Results of Proficiency Test
Overall migration (fcm)
October 2016

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

On request of a number of participants in the iis PT program it was decided to start PTs on food contact materials (fcm) in 2012. Since 2012 iis has organised a PT on Overall Migration every year. During the contact of the food contact materials with the 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, expressed in mg/dm^2 to be 10. 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.
It was decided to continue with the interlaboratory study for the determination of Overall migration on food contact materials in the annual proficiency testing program 2016/2017. In this interlaboratory study 54 laboratories from 21 different countries did register for participation (see appendix 4). In this report, the results of the 2016 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. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send 2 different samples which were tested and found to be positive on migration. The first samples were three identical bowls, labelled #16615. The second sample was a spatula, labelled #16616. Furthermore, a number of test conditions (migration method, type of simulant, exposure time and temperature) were prescribed to be used for both samples. Participants were also requested to report some of the test conditions that the laboratory had used.

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 April 2014 (iis-protocol, version 3.3). 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 first sample, a batch of polypropylene salad bowls for single use in the food industry that gave positive test results for Overall Migration was selected.
The homogeneity of the batch was checked by determination of the Overall Migration (48 hrs. at 70°C and 3% acetic acid as simulant) on three sets of three stratified randomly selected samples.

3 sets of 3 samples	Overall Migration in mg/dm^2	Overall Migration in mg/dm^2 average per set
Sample 1	14.50	13.7
Sample 2	13.10	
Sample 3	13.50	
Sample 4	12.40	13.7
Sample 5	14.00	
Sample 6	14.60	
Sample 7	14.00	13.5
Sample 8	14.10	
Sample 9	12.40	

Table 1: homogeneity test results of the subsamples #16615

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

	Overall Migration in mg/dm^2
r(observed)	0.3
reference test method	EN1186-9:2002
0.3xR(reference test method)	0.9
R(reference test method)	2.9

Table 2: evaluation of the repeatability of subsamples #16615

The calculated repeatability for Overall Migration on the three sets of three samples #16615 is in good agreement with the estimated target, calculated using EN1186-9 precision data. Therefore homogeneity of the subsamples #16615 was assumed.

The second sample, a spatula for multiple uses in the food industry that gave positive test results for Overall Migration was selected.
The homogeneity of the batch was checked by determination of the Overall Migration (2 hrs. at 100°C and 3% acetic acid as simulant) on four sets of three stratified randomly selected samples.

4 sets of 3 samples	Overall Migration in mg/dm^2	Overall Migration in mg/dm^2 average per set
Sample 1	13.8	15.1
Sample 2	17.2	
Sample 3	14.3	
Sample 4	13.8	14.9
Sample 5	16.2	
Sample 6	14.6	
Sample 7	14.0	14.9
Sample 8	16.0	
Sample 9	14.6	
Sample 10	14.1	14.7
Sample 11	14.8	
Sample 12	15.1	

Table 3: homogeneity test results of subsamples #16616

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

	Overall Migration in mg/dm^2
r(observed)	0.5
Reference test method	EN1186-3:2002
0.3xR(reference test method)	1.0
R(reference test method)	3.2

Table 4: evaluation of the repeatability of subsamples #16616

The calculated repeatability for Overall Migration on the samples #16616 is in good agreement with the estimated target, calculated using EN1186-3 precision data, therefore homogeneity of the samples #16616 was assumed.

To each of the participating laboratories one set of samples #16615 (three identical bowls) and one sample #16616 (spatula) was sent on September 7, 2016.

2.5 ANALYSES

The participants were requested to determine Overall Migration on both samples using the prescribed test conditions (for sample #16615: article filling, 72 hrs at 80°C and 3% acetic acid as simulant and for sample #16616: total immersion, 2hrs at 100°C and 3% acetic acid as simulant). 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’ results,

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

To get comparable results a detailed report form, on which the units were prescribed as well as the reference test methods and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/.
The laboratories were also requested to confirm the sample receipt on the same data entry portal together with some details of the test methods used.

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 reanalysis). 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 April 2014 (iis-protocol, version 3.3).
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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the test results 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. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.
Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis.
The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.
Furthermore, Kernel Density Graphs were made. 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 of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8.

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:

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

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:

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

4 EVALUATION

In this interlaboratory study, no problems were encountered with the dispatch of the samples. One participant reported test results after the final reporting date and five other participants did not report any test results at all. Finally, 49 of the 54 participants submitted test results. These 49 laboratories reported 165 numerical test results. Observed were 10 statistically outlying results, which is 6.1%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

For the determination of Overall Migration (also called Global migration or Total Migration) by article filling used for the salad bowl, the EN1186 method series part 9 is considered to be the official EC test method. In this PT, as mentioned in the letter of instructions, 3% acetic acid was used as simulant for 72 hrs. at 80°C. The target reproducibility was estimated from the EN1186-9 (Annex A) reproducibility of 2.3 mg/dm^2 for simulant B at a migration level of 10.7 mg/dm^2.
For the determination of Overall Migration by total immersion used for the spatula, the EN1186 method series part 3 is considered to be the official EC test method. In this PT, as mentioned in the letter of instructions, 3% acetic acid was used as simulant for 2 hrs. at 100°C.
The target reproducibility was estimated from the EN1186-3 (Annex A) reproducibility of 2.3 mg/dm^2 for simulant B at a migration level of 10.7 mg/dm^2.

Nearly all of the participants reported to have used respectively part 9 for the bowl and part 3 of the EN1186 test method for the spatula. The reported details of the methods that were used by the participants are listed in appendix 2 (Bowl) and appendix 3 (Spatula).

4.1 EVALUATION OF THE REPORTED TEST RESULTS PER SAMPLE AND PER TEST

In this section the results are discussed per sample and per test. Some test results were excluded, the reasons for this are discussed in paragraph 5.

Sample #16615: BOWL

Residue in mg: These intermediate results were not evaluated as they are in principle dependent on the size of the volume of simulant used.
Fifteen participants reported the residue of the part of simulant that was evaporated (residue based on e.g. 100 or 200 ml), although it was requested to report the total residue after all simulant was evaporated.
An overview of the reported test results in mg can be found in appendix 2.

Migration in mg/dm^2: This determination was problematic. Two statistical outliers were observed and eight other suspect test results were excluded. The results were excluded because a too low volume and/or a too large or a too small surface area was used, see also the discussion in paragraph 5. The calculated reproducibility after rejection of the suspect data is not in agreement with the target reproducibility estimated from EN1186-9:02.

Migration in mg/kg: The determination of overall migration in mg/kg food simulant was problematic. Five statistical outliers were observed and four other suspect test results were excluded. The calculated reproducibility, after rejection of

the suspect data was not in agreement with the target reproducibility estimated from EN1186-9:02.
Two participants did not report migration in mg/kg food simulant, but only a test result in mg/L food simulant. These two test results have been included in this evaluation because the specific gravity of the food simulant may be considered to be one, as stated in method EN1186-9:02.
One laboratory used a factor 6 to calculate the migration in mg/kg from the migration in mg/dm^2. This test result was excluded from the statistical evaluation because the use of a factor 6 is not in line with the EN1186-9.
Furthermore, test results reported by two participants that used a volume of food simulant smaller than 1000 ml were excluded from the evaluation. See also the discussion in paragraph 5.

Migration in mg/L: These test results were not evaluated because EN1186-9 test results should be reported either in mg/dm^2 or in mg/kg food simulant in accordance with EU 10-2011.
An overview of the reported test results in mg/L is given in appendix 2.

Sample #16616: SPATULA

Residue in mg: These intermediate results were not evaluated as they are in principle dependent on the size of the volume of simulant used.
Nine participants reported the residue of the part of simulant that was evaporated (residue based on e.g. 9 - 95 ml), although it was requested to report the total residue after all simulant was evaporated,
An overview of the reported test results in mg can be found in appendix 3.

Migration in mg/dm^2: This determination was problematic. Two statistical outliers were observed and eight other suspect test results were excluded, see the discussion in paragraph 5. The results were excluded because a too large or too small volume and/or a too large or too small surface area was used. After rejection of the suspect data, the calculated reproducibility is not in agreement with the target reproducibility estimated from EN1186-3:02.

Migration in mg/kg: These test results were not evaluated because EN1186-3 test results should be reported in mg/dm^2 in accordance with the test method and the regulations 90/128/EEG and EU 10-2011.
An overview of the reported test results in mg/kg is given in appendix 3.

Migration in mg/L: These test results were not evaluated because EN1186-3 test results should be reported in mg/dm^2 in accordance with the test method and the regulations 90/128/EEG and EU 10-2011.
An overview of the reported test results in mg/L is given in appendix 3.

4.2 PERFORMANCE EVALUATION OF THE GROUP OF LABORATORIES

The calculated reproducibilities and the target reproducibilities derived from the literature standard methods here resp. EN1186-9 and EN1189-3 are compared in the next tables.

BOWL	unit	n	Average	2.8 * sd	R (target)
Overall migration	mg/dm^2	34	14.47	6.87	3.11
Overall migration	mg/kg	31	57.44	27.49	12.35

Table 5: performance overview for samples #16615

SPATULA	unit	n	Average	2.8 * sd	R (target)
Overall migration	mg/dm^2	38	24.44	19.63	9.10

Table 6: performance overview for samples #16616

4.3 COMPARISON 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 is visualized in table 4.

	article filling	total immersion	EN1186
2012	18%[3]	----	17% (part 8)
2013	----	25-30%[2]	11% (part 3)
2014	18%[3]	----	17% (part 8)
2015	14%[3]	-----	8% (part 9)
2016	17% [3]	29% [1]	8% (9) – 13% (3)

Table 7: comparison of the relative uncertainties for Overall Migration in mg/dm^2 in the previous PTs and in the present PT

[1] A single test item was used

[2] Two test items were used and the average of two test results was reported

[3] Three test items were used and the average of three test results was reported

No quality improvement was yet observed over the years. An explanation may be that the group of participating laboratories is varying strongly and each year new laboratories participate and others no longer participate. Also the test items used vary. This year a bowl and a spatula were used and in the past another type of bowl, simple plates and gloves were used.

5 DISCUSSION

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 done 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 preset conditions in this PT were:

Sample ID	**#16615**	**#16616**
Simulant	3% acetic acid	3% acetic acid
Exposure time	72 hrs	2 hrs
Exposure temperature	80.0 °C	100 °C
Migration method	Article filling	Total immersion
Simulant volume	as per method used	as per method used

Table 8: preset test conditions used in this PT

Test method EN1186-3 describes only reporting in mg/dm^2. Test method EN1186-9 describes reporting in either mg/dm^2 or in mg/kg food simulant. In previous PTs it was sometimes unclear in which unit a test result was reported. Therefore, it was allowed to report three migration results (mg/dm^2, mg/kg food and mg/L food simulant).
The participants were requested to report additional details regarding preparation, residue, surface area, simulant volume (total and used for evaporation) and details about the evaporation step (see appendices 2 and 3).
Using these intermediate reported test results and the reported test details, it was possible to check the calculations done by the laboratories. This revealed that several calculation errors were present. A number of laboratories corrected the calculation errors; see the original and the revised test results in appendix 1.

Sample #16615: BOWL

Preparation

Surprisingly six participants reported to have used water to clean the test items prior to use. Method EN1186-9 states in paragraph 6.1: “under no circumstances wash the sample with water or solvent”.

Determination of volume of simulant used

The amount of simulant used by each participant varied from 200 – 1450 ml, see appendix 2.
In method EN1186-9 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 will result in a much different volume to surface ratio than using a large simulant volume. Based on this, the test results of the participants, which used a simulant volume smaller than 1000 ml, were excluded from the statistical evaluation.
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. iis measured the maximum volume to 0.5 cm below the top for this sample as 1350 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. None of the participants reported a volume above 1450 ml. No test results were excluded in this PT for use of a too large volume.

Determination of the contact surface used

Three salad bowls were sent to every participant for the overall migration test. The surface area of the bowls could be determined using one sample 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 2.06 – 6.4 dm^2, see appendix 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.

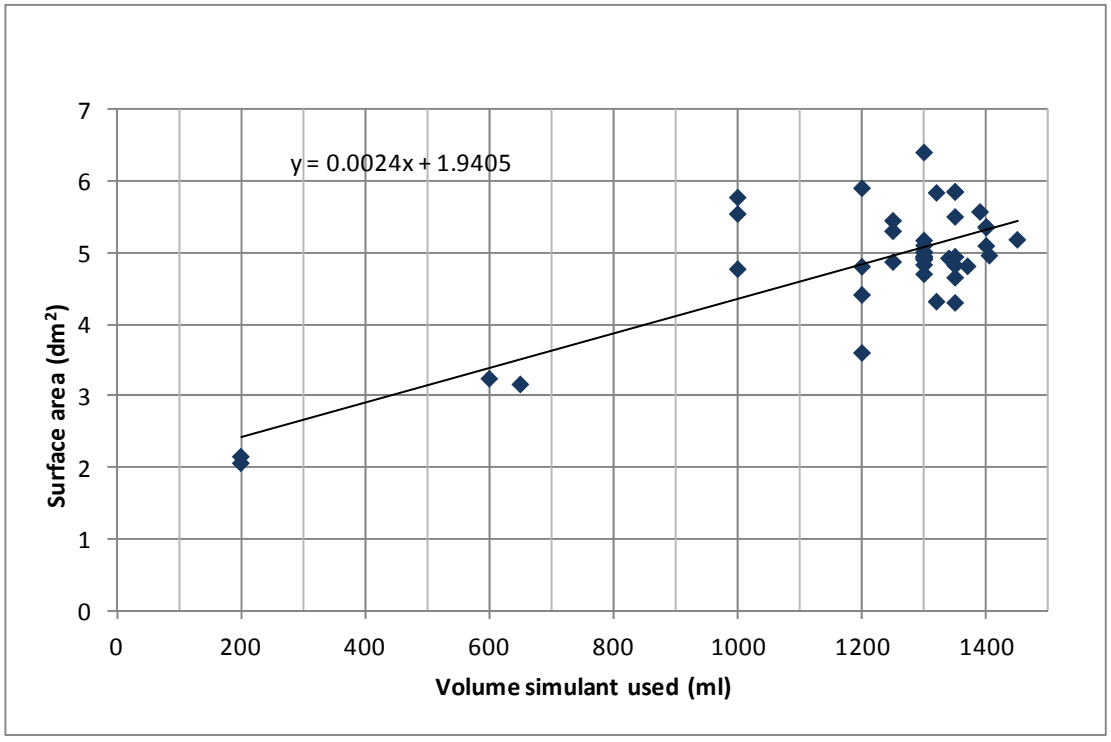


Figure 1: reported surface area versus volume of simulant used by participants

The maximum surface area (to 0.5 cm below the top) for sample #16615 was determined by iis to be approx. 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. From this, the total surface area of the maximum volume of the bowl could be estimated to be 5.85 dm^2 using 1350 ml of simulant.
Using this information, the test results for the Overall Migration in mg/dm^2 based on a surface area below 4.5 and above 6.2 dm^2 were excluded.

Calculation of Overall Migration in mg/dm^2

According to method EN1186-9, 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 dividing it by the surface area in dm^2. Eleven participants reported the sample mass in mg for a part of the evaporated simulant, but the total residue result could be calculated by iis from the reported total volume of simulant used and the partial volume of the simulant evaporated.

Calculation of Overall Migration in mg/kg food simulant

One laboratory used a factor of 6 in the calculation from mg/dm^2 to mg/kg food simulant. This method of calculation is not mentioned in method EN1186-9. Therefore the test result from this calculation method was excluded from the statistical evaluation. Method EN1186-1 does mention use of a factor of 6 dm^2 to 1 kg (see paragraph 12.1.2 of the method), but only for unknown surface to volume ratios, which is obviously not the case in this PT. Furthermore EN1186-1 states in paragraph 9.7 that the method for article filling is given in EN1186-9.

In EN1186-9, there are two ways of calculation of the Overall Migration in mg/kg food simulant. The first, as per formula 2 in paragraph 8.1.1 of EN1186-9, describes a division of the mass of all residue in milligrams by the volume in litres and reporting this value as mg/kg (assuming the specific gravity of the simulant by convention to be one). The second way, as per formula 4 in paragraph 8.1.3 of EN1186-9, describes a division of the mass in milligrams of the residue of 200 ml of simulant times the factor of 5, again reporting the value in mg/kg. When grams are used instead of milligrams also a factor of 1000 is applied.
All reporting participants, except one which used a factor 6, used either the first or the second formula to determine the Overall Migration in mg/kg food simulant.
The contact surface is irrelevant in the calculation of the test results expressed in mg/kg food simulant. Therefore one would expect to find a smaller variation in the test results in mg/kg food simulant than in the test results in dm^2 as the latter do contain the uncertainty in the contact surface estimation. Surprisingly, there is no significant difference between the two variations: RSD 17.0% *vs* 17.0%.

Calculation of Overall Migration in mg/L food simulant

EN1186-9 does not describe reporting migration in mg/L, but in paragraph 8.1.1 under formula 2 is noted that the specific gravity of the simulant is considered to be one. Thus the amount of mg/kg will be the same as in mg/L. All participants reported either the same test result for both migration in mg/kg and in mg/L or reported one test result expressed in one of the two units.

Limits for overall migration from EU regulation No 10/2011

This EU regulation describes in article 12 that the limit for overall migration is 10 mg/dm2. According to this limit 95% of the participants would have rejected the salad bowl for food use. Should this salad bowl be used in applications that will allow infants and children's food to come in contact, then an overall migration limit of 60 mg/kg is applied. According to this limit and based on the reported test results only 43% of the participants would have rejected the salad bowl, while 57% would have accepted it for food use. The large difference in rejection percentage for the salad bowl depending on the application and/or regulation is remarkable. One would expect that the rejection percentage for infants and children would have been higher than 95% and not much lower.

Sample #16616: SPATULA

Preparation

Surprisingly five participants reported to have used water to clean the test items prior to use. Method EN1186-3 states in paragraph 3.4.1: “under no circumstances wash the sample with water or solvent”.

Determination of volume of simulant used

The amount of simulant used by the participants varies from 80 – 2750 ml (!), see appendix 3. Test method EN1186-3 mentions that a specimen of approx 1 dm^2 is to be immersed into 100 mL of simulant (= 1 dm^2/100mL). Remarkably only twelve participants used a ratio near the optimal ratio of 1 dm^2/100mL (the continuous line in below graph). The wide range of used ratios is from 72 – 1650 ml per dm^2. Therefore, it was investigated whether this ratio may be of significant influence on the dispersion of the overall migration test results. In appendix 1, the evaluation of the test results based on a migration with dm^2/100mL are presented. From this evaluation it became clear that the influence of the use of different ratios may not be significant (this was also found in a previous proficiency test on Overall migration iis13P05GM, method: total immersion).

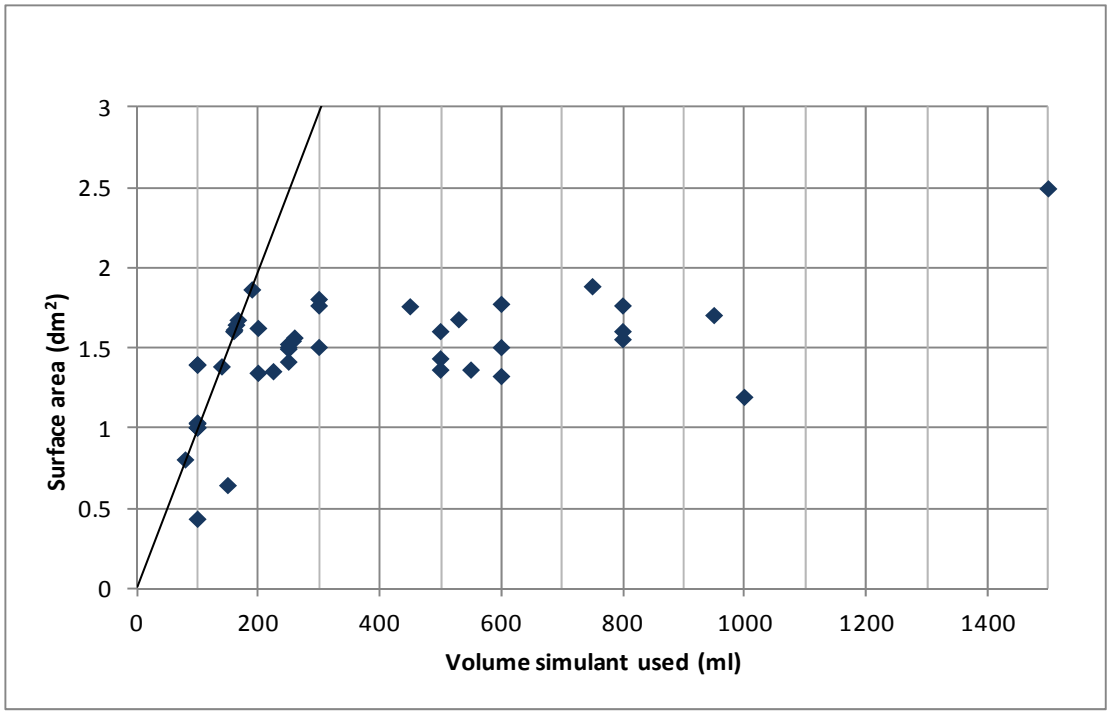


Figure 2: reported surface area versus volume of simulant used by participants

Many laboratories reported to have evaporated all simulant and only few reported to have evaporated part of the simulant and calculated the total amount of residue. It is unknown whether evaporation of the simulant was performed in the migration container or in another container. In case of low soluble components this lead to different migration results.

Determination of the contact surface used

One spatula was sent to each participant for the overall migration test. The contact surface used as reported by the participants varies from 0.1186 – 2.49 dm^2, with one exceptional value of 80.3 dm^2 (probably a unit error), see appendix 3. A large variation is observed when the reported surface area is compared with the used simulant volume (see figure 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. Five participants reported the sample mass in mg for a part of the evaporated simulant, but the total residue result could be calculated by iis from the reported total volume of simulant used and the partial volume of the simulant evaporated.

Calculation of Overall Migration in mg/kg food simulant

EN1186-3 does not describe reporting migration in mg/kg. However, 37 laboratories reported a test result in mg/kg food simulant of which 13 used a conversion factor of 6 dm^2/kg in accordance with EN1186-1, paragraph 12.1, while 18 other participants used a calculation comparable to the calculation mentioned in method EN1186-9, see appendix 3. The variety of calculation methods used lead to a large variation in the test results in mg/kg.

Calculation of Overall Migration in mg/L food simulant

EN1186-3 does not describe reporting migration in mg/L. However, 30 participants, except one, reported the same numerical result for both the migration in mg/kg and mg/L.

Limits for overall migration from EU regulation No 10/2011

This EU regulation describes in article 12 that the limit for overall migration is 10 mg/dm2. According to this limit 92% of the participants would have rejected the spatula. Should this spatula be used in applications that will allow infants and children's food to come in contact, then an overall migration limit of 60 mg/kg is applied. Using this limit and based on the reported results, 82% of the participants would have rejected the spatula, while 18% would have accepted it.

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 predetermined but will be selected by the individual laboratories. The high variation in the amount of simulant volume used and/or in the determined surface area will also have a negative effect on the variation of the test results.

The salad bowl was also used in the proficiency test of 2015. Although the test conditions slightly deviate and some improvement was made as the number of excluded laboratories (16 vs 8 for mg/dm^2 and 19 vs 4 for mg/kg) and statistical outliers decreased, this means that more laboratories followed the same test method and therefore may be better compared to each other. Regretfully, the uncertainties were somewhat larger compared with the previous proficiency test.

In 2013 a proficiency test on Overall migration (total immersion) was organized, which can be compared with sample #16616 (Spatula) used in the 2016 PT. Regretfully, no improvement was visible. The variation of the 2016 PT is smaller, but nine results were excluded for statistical evaluation in the 2016 PT, compared to none in 2013.

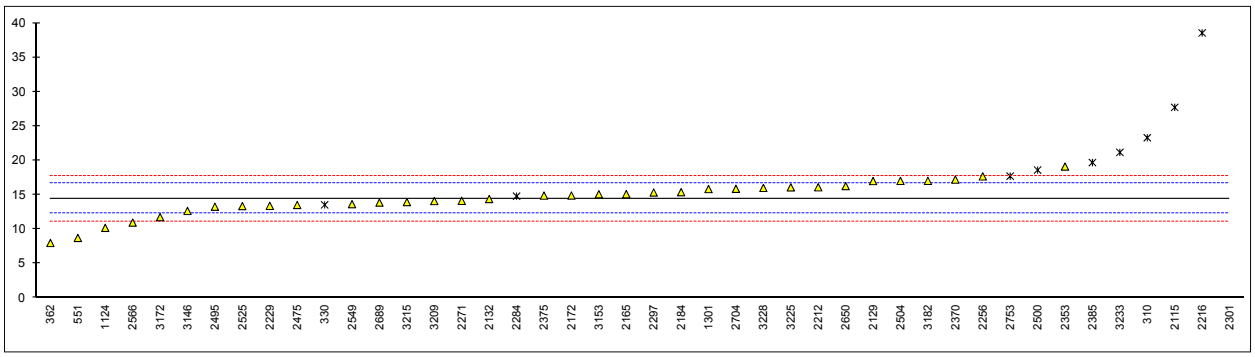
Each laboratory has to evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and the quality of the analytical results.

APPENDIX 1:

Determination of Overall Migration on sample #16615 (Bowl); results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
310	EN1186-9	23.3	ex	7.96	Excluded due to low volume, see §5
330	EN1186-9	13.535	ex	-0.84	Excluded due to low volume, see §5
362	EN1186-9	8.00		-5.82	
452		-----		-----	
551	EN1186-9	8.725	C	-5.17	First reported 6.137
622		-----		-----	
1124	EN1186-9	10.19		-3.85	
1179		-----		-----	
1301		15.84		1.24	
2115	EN1186-9	27.72	ex	11.94	Excluded due to large surface area, see §5
2129	EN1186-9	17		2.28	
2132	EN1186-9	14.39		-0.07	
2156		-----		-----	
2165	EN1186-9	15.1		0.57	
2172	EN1186-9	14.90		0.39	
2184	EN1186-9	15.3846		0.83	
2212	21 CFR175.300	16.1		1.47	
2216	21 CFR175.300	38.549	R(0.01)	21.69	
2229	EN1186-9	13.4		-0.96	
2256	EN1186-9	17.68		2.90	
2271	EN1186-9	14.12		-0.31	
2284	EN1186-9	14.8	ex	0.30	Excluded due to small surface area, see §5
2297	EN1186-9	15.34		0.79	
2301	EN1186-9	99.745	R(0.01)	76.80	reported small surface area
2353	EN1186-9	19.11		4.18	
2370	EN1186-9	17.22		2.48	
2375	EN1186-9	14.89		0.38	
2385	EN1186-9	19.69	ex	4.71	Excluded due to low volume, see §5
2463		-----		-----	
2475	EN1186-9	13.52		-0.85	
2495	EN1186-9	13.267		-1.08	
2500	EN1186-9	18.6	ex	3.72	Excluded due to small surface area, see §5
2504	EN1186-9	17.024		2.30	
2525	EN1186-9	13.36		-1.00	
2549	EN1186-9	13.64		-0.74	
2566	EN1186-9	10.961		-3.16	
2650	EN1186-9	16.27		1.63	
2689	EN1186-9	13.87		-0.54	
2704	EN1186-9	15.873		1.27	
2705		-----		-----	
2707		-----		-----	
2747		-----		-----	
2753	EN1186-9	17.71	ex	2.92	Excluded due to small surface area, see §5
2764		-----		-----	
3146	EN1186-9	12.657		-1.63	
3153	EN1186-9	15.08		0.55	
3172	EN1186-9	11.752		-2.44	
3182	EN1186-9	17.03		2.31	
3209	EN1186-9	14.09		-0.34	
3215	EN1186-9	13.94		-0.47	
3225	EN1186-9	16.0858		1.46	
3228	EN1186-9	16.0		1.38	
3233	EN1186-9	21.17	ex	6.04	Excluded due to low volume, see §5
3237		-----		-----	

normality	OK	
n	34	
outliers	2 (+8 excl)	
mean (n)	14.465	
st.dev. (n)	2.4544	RSD% = 17%
R(calc.)	6.872	
R(EN1186-9:02)	3.109	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35
30
25
20
15
10
5
0
362
551
1124
2566
3172
3146
2495
2525
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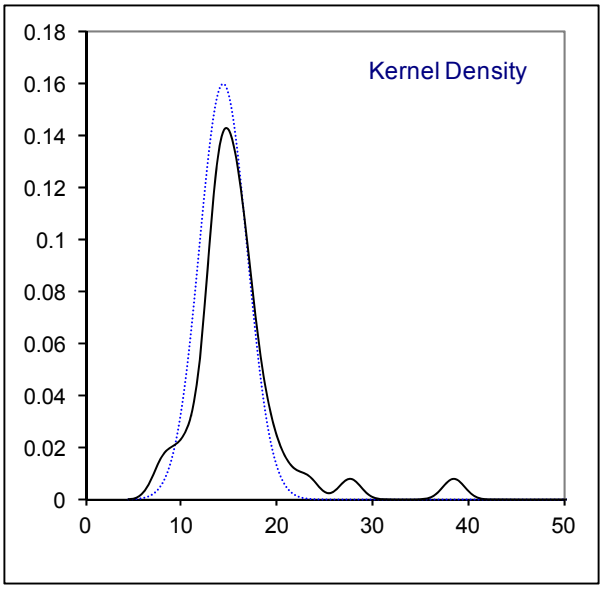
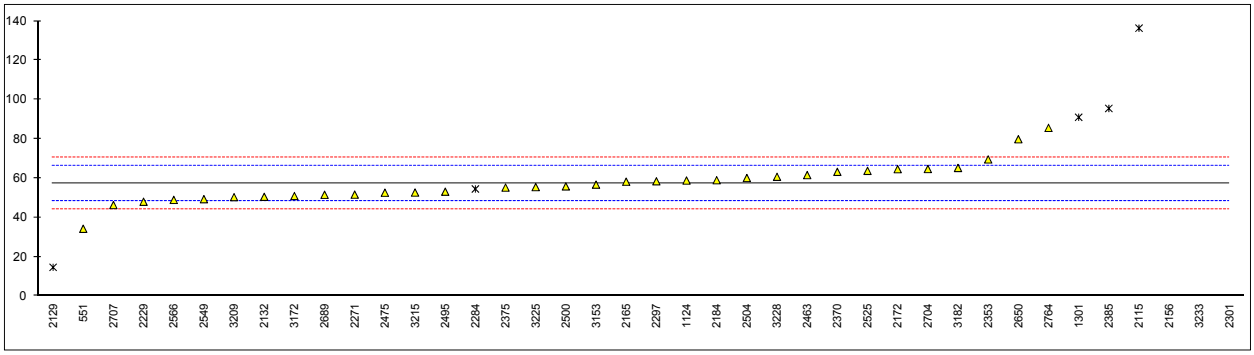
330
2549
2689
3215
3209
2271
2132
2284
2375
2172
3153
2165
2297
2184
1301
2704
3228
3225
2212
2650
2129
2504
3182
2370
2256
2753
2500
2353
2385
3233
310
2115
2216
2301


Kernel Density
0.18
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
0
10
20
30
40
50

Determination of Overall Migration on sample #16615 (Bowl); results in mg/kg food stimulant

lab	method	value	mark	z(targ)	remarks
310		-----		-----	
330		-----		-----	
362		-----		-----	
452		-----		-----	
551	EN1186-9	34.231	C	-5.26	First reported 24.077
622		-----		-----	
1124	EN1186-9	58.80		0.32	
1179		-----		-----	
1301	EN1186-9	91	C,ex	7.63	excluded used factor 6, see§5, first reported 210.73
2115	EN1186-9	136.36	R(0.01)	17.93	
2129	EN1186-9	14.6	C,E,ex	-9.71	excluded, results could not be calc by iis, f.r. 146
2132	EN1186-9	50.51		-1.56	
2156	EN1186-9	158.3	R(0.01)	22.91	
2165	EN1186-9	58.2		0.18	
2172	EN1186-9	64.59		1.64	
2184	EN1186-9	58.9615		0.36	
2212		-----		-----	
2216		-----		-----	
2229	EN1186-9	48.0		-2.13	
2256		-----		-----	
2271	EN1186-9	51.62		-1.31	
2284	EN1186-9	54.5	E,ex	-0.65	excluded, results could not be calc by iis
2297	EN1186-9	58.40		0.23	
2301	EN1186-9	326.136	R(0.01)	61.00	
2353	EN1186-9	69.55		2.76	
2370	EN1186-9	63.21		1.32	Reported only in mg/L
2375	EN1186-9	55.22		-0.49	
2385	EN1186-9	95.50	R(0.01)	8.65	Reported use of low volume and reported only in mg/L
2463	EN1186-9	61.55		0.95	
2475	EN1186-9	52.60		-1.09	
2495	EN1186-9	53.111		-0.97	
2500	EN1186-9	55.8		-0.36	
2504	EN1186-9	60.070	C	0.61	First reported 102.145
2525	EN1186-9	63.73		1.44	
2549	EN1186-9	49.31		-1.83	
2566	EN1186-9	48.93		-1.92	
2650	EN1186-9	79.83		5.09	
2689	EN1186-9	51.53		-1.33	
2704	EN1186-9	64.67		1.65	
2705		-----		-----	
2707	EN1186-9	46.3		-2.52	
2747		-----		-----	
2753		-----		-----	
2764	EN1186-9	85.6	C	6.40	First reported 212.5
3146		-----		-----	
3153	EN1186-9	56.69		-0.16	
3172	EN1186-9	50.925		-1.47	
3182	EN1186-9	65.20		1.77	
3209	EN1186-9	50.34		-1.60	
3215	EN1186-9	52.69		-1.07	
3225	EN1186-9	55.4568		-0.44	
3228	EN1186-9	60.7		0.75	
3233	EN1186-9	218.00	R(0.01)	36.46	Reported use of low volume
3237		-----		-----	

normality	not OK		
n	32		
outliers	5 (+ 3 excl)		
mean (n)	57.385	RSD% =	17%
st.dev. (n)	9.6626		
R(calc.)	27.055		
R(EN1186-9:02)	12.335		


140
120
100
80
60
40
20
0
2129
551
2707
2229
2566
2549
3209
2132
3172
2689
2271
2475
3215
2495
2284
2375
3225
2500
3153
2165
2297
1124
2184
2504
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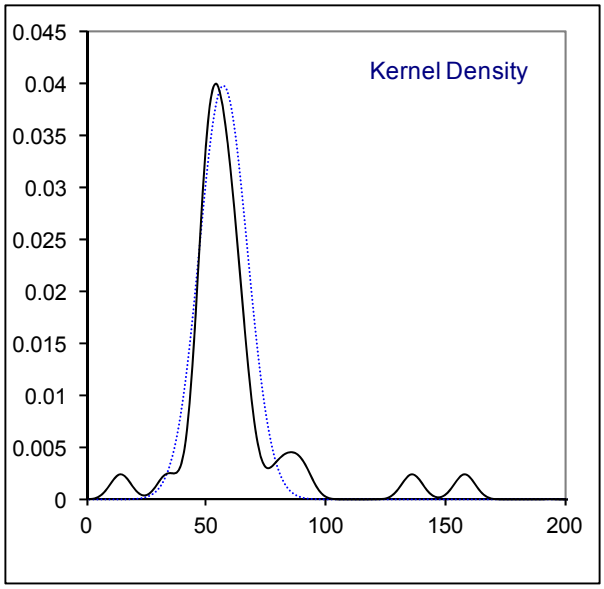
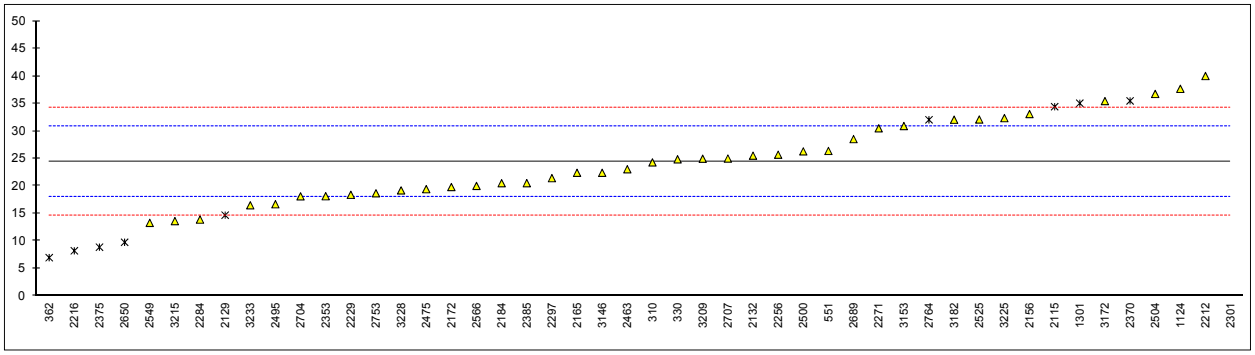
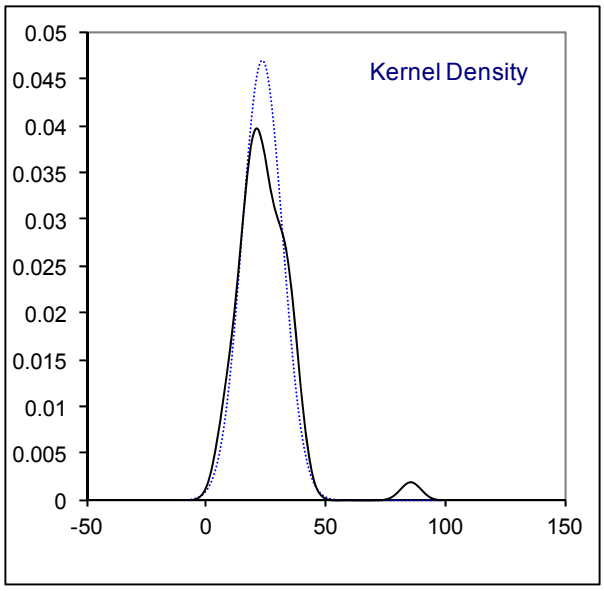
2650
2764
1301
2385
2115
2156
3233
2301


Kernel Density
0.045
0.04
0.035
0.03
0.025
0.02
0.015
0.01
0.005
0
0
50
100
150
200

Determination of Overall Migration on sample #16616 (Spatula); results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
310	EN1186-3	24.3		-0.04	
330	EN1186-3	24.857		0.13	
362		6.97	R(0.05)	-5.38	
452		-----		-----	
551	EN1186-3	26.375		0.60	
622		-----		-----	
1124	EN1186-3	37.68		4.08	
1179		-----		-----	
1301	EN1186-3	35.05	ex	3.27	Result excluded due to small contact surface area
2115	EN1186-3	34.42	ex	3.07	Result excluded, due to small contact surface area
2129	EN1186-3	14.7	E,ex	-3.00	excluded, results could not be calc by iis
2132	EN1186-3	25.52		0.33	
2156	EN1186-3	33.09		2.66	
2165	EN1186-3	22.4		-0.63	
2172	EN1186-3	19.81		-1.42	
2184	EN1186-3	20.5128		-1.21	
2212	21 CFR175.300	40.0		4.79	
2216	21 CFR175.300	8.2191	ex	-4.99	Result excluded due to small contact surface area
2229	EN1186-3	18.4		-1.86	
2256	EN1186-3	25.69		0.39	
2271	EN1186-3	30.5		1.87	
2284	EN1186-3	13.9		-3.24	
2297	EN1186-3	21.43		-0.93	
2301	EN1186-3	85.752	R(0.01)	18.87	Reported to have used an excessive amount of simulant
2353	EN1186-3	18.15		-1.93	
2370	EN1186-3	35.48	ex	3.40	Result excluded, evaporated only 9 ml
2375	EN1186-3	8.88	ex	-4.79	Result excluded, used excessive amount of simulant
2385	EN1186-3	20.52		-1.21	
2463	EN1186-3	23.05		-0.43	
2475	EN1186-3	19.43		-1.54	
2495	EN1186-3	16.686		-2.39	
2500	EN1186-3	26.3		0.57	
2504	EN1186-3	36.745		3.79	
2525	EN1186-3	32.1		2.36	
2549	EN1186-3	13.30		-3.43	
2566	EN1186-3	20.02		-1.36	
2650	EN1186-3	9.77	C,ex	-4.51	Result excluded due to large contact surface area, f.r. 0.51
2689	EN1186-3	28.53		1.26	
2704	EN1186-3	18.13		-1.94	
2705		-----		-----	
2707	EN1186-3	25.0		0.17	
2747		-----		-----	
2753	EN1186-3	18.68		-1.77	
2764	In house	32.04	ex	2.34	Result excluded due to small contact surface area
3146	EN1186-3	22.4		-0.63	
3153	EN1186-3	30.91		1.99	
3172	EN1186-3	35.44		3.39	
3182	EN1186-3	32.06		2.35	
3209	EN1186-3	24.97		0.16	
3215	EN1186-3	13.62		-3.33	
3225	EN1186-3	32.3490		2.44	
3228	EN1186-3	19.2		-1.61	
3233	EN1186-3	16.50		-2.44	
3237		-----		-----	
					Only 1dm^2 :100 ml ratio
	normality	OK			OK
	n	38			12
	outliers	2 (+8 excl)			0
	mean (n)	24.436			24.213
	st.dev. (n)	7.0098	RSD%=29%		7.9295
	R(calc.)	19.627			22.203
	R(EN1186-3:02)	9.098			9.015


50
45
40
35
30
25
20
15
10
5
0
362 2216 2375 2650 2549 3215 2284 2129 3233 2495 2704 2353 2229 2753 3228 2475 2172 2566 2184 2385 2297 2165 3146 2463 310 330 3209 2707 2132 2256 2500 551 2689 2271 3153 2764 3182 2525 3225 2156 2115 1301 3172 2370 2504 1124 2212 2301


Kernel Density
0.05
0.045
0.04
0.035
0.03
0.025
0.02
0.015
0.01
0.005
0
-50
0
50
100
150

APPENDIX 2

Details BOWL reported by participating laboratories on residue, volume of simulant and surface area

lab	reported residue (mg)	volume simulant (ml)	volume simulant evap. (ml)	surface area (dm^2)	How was the evaporation done?
310	12.60	600	100	3.24	---
330	29.1	200	200	2.15	Evaporation of simulant in several small volumes
362	44.3	1000	1000	5.54	---
452	-----				---
551	44.50	1300	1300	5.1	Evaporation of simulant in several small volumes
622	223.34	1000	± 60	Art. filling	Evaporation of simulant in one step
1124	58.80	1000	1000	5.77	Evaporation of simulant in several small volumes
1179	-----				---
1301	-----	1100	200	1100	---
2115	-----	1300	50	6.4	Evaporation of simulant in several small volumes
2129	5.84	1250	50	5.3	Evaporation of simulant in one step
2132	69.20	1370	50	4.81	Evaporation of simulant in several small volumes
2156	213.6	1350	200	--	Evaporation of simulant in one step
2165	75.7	1300	1300	5.01	Evaporation of simulant in several small volumes
2172	87.2	1350	1350	5.85	Evaporation of simulant in several small volumes
2184	76.0	1300	1300	4.94	Evaporation of simulant in one step
2212	85.8	1400	200	5.35	Evaporation of simulant in one step
2216	199.3	1300	1300	5.17	---
2229	64.8	1350	1350	4.82	Evaporation of simulant in one step
2256	15.42	1250	200	5.4454	Evaporation of simulant in one step
2271	69.71	1350	100	4.94	---
2284	130.08	1200	500	4.41	Evaporation of simulant in several small volumes
2297	75.92	1300	1300	4.95	Evaporation of simulant in one step
2301	430.500	1320	50	4.316	---
2353	97.37	1400	200	5.0941	Evaporation of simulant in several small volumes
2370	84.7	1340	6	4.92	Evaporation of simulant in one step
2375	0.0805	1300	200	5	Evaporation of simulant in several small volumes
2385	20.1	650	200	3.16	Evaporation of simulant in one step
2463	12.31	1300	200		Evaporation of simulant in one step
2475	-----	1250	200	4.87	Evaporation of simulant in one step
2495	63.73	1200	1200	4.804	Evaporation of simulant in one step
2500	66.9	1200	200	3.6	Evaporation of simulant in one step
2504	30.033	1405	500	4.96	Evaporation of simulant in one step
2525	63.73	1000	1000	4,77	Evaporation of simulant in several small volumes
2549	64.10	1300	1300	4.7	Evaporation of simulant in one step
2566	63.96	1320	nil	5.835	Evaporation of simulant in several small volumes
2650	96	1200	200	5.9	Evaporation of simulant in several small volumes
2689	66.99	1300	250	4.83	Evaporation of simulant in one step
2704	87.3	1350	200	5.5	Evaporation of simulant in several small volumes
2705	-----				---
2707	-----	1350	200	4.94	Evaporation of simulant in one step
2747	-----				---
2753	76.17	1350	1350	4.3	Evaporation of simulant in several small volumes
2764	-----	1400	200		Evaporation of simulant in several small volumes
3146	70.5	1390	0	5.57	Evaporation of simulant in one step
3153	73.7	1300	1300	4.9	Evaporation of simulant in one step
3172	68.75	1350	1350	5.85	Evaporation of simulant in one step
3182	92.120	1400	500	5.36	Evaporation of simulant in one step
3209	73.10	1450	1450	5.18	Evaporation of simulant in one step
3215	68.50	1300	1300	4.914	Evaporation of simulant in one step
3225	75.67	1350	1350	4.65	Evaporation of simulant in one step
3228	78.88	1300	200	4.93	Evaporation of simulant in one step
3233	21.80	200	100	2.06	Evaporation of simulant in one step
3237	-----				---

Details reported by the participating laboratories on preparation and evaporation

lab	Was sample cleaned prior to the migration step?	cleaned with
310	No	
330	No	
362	No	
452	---	
551	Yes	with a lint-free cloth
622	No	
1124	Yes	Rinsed with distilled water
1179	---	
1301	Yes	
2115	No	
2129	No	
2132	Yes	Lint-free cloth
2156	Yes	Soft Brush
2165	No	
2172	No	
2184	No	
2212	Yes	Wipe sample with lint-free cloth gently
2216	No	
2229	No	
2256	Yes	wiped gently with a lint-free cloth.
2271	Yes	1*100ml
2284	No	
2297	Yes	
2301	Yes	Cleaned prior the sample with Dist. water, 2*500mL and 1*320mL
2353	Yes	Brush to remove dust
2370	Yes	The sample was purged with comp. air to remove any dust on it.
2375	No	
2385	Yes	Water
2463	Yes	
2475	No	
2495	Yes	cleaned with dry paper
2500	No	
2504	Yes	clean with tissue paper
2525	No	
2549	No	
2566	Yes	with the lint free cloth
2650	No	
2689	No	
2704	No	
2705	---	
2707	No	
2747	---	
2753	Yes	water
2764	Yes	
3146	No	
3153	Yes	Wiping the sample with a lint-free cloth
3172	No	
3182	Yes	Wipe with kimwipe paper
3209	Yes	Distilled water
3215	No	
3225	Yes	Cleaned by gently wiping with Kimwipes
3228	Yes	The sample was cleaned with soft cloth
3233	Yes	the sample was cleaned with a lint-free cloth
3237	---	

Calculations used by participating laboratories on Overall Migration on sample #16615 in mg/kg and mg/L

lab	method of calculation	reported result migration (mg/kg)	method of calculation	reported result migration (mg/L)	remarks
310	-----	-----	-----	-----	
330	-----	-----	-----	-----	
362	-----	-----	-----	-----	
452	-----	-----	-----	-----	
551	EN1186-9	34.231	-----	34.231	mg/kg = mg/L
622	-----	-----	-----	-----	
1124	EN1186-9	58.80	-----	58.80	mg/kg = mg/L
1179	-----	-----	-----	-----	
1301	Factor 6	91	-----	-----	calculation error: iis calc: 95 mg/kg
2115	-----	136.36	-----	136.36	mg/kg = mg/L
2129	-----	14.6	-----	-----	results could not be calculated by iis
2132	EN1186-9	50.51	-----	50.51	mg/kg = mg/L
2156	EN1186-9	158.3	-----	158.3	mg/kg = mg/L
2165	EN1186-9	58.2	-----	58.2	mg/kg = mg/L
2172	EN1186-9	64.59	-----	64.59	mg/kg = mg/L
2184	EN1186-9	58.9615	-----	58.9615	mg/kg = mg/L
2212	-----	-----	-----	-----	
2216	-----	-----	-----	-----	
2229	EN1186-9	48.0	-----	48.0	mg/kg = mg/L
2256	-----	-----	-----	-----	
2271	EN1186-9	51.62	-----	51.62	mg/kg = mg/L
2284		54.5	-----	54.5	results could not be calculated by iis
2297	EN1186-9	58.40	-----	58.40	mg/kg = mg/L
2301	EN1186-9	326.136	-----	326.136	mg/kg = mg/L
2353	EN1186-9	69.55	-----	69.55	mg/kg = mg/L
2370		-----	EN1186-9	63.21	mg/kg = mg/L
2375	EN1186-9	55.22	-----	55.22	calculation error: iis calc.: 57.25 mg/kg; mg/kg = mg/L
2385		-----	EN1186-9	95.50	calculation error: iis calc.: 100.5 mg/kg;
2463	EN1186-9	61.55	-----	-----	
2475	EN1186-9	52.60	-----	52.60	mg/kg = mg/L
2495	EN1186-9	53.111	-----	53.111	mg/kg = mg/L
2500	EN1186-9	55.8	-----	-----	
2504	EN1186-9	60.070	-----	60.070	mg/kg = mg/L
2525	EN1186-9	63.73	-----	63.73	mg/kg = mg/L
2549	EN1186-9	49.31	-----	49.31	mg/kg = mg/L
2566	EN1186-9	48.93	-----	48.93	mg/kg = mg/L
2650	EN1186-9	79.83	-----	79.83	mg/kg = mg/L
2689	EN1186-9	51.53	-----	51.53	mg/kg = mg/L
2704	EN1186-9	64.67	-----	64.67	mg/kg = mg/L
2705	-----	-----	-----	-----	
2707	EN1186-9	46.3	-----	-----	
2747	-----	-----	-----	-----	
2753	-----	-----	-----	-----	
2764	EN1186-9	85.6	-----	-----	
3146	-----	-----	-----	-----	
3153	EN1186-9	56.69	-----	56.69	mg/kg = mg/L
3172	EN1186-9	50.925	-----	50.925	mg/kg = mg/L
3182	EN1186-9	65.20	-----	65.20	mg/kg = mg/L
3209	EN1186-9	50.34	-----	50.34	mg/kg = mg/L
3215	EN1186-9	52.69	-----	52.69	mg/kg = mg/L
3225	EN1186-9	55.4568	-----	-----	
3228	EN1186-9	60.7	-----	60.7	mg/kg = mg/L
3233	EN1186-9	218.00	-----	218.00	mg/kg = mg/L
3237	-----	-----	-----	-----	

APPENDIX 3

Details SPATULA reported by participating laboratories on residue, volume of simulant and surface area

lab	reported residue (mg)	volume simulant (ml)	volume simulant evap. (ml)	surface area (dm^2)	Ratio (dm^2/ 100ml)	How was the evaporation done?
310	19.65	200	100	1.62	0.81	Evaporation of simulant in one step
330	20.567	80	80	0.8	1.00	Evaporation of simulant in several small volumes
362	8.30	1000	1000	1.19	0.12	---
452	--				-----	---
551	42.2	160	160	1.6	1.00	Evaporation of simulant in one step
622	253.33	130	30	--	-----	Evaporation of simulant in one step
1124	52.00	140	140	1.38	0.99	Evaporation of simulant in several small volumes
1179	--	--	--	--	-----	---
1301	--	150	150	0.64	0.43	---
2115	--	100	-	0.43	0.43	Evaporation of simulant in several small volumes
2129	4.7	500	50	1.6	0.32	Evaporation of simulant in one step
2132	38.20	600	600	1.5	0.25	Evaporation of simulant in several small volumes
2156	46.00	100	95	1.39	1.39	Evaporation of simulant in one step
2165	34.4	257	257	1.54	0.60	Evaporation of simulant in several small volumes
2172	31.9	161	161	1.61	1.00	Evaporation of simulant in one step
2184	32.0	260	260	1.56	0.60	Evaporation of simulant in one step
2212	40.6	100	100	1.03	1.03	Evaporation of simulant in one step
2216	--			0.657	-----	---
2229	30.2	164	164	1.64	1.00	Evaporation of simulant in one step
2256	35.76	100	100	1.3935	1.39	Evaporation of simulant in one step
2271	41.2	225	100	1.35	0.60	
2284	25.9	190	190	1.86	0.98	Evaporation of simulant in one step
2297	30.22	250	250	1.41	0.56	Evaporation of simulant in one step
2301	143.000	2750	50	1.667	0.06	
2353	27.24	300	300	1.5012	0.50	Evaporation of simulant in several small volumes
2370	62.27	450	9	1.755	0.39	Evaporation of simulant in one step
2375	4.42	1500	200	2.49	0.17	Evaporation of simulant in several small volumes
2385	27.5	200	200	1.34	0.67	Evaporation of simulant in one step
2463	23.63	100	100	1.025	1.03	Evaporation of simulant in one step
2475	34.2	800	800	1.76	0.22	Evaporation of simulant in several small volumes
2495	22.69	500	500	1.36	0.27	Evaporation of simulant in one step
2500	26.3	100	100	1	1.00	Evaporation of simulant in one step
2504	32.3	600	400	1.32	0.22	
2525	45.8	500	500	1.43	0.29	Evaporation of simulant in several small volumes
2549	25.00	750	750	1.88	0.25	Evaporation of simulant in one step
2566	35.25	300	20	1.76	0.59	Evaporation of simulant in one step
2650	40.84	800	800	80.3	10.04	Evaporation of simulant in several small volumes
2689	42.79	250	250	1.5	0.60	Evaporation of simulant in one step
2704	32.1	600	200	1.77	0.30	Evaporation of simulant in several small volumes
2705	--				-----	---
2707	--	100		1	1.00	Evaporation of simulant in one step
2747	--				-----	---
2753	31.20	167	167	1.67	1.00	Evaporation of simulant in several small volumes
2764	--			0.1186	-----	Evaporation of simulant in one step
3146	38	950	950	1.7	0.18	Evaporation of simulant in one step
3153	50.8	800	800	1.6	0.20	Evaporation of simulant in one step
3172	48.2	550	550	1.36	0.25	Evaporation of simulant in one step
3182	50.2	800	800	1.55	0.19	Evaporation of simulant in one step
3209	45.00	300	300	1.8	0.60	Evaporation of simulant in one step
3215	22.80	530	530	1.674	0.32	Evaporation of simulant in one step
3225	48.2	250	250	1.49	0.60	Evaporation of simulant in one step
3228	29.18	250	250	1.52	0.61	Evaporation of simulant in one step
3233	16.50	100	100	1	1.00	Evaporation of simulant in one step
3237	--	--	--	--	--	---

Details reported by the participating laboratories on preparation and evaporation

lab	Was sample cleaned prior to the migration step?	cleaned with
310	No	
330	No	
362	No	
452	---	
551	Yes	with a lint free cloth
622	No	
1124	No	
1179	---	
1301	Yes	
2115	No	
2129	No	
2132	Yes	Lint-free cloth
2156	Yes	soft brush
2165	No	
2172	No	
2184	No	
2212	Yes	Wipe the sample with lint-free cloth gently
2216	No	
2229	No	
2256	Yes	The sample was wiped gently with a lint-free cloth.
2271	Yes	1*100
2284	No	
2297	Yes	
2301	Yes	With Distilled water, (5x500) mL and (1x200) mL
2353	Yes	Brush to remove dust
2370	Yes	purged with compressed air stream to remove any dust
2375	No	-
2385	Yes	Water
2463	Yes	
2475	No	
2495	Yes	cleaned with dry paper
2500	No	
2504	Yes	Clean with Tissue paper, evaporation 1x400 mL
2525	No	
2549	No	
2566	Yes	with lint free cloth
2650	No	
2689	No	
2704	No	
2705	---	
2707	No	
2747	---	
2753	Yes	Water
2764	Yes	
3146	No	
3153	Yes	Wiping the sample with lint-free cloth
3172	No	
3182	Yes	With kimwipe paper
3209	Yes	Distilled water
3215	No	
3225	Yes	Cleaned by gently wiping with Kimwipes
3228	Yes	cleaned with soft cloth.
3233	Yes	The sample was cleaned with a lint-free cloth
3237	---	

Calculations used by participating laboratories on Overall Migration on sample #16616 in mg/kg and mg/L

lab	method of calculation	reported result migration (mg/kg)	method of calculation	reported result migration (mg/L)	remarks
310	-----	-----	-----	-----	
330	-----	-----	-----	-----	
362	-----	-----	-----	-----	
452	-----	-----	-----	-----	
551	EN1186-9	263.75	-----	263.75	mg/kg = mg/L
622	-----	-----	-----	-----	
1124	EN1186-9	269.14	-----	269.14	mg/kg = mg/L
1179	-----	-----	-----	-----	
1301	-----	-----	-----	-----	
2115	-----	-----	-----	-----	
2129	-----	47	-----	-----	result could not be calculated by iis
2132	EN1186-9	63.67	-----	63.67	mg/kg = mg/L
2156	EN1186-9	460.0	-----	460.0	mg/kg = mg/L
2165	Factor 6	134.2	-----	134.2	mg/kg = mg/L
2172	EN1186-9	198.1	-----	198.1	mg/kg = mg/L
2184	Factor 6	123.0769	-----	123.0769	mg/kg = mg/L
2212	-----	-----	-----	-----	
2216	-----	-----	-----	-----	
2229	Factor 6	110.5	-----	-----	
2256	-----	-----	-----	-----	
2271	Factor 6	183.1	-----	183.1	mg/kg = mg/L
2284	EN1189-9	136.3	-----	136.3	mg/kg = mg/L
2297	EN1186-9	120.9	-----	120.9	mg/kg = mg/L
2301	EN1186-9	52.000	-----	52.000	mg/kg = mg/L
2353	Factor 6	108.9	-----	108.9	mg/kg = mg/L
2370	-----	-----	-----	138.4	result could not be calculated by iis
2375	Factor 6	53.86	-----	53.86	mg/kg = mg/L
2385	-----	-----	EN1186-9	137.5	
2463	-----	-----	-----	-----	
2475	-----	-----	-----	-----	
2495	EN1186-9	45.387	-----	45.387	mg/kg = mg/L
2500	-----	-----	-----	-----	
2504	Factor 6	220.470	-----	12.9200	
2525	EN1186-9	91.6	-----	91.6	mg/kg = mg/L
2549	EN1186-9	33.33	-----	33.33	mg/kg = mg/L
2566	EN1186-9	66.73	-----	66.73	mg/kg = mg/L
2650	EN1186-9	51.05	-----	51.05	mg/kg = mg/L
2689	Factor 6	171.16	-----	171.16	mg/kg = mg/L
2704	Factor 6	108.6	-----	108.6	mg/kg = mg/L
2705	-----	-----	-----	-----	
2707	-----	-----	-----	-----	
2747	-----	-----	-----	-----	
2753	-----	-----	-----	-----	
2764	-----	-----	-----	-----	
3146	-----	-----	-----	-----	
3153	EN1186-9	63.50	-----	63.50	mg/kg = mg/L
3172	Factor 6	212.64	-----	212.64	mg/kg = mg/L
3182	EN1186-9	62.13	-----	62.13	mg/kg = mg/L
3209	Factor 6	149.83	-----	149.83	mg/kg = mg/L
3215	EN1186-9	43.02	-----	43.02	mg/kg = mg/L
3225	EN1186-9	192.8000	-----	-----	
3228	Factor 6	115.2	-----	115.2	mg/kg = mg/L
3233	Factor 6	99.00	-----	99.00	mg/kg = mg/L
3237	-----	-----	-----	-----	

APPENDIX 4

Number of participating laboratories per country

1 lab in	BRAZIL
1 lab in	BULGARIA
3 labs in	FRANCE
4 labs in	GERMANY
6 labs in	HONG KONG
2 labs in	INDIA
2 labs in	INDONESIA
4 labs in	ITALY
1 lab in	LATVIA
1 lab in	LUXEMBOURG
1 lab in	MALAYSIA
15 labs in	P.R. of CHINA
1 lab in	QATAR
1 lab in	SPAIN
1 lab in	TAIWAN R.O.C.
2 labs in	THAILAND
2 labs in	THE NETHERLANDS
2 labs in	TURKEY
2 labs in	U.A.E.
1 lab in	U.S.A.
1 lab in	UNITED KINGDOM

APPENDIX 5

Abbreviations:

C = final 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
n.a. = not applicable
E = probably an error in calculation
W = test result withdrawn on request of participant
f.r. = first reported

Literature:

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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
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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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