339	In house	635.43	0.635		0.635	C	-2.46	lab correction µg/L > mg/L
2108	EN71-11	5.845	2.923		2.923	C,G(0.05)	11.35	lab correction one-sided>two-sided
2115	EN71-11	1.40		0.70	0.70	C	-2.07	iis correction
2137	EN71-11	1.605			1.605		3.39	
2172	EN71-11	0.735	0.690		0.690	C	-2.13	lab correction from 23.18->10 cm^2
2213	EN71-11	1.2804	1.26		1.26	C	1.31	lab correction
2256	EN71-11	0.735			0.735		-1.85	
2386	EN71-11	7.362	1.2		1.2	C	0.95	lab correction from 62->10 cm^2
2805	EN71-11	1.72			1.72		4.09	
2834		-----			-----		-----	
2861	EN71-11	2.505724		1.252862	1.252862	C	1.27	iis correction
3172		-----			-----		-----	
3200	EN71-11	0.580			0.580		-2.79	
3209	EN71-11	0.741			0.741		-1.82	
3233	EN71-11	1.39			1.39		2.10	
3238	EN71-11	0.19	0.077		0.077	C,G(0.05)	-5.82	lab correction

normality	OK	
n	12	
outliers	2	
mean (n)	1.042	
st.dev. (n)	0.4067	
R(calc.)	1.139	
st.dev.(Horwitz)	0.1657	
R(Horwitz)	0.464	Compare R(EN71-11) = 0.131

*) iis converted one-sided test result to two-sided by multiplying with factor: 10 cm^2/20 cm^2





APPENDIX 2 Surface area calculations and analytical details

Determination of surface on sample #18655 (Thermal Paper);

lab	length (cm)	width (cm)	thickness	surface	surface (cm^2)	iis calc. two-sided*
110	-----	-----	-----	-----	-----	-----
339	1	10	-----	one-sided C	10	20
2108	5	2	-----	two-sided	10	20
2115	10	1	-----	one-sided	-----	20
2137	5	1	-----	two-sided	10	10
2172	5	1	-----	two-sided	10	10
2213	5 C	5 C	-----	two-sided	5*5 C	50
2256	5	2	-----	one-sided	10	20
2386	10	10	0.0005	one-sided	100	200
2805	5	2	-----	one-sided	10	20
2834	5.0	1.0	-----	two-sided	10	10
2861	4.939 C	1.106	0.005	two-sided	10.925068	10.925
3172	5	2	-----	two-sided	10	20
3200	2.50	2.00	<0.10cm	two-sided	10.00	10
3209	5	1	<0.1cm	two-sided	10	10
3233	5	1	-----	two-sided	10	10
3238	5 C	1 C	0.006	two-sided	10	10

*) iis calculated length x width, no thickness because the sample thickness is <0.1 cm

Lab 339 first reported: two-sided
Lab 2213 first reported for length: 10, for width: 10 and for surface: 10*10
Lab 2861 first reported for length: 4.939
Lab 3238 first reported for length: 3.2 and for width: 3.2

Determination of surface on sample #18656 (PE strips);

lab	length (cm)	width (cm)	thickness	surface	surface (cm^2)	iis calc. two-sided*	result corr.	final calc. surface	dev. from 10 cm^2
110	-----	-----	-----	-----	-----	-----	-----	-----	-----
339	1 C	5	-----	two-sided	10	11.8	-	11.8	18%
2108	10	1	1 mm	two-sided	10	23.3	yes, x 10/20	11.65	17%
2115	10	1	1-2 mm	one-sided	10.2	22.1	yes, x 10/20	11.05	11%
2137	3.8 C	1.3 C	0.1	two-sided	10	11.41	-	11.41	14%
2172	9.95 C	1	0.15	two-sided	10.65	23.18	yes, x 10/23.18	10	0%
2213	5.5	1	0.15	two-sided	1*5.5	12.95	-	12.95	30%
2256	3.6	1.2	0.155	two-sided C	10.13	10.08	-	10.08	1%
2386	1	12	0.15	two-sided	62**	55.8	yes, x 10/62	10	0%
2805	3.889	1.286	-----	two-sided	10.003	11.56	-	11.56	16%
2834	-----	-----	-----	-----	-----	-----	-----	-----	-----
2861	1.288	5.648 C	0.15	one-sided	9.355424 C	16.63	yes, x 10/20	8.32	-17%
3172	-----	-----	-----	-----	-----	-----	-----	-----	-----
3200	3.82	1.28	0.15 cm	two-sided	11.31	11.31	-	11.31	13%
3209	3.34	1.29	1.5MM	two-sided	10.01	10.01	-	10.01	0%
3233	3.6	1.3	1mm	two-sided	10	10.83	-	10.83	8%
3238	3.2 C	1.3	0.153	two-sided	9.697 C	9.67	-	9.67	-3%

*) iis calculated with a thickness of 0.15 cm
**) maximum area of 1 strip of 12.5 x 1.3 x 0.15 is 36.64, both strips were used

Lab 339 first reported for length: 2
Lab 2137 first reported for length: 5 and width: 1
Lab 2172 first reported for length: 4.5
Lab 2256 first reported: one-sided
Lab 2861 first reported for width: 7.263 and for surface: 9.354744
Lab 3238 first reported for length: 7.7 and for surface: 10

Analytical details

lab	ISO17025 accredited	sample precleaned	#18655 volume simulant	temp. simulant	rotation speed	time used	#18656 volume simulant	temp. simulant	rotation speed	time used
110	---	---	-----	-----	-----	-----	-----	-----	-----	-----
339	No	No	100	20	-----	60	100	20	-----	60
2108	Yes	No	100	22	60	60	100	22	60	60
2115	No	No	100	25	60	60	100	25	60	60
2137	Yes	No	100	20	60	60	100	20	60	60
2172	---	---	100	20	60	60	100	20	60	60
2213	Yes	No	100	20	60	60	40	20	60	60
2256	Yes	Yes, brush	100	20	60	60	100	20	60	60
2386	Yes	No	100	20	60	60	100	20	60	60
2805	No	No	100	20	60	60	100	20	60	60
2834	Yes	No	100	20	60	60	-----	-----	-----	-----
2861	Yes	No	100	20	60	60	100	20	60	60
3172	---	---	100	25	60	60	-----	-----	-----	-----
3200	Yes	No	100.00	20.0	60	60	113.0	20.0	60	60
3209	Yes	No	100	20	60	60	100	20	60	60
3233	No	No	100	20	60	60	100	20	60	60
3238	No	No	100	20	60	60	100	20	60	60

APPENDIX 3

Number of participating laboratories per country

3 labs in FRANCE

3 labs in GERMANY

1 lab in INDIA

2 labs in ITALY

1 lab in KOREA

4 labs in P.R. of CHINA

1 lab in TURKEY

1 lab in U.S.A.

1 lab in VIETNAM

APPENDIX 4

Abbreviations:

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= possibly 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, June 2018
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, No 4 January 2001.
15 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry 2002, Analyst 2002, 127, 1359-1364
16 ISO 13528:15, Statistical methods for use in proficiency testing by interlaboratory comparison
17 https://chemicalwatch.com/44942/bpa-poised-for-classification-as-category-1-reprotoxin
18 Annex XVII to REACH Regulation 1907/2006
19 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, T*echnometrics*, 25(2), 165-172, (1983)