Results of Proficiency Test
Overall Migration (fcm)
September 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

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 (EC) No. 10/2011 (lit. 18 and lit. 19) 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.

Since 2012, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Overall Migration every year. During the annual proficiency testing program 2019/2020, it was decided to continue with the proficiency test for the determination of Overall Migration on food contact materials.
In this interlaboratory study 44 laboratories from 18 different countries registered for participation. See appendix 4 for the number of participants per country. In this report, the results of the 2019 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 analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send 1 sample, a blue PTFE square plate labelled #19620, positive on Overall Migration. Furthermore, a number of test conditions (type of simulant, exposure time, exposure volume, migration method, simulant volume and details about the contact surface testing) were prescribed. 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

The batch for sample #19620 (a blue PTFE square plate, 10 x 10 x 0.2cm) was used in a previous test of Overall Migration on food contact materials (iis13P05GM). Therefore, homogeneity of the subsamples #19620 was assumed.

To each of the participating laboratories one sample #19620 was sent on October 4, 2019.

2.5 ANALYSES

The participants were requested to determine Overall Migration on sample #19620 using the prescribed test conditions (total immersion, repeated use with double contact surface testing and distilled Water as simulant for 2 hrs. at 70°C.)
In daily practise, not just one item, but more items for testing would have been sent. However, this sample is positive and especially prepared for this proficiency test. This means that one item of the sample is sufficient for the determination of Overall Migration. The participants were requested to report the test results of all three successive migration steps. 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 sample as if it was a routine sample 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 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 appropriate reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

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

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

According to ISO5725 the original test results per determination were submitted to Dixon’s, Grubbs’ and/or Rosner’s outlier tests. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ test and by R(0.01) for the Rosner’s test. Stragglers are marked by D(0.05) for the Dixon’s test, by G(0.05) or DG(0.05) for the Grubbs’ test and by R(0.05) for the Rosner’s test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1, was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to 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 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, e.g. EN reproducibilities, 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. In some cases, a reproducibility based on former iis proficiency tests could be used.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test 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. The usual interpretation of z-scores is as follows:

	| z | < 1	good
1 <	| z | < 2	satisfactory
2 <	| z | < 3	questionable
3 <	| z |	unsatisfactory

4 EVALUATION

In this interlaboratory study, no problems were encountered with the dispatch of the samples. One participant did not report any test results at all and two participants reported test results after the final reporting date. Finally, the 43 reporting laboratories reported 122 numerical test results for Overall Migration per contact surface. Observed were 5 outlying test results, which is 4.1%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION 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 listed in appendix 5.

In the past, iis has found that the Overall and Specific migration methods, limits and calculations are mixed up and used inappropriately by participants. So iis issued a White Paper on this subject in February 2018 (White Paper on the determination of Overall and Specific migration on food contact materials, lit. 20) 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 total immersion, the EN1186 method series part 3 is considered to be the official EC test method. In method EN1186-3 it is described that three samples are needed. In this 2019 PT only one sample (a PTFE square plate) was sent for the determination of Overall Migration as the sample was positive on Overall Migration.
The target reproducibility used for statistical evaluation was estimated from the EN1186-3 (Annex A) reproducibility of simulants A, B and C (based on 3 replicates).

In the evaluation of the test results of the 2019 PT some test results were excluded from statistical calculation in case:
- the participant has 1 or more outliers in Overall Migration (per contact surface) or
- it was not clear if the overall migration test was done with double contact surface testing (see also discussion in paragraph 4.4).

Overall Migration - 1st step in mg/dm^2: This determination was problematic. Two statistical outliers were observed and five other test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the target reproducibility estimated from EN1186-3:02.

Overall Migration – 2nd step in mg/dm^2: This determination was not problematic. Two statistical outliers were observed and four other test results were excluded. However, the calculated reproducibility after rejection of the suspect data is in agreement with the target reproducibility estimated from EN1186-3:02.

Overall Migration – 3rd step in mg/dm^2: This determination was problematic. One statistical outlier was observed and five other test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the target reproducibility estimated from EN1186-3:02.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility of the relevant reference method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average test result, the calculated reproducibility (2.8 * standard deviation) and the estimated target reproducibility derived from the literature test method EN1186-3:02 are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R (lit)
Overall Migration – 1st step	mg/dm^2	35	24.30	13.31	11.72
Overall Migration – 2nd step	mg/dm^2	33	18.15	8.22	8.76
Overall Migration – 3rd step	mg/dm^2	35	11.84	7.39	5.71

Table 1: reproducibilities of tests on sample #19620

Without further statistical calculations, it can be concluded that there is only for the 2nd Overall Migration step a good compliance of the group of participating laboratories with the target reproducibility estimated from EN1186-3:02.

4.3 COMPARISON OF PROFICIENCY TEST OF SEPTEMBER 2019 AGAINST PREVIOUS PTS

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

	article filling	total immersion	EN1186
2013	----	25-30% (2)	11% (part 3)
2014	18% (1)	----	17% (part 8)
2015	14% (1)	-----	8% (part 9)
2016	17% (1)	29% (3)	8% (part 9) – 13% (part 3)
2017	----	32-36% (3)	17% (part 3)
2018	13-17% (3)	-----	17% (part 9)
2019	-----	16-22% (3)	17% (part 3)

Table 2: development of the uncertainties Overall Migration in mg/dm^2 over the years

(1) Three test items were used and the average of three test results was reported

(2) Two test items were used and the average of two test results was reported

(3) A single test item was used

In the 2019 PT as well in the 2013 PT the same batch of square plates was used. It can be concluded that there is an improvement in the uncertainty when the 2019 PT is compared with the 2013 PT. In general it can be concluded that there is an improvement for Overall Migration via total immersion when the 2019 PT is compared with the previous PTs.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

Before the start of this PT it was clear that a wide range of test results would be reported when the choice of the test conditions would have been selected by the participating laboratories. Therefore, a set of prescribed test conditions (known to give a positive test result) was given together with the instructions to all participants:

Sample ID	#19620
Simulant	Distilled Water
Exposure time	2 hours
Exposure temperature	70°C
Migration method	Total immersion, repeated use
Simulant volume	As per method used
Contact surface testing	Double

Table 3: prescribed test conditions used in this PT

The participants were requested to report the test results of all three successive migration steps. Additional details regarding preparation, residue, surface area, simulant volume and details about the evaporation step were also requested to be reported (see appendices 2 and 3).

Test method and accreditation

About 75% of the reporting participants mentioned to have used test method EN1186-3, a few participants mentioned to have used test method EN1186-1.
About 88% of the reporting participants mentioned that they are accredited for this test.

Preparation

Twenty-four participants reported not to clean the sample and ten participants used a lint free cloth/tissue or soft brush before the determination of the Overall Migration. Method EN1186-3 states in paragraph 3.41: “Before preparing test specimens, remove any surface contamination from the sample by gently wiping it with a lint free cloth, or by brushing with a soft brush.”
Surprisingly a few participants reported to have used water, a detergent/soap or an ethanol solution to clean the test item prior to use. Method EN1186-3 states in paragraph 3.41: “under no circumstances wash the sample with water or solvent”. However, in general can be concluded that it appears that these cleansing methods has a negligible effect on the Overall Migration in mg/dm^2.

Ratio dm^2 per 100 mL, contact surface and volume of simulant

Method EN1186-1 states in paragraph 9.3: “that the surface to volume ratio in the total immersion test is conventionally 1 dm^2 of food contact area to 100 mL of food simulant.”, also in method EN1186-3 the ratio of 1 dm^2/100mL is mentioned. In appendix 1, the statistical evaluation based on a migration with a reported ratio of 1 dm^2/100mL are also presented. When the group using the ratio 1dm^2/100mL is evaluated separately, the calculated reproducibility is in the 1st and 2nd Overall Migration step in line with the calculated reproducibility of the whole group.

Sample #19620 was a blue PTFE square plate of 10 x 10 x 0.2cm. When the whole plate was used for the total migration and when there was conducted a double contact surface testing (as prescribed), the exposed contact surface area should be at least 2 dm^2. Participants with a contact surface area below 2 dm^2 and no clear indication that the migration was done via double contact surface testing were excluded form the statistical evaluation of the Overall Migration in mg/dm^2.

Calculation of Overall Migration in mg/dm^2

According to method EN1186-3, the Overall Migration in mg/dm^2 should be calculated taking the mass residue after evaporation of all simulant and corrected for a blank sample mass in mg by division of the surface area in dm^2. A few participants reported a test result of Overall Migration in mg/dm^2 which is not in line with the reported residue (mg) and the reported surface area (dm^2). Some other test results of Overall Migration in mg/dm^2 were corrected without correction of the reported residue (mg) or the surface area (dm^2)

Evaporation: temperature and time

After exposure of the plate to the simulant for the selected time, the simulant must be evaporated to dryness. The reported evaporation temperature varied from 70 to 350°C. About 60% of the reporting participants used an evaporation temperature between 100°C and 150 °C. The reported evaporation time varied from 60 to 2880 minutes. About 50% of the reporting participants used an evaporation time less than 240 minutes.
The differences in evaporation temperature and time did not appear to be of influence.

5 DISCUSSION

Total immersion, repeated use

The prescribed test migration method for this PT was total immersion with repeated use. Therefore, it was requested to report the Overall Migration (mg/dm^2) for three successive migration steps.
In this PT, the average Overall Migration in (mg/dm^2) decreased in each successive migration step (step 1: 24 mg/dm^2, step 2: 18 mg/dm^2 and step 3: 12 mg/dm^2). Almost all participants reported a decrease in the three successive steps.

Limits for Overall Migration from EU regulation No 10/2011

The EU regulation describes in article 12 that the limit for Overall Migration is 10 mg/dm^2. In this 2019 PT the sample #19620 was tested in three successive contact periods, using a new portion of simulant for each exposure period. The Overall Migration found in the third migration step should comply the limit for Overall Migration (10 mg/dm^2).
According to this limit thirty-one participants would have rejected sample #19620. However, ten participants would have accepted sample #19620 based on the test result of the third migration step. Based on only the first step almost all participants would have rejected sample #19620.

2019 iis PT vs 2013 iis PT

Sample #19620 was also used in a previous iis PT; labelled as sample #13183 in iis13P05GM.

		Sample #19620			Sample #13183		
Parameter	unit	n	average	2.8 * sd	n	average	2.8 * sd
Overall Migration – 1st step	mg/dm^2	35	24.30	13.31	53	26.46	9.70
Overall Migration – 2nd step	mg/dm^2	33	18.15	8.22	54	18.72	13.35
Overall Migration – 3rd step	mg/dm^2	35	11.84	7.39	52	12.17	10.33

Table 4: comparison sample #19620 vs #13183

The average test results of the 2019 PT are comparable with the average test results of the 2013 PT. The variation of the 1st step of the 2019 PT is somewhat larger than in the 2013 PT, whereas the variation of the 2nd and the 3rd step are smaller (see also table 2).

6 CONCLUSION

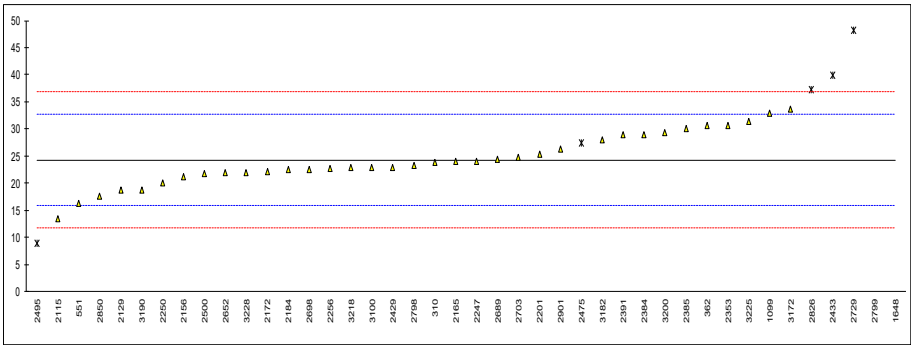
It is to be expected that the variation of the migration test results in real life practise will be larger than observed in this PT as the test conditions like time, temperature, etc. will not be prescribed but will be selected by the individual laboratories.
Each laboratory has to evaluate its performance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

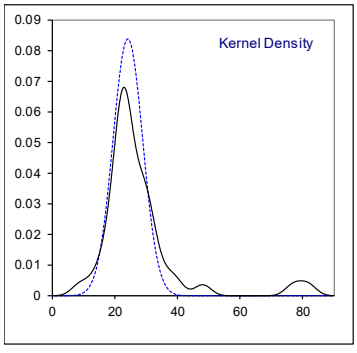
APPENDIX 1

Determination of 1st Overall Migration (per contact surface) on sample #19620; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
310	EN1186-3	23.85	C	-0.11	first reported 47.7
362	EN1186-3	30.55	C	1.49	reported 61.1
551	EN1186-3	16.203		-1.93	
1099	EN1186-3	32.9159		2.06	
1648	EN1186-3	81.832	ex	13.74	excluded see § 4.1
2115	EN1186-1	13.41		-2.60	
2129	EN1186-3	18.67		-1.34	
2156	EN1186-3	21.200		-0.74	
2165	GB31604.8	23.942		-0.09	
2172	EN1186-3	22.12		-0.52	
2184	EN1186-3	22.45		-0.44	
2201	EN1186-3	25.367		0.26	
2247	EN1186-1	24.01		-0.07	
2250	EN1186-3	20.133		-1.00	
2256		22.76		-0.37	
2353	EN1186-3	30.555		1.49	
2384	EN1186-3	28.93		1.11	
2385	EN1186-3	30.14		1.40	
2391	EN1186-3	28.846		1.09	
2429	EN1186-3	22.95		-0.32	
2433	EN1186-3	39.8	ex, C	3.70	first reported 45.400, excluded see § 4.1
2475	EN1186	27.4	ex	0.74	excluded see § 4.1
2495	EN1186-3	8.929	ex	-3.67	excluded as outlier in 2nd Overall Migration (per contact surface)
2500	EN1186-9	21.83		-0.59	
2652	EN1186-3	21.861		-0.58	
2689	EN1186-3	24.33		0.01	
2698	EN1186-3	22.596		-0.41	
2703	EN1186-3	24.8		0.12	
2729	National Standard	48.2	R(0.01)	5.71	
2798	EN1186-3	23.35		-0.23	
2799	EN1186-3	77.2	R(0.01)	12.64	
2826	EN1186-3	37.3	ex	3.11	excluded see § 4.1
2850	GUUE L12+230/22	17.65		-1.59	
2897		-----		-----	
2901	EN1186-1	26.3750		0.50	
3100	EN1186-3	22.895		-0.34	
3118		-----		-----	
3172	EN1186-3	33.68		2.24	
3182	EN1186-3	27.915		0.86	
3190	EN1186-3	18.75		-1.33	
3200	EN1186-3	29.350		1.21	
3218	EN1186-3	22.80		-0.36	
3225	EN1186-1/-3	31.4		1.70	
3228	EN1186-3	21.9		-0.57	

			1 dm^2/100mL only	
normality	OK		OK	
n	35		20	
outliers	2 (+5 ex)		0 (+2 ex)	
mean (n)	24.300		23.378	
st.dev. (n)	4.7520	RSD = 20%	4.8018	RSD = 21%
R(calc.)	13.306		13.445	
st.dev.(EN1186-3:02)	4.1861		4.0273	
R(EN1186-3:02)	11.721		11.276	

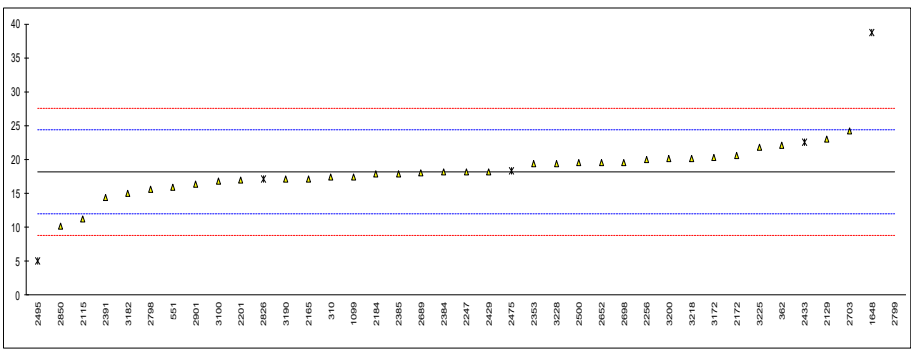


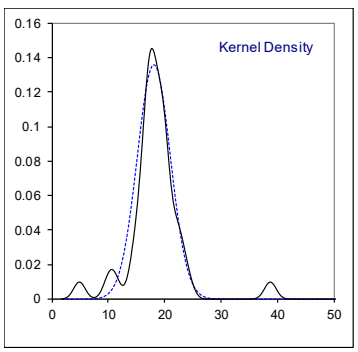


Determination of 2nd Overall Migration (per contact surface) on sample #19620; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
310	EN1186-3	17.43	C	-0.23	first reported 34.9
362	EN1186-3	22.05	C	1.25	reported 44.1
551	EN1186-3	15.964		-0.70	
1099	EN1186-3	17.4682		-0.22	
1648	EN1186-3	38.762	ex	6.59	excluded see § 4.1
2115	EN1186-1	11.25		-2.21	
2129	EN1186-3	23		1.55	
2156		-----		-----	
2165	GB31604.8	17.115		-0.33	
2172	EN1186-3	20.60		0.78	
2184	EN1186-3	17.79		-0.12	
2201	EN1186-3	16.933		-0.39	
2247	EN1186-1	18.15		0.00	
2250		-----		-----	
2256		20.04		0.60	
2353	EN1186-3	19.360		0.39	
2384	EN1186-3	18.13		-0.01	
2385	EN1186-3	17.82		-0.11	
2391	EN1186-3	14.423		-1.19	
2429	EN1186-3	18.18		0.01	
2433	EN1186-3	22.600	ex	1.42	excluded see § 4.1
2475	EN1186	18.3	ex	0.05	excluded see § 4.1
2495	EN1186-3	4.976	R(0.01)	-4.21	
2500	EN1186-9	19.47		0.42	
2652	EN1186-3	19.515		0.44	
2689	EN1186-3	18.08		-0.02	
2698	EN1186-3	19.519		0.44	
2703	EN1186-3	24.15		1.92	
2729		-----		-----	
2798	EN1186-3	15.60		-0.82	
2799	EN1186-3	76.4	R(0.01)	18.63	
2826	EN1186-3	17.1	ex	-0.34	excluded see § 4.1
2850	GUUE L12+230/22	10.15		-2.56	
2897		-----		-----	
2901	EN1186-1	16.3250		-0.58	
3100	EN1186-3	16.747		-0.45	
3118		-----		-----	
3172	EN1186-3	20.22		0.66	
3182	EN1186-3	14.929		-1.03	
3190	EN1186-3	17.10		-0.34	
3200	EN1186-3	20.150		0.64	
3218	EN1186-3	20.15		0.64	
3225	EN1186-1/-3	21.8		1.17	
3228	EN1186-3	19.4		0.40	

				1 dm^2/100mL only	
	normality	suspect		suspect	
	n	33		19	
	outliers	2 (+4 ex)		0 (+2 ex)	
	mean (n)	18.152		17.730	
	st.dev. (n)	2.9356	RSD = 16%	3.1241	RSD = 18 %
	R(calc.)	8.220		8.748	
	st.dev.(EN1186-3:02)	3.1270		3.0544	
	R(EN1186-3:02)	8.756		8.552	

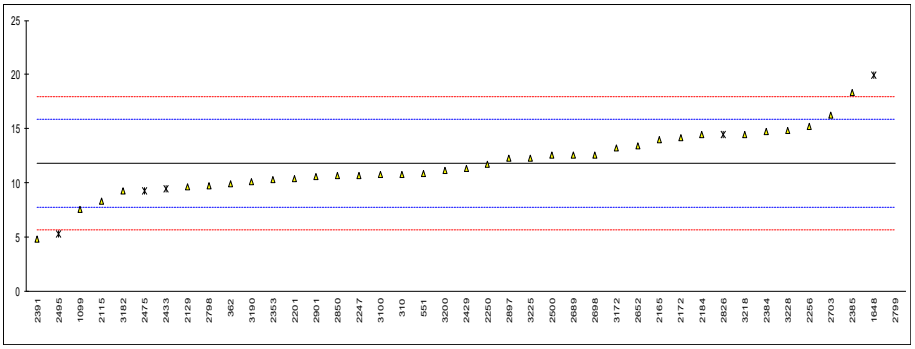


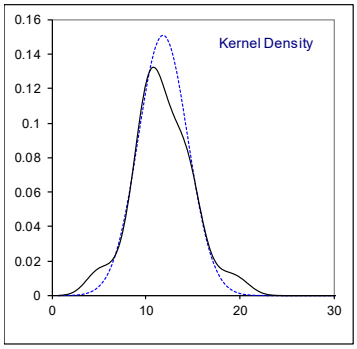


Determination of 3rd Overall Migration (per contact surface) on sample #19620; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
310	EN1186-3	10.83	C	-0.49	first reported 21.7
362	EN1186-3	9.90	C	-0.95	reported 19.8
551	EN1186-3	10.882		-0.47	
1099	EN1186-3	7.5318		-2.11	
1648	EN1186-3	19.950	ex	3.98	excluded see § 4.1
2115	EN1186-1	8.31		-1.73	
2129	EN1186-3	9.67		-1.06	
2156		-----		-----	
2165	GB31604.8	14.038		1.08	
2172	EN1186-3	14.20		1.16	
2184	EN1186-3	14.47		1.29	
2201	EN1186-3	10.442		-0.68	
2247	EN1186-1	10.66		-0.58	
2250	EN1186-3	11.744		-0.05	
2256		15.21		1.65	
2353	EN1186-3	10.310		-0.75	
2384	EN1186-3	14.73		1.42	
2385	EN1186-3	18.32		3.18	
2391	EN1186-3	4.808		-3.45	
2429	EN1186-3	11.38		-0.22	
2433	EN1186-3	9.50	ex	-1.15	excluded see § 4.1
2475	EN1186	9.3	ex	-1.24	excluded see § 4.1
2495	EN1186-3	5.264	ex	-3.22	excluded as outlier in 2nd Overall Migration (per contact surface)
2500	EN1186-9	12.55		0.35	
2652	EN1186-3	13.432		0.78	
2689	EN1186-3	12.55		0.35	
2698	EN1186-3	12.596		0.37	
2703	EN1186-3	16.3		2.19	
2729		-----		-----	
2798	EN1186-3	9.70		-1.05	
2799	EN1186-3	71.3	R(0.01)	29.16	
2826	EN1186-3	14.5	ex	1.31	excluded see § 4.1
2850	GUUE L12+230/22	10.65		-0.58	
2897		12.27		0.21	
2901	EN1186-1	10.6250		-0.59	
3100	EN1186-3	10.798		-0.51	
3118		-----		-----	
3172	EN1186-3	13.22		0.68	
3182	EN1186-3	9.242		-1.27	
3190	EN1186-3	10.15		-0.83	
3200	EN1186-3	11.150		-0.34	
3218	EN1186-3	14.50		1.31	
3225	EN1186-1/-3	12.3		0.23	
3228	EN1186-3	14.8		1.45	

			1 dm^2/100mL only
normality	OK		OK
n	35		20
outliers	1 (+5 ex)		0 (+1 ex)
mean (n)	11.836		11.286
st.dev. (n)	2.6379	RSD = 22%	1.5575 RSD = 14 %
R(calc.)	7.386		4.361
st.dev.(EN1186-3:02)	2.0390		1.9443
R(EN1186-3:02)	5.709		5.444





APPENDIX 2

Details for calculation of Overall Migration on sample #19620: 1st step

lab	total residue (mg)	mark	surface area (dm^2)	mark	volume simulant (ml)	mark	iis calc. ratio area/simulant (dm^2 / 100 ml)	remarks
310	52		1.09		100		1.09	surface=1 side?, test=2 side
362	61.1	C	1		100		1.00	fr 0.0611, surface=1 side?, test=2 side
551	33.80		2.086		200		1.04	
1099	72.42		2.20		100		2.20	
1648	83.9		2.02		100		2.02	
2115	27.9		2.08		200		1.04	
2129	56		2		330		0.61	
2156	44.100		2.080		100.000		2.08	
2165	49.8		2.08		347		0.60	
2172	44.24		2.00		200		1.00	
2184	46.7		2.08		345		0.60	
2201	50.733		2.0		200		1.00	
2247	50.0		2.083		200.0		1.04	
2250	45.1		2.0861		200		1.04	
2256	48.02		2.11		352		0.60	
2353	30.555		1.00		100		1.00	
2384	32.4		1.12		100		1.12	
2385	36.2		2		333		0.60	
2391	60.0		2.08		100		2.08	
2429	50.5		2.20		367		0.60	
2433	22.700		0.50		100		0.50	
2475	27.8		1		100		1.00	
2495	18.57		2.08		300		0.69	
2500	45.4		2.08		315		0.66	
2652	43.72		1.9994		200		1.00	
2689	50.6		2.08		200		1.04	
2698	47.000		2.080		208.000		1.00	
2703	49.6		2		100		2.00	
2729	48.2		1		200		0.50	
2798	46.7		2		200		1.00	
2799	77.2		1		400		0.25	
2826	20.1		0.539		90		0.60	
2850	35.4		2		200		1.00	
2897	-----		-----		-----		-----	
2901	52.75	C	2		200		1.00	first reported 0.05275
3100	45.8		2.0004		200		1.00	
3118	-----		-----		-----		-----	
3172	70.8		2.1021		350		0.60	
3182	58.9		2.11		900.0		0.23	
3190	37.5		2		200		1.00	
3200	58.70		2.00		200.0		1.00	
3218	46.00		2.00		200		1.00	
3225	31.4		1.00		100		1.00	
3228	45.6		2.08		347		0.60	

Details for calculation of Overall Migration on sample #19620: 2nd step

lab	total residue (mg)	mark	surface area (dm^2)	mark	volume simulant (ml)	mark	iis calc. ratio area/simulant (dm^2 / 100 ml)	remarks
310	38		1.09		100		1.09	surface=1 side?, test=2 side
362	44.1	C	1		100		1.00	fr 0.0441, surface=1 side?, test=2 side
551	33.30		2.086		200		1.04	
1099	38.43		2.20		100		2.20	
1648	40.4		2.02		100		2.02	
2115	23.41		2.08		200		1.04	
2129	69		2		330		0.61	
2156	-----		-----		-----		-----	
2165	35.6		2.08		347		0.60	
2172	41.20		2.00		200		1.00	
2184	37.0		2.08		345		0.60	
2201	33.867		2.0		200		1.00	
2247	37.8		2.083		200.0		1.04	
2250	-----		2.0861		200		1.04	
2256	42.28		2.11		352		0.60	
2353	19.360		1.00		100		1.00	
2384	20.3		1.12		100		1.12	
2385	21.4		2		333		0.60	
2391	30.0		2.08		100		2.08	
2429	46.6		2.20		367		0.60	
2433	11.300		0.50		100		0.50	
2475	18.7		1		100		1.00	
2495	10.35		2.08		300		0.69	
2500	40.5		2.08		315		0.66	
2652	39.07		1.9994		200		1.00	
2689	37.6		2.08		200		1.04	
2698	40.600		2.080		208.000		1.00	
2703	48.3		2		100		2.00	
2729	-----		-----		-----		-----	
2798	31.2		2		200		1.00	
2799	76.4		1		400		0.25	
2826	9.2		0.539		90		0.60	
2850	20.4		2		200		1.00	
2897	-----		-----		-----		-----	
2901	32.65	C	2		200		1.00	first reported 0.03265
3100	33.5		2.0004		200		1.00	
3118	-----		-----		-----		-----	
3172	42.5		2.1021		350		0.60	
3182	31.5		2.11		900.0		0.23	
3190	34.2		2		200		1.00	
3200	40.30		2.00		200.0		1.00	
3218	40.70		2.00		200		1.00	
3225	21.8		1.00		100		1.00	
3228	40.3		2.08		347		0.60	

Details for calculation of Overall Migration on sample #19620: 3rd step

lab	total residue (mg)	mark	surface area (dm^2)	mark	volume simulant (ml)	mark	iis calc. ratio area/simulant (dm^2 / 100 ml)	remarks
310	23.6		1.09		100		1.09	surface=1 side?, test=2 side
362	19.8	C	1		100		1.00	fr 0.0198, surface=1 side?, test=2 side
551	22.70		2.086		200		1.04	
1099	16.57		2.20		100		2.20	
1648	21.4		2.02		100		2.02	
2115	17.29		2.08		200		1.04	
2129	29		2		330		0.61	
2156	-----		-----		-----		-----	
2165	29.2		2.08		347		0.60	
2172	28.40		2.00		200		1.00	
2184	30.1		2.08		345		0.60	
2201	20.883		2.0		200		1.00	
2247	22.2		2.083		200.0		1.04	
2250	26.5		2.0861		200		1.04	
2256	30.1		2.11		352		0.60	
2353	10.310		1.00		100		1.00	
2384	16.5		1.12		100		1.12	
2385	22.0		2		333		0.60	
2391	10.0		2.08		100		2.08	
2429	33.6		2.20		367		0.60	
2433	4.80		0.50		100		0.50	
2475	9.7		1		100		1.00	
2495	10.95		2.08		300		0.69	
2500	26.1		2.08		315		0.66	
2652	27.40		1.9994		200		1.00	
2689	26.1		2.08		200		1.04	
2698	26.200		2.080		208.000		1.00	
2703	32.6		2		100		2.00	
2729	-----		-----		-----		-----	
2798	19.4		2		200		1.00	
2799	71.3		1		400		0.25	
2826	7.8		0.539		90		0.60	
2850	21.4		2		200		1.00	
2897	25.03		2.04		100		2.04	
2901	21.25	C	2		200		1.00	first reported 0.02125
3100	21.6		2.0004		200		1.00	
3118	-----		-----		-----		-----	
3172	27.8		2.1021		350		0.60	
3182	19.5		2.11		900.0		0.23	
3190	20.3		2		200		1.00	
3200	22.30		2.00		200.0		1.00	
3218	29.40		2.00		200		1.00	
3225	12.3		1.00		100		1.00	
3228	30.8		2.08		347		0.60	

APPENDIX 3
Summary of reported analytical details

lab	ISO17025 accredited	Cleaned sample prior to migration step	Equipment used	Evaporation time (min)			Evaporation temperature (°C)		
				1st step	2nd step	3rd step	1st step	2nd step	3rd step
310	No	No	Oven	Overnight	Overnight	Overnight	105	105	105
362	Yes	Yes: soft brush	Incubator	190	190	190	100	100	100
551	Yes	Yes: lint-free cloth	Oven	180	180	240	110	110	110
1099	Yes	Yes: dry lint-free cloth.	Oven	circa: 480	circa: 480	circa: 480	180-200	180-200	180-200
1648	Yes	Yes: lint free cloth	Oven	135	118	85	250	250	250
2115	Yes	No	Oven	120	120	120	70	70	70
2129	Yes	No	Oven	120	120	120	70	70	70
2156	Yes	No	Oven	240			110		
2165	Yes	Yes: lint free cloth	Oven	over night	over night	over night	105	105	105
2172	Yes	No	Oven	120	120	120	105	105	105
2184	Yes	No	Oven	1440	1440	1440	105	105	105
2201	Yes	No	Oven	180	180	180	90	90	90
2247	Yes	Yes: lint free tissue paper	Oven	270	270	270	100-105	100-105	100-105
2250	Yes	No	Oven	n.d.	n.d.	n.d.	100	100	100
2256	Yes	No	Oven	240	220	220	100	100	100
2353	Yes	No	Oven	~180	~180	~180	100	100	100
2384	Yes	Yes: lint-free tissue	Oven	120-240	120-240	120-240	220	220	220
2385	Yes	No	Oven	480	480	480	105	105	105
2391	No	Yes: lens tissue	Oven	111	113	104	94.0	96.0	94.6
2429	Yes	Yes: 10% ethanol solution	Oven	120	120	120	105	105	105
2433	Yes	No	Oven	90	90	90	150	150	150
2475	Yes	No	Oven	1080-1night	1080-1night	1080-1night	105	105	105
2495	Yes	Yes: soap	Oven	300	300	300	130	130	130
2500	Yes	No	Oven	120	120	120	70	70	70
2652	Yes	No	Oven						
2689	Yes	No	Incubator	600	600	600	105	105	105
2698	Yes	Yes: water	Oven	120	120	120	105	105	105
2703	Yes	No	Incubator	*)	*)	*)	82	82	82
2729	No	No	Oven	120			70		
2798	Yes	Yes: lint free cloth	Oven	360	360	360	105	105	105
2799	Yes	No	Oven	240	240	240	105	105	105
2826	Yes	No	Oven	60	60	60			
2850	Yes	No	Oven	2880	2880	2880	103	103	103
2897	Yes	Yes	Oven			*)			105
2901	No	No	Oven	180	180	120	100	100	100
3100	Yes	Yes: distilled water	Oven	132	135	134	105	105	105
3118	---	---	---						
3172	---	---	---						
3182	Yes	Yes: DI water	Oven	295	300	299	98	98	98
3190	Yes	Yes: washed by detergent	Oven	360	360	360	*)	*)	*)
3200	Yes	No	Oven	480	480	480	105	105	105
3218	Yes	Yes	Oven	120	120	120	350	350	350
3225	Yes	No	Oven	720	720	720	105	105	105
3228	Yes	Yes: lint-free cloth	Oven	overnight	overnight	overnight	105	105	105

*)
Lab 2703 – evaporation time (min):
1st step: Exposure time = 120 minutes Evaporation time = 208 minutes
2nd step: Exposure time = 123 minutes Evaporation time - Samples left to evaporate over approx. 60 hour period
3rd step: Exposure time = 122 minutes Evaporation time - Samples left to evaporate over approx. 60 hour period

Lab 2897 – evaporation time (min):
3rd step: 60 minutes on heating plate and 1 night in oven (105 °C)

Lab 3190 – evaporation temperature (°C):
1st, 2nd and 3rd step: 1,hot plate over 200°C till less than 10 ml; 2,oven 105 to dryness

n.d. = not determined

APPENDIX 4

Number of participants per country

2 labs in	BRAZIL
1 lab in	BULGARIA
1 lab in	FRANCE
3 labs in	GERMANY
1 lab in	GREECE
4 labs in	HONG KONG
1 lab in	INDIA
1 lab in	INDONESIA
5 labs in	ITALY
3 labs in	MALAYSIA
15 labs in	P.R. of CHINA
1 lab in	PHILIPPINES
1 lab in	POLAND
1 lab in	SOUTH KOREA
1 lab in	THAILAND
1 lab in	THE NETHERLANDS
1 lab in	UNITED ARAB EMIRATES
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 = possibly an error in calculations
ex = test result excluded from statistical evaluation
n.a. = not applicable
n.d. = not detected
n.e. = not evaluated
fr. = first reported

Literature

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2 EN 1186-1:02 - Guide to the selection of conditions and test methods for Overall Migration
3 EN 1186-3:02 - Test methods for Overall Migration into aqueous simulant by total immersion
4 EN 1186-8:02 - Test methods for Overall Migration into olive oil by article filling
5 EN 1186-9:02 - Test methods for Overall Migration into aqueous simulant by article filling
6 ASTM E1301:03
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9 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
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18 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
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20 White paper on the determinations of Overall and Specific migration on food contact materials, February 2018, www.iisnl.com/pdf/WhitePaper.pdf (or www.iisnl.com on News and Report page in News)