Results of Proficiency Test
Specific migration (fcm)
September 2014

CONTENTS

Appendices:

1   INTRODUCTION

On request of a number of participants in the iis PT program it was decided to start PTs on food contact materials in 2012. This PT was repeated in 2013 and 2014.

During the contact of materials, like kitchenware, with food, 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 (EC) 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. The determination of specific migration requires additional analytical testing following the migration step, while the determination of the overall migration requires weighing as only quantitative analytical technique. This makes the specific migration of BPA from food contact materials more difficult than determination of the overall migration.

In the interlaboratory study of October 2014, 47 laboratories from 17 different countries participated (See appendix 4). In this report, the results of the 2014 proficiency test are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2   SET-UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. 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, that was known to give a measurable test result, labelled #14181, and to prescribe a number of test conditions (migration method, type of simulant, exposure time and temperature) to be used. Participants were also requested to report some intermediate test results.

2.1   ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in accordance with ISO/IEC 17043:2010, (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie, see also www.RVA.nl). This PT falls in the accreditation scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially 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 was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol can be downloaded from the iis website www.iisnl.com.

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 plastic plates containing Bisphenol A (BPA) for repetitive use in the food industry that gave positive test results for specific migration of BPA was selected.
The homogeneity of the batch was checked by determination of the Specific Migration of BPA on 8 stratified randomly selected plates.

	concentration of BPA in simulant in mg/l, 1st contact step, #14181
Sample 1	1.8
Sample 2	2.0
Sample 3	1.9
Sample 4	2.2
Sample 5	1.9
Sample 6	1.9
Sample 7	2.2
Sample 8	2.1

Table 1: results of the homogeneity test on the subsamples #14181

The repeatability for Specific Migration on the eight samples #14181 is in agreement with the repeatability of the laboratory performing the tests. Therefore, homogeneity of the samples #14181 was assumed.

To each of the participating laboratories one sample #14181 was sent on September 10, 2014.

2.5 ANALYSIS

The participants were requested to determine the Specific Migration of Bisphenol A (BPA) on the sample using the prescribed test conditions (total immersion, 2.0 hrs @100°C and 3% Acetic Acid as simulant). It was requested to report the analytical results using the indicated units on the report form and to use a minimum number of digits and not to round the results more. 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 results a detailed report form, on which the units were prescribed, was sent together with the sample. Also, a letter of instructions was added to the package.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were received.
The original data are tabulated per sample in the appendix 1 of this report.
The laboratories are represented by the code numbers.

Directly after the deadline, a reminder was sent to those laboratories that did not report results at that moment.
Shortly after the deadline, the available results were screened for suspect data. A 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 results. Additional or corrected results are used for the data analysis and the original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

The statistical calculations were performed as described in the procedures in the report 'iis Interlaboratory Studies, Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3).

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'.
After removal of outliers, this check was repeated. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon, Grubbs and or Rosner General ESD outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test and by R(0.01) for the Rosner General ESD test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test and by R(0.05) for the Rosner General ESD test (ref. 17). Both outliers and stragglers were not included in the calculations of averages and standard deviations. Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

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 analysis results are plotted. The corresponding laboratory numbers are under the X-axis.
The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle. Furthermore, Kernel Density

Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 4; refs.14 and 15). 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 spread of 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.
The z-scores were calculated in accordance with:

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

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

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used.
This should be done in order to evaluate the fit-for-useness of the reported test result, see also appendix 4; ref. 16.

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:

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

4   EVALUATION

In this interlaboratory study, no problems were encountered with the dispatch of the samples. Three participants reported test results after the final reporting date. Seven other participants did not report any test results at all. Thus, 41 of the 48 participants submitted test results. These 41 laboratories reported 357 numerical test results, of which 113 for Specific Migration in mg/dm^2. Observed were 12 outlying test results, which is 3.4% (5 for the results in mg/dm^2, or 4.4%). In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

A non Gaussian distribution was only observed for the reported specific migration results in mg/dm^2 for the third contact. Therefore this statistical evaluation should be used with due care.

For the determination of Specific Migration, several standardised test methods exist. The most relevant literature is the CEN/TS13130 parts 1 and 13. In CEN/TS13130-1 is described how the specific migration test may be performed. In CEN/TS13130-13 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. Therefore it is not strange to find that the repeatability of CEN/TS13130-13 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.

All reported results of laboratory 2217 were strongly deviating due to an unresolved error. Also a calculation error was observed in the specific migration results in mg/dm^2 for this laboratory. Two other laboratories (2497 and 3151) also appeared to have made a calculation error in the specific migration test result in mg/dm^2. These test results were excluded from the data prior to the statistical analysis.

4.1   PERFORMANCE EVALUATION OF THE GROUP OF LABORATORIES

The calculated reproducibilities and the target reproducibilities are compared in the next table.

	unit	n	Average	2.8 * sd	R (target)
Specific migration, 1st contact	mg/dm^2	38	0.54	0.68	0.27
Specific migration, 2nd contact	mg/dm^2	35	0.27	0.33	0.15
Specific migration, 3rd contact	mg/dm^2	34	0.19	0.28	0.11

Table 2: performance overview for sample #14181

4.2 EVALUATION

No significant differences were observed between the results of the 1st, the 2nd and 3rd contact.

Concentration BPA in simulant in mg/l:
> These intermediate results were not evaluated as they are in principle dependent of the amount of simulant used. When the total immersion method is used, use of a large amount of simulant will automatically give a lower BPA concentration than use of a small amount of simulant.

Specific migration of BPA in mg/dm^2:
> This determination may be very problematic. A wide range of test results was reported, e.g. for the 3rd contact: 0.02 – 4.78 mg/dm^2. In total five statistical outliers were detected. The three calculated reproducibilities, after rejection of the statistical outliers, are all not at all in agreement with the target reproducibilities estimated from the Horwitz equation.

Specific migration of BPA in mg/kg:
> These test results were not evaluated as they are in principle dependent of the choice of the factor that was used for the conversion of the Specific Migration in mg/dm^2 to mg/kg. See also the discussion in paragraph 5.

4.3 EVALUATION OF THE TEST METHODS USED

Most participants reported to have used as test method EN13130 (part 1 or 13). Also a small number of in house methods were reported.
The reported details that were used by the participants (volume of simulant and contact surface) as well as the actual BPA concentrations measured in the simulant for each of the three migration steps are listed in appendix 2.

4.4 COMPARISON WITH PREVIOUS PROFICIENCY TESTS

The number of participants increased from 38 in 2012 to 47 in this round. The percentage of outliers decreased over the years from 3.8% in 2012 to 3.4% of the numerical results in 2014.

The evolution of the uncertainty for Specific Migration in mg/dm^2 as observed in this proficiency scheme and the comparison with the findings in previous rounds is visualized in table 3.

	formaldehyde via article filling	BPA via total immersion
2012	41 - 47%	----
2013	41 – 61%	----
2014	----	44 – 52%

Table 3: comparison of the uncertainties in % for Specific Migration in mg/dm^2 in the previous rounds and in the present round

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 preset conditions were:

Simulant	3% acetic acid
Exposure time	2.0 hrs
Exposure temperature	100.0 °C
Migration method	Total immersion

Table 4: preset test conditions used in this PT

Not only a migration result was to be reported, but the participants were requested to report also the intermediate BPA concentration in the simulant per migration step. The reported BPA concentrations are listed in appendix 2. Using these intermediate test results it was possible to check the calculations done by the laboratories.
This revealed that initially indeed in the reported test results of a number of laboratories calculation errors were present. These test results were excluded from the statistical calculations.

It may be interesting to know whether the large variance in simulant volume may be of influence on the spread of Specific Migration test results, in other words whether the residual correlation may be significant. In below table 5, the evaluations of the test results based on a use of 20-163 ml of simulant and of 250-550 ml simulant are given.

	with simulant volumes between 20 - 163 ml	with simulant volumes between 250 - 550 ml
Specific migration 1st step in mg/dm^2	0.53	0.52
Specific migration 2nd step in mg/dm^2	0.24	0.27
Specific migration 3rd step in mg/dm^2	0.19	0.17

Table 5: influence of volume of simulant used on Specific Migration of BPA

From this evaluation it may be concluded that the influence of the use of different volumes of simulant is small and not significant.

The amount of simulant used by each participant varied from 20 – 550 ml, and the contact surface varied from 0.19 – 1.64 dm^2, see appendix 2.
Thus, the amount of simulant used per dm^2 contact surface varied from 12.7 - 335 ml/dm^2 (see appendix 2), while on average 165 ml/dm^2 was used. This average is in good agreement with the requirement of EN13130-1, where 100 ml shall be used for each 0.6 dm^2 (or 167 ml/dm^2):
quote
Where the surface-to-volume ratio to be used in contact with foodstuff is not known, conventional exposure conditions shall be used, i.e. 0,6 dm² of surface area of plastics in contact with 100 g of foodstuff or 100ml of food simulant.
unquote

It is remarkable to see that the range of reported contact surfaces was from 0.1884 – 1.64 dm^2. The surface of the disc was easy to determine. The disc was approx 1.6 dm^2, with a thickness of 0.75 mm. When the reported surfaces are closely examined (see appendix 2), it can be found that

27 laboratories reported a surface closely to 1.6 dm^2 (1.57 – 1.63) and 14 other laboratories reported a surface smaller than 0.85 dm^2, of which 5 exactly half of the total surface. Maybe those laboratories did not use the whole disc, but cut the disc in half or in smaller pieces. No correlation could be found between the reported contact surface and the measured BPA concentration.

The Specific Migration results in mg/kg show a larger spread than the results for Specific Migration in mg/dm^2. Upon investigation for the reason of the increase in spread, it was found that nine laboratories used the conventional factor 6 dm^2/kg in the calculation of the specific migration in mg/kg from the migration in mg/dm^2 cfr. EN13130-1:2004, paragraphs 4.7, 10.2 and 13.1.1. Another 24 laboratories obviously reported results in mg/L food simulant, not in mg/kg food as requested.

From the reported test results is was clear that for about 80% of the laboratories the results for 1st migration > 2nd migration > 3rd migration and even 90% of the laboratories reported 3rd migration < 1st migration. This is seen often when 3 subsequent migration steps are measured and the effect may be attributed to exhaustion of the migrating component.
Only one laboratory reported the results for 1st migration < 2nd migration < 3rd migration, the last two results being statistical outliers.

Each laboratory has to 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

Specific Migration of BPA, 1st contact on sample #14181; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
310		-----		-----	
330	EN13130-13	0.72		1.90	
362	INH-537	0.36		-1.90	
623	EN13130-13	0.475		-0.68	
2115	EN13130-1/13	0.1050		-4.59	
2129	CEN/TS13130-13/EN13130-1	0.51		-0.31	
2156	EN13130-13	0.27		-2.85	
2165	EN13130-13	0.40		-1.48	
2172	EN13130-13	0.54		0.00	
2184	EN13130-13	0.374		-1.75	
2190	EN13130-13	0.101		-4.63	
2196	EN13130-13	0.032		-5.36	
2215	EN13130-13	0.58		0.42	
2217	in house	14.48	E,R(0.01)	147.09	see §4.1, iis calculated 12.452
2229		-----		-----	
2241	EN13130-13	0.73		2.01	
2256	EN13130-13	0.57		0.32	
2271	EN13130-13	0.73		2.01	
2284	EN13130-13	0.55		0.11	
2309		-----		-----	
2353	EN13130-13	0.51		-0.31	
2372	EN13130-13	0.84		3.17	
2386	EN13130-1/in house	0.81		2.85	
2391		-----		-----	
2403		-----		-----	
2441	EN13130-13	0.33		-2.21	
2495		-----		-----	
2497	EN13130-13	0.125	E, ex	-4.38	see §4.1, iis calculated 0.192
2504	EN13130-13	0.80		2.75	
2510	EN13130-1	0.70		1.69	
2551	EN13130-13	1.2	C	6.97	first reported 0.005873 mg/dm^2
2594	EN13130-13	0.937		4.19	
2599	CEN/TS13130-13	0.43		-1.16	
2616	CEN/TS13130-13	0.680		1.48	
2627	EN13130-13	0.05		-5.17	
3100	EN13130-13	0.60		0.63	
3146	EN13130-1/in house	0.76		2.32	
3151	EN13130-13	0.48	E, ex	-0.63	see §4.1, iis calculated 0.344
3153		-----		-----	
3172	EN13130-13	0.51		-0.31	
3182	EN13130-13	0.46		-0.84	
3185	EN13130-13	0.64		1.06	
3209	EN13130-13	0.57		0.32	
3218	EN13130-13	0.58		0.42	
3225	EN13130-13	0.50		-0.42	
3228	EN13130-13	0.36		-1.90	
3233	in house	0.53		-0.10	
3246	EN13130-13	0.67		1.37	
	normality	OK			
	n	38			
	outliers	1	+ 2 excl.		
	mean (n)	0.5398			
	st.dev. (n)	0.24349			
	R(calc.)	0.6818			
	R(Horwitz)	0.2654		Compare R(EN13130-13) = 0.0688	
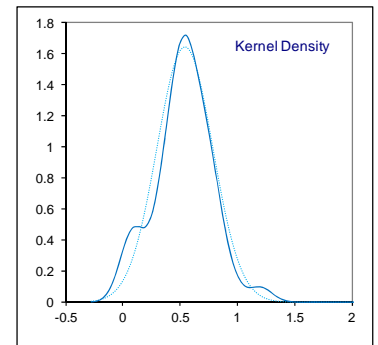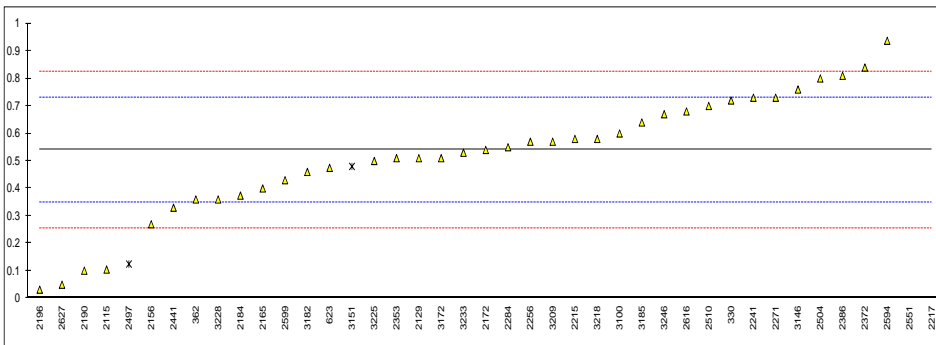
Specific Migration of BPA, 1st contact on sample #14181; results in mg/kg

lab	method	value	mark	z(targ)	conventional factor	remarks
310		-----		-----		
330	EN13130-13	4.30		-----		reported in mg/L
362	INH-537	4.30		-----		reported in mg/L
623	EN13130-13	2.85		-----		reported in mg/L
2115	EN13130-1/13	0.3393		-----		
2129	CEN/TS13130-13/EN13130-1	3.06		-----	used conventional factor 6	
2156	EN13130-13	0.86		-----		
2165	EN13130-13	2.42		-----		
2172	EN13130-13	3.22		-----		reported in mg/L
2184	EN13130-13	2.26		-----		reported in mg/L
2190	EN13130-13	1.01		-----		reported in mg/L
2196	EN13130-13	0.19		-----		reported in mg/L
2215	EN13130-13	3.48		-----	used conventional factor 6	
2217	in house	74.70		-----	used conventional factor 6	
2229		-----		-----		
2241	EN13130-13	4.38		-----	used conventional factor 6	
2256	EN13130-13	3.43		-----		reported in mg/L
2271	EN13130-13	4.39		-----		reported in mg/L
2284	EN13130-13	3.31		-----		reported in mg/L
2309		-----		-----		
2353	EN13130-13	3.07		-----		reported in mg/L
2372	EN13130-13	5.04		-----	used conventional factor 6	
2386	EN13130-1/in house	4.87		-----		reported in mg/L
2391		-----		-----		
2403		-----		-----		
2441	EN13130-13	1.99		-----		reported in mg/L
2495		-----		-----		
2497	EN13130-13	0.75		-----		
2504	EN13130-13	4.79		-----		
2510	EN13130-1	4.20		-----	used conventional factor 6	
2551	EN13130-13	9.5		-----		reported in mg/L
2594	EN13130-13	5.62		-----		
2599	CEN/TS13130-13	4.47		-----		reported in mg/L
2616	CEN/TS13130-13	4.080		-----	used conventional factor 6	
2627	EN13130-13	0.21		-----		reported in mg/L
3100	EN13130-13	3.62		-----		reported in mg/L
3146	EN13130-1/in house	4.59		-----	used conventional factor 6	
3151	EN13130-13	2.85		-----		
3153		-----		-----		
3172	EN13130-13	4.83		-----		
3182	EN13130-13	2.76		-----	used conventional factor 6	
3185	EN13130-13	3.84		-----		reported in mg/L
3209	EN13130-13	3.46		-----		reported in mg/L
3218	EN13130-13	3.49		-----		reported in mg/L
3225	EN13130-13	2.98		-----		reported in mg/L
3228	EN13130-13	2.18		-----		reported in mg/L
3233	in house	3.18		-----		reported in mg/L
3246	EN13130-13	4.06		-----		reported in mg/L

Specific Migration of BPA, 2nd contact on sample #14181; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
310		-----		-----	
330		-----		-----	
362	INH-537	0.13		-2.68	
623	EN13130-13	0.263		-0.17	
2115	EN13130-1/13	0.1606		-2.10	
2129	CEN/TS13130-13/EN13130-1	0.27		-0.04	
2156	EN13130-13	0.23		-0.79	
2165	EN13130-13	0.28		0.15	
2172	EN13130-13	0.28		0.15	
2184	EN13130-13	0.268		-0.07	
2190	EN13130-13	0.093		-3.38	
2196	EN13130-13	0.027		-4.63	
2215	EN13130-13	0.27		-0.04	
2217	in house	5.62	R(0.01)	101.06	
2229		-----		-----	
2241	EN13130-13	0.27		-0.04	
2256	EN13130-13	0.27		-0.04	
2271	EN13130-13	0.30		0.53	
2284	EN13130-13	0.26		-0.22	
2309		-----		-----	
2353	EN13130-13	0.31		0.72	
2372	EN13130-13	1.32	C, R(0.01)	19.81	first reported 0.86 mg/dm^2
2386	EN13130-1/in house	0.35		1.48	
2391		-----		-----	
2403		-----		-----	
2441	EN13130-13	0.21		-1.17	
2495		-----		-----	
2497	EN13130-13	0.052	E, ex, C	-4.15	first reported 0.026 mg/dm^2
2504	EN13130-13	0.39		2.23	
2510	EN13130-1	0.34		1.29	
2551	EN13130-13	0.45	C	3.37	first reported 0.494415 mg/dm^2
2594	EN13130-13	0.412		2.65	
2599		-----		-----	
2616	CEN/TS13130-13	0.322		0.95	
2627	EN13130-13	0.03		-4.57	
3100	EN13130-13	0.29		0.34	
3146	EN13130-1/in house	0.08		-3.63	
3151	EN13130-13	0.25	E, ex	-0.41	see §4.1, iis calculated 0.172
3153		-----		-----	
3172	EN13130-13	0.38	C	2.04	first reported 0.26 mg/dm^2
3182	EN13130-13	0.23		-0.79	
3185	EN13130-13	0.27		-0.04	
3209	EN13130-13	0.29		0.34	
3218	EN13130-13	0.38		2.04	
3225	EN13130-13	0.10		-3.25	
3228	EN13130-13	0.30		0.53	
3233	in house	0.58		5.82	
3246	EN13130-13	0.43		2.99	

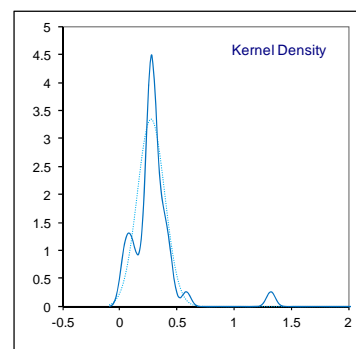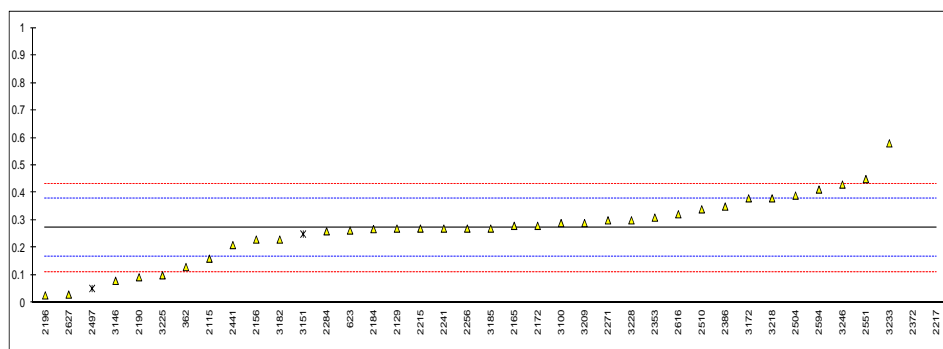| | | | | |
|---|---|---|---|
| normality | OK | | |
| n | 35 | | |
| outliers | 2 | +2 excl. | |
| mean (n) | 0.2719 | | |
| st.dev. (n) | 0.11914 | | |
| R(calc.) | 0.3336 | | |
| R(Horwitz) | 0.1482 | | |

Specific Migration of BPA, 2nd contact on sample #14181; results in mg/kg

lab	method	value	mark	z(targ)	conventional factor	remarks
310		-----		-----		
330		-----		-----		
362	INH-537	1.52		-----		reported in mg/L
623	EN13130-13	1.58		-----		reported in mg/L
2115	EN13130-1/13	0.5191		-----		
2129	CEN/TS13130-13/EN13130-1	1.62		-----	used conventional factor 6	
2156	EN13130-13	0.73		-----		
2165	EN13130-13	1.70		-----		
2172	EN13130-13	1.69		-----		reported in mg/L
2184	EN13130-13	1.62		-----		reported in mg/L
2190	EN13130-13	0.93		-----		reported in mg/L
2196	EN13130-13	0.16		-----		reported in mg/L
2215	EN13130-13	1.62		-----	used conventional factor 6	
2217	in house	33.72		-----	used conventional factor 6	
2229		-----		-----		
2241	EN13130-13	1.62		-----	used conventional factor 6	
2256	EN13130-13	1.60		-----		reported in mg/L
2271	EN13130-13	1.79		-----		reported in mg/L
2284	EN13130-13	1.57		-----		reported in mg/L
2309		-----		-----		
2353	EN13130-13	1.85		-----		reported in mg/L
2372	EN13130-13	7.92		-----	used conventional factor 6	
2386	EN13130-1/in house	2.09		-----		reported in mg/L
2391		-----		-----		
2403		-----		-----		
2441	EN13130-13	1.23		-----		reported in mg/L
2495		-----		-----		
2497	EN13130-13	0.291		-----		
2504	EN13130-13	2.32		-----		
2510	EN13130-1	2.04		-----	used conventional factor 6	
2551	EN13130-13	3.5		-----		reported in mg/L
2594	EN13130-13	2.47		-----		
2599		-----		-----		
2616	CEN/TS13130-13	1.935		-----	used conventional factor 6	
2627	EN13130-13	0.12		-----		reported in mg/L
3100	EN13130-13	1.76		-----		reported in mg/L
3146	EN13130-1/in house	0.48		-----	used conventional factor 6	
3151	EN13130-13	1.47		-----		
3153		-----		-----		
3172	EN13130-13	3.53		-----		
3182	EN13130-13	1.38		-----	used conventional factor 6	
3185	EN13130-13	1.61		-----		reported in mg/L
3209	EN13130-13	1.77		-----		reported in mg/L
3218	EN13130-13	2.25		-----		reported in mg/L
3225	EN13130-13	0.59		-----		reported in mg/L
3228	EN13130-13	1.82		-----		reported in mg/L
3233	in house	3.53		-----		reported in mg/L
3246	EN13130-13	2.62		-----		reported in mg/L

Specific Migration of BPA, 3rd contact on sample #14181; results in mg/dm²

lab	method	value	mark	z(targ)	remarks
310		-----		-----	
330		-----		-----	
362	INH-537	0.11		-2.08	
623	EN13130-13	0.198		0.16	
2115	EN13130-1/13	0.070		-3.10	
2129	CEN/TS13130-13/EN13130-1	0.28		2.24	
2156	EN13130-13	0.15	E, ex	-1.06	see §4.1, iis calculated 0.185
2165	EN13130-13	0.16		-0.81	
2172	EN13130-13	0.16		-0.81	
2184	EN13130-13	0.156		-0.91	
2190	EN13130-13	0.074		-2.99	
2196	EN13130-13	0.034		-4.01	
2215	EN13130-13	0.17		-0.56	
2217	in house	4.78	R(0.01)	116.56	
2229		-----		-----	
2241	EN13130-13	0.21		0.46	
2256	EN13130-13	0.16		-0.81	
2271	EN13130-13	0.21		0.46	
2284	EN13130-13	0.16		-0.81	
2309		-----		-----	
2353	EN13130-13	0.18		-0.30	
2372	EN13130-13	2.09	C, R(0.01)	48.22	first reported 0.86 mg/dm²
2386	EN13130-1/in house	0.27		1.98	
2391		-----		-----	
2403		-----		-----	
2441	EN13130-13	0.14		-1.32	
2495		-----		-----	
2497	EN13130-13	0.046	E, ex, C	-3.71	first reported 0.023 mg/dm², iis calculated 0.0.036
2504	EN13130-13	0.37		4.53	
2510	EN13130-1	0.24		1.22	
2551	EN13130-13	0.50	C	7.83	first reported 0.94586 mg/dm²
2594	EN13130-13	0.249		1.45	
2599		-----		-----	
2616	CEN/TS13130-13	0.243		1.30	
2627	EN13130-13	0.02		-4.37	
3100	EN13130-13	0.20		0.21	
3146	EN13130-1/in house	0.10		-2.33	
3151	EN13130-13	0.16	E, ex	-0.81	see §4.1, iis calculated 0.112
3153		-----		-----	
3172	EN13130-13	0.26	C	1.73	first reported 0.38 mg/dm²
3182	EN13130-13	0.13		-1.57	
3185	EN13130-13	0.18		-0.30	
3209	EN13130-13	0.16		-0.81	
3218	EN13130-13	0.20		0.21	
3225	EN13130-13	0.08		-2.84	
3228	EN13130-13	0.16		-0.81	
3233	in house	0.37		4.53	
3246	EN13130-13	0.32		3.25	

normality	suspect		
n	34		
outliers	2	+3 excl.	
mean (n)	0.1919		
st.dev. (n)	0.09968		
R(calc.)	0.2791		
R(Horwitz)	0.1102		
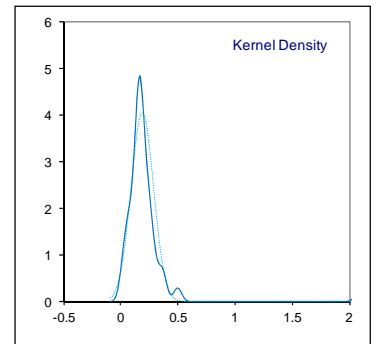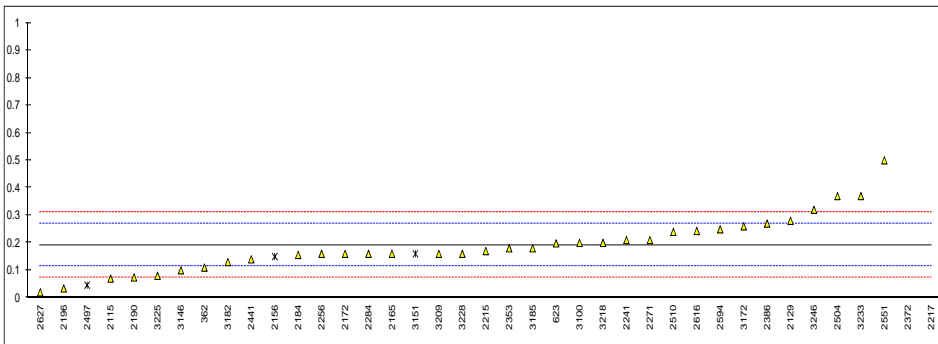
Specific Migration of BPA, 3rd contact on sample #14181; results in mg/kg

lab	method	value	mark	z(targ)	conventional factor	remarks
310		-----		-----		
330		-----		-----		
362	INH-537	1.26		-----		reported in mg/L
623	EN13130-13	1.19		-----		reported in mg/L
2115	EN13130-1/13	0.226		-----		
2129	CEN/TS13130-13/EN13130-1	1.68		-----	used conventional factor 6	
2156	EN13130-13	0.59		-----		
2165	EN13130-13	0.95		-----		
2172	EN13130-13	0.97		-----		reported in mg/L
2184	EN13130-13	0.94		-----		reported in mg/L
2190	EN13130-13	0.74		-----		reported in mg/L
2196	EN13130-13	0.20		-----		reported in mg/L
2215	EN13130-13	0.99		-----	used conventional factor 6	
2217	in house	28.68		-----	used conventional factor 6	
2229		-----		-----		
2241	EN13130-13	1.28		-----	used conventional factor 6	
2256	EN13130-13	0.93		-----		reported in mg/L
2271	EN13130-13	1.24		-----		reported in mg/L
2284	EN13130-13	0.96		-----		reported in mg/L
2309		-----		-----		
2353	EN13130-13	1.07		-----		reported in mg/L
2372	EN13130-13	12.54		-----	used conventional factor 6	
2386	EN13130-1/in house	1.60		-----		reported in mg/L
2391		-----		-----		
2403		-----		-----		
2441	EN13130-13	0.82		-----		reported in mg/L
2495		-----		-----		
2497	EN13130-13	0.276		-----		
2504	EN13130-13	2.20		-----		
2510	EN13130-1	1.44		-----	used conventional factor 6	
2551	EN13130-13	3.9		-----		reported in mg/L
2594	EN13130-13	1.50		-----		
2599		-----		-----		
2616	CEN/TS13130-13	1.459		-----	used conventional factor 6	
2627	EN13130-13	0.08		-----		reported in mg/L
3100	EN13130-13	1.21		-----		reported in mg/L
3146	EN13130-1/in house	0.57		-----	used conventional factor 6	
3151	EN13130-13	0.95		-----		
3153		-----		-----		
3172	EN13130-13	2.46		-----		
3182	EN13130-13	0.81		-----	used conventional factor 6	
3185	EN13130-13	1.10		-----		reported in mg/L
3209	EN13130-13	1.00		-----		reported in mg/L
3218	EN13130-13	1.20		-----		reported in mg/L
3225	EN13130-13	0.50		-----		reported in mg/L
3228	EN13130-13	0.95		-----		reported in mg/L
3233	in house	2.25		-----		reported in mg/L
3246	EN13130-13	1.97		-----		reported in mg/L

APPENDIX 2

Details reported by the participating laboratories

lab	first migration step				second migration step				third migration step			
	ml	surf	ratio	BPA	ml	surf	ratio	BPA	ml	surf	ratio	BPA
310	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
330	100	0.6	166.7	4.30	-----	-----	-----	-----	-----	-----	-----	-----
362	130	1.56	83.3	4.30	130	1.56	83.3	1.52	130	1.56	83.3	1.26
623	265	1.59	166.7	2.85	265	1.59	166.7	1.58	265	1.59	166.7	1.19
2115	250	0.8085	309.2	0.3396	250	0.8085	309.2	0.5195	250	0.8085	309.2	0.227
2129	100	1.6	62.5	8.2	100	1.6	62.5	4.28	100	1.6	62.5	4.49
2156	500	1.571	318.3	0.85	500	1.571	318.3	0.71	400	1.571	318.3	0.58
2165	260	1.57	165.6	2.44	260	1.57	165.6	1.71	260	1.57	165.6	0.96
2172	262	1.57	166.9	3.22	262	1.57	166.9	1.69	262	1.57	166.9	0.97
2184	260	1.57	165.6	2.26	260	1.57	165.6	1.62	260	1.57	165.6	0.94
2190	157	1.57	100.0	1.01	157	1.57	100.0	0.93	157	1.57	100.0	0.74
2196	134	0.8	167.5	0.19	134	0.8	167.5	0.16	134	0.8	167.5	0.20
2215	272	1.63	166.9	3.48	272	1.63	166.9	1.62	272	1.63	166.9	0.99
2217	100	1.57	63.7	195.50	100	1.57	63.7	88.26	100	1.57	63.7	75.11
2229	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2241	65	0.39	166.7	4.38	65	0.39	166.7	1.62	65	0.39	166.7	1.28
2256	260	1.58	164.6	3.43	260	1.58	164.6	1.60	260	1.58	164.6	0.93
2271	265	1.586	167.1	4.39	265	1.586	167.1	1.79	265	1.586	167.1	1.24
2284	262	1.57	166.9	3.31	262	1.57	166.9	1.57	262	1.57	166.9	0.96
2309	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2353	262	1.5708	166.8	3.07	262	1.5708	166.8	1.85	262	1.5708	166.8	1.07
2372	30	0.1884	159.2	5.29	30	0.1884	159.2	5.40	30	0.1884	159.2	5.45
2386	130	0.78	166.7	4.87	130	0.78	166.7	2.09	130	0.78	166.7	1.60
2391	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2403	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2441	262	1.57	166.9	1.99	262	1.57	166.9	1.23	262	1.57	166.9	0.82
2495	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2497	20	1.57	12.7	15.07	20	1.57	12.7	3.05	20	1.57	12.7	2.79
2504	163	1.63	100.0	7.98	163	1.63	100.0	3.86	163	1.63	100.0	3.67
2510	550	1.64	335.4	2.09	527	1.64	321.3	1.06	542	1.64	321.3	0.74
2551	100	0.785	127.4	0.0461	100	0.785	127.4	3.88116	100	0.785	127.4	7.425
2594	261	1.5708	166.2	5.64	261	1.5708	166.2	2.48	261	1.5708	166.2	1.50
2599	150	1.57	95.5	4.47	-----	-----	-----	-----	-----	-----	-----	-----
2616	374	1.626	230.0	2.957	368	1.626	226.3	1.425	370	1.626	226.3	1.069
2627	100	0.41	243.9	0.21	100	0.41	243.9	0.12	100	0.41	243.9	0.08
3100	67	0.40	167.5	3.62	67	0.40	167.5	1.76	67	0.40	167.5	1.21
3146	500	1.57	318.5	2.40	500	1.57	318.5	0.25	500	1.57	318.5	0.30
3151	30	0.223	134.5	2.56	30	0.223	134.5	1.28	30	0.223	134.5	0.83
3153	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3172	159	1.59	100.0	5.07	159	1.59	100.0	2.58	159	1.59	100.0	3.71
3182	160	1.6	100.0	4.60	160	1.6	100.0	2.29	160	1.6	100.0	1.35
3185	67	0.40	167.5	3.84	67	0.40	167.5	1.61	67	0.40	167.5	1.10
3209	260	1.57	165.6	3.46	260	1.57	165.6	1.77	260	1.57	165.6	1.00
3218	67	0.4	167.5	3.49	67	0.4	167.5	2.25	67	0.4	167.5	1.20
3225	32	0.19	168.4	2.98	32	0.19	168.4	0.59	32	0.19	168.4	0.50
3228	260	1.57	165.6	2.18	260	1.57	165.6	1.82	260	1.57	165.6	0.95
3233	260	1.57	165.6	3.18	260	1.57	165.6	3.53	260	1.57	165.6	2.25
3246	130	0.785	165.6	4.06	130	0.785	165.6	2.62	130	0.785	165.6	1.97

The abbreviations used in above table are as follows:

ml = simulant used in the nth migration step in millilitres

surf = contact surface used in the nth migration step in dm^2

ratio = amount of simulant used in the nth migration step per dm^2 surface in ml/dm^2

BPA = BPA concentration measured after the nth migration step in mg/l

APPENDIX 3

Number of participating laboratories per country

  1 lab in BULGARIA

  3 labs in FRANCE

  4 labs in GERMANY

  4 labs in HONG KONG

  1 lab in HUNGARY

  1 lab in INDIA

  1 lab in INDONESIA

  1 lab in IRELAND

  5 labs in ITALY

  1 lab in MALAYSIA

18 labs in P.R. of CHINA

  1 lab in SAUDI ARABIA

  1 lab in SWEDEN

  1 lab in TAIWAN R.O.C.

  2 labs in THAILAND

  1 lab in THE NETHERLANDS

  1 lab in VIETNAM

APPENDIX 4

Abbreviations:

C	= final result after checking of first reported suspect result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner outlier test
R(0.05)	= straggler in Rosner outlier test
n.a.	= not applicable
E	= possible calculation error
W	= result was withdrawn

Literature:

1   iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, April 2014

2   EN13130-1 Materials and articles in contact with foodstuffs – Plastics substances subject to limitation - Part 1: Guide to test methods for the specific migration of substances from plastics to foods and food simulants and the determination of substances in plastics and the selection of conditions of exposure to food simulants

3   CEN/TS 13130-13 Materials and articles in contact with foodstuffs – Plastics substances subject to limitation - Part 13: Determination of 2,2-bis(4-hydroxyphenyl)propane (Bisphenol A) in food simulants

4   ASTM E178-02

5   ASTM E1301-03

6   ISO 5725-86

7   ISO 5725, parts 1-6, 1994

8   M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)

9   W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)

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12   P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)

13   J.N. Miller, Analyst, 118, 455, (1993)

14   Analytical Methods Committee Technical Brief, No4 January 2001

15   The Royal Society of Chemistry 2002, Analyst 2002, 127 page1359-1364, P.J. Lowthian and M. Thompson. (see http://www.rsc.org/suppdata/an/b2/b205600n/)

16   R.G. Visser, Reliability of proficiency test results for metals and phthalates in plastics, Accred Qual Assur, 14:29-34 (2009)

17   EUR 24815 EN 2011 - Technical guidelines on testing the migration of primary aromatic amines from polyamide kitchenware and of BPA from melamine kitchenware, JRC (2011)

18   Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), pp. 165-172, (1983)