Results of Proficiency Test
Overall Migration (fcm)
October 2020

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

During the contact of materials with food, molecules can migrate from the food contact material to the food. Because of this, in many countries regulations are made to ensure food safety. The framework Regulation (EU) No. 10/2011 (lit. 18 and lit. 19) applies to all food contact materials and describes a large number of requirements, e.g. limits for Overall Migration and specific limits for certain constituents. Article 12 of this regulation describes the Overall Migration limit, which is 10 mg/dm^2. Only when determined for food contact intended for infants and children, the Overall Migration is expressed in mg/kg food simulant with a limit of 60 mg/kg food simulant. The determination of Specific Migration requires additional analytical testing following the migration step, while the determination of the Overall (also called global or total) Migration requires weighing as only quantitative analytical technique. In September 2020, the 15th amendment of this EU 10/2011 was published and will be implemented in 2021 for new products. This amendment especially describes methods for repeated use articles, how to test and to reject them.

Since 2012, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Overall Migration every year. During the annual proficiency testing program 2020/2021, it was decided to continue the proficiency test for the determination of Overall Migration on food contact materials.
In this interlaboratory study 48 laboratories from 19 different countries registered for participation. See appendix 4 for the number of participants per country. In this report, the results of this proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send one sample (a set of three items) labelled #20675 positive on Overall Migration. Furthermore, a number of test conditions (type of simulant, exposure time, exposure volume, migration method, simulant volume and details about the contact surface testing) were prescribed. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

A batch of white plastic knives was selected and purchased from the market. The knives were positive for Overall Migration. Randomly from the batch 60 sets of three knives were put into a bag and labelled #20675. The homogeneity of the subsamples was checked by determination of Overall Migration according to EN1186 on 6 stratified randomly selected subsamples with the following conditions: total immersion, 3% Acetic Acid, 2 hours at 70°C.

	Overall Migration mg/dm^2
Sample #20675-1	15.98
Sample #20675-2	15.20
Sample #20675-3	15.29
Sample #20675-4	14.51
Sample #20675-5	14.90
Sample #20675-6	16.18

Table 1: homogeneity test results of subsamples #20675

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

	Overall Migration mg/dm^2
r (observed)	1.78
reference test method	EN1186-3
0.3 * R (reference test method)	2.22

Table 2: evaluation of the repeatability of subsamples #20675

The calculated reproducibility was in agreement with 0.3 times the reproducibility of the target test method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample, a set of three knives, labelled #20675 was sent on September 9, 2020.

2.5 ANALYZES

The participants were requested to determine Overall Migration on sample #20675 using the prescribed test conditions (total immersion, single use and 3% Acetic Acid as simulant for 2 hours at 70°C). Each participant received three knives to be tested separately, where also the average of the three tests was requested.
It was also requested to report if the laboratory was accredited for this test and to report some analytical details.

It was explicitly requested to treat the sample as if it was a routine sample and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report ‘less than’ test results, which are above the detection limit, because such results cannot be used for meaningful statistical evaluations.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in 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 June 2018 (iis-protocol, version 3.5).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

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

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

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. EN reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

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

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare. The usual interpretation of z-scores is as follows:

$\lvert z \rvert < 1$	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. Two participants did not report any test results at all and two participants reported test results after the final reporting date. Finally, the 46 reporting laboratories reported 45 numerical test results for the Average Overall Migration per contact surface. Observed were 2 outlying test results, which is 4.4%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

The original data set proved to have a normal Gaussian distribution.

4.1 EVALUATION OF THE TEST RESULTS

In this section the reported test results are discussed. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 5.

In the past iis has observed that the Overall and Specific Migration methods the limits and the calculations are mixed up and used inappropriately by participants. Therefore, iis issued a White Paper on this subject in February 2018 (see lit. 20) to help participants understand the differences between the two methods, the units used for reporting and the regulated limits.

For the determination of Overall Migration (also called Global or Total Migration) on food contact material by total immersion, the EN1186 method series part 3 is considered to be the official EC test method. 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).

Overall Migration: This determination was not problematic. Two statistical outliers were observed and one other test result was excluded. The calculated reproducibility after rejection of the suspect data is in full agreement with the target reproducibility estimated from EN1186-3:02.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	2.8 * sd	R(lit)
Average Overall Migration	mg/dm^2	42	21.55	11.17	10.39

Table 3: reproducibility of tests on sample #20675

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

4.3 COMPARISON OF PROFICIENCY TEST OF OCTOBER 2020 AGAINST PREVIOUS PTS

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

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

Table 4: development of the uncertainties over the years

The uncertainty observed in this PT is in line with the uncertainties observed in previous PTs for total immersion.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

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

Sample ID	#20675
Simulant	3% Acetic Acid
Exposure time	2 hours
Exposure temperature	70°C
Migration method	Total immersion, single use
Simulant volume	As per method used

Table 5: prescribed test conditions used in this PT

The participants were requested to report the intermediate test results for the three knives and the average Overall Migration. Additional details regarding preparation, residue, surface area, simulant volume and details about the evaporation step were also requested. See appendices 2 and 3 for the reported details.

<u>Test method and accreditation</u>
About 95% of the reporting participants mentioned to have used test method EN1186-3. From the reporting participants about 80% mentioned that they are accredited for this test.

Preparation

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

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

Method EN1186-1 states in paragraph 9.3: “that the surface to volume ratio in the total immersion test is conventionally 1 dm^2 of food contact area to 100 mL of food simulant.”, also in method EN1186-3 the ratio of 1 dm^2/100mL is mentioned. In appendix 2 the ratio calculated by iis is given based on the reported details of the participants.
Only fifteen of the reporting participants used a surface to volume ratio of 1 dm^2/100mL, on average over all participants the volume to surface ratio was nearly 2 dm^2/100mL. Remarkably, this did not have an effect on the Overall Migration test results.

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). These are marked as bold in appendix 2. Some other test results of Overall Migration in mg/dm^2 were corrected without correction of the reported residue (mg) or the surface area (dm^2). For these tests iis did not calculate the Overall Migration.

Evaporation: temperature and time

After exposure of the plate to the simulant for the selected time, the simulant must be evaporated to dryness. The reported evaporation temperature varied from 90 to 400°C. About 60% of the reporting participants used an evaporation temperature between 100°C and 150 °C. The reported evaporation time varied from 16 to 1440 minutes. About 50% of the reporting participants used an evaporation time less than 240 minutes.
The differences in evaporation temperature and time did not appear to be of influence on the test results of the samples in this PT.

5 DISCUSSION

Total immersion, single use, three articles

The prescribed test migration method for this PT was total immersion. A set of three knives was sent to the participants to be reported separately. Further, the average Overall Migration was requested. One laboratory reported the test results of the three knives but did not report the average Overall Migration. One participant reported the test result of one knife as the average Overall Migration and this test value was excluded from the statistical evaluation.

Limits for Overall Migration from EU regulation No 10/2011

The EU regulation describes in article 12 that the limit for Overall Migration is 10 mg/dm^2. In this PT the Overall Migration found should comply the limit for Overall Migration. According to this limit all reporting participants would have rejected sample #20675.

6 CONCLUSION

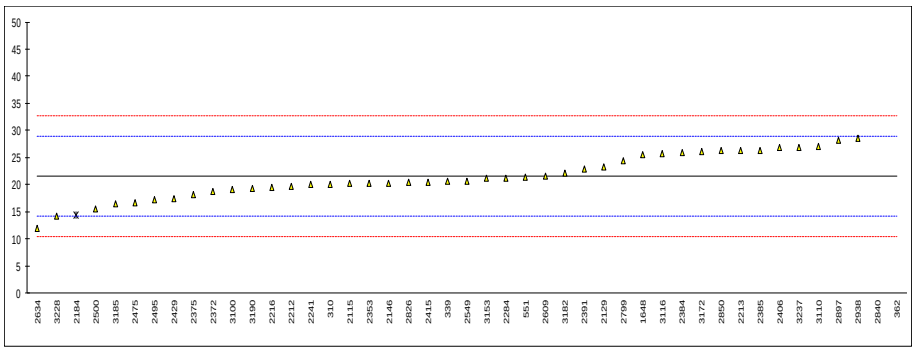
It is to be expected that the variation of the migration test results in real life practice will be larger than observed in this PT as the test conditions like time, temperature, etc. will not be prescribed but will be selected by the individual laboratories.

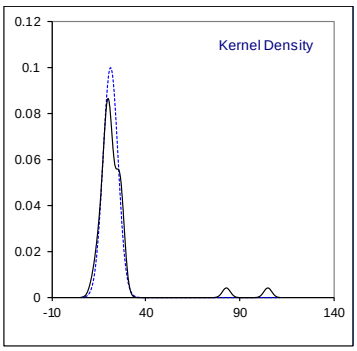
Each laboratory has to evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and the quality of the analytical results.

APPENDIX 1

Average Overall Migration (per contact surface) on sample #20675; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
310	EN1186-3	20.1		-0.39	
339	EN1186-3	20.54		-0.27	
362	EN1186-3	104.85	C,R(0.01)	22.44	first reported: 52.42
551	EN1186-3	21.474		-0.02	
1648	EN1186-3	25.530		1.07	
2115	EN1186-3	20.23		-0.35	
2129	EN1186-3	23.3		0.47	
2146	EN1186-3	20.342		-0.32	
2165		-----		-----	
2184	EN1186-3	14.33	ex	-1.94	test result excluded, only result of one knife
2212	CFR175.300	19.62		-0.52	
2213	EN1186	26.32		1.29	
2216		19.400		-0.58	
2236		-----		-----	
2241	EN1186-3	20.089		-0.39	
2284	EN1186-3	21.19		-0.10	
2353	EN1186-3	20.321		-0.33	
2372	EN1186-3	18.64601262		-0.78	
2375	EN1186-3	18.15		-0.92	
2384	EN1186-3	25.97		1.19	
2385		26.327		1.29	average was not reported but calculated by iis
2391	EN1186-3	22.857		0.35	
2406	EN1186-3	26.907		1.44	
2415	EN1186-3	20.444		-0.30	
2429	EN1186-3	17.4		-1.12	
2475	EN1186-3	16.63		-1.32	
2495	EN1186-3	17.24		-1.16	
2500	EN1186-3	15.574		-1.61	
2549	EN1186-3	20.63		-0.25	
2609	EN1186-3	21.556		0.00	
2634	EN1186-3	12.0		-2.57	
2799	EN1186-3	24.4737	C	0.79	first reported: 49.0765
2826	EN1186-3	20.388		-0.31	
2840	EN1186-3	82.9	R(0.01)	16.53	
2850	EN1186-3	26.3		1.28	
2897	EN1186-3	28.09		1.76	
2938	INH-31	28.47		1.87	
3100	EN1186-3	19.183		-0.64	
3110	EN1186-3	27		1.47	
3116	EN1186-3	25.80		1.15	
3153	EN1186-3	21.158		-0.10	
3172	EN1186-3	26.11		1.23	
3182	EN1186-3	22.20		0.18	
3185	EN1186-3	16.5		-1.36	
3190	EN1186-3	19.37		-0.59	
3218		-----		-----	
3228	EN1186-3	14.2		-1.98	
3237	EN1186-3	26.93		1.45	
	normality	OK			
	n	42			
	outliers	2 (+1ex)			
	mean (n)	21.547			
	st.dev. (n)	3.9906	RSD = 19%		
	R(calc.)	11.174			
	st.dev.(EN1186-3:02)	3.7118			
	R(EN1186-3:02)	10.393			





APPENDIX 2

Determination of Overall Migration on sample #20675; results in mg/dm^2

lab	Knife 1		Knife 2		Knife 3	
	reported	iis calculated	reported	iis calculated	reported	iis calculated
310	19.9	19.880	19.9	19.880	20.5	20.566
339	20.109	20.870	19.457	20.217	22.065	22.826
362	107.27	-----	101.82	-----	105.45	-----
551	**22.115**	**23.846**	**20.577**	**22.308**	**21.731**	**23.462**
1648	**25.682**	**27.444**	**25.909**	**27.465**	**25.000**	**26.794**
2115	19.5	19.500	19.7	19.700	21.4	21.200
2129	**21**	**12.800**	**24**	**14.600**	**25**	**15.200**
2146	17.436	17.391	21.282	21.228	22.308	22.251
2165	14.583	14.583	-----	-----	-----	-----
2184	**14.33**	**9.885**	-----	-----	-----	-----
2212	19.35	19.355	19.15	19.153	20.36	20.363
2213	26.8	26.881	25.75	25.751	26.4	26.203
2216	20.144	20.144	17.794	18.564	20.312	21.140
2236	-----	-----	-----	-----	-----	-----
2241	20.431	20.329	18.686	18.686	21.150	21.150
2284	21.19	21.190	20.95	20.952	21.43	21.429
2353	20.045	20.045	20.913	20.913	20.004	20.004
2372	19.57831325	20.009	17.85714286	18.287	18.50258176	18.933
2375	19.05	19.909	18.21	19.091	17.20	18.073
2384	27.65	27.566	25.00	24.927	25.29	25.220
2385	26.67	26.667	27.44	27.436	24.87	24.872
2391	22.619	22.619	22.857	22.857	23.095	23.095
2406	26.072	26.072	29.080	29.080	25.570	25.570
2415	21.185	21.178	20.444	20.444	19.704	19.711
2429	17.2	17.193	17.2	17.193	17.8	17.857
2475	17.09	17.091	15.27	15.273	17.45	17.455
2495	16.84	16.906	17.19	17.193	17.68	17.684
2500	15.517	15.517	15.172	15.172	16.034	16.034
2549	21.90	21.905	19.52	19.524	20.47	20.476
2609	21.627	22.056	21.199	21.627	21.842	22.270
2634	14.7	14.667	6.7	6.667	14.7	14.667
2799	**23.9474**	**64.380**	**27.3684**	**71.240**	**22.1053**	**60.686**
2826	20.859	20.808	20.455	20.404	19.850	19.798
2840	49.7	49.737	119.5	119.474	79.6	79.737
2850	26.9	26.857	26.3	26.286	25.7	25.714
2897	**29.79**	**37.167**	**28.43**	**35.810**	**26.05**	**33.429**
2938	34.938	34.928	22.488	22.488	27.99	27.990
3100	19.057	19.057	19.434	19.434	19.057	19.057
3110	27	-----	28	-----	27	-----
3116	26.72	26.722	25.90	25.895	24.79	24.793
3153	20.559	20.559	21.357	21.357	21.557	21.557
3172	26.43	26.429	25.95	25.952	25.95	25.952
3182	22.55	22.553	22.55	22.553	21.49	21.489
3185	15.41	15.410	16.72	16.721	17.54	17.541
3190	19.04	19.038	19.31	19.309	19.76	19.758
3218	-----	-----	-----	-----	-----	-----
3228	14.1	14.091	14.8	14.773	13.6	13.636
3237	27.63	27.625	26.18	26.175	27	27.000

The figures in bold show a calculation difference between the reported test result and the result iis calculated of larger than 1. Difference below one can also be caused by rounding issues in the reported raw data.

Lab 362 first reported for knife 1: 52.64, for knife 2: 50.91 and for knife 3: 52.73
Lab 2799 first reported for knife 1: 48.024, for knife 2: 54.8812 and for knife 3: 44.327

Reported details for calculation of Overall Migration on sample #20675

lab	total residue (mg) knife 1	total residue (mg) knife 2	total residue (mg) knife 3	surface area (dm^2)	volume simulant (mL)	iis calc. ratio area/simulant (dm^2 / 100 mL)
310	11.60	11.60	12.0	0.5835	100	1.71
339	9.6	9.3	10.5	0.46	50	1.09
362	-----	-----	-----	0.11	11	1.00
551	12.40	11.60	12.20	0.520	63.0	1.21
1648	12.1	12.2	11.8	0.4409	100	2.27
2115	9.75	9.85	10.6	0.5	100	2.00
2129	6.4	7.3	7.6	0.5	80	1.60
2146	6.8	8.3	8.7	0.391	100	2.56
2165	7.0	-----	-----	0.48	80	1.67
2184	8.6	-----	-----	0.87	145	1.67
2212	9.6	9.5	10.1	0.496	49	0.99
2213	11.9	11.4	11.6	0.4427	170	3.84
2216	12.0	10.6	12.1	0.59572/0.57/0.57	340.7	5.72 – 5.96
2236	-----	-----	-----	-----	-----	-----
2241	9.9	9.1	10.3	0.487	50	1.03
2284	8.9	8.8	9.0	0.42	42	1.00
2353	9.70	10.12	9.68	0.4839	48	0.99
2372	9.3	8.5	8.8	0.4648	46	0.99
2375	0.01095	0.0105	0.00994	0.55	100	1.82
2384	9.4	8.5	8.6	0.341	100	2.93
2385	10.4	10.7	9.7	0.39	65	1.67
2391	0.0095	0.0096	0.0097	0.42	130	3.10
2406	10.4	11.6	10.2	0.3989	60	1.50
2415	9.53	9.20	8.87	0.45	62.5/62/63	1.38 – 1.40
2429	9.8	9.8	10.0	0.57	57.0	1.00
2475	9.4	8.4	9.6	0.55	100	1.82
2495	8.25	8.39	8.63	0.488	65.0	1.33
2500	9.0	8.8	9.3	0.58	97	1.67
2549	4.6	4.1	4.3	0.210	30	1.43
2609	10.3	10.1	10.4	0.467	70.0	1.50
2634	2.2	1.0	2.2	0.15	100	6.67
2799	0.0122	0.0135	0.0115	0.1895	130	6.86
2826	10.3	10.1	9.8	0.495	50	1.01
2840	18.9	45.4	30.3	0.38	100	2.63
2850	9.4	9.2	9.0	0.35	35	1.00
2897	15.61	15.04	14.04	0.42	80	1.90
2938	14.6	9.4	11.7	0.418	100	2.39
3100	10.1	10.3	10.1	0.53	53	1.00
3110	-----	-----	-----	-----	-----	-----
3116	9.7	9.4	9.0	0.363	36	0.99
3153	10.3	10.7	10.8	0.501	50	1.00
3172	11.1	10.9	10.9	0.42	70	1.67
3182	10.60	10.60	10.10	0.47	100.00	2.13
3185	9.4	10.2	10.7	0.61	61	1.00
3190	9.5	9.5	9.8	0.499/0.49/0.50	50	1.00 – 1.02
3218	-----	-----	-----	-----	-----	-----
3228	6.2	6.5	6.0	0.44	73	1.66
3237	11.05	10.47	10.8	0.4	110	2.75

APPENDIX 3 Summary of reported analytical details

lab	ISO17025 accredited	Cleaned prior to migration step	Sample	Equipment	Evaporation time (min)	Evaporation temperature (°C)
310	No	No	as received	Oven	o.n.	105
339	Yes	No	further cut	Oven		
362	Yes	Yes	as received	Incubator	30 min	100
551	Yes	No	as received	Oven		
1648	No	Yes, with a cloth	as received	Oven	About 120 min.	250-270°C
2115	Yes	No	as received	Incubator	4 h	90°C
2129	Yes	No	as received	Oven	about 1 hr	about 300°C
2146	Yes	Yes, with a brush	as received	Incubator	Evap. 360 min.	***)
2165	Yes	No	as received	Oven	Overnight	105
2184	Yes	No	used 2 pieces	Oven	720	100
2212	Yes	Yes, D.I.	as received	Oven	NA	NA
2213	Yes	Yes	as received	Oven	-----	-----
2216	No	No	as received	Oven	102	416
2236	---	---	---	---		
2241	Yes	No	as received	Oven	2.5h	150°C
2284	Yes	Yes, with a non-woven cloth	as received	Oven	12h	105 °C
2353	Yes	No	as received	Oven	30 min.	105 C
2372	Yes	No	as received	Oven	About 60 min.	260 °C.
2375	Yes	No	as received	Oven	-	-
2384	Yes	Yes, with lint-free tissue	as received	Oven	180-240	200
2385	Yes	No	---	Oven		
2391	No	Yes, with lint-free tissue	as received	Oven	160min	98°C
2406	No	No	further cut	Oven	Not less than 480	105°C
2415	Yes	No	as received	Oven	480	100-102
2429	Yes	No	further cut	Water bath	124 minutes	280°C
2475	Yes	No	as received	Oven	1080	105
2495	Yes	Yes, with water and soap	as received	Oven	*)	105°C
2500	Yes	No	as received	Oven	120 min.	105°C
2549	Yes	No	as received	Oven	120 min	150°C
2609	No	No	as received	Water bath	3 hours	100℃
2634	Yes	No	as received	Oven	30-45 min	evap. by hot plate
2799	Yes	No	as received	Oven		
2826	Yes	No	as received	Oven	16 mins	100°C
2840	No	No	as received	Oven/Waterbath	-----	100°C
2850	Yes	No	as received	Incubator	1440	105
2897	Yes	Yes	as received	Oven	1 hour	105°C
2938	No	No	as received	Oven	95	105°C
3100	Yes	Yes, with lint free cloth	as received	Oven	One hour	105°C
3110	Yes	---	---	---		
3116	No	No	further cut	Oven	7	93
3153	Yes	No	as received	Oven	40 minutes	95°C
3172	---	---	---	---		
3182	Yes	No	as received	Oven	**)	**)
3185	Yes	Yes, with distilled water	as received	Oven	120 min.	105 °C
3190	Yes	No	as received	Oven	About 2 hrs	About 200°C
3218	---	---	---	---		
3228	Yes	No	as received	Oven	more than 12h	105oC
3237	Yes	Yes, with dust free cloth	as received	Oven		

*) lab 2495: We put direclty total volume of simulant in quartz capsules
**) lab 3182: time: Knife 1: 2 hours 11 minutes, Knife 2: 2 hours 17 minutes, Knife 3: 2 hours 24 minutes
temperature: Knife 1 : 98 °C, Knife 2 & Knife 3 : 96 °C
***) lab 2146: Evaporation was done with a hot plate at a temperature of 190 °C.

APPENDIX 4

Number of participants per country

2 labs in BRAZIL
1 lab in BULGARIA
1 lab in FINLAND
2 labs in FRANCE
2 labs in GERMANY
1 lab in GREECE
8 labs in HONG KONG
2 labs in INDIA
1 lab in ISRAEL
5 labs in ITALY
2 labs in MALAYSIA
11 labs in P.R. of CHINA
1 lab in TAIWAN
1 lab in THAILAND
1 lab in THE NETHERLANDS
2 labs in TURKEY
2 labs in U.S.A.
2 labs in UNITED ARAB EMIRATES
1 lab in VIETNAM

APPENDIX 5

Abbreviations

C = final test result after checking of first reported suspect test result
D(0.01) = outlier in Dixon's outlier test
D(0.05) = straggler in Dixon's outlier test
G(0.01) = outlier in Grubbs' outlier test
G(0.05) = straggler in Grubbs' outlier test
DG(0.01) = outlier in Double Grubbs' outlier test
DG(0.05) = straggler in Double Grubbs' outlier test
R(0.01) = outlier in Rosner's outlier test
R(0.05) = straggler in Rosner's outlier test
E = a difference in calculations
ex = test result excluded from statistical evaluation
n.a. = not applicable
n.d. = not detected
n.e. = not evaluated
fr. = first reported

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