Results of Proficiency Test
Overall Migration (fcm)
September 2018

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis18P09GM

January 2019

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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 (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, a proficiency test for Overall Migration is organised by the Institute for Interlaboratory Studies (iis) every year. During the annual proficiency testing program 2018/2019, it was decided to continue with the proficiency test for the determination of Overall Migration of food contact materials.
In this interlaboratory study 49 laboratories from 19 different countries registered for participation. See appendix 4 for the number of participants per country. In this report, the results of the 2018 proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET-UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send 1 sample (a pink cup, labelled #18615), positive on Overall Migration. Furthermore, a number of test conditions (migration method, type of simulant, exposure time and temperature) 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/IEC 17043:2010 (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 organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). 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 50 pink Poly Propylene cups, fortified with Calcium Carbonate was prepared by a third party and labelled #18615. The homogeneity of the batch was checked by determination of the Overall Migration (1st step) on 8 stratified randomly selected test items.

	Overall Migration (1st step) in mg/dm^2 (3% acetic acid, 200 ml fill-up, 2 hrs. at 100°C)
Sample #18615-1	15.16
Sample #18615-2	15.88
Sample #18615-3	15.95
Sample #18615-4	15.02
Sample #18615-5	14.88
Sample #18615-6	16.24
Sample #18615-7	15.45
Sample #18615-8	15.67

Table 1: homogeneity test results of subsamples #18615

From the above test results, the observed repeatability was calculated and compared with 0.3 times the target repeatability in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Overall Migration (1st step) in mg/dm^2 (3% acetic acid, 200 ml fill-up, 2 hrs. at 100°C)
r(observed)	1.4
reference test method	EN1186-9:2002
r(reference test method)	1.6

Table 2: evaluation of the repeatability of subsamples #18615

The calculated repeatability was in agreement with the corresponding repeatability of the reference test method. Therefore, homogeneity of the samples #18615 was assumed.

To each of the participating laboratories one sample #18615 (pink cup) was sent on September 5, 2018.

2.5 ANALYSES

The participants were requested to determine Overall Migration on sample #18615 (Poly Propylene cup) using the prescribed test conditions (article filling, repeated use as migration method, 2hrs at 100°C and 3% M/V acetic acid as simulant).
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.
It was also requested to report some analytical details and if the laboratory was accredited.

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’ results, which are above the detection limit, because such results can’t be used for meaningful statistical calculations.

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 reanalyses). 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 ISO 5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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

3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.
Furthermore, Kernel Density Graphs were made. 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. Four participants did not report any test results at all and two participants reported results after the final reporting date. Finally, the 45 reporting laboratories reported 133 numerical test results for Overall Migration per contact surface. Observed were 39 outlying test results, which is 29%.
In proficiency studies, outlier percentages of 3% - 7.5% are quite normal. For all three migration steps a bimodal distribution of the data was observed. Since this sample was made to be positive and a group of laboratories did not find this positive amount of BPA in the sample, it was decided to allow a higher number of outliers than is normally done. In this way, the lower test results were not used for the determination of the mean.

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 REPORTED TEST RESULTS

In this section the 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 methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 5.

In this PT, as mentioned in the letter of instructions, the migration method was article filling for repeated use and 3% M/V acetic acid as simulant for 2 hrs. at 100°C. The participants were requested to report the test results of all three successive migration steps.

For the determination of Overall Migration (also called Global migration or Total Migration) by article filling, the EN1186 method series part 9 is considered to be the official EC test method. In Method EN1186-9 it is described that five samples are needed: two samples to determine the surface area and three sample for the migration test. In this 2018 PT only one sample (a Poly Propylene cup) was available for both surface area determination and the migration test. Nearly all participants reported to have used part 9 of the EN1186 test method for the cup (sample #18615). The reported details of the methods that were used by the participants are listed in appendices 2 and 3.

The target reproducibility used for statistical evaluation was estimated from the EN1186-9 (Annex A) reproducibility of simulants A, B and C (based on 3 replicates).

Overall Migration - 1st step in mg/dm^2: This determination was problematic for a number of laboratories. Eleven (!) statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the target reproducibility estimated from EN1186-9:02.

Overall Migration – 2nd step in mg/dm^2: This determination was problematic for a number of laboratories. Fifteen (!) statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the target reproducibility estimated from EN1186-9:02.

Overall Migration – 3rd step in mg/dm^2: This determination was problematic for a number of laboratories. Fourteen (!) statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the target reproducibility estimated from EN1186-9:02.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

The calculated reproducibilities (2.8 * sd) and the target reproducibilities derived from the reference test method EN1186-9 are compared in the next table.

	unit	n	average	2.8 * sd	R (target)
Overall Migration – 1st step	mg/dm^2	34	11.09	4.13	5.35
Overall Migration – 2nd step	mg/dm^2	29	6.60	3.20	3.18
Overall Migration – 3rd step	mg/dm^2	30	4.82	2.28	2.32

Table 3: performance overview for samples #18615

Without further statistical calculations, it can be concluded that there is a good compliance of the group of participating laboratories (after removal of the outliers) with the target reproducibility estimated from EN1186-9:02.

4.3 COMPARISON OF PROFICIENCY TEST OF SEPTEMBER 2018 AGAINST PREVIOUS PTS

The uncertainties of the test results of the Overall Migration test results in mg/dm^2 in the iis18P09GM are listed in the next table and are in line with previous proficiency tests.
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 are given in table 4.

	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)

Table 4: comparison of the relative uncertainties for 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

No quality improvement was observed over the years. A possible explanation may be that the test items used vary. This year a cup was used and in the past a spoon, a spatula, a bowl, square plates and gloves.

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 predetermined test conditions (known to give a positive test result) was given together with the instructions to all participants. These conditions in this PT were:

Sample ID	#18615
Simulant	3% M/V acetic acid
Exposure time	2 hours
Exposure temperature	100.0 °C
Migration method	Article filling, repeated use
Simulant volume	as per method used

Table 5: 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).

Preparation

Surprisingly, nine participants reported to have used water to clean the test item prior to use and one participant reported to have cleaned the cup with 3% Acetic Acid. Method EN1186-9 states in paragraph 6.1: “under no circumstances wash the sample with water or solvent”. Five of the participants that used water for cleaning had outlying results (some high, some low). It does not appear that using water to clean has a major effect on the test result. However, the participant that cleaned the cup with Acetic Acid found very low test results (at least 10 times lower).

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

With article filling the surface to volume ratio varies with the volume that is chosen to fill the article. Since the chosen volume of the simulant did not vary much (200 to 260 mL), the ratio found for the laboratories varied between 0.62 and 0.72. One laboratory used only 110 ml of volume, resulting in a higher ratio and one laboratory used a ratio of 0.6, which is a ratio more consistent with total immersion tests than with article filling.

Calculation of Overall Migration in mg/dm^2

The calculation was checked from the reported results for total residue, contact surface and simulant volume. The majority of the participants calculated the Overall Migration with the equation of paragraph 8.1.1 of EN1186-9. Seven participants calculated the migration with the equation of paragraph 8.1.2 of EN1186-9. The influence of this difference in calculation is small and only relevant for those laboratories that have found a high total residue. Recalculating these higher total residues with the equation of paragraph 8.1.1 will still result in a (too) high migration result.

One participant 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).

Evaporation: temperature, time and volume

After exposure of the cup to the simulant for the selected time, the simulant must be evaporated to dryness. Most laboratories reported to have evaporated all simulant in one step and two participants reported to have used the distillation method, mentioned in paragraph 7.2.3 of method EN1186-9.

Most participants used an evaporation temperature around 100 °C. This is according to method EN1186-9. Some participants used temperatures up to 350°C for evaporation. The time needed to evaporate each simulant solution has a wide range from 30 minutes to 60 hours (!). These differences did not appear to be of influence.

However, the majority of the participants use the distillation method, using a hotplate, steam bath or other heating device to evaporate to a low volume. This could give a risk by sputtering or overheating the residues, resulting in loss of residue and as a consequence a lower Overall Migration test result.

5 DISCUSSION

Total migration, repeated use

A cup is in general an article for multiple 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: 11 mg/dm^2, step 2: 7 mg/dm^2 and step 3: 5 mg/dm^2). The majority of the laboratories reported also 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 2018 PT the cup 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 all participants, but one, who did conduct the three migration steps would not have rejected the cup based on the test result of the third migration step.

Based on only the first step, twenty-six participants would have rejected the cup and nineteen would not have rejected the cup.

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 predetermined but will be selected by the individual laboratories.

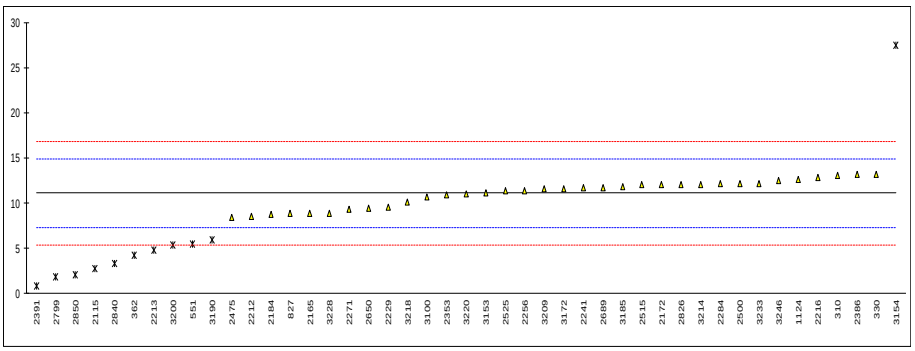
Each laboratory has to evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and the quality of the analytical results.

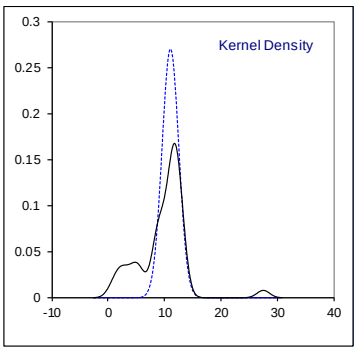
Finally, a request from our side: in case your laboratory can help the Institute for Interlaboratory Studies (iis) with suitable positive material for the determination of Overall Migration on food contact materials, you are kindly requested to contact the Institute for Interlaboratory Studies (iisnl@sgs.com).

APPENDIX 1

Determination of 1st Overall Migration on sample #18615; results in mg/dm^2 per contact surface

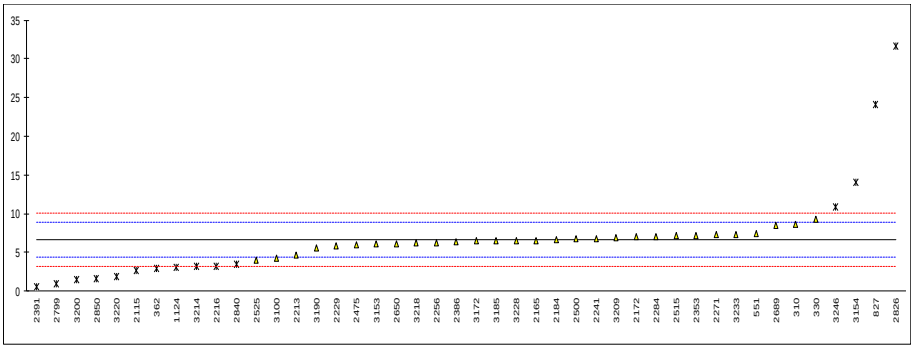
lab	method	value	mark	z(targ)	remarks
310	EN1186-9	13.0		1.00	
330	EN1186-9	13.19		1.10	
362	EN1186-9	4.22	R(0.05)	-3.60	
551	EN1186-9	5.488	R(0.05)	-2.93	
827	EN1186-9	8.83		-1.18	
1099		-----		-----	
1124	EN1186-9	12.61		0.79	
2115	EN1168	2.781	R(0.05)	-4.35	
2165	EN1186-9	8.90		-1.15	
2172	EN1186-9	12.0		0.48	
2184	EN1186-9	8.7135		-1.24	
2212	CFR175.300	8.5		-1.36	
2213	EN1186-9	4.78	R(0.05)	-3.30	
2216	21CFR175.300Mod.	12.78332		0.89	
2229	EN1186-9	9.509		-0.83	
2241	EN1186-9	11.69		0.31	
2256	EN1186-9	11.40		0.16	
2271	EN1186-9	9.325		-0.92	
2284	EN1186-9	12.10		0.53	
2353	EN1186-9	10.860		-0.12	
2386	EN1186-9	13.17		1.09	
2391	EN1186-9	0.855	R(0.05)	-5.36	
2475	EN1186	8.414		-1.40	
2495		-----		-----	
2500	EN1186-9	12.12		0.54	
2515	EN1186-9	11.985		0.47	
2525	EN1186-9	11.368		0.14	
2650	EN1186-9	9.427		-0.87	
2689	EN1186-9	11.739		0.34	
2799	EN1186-9	1.83	R(0.05)	-4.85	
2826	EN1186-9	12		0.48	
2840	EN1186-9	3.259	R(0.05)	-4.10	
2850	INH-10	2.10	R(0.05)	-4.71	
3100	EN1186-9	10.69		-0.21	
3153	EN1186-1/-9	11.12		0.01	
3154	EN1186-9	27.48	C,R(0.01)	8.58	first reported: 0.0275
3163		-----		-----	
3172	EN1186-9	11.60		0.27	
3185	EN1186-9	11.82		0.38	
3190	EN1186-3	5.966	R(0.05)	-2.68	
3200	EN1186-9	5.36	R(0.05)	-3.00	
3209	EN1186-3	11.55		0.24	
3214	EN1186-9	12.01		0.48	
3218	EN1186-9	10.120		-0.51	
3220	EN1186-9	10.9687		-0.06	
3228	EN1186-9	8.90		-1.15	
3233	EN1186-9	12.2		0.58	
3237		-----		-----	
3246	EN1186-9	12.500		0.74	
	normality	OK			
	n	34			
	outliers	11			
	mean (n)	11.092			
	st.dev. (n)	1.4760	RSD=13%		
	R(calc.)	4.133			
	st.dev.(EN1186-9:02)	1.9107			
	R(EN1186-9:02)	5.350			

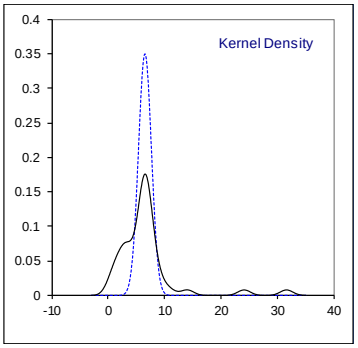




Determination of 2nd Overall Migration on sample #18615; results in mg/dm^2 per contact surface

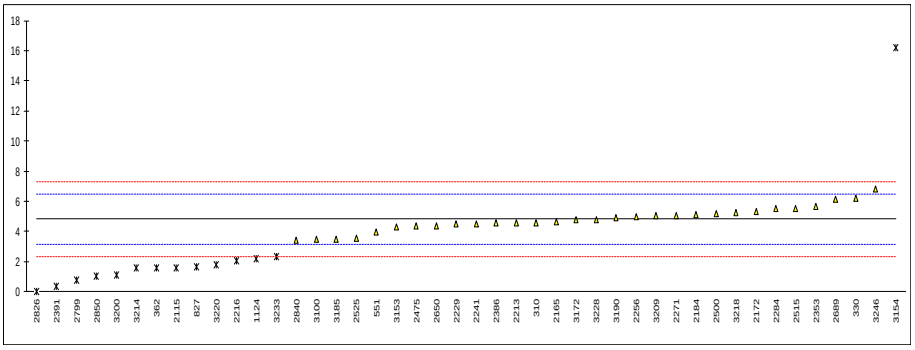
lab	method	value	mark	z(targ)	remarks
310	EN1186-9	8.6		1.76	
330	EN1186-9	9.24		2.32	
362	EN1186-9	2.98	R(0.05)	-3.18	
551	EN1186-9	7.439		0.74	
827	EN1186-9	24.11	R(0.01)	15.40	
1099		-----		-----	
1124	EN1186-9	3.09	R(0.05)	-3.09	
2115	EN1168	2.72	R(0.05)	-3.41	
2165	EN1186-9	6.57		-0.03	
2172	EN1186-9	7.04		0.39	
2184	EN1186-9	6.6667		0.06	
2212		-----		-----	
2213	EN1186-9	4.59		-1.77	
2216	21CFR175.300Mod.	3.263826	R(0.05)	-2.93	
2229	EN1186-9	5.828		-0.68	
2241	EN1186-9	6.76		0.14	
2256	EN1186-9	6.3		-0.26	
2271	EN1186-9	7.239		0.56	
2284	EN1186-9	7.10		0.44	
2353	EN1186-9	7.227		0.55	
2386	EN1186-9	6.37		-0.20	
2391	EN1186-9	0.513	R(0.05)	-5.35	
2475	EN1186	6.0		-0.53	
2495		-----		-----	
2500	EN1186-9	6.72		0.10	
2515	EN1186-9	7.116		0.45	
2525	EN1186-9	4.018		-2.27	
2650	EN1186-9	6.152		-0.39	
2689	EN1186-9	8.509		1.68	
2799	EN1186-9	0.952	R(0.05)	-4.97	
2826	EN1186-9	31.6	R(0.01)	21.98	
2840	EN1186-9	3.443	R(0.05)	-2.78	
2850	INH-10	1.59	R(0.05)	-4.41	
3100	EN1186-9	4.25		-2.07	
3153	EN1186-1/-9	6.14		-0.41	
3154	EN1186-9	14.08	C,R(0.05)	6.58	first reported: 0.0141
3163		-----		-----	
3172	EN1186-9	6.47		-0.12	
3185	EN1186-9	6.50		-0.09	
3190	EN1186-3	5.531		-0.94	
3200	EN1186-9	1.49	R(0.05)	-4.49	
3209	EN1186-3	6.91		0.27	
3214	EN1186-9	3.16	R(0.05)	-3.03	
3218	EN1186-9	6.287		-0.28	
3220	EN1186-9	1.8404	R(0.05)	-4.19	
3228	EN1186-9	6.53		-0.06	
3233	EN1186-9	7.33		0.64	
3237		-----		-----	
3246	EN1186-9	10.833	R(0.05)	3.72	
	normality	suspect			
	n	29			
	outliers	15			
	mean (n)	6.601			
	st.dev. (n)	1.1420	RSD=17%		
	R(calc.)	3.198			
	st.dev.(EN1186-9:02)	1.1372			
	R(EN1186-9:02)	3.184			

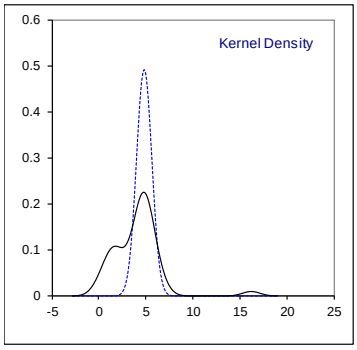




Determination of 3rd Overall Migration on sample #18615; results in mg/dm^2 per contact surface

lab	method	value	mark	z(targ)	remarks
310	EN1186-9	4.6		-0.26	
330	EN1186-9	6.17		1.63	
362	EN1186-9	1.61	R(0.05)	-3.87	
551	EN1186-9	3.963		-1.03	
827	EN1186-9	1.66	R(0.05)	-3.80	
1099		-----		-----	
1124	EN1186-9	2.18	R(0.05)	-3.18	
2115	EN1168	1.61	R(0.05)	-3.87	
2165	EN1186-9	4.65		-0.20	
2172	EN1186-9	5.31		0.59	
2184	EN1186-9	5.0877		0.32	
2212		-----		-----	
2213	EN1186-9	4.54		-0.34	
2216	21CFR175.300Mod.	2.08522	R(0.05)	-3.29	
2229	EN1186-9	4.479		-0.41	
2241	EN1186-9	4.51		-0.37	
2256	EN1186-9	5.0		0.22	
2271	EN1186-9	5.03		0.26	
2284	EN1186-9	5.50		0.82	
2353	EN1186-9	5.684		1.04	
2386	EN1186-9	4.54		-0.34	
2391	EN1186-9	0.342	R(0.05)	-5.39	
2475	EN1186	4.345		-0.57	
2495		-----		-----	
2500	EN1186-9	5.21		0.47	
2515	EN1186-9	5.555		0.89	
2525	EN1186-9	3.564		-1.51	
2650	EN1186-9	4.396		-0.51	
2689	EN1186-9	6.149		1.60	
2799	EN1186-9	0.732	R(0.05)	-4.92	
2826	EN1186-9	0	R(0.01)	-5.80	
2840	EN1186-9	3.42377		-1.68	
2850	INH-10	1.02	R(0.05)	-4.58	
3100	EN1186-9	3.45		-1.65	
3153	EN1186-1/-9	4.32		-0.60	
3154	EN1186-9	16.19	C,R(0.01)	13.70	first reported: 1.47
3163		-----		-----	
3172	EN1186-9	4.74		-0.09	
3185	EN1186-9	3.50		-1.59	
3190	EN1186-3	4.910		0.11	
3200	EN1186-9	1.07	R(0.05)	-4.52	
3209	EN1186-3	5.02		0.24	
3214	EN1186-9	1.56	R(0.05)	-3.93	
3218	EN1186-9	5.269		0.54	
3220	EN1186-9	1.7668	R(0.05)	-3.68	
3228	EN1186-9	4.80		-0.02	
3233	EN1186-9	2.33	R(0.05)	-3.00	
3237		-----		-----	
3246	EN1186-9	6.833		2.43	
	normality	OK			
	n	30			
	outliers	14			
	mean (n)	4.818			
	st.dev. (n)	0.8130	RSD=17%		
	R(calc.)	2.276			
	st.dev.(EN1186-9:02)	0.8300			
	R(EN1186-9:02)	2.324			





APPENDIX 2:

Details for calculation of Overall Migration on sample #18615: 1st step

lab	total residue (mg)		surface area (dm^2)	volume simulant (ml)		iis calculation ratio area/simulant (dm^2 / 100 ml)	remarks
310	20.1		1.56	220		0.7091	
330	17.6		1.63	250		0.6520	
362	6.8	C	1.61	250		0.6440	first reported: 0.0068
551	9.00		1.65	240		0.6875	
827	11.5		1.63	250		0.6520	
1099	-----		-----	-----		-----	
1124	20.80		1.65	230		0.7174	
2115	4.5		1.618	242		0.6686	
2165	15.3		1.72	250		0.6880	
2172	19.5		1.62	250		0.6480	
2184	14.9		1.71	250		0.6840	
2212	14.0		1.6419	250		0.6568	
2213	7.7		1.61	230		0.7000	
2216	14.5		1.103	110		1.0027	
2229	15.5		1.63	250		0.6520	
2241	18.94		1.62	250		0.6480	
2256	18.0		1.6213	250		0.6485	
2271	15.2		1.63	250		0.6520	
2284	19.60		1.62	250		0.6480	
2353	13.66	C	1.5723	250		0.6289	first reported: 0.01366
2386	21.60		1.64	240		0.6833	
2391	1.5		1.755	260		0.6750	
2475	12.2		1.74	240		0.7250	
2495	-----		-----	-----		-----	
2500	19.4		1.642	250		0.6568	
2515	19.2		1.602	250.0		0.6408	
2525	18.400		1.616	240		0.6733	
2650	15.46		1.64	250		0.6560	
2689	18.9		1.61	245		0.6571	
2799	2.5	C	1.3655	200		0.6828	first reported: 0.0025
2826	17.4		1.76	250		0.7040	
2840	5.3		1.62624	250		0.6505	
2850	3.9		1.57	230		0.6826	
3100	15.02	C	1.756	250		0.7024	first reported: 0.01502
3153	18.30		1.65	250		0.6600	
3154	40.4	C	1.47	230		0.6391	first reported: 0.0404
3163	-----		-----	-----		-----	
3172	18.1		1.56	230	C	0.6783	first reported: 2.30
3185	16.2		1.58	230		0.6870	
3190	9.6		1.609	240		0.6704	
3200	9.00		1.68	245.0		0.6857	
3209	18.592		1.61	250		0.6440	
3214	18.73		1.56	230		0.6783	
3218	16.9		1.670	240		0.6958	
3220	14.9		1.3584	200		0.6792	
3228	15.4		1.73	250		0.6920	
3233	7.30		1.67	246	C	0.6789	first reported: 0.246
3237	-----		-----	-----		-----	
3246	16.65		1.332	222		0.6000	

Details for calculation of Overall Migration on sample #18615: 2nd step

lab	total residue (mg)		surface area (dm^2)	volume simulant (ml)		iis calculation ratio area/simulant (dm^2 / 100 ml)	remarks
310	13.6		1.56	220		0.7091	
330	12.5		1.63	250		0.6520	
362	4.8	C	1.61	250		0.6440	first reported: 0.0048
551	12.20		1.65	240		0.6875	
827	31.4		1.63	250		0.6520	
1099	-----		-----	-----		-----	
1124	5.10		1.65	230		0.7174	
2115	4.4		1.618	242		0.6686	
2165	11.3		1.72	250		0.6880	
2172	11.4		1.62	250		0.6480	
2184	11.4		1.71	250		0.6840	
2212	-----		-----	-----		n	
2213	7.4		1.61	230		0.7000	
2216	4.2		1.103	110		1.0027	
2229	9.5		1.63	250		0.6520	
2241	10.95		1.62	250		0.6480	
2256	10.2		1.6213	250		0.6485	
2271	11.8		1.63	250		0.6520	
2284	11.51		1.62	250		0.6480	
2353	9.09	C	1.5723	250		0.6289	first reported: 0.00909
2386	10.45		1.64	240		0.6833	
2391	0.9		1.755	260		0.6750	
2475	8.7		1.74	240		0.7250	
2495	-----		-----	-----		-----	
2500	11.0		1.642	250		0.6568	
2515	11.4		1.602	250.0		0.6408	
2525	6.200		1.543	220		0.7014	
2650	10.09		1.64	250		0.6560	
2689	13.7		1.61	245		0.6571	
2799	1.3	C	1.3655	200		0.6828	first reported: 0.0013
2826	44.4		1.76	250		0.7040	
2840	5.4		1.56816	240		0.6534	
2850	3.1		1.57	230		0.6826	
3100	5.97	C	1.756	250		0.7024	first reported: 0.00597
3153	10.10		1.65	250		0.6600	
3154	20.7	C	1.47	230		0.6391	first reported: 0.0207
3163	-----		-----	-----		-----	
3172	10.1		1.56	230	C	0.6783	first reported: 2.30
3185	8.9		1.58	230		0.6870	
3190	8.9		1.609	240		0.6704	
3200	2.60		1.68	245.0		0.6857	
3209	11.112		1.61	250		0.6440	
3214	4.93		1.56	230		0.6783	
3218	10.5		1.670	240		0.6958	
3220	2.5		1.3584	200		0.6792	
3228	11.3		1.73	250		0.6920	
3233	4.40		1.67	246	C	0.6789	first reported: 0.246
3237	-----		-----	-----		-----	
3246	14.430		1.332	222		0.6000	

Details for calculation of Overall Migration on sample #18615: 3rd step

lab	total residue (mg)		surface area (dm^2)		volume simulant (ml)		iis calculation ratio area/simulant (dm^2 / 100 ml)	remarks
310	7.4		1.56		220		0.7091	
330	8.4		1.63		250		0.6520	
362	2.6	C	1.61		250		0.6440	first reported: 0.0026
551	6.5		1.65		240		0.6875	
827	2.2		1.63		250		0.6520	
1099	-----		-----		-----		-----	
1124	3.60		1.65		230		0.7174	
2115	2.6		1.618		242		0.6686	
2165	8.0		1.72		250		0.6880	
2172	8.60		1.62		250		0.6480	
2184	8.7		1.71		250		0.6840	
2212	-----		-----		-----		-----	
2213	7.3		1.61		230		0.7000	
2216	2.6		1.103		110		1.0027	
2229	7.3		1.63		250		0.6520	
2241	7.30		1.62		250		0.6480	
2256	8.1		1.6213		250		0.6485	
2271	8.2		1.63		250		0.6520	
2284	8.91		1.62		250		0.6480	
2353	7.15	C	1.5723		250		0.6289	first reported: 0.00715
2386	7.45		1.64		240		0.6833	
2391	0.6		1.755		260		0.6750	
2475	6.3		1.74		240		0.7250	
2495	-----		-----		-----		-----	
2500	8.6		1.642		250		0.6568	
2515	8.9		1.602		250.0		0.6408	
2525	5.500		1.543		220		0.7014	
2650	7.21		1.64		250		0.6560	
2689	9.9		1.61		245		0.6571	
2799	1	C	1.3655		200		0.6828	first reported: 0.0010
2826	0		1.76		250		0.7040	
2840	5.3		1.54799		230		0.6730	
2850	2.2		1.57		230		0.6826	
3100	4.85	C	1.756		250		0.7024	first reported: 0.00485
3153	7.10		1.65		250		0.6600	
3154	23.8	C	1.47	C	230		0.6391	first reported: 0.0238, 0.0162
3163	-----		-----		-----		-----	
3172	7.4		1.56		230	C	0.6783	first reported: 2.30
3185	4.8		1.58		230		0.6870	
3190	7.9		1.609		240		0.6704	
3200	1.90		1.68		245.0		0.6857	
3209	8.083		1.61		250		0.6440	
3214	2.43		1.56		230		0.6783	
3218	8.8		1.670		240		0.6958	
3220	2.4		1.3584		200		0.6792	
3228	8.3		1.73		250		0.6920	
3233	1.40		1.67		246	C	0.6789	first reported: 0.246
3237	-----		-----		-----		-----	
3246	9.102		1.332		222		0.6000	

APPENDIX 3

Additional reported analytical details on sample #18615

lab	ISO17025 accredited for this test	Cleaned prior to migration step	preheated simulant to 100°C	surface to volume ratio (dm^2/ml)
310	No	No	Yes	7.091
330	Yes	Yes	Yes	1,63dm2 / 250ml
362	Yes	Yes, with soft brush	Yes	0.0064
551	Yes	Yes	Yes	0,0068 dm2/mL
827	No	No	Yes	1.63dm2/250mL = 0.00652(dm2/mL)
1099	---	---	---	---
1124	Yes	No	Yes	1.65/230
2115	Yes	No	Yes	0.0067
2165	No	No	Yes	1.72 dm2/250ml
2172	Yes	No	Yes	0.00648
2184	Yes	No	Yes	1.71dm2 / 250ml
2212	Yes	Yes, wipe with lint-free cloth gently	Yes	
2213	No	Yes, with distilled water	Yes	0.007
2216	Yes	Yes, with a brush and hot (> 88C) water	Yes	1/100
2229	Yes	Yes	Yes	
2241	Yes	No	Yes	1.6/250
2256	Yes	Yes, with soft brush	Yes	0.006485
2271	Yes	No	Yes	0.00652
2284	Yes	Yes, with non woven fabric	Yes	1.62dm2/250ml
2353	Yes	Yes, with brush	Yes	1.5723 dm2/250ml
2386	Yes	Yes, with water	Yes	1,64/240
2391	No	Yes, with 70ml of 3% (w/v) Acetic acid	No	0.0068
2475	Yes	No	Yes	0.073
2495	---	---	---	---
2500	Yes	No	Yes	0.0066
2515	Yes	Yes, wipe with lint-free cloth gently	Yes	0.0064
2525	Yes	No	Yes	6,7 (1st step); 6,8 (2nd, 3rd step)
2650	Yes	No	Yes	1.6 / 250
2689	Yes	Yes, with DI water	Yes	0.00654
2799	No	No	Yes	0.0068
2826	Yes	No	Yes	0.00704
2840	Yes	No	Yes	1) 0,0065; 2) 0,0065; 3) 0,0067
2850	Yes	Yes, with water	Yes	1.57/230
3100	Yes	Yes, with distilled water	Yes	0.00702dm2/mL
3153	Yes	Yes, with lint-free cloth	Yes	1.6 dm2 / 250ml
3154	---	---	---	---
3163	---	---	---	---
3172	Yes	No	No	6.78
3185	Yes	Yes, with distilled water	Yes	1.58dm^2 / 230mL
3190	Yes	Yes, with soft brush	Yes	1.6/240
3200	Yes	Yes	Yes	0.0066
3209	Yes	Yes	Yes	1dm2:156mL
3214	No	Yes, with non woven paper	Yes	0.00678
3218	Yes	No	Yes	1.670dm2/240mL
3220	No	Yes, with distilled water	Yes	0.006792 dm^2/mL
3228	Yes	Yes, with lint-free cloth	Yes	1.73/250
3233	No	Yes, with lint-free cloth	Yes	1.67/0.246 = 6.79
3237	---	---	---	---
3246	Yes	Yes, with distilled water	No	0.6

Additional reported analytical details on sample #18615– continued

lab	evaporation of simulant	duration of evaporation (min)	temperature of evaporation (°C)
310	Directly evaporated from a dish (Evap. method)	Overnight	105
330	Directly evaporated from a dish (Evap. method)	8h	95°C
362	Directly evaporated from a dish (Evap. method)	360	100
551	Directly evaporated from a dish (Evap. method)	Average of 7h30	130 ºC
827	Directly evaporated from a dish (Evap. method)	840min (overnight)	105
1099	---	---	---
1124	Directly evaporated from a dish (Evap. method)	200	100
2115	Directly evaporated from a dish (Evap. method)	3600	170
2165	Directly evaporated from a dish (Evap. method)	600	105
2172	Directly evaporated from a dish (Evap. method)	120 min hotplate and 60 min oven	105
2184	Directly evaporated from a dish (Evap. method)	960 mins	105°C
2212	---	---	---
2213	Directly evaporated from a dish (Evap. method)	120	100.2
2216	Directly evaporated from a dish (Evap. method)	30 (hotplate followed by oven)	105
2229	Directly evaporated from a dish (Evap. method)	240	110
2241	Directly evaporated from a dish (Evap. method)	270 min hot plate and 30min oven	280
2256	First distilled before further evap. (Dist. method)	120 minutes	100°C
2271	Directly evaporated from a dish (Evap. method)	180	180
2284	Directly evaporated from a dish (Evap. method)	about 800min	105
2353	Directly evaporated from a dish (Evap. method)	Around 300min	Around 72.5
2386	Directly evaporated from a dish (Evap. method)	over night	105
2391	Directly evaporated from a dish (Evap. method)	390	100
2475	Directly evaporated from a dish (Evap. method)	1440	110
2495	---	---	---
2500	Directly evaporated from a dish (Evap. method)	120	100
2515	Directly evaporated from a dish (Evap. method)	360	150 °C of hot plate
2525	Directly evaporated from a dish (Evap. method)	about 90 min (hotplate)	300 °C
2650	Directly evaporated from a dish (Evap. method)	240	105
2689	Directly evaporated from a dish (Evap. method)	720	105
2799	Directly evaporated from a dish (Evap. method)	180	105
2826	Directly evaporated from a dish (Evap. method)	210	100
2840	Directly evaporated from a dish (Evap. method)	1) 04h:21m; 2) 02h:06m; 3) 02h:56m	80°C
2850	Directly evaporated from a dish (Evap. method)	2880	103
3100	Directly evaporated from a dish (Evap. method)	120min	105
3153	Directly evaporated from a dish (Evap. method)	6 hours	100°C
3154	---	---	---
3163	---	---	---
3172	Directly evaporated from a dish (Evap. method)	360	100
3185	Directly evaporated from a dish (Evap. method)	150 minutes	105°C
3190	Directly evaporated from a dish (Evap. method)	about 240min	about 300°C
3200	First distilled before further evap. (Dist. method)	600	105
3209	Directly evaporated from a dish (Evap. method)	240	100
3214	Directly evaporated from a dish (Evap. method)	480	105
3218	Directly evaporated from a dish (Evap. method)	120min	350°C
3220	Directly evaporated from a dish (Evap. method)	4hrs.	110 °C
3228	Directly evaporated from a dish (Evap. method)	960	105
3233	Directly evaporated from a dish (Evap. method)	/	105°C
3237	---	---	---
3246	Directly evaporated from a dish (Evap. method)	240 minutes	200°C

APPENDIX 4

Number of participating laboratories per country

2 labs in	BRAZIL
1 lab in	BULGARIA
3 labs in	FRANCE
3 labs in	GERMANY
5 labs in	HONG KONG
2 labs in	INDIA
4 labs in	ITALY
1 lab in	KOREA
1 lab in	LATVIA
1 lab in	MALAYSIA
16 labs in	P.R. of CHINA
1 lab in	POLAND
1 lab in	SPAIN
1 lab in	TAIWAN R.O.C.
2 labs in	THE NETHERLANDS
1 lab in	TURKEY
1 lab in	U.S.A.
1 lab in	UNITED ARAB EMIRATES
2 labs 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
ex = test result excluded from the statistical evaluation
n.a. = not applicable
n.e. = not evaluated
n.d. = not detected
fr. = first reported

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
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
7 ISO 5725:86
8 ISO 5725, parts 1-6, 1994
9 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
10 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
11 IP 367:96
12 DIN 38402 T41/42
13 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
14 J.N. Miller, Analyst, 118, 455, (1993)
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