Institute for
Interlaboratory Studies

Results of Proficiency Test
Overall Migration on
Food Contact Materials
October 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

During the contact of materials with food, molecules can migrate from the food contact material to the food. Because of this, in many countries regulations are made to ensure food safety. The framework Regulation (EU) No. 10/2011 (lit. 13 and lit. 14) 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. Article 12 of this regulation describes the Overall Migration limit, which is 10 mg/dm^2. Only when determined for food contact intended for infants and children, the Overall Migration is expressed in mg/kg food simulant with a limit of 60 mg/kg food simulant. 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.
In September 2020 the 15th amendment of this EU 10/2011 (lit. 15) was published. This amendment especially describes methods for repeated use articles, how to test and to reject them.

Since 2012 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Overall Migration on Food Contact Materials every year. During the annual proficiency testing program 2022/2023 it was decided to continue the proficiency test for the determination of Overall Migration on Food Contact Materials.

In this interlaboratory study 50 laboratories in 19 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the Overall Migration 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/IEC17025 accredited laboratory.
It was decided to send one sample, a set of three identical items, labelled #22715 positive on Overall Migration.
The participants were requested 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/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under 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 organization 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). 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 black polypropylene salad bowls for single use was selected. The salad bowls were positive for Overall Migration. Randomly from the batch 70 sets of three salad bowls were put into a bag and labelled #22715.
The homogeneity of the subsamples was checked by determination of Overall Migration in accordance with to GB31604.8 on three sets of three stratified randomly selected subsamples with the following conditions: article filling, 3% M/V Acetic Acid, 48 hours at 70°C.

	Overall Migration mg/dm^2	Overall Migration, average per set mg/dm^2
sample #22715-1	10.69	11.43
sample #22715-2	11.50	
sample #22715-3	12.08	
sample #22715-4	11.41	10.53
sample #22715-5	9.62	
sample #22715-6	10.57	
sample #22715-7	11.66	11.32
sample #22715-8	11.26	
sample #22715-9	11.04	

Table 1: homogeneity test results of subsamples #22715

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Overall Migration, average per set mg/dm^2
r (observed)	1.37
reference test method	EN1186-9:02
0.3 * R (reference test method)	1.61

Table 2: evaluation of the repeatability of subsamples #22715

The calculated repeatability is in agreement with 0.3 times the estimated reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample, a set of three salad bowls, labelled #22715 was sent on September 7, 2022.

2.5 ANALYZES

The participants were requested to determine Overall Migration using the prescribed test conditions (article filling, single use and 3% M/V Acetic Acid as simulant for 48 hours at 70 °C). Each participant received three salad bowls to be tested separately, where also the average of the three tests was requested.
It was also requested to report if the laboratory was accredited for this test and to report some analytical details.

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’ test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) 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 appendices 1 and 2 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 reanalyzes). Additional or

corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendices 1 and 2. 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 organization 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.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. 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 visualize 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. This 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

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 (derived from e.g. ISO or ASTM test methods), 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 case no literature reproducibility was available, other target values were 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 in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated 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. 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 proficiency test some problems were encountered with the dispatch of the samples. Two participants reported test results after the final reporting date and four other participants did not report any test results. Not all participants were able to report all tests requested. In total 46 participants reported 46 numerical test results for average Overall Migration per contact surface. Observed were 2 outlying test results, which is 4.3%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

The data set did not prove to have a normal Gaussian distribution and is referred to as “suspect”. The statistical evaluation of this data set should be used with due care, see also paragraph 3.1.

4.1 EVALUATION OF THE TEST RESULTS

In this section the reported test results are discussed. The test methods 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 tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 5.

In the past iis has observed that for the Overall and Specific Migration methods the limits and the calculations are mixed up by participants. Therefore, iis issued a White Paper on this subject in February 2018 (see lit. 16) to help participants understand the differences between the two methods, the units used for reporting and the regulated limits.

For the determination of Overall Migration (also called Global or Total Migration) on food contact material by article filling, the EN1186 method is considered to be the official EC test method. In method EN1186-9:02 it is described that five samples are needed: two samples to determine the surface area and three sample for the migration test. In this 2022 PT only a set of three samples (salad bowls) were available for both surface area determination and the migration test.

The target reproducibility used for statistical evaluation was estimated from method EN1186-9:02 (Annex A) reproducibility of simulants A, B and C (based on 3 replicates). Medio August 2022 the method EN1186-9:02 was superseded with method EN1186-3:2022 “Materials and articles in contact with foodstuffs – Plastics – Part 3: Test methods for overall migration in evaporable simulants”. The target reproducibility mentioned in EN1186-9:02 has remained the same in EN1186-3:2022. Therefore, method EN1186-9:02 is still used as reference method in this PT. The PT started in September and presumable the participants are not yet transferred to EN1186-3:2022 already.

Overall Migration: This determination may be problematic for a number of laboratories. Two statistical outliers were observed and ten other test results were excluded. The calculated reproducibility after rejection of the suspect data is in full agreement with the target reproducibility estimated from EN1186-9:02.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as 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 derived from the reference method is presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Overall Migration	mg/dm^2	34	13.33	6.13	6.43

Table 3: reproducibility of tests on sample #22715

Without further statistical calculations it can be concluded that for Overall Migration per contact surface there is a good compliance of the group of participating laboratories with the target reproducibility estimated from EN1186-9:02.

4.3 COMPARISON OF THE PROFICIENCY TEST OF OCTOBER 2022 WITH PREVIOUS PTS

	October 2022	October 2021	October 2020	October 2019	October 2018
Number of reporting laboratories	46	44	46	49	45
Number of test results	46	131	45	122	133
Number of statistical outliers	2	7	2	5	39
Percentage of statistical outliers	4.3%	5.3%	4.4%	4.1%	29%

Table 4: comparison with previous proficiency tests

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

The performance of the determinations of the proficiency test was compared to uncertainties observed in PTs over the years, expressed as relative standard deviation (RSD) of the PTS, see next table.

year	article filling	total immersion	# of items	EN1186:02
2013	-----	25-30%	2	11% (part 3)
2014	18%	-----	3	17% (part 8)
2015	14%	-----	3	8% (part 9)
2016	17%	29%	3 – 1	8% (part 9) – 13% (part 3)
2017	-----	32-36%	1	17% (part 3)
2018	13-17%	-----	1	17% (part 9)
2019	-----	16-22%	1	17% (part 3)
2020	-----	19%	3	17% (part 3)
2021	13-21%	-----	1	17% (part 9)
2022	16%	-----	3	17% (part 9)

Table 5: development of the uncertainties over the years

The uncertainty observed in this PT is in line with the uncertainties observed in previous PTs for article filling.

Sample #22715 was also used in a previous iis PT as sample #15180 in the PT iis15P09GM. The result of this 2022 PT (sample #22715) is in line with the result of the 2015 PT (sample #15180)

	unit	sample #22715			sample #15180		
		n	average	R(calc)	n	average	R(calc)
Overall Migration	mg/dm^2	34	13.33	6.13	35	12.63	4.90

Table 6: comparison of sample #22715 with #15180

4.4 EVALUATION OF THE ANALYTICAL DETAILS

The reported details of the determination of Overall Migration per contact surface area of each bowl and the reported analytical details that were used by the participants are listed in appendices 2 and 3. Based on the reported details the following is observed:

- About 85% of the reporting participants mentioned to have used test method EN1186-9. From the reporting participants about 85% mentioned that they are accredited for this test.
- About 75% of the participants reported not to clean the sample and a few participants used a cloth or brush to clean the sample prior to the migration step. A few participants reported to have used water and/or a detergent/soap to clean the sample prior to use. Method EN1186-9:02 states in paragraph 6.1: “under no circumstances wash the sample with water or solvent”. However, in general can be concluded that it appears that these cleansing methods have a negligible effect on the Overall Migration in mg/dm^2.
- Almost all participants reported to have heated the simulant to 70 °C before the sample was filled with simulant.
- The amount of simulant used by each participant varied from 100 – 1450 mL. In test method EN1186-9:02 is mentioned that a specimen should be filled to within 0.5 cm from the top. This should lead to a large volume of simulant and consequently also a large contact surface.
- Looking at the test item, a salad bowl, with a relatively large round bottom, rounded corners and only near the top almost square with a distinctive rim, it is obvious that using a lower simulant volume it will result in a different volume to surface ratio than using a large simulant volume. Therefore, it was decided to exclude for statistical evaluation the test results of participants which used a simulant volume smaller than 1000 mL.
- When the bowl was filled to the top edge, a volume of 1500 mL was found. In order to cover the bowl during the test (to avoid dust particles falling in and to prevent evaporation of the simulant), the test item should not be filled to the top edge.
- iis measured the maximum volume to 0.5 cm below the top for this sample as 1350 mL. Based on a maximum volume of 1350 mL simulant the total surface area of the bowl could be estimated to be 5.85 dm^2. Therefore, it was decided to exclude test results for statistical evaluation with a reported contact surface area below 4.5 or above 6.2 dm^2.
- None of the participants reported a volume above 1450 mL. In this PT none of the test results were excluded for using a too large volume.

- After exposure of the salad bowl to the simulant for the selected time, the simulant must be evaporated to low volume. About 85% of the reported participants directly evaporated the simulant from a dish (evaporation method) and about 10% first distilled the simulant before further evaporation (distillation method).
- The reported time needed for evaporation of the simulant to low volume varied from 40 minutes to 24 hours. The reported temperature varied from 70 °C to 300 °C.

As the calculated reproducibility of the Overall Migration is in full agreement with the requirements of the target reproducibility, no separate statistical analysis on the evaporation temperature and time has been performed.

One participant reported the test results of Overall Migration per contact surface in mg/kg instead of the requested mg/dm^2. The test result of this participant was excluded for statistical calculations.

5 DISCUSSION

In this PT a set of three identical salad bowls were sent to each participant. The surface area of the bowls could be determined using one test item before the start of the migration test, but the area could also be determined on a bowl after finishing the migration test. The contact surface used as reported by the participants varies from 1.67 – 10.7728 dm^2. A large variation is observed when the reported surface area is compared to the used simulant volume (see figure 1). Very different surface areas were reported for the same used volumes.

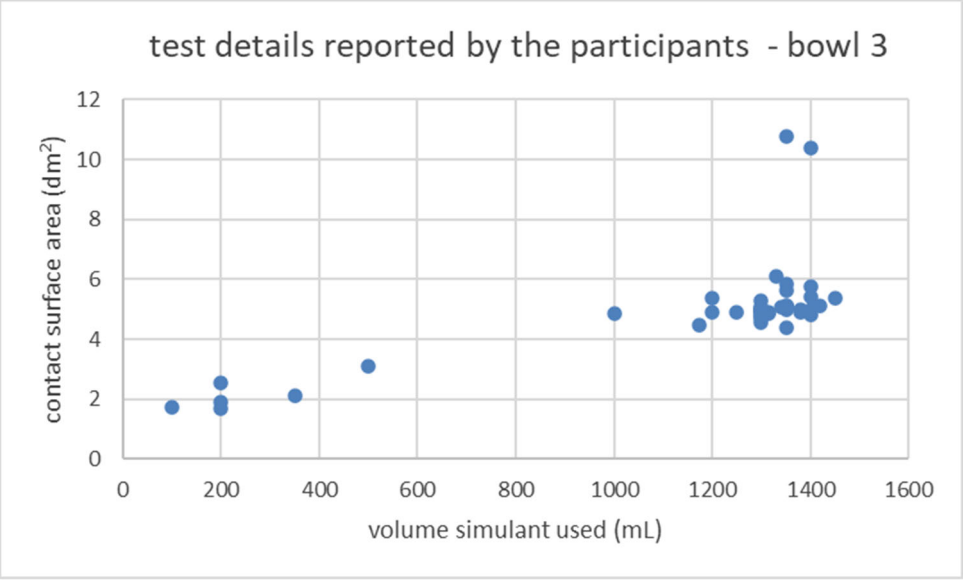


Figure 1: reported surface area versus volume of simulant used by participants

The test item has an edge about 0.5 cm below the top, which according to the method, would be the maximum level to fill the test item. The maximum surface area to 0.5 cm below the top for sample #22715 was determined by iis to be approximately 5.85 dm^2. This was done in two ways. First, by measurement with a ruler and the approximation that some rounded parts are squares/triangles. The second way was cutting of the 0.5 cm top edge of the bowl, weighing the remaining bowl, cutting a square sample out of the bowl and determination of the weight/surface ratio.

The EU regulation number 10/2011 describes in article 12 that the limit for Overall Migration is 10 mg/dm^2. According to this limit almost all of the reporting participants would have rejected sample #22715 and three participants would have accepted sample #22715.

6 CONCLUSION

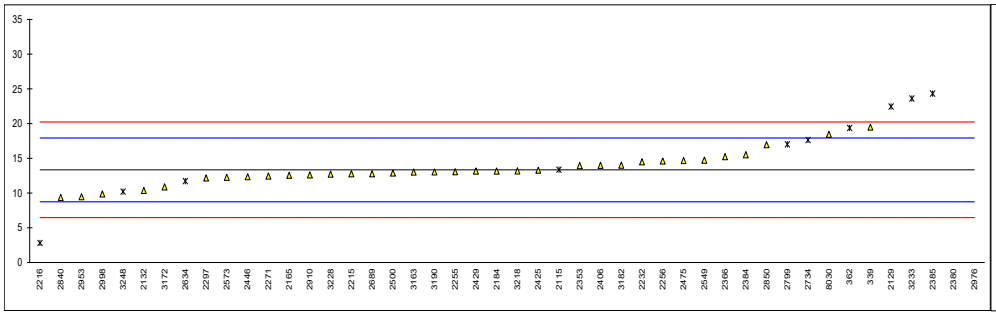
It is to be expected that the variation of the migration test results in real life practice will be larger than observed in this PT as the test conditions like time, temperature, etc. will not be prescribed but will be selected by the individual laboratories.

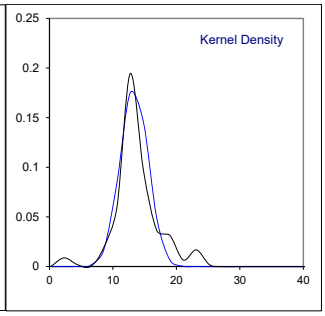
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1

Average Overall Migration (per contact surface) on sample #22715; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
310		-----		-----	
339	EN1186-9	19.47842056		2.68	
362	EN1186-9	19.36	ex	2.63	excl. due to low volume and contact surface area, see §4.4
551		-----		-----	
2115	EN1186-9	13.35	ex	0.01	excl. due to low volume and contact surface area, see §4.4
2129	EN1186-9	22.444	ex	3.97	excl. due to low volume and contact surface area, see §4.4
2132	EN1186-9	10.37		-1.29	
2165	EN1186-9/GB31604.8	12.5475		-0.34	
2184	EN1186-9	13.152		-0.08	
2215	EN1186-9	12.756		-0.25	
2216	In house	2.8	ex	-4.59	excluded due to large contact surface area, see §4.4
2232	EN1186-9	14.481		0.50	
2255	EN1186-9	13.05		-0.12	
2256	EN1186-9	14.59		0.55	
2271	EN1186-9	12.43		-0.39	
2297	EN1186-9	12.164		-0.51	
2320		-----		-----	
2353	EN1186-9	13.94		0.27	
2366	EN1186-9	15.23		0.83	
2380	EN1186-9	481.21	C,R(0.01)	203.77	first reported 200.79
2384	EN1186-9	15.5		0.95	
2385	EN1186-9	24.3	R(0.01)	4.78	samples deformed under heat due to the glass plate
2406	EN1186-9	13.988		0.29	
2425	EN1186-9	13.28		-0.02	
2429	EN1186-9	13.15		-0.08	
2446	EN1186-9	12.33		-0.43	
2475	EN1186	14.673		0.59	
2500	EN1186-9	12.87		-0.20	
2549	EN1186-9	14.721		0.61	
2573	EN1186-9	12.253		-0.47	
2634	EN1186-9	11.7	ex	-0.71	excl. due to low volume and contact surface area, see §4.4
2689	EN1186-9	12.763		-0.25	
2703		-----		-----	
2734	EN1186	17.613	ex	1.87	excl. due to low volume and contact surface area, see §4.4
2799	EN1186-9	17	ex	1.60	excluded as test results is reported in mg/kg, , see §4.4
2840	EN1186-1	9.35	C	-1.73	reported 7.606, average was calculated by iis
2850	EN1186-9	16.95		1.58	
2910	EN1186-9	12.593		-0.32	
2953	EN1186-9	9.446	C	-1.69	first reported 94.43
2976	EN1186-9	529.18	ex	224.66	excluded due to low contact surface area, see §4.4
2998	In house	9.85	C	-1.51	first reported 4.79
3163	EN1186-9	13.0		-0.14	
3172	EN1186-9	10.8805		-1.07	
3182	EN1186-9	14.00		0.29	
3190	EN1186-9	13.032		-0.13	
3218	EN1186-9	13.181		-0.06	
3228	EN1186-9	12.72		-0.27	
3233	EN1186-9	23.576	ex	4.46	excl. due to low volume and contact surface area, see §4.4
3248	EN1186-9	10.2	ex,C	-1.36	excl due to large contact surface area, see §4.4, f.r. 4.73
8030	EN1186-9	18.45		2.23	
	normality	suspect			
	n	34			
	outliers	2 (+ 10ex)			
	mean (n)	13.329			
	st.dev. (n)	2.1897	RSD = 16%		
	R(calc.)	6.131			
	st.dev.(EN1186-9:02)	2.2961			
	R(EN1186-9:02)	6.429			





APPENDIX 2

Details on reported intermediate test results on sample #22715: salad bowl 1

lab	total residue (mg)		surface area (dm^2)		volume simulant (ml)	Overall Migration (mg/dm^2)		remarks
310	-----		-----		-----	-----		
339	17.6		4.49		1174	21.09081849		
362	32.5		1.67		200	19.46		
551	-----		-----		-----	-----		
2115	29.8		2.53		200	11.78		
2129	7.4		2.3		280	24.667		
2132	8.7		5.76		1400	10.57		
2165	62.5		5.09		1350	12.2790		
2184	70.60		5.08		1350	13.897		
2215	69.16		5.4		1400	12.807		
2216	0.029		10.7728		1350	2.7		
2232	77.8		5.612		1350	13.863		
2255	64.211		4.920		1315	13.051		
2256	12		5.27		1300	14.80		
2271	64.1		5.10		1350	12.57		
2297	65.1		5.36		1450	12.146		
2320	-----		-----		-----	-----		
2353	9.68		4.8980		1380	13.66		
2366	76.3		5.01		1380	15.23		
2380	0.1381		4.833		1400	484.51	C	first reported 200.020 mg/dm^2
2384	6.1		5.357		1200	13.7		
2385	121.1	C	5		1400	24.2		first reported 17.3 mg
2406	71		5.1264		1420	13.850		
2425	65.91		4.96		1300	13.29		
2429	65.5		4.95		1300.0	13.23		
2446	80.8		5.062		1340	15.96		
2475	11.4		4.87		1315	14.99		
2500	60.6		4.7		1300	12.89		
2549	75.1		4.95		1300	15.172		
2573	612		5.03		1300	12.167		
2634	33.9		1.75		100	19.4		
2689	61.3		4.9		1200	12.510		
2703	-----		-----		-----	-----		
2734	54.6		3.1		500	17.613		
2799	0.0177		-----		1000	16.5		reported overall migration in mg/kg
2840	45.700		4.88	C	1000	9.36	C	first reported 6 dm^2 and 7.62 mg/dm^2
2850	78.5		4.58		1300	17.14		
2910	63.4		5.096		1350	12.431		
2953	551.1		6.121		1330	9.03	C	first reported 90.03 mg/dm^2
2976	2261.25		4.37		1350	517.44		
2998	50.9		5.0	C	1300	10.18	C	first reported 10.28 dm^2 and 4.95 mg/dm^2
3163	51		5		1350	13.8		
3172	63.7		5.83		1350	10.9262		
3182	11.2		5.09		1300.00	14.30		
3190	0.0625		4.76		1300	13.109		
3218	64.30		4.800		1300	13.406		
3228	65.5		5.06		1350	12.94		
3233	23.3		1.92		200	24.270		
3248	0.0502		10.4		1400	10.4	C	first reported 4.83 mg/dm^2
8030	94.28		4.89		1250	19.28		

Details on reported intermediate test results on sample #22715: salad bowl 2

lab	total residue (mg)	surface area (dm^2)	volume simulant (ml)	Overall Migration (mg/dm^2)	remarks
310	-----	-----	-----	-----	
339	16.6	4.49	1174	19.78346882	
362	34.30	1.67	200	20.54	
551	-----	-----	-----	-----	
2115	33.5	2.53	200	13.24	
2129	6.3	2.1	350	21.000	
2132	8.5	5.76	1400	10.33	
2165	64.2	5.09	1350	12.6130	
2184	65.40	5.08	1350	12.874	
2215	72	5.4	1400	13.333	
2216	0.0286	10.7728	1350	2.7	
2232	80.4	5.612	1350	14.326	
2255	64.201	4.920	1315	13.048	
2256	11.8	5.27	1300	14.55	
2271	62.1	5.11	1350	12.15	
2297	64.8	5.36	1450	12.089	
2320	-----	-----	-----	-----	
2353	9.93	4.8980	1380	14.02	
2366	74.7	5.01	1380	14.91	
2380	0.1384	4.833	1400	481.10 C	first reported 200.46 mg/dm^2
2384	7.2	5.357	1200	16.1	
2385	126.0 C	5	1400	25.2	first reported 18.0 mg
2406	71.71	5.1264	1420	13.988	
2425	66.73	4.96	1300	13.45	
2429	64.45	4.95	1300.0	13.02	
2446	54.2	5.062	1340	10.71	
2475	10.7	4.87	1315	14.04	
2500	62.1	4.7	1300	13.21	
2549	73	4.95	1300	14.747	
2573	619	5.03	1300	12.306	
2634	11.4	1.75	100	6.5	
2689	62.7	4.9	1200	12.796	
2703	-----	-----	-----	-----	
2734	55.5	3.1	500	17.903	
2799	0.0182	-----	1000	17	reported overall migration in mg/kg
2840	47.400	4.88 C	1000	9.71 C	first reported 6 dm^2 and 7.90 mg/dm^2
2850	74.9	4.58	1300	16.36	
2910	64.0	5.096	1350	12.549	
2953	626.1	6.121	1330	10.228 C	first reported 102.28 mg/dm^2
2976	2328.75	4.37	1350	532.89	
2998	47.7	5.0	1300	9.54 C	first reported 10.28 dm^2 and 4.64 mg/dm^2
3163	29	5	1350	7.9	
3172	62.7	5.83	1350	10.7547	
3182	10.8	5.09	1300.00	13.79	
3190	0.0613	4.76	1300	12.857	
3218	63.70	4.800	1300	13.271	
3228	64.8	5.06	1350	12.67	
3233	20.0	1.92	200	20.833	
3248	0.0496	10.4	1400	10.3 C	first reported 4.77 mg/dm^2
8030	87.58	4.89	1250	17.91	

Details on reported intermediate test results on sample #22715: salad bowl 3

lab	total residue (mg)	surface area (dm^2)	volume simulant (ml)	Overall Migration (mg/dm^2)	remarks
310	-----	-----	-----	-----	
339	14.9	4.49	1174	17.56097439	
362	30.2	1.67	200	18.08	
551	-----	-----	-----	-----	
2115	38.0	2.53	200	15.02	
2129	6.5	2.1	350	21.667	
2132	8.4	5.76	1400	10.21	
2165	64.9	5.09	1350	12.7505	
2184	64.60	5.08	1350	12.716	
2215	65.48	5.4	1400	12.126	
2216	0.0314	10.7728	1350	2.9	
2232	85.6	5.612	1350	15.253	
2255	64.1992	4.920	1315	13.048	
2256	11.7	5.27	1300	14.43	
2271	64.5	5.13	1350	12.57	
2297	65.7	5.36	1450	12.257	
2320	-----	-----	-----	-----	
2353	10.01	4.8980	1380	14.13	
2366	77.9	5.01	1380	15.55	
2380	0.1394	4.833	1400	478.03 C	first reported 201.90 mg/dm^2
2384	7.4	5.357	1200	16.6	
2385	116.9 C	5	1400	23.4	first reported 16.7 mg
2406	72.42	5.1264	1420	14.127	
2425	64.92	4.96	1300	13.09	
2429	65.34	4.95	1300.0	13.20	
2446	52.3	5.062	1340	10.33	
2475	11.4	4.87	1315	14.99	
2500	58.8	4.7	1300	12.51	
2549	70.5	4.95	1300	14.242	
2573	618	5.03	1300	12.286	
2634	13.8	1.75	100	9.3	
2689	63.6	4.9	1200	12.979	
2703	-----	-----	-----	-----	
2734	53.7	3.1	500	17.323	
2799	0.0182	-----	1000	17	reported overall migration in mg/kg
2840	43.800	4.88 C	1000	8.98 C	first reported 6 dm^2 and 7.30 mg/dm^2
2850	79.4	4.58	1300	17.34	
2910	65.3	5.096	1350	12.804	
2953	555.8	6.121	1330	9.08 C	first reported 90.8 mg/dm^2
2976	2347.65	4.37	1350	537.22	
2998	49.1	5.0 C	1300	9.82 C	first reported 10.28 dm^2 and 4.78 mg/dm^2
3163	64	5	1350	17.3	
3172	63.9	5.83	1350	10.9605	
3182	10.9	5.09	1300.00	13.92	
3190	0.0626	4.76	1300	13.130	
3218	61.80	4.800	1300	12.865	
3228	63.5	5.06	1350	12.54	
3233	24.6	1.92	200	25.625	
3248	0.0477	10.4	1400	9.92 C	first reported 4.59 mg/dm^2
8030	88.85	4.89	1250	18.17	

APPENDIX 3

Summary of reported analytical details

lab	ISO17025 accredited	sample cleaned prior to migration step	simulant heated to 70 °C	ratio surface/volume (dm²/mL)
310	---	---	---	
339	Yes	No	No	4,49dm²/1174ml
362	Yes	Yes	Yes	1:100
551	---	---	---	
2115	Yes	No	Yes	1:1
2129	Yes	No	Yes	2,3 dm²/280 mL 2,1 dm²/350 mL 2,1 dm²/350 mL
2132	Yes	Yes, with DI	Yes	0.0041
2165	Yes	No	Yes	5.09dm2/1350 mL = 0.00377dm2/mL
2184	Yes	No	Yes	5.08 dm2/1350 ml
2215	Yes	Yes, brushing with a soft brush	Yes	5.4/1400
2216	No	No	Yes	0.008
2232	No	No	Yes	0.0042
2255	No	No	Yes	0.004
2256	Yes	No	Yes	0.0085
2271	Yes	No	Yes	0.00379 dm2/mL
2297	Yes	No	Yes	5.36dm2-1450mL
2320	---	---	---	
2353	Yes	No	Yes	0.0003549
2366	Yes	No	Yes	
2380	Yes	No	Yes	4.833 - 1400
2384	Yes	No	Yes	0.00446
2385	Yes	No	Yes	5/1400
2406	No	Yes, clean with brush	Yes	5.1264 dm2/ 1420 mL
2425	Yes	Yes	Yes	0.004 (5.5 dm2/1300 mL)
2429	Yes	Yes, soft cloth	Yes	S/V=5.00dm2/1300.0mL
2446	Yes	No	Yes	0.00377
2475	Yes	No	Yes	4.87/1315
2500	Yes	No	Yes	4.7dm2/1300mL
2549	Yes	No	Yes	4.95 dm2/1300 ml
2573	Yes	No	Yes	5.03/1300
2634	Yes	No	Yes	1.75DM2/100ML
2689	Yes	Yes	Yes	4.9 dm2/ 1200 mL
2703	---	---	---	
2734	Yes	No	Yes	0.006
2799	Yes	No	Yes	N/A
2840	Yes	No	No	According to EN 13130, 6 dm² corresponds to 1 kg of simulant, which to 1000 ml, so the ratio is 6 dm² /1000 ml.
2850	Yes	No	Yes	0.0035
2910	Yes	No	Yes	5.10dm2/1350ml
2953	Yes	Yes	Yes	
2976	Yes	Yes, washed with detergent in water	Yes	0.003237037
2998	Yes	Yes, washing with soap, rinsing with mains water and distilled water	Yes	0,008
3163	No	No	Yes	5/1350
3172	Yes	No	Yes	0.004319
3182	Yes	No	Yes	(5.09*1300)/200
3190	Yes	No	Yes	4.76 / 1300
3218	Yes	Yes, rinse with distilled water	Yes	4.80dm2 : 1300mL
3228	Yes	No	Yes	0.00375dm2/ml
3233	No	No	Yes	384
3248	Yes	No	Yes	1:1
8030	Yes	No	Yes	0.0039 dm2/ml

Summary of reported analytical details – continued --

lab	simulant evaporated in a dish or first distilled	evaporation time (min)	evaporation temperature (°C)
310	---		
339	Directly evaporated from a dish (Evaporation method)	between 240 and 300 min	95°C
362	Directly evaporated from a dish (Evaporation method)	190	100
551	---		
2115	Directly evaporated from a dish (Evaporation method)	24 h	150 °C
2129	Other, 50 mL removed from the total volume for the evaporation	180 minutes	
2132	Other, place in oven until completely dried	More that 480 minutes	105
2165	Directly evaporated from a dish (Evaporation method)	over night.	105 °C
2184	Directly evaporated from a dish (Evaporation method)	overnight	105°C
2215	First distilled before further evaporation (Distillation method)	4h	100℃
2216	Directly evaporated from a dish (Evaporation method)	180	250
2232	Directly evaporated from a dish (Evaporation method)	480-500 minutes	100
2255	Directly evaporated from a dish (Evaporation method)	4 hrs (approx.)	105-107
2256	Other, took 200mL migration solution to evaporate, and added the ratio back in calculation.	180 mins	100℃
2271	Directly evaporated from a dish (Evaporation method)	2hours	150~280°C
2297	Directly evaporated from a dish (Evaporation method)	480	120
2320	---		
2353	First distilled before further evaporation (Distillation method)	4 hrs	105°C
2366	---		
2380	Directly evaporated from a dish (Evaporation method)	270 Minutes	95 °C
2384	Directly evaporated from a dish (Evaporation method)	240 minutes	220°C
2385	Directly evaporated from a dish (Evaporation method)	over night	105
2406	Directly evaporated from a dish (Evaporation method)	~180 mins	~100 °C
2425	Directly evaporated from a dish (Evaporation method)	4 hours	150°C
2429	Directly evaporated from a dish (Evaporation method)	About 2.5 hours.	270 °C
2446	Directly evaporated from a dish (Evaporation method)	960	ca. 150-225°C
2475	Directly evaporated from a dish (Evaporation method)	1080	105
2500	First distilled before further evaporation (Distillation method)		
2549	Directly evaporated from a dish (Evaporation method)	120 mins	150°C
2573	Directly evaporated from a dish (Evaporation method)	400 minutes	250℃-300℃
2634	Directly evaporated from a dish (Evaporation method)		
2689	Directly evaporated from a dish (Evaporation method)	12 hours	100 ℃
2703	---		
2734	Directly evaporated from a dish (Evaporation method)	40	
2799	Directly evaporated from a dish (Evaporation method)	40-60 minutes	100
2840	Directly evaporated from a dish (Evaporation method)	approx. 9 h	<=82°C
2850	Directly evaporated from a dish (Evaporation method)	7200 minutes	100°C
2910	First distilled before further evaporation (Distillation method)	7h	300°C
2953	Directly evaporated from a dish (Evaporation method)		105
2976	Directly evaporated from a dish (Evaporation method)	12 hours	105
2998	Directly evaporated from a dish (Evaporation method)	1440	105°C
3163	Directly evaporated from a dish (Evaporation method)	200	70
3172	Directly evaporated from a dish (Evaporation method)	1200	70
3182	Directly evaporated from a dish (Evaporation method)	1st piece: 1h 25min 32sec 2nd piece: 1h 21min 15sec 3rd piece: 1h 23min 36sec	98 °C
3190	Directly evaporated from a dish (Evaporation method)	About 500 minutes.	105
3218	Directly evaporated from a dish (Evaporation method)	180 minutes	105°C
3228	Directly evaporated from a dish (Evaporation method)	720 minutes	105
3233	Directly evaporated from a dish (Evaporation method)	1230	105
3248	Directly evaporated from a dish (Evaporation method)	4 hours	100°C
8030	Directly evaporated from a dish (Evaporation method)	1 day	100 C

APPENDIX 4

Number of participants per country

3 labs in BANGLADESH
2 labs in BRAZIL
1 lab in BULGARIA
3 labs in FRANCE
3 labs in GERMANY
5 labs in HONG KONG
1 lab in INDIA
1 lab in ISRAEL
6 labs in ITALY
1 lab in MALAYSIA
14 labs in P.R. of CHINA
1 lab in SINGAPORE
1 lab in SRI LANKA
2 labs in THAILAND
2 labs in THE NETHERLANDS
1 lab in U.S.A.
1 lab in UNITED ARAB EMIRATES
1 lab in UNITED KINGDOM
1 lab in VIETNAM

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	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?

Literature

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 ISO5725:86
3 ISO5725 parts 1-6:94
4 ISO13528:05
5 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
8 J.N. Miller, Analyst, 118, 455, (1993)
9 Analytical Methods Committee, Technical Brief, No 4, January 2001
10 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)
13 Commission regulation (EU) No 10/2011 of January 2011 on plastic materials and articles intended to come into contact with food, published in the official journal of the EU on the 15th of Janary 2011
14 Union Guidelines on Regulation (EU) No 10/2011 on plastic materials and articles intended to come into contact with food, 21st of February 2014, website: ec.europa.eu
15 Commission regulation (EU) No 2020/1245 of September 2020 amending and correcting Regulation (EU) No 10/2011 on plastic materials and articles intended to come into contact with food, published in the official journal of the EU on the 3rd of September 2020
16 iis memo 1801: White paper on the determinations of Overall and Specific migration on food contact materials, February 2018, www.iisnl.com