Results of Proficiency Test
Specific migration (fcm)
September 2017

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2012, the Institute of Interlaboratory Studies (iis) organizes a proficiency test (PT) scheme for food contact materials. During the annual proficiency testing program 2017/2018, it was decided to continue the proficiency test for the determination of Specific Migration in food contact materials. It was decided to organize a PT on the specific migration of 2,2-bis(4-hydroxyphenyl)propane (Bisphenol A or BPA).
During the contact of food with materials like kitchenware, molecules can migrate from the material to the food. Because of this, in many countries regulations are made to ensure food safety. The framework Regulation (EU) No. 1935/2004 applies to all food contact materials and describes a large number of requirements, e.g. limits for overall migration and specific limits for certain constituents. Supplementary, regulation (Eu) No 10/2011 describes a specific migration limit of 0.6 mg/kg food for Bisphenol A.
The determination of specific migration requires additional analytical testing following the migration step, while the determination of the overall (also called global, or total) migration requires weighing as only quantitative analytical technique. This makes the specific migration from food contact materials more difficult than determination of the overall migration.

In this interlaboratory study 32 laboratories in 18 different countries registered for participation (see appendix 4). 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 one sample, a cup, labelled #17625, artificially fortified with Bisphenol A and to prescribe a number of test conditions (migration method, type of simulant, exposure time and temperature). Participants were also requested to report some intermediate test results and to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls in the accredited scope. 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 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 Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

A batch of 45 colourless Polycarbonate (food) cups artificially fortified with Bisphenol A was prepared by a third party and labelled #17625. The homogeneity was checked by determination of the specific migration of BPA on 8 stratified randomly selected plates.

	migration of BPA in mg/l food simulant (200 ml 95% Ethanol, 120 min at 70°C)
Sample #17625-1	0.147
Sample #17625-2	0.149
Sample #17625-3	0.158
Sample #17625-4	0.143
Sample #17625-5	0.165
Sample #17625-6	0.157
Sample #17625-7	0.147
Sample #17625-8	0.162

Table 1: homogeneity test results on the subsamples #17625

From the above test results, the repeatability was calculated and compared to 0.3 times the corresponding reproducibility of the reference method in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	migration of BPA in mg/l food simulant (200 ml 95% Ethanol, 120 min at 70°C)
r(observed)	0.02
reference method	Horwitz
0.3 x R (reference method)	0.03

Table 2: evaluation of the repeatability of subsamples #17625

The calculated repeatability was in good agreement with 0.3 times the corresponding reproducibility of the reference method, estimated from the Horwitz equation. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample #17625 was sent on September 6, 2017.

2.5 ANALYSES

The participants were requested to determine the specific migration of 2,2-bis(4-hydroxyphenyl)propane (Bisphenol A or BPA) on sample #17625 using the prescribed test conditions (article filling, repeated use, 2 hrs at 70°C and 95% Ethanol as simulant).

It was explicitly requested to treat the sample as a routine sample and to report the analytical results using the indicated units on the report form in the data entry portal and not to round the 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 cannot be used for meaningful statistical calculations.

To get comparable test results a detailed report form and a letter of instructions are prepared. 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 were 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 sample and per component in the appendix 1 of this report. The laboratories are represented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that did not report 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 reanalyses). Additional or corrected test results are used for the data analysis and the original test results are placed under 'Remarks' in the test 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.

In accordance to ISO 5725 the original test results per determination were submitted subsequently 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 significant 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 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 literature reproducibility by division with 2.8. In general, when no literature reproducibility is available, another target may be used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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

The z-scores were calculated in accordance with:

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

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.
Absolute values for z<2 are very common and absolute values for z>3 are very rare. Therefore the usual interpretation of z-scores is as follows:

	$\lvert z \rvert$	< 1	good
1 <	$\lvert z \rvert$	< 2	satisfactory
2 <	$\lvert z \rvert$	< 3	questionable
3 <	$\lvert z \rvert$		unsatisfactory

4 EVALUATION

In this interlaboratory study, no problems were encountered with the dispatch of the samples. No participants reported test results after the final reporting date, but five participants did not report any test results at all. Thus, 27 of the 32 participants submitted test results.
In total over 380 (intermediate) results were reported, of which 148 test results in both mg/dm^2 and mg/kg food simulant. In total eleven statistical outliers were observed, which is 7.4% of the 148 test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

For the determination of Specific Migration, several standardised test methods exist. The most relevant literature is the EN13130 parts 1 and 13. In EN13130-1 is described how the specific migration test may be performed. In EN13130-13 the repeatability is given in the precision statement. However, this guideline describes only the analytical determination of BPA in the simulant (e.g. aqueous simulant by HPLC and fluorescence as detection), but not the migration test. The repeatability of EN13130-13 appears not to be very realistic as it is much smaller than the corresponding Horwitz value ($r_{CEN/TS13130-13:05}$ =0.18 mg/l, compare with $r_{Horwitz}$ = 0.50 mg/l (1.51/3), both at a level of 4 mg/l BPA). Therefore, it was decided to estimate the target reproducibilities from the Horwitz equation.

About 67% of the participants reported to have used test method EN13130-13 for the specific migration of BPA and 22% of the participants reported to have used an ‘in house’ method. Another two participants reported to have used test method EN13130-1 and one reported EN13130-1+GB/T23296 as test method.

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.

4.1 EVALUATION PER TEST

In this paragraph, the test results are discussed per test.
It was possible to repeat the calculations from most participants using the intermediate test results (see appendix 2) as reported by the participants. The calculations from laboratories 362, 2714 and 3216 could not be repeated.
Presumably participant 362 made a transition error in the value of the contact surface (see also remark in appendix 2) and the test results were therefore not excluded from the statistical evaluations. Participant 2714 reported very deviating test results which were all indicated as statistical outliers and therefore these were not used in the statistical evaluations.
Participant 3216 did not report the contact surface and therefore the calculation could not be repeated. This participant explained that it had converted the measured concentration BPA in mg/l in simulant solution to mg/kg by using the density of the simulant solution. And subsequently converted this to mg/dm^2 by dividing by 6. In Directive 8.2/711/EEC it is stated that the specific gravity of all the simulants should be conventionally be assumed to 1. Therefore, the test results of this participant were suspect and were excluded from the statistical evaluations.

Specific migration of BPA in mg/dm^2 contact surface:
This determination may be problematic for step 1 and step 3. One statistical outlier was observed in step 1 and two statistical outliers were observed in step 3. The calculated reproducibilities of steps 1 and 3 after rejection of the suspect data are not in agreement with the estimated reproducibilities using the Horwitz equation.
Remarkebly, this determination may be not problematic for step 2. One statistical outlier was observed. The calculated reproducibility after rejection of the suspect data is in agreement with the estimated reproducibility using the Horwitz equation.

Specific migration of BPA in mg/kg food simulant:
The migration results in mg/kg food simulant are obtained by multiplication of the specific migration in mg/dm^2 with the conventional conversion factor 6 (except for participant 3216 as explained above).
This determination may be problematic. One statistical outlier was observed in steps 1 and 2 and five statistical outliers were observed in step 3. The calculated reproducibilities of steps 1, 2 and 3 after rejection of the statistical outliers are not in agreement with the estimated reproducibilities using the Horwitz equation.

4.2 PERFORMANCE EVALUATION OF THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant reference method, here Horwitz, and the reproducibility as found for the group of participating laboratories. The target reproducibilities derived from reference methods are compared in the next two tables.

Specific Migration	unit	n	Average	2.8 * sd	R (target)
BPA, Step 1	mg/dm^2	24	0.0255	0.0319	0.0199
BPA, Step 2	mg/dm^2	23	0.0133	0.0124	0.0114
BPA, Step 3	mg/dm^2	23	0.0100	0.0140	0.0090

Table 3: Reproducibilities of tests on sample #17625 in mg/dm^2

Specific Migration	unit	n	Average	2.8 * sd	R (target)
BPA, Step 1	mg/kg fs	22	0.182	0.224	0.105
BPA, Step 2	mg/kg fs	21	0.092	0.100	0.059
BPA, Step 3	mg/kg fs	18	0.057	0.069	0.039

Table 4: Reproducibilities of tests on sample #17625 in mg/kg food simulant (fs)

Without further statistical calculations, it can be concluded that there is not a good compliance of the group of laboratories with the relevant target reproducibility (see for discussion paragraph 4.1 and 5).

4.3 COMPARISON OF PROFICIENCY TEST OF SEPTEMBER 2017 TO PREVIOUS PROFICIENCY TESTS

The evolution of the uncertainty for Specific Migration in mg/dm^2 and/or mg/kg as observed in this proficiency scheme and the comparison with the findings in previous rounds is listed in table 5.

	BPA via article filling	Metals via total immersion	DEHP via total immersion	BPA via total immersion	Formalde hyde via article filling	Target (Horwitz)	Conc range
2012	----	----	----	----	41 - 47%	14-20%	0.2 - 3
2013	----	----	----	----	41 - 61%	14-20%	0.2 - 3
2014	----	----	----	44 - 52%	----	14-20%	0.2 - 3
2015	----	----	34 - 40%	----	----	14-20%	0.2 - 3
2016	----	29 - 30%	----	----	----	14-20%	0.2 - 3
2017	33 - 50%	----	----	----	----	20-33%	0.009 – 0.2

Table 5: comparison of the uncertainties in % for Specific Migration in mg/dm^2 and/or mg/kg in the previous and present PT

From the above table, it is clear that the performance of this PT does not show improvement compared to the PTs of the last years. It also shows that the strict requirements, estimated from the Horwitz equation are not met.
When looking at the group of laboratories that reported analytical details in line to test method EN13130-1, the uncertainties for BPA determination the vary from 21 to 45%, which partly meet the target requirements (see for more discussion paragraph 5).

4.4 EVALUATION OF THE ANALYTICAL DETAILS

The reported analytical details that were used by the participants are listed in appendix 3. About 59% reported to be accredited for the determination of the specific migration of BPA and 37% reported not to be accredited. It appeared that the variation in the reported test results by the accredited participants is much lower than the variation in the reported test results by the not accredited participants. Also, the mean BPA values obtained by the accredited group is much higher, see overview in table 6. For step 1 this difference is statistically different at 99% (t-test).

Step	Accredited Mean in mg/dm^2	Accredited RSD %	Not Accredited Mean in mg/dm^2	Not Accredited RSD %
1	0.0275	38%	0.0155	48%
2	0.0140	26%	0.0098	64%
3	0.0104	47%	0.0084	72%

Table 6: comparison over accredited and not-accredited labs for Specific Migration in mg/dm^2

About 59% reported not to clean the sample before the determination of the specific migration of BPA and 37% reported to clean the cup. Four participants reported to clean the cup with water which is not in line with test method EN13130-1 paragraph 15.5. Five participants reported to clean the cup with lint-free cloth or brush or paper.

The reported surface to volume ratio in the additional questionnaire varies from 0.0060 to 0.0081 dm^2/ml. The average surface to volume ratio was 0.0071 dm^2/ml. Each participant used the same ratio over the three migration steps. One participant did change the amount of simulant but the ratio remained the same.

Almost all participants (89%) preheated the simulant solution. Two participants mentioned not to preheat the simulant solution.

5 DISCUSSION

Before the start of this PT, it was assumed that a wide range of test results would be reported when the choice of the test conditions would have been left to the participating laboratories. Therefore, a set of predetermined test conditions was given together with the instructions to all participants. These pre-set conditions were:

Simulant	95% Ethanol
Exposure time	2 hours (120 min)
Exposure temperature	70.0 °C
Migration method	Article filling, repeated use

Table 7: test conditions described for this PT

From the reported details, it became clear that most participants followed the test method that was reported. Several deviating conditions (like rinsing with water or not pre-heating the simulant solution) were used. These deviations may have an influence on the variation in the test results as observed. Therefore, it was decided to investigate what the variation will become when test results of the deviating conditions are excluded. The selection (called “selected set”) was based on the exclusion of: simulant not preheated (2 participants), (might have) used density instead of factor 1 (2 participants), rinsed with water (3 participants).

Specific Migration	unit	n	Average	2.8 * sd	R (target)
BPA, Step 1	mg/dm^2	24	0.0255	0.0319	0.0199
“selected set”	mg/dm^2	18	0.0247	0.0266	0.0193
BPA, Step 2	mg/dm^2	23	0.0133	0.0124	0.0114
“selected set”	mg/dm^2	18	0.0132	0.0078	0.0114
BPA, Step 3	mg/dm^2	23	0.0100	0.0140	0.0090
“selected set”	mg/dm^2	19	0.0100	0.0127	0.0090

Table 8: Reproducibilities of tests on sample #17625 in mg/dm^2

Specific Migration	unit	n	Average	2.8 * sd	R (target)
BPA, Step 1	mg/kg food	22	0.182	0.224	0.105
“selected set”	mg/kg food	16	0.179	0.208	0.104
BPA, Step 2	mg/kg food	21	0.092	0.100	0.059
“selected set”	mg/kg food	16	0.094	0.084	0.060
BPA, Step 3	mg/kg food	18	0.057	0.069	0.039
“selected set”	mg/kg food	14	0.054	0.049	0.038

Table 9: Reproducibilities of tests on sample #17625 in mg/kg food simulant

The deviating conditions obviously did have some effect on the observed variations but not on the averages. The effect of accreditation on the average and the variation compared to the group of not accredited laboratories is larger (see table 6 or appendix 1).

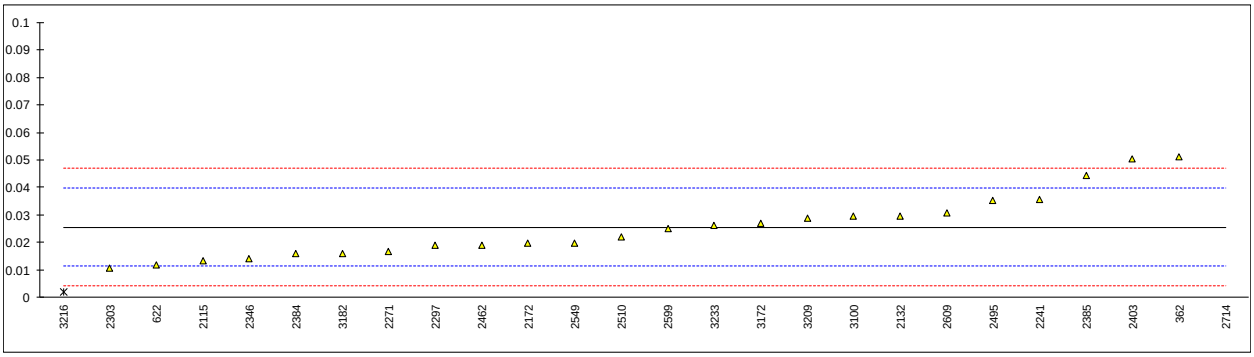
Each laboratory should evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and the quality of the analytical results.

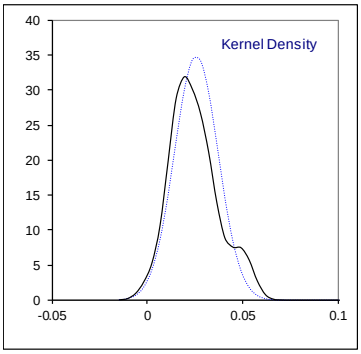
APPENDIX 1

Determination of 1st Specific Migration of BPA on sample #17625; results in mg/dm^2 per contact surface

lab	method	value	mark	z(targ)	remarks
310		-----		-----	
362	EN13130-13	0.051	C	3.59	first reported: 0.1195
622	In house	0.012		-1.91	
2115	EN13130-13	0.0135		-1.69	
2132	EN13130-13	0.02979		0.60	
2172	EN13130-13	0.0198		-0.81	
2241	EN13130-13	0.0356		1.42	
2271	EN13130-13	0.0168		-1.23	
2297	EN13130-13	0.0189		-0.93	
2303	EN13130-13	0.0108		-2.08	
2346	In house	0.014		-1.62	
2370		-----		-----	
2384	INH-537	0.016		-1.34	
2385	In house	0.04430		2.65	
2403	EN13130-13	0.0504		3.51	
2433		-----		-----	
2462	EN13130-13	0.0190		-0.92	
2495	EN13130-13	0.03526		1.37	
2504		-----		-----	
2510	In house	0.022	C	-0.50	first reported: 0.088
2549	EN13130-13	0.0199		-0.79	
2599	EN13130-13	0.025		-0.07	
2609	EN13130-1	0.03079		0.74	plus method GB/T23296
2714	EN13130-13	1.05436	C,R(0.01)	145.07	first reported: 0.1952526
2780		-----		-----	
3100	EN13130-13	0.0295		0.56	
3163		-----		-----	
3172	EN13130-13	0.027		0.21	
3182	EN13130-13	0.016		-1.34	
3209	EN13130-13	0.029	C	0.49	first reported: 56.72
3216	EN13130-1	0.0019	ex	-3.33	
3233	In house	0.0261		0.08	

			Accredited labs only	Selected test results, see § 5
	normality	OK	OK	not OK
	n	24	16	18
	outliers	1+1ex	0	0
	mean (n)	0.02552	0.02748	0.02471
	st.dev. (n)	0.011404 RSD=45%	0.010467 RSD=38%	0.009493 RSD=38%
	R(calc.)	0.03193	0.02931	0.02658
	st.dev.(Horwitz)	0.007092	0.007552	0.006901
	R(Horwitz)	0.01986	0.021147	0.01932

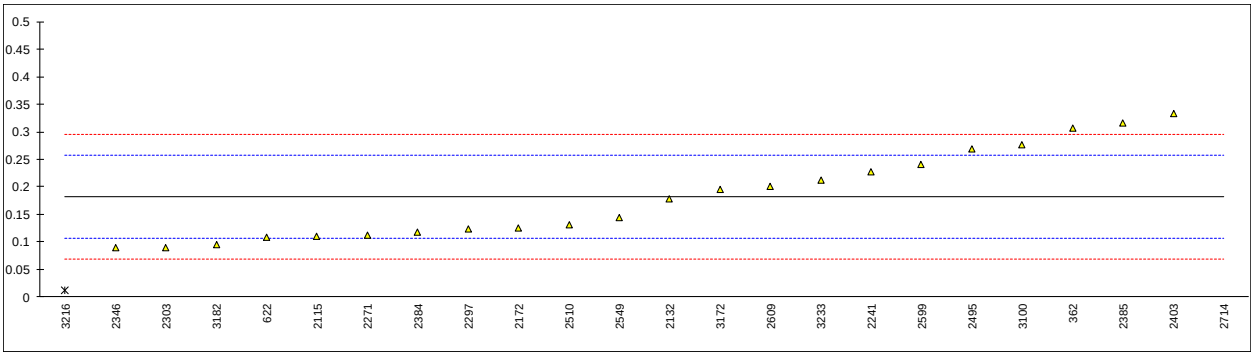


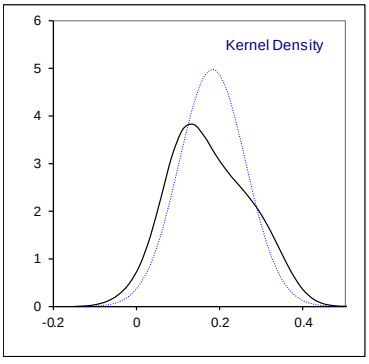


Determination of 1st Specific Migration of BPA on sample #17625; results in mg/kg food stimulant

lab	method	value	mark	z(targ)	remarks
310		-----		-----	
362	EN13130-13	0.307		3.32	
622	In house	0.109		-1.94	
2115	EN13130-13	0.11		-1.91	
2132	EN13130-13	0.1787		-0.09	
2172	EN13130-13	0.1253		-1.51	
2241	EN13130-13	0.228		1.22	
2271	EN13130-13	0.113		-1.84	
2297	EN13130-13	0.123		-1.57	
2303	EN13130-13	0.0897		-2.45	
2346	In house	0.089		-2.47	
2370		-----		-----	
2384	INH-537	0.117		-1.73	
2385	In house	0.31645		3.57	
2403	EN13130-13	0.333		4.01	
2433		-----		-----	
2462	EN13130-13	-----		-----	
2495	EN13130-13	0.2685		2.29	
2504		-----		-----	
2510	In house	0.132		-1.33	
2549	EN13130-13	0.145		-0.99	
2599	EN13130-13	0.24		1.54	
2609	EN13130-1	0.2015		0.52	plus method GB/T23296
2714	EN13130-13	6.32616	C,R(0.01)	163.19	first reported: 1.1715156
2780		-----		-----	
3100	EN13130-13	0.2767		2.51	
3163		-----		-----	
3172	EN13130-13	0.196		0.37	
3182	EN13130-13	0.0959		-2.29	
3209	EN13130-13	-----		-----	
3216	EN13130-1	0.0115	ex	-4.53	
3233	In house	0.2114		0.78	

			Accredited labs only	Selected test results, see § 5
	normality	OK	OK	OK
	n	22	14	16
	outliers	1+1ex	0	0
	mean (n)	0.18210	0.20151	0.17914
	st.dev. (n)	0.080039 RSD=44%	0.079054 RSD=39%	0.074113 RSD=41%
	R(calc.)	0.22411	0.22135	0.20752
	st.dev.(Horwitz)	0.037650	0.041033	0.037129
	R(Horwitz)	0.10542	0.11489	0.10396

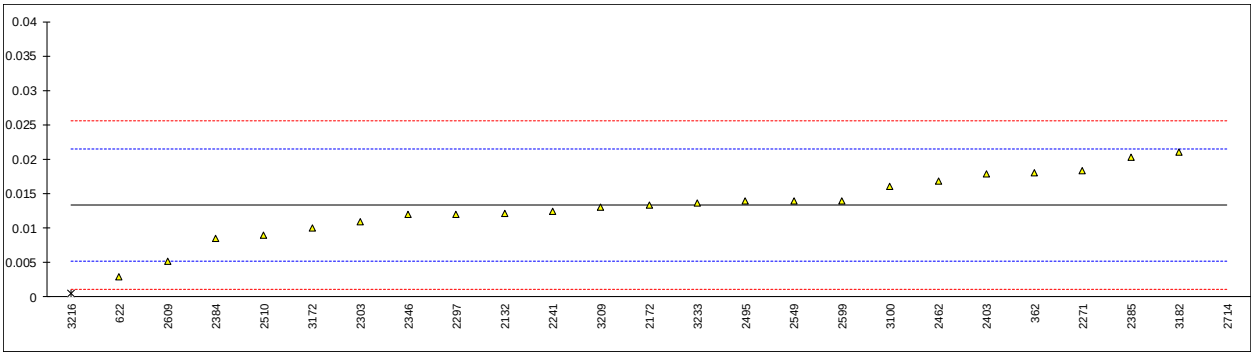


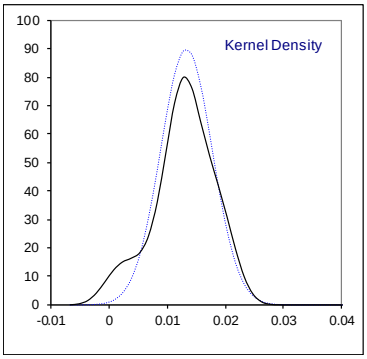


Determination of 2nd Specific Migration of BPA on sample #17625; results in mg/dm^2 per contact surface

lab	method	value	mark	z(targ)	remarks
310		-----		-----	
362	EN13130-13	0.018	C	1.16	first reported: 0.041
622	In house	0.0029		-2.55	
2115	EN13130-13	< 0.1		-----	
2132	EN13130-13	0.01221		-0.27	
2172	EN13130-13	0.0133		0.00	
2241	EN13130-13	0.0125		-0.19	
2271	EN13130-13	0.0183		1.23	
2297	EN13130-13	0.0120		-0.32	
2303	EN13130-13	0.0109		-0.59	
2346	In house	0.012		-0.32	
2370		-----		-----	
2384	INH-537	0.0086		-1.15	
2385	In house	0.02036		1.73	
2403	EN13130-13	0.0179		1.13	
2433		-----		-----	
2462	EN13130-13	0.0168		0.86	
2495	EN13130-13	0.01392		0.15	
2504		-----		-----	
2510	In house	0.00897	C	-1.06	first reported: 0.036
2549	EN13130-13	0.0140		0.17	
2599	EN13130-13	0.014		0.17	
2609	EN13130-1	0.005270		-1.97	plus method GB/T23296
2714	EN13130-13	0.36788	C,R(0.01)	87.01	first reported: 0.072133
2780		-----		-----	
3100	EN13130-13	0.0161		0.69	
3163		-----		-----	
3172	EN13130-13	0.01		-0.81	
3182	EN13130-13	0.021		1.89	
3209	EN13130-13	0.013	C	-0.07	first reported: 26.62
3216	EN13130-1	0.0006	ex	-3.11	
3233	In house	0.0137		0.10	

			Accredited labs only	Selected test results, see § 5
	normality	OK	suspect	OK
	n	23	15	18
	outliers	1+1ex	0	0
	mean (n)	0.01329	0.01398	0.01323
	st.dev. (n)	0.004444 RSD=33%	0.003680 RSD=26%	0.002770 RSD=21%
	R(calc.)	0.01244	0.01030	0.00776
	st.dev.(Horwitz)	0.004075	0.004254	0.004059
	R(Horwitz)	0.01141	0.011910	0.01137

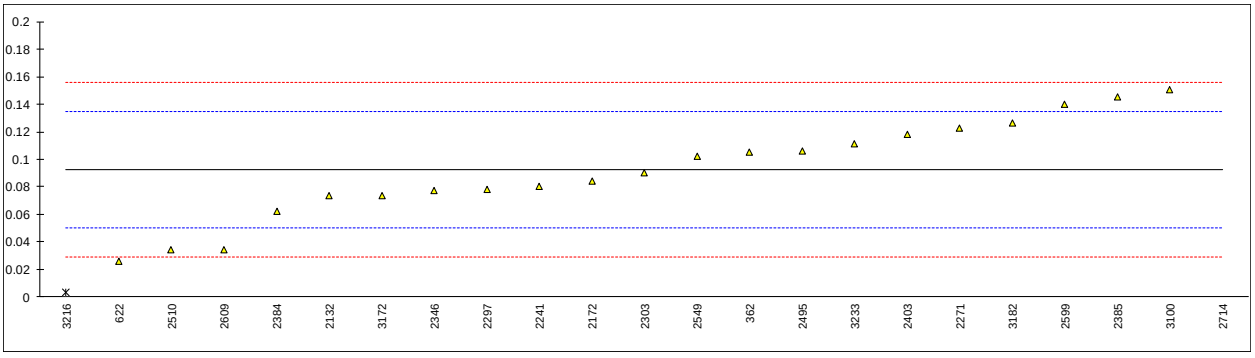


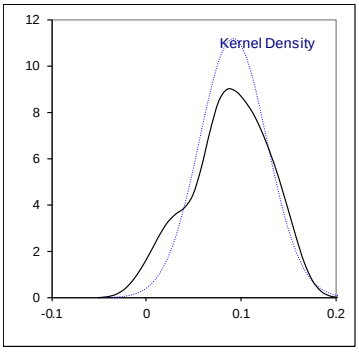


Determination of 2nd Specific Migration of BPA on sample #17625; results in mg/kg food stimulant

lab	method	value	mark	z(targ)	remarks
310		-----		-----	
362	EN13130-13	0.105		0.60	
622	In house	0.026		-3.14	
2115	EN13130-13	< 0.01		<-3.89	Possibly a false negative test result?
2132	EN13130-13	0.07324		-0.91	
2172	EN13130-13	0.084		-0.40	
2241	EN13130-13	0.080		-0.59	
2271	EN13130-13	0.123		1.45	
2297	EN13130-13	0.078		-0.68	
2303	EN13130-13	0.0902		-0.10	
2346	In house	0.077		-0.73	
2370		-----		-----	
2384	INH-537	0.062		-1.44	
2385	In house	0.14545		2.51	
2403	EN13130-13	0.118		1.21	
2433		-----		-----	
2462	EN13130-13	-----		-----	
2495	EN13130-13	0.1060		0.64	
2504		-----		-----	
2510	In house	0.034		-2.76	
2549	EN13130-13	0.102		0.45	
2599	EN13130-13	0.14		2.25	
2609	EN13130-1	0.03449		-2.74	plus method GB/T23296
2714	EN13130-13	2.20728	C,R(0.01)	99.97	first reported: 0.432798
2780		-----		-----	
3100	EN13130-13	0.1507		2.76	
3163		-----		-----	
3172	EN13130-13	0.0733		-0.90	
3182	EN13130-13	0.1265		1.61	
3209	EN13130-13	-----		-----	
3216	EN13130-1	0.0036	ex	-4.20	
3233	In house	0.1113		0.89	

			Accredited labs only	Selected test results, see § 5
	normality	OK	OK	OK
	n	21	13	16
	outliers	1+1ex	0	0
	mean (n)	0.09239	0.10055	0.09392
	st.dev. (n)	0.035728 RSD=39%	0.034194 RSD=34%	0.030104 RSD=32%
	R(calc.)	0.10004	0.09574	0.08429
	st.dev.(Horwitz)	0.021156	0.022733	0.021453
	R(Horwitz)	0.05924	0.063653	0.06007

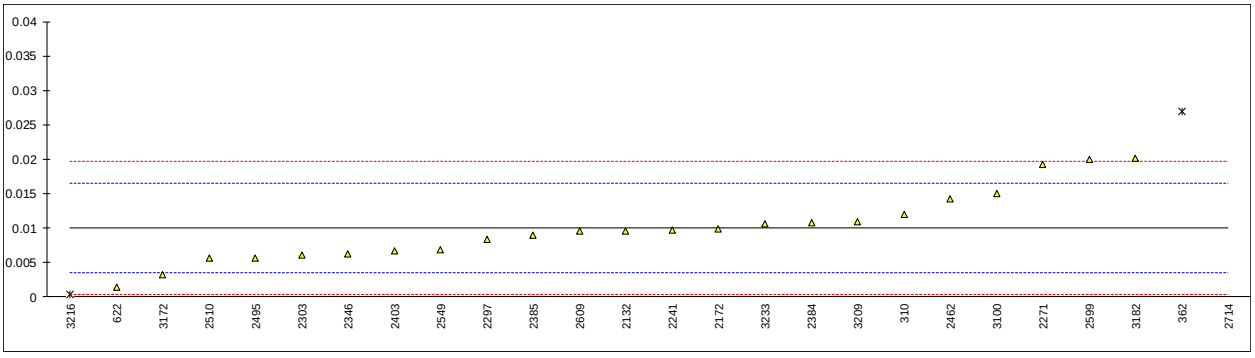


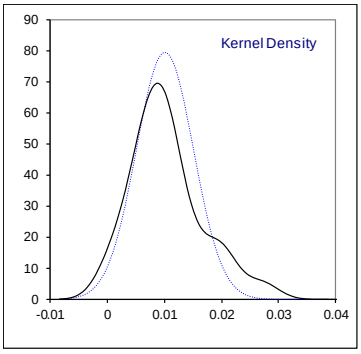


Determination of 3rd Specific Migration of BPA on sample #17625; results in mg/dm^2 per contact surface

lab	method	value	mark	z(targ)	remarks
310		0.012		0.61	
362	EN13130-13	0.027	C,R(0.01)	5.28	first reported: 0.062
622	In house	0.0014		-2.69	
2115	EN13130-13	< 0.1		-----	
2132	EN13130-13	0.009627		-0.13	
2172	EN13130-13	0.00996		-0.03	
2241	EN13130-13	0.0097		-0.11	
2271	EN13130-13	0.0192		2.85	
2297	EN13130-13	0.0083		-0.54	
2303	EN13130-13	0.0061		-1.23	
2346	In house	0.0062		-1.20	
2370		-----		-----	
2384	INH-537	0.0108		0.24	
2385	In house	0.00894		-0.34	
2403	EN13130-13	0.0067		-1.04	
2433		-----		-----	
2462	EN13130-13	0.0143		1.33	
2495	EN13130-13	0.00566		-1.36	
2504		-----		-----	
2510	In house	0.00559	C	-1.39	first reported: 0.0022
2549	EN13130-13	0.0068		-1.01	
2599	EN13130-13	0.020		3.10	
2609	EN13130-1	0.009574		-0.15	plus method GB/T23296
2714	EN13130-13	0.17284	C,R(0.01)	50.70	first reported: 0.0249057
2780		-----		-----	
3100	EN13130-13	0.0150		1.54	
3163		-----		-----	
3172	EN13130-13	0.0032		-2.13	
3182	EN13130-13	0.0202		3.16	
3209	EN13130-13	0.011	C	0.30	first reported: 21.88
3216	EN13130-1	0.0003	ex	-3.03	
3233	In house	0.0107		0.21	

			Accredited labs only	Selected test results, see § 5
	normality	OK	OK	OK
	n	23	15	19
	outliers	2+1ex	0	0
	mean (n)	0.01004	0.01039	0.01004
	st.dev. (n)	0.005009 RSD=50%	0.004848 RSD=47%	0.004550 RSD=45%
	R(calc.)	0.01402	0.01357	0.01274
	st.dev.(Horwitz)	0.003211	0.003306	0.003211
	R(Horwitz)	0.00899	0.00926	0.00899

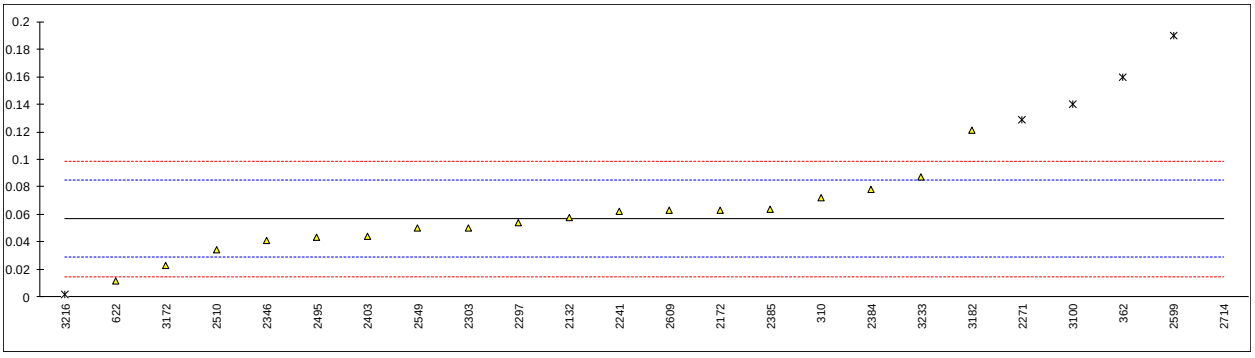


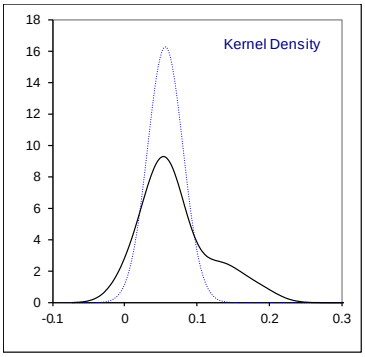


Determination of 3rd Overall Migration of BPA on sample #17625; results in mg/kg food stimulant

lab	method	value	mark	z(targ)	remarks
310		0.072		1.10	
362	EN13130-13	0.1597	R(0.05)	7.39	
622	In house	0.012		-3.20	
2115	EN13130-13	< 0.01		<-3.34	Possibly a false negative test result?
2132	EN13130-13	0.05776		0.08	
2172	EN13130-13	0.063		0.46	
2241	EN13130-13	0.062		0.39	
2271	EN13130-13	0.129	R(0.01)	5.19	
2297	EN13130-13	0.054		-0.19	
2303	EN13130-13	0.0505		-0.44	
2346	In house	0.041		-1.12	
2370		-----		-----	
2384	INH-537	0.0778		1.52	
2385	In house	0.06383		0.52	
2403	EN13130-13	0.0444		-0.88	
2433		-----		-----	
2462	EN13130-13	-----		-----	
2495	EN13130-13	0.0431		-0.97	
2504		-----		-----	
2510	In house	0.034		-1.62	
2549	EN13130-13	0.050		-0.47	
2599	EN13130-13	0.19	R(0.01)	9.56	
2609	EN13130-1	0.06266		0.43	plus method GB/T23296
2714	EN13130-13	1.03704	C,R(0.01)	70.25	first reported: 0.1494342
2780		-----		-----	
3100	EN13130-13	0.1402	R(0.05)	5.99	
3163		-----		-----	
3172	EN13130-13	0.023		-2.41	
3182	EN13130-13	0.1212		4.63	
3209	EN13130-13	-----		-----	
3216	EN13130-1	0.0018	ex	-3.93	
3233	In house	0.0869		2.17	

			Accredited labs only	Selected test results, see § 5
	normality	suspect	OK	OK
	n	18	10	14
	outliers	5+1ex	3	3
	mean (n)	0.05662	0.05108	0.05425
	st.dev. (n)	0.024530 RSD=43%	0.013342 RSD=26%	0.017321 RSD=32%
	R(calc.)	0.06868	0.03736	0.04850
	st.dev.(Horwitz)	0.013957	0.012788	0.013459
	R(Horwitz)	0.03908	0.03581	0.03769





APPENDIX 2

Details on final concentration, surface area and volume of simulant reported per step

lab	1st Final conc. (mg/l)	1st surface area (dm^2)	1st volume simulant (ml)	2nd Final conc. (mg/l)	2nd surface area (dm^2)	2nd volume simulant (ml)	3rd Final conc. (mg/l)	3rd surface area (dm^2)	3rd volume simulant (ml)
310	-----	-----	-----	-----	-----	-----	0.09	1.5	200
362	0.384	0.675 *)	210	0.132	0.675 *)	210	0.20	0.675 *)	210
622	0.086	1.755	250	0.020	1.755	250	0.010	1.755	250
2115	0.11	1.22	150	< 0.1	1.22	150	< 0.1	1.22	150
2132	0.1990	1.670	250	0.08156	1.670	250	0.06432	1.670	250
2172	0.1253	1.58	250	0.084	1.58	250	0.063	1.58	250
2241	0.228	1.59	250	0.080	1.59	250	0.062	1.59	250
2271	0.113	1.68	250	0.123	1.68	250	0.129	1.68	250
2297	0.123	1.63	250	0.078	1.63	250	0.054	1.63	250
2303	0.0897	1.82	220	0.0902	1.82	220	0.0505	1.82	220
2346	0.089	1.6467	250	0.077	1.6467	250	0.041	1.6467	250
2370	-----	-----	-----	-----	-----	-----	-----	-----	-----
2384	0.117	1.6504	230	0.062	1.6504	230	0.0778	1.6504	230
2385	0.31645	1.5	210	0.14545	1.5	210	0.06383	1.5	210
2403	0.333	1.650	250	0.118	1.650	250	0.0444	1.650	250
2433	-----	-----	-----	-----	-----	-----	-----	-----	-----
2462	0.134	1.62	230.0	0.118	1.62	230.0	0.101	1.62	230.0
2495	0.2685	1.5230	200	0.1060	1.5230	200	0.0431	1.5230	200
2504	-----	-----	-----	-----	-----	-----	-----	-----	-----
2510	0.15	1.70	250	0.061	1.70	250	0.038	1.70	250
2549	0.145	1.456	200	0.102	1.456	200	0.050	1.456	200
2599	0.191	1.5	200	0.107	1.5	200	0.153	1.5	200
2609	0.2015	1.7017	260	0.03449	1.7017	260	0.06266	1.7017	260
2714	1.2363888	1.26645	200.0	0.4567647	1.5514026	245.0	0.1577089	1.58306	250.0
2780	-----	-----	-----	-----	-----	-----	-----	-----	-----
3100	0.2241	1.29	170	0.1221	1.29	170	0.1135	1.29	170
3163	-----	-----	-----	-----	-----	-----	-----	-----	-----
3172	0.196	1.60	220	0.0733	1.60	220	0.023	1.60	220
3182	0.110	1.72	250	0.145	1.72	250	0.139	1.72	250
3209	0.210 C	1.620	230	0.090 C	1.620	230	0.075 C	1.620	230
3216	0.00954	-----	200	0.0030	-----	200	0.0015	-----	200
3233	0.2114	1.62	200	0.1113	1.62	200	0.0869	1.62	200

*) Lab 362 presumably a typo in surface area; the calculation was correct when 1.675 was used instead of 0.675

C: Lab 3209 first reported respectively: 0.0399; 0.0187; 0.0154

APPENDIX 3

Analytical Details

lab	accredited acc. ISO/IEC17025 for this test	sample cleaned prior to the migration step	Cleaned with	the surface-to-volume ratio (in dm² / mL)	simulant preheated before use
310	No	No		7.5 (? in calc. 0.0075 dm2/ml))	Yes
362	---	---			---
622	No	No		1.7548 dm^2/250mL = 0.007 dm^2/mL	No
2115	Yes	No		1.22 dm^2/150mL = 0.0081 dm^2/mL	No
2132	Yes	Yes	lint-free cloth	0.00668 dm^2/mL	Yes
2172	Yes	No		1.58 dm^2/250mL = 0.0063 dm^2/mL	Yes
2241	Yes	No		1.6 dm^2/250ml = 0.0064 dm^2/mL	Yes
2271	Yes	No		0.0067 dm^2/mL	Yes
2297	No	Yes		6.5 dm^2/1000mL = 0.0065 dm^2/mL	Yes
2303	No	No			Yes
2346	Yes	Yes	brush	1.6467 dm^2/250 ml = 0.0066 dm^2/mL	Yes
2370	---	---			---
2384	No	Yes	kimwipes	0.007176 dm^2/mL	Yes
2385	Yes	Yes	water		Yes
2403	Yes	No		1.65 dm^2/250 mL = 0.0066 dm^2/mL	Yes
2433	---	---			---
2462	Yes	Yes	paper	Article filling	Yes
2495	Yes	No		0.0076 dm^2/mL	Yes
2504	---	---			---
2510	No	No		0.0068 dm^2/mL	Yes
2549	Yes	No		0.007 dm^2/mL	Yes
2599	Yes	No			Yes
2609	Yes	Yes	Milli Q water		Yes
2714	No	Yes	lint-free cloth	167ml/dm^2 = 0.0060 dm^2/mL	Yes
2780	---	---			---
3100	Yes	Yes	water	1.29 dm^2/170mL = 0.0076 dm^2/mL	Yes
3163	---	---			---
3172	Yes	No		0.00727 dm^2/mL	Yes
3182	No	Yes	Milli Q water	1.72 dm^2/250mL = 0.0068 dm^2/mL	Yes
3209	Yes	No		1:1.4 (cm^2/mL?=0.0071 dm^2/mL)	Yes
3216	No	No			Yes
3233	No	No		1.62dm^2/200mL = 0.0081 dm^2/mL	Yes

Lab 2495 presumably a typo in surface to volume ratio; reported 0.076, in calc. 0.0076 dm^2/ml was used

APPENDIX 4

Number of participating laboratories per country

2 labs in BRAZIL
1 lab in BULGARIA
1 lab in FRANCE
1 lab in GERMANY
2 labs in HONG KONG
1 lab in INDIA
1 lab in INDONESIA
1 lab in IRELAND
3 labs in ITALY
1 lab in MALAYSIA
9 labs in P.R. of CHINA
1 lab in PHILIPPINES
1 lab in SPAIN
1 lab in SWEDEN
1 lab in TAIWAN R.O.C.
2 labs in THAILAND
2 labs in THE NETHERLANDS
1 lab in UNITED KINGDOM

APPENDIX 5

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 = probably an error in calculation
U = test result probably reported in a different unit
W = test result withdrawn on request of the participant
ex = test result excluded from statistical evaluation
n.a. = not applicable
n.e. = not evaluated
n.d. = not detected
fr. = first reported

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, March 2017
2 EN13130-1 Materials and articles in contact with foodstuffs – Plastics substances subject to limitation
3 EN13130-13 Materials and articles in contact with foodstuffs – Plastics substances subject to limitation - Determination of 2,2-bis(4-hydroxyphenyl)propane (Bisphenol A) in food simulants
4 Commission regulation (EU) No 1935/2004 of 27 October 2004, on materials and articles intended to come into contact with food
5 Commission regulation (EU) No 10/2011 of 14 January 2011 on plastic materials and articles intended to come into contact with food
6 ASTM E178:02
7 ASTM E1301:03
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9 ISO 5725, parts 1-6, 1994
10 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
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18 R.G. Visser, Accred Qual Assur, 14:29-34 (2009)
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