Results of Proficiency Test
Overall migration (fcm)
September 2017

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. G.A. Oosterlaken-Buijs
Correctors: dr. R.G. Visser & ing. R.J. Starink

Report: iis17P09GM

December 2017

CONTENTS

1 INTRODUCTION

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.

Since 2012, a proficiency test for Overall migration is organised every year by the Institute for Interlaboratory Studies (iis). During the annual proficiency testing program 2017/2018, it was decided to continue with the interlaboratory study for the determination of Overall migration on food contact materials.
In this interlaboratory study 47 laboratories from 22 different countries registered for participation. See appendix 5 for the number of participants per country. In this report, the results of the 2017 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 1 sample (a melamine spoon, labelled #17620) which was tested and found to be positive on migration. Furthermore, a number of test conditions (migration method, type of simulant, exposure time and temperature) were prescribed to be used. 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 March 2017 (iis-protocol, version 3.4). 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 melamine spoons for multiple 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 (1st step) on 8 stratified randomly selected test items.

	Overall Migration (1st step) in mg/dm^2 (3% acetic acid, 4 hrs. at 100°C)
Sample #17620-1	33.1
Sample #17620-2	34.3
Sample #17620-3	31.9
Sample #17620-4	34.3
Sample #17620-5	30.7
Sample #17620-6	34.9
Sample #17620-7	31.3
Sample #17620-8	35.5

Table 1: homogeneity test results of subsamples #17620

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 (1st step) in mg/dm^2 (3% acetic acid, 4 hrs. at 100°C)
r(observed)	5.0
reference test method	EN1186-9:2002
0.3xR(reference test method)	4.8
R(reference test method)	16.0

Table 2: evaluation of the repeatability of subsamples #17620

The calculated repeatability was in agreement with 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the samples #17620 was assumed.

To each of the participating laboratories one sample #17620 (spoon) was sent on September 6, 2017.

2.5 ANALYSES

The participants were requested to determine Overall Migration on sample #17620 (melamine spoon) using the prescribed test conditions (total immersion, repeated use as migration method, 4hrs at 100°C and 3% M/V acetic acid as simulant).
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 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 March 2017 (iis-protocol, version 3.4).
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. 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 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. Two participants did not report any test results at all and all other participants reported in time. Finally, the 45 reporting laboratories reported 124 numerical test results for overall migration per contact surface and 117 numerical test results for overall migration per kg food simulant. For overall migration per contact surface 4 statistically outlying results were observed, which is 3.2%. 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 total immersion used for the spoon, the EN1186 method series part 3 is considered to be the official EC test method. The target reproducibility used for statistical evaluation was estimated from the EN1186-3 (Annex A) reproducibility of simulants A, B and C (based on 3 replicates).

In Method EN1186-3 it is described that five samples are needed for articles of irregular shape: two samples to determine the surface area and three sample for the migration test. In this 2017 PT only one sample (a melamine spoon) was available for both surface area determination and the migration test.
In this PT, as mentioned in the letter of instructions, the migration method was total immersion for repeated use and 3% M/V acetic acid as simulant for 4 hrs. at 100°C. The participants were requested to report the test results of all three successive migration steps.

Nearly all participants reported to have used part 3 of the EN1186 test method for the spoon (sample #17620). The reported details of the methods that were used by the participants are listed in appendices 2 and 4.

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 6.

Residue in mg: These intermediate results were not evaluated as they are in principle dependent on the size of the volume of simulant used.
Four participants reported to have evaporated only a part of the simulant used (see appendices 2 and 4), although the EN1189-3 test method describes that all of the simulant used should be evaporated to determine the residue.
An overview of the reported test results per step in mg can be found in appendix 2.

Migration in mg/dm^2: This determination was problematic. Some test results were excluded because deviation ratios (surface area/volume) were used (see paragraph 4.4).
Overall migration - 1st step:
Two statistical outliers were observed and eleven test results were excluded, see the discussion in paragraph 4.4. The calculated reproducibility after rejection of the suspect data is not in agreement with the target reproducibility estimated from EN1186-3:02.
Overall migration – 2nd step:
One statistical outlier was observed and nine test results were excluded, see the discussion in paragraph 4.4. The calculated reproducibility after rejection of the suspect data is not in agreement with the target reproducibility estimated from EN1186-3:02.

Overall migration – 3rd step:
One statistical outlier was observed and nine test results were excluded, see the discussion in paragraph 4.4. The calculated reproducibility after rejection of the suspect data 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 per step in mg/kg is given in appendices 2 and 3.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

The calculated reproducibilities and the target reproducibilities derived from the literature standard method EN1189-3 are compared in the next tables.

	unit	n	Average	2.8 * sd	R (target)
Overall migration – 1st step	mg/dm^2	31	30.88	27.57	14.89
Overall migration – 2nd step	mg/dm^2	29	55.42	51.91	26.73
Overall migration – 3rd step	mg/dm^2	31	64.16	64.44	30.95

Table 3: performance overview for samples #17620

Without further statistical calculations, it can be concluded that there is no good compliance of the group of participating laboratories with the target reproducibility estimated from EN1186-3:02.

4.3 COMPARISON OF PROFICIENCY TEST OF SEPTEMBER 2017 AGAINST PREVIOUS PTS

The uncertainties in the test results of the determined Overall migration in mg/dm^2 in the iis17P09GM are listed in the next table and are comparable 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
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)

Table 4: comparison of the relative uncertainties for Overall Migration in mg/dm^2 in the previous PTs and in the present PT

(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 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 spoon was used and in the past a spatula, a bowl, square plates and gloves were used.

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 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.
The preset conditions in this PT were:

Sample ID	#17620
Simulant	3% M/V acetic acid
Exposure time	4 hours
Exposure temperature	100.0 °C
Migration method	Total immersion, repeated use
Simulant volume	as per method used

Table 5: preset test conditions used in this PT

The participants were requested to treat the sample as it was a routine sample and analyse it in the way they normally do in day-to-day circumstances with in mind the conditions mentioned in the table above.
The migration method was total immersion for repeated usage in three steps were 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 4).

Preparation
Surprisingly a few 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”.

Ratio ml per dm^2
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). In this PT only fourteen participants used a ratio near the standard ratio of 1 dm^2/100mL (the continuous line in figure 1). A wide range of ratios was used, from 0.06 – 1.01 dm^2/100mL (see appendix 2)
Test results from participants that used a ratio of surface area/volume of simulant significantly deviating from the standard ratio of 1 dm^2/100mL were excluded from the statistical calculations (see appendix 1).

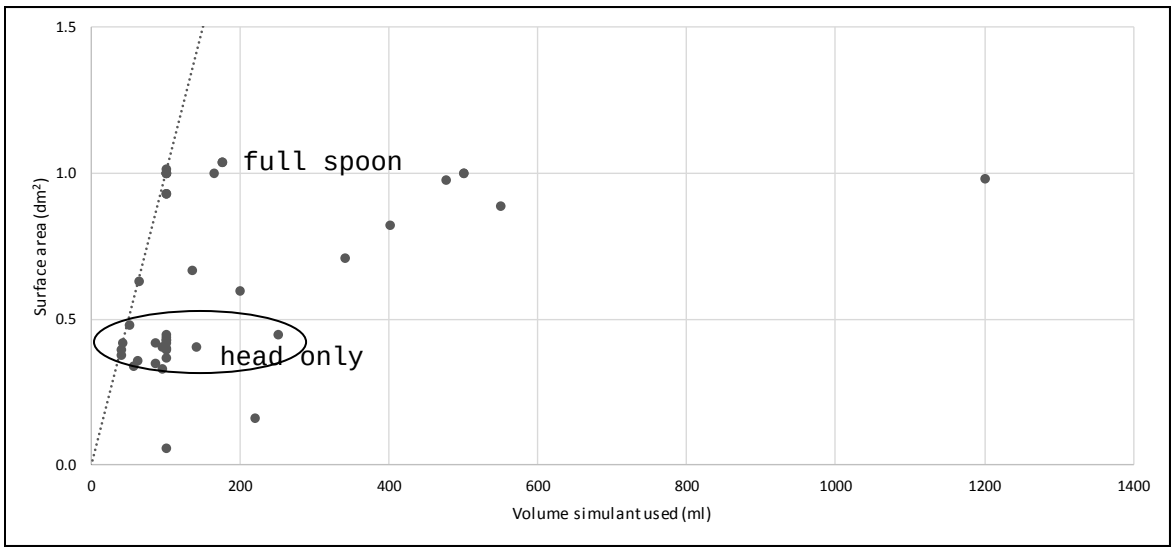


Figure 1: reported surface area versus volume of simulant used by participants

Contact surface used

One spoon was sent to each participant for the overall migration test. The contact surface used as reported by the participants varies from 0.055 (!) – 1.040 dm^2 (see appendix 2). The whole spoon (head + handle) had a surface area of about 1 dm^2 and the surface area of the head was about 0.33 dm^2 (without the triangle part between head and handle).

Volume of simulant used

The amount of simulant used in this PT varied from 40 – 1200 ml (!), see appendix 2.
The volume of simulant used may depend on:
- the portion of the spoon which is exposed to the simulant,
- the type of vessel used for exposure of the spoon to the simulant.

In case the whole spoon is used, the width and/or size of the vessel most likely determines the volume of simulant used and not the ratio of surface area/volume of simulant as mentioned in test method EN1186-3. However, in case a part of the spoon (e.g. only the head) is used for overall migration it is easier to find a vessel that complies the ratio of surface area/volume of simulant as mentioned in test method EN1186-3 (= 1 dm^2/100mL).

Calculation of Overall Migration in mg/dm^2

According to method EN1186-3, the Overall Migration in mg/dm^2 should be calculated taking the mass residue after evaporation of all simulant and corrected for a blank sample mass in mg by division of the surface area in dm^2. A few participants reported a test result of Overall Migration in mg/dm^2 which is not in line with the reported residue (mg) and the reported surface area (dm^2).

Calculation of Overall Migration in mg/kg food simulant

When an article is used for contact with food for children, the overall migration is expressed in mg/kg food simulant. Part of the participants used a conversion factor of 6 dm^2/kg in accordance with EN1186-1, paragraph 12.1, while another part of the participants used a calculation comparable to the calculation mentioned in method EN1186-9. The variety of calculation methods used, lead to a large variation in the test results in mg/kg food simulant (see appendix 3).

Evaporation: temperature, time and volume

After exposure of the spoon to the simulant for the selected time, the simulant must be evaporated to dryness. Many laboratories reported to have evaporated all simulant in one step and only few reported to have evaporated the simulant in several small volumina. Four participants reported to have evaporated only a part of the simulant used (see appendices 2 and 4), although the EN1189-3 test method describes that all simulant should be evaporated to determine the 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 may lead to different migration results.

Almost all participants used an evaporation temperature around 100 °C. This is according to method EN1186-3. The time needed to evaporate each simulant solution has a wide range from 20 minutes to 48 hours (!).

5 DISCUSSION

Total migration, repeated use

A spoon 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.

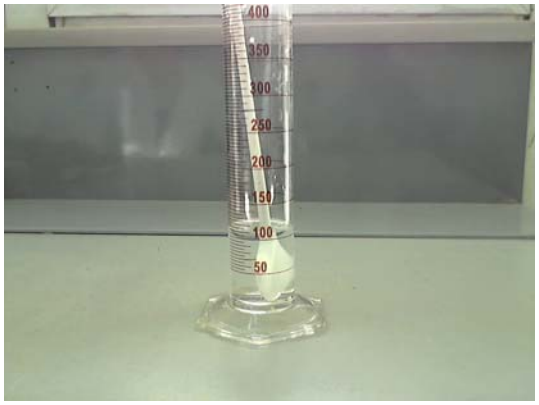
In this PT the average Overall migration in (mg/dm^2) did increase in each successive migration step (step 1: 30.88 mg/dm^2, step 2: 55.42 mg/dm^2 and step 3: 64.16 mg/dm^2). This may be unexpected for some of the participating laboratories, but in literature has been published that occasionally migration may increase upon successive use (lit. 18).

Test item

The test item investigated may have caused some problems to the laboratories. Before starting the migration test, the laboratory had to decide to use the full spoon in order to have a contact surface of approx. 1 dm^2, or to use only the head of the spoon. When the full spoon was selected, it may have been a problem to find a container that would cover the full spoon with only 100 ml of food simulant. Only 7 laboratories reported to have been able to do so. Regretfully none of these laboratories sent a picture of this.

When the head of the spoon was selected, a container must be found that covers the head of the spoon only with approx. 40 ml of food simulant. Only 5 laboratories reported to have done so. Regretfully none of these laboratories did send a picture of this.

Other laboratories simulated a real-life situation and immersed the head of the spoon in a minimum of liquid. This resulted in the use of approx. 100 ml food simulant and a ratio of 0.4 dm^2/100 ml. Several laboratories did send a picture of this, see for example below picture.



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/dm^2.
In this 2017 PT the spoon 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 who have conducted the three migration steps would have rejected the spoon based on the test result of the third migration step.
A few participants did not execute the second and/or the third migration step. These participants also would have rejected the spoon based on the first migration step.

Should this spoon 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, almost all participants would have rejected the spoon.
One participant would reject the spoon based on overall migration in mg/dm^2 but would accept the spoon based on overall migration in mg/kg food simulant. And a few participants did only report overall migration in mg/dm^2.

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.

In 2013 and 2015 a proficiency test on Overall migration (total immersion) was organized, which can be compared with sample #17620 (spoon) used in the 2017 PT. Regretfully, no improvement was visible: the variation of the 2017 PT was larger than in previous PTs.

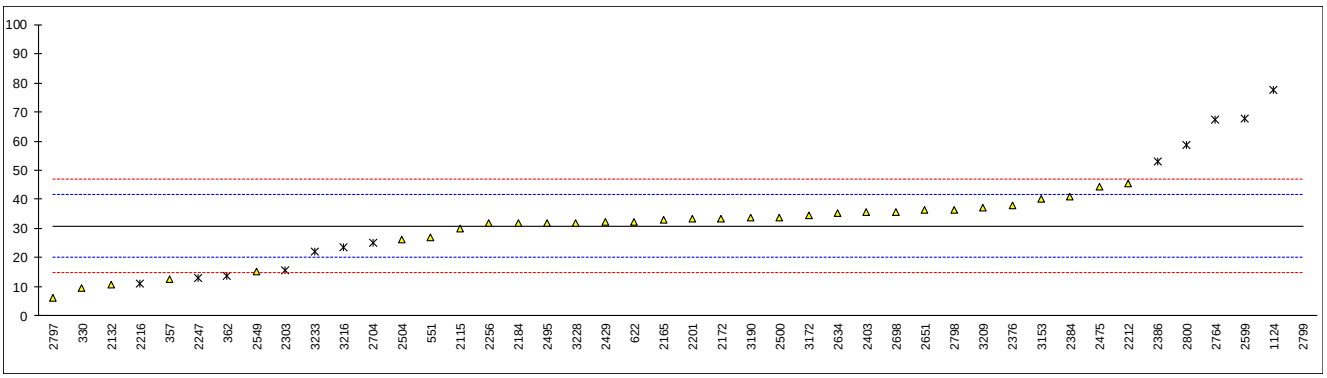
Each laboratory has to evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and the quality of the analytical results.

Finally a request from our site: 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.

APPENDIX 1

Determination of 1st Overall Migration on sample #17620; results in mg/dm^2 per contact surface

lab	method	value	mark	z(targ)	remarks
310		-----		-----	
330	EN1186-3	9.8		-3.96	
357	EN1186-3	12.60		-3.44	
362		13.8	ex	-3.21	Excluded due to low dm^2/ml ratio
551	EN1186-3	27.143		-0.70	
622	In house	32.33		0.27	
1124	EN1186-3	77.46	R(0.05)	8.76	
2115	EN1186-3	30.04	C	-0.16	First reported: 2.63
2132	EN1186-3	10.755		-3.78	
2165	EN1186-3/GB31604.8	33.17		0.43	
2172	EN1186-3	33.26		0.45	
2184	EN1186-3	32.00		0.21	
2201	EN1186-3	33.24		0.44	
2212	CFR175.300	45.6		2.77	
2216	21CFR175.300	11.16	C,ex	-3.71	First reported: 0.043433, Excluded due to low dm^2/ml ratio
2247	EN1186-3	13.15	ex	-3.33	Excluded due to low dm^2/ml ratio
2256	EN1186-3	32.0		0.21	
2303	EN1186-3	15.517	ex	-2.89	Excluded due to low dm^2/ml ratio
2370		-----		-----	
2376	EN1186-3	37.81		1.30	
2384	EN1186-3	41		1.90	
2386	EN1186-3	52.88	ex	4.14	Excluded due to low dm^2/ml ratio
2403	EN1186-3	35.48		0.86	
2429	EN1186-3	32.14		0.24	
2475	EN1186-3	44.44		2.55	
2495	EN1186-3	32.0		0.21	
2500	EN1186-3	33.632		0.52	
2504	EN1186-3	26.400		-0.84	
2549	EN1186-3	15.45		-2.90	
2599	EN1186-3	67.6	ex	6.90	Excluded due to low dm^2/ml ratio
2634	In house	35.2		0.81	
2651	EN1186-3	36.25		1.01	
2698	EN1186-3	35.48		0.86	
2704	EN1186-3	25.11	ex	-1.08	Excluded due to low dm^2/ml ratio
2764		67.2	R(0.05)	6.83	
2797	EN1186-3	6.314		-4.62	
2798	EN1186-3	36.58		1.07	
2799	EN1186-3	253.859	ex	41.92	Excluded due to low dm^2/ml ratio, reported small surface area
2800	EN1186-3	58.64	ex	5.22	Excluded: reported problems with lost
3153	EN1186-3	40.069		1.73	
3163		-----		-----	
3172	EN1186-3	34.44		0.67	
3190	EN1186-3	33.61		0.51	
3209	EN1186-3	37.04		1.16	
3216	EN1186-3	23.46	ex	-1.39	Ex. due to low dm^2/ml ratio, reported area differs from area photo
3228	EN1186-3	32.0		0.21	
3233	EN1186-3	22.03	ex	-1.66	Excluded due to low dm^2/ml ratio
					Only 1 dm^2 : 100 ml ratio
	normality	suspect			not OK
	n	31			13
	outliers	2 (+11 ex)			1
	mean (n)	30.880			34.929
	st.dev. (n)	9.8462	RSD%=32%		14.2319 RSD%=41%
	R(calc.)	27.569			39.849
	st.dev.(EN1186-3:02)	5.3196			6.0172
	R(EN1186-3:02)	14.895			16.848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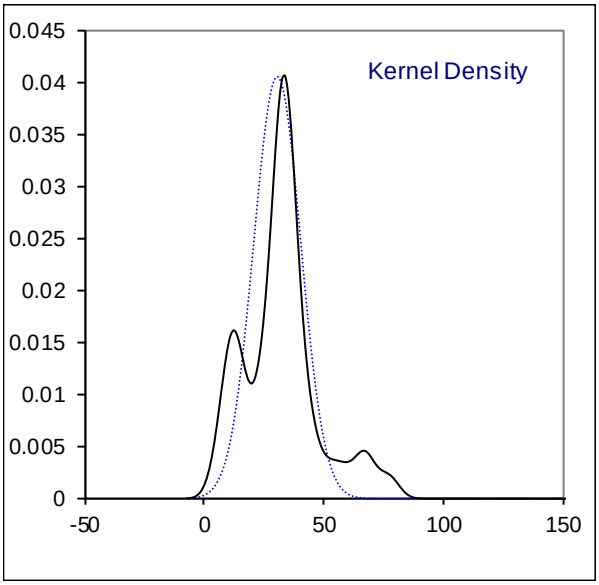
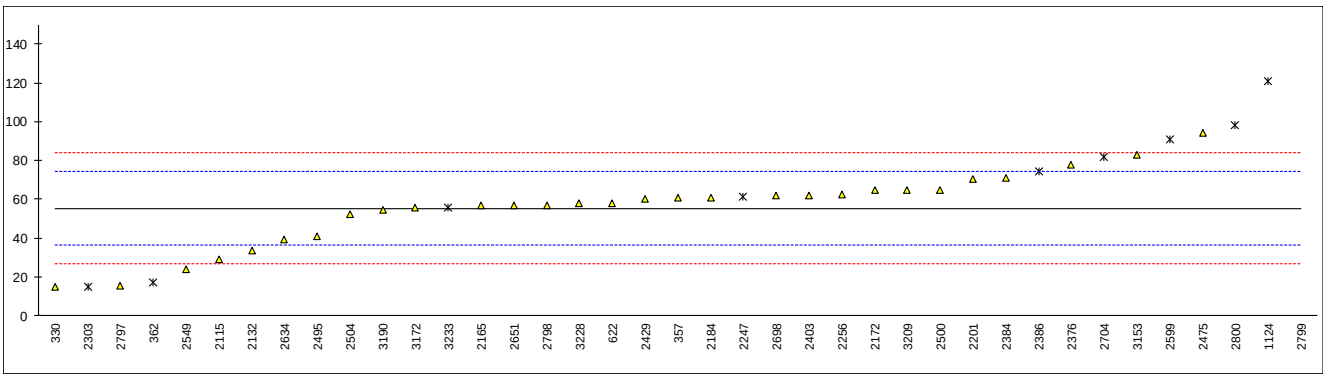
10
0
2797 330 2132 2216 357 2247 362 2549 2303 3233 3216 2704 2504 551 2115 2256 2184 2495 3228 2429 622 2165 2201 2172 3190 2500 3172 2634 2403 2698 2651 2798 3209 2376 3153 2384 2475 2212 2386 2800 2764 2599 1124 2799


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

Determination of 2nd Overall Migration on sample #17620; results in mg/dm^2 per contact surface

lab	method	value	mark	z(targ)	remarks
310		-----		-----	
330	EN1186-3	14.8		-4.31	
357	EN1186-3	60.74		0.32	
362		17.4	C,ex	-4.05	First reported: 2.90, Excluded due to low dm^2/ml ratio
551		-----		-----	
622	In house	58.11		0.05	
1124	EN1186-3	120.64	R(0.05)	6.35	
2115	EN1186-3	28.96	C	-2.89	First reported: 1.25
2132	EN1186-3	33.645		-2.41	
2165	EN1186-3/GB31604.8	56.82		-0.08	
2172	EN1186-3	64.82		0.73	
2184	EN1186-3	61.00		0.34	
2201	EN1186-3	70.30		1.28	
2212		-----		-----	
2216		-----		-----	
2247	EN1186-3	61.24	ex	0.37	Excluded due to low dm^2/ml ratio
2256	EN1186-3	62.6		0.50	
2303	EN1186-3	15.212	ex	-4.27	Excluded due to low dm^2/ml ratio
2370		-----		-----	
2376	EN1186-3	77.69		2.03	
2384	EN1186-3	71		1.35	
2386	EN1186-3	74.63	ex	1.72	Excluded due to low dm^2/ml ratio
2403	EN1186-3	62.15		0.46	
2429	EN1186-3	60.24		0.27	
2475	EN1186-3	94.31		3.70	
2495	EN1186-3	41.1		-1.66	
2500	EN1186-3	65.070		0.75	
2504	EN1186-3	52.633		-0.50	
2549	EN1186-3	24.32		-3.35	
2599	EN1186-3	90.7	ex	3.34	Excluded due to low dm^2/ml ratio
2634	In house	39.2		-1.85	
2651	EN1186-3	56.875		-0.07	
2698	EN1186-3	62.14		0.46	
2704	EN1186-3	81.67	ex	2.43	Excluded due to low dm^2/ml ratio
2764		-----		-----	
2797	EN1186-3	15.407		-4.25	
2798	EN1186-3	57.11		-0.05	
2799	EN1186-3	292.947	ex	23.72	Excluded due to low dm^2/ml ratio, reported small surface area
2800	EN1186-3	98.36	ex	4.11	Excluded: reported problems with lost
3153	EN1186-3	83.088		2.57	
3163		-----		-----	
3172	EN1186-3	55.53		-0.21	
3190	EN1186-3	54.72		-0.29	
3209	EN1186-3	64.84		0.73	
3216		-----		-----	
3228	EN1186-3	58.0		0.04	
3233	EN1186-3	56.03	ex	-0.16	Excluded due to low dm^2/ml ratio
					Only 1 dm^2 : 100 ml ratio
	normality	OK			not OK
	n	29			11
	outliers	1 (+9 ex)			1
	mean (n)	55.421			55.914
	st.dev. (n)	18.5383	RSD%=33%		16.6775 RSD%=30%
	R(calc.)	51.907			46.697
	st.dev.(EN1186-3:02)	9.5474			9.6322
	R(EN1186-3:02)	26.733			26.970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362
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2504
3190
3172
3233
2165
2651
2798
3228
622
2429
357
2184
2247
2698
2403
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2172
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2201
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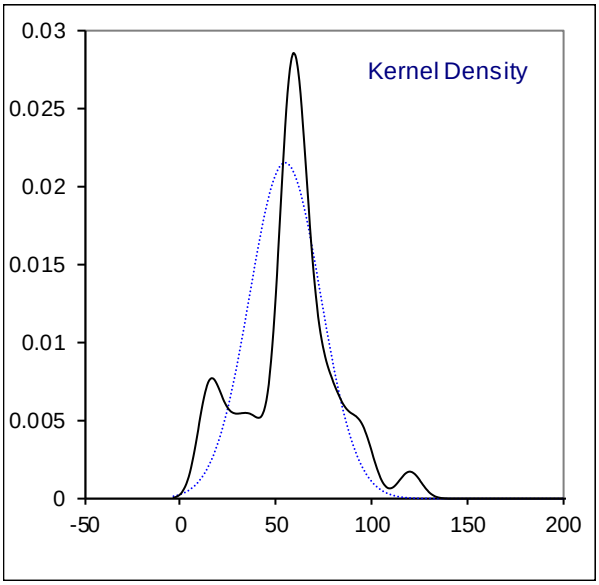
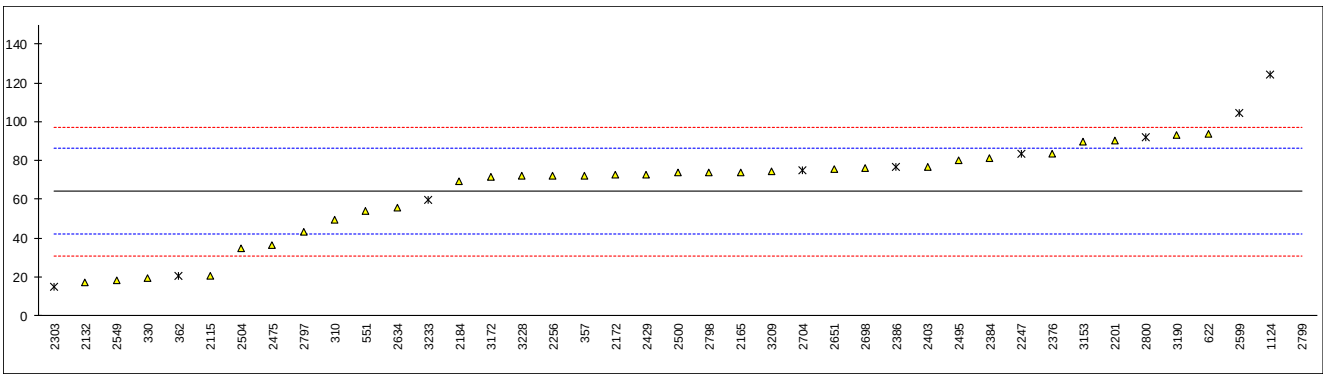
2376
2704
3153
2599
2475
2800
1124
2799


Kernel Density
0.03
0.025
0.02
0.015
0.01
0.005
0
-50
0
50
100
150
200

Determination of 3rd Overall Migration on sample #17620; results in mg/dm^2 per contact surface

lab	method	value	mark	z(targ)	remarks
310	EN1186-3	49.5		-1.45	
330	EN1186-3	19.4		-4.10	
357	EN1186-3	72.45		0.56	
362		20.4	C,ex	-4.01	First reported: 3.03, Excluded due to low dm^2/ml ratio
551	EN1186-3	54.286		-1.03	
622	In house	93.87		2.45	
1124	EN1186-3	124.29	R(0.05)	5.12	
2115	EN1186-3	20.57	C	-4.00	First reported: 0.83
2132	EN1186-3	17.069		-4.30	
2165	EN1186-3/GB31604.8	74.13		0.71	
2172	EN1186-3	72.52		0.57	
2184	EN1186-3	69.10		0.27	
2201	EN1186-3	90.01		2.11	
2212		-----		-----	
2216		-----		-----	
2247	EN1186-3	83.71	ex	1.55	Excluded due to low dm^2/ml ratio
2256	EN1186-3	72.4		0.56	
2303	EN1186-3	15.161	ex	-4.47	Excluded due to low dm^2/ml ratio
2370		-----		-----	
2376	EN1186-3	83.76		1.56	
2384	EN1186-3	81.25		1.34	
2386	EN1186-3	76.63	ex	0.93	Excluded due to low dm^2/ml ratio
2403	EN1186-3	76.77		0.94	
2429	EN1186-3	72.62		0.58	
2475	EN1186-3	36.31		-2.61	
2495	EN1186-3	79.9		1.22	
2500	EN1186-3	73.653		0.67	
2504	EN1186-3	34.755		-2.75	
2549	EN1186-3	18.64		-4.17	
2599	EN1186-3	104.7	ex	3.40	Excluded due to low dm^2/ml ratio
2634	In house	55.5		-0.93	
2651	EN1186-3	75.833		0.86	
2698	EN1186-3	76.19		0.89	
2704	EN1186-3	75.00	ex	0.79	Excluded due to low dm^2/ml ratio
2764		-----		-----	
2797	EN1186-3	43.1905		-2.01	
2798	EN1186-3	73.68		0.67	
2799	EN1186-3	221.118	ex	13.63	Excluded due to low dm^2/ml ratio, reported small surface area
2800	EN1186-3	91.71	ex	2.26	Excluded: reported problems with lost
3153	EN1186-3	89.971		2.10	
3163		-----		-----	
3172	EN1186-3	71.74		0.50	
3190	EN1186-3	93.06		2.38	
3209	EN1186-3	74.68		0.76	
3216		-----		-----	
3228	EN1186-3	72.0		0.52	
3233	EN1186-3	59.86	ex	-0.54	Excluded due to low dm^2/ml ratio
					Only 1 dm^2 : 100 ml ratio
	normality	OK			suspect
	n	31			11
	outliers	1 (+9 ex)			1
	mean (n)	64.155			64.859
	st.dev. (n)	23.0138	RSD%=36%		23.1882 RSD%=36%
	R(calc.)	64.439			64.927
	st.dev.(EN1186-3:02)	11.0520			11.1732
	R(EN1186-3:02)	30.945			31.285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80
60
40
20
0
2303
2132
2549
330
362
2115
2504
2475
2797
310
551
2634
3233
2184
3172
3228
2256
357
2172
2429
2500
2798
2165
3209
2704
2651
2698
2386
2403
2495
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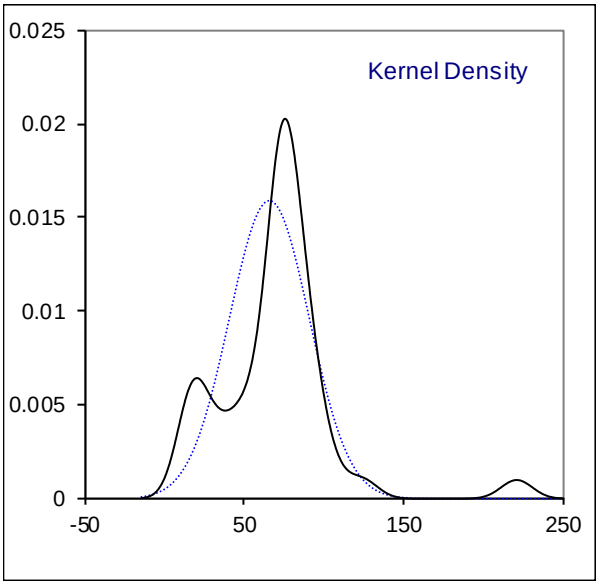
3153
2201
2800
3190
622
2599
1124
2799


Kernel Density
0.025
0.02
0.015
0.01
0.005
0
-50
50
150
250

APPENDIX 2: reported details per step

Reported details on overall migration on sample #17620: 1st step

lab	total residue (mg)	overall migration (mg/kg)	surface area (dm^2)	volume simulant (ml)	iis calculation ratio area/simulant (dm^2 / 100 ml)	remarks
310	-----	-----	-----	-----	-----	
330	10.0	98.0	1.0	100.0	1.00	
357	5.29	75.57	0.42	100	0.42	
362	11.4	28.5	0.824	400	0.21	
551	9.5	110.465	0.35	86	0.41	
622	21.70	162.26	0.67	135	0.50	
1124	48.80	464.76	0.63	63	1.00	
2115	9.99 C	105.16 C	0.33 C	95 C	0.35	First reported: 3.15, 21.00, 1.2, 150
2132	10.900	109.000	1.0135	100	1.01	
2165	34.5	199.04	1.04	175	0.59	
2172	33.26	199.56	1.00	100	1.00	
2184	32.00	192.00	1.00	165.0	0.61	
2201	11.3	198.24	0.339	57	0.59	
2212	20.5	-----	0.45	120 *)	0.38 *)	*) see analytical details appendix 4
2216	10.2	-----	0.977488	476	0.21	
2247	11.7	21.27	0.89	550	0.16	
2256	32.1	192.0	1.002	100	1.00	
2303	39.60	93.101	0.9828	1200	0.08	
2370	-----	-----	-----	-----	-----	
2376	16.95	226.86	0.4483	100	0.45	
2384	16.4	246	0.3958	40	0.99	
2386	56.5	113	1	500	0.20	
2403	33.0	212.88	0.932	100	0.93	
2429	13.5	192.84	0.422	85.0	0.50	
2475	16.4	164 C	0.369	100	0.37	First reported: 444
2495	13.71	191.7	0.4291	100	0.43	
2500	33.7	201.792	1.002	100.00	1.00	
2504	15.800	79.000	0.60	200	0.30	
2549	6.8	92.7	0.44	100	0.44	
2599	66.2	135	1	500	0.20	
2634	14.8	148	0.4	100	0.40	
2651	17.8	217.5	0.48	50	0.96	
2698	14.9	212.88	0.42	42	1.00	
2704	11.25	150.66	0.45	250	0.18	
2764	-----	-----	-----	-----	-----	
2797	2.51	37.884	0.39592	100	0.40	
2798	13.9	219.48	0.38	40	0.95	
2799	14.8	148	0.0583	100	0.06	
2800	23.77	252.87	0.4053	94	0.43	Reported: problems with lost
3153	16.3	240.4	0.4068	140	0.29	
3163	-----	-----	-----	-----	-----	
3172	14.91	206.61	0.43	100	0.43	
3190	12.1	-----	0.36	61	0.59	
3209	34.6	222.26	0.93	100	0.93	
3216	0.3	-----	0.1620	220	0.07	
3228	33.3	192.0	1.04	175	0.59	
3233	4.6	46.0	0.71	340	0.21	

Reported details on overall migration on sample #17620: 2nd step

lab	total residue (mg)		overall migration (mg/kg)		surface area (dm^2)		volume simulant (ml)		iis calculation ratio area/simulant (dm^2 / 100 ml)	remarks
310	-----		-----		-----		-----		-----	
330	15.1		148.0		1.0		100.0		1.00	
357	25.51		364.43		0.42		100		0.42	
362	14.3	C	35.8	C	0.824		400		0.21	First reported: 2.90, 3.52
551	-----		-----		-----		-----		-----	
622	39.00		291.62		0.67		135		0.50	
1124	76.00		723.84		0.63		63		1.00	
2115	9.63	C	101.37	C	0.33	C	95	C	0.35	First reported: 1.50, 10.00, 1.2, 150
2132	34.100		341.000		1.0135		100		1.01	
2165	59.1		340.96		1.04		175		0.59	
2172	64.82		388.92		1.00		100		1.00	
2184	61.00		366.00		1.00		165.0		0.61	
2201	23.9		419.30		0.339		57		0.59	
2212	-----		-----		-----		-----		-----	
2216	-----		-----		-----		-----		-----	
2247	54.5		99.09		0.89		550		0.16	
2256	62.7		375.6		1.002		100		1.00	
2303	42.10		91.270		0.9828		1200		0.08	
2370	-----		-----		-----		-----		-----	
2376	34.83		466.16		0.4483		100		0.45	
2384	28.4		426		0.3958		40		0.99	
2386	78.25		156.5		1		500		0.20	
2403	57.8		372.90		0.932		100		0.93	
2429	25.3		361.44		0.422		85.0		0.50	
2475	34.8		348		0.369		100		0.37	
2495	17.65		246.8		0.4291		100		0.43	
2500	65.2		390.419		1.002		100.00		1.00	
2504	31.500		157.50		0.60		200		0.30	
2549	10.7		145.9		0.44		100		0.44	
2599	90.7		181		1		500		0.20	
2634	16.5		165		0.4		100		0.40	
2651	28.1		341.25		0.48		50		0.96	
2698	26.1		372.84		0.42		42		1.00	
2704	36.75		489.99		0.45		250		0.18	
2764	-----		-----		-----		-----		-----	
2797	6.11		92.442		0.39592		100		0.40	
2798	21.7		342.66		0.38		40		0.95	
2799	16.2		162		0.0553		100		0.06	
2800	39.87		442.97		0.4053		94		0.43	Reported: problems with lost
3153	33.8		498.5		0.4068		140		0.29	
3163	-----		-----		-----		-----		-----	
3172	23.98		333.15		0.43		100		0.43	
3190	19.7		-----		0.36		61		0.59	
3209	60.4		389.03		0.93		100		0.93	
3216	-----		-----		-----		-----		-----	
3228	60.3		348.0		1.04		175		0.59	
3233	11.7		117		0.71		340		0.21	

Reported details on overall migration on sample #17620: 3rd step

lab	total residue (mg)		overall migration (mg/kg)		surface area (dm^2)		volume simulant (ml)		iis calculation ratio area/simulant (dm^2 / 100 ml)	remarks
310	46.5		297.6		0.94		130		0.72	
330	19.7		194.0		1.0		100.0		1.00	
357	30.43		434.71		0.42		100		0.42	
362	16.8	C	42.0	C	0.824		400		0.21	First reported: 2.5, 6.25
551	19.0		220.93		0.35		86		0.41	
622	63.00		471.07		0.67		135		0.50	
1124	78.30		745.74		0.63		63		1.00	
2115	6.84	C	72.00	C	0.33	C	95	C	0.35	First reported: 1.00, 6.67, 1.2, 150
2132	17.300		170.692		1.0135		100		1.01	
2165	77.1		444.81		1.04		175		0.59	
2172	72.52		435.12		1.00		100		1.00	
2184	69.10		414.60		1.00		165.0		0.61	
2201	30.6		536.84		0.339		57		0.59	
2212	-----		-----		-----		-----		-----	
2216	-----		-----		-----		-----		-----	
2247	74.5		152.2		0.89		550		0.16	
2256	72.5		434.4		1.002		100		1.00	
2303	38.30		90.965		0.9828		1200		0.08	
2370	-----		-----		-----		-----		-----	
2376	37.55		502.56		0.4483		100		0.45	
2384	32.5		487.50		0.3958		40		0.99	
2386	80		160		1		500		0.20	
2403	71.4		460.62		0.932		100		0.93	
2429	30.5		435.72		0.422		85.0		0.50	
2475	13.4		134		0.369		100		0.37	
2495	34.29		479.5		0.4291		100		0.43	
2500	73.8		441.916		1.002		100.00		1.00	
2504	20.800		104.000		0.60		200		0.30	
2549	8.2		111.8		0.44		100		0.44	
2599	102.6		209		1		500		0.20	
2634	23.3		233		0.4		100		0.40	
2651	37.2		454.998		0.48		50		0.96	
2698	42.0		457.14		0.42		42		1.00	
2704	33.75		450.00		0.45		250.		0.18	
2764	-----		-----		-----		-----		-----	
2797	17.11		259.143		0.39592		100		0.40	
2798	28.0		442.08		0.38		40		0.95	
2799	17.8		178		0.0805		100		0.08	
2800	37.17		422.39		0.4053	C	88		0.46	fr.: 91.71, reported: problems with lost
3153	36.6		539.8		0.4068		140		0.29	
3163	-----		-----		-----		-----		-----	
3172	30.95		430.42		0.43		100		0.43	
3190	33.5		-----		0.36		61		0.59	
3209	69.7		448.06		0.93		100		0.93	
3216	-----		-----		-----		-----		-----	
3228	74.9		432.0		1.04		175		0.59	
3233	12.5		125		0.71		340		0.21	

APPENDIX 3

Calculations used on sample #17620: overall migration (per kg food simulant) in mg/kg

lab	1st step reported (mg/kg)		2nd step reported (mg/kg)		3rd step reported (mg/kg)		method of calculation	remarks
310	-----		-----		297.6		Factor 6 ?	This kind of article results in mg/dm^2 and not in mg/kg
330	98.0		148.0		194.0		?	
357	75.57		364.43		434.71		Factor 6	
362	28.5		35.8	C	42.0	C	EN1186-9	First reported = 3.52, 6.25
551	110.465		-----		220.93		EN1186-9	
622	162.26		291.62		471.07		?	
1124	464.76		723.84		745.74		Factor 6	
2115	105.16	C	101.37	C	72.00	C	EN1186-9	First reported = 21.00, 10.00, 6.67
2132	109.000		341.000		170.692		EN1186-9	3rd step: EN1186-9 ?
2165	199.04		340.96		444.81		Factor 6	
2172	199.56		388.92		435.12		Factor 6	
2184	192.00		366.00		414.60		Factor 6	
2201	198.24		419.30		536.84		EN1186-9	
2212	-----		-----		-----		-----	
2216	-----		-----		-----		-----	
2247	21.27		99.09		152.2		EN1186-9	3rd step: EN1186-9 ?
2256	192.0		375.6		434.4		Factor 6 ?	
2303	93.101		91.270		90.965		?	
2370	-----		-----		-----		-----	
2376	226.86		466.16		502.56		Factor 6	
2384	246		426		487.50		?	
2386	113		156.5		160		EN1186-9	
2403	212.88		372.90		460.62		Factor 6 ?	
2429	192.84		361.44		435.72		Factor 6 ?	
2475	164	C	348		134		EN1186-9	First reported = 444
2495	191.7		246.8		479.5		Factor 6	
2500	201.792		390.419		441.916		Factor 6	
2504	79.000		157.50		104.000		EN1186-9	
2549	92.7		145.9		111.8		Factor 6	
2599	135		181		209		?	
2634	148		165		233		EN1186-9	
2651	217.5		341.25		454.998		?	
2698	212.88		372.84		457.14		Factor 6	3rd step: factor 6 ?
2704	150.66		489.99		450.00		Factor 6	
2764	-----		-----		-----			
2797	37.884		92.442		259.143		Factor 6 ?	
2798	219.48		342.66		442.08		Factor 6	
2799	148		162		178		EN1186-9	
2800	252.87		442.97		422.39		EN1186-9	2rd step: EN1186-9 ?
3153	240.4		498.5		539.8		Factor 6	
3163	-----		-----		-----		-----	
3172	206.61		333.15		430.42		?	
3190	-----		-----		-----		-----	
3209	222.26		389.03		448.06		?	
3216	-----		-----		-----		-----	
3228	192.0		348.0		432.0		Factor 6	
3233	46.0		117		125		?	

APPENDIX 4: additional reported details

Reported analytical details on sample #17620

lab	ISO17025 accredited for this test	surface to volume ratio (dm^2/ml)	volume simulant evaporated (ml)	evaporation of simulant
310	No	7.2	130	Evaporation of simulant in one step
330	Yes	1 dm2 / 100ml	100 ml	Evaporation of simulant in one step
357	No	0,42 dm2/100 ml)	100 ml	Evaporation of simulant in several small volumina
362	---	---	---	---
551	Yes	4.07	86	Evaporation of simulant in one step
622	No	0.01 dm2/2 mL	135 mL	Evaporation of simulant in one step
1124	Yes	0.63 dm2/63 mL	63	Evaporation of simulant in one step
2115	Yes	0.33 dm2 /95ml *)	95 ml *)	Evaporation of simulant in one step
2132	Yes	0.01014	100	Evaporation of simulant in one step
2165	No	6 dm2:1000ml	175	Evaporation of simulant in several small volumina
2172	Yes	1.00dm2/100mL	100mL	Evaporation of simulant in several small volumina
2184	Yes	1 dm2: 165ml	165ml	Evaporation of simulant in one step
2201	Yes	1dm2/167ml	57	Evaporation of simulant in one step
2212	Yes	0.0038	120mL	Evaporation of simulant in several small volumina
2216	Yes **)	0.002054	476	Evaporation of simulant in one step
2247	Yes	1 dm2: 618 ml	complete (550ml)	Evaporation of simulant in several small volumina
2256	Yes	1.0dm2 to 100ml	100ml	Evaporation of simulant in one step
2303	Yes	0.0008	1220	Evaporation of simulant in several small volumina
2370	---	---	---	---
2376	Yes	0.4483 dm2 / 100 mL	100ml	Evaporation of simulant in several small volumina
2384	Yes	0.01	40	Evaporation of simulant in one step
2386	Yes	1/500	500	Evaporation of simulant in one step
2403	Yes	1:100	5 ml	Evaporation of simulant in one step
2429	Yes	0.42dm2=85mL	85mL	Evaporation of simulant in one step
2475	Yes	0.369/100	100	Evaporation of simulant in one step
2495	Yes	0.0043	100	Evaporation of simulant in one step
2500	Yes	0.6dm2 to 100mL	100	Evaporation of simulant in one step
2504	Yes	0.003	200	Evaporation of simulant in one step
2549	Yes	0.0044	100	Evaporation of simulant in one step
2599	Yes	---	100%	Evaporation of simulant in several small volumina
2634	Yes	0.4	83.2	Evaporation of simulant in one step
2651	Yes	0.48in dm^2/ 50mL	50ml	Evaporation of simulant in one step
2698	Yes	1/100	0	Evaporation of simulant in one step
2704	No	0,0018	100	Evaporation of simulant in several small volumina
2764	Yes	---	---	Evaporation of simulant in one step
2797	No	0.004 (0.4dm2/100mL)	100mL	Evaporation of simulant in one step
2798	Yes	0.38 dm^2-40mL	40mL	Evaporation of simulant in several small volumina
2799	No	------	100 ml	Evaporation of simulant in one step
2800	Yes	0.41dm2/100ml	about 90 ml	---
3153	Yes	0.41 dm2 / 140 ml	140 ml	Evaporation of simulant in one step
3163	---		---	---
3172	Yes	0,0043	100	Evaporation of simulant in one step
3190	Yes	0.36dm^2 / 61mL	61ml	Evaporation of the simulant by sand bath
3209	Yes	1:1	20mL	Evaporation of simulant in several small volumina
3216	No	0,16dm2 /220mL	200 mL	Evaporation of simulant in one step
3228	Yes	1.04dm2/175mL	175ml	Evaporation of simulant in one step
3233	No	0.71/340 = 0.0021	100	Evaporation of simulant in one step

*)
Lab 2115: Reported 1.2 dm2 /150ml, 150ml (lab 2115 corrected surface area and volume simulant for all 3 steps)

**)
Lab 2216: Accreditation according to US 21CFR Part 175.300

Reported analytical details on sample #17620– continued

lab	duration of evaporation (min)	temperature of evaporation (°C)	sample cleaned prior the test?	remarks
310	---	---	No	
330	about 1 day	95°C	No	Sample part has been cut
357	Overnight	100 C overnight + 105 C to stable the mass	Yes	Cleaned: wiped with soft cloth
362	---	---	---	
551	90 min	>105°C	Yes	Cleaned: with a lint free cloth
622	3 hours	100°C	No	
1124	70	82	Yes	Cleaned: LInt-free cloth
2115	240 min	105°C	No	
2132	480mins	105°C	Yes	Cleaned: lint-free cloth.
2165	720min	105	No	
2172	50 min	105°C	No	
2184	180 mins	/	No	/
2201	60 minutes	100	Yes	Cleaned with water under no circumstances prior the migration.
2212	3 hours	105	Yes	Cleaned: wipe sample with lint-free cloth gently
2216	---	---	Yes	Cleaned: with 90 C distilled water prior to extraction
2247	400 to 500 min	110	Yes	Cleaned: wiped with dry fabric; Evaporation done 3X 200
2256	90mins	105	No	
2303	1260	100	No	
2370	---	---	---	
2376	140 mins	95.7 C	Yes	Cleaned: brush to remove dust
2384	240 - 360	110	Yes	Cleaned: with lint-free cloth before proceed with analysis.
2386	over night	105°C	Yes	Cleaned with water
2403	60 min	300 °C	No	The sample quantity is too less to meet the test requirements
2429	240minutes	105	Yes	Cleaned: with water prior to the migration step.
2475	---	105-110	No	
2495	about 30	110°C	Yes	the 2nd evaporation step could be affected by small bumping
2500	90minutes	105°C	No	
2504	4 hr	90-110	No	
2549	3 hours	150	No	
2599	about 12 h	105	No	Difficult to add any pictures of the spoon here
2634	60 min	---	No	
2651	20 minutes	105℃	Yes	Cleaned: with distilled water during preparation
2698	2 hours	110	Yes	
2704	200	---	No	
2764	---	---	---	
2797	480	100	No	
2798	180 minutes	105℃	Yes	Cleaned: wiping it with a lint free cloth
2799	80 minutes	100	No	
2800	about 48h	---	Yes	Cleaned: water, cloth. The sample contained visible dirt
3153	About 180 min.	At about 100°C	Yes	Cleaned: with lint-free cloth
3163	---	---	---	
3172	720	100	No	
3190	about 3 ½ hours	about 90°C	Yes	Cleaned: by water
3209	240	100	Yes	
3216	About 8h in oven	100ºC	No	
3228	960min	105°C	Yes	Cleaned: with lint free cloth prior to the migration step.
3233	---	105	No	

APPENDIX 5

Number of participating laboratories per country

1 lab in	BRAZIL
1 lab in	BULGARIA
1 lab in	CROATIA
2 labs in	FINLAND
3 labs in	FRANCE
1 lab in	GERMANY
5 labs in	HONG KONG
2 labs in	INDIA
1 lab in	INDONESIA
1 lab in	ISRAEL
4 labs in	ITALY
1 lab in	LATVIA
1 lab in	MALAYSIA
13 labs in	P.R. of CHINA
1 lab in	SPAIN
1 lab in	SWEDEN
1 lab in	TAIWAN R.O.C.
1 lab in	THAILAND
2 labs in	THE NETHERLANDS
1 lab in	U.S.A.
2 labs in	UNITED ARAB EMIRATES
1 lab in	UNITED KINGDOM

APPENDIX 6

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:

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2 EN 1186-1:02 - Guide to the selection of conditions and test methods for overall migration
3 EN 1186-3:02 - Test methods for overall migration into aqueous simulant by total immersion
4 EN 1186-8:02 - Test methods for overall migration into olive oil by article filling
5 EN 1186-9:02 - Test methods for overall migration into aqueous simulant by article filling
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9 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
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18 Commission regulation (EU) No 10/2011 of January 2011 on plastic materials and articles intended to come into contact with food, published in the official journal of the EU on the 15th of Janary 2011
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