Results of Proficiency Test
Overall Migration on
Food Contact Materials
October 2021

CONTENTS

1   INTRODUCTION

During the contact of materials with food, molecules can migrate from the food contact material to the food. Because of this, in many countries regulations are made to ensure food safety. The framework Regulation (EU) No. 10/2011 (lit. 13 – lit. 15) 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 to EU 10/2011 was officially published as 'Commission Regulation (EU) 2020/1245 amending and correcting Regulation (EU) No 10/2011 on plastic materials and articles intended to come into contact with food' (lit. 15). 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 2021/2022 it was decided to continue the proficiency test for the determination of Overall Migration on Food Contact Materials.

In this interlaboratory study 47 laboratories from 18 different countries registered for participation. See appendix 4 for the number of participants per country. In this report the results of the Overall Migration 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 Polypropylene cup sample labelled #21720 positive on Overall Migration.
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 yellow Polypropylene cups fortified with Calcium Carbonate was prepared by a third party. The cups were labelled #21720.

The homogeneity of the subsamples was checked by determination of Overall Migration (1st step only) according to EN1186-9 on 8 stratified randomly selected subsamples with the following conditions: article filling, 3% Acetic Acid, 2 hours at 100°C.

	Overall Migration (1st step) mg/dm^2
sample #21720-1	20.24
sample #21720-2	20.10
sample #21720-3	19.79
sample #21720-4	20.82
sample #21720-5	19.93
sample #21720-6	19.12
sample #21720-7	19.41
sample #21720-8	19.76

Table 1: homogeneity test results of subsamples #21720

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 (1st step) mg/dm^2
r (observed)	1.45
reference test method	EN1186-9:02
0.3 x R (reference test method)	2.88

Table 2: evaluation of the repeatability of subsamples #21720

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

To each of the participants one sample labelled #21720 was sent on September 8, 2021.

2.5 ANALYZES

The participants were requested to determine Overall Migration and to report the test results of three successive migration steps. 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 test conditions known to give a positive test result was prescribed (article filling, repeated use as migration method and 3% M/V Acetic Acid as simulant for 2 hours at 100°C).
In daily practice, not just one item, but more items for testing would have been sent. However, this sample is positive and especially prepared for determination of Overall Migration. This means that one item of the sample is sufficient for the determination of Overall Migration.
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 test 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 reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the 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.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

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, derived from e.g. ISO or ASTM test method 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, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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. Therefore, 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 some problems were encountered with the dispatch of the samples. Three participants reported test results after the final reporting date and three other participants did not report any test results. Not all participants were able to report all tests requested.
In total 44 participants reported 131 numerical test results. Observed were 7 outlying test results, which is 5.3%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

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

4.1   EVALUATION PER TEST

In this section the reported test results are discussed per test. 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. The abbreviations, used in these tables, are explained in appendix 5.

In the past iis has observed that the Overall and Specific Migration methods, limits and calculations are mixed up by participants. Therefore, iis issued a White Paper on this subject in February 2018 (see lit. 16) 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 article filling, the EN1186 method series part 9 is considered to be the official EC test method. In method EN1186-9:02 it is described that five samples are needed: two samples to determine the surface area and three sample for the migration test. In this 2021 PT only one sample (a Polypropylene cup) was available for both surface area determination and the migration test.

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

Overall Migration - 1st step in mg/dm^2: This determination may be problematic for a number of laboratories. Four statistical outliers were observed and three other test results were excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the target reproducibility estimated from EN1186-9:02.

Overall Migration – 2nd step in mg/dm^2: This determination was not problematic. Two statistical outliers were observed and two other test results were excluded. The calculated reproducibility after rejection of the suspect data is in full agreement with the target reproducibility estimated from EN1186-9:02.

Overall Migration – 3rd step in mg/dm^2: This determination was problematic. One statistical outlier was observed and three other test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the target reproducibility estimated from EN1186-9:02.

4.2   PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test 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)
Overall Migration – 1st step	mg/dm^2	37	16.05	5.75	7.74
Overall Migration – 2nd step	mg/dm^2	40	11.06	5.18	5.34
Overall Migration – 3rd step	mg/dm^2	39	8.35	4.86	4.03

Table 3: reproducibility of tests on sample #21720

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

4.3   COMPARISON OF PROFICIENCY TEST OF OCTOBER 2021 AGAINST PREVIOUS PTS

The uncertainty for Overall Migration in mg/dm^2 as observed in this PT and the comparison with the findings in previous rounds is compared to former Overall Migration proficiency tests 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)
2021	13-21%		1	17% (part 9)

Table 4: development of the uncertainties over the years

The uncertainty observed in this PT is almost in line with the uncertainties observed in previous PTs for article filling (the 3rd Overall Migration step with an observed RSD of 21% is slightly higher).

4.4　EVALUATION OF THE ANALYTICAL DETAILS

The reported details of the three successive migration steps and the reported analytical details that were used by the participants are listed in appendices 2 and 3.

Test method and accreditation
About 85% of the reporting participants mentioned to have used test method EN1186-9. From the reporting participants about 90% mentioned that they are accredited for this test.

Preparation
Twenty participants reported not to clean the sample and thirteen participants used a (lint free) cloth/tissue before the determination of the Overall Migration.
A few participants reported to have used water and/or a detergent/soap to clean the test item prior to use. Method EN1186-9 states in paragraph 6.1: "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.

All participants reported to have heated the simulant heated to 100°C before the sample was filled with simulant.

Ratio dm^2 per 100 mL, contact surface area and volume of simulant
With article filling the surface to volume ratio varies with the volume that is chosen to fill the article. The chosen volume of simulant differs from 190mL to 260mL and most of the participants found a ratio which varied between 0.53 and 0.87 dm^2/100mL. Four participants used a ratio which was significantly different, the test results of these participants where therefore excluded for the statistical evaluation.

Evaporation
After exposure of the cup to the simulant for the selected time, the simulant must be evaporated to low volume. About 85% of the reported participants directly evaporated the simulant in a dish (evaporation method) and about 15% first distilled the simulant before further evaporation (distillation method).
The reported evaporation time of the simulant to low volume varied from 30 minutes to overnight. The reported temperature varied from 100°C to more than 250°C. These differences did not appear to be of influence in this PT.

Calculation of Overall Migration in mg/dm^2
The calculation was checked from the reported results for total residue, contact surface area and volume of simulant. The majority of the participants calculated the Overall Migration with the equation of paragraph 8.1.1 of EN1186-9. Some participants calculated the migration with the equation of paragraph 8.1.2 of EN1186-9. The influence of this difference in calculation is small and only relevant for those laboratories that have found a high total residue.
For some participants the Overall Migration calculated by iis is not in line with the reported total residue (mg) and the reported surface area (dm^2) using equation of paragraph 8.1.1 nor using equation of paragraph 8.1.2. One participant reported possibly the total residue in grams and not in milligrams.

5   DISCUSSION

Total immersion, repeated use, one article
A cup is in general an article for multiple use. Therefore, it was requested to report the Overall Migration (mg/dm^2) for three successive migration steps.
In this PT, the average Overall Migration in (mg/dm^2) decreased in each successive migration step (step 1: 16 mg/dm^2, step 2: 11 mg/dm^2 and step 3: 8 mg/dm^2). The majority of the laboratories (except three participants) reported also a decrease in the three successive steps.

Limits for Overall Migration from EU regulation No 10/2011 and 15th amendment to EU 10/2011
The new EU regulation 1245/2020, the 15th amendment of EU10/2011 has been approved in September 2020 and should be implemented before March 27, 2021 for new products and in 2022 for existing products. In this amendment a few approval regulations are mentioned. For repeated use the migration test result should be less for each following step (OM1>OM2>OM3). The third step will be leading for the end result of approval of the sample.

The EU regulation describes in article 12 that the limit for Overall Migration is 10 mg/dm^2.
In this 2021 PT the cup was tested in three successive contact periods, using a new portion of simulant for each exposure period. The Overall Migration found in the third migration step should comply the limit for Overall Migration (10 mg/dm^2).
According to this limit eight participants would have rejected the cup based on the test result of the third migration step.
Two of the three participants who didn't have a lower Overall Migration (OM) in a next migration step (and not in compliance with OM1 > OM2 > OM3), found in the third migration step an Overall Migration > 10 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 prescribed but will be selected by the individual laboratories.

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

APPENDIX 1

Determination of 1st Overall Migration (per contact surface) on sample #21720; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
310		-----		-----	
339	EN1186-9	18.43		0.86	
551	EN1186	16.79		0.27	
840	EN1186	3.86	R(0.05)	-4.41	
1648	EN1186-9	17.950		0.69	
2115	EN1186-9	16.09		0.01	
2132	EN1186-9	16.60		0.20	
2165	GB31604-8/EN1186-9	14.224		-0.66	
2184	EN1186-9	14.113		-0.70	
2241	EN1186-9	17.42		0.49	
2247	EN1186-9	18.32		0.82	
2256	EN1186-9	13.27		-1.01	
2353	EN1186-9	18.47		0.87	
2369	EN1186-9	17.973	ex	0.69	test result excluded *)
2375	EN1186-9	13.653		-0.87	
2379	EN1186	14.221		-0.66	
2384	EN1186-9	19.6875		1.31	
2385		16.28		0.08	
2429	EN1186-3	13.94		-0.76	
2446	EN1186-9	16.44		0.14	
2475	EN1186-9	13.192		-1.03	
2504	EN1186-9	17.582		0.55	
2525	EN1186-9	17.84		0.65	
2703		-----		-----	
2729	EN1186-1	35.6595	C,R(0.01)	7.09	first reported 32.9514
2799	EN1186-9	8.7565	ex	-2.64	test result excluded *)
2826	EN1186-9	17.058		0.36	
2850	EN1186-3	7.3	R(0.05)	-3.16	
2867	EN1186-9	14.430		-0.59	
2892	EN1186-9	14.188		-0.67	
2901	EN1186-9	17.688		0.59	
2910	EN1186-9	14.329		-0.62	
2958		-----		-----	
2976	EN1186-9	6.788	R(0.05)	-3.35	
3100	EN1186-9	14.742		-0.47	
3116	EN1186-9	15.10		-0.34	
3122	EN1186-9	11.7	ex	-1.57	test result excluded *)
3153	EN1186-9	16.189		0.05	
3172	EN1186-9	18.1514		0.76	
3182	EN1186-9	14.36		-0.61	
3185	EN1186-9	21.484		1.96	
3190	EN1186-9	15.77		-0.10	
3218	EN1186-9	15.756		-0.11	
3225	EN1186-9	18.4		0.85	
3228	EN1186-9	14.2		-0.67	
3246	EN1186-9	14.759		-0.47	
3248	EN1186-9	12.7976		-1.18	
	normality	OK			
	n	37			
	outliers	4 + 3ex			
	mean (n)	16.052			
	st.dev. (n)	2.0552	RSD = 13%		
	R(calc.)	5.755			
	st.dev.(EN1186-9:02)	2.7652			
	R(EN1186-9:02)	7.743			

*) test result excluded for it is an outlier in ratio contact surface area per volume of simulant (dm^2/100mL), see appendix 2
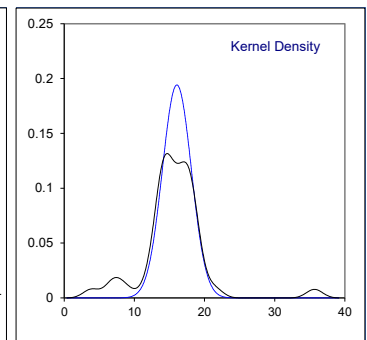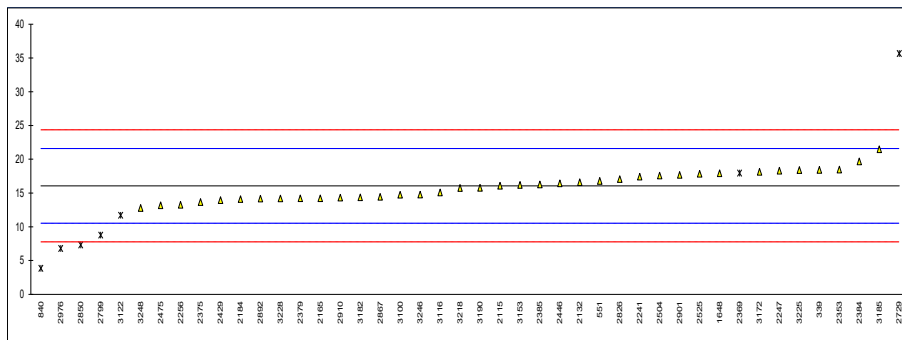
Determination of 2nd Overall Migration (per contact surface) on sample #21720; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
310		-----		-----	
339	EN1186-9	14.95		2.04	
551	EN1186	9.372		-0.89	
840	EN1186	2.99	R(0.05)	-4.24	
1648	EN1186-9	9.627		-0.75	
2115	EN1186-9	12.56		0.79	
2132	EN1186-9	12.68		0.85	
2165	GB31604-8/EN1186-9	11.988		0.49	
2184	EN1186-9	11.835		0.41	
2241	EN1186-9	11.53		0.25	
2247	EN1186-9	10.87		-0.10	
2256	EN1186-9	9.52		-0.81	
2353	EN1186-9	14.20		1.65	
2369	EN1186-9	40.135	R(0.01)	15.25	
2375	EN1186-9	9.831		-0.65	
2379	EN1186	11.429		0.19	
2384	EN1186-9	11.5625		0.26	
2385		10.32		-0.39	
2429	EN1186-3	10.34		-0.38	
2446	EN1186-9	8.53		-1.33	
2475	EN1186-9	8.362		-1.42	
2504	EN1186-9	13.133		1.09	
2525	EN1186-9	8.29		-1.45	
2703		-----		-----	
2729	EN1186-1	12.8270		0.93	
2799	EN1186-9	5.1295	ex	-3.11	test result excluded *)
2826	EN1186-9	10.829		-0.12	
2850	EN1186-3	11.3		0.12	
2867	EN1186-9	11.582		0.27	
2892	EN1186-9	11.062		0.00	
2901	EN1186-9	10.462		-0.32	
2910	EN1186-9	11.890		0.43	
2958		-----		-----	
2976	EN1186-9	6.641		-2.32	
3100	EN1186-9	11.550		0.26	
3116	EN1186-9	13.30		1.17	
3122	EN1186-9	9.6	ex	-0.77	test result excluded *)
3153	EN1186-9	11.245		0.10	
3172	EN1186-9	8.7012		-1.24	
3182	EN1186-9	9.14		-1.01	
3185	EN1186-9	13.281		1.16	
3190	EN1186-9	10.88		-0.10	
3218	EN1186-9	12.267		0.63	
3225	EN1186-9	13.1		1.07	
3228	EN1186-9	12.7		0.86	
3246	EN1186-9	11.500		0.23	
3248	EN1186-9	7.3214		-1.96	

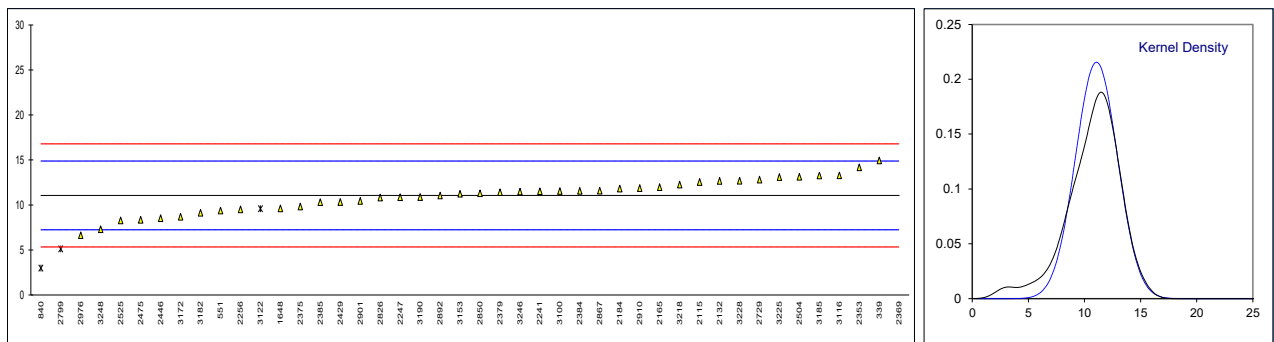|				
	normality	OK		
	n	40		
	outliers	2 + 2ex		
	mean (n)	11.063		
	st.dev. (n)	1.8504	RSD = 17 %	
	R(calc.)	5.181		
	st.dev.(EN1186-9:02)	1.9058		
	R(EN1186-9:02)	5.336		

*) test result excluded for it is an outlier in ratio contact surface area per volume of simulant (dm^2/100mL), see appendix 2

Determination of 3rd Overall Migration (per contact surface) on sample #21720; results in mg/dm²

lab	method	value	mark	z(targ)	remarks
310		-----		-----	
339	EN1186-9	10.88		1.76	
551	EN1186	11.573		2.24	
840	EN1186	2.20	ex	-4.28	test result excluded *)
1648	EN1186-9	6.397		-1.36	
2115	EN1186-9	9.74		0.97	
2132	EN1186-9	10.49		1.49	
2165	GB31604-8/EN1186-9	9.814		1.02	
2184	EN1186-9	9.873		1.06	
2241	EN1186-9	8.84		0.34	
2247	EN1186-9	8.50		0.10	
2256	EN1186-9	6.39		-1.36	
2353	EN1186-9	10.44		1.45	
2369	EN1186-9	20.541	R(0.01)	8.48	
2375	EN1186-9	7.360		-0.69	
2379	EN1186	10.135		1.24	
2384	EN1186-9	8.90625		0.39	
2385		7.67		-0.47	
2429	EN1186-3	6.91		-1.00	
2446	EN1186-9	6.05		-1.60	
2475	EN1186-9	5.911		-1.70	
2504	EN1186-9	9.114		0.53	
2525	EN1186-9	5.59		-1.92	
2703		-----		-----	
2729	EN1186-1	6.6270		-1.20	
2799	EN1186-9	3.7824	ex	-3.18	test result excluded *)
2826	EN1186-9	5.5916		-1.92	
2850	EN1186-3	4.7		-2.54	
2867	EN1186-9	9.747		0.97	
2892	EN1186-9	7.625		-0.50	
2901		-----		-----	
2910	EN1186-9	9.817		1.02	
2958		-----		-----	
2976	EN1186-9	6.493		-1.29	
3100	EN1186-9	9.727		0.96	
3116	EN1186-9	10.15		1.25	
3122	EN1186-9	8.0	ex	-0.24	test result excluded *)
3153	EN1186-9	8.504		0.11	
3172	EN1186-9	7.8944		-0.32	
3182	EN1186-9	7.67		-0.47	
3185	EN1186-9	8.359		0.01	
3190	EN1186-9	8.32		-0.02	
3218	EN1186-9	8.547		0.14	
3225	EN1186-9	10.6		1.56	
3228	EN1186-9	9.9		1.08	
3246	EN1186-9	8.034		-0.22	
3248	EN1186-9	6.726		-1.13	

	normality	OK		
	n	39		
	outliers	1 + 3ex		
	mean (n)	8.349		
	st.dev. (n)	1.7362		
	R(calc.)	4.861	RSD = 21%	
	st.dev.(EN1186-9:02)	1.4383		
	R(EN1186-9:02)	4.027		

*) test result excluded for it is an outlier in ratio contact surface area per volume of simulant (dm²/100mL), see appendix 2
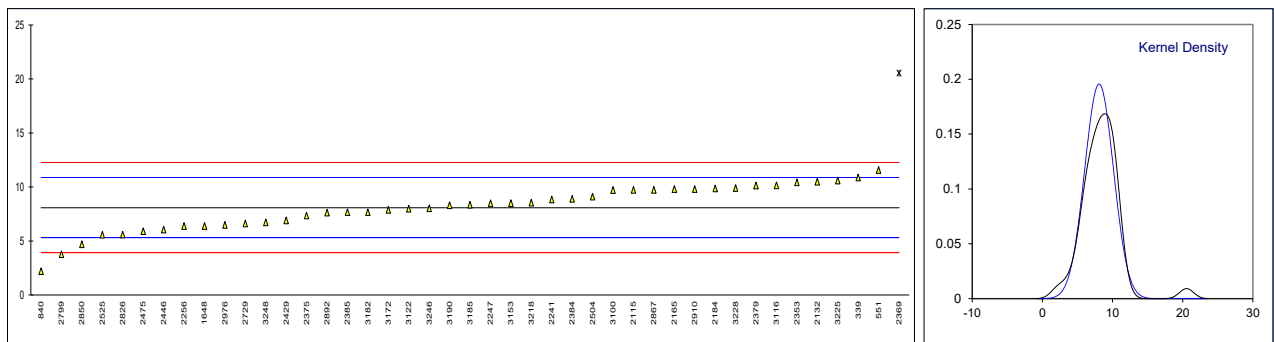
APPENDIX 2

Details for calculation of Overall Migration on sample #21720; results in mg/dm^2

	1st step			2nd step			3rd step		
	reported	iis calculated with equation of EN1186-9		reported	iis calculated with equation of EN1186-9		reported	iis calculated with equation of EN1186-9	
lab		8.1.1	8.1.2		8.1.1	8.1.2		8.1.1	8.1.2
310	-----			-----			-----		
339	18.43		18.7121	14.95		15.0758	10.88		11.2121
551	16.79	**18.8264**	**21.6504**	9.372	**12.3064**	**14.1524**	11.573	**13.7734**	**15.8394**
840	3.86		3.8621	2.99		2.9900	2.20	1.7608	
1648	17.950	19.2045	**23.0454**	9.627	**10.8763**	**13.0516**	6.397	**7.6445**	**9.1734**
2115	16.09	16.0897		12.56	12.5641		9.74	9.7436	
2132	16.60	16.6000		12.68	12.6800		10.49	10.4900	
2165	14.224	14.2236		11.988	11.9876		9.814	9.8137	
2184	14.113	14.1139		11.835	11.8354		9.873	9.8734	
2241	17.42	17.4235		11.53	11.5294		8.84	8.8412	
2247	18.32	18.3237		10.87	10.8671		8.50	8.4971	
2256	13.27	13.2667		9.52	9.5159		6.39	6.3902	
2353	18.47		18.4662	14.20		14.1951	10.44		10.4422
2369	17.973	**0.0180** *)	**0.0225** *)	40.135	**0.0401** *)	**0.0502** *)	20.541	**0.0205** *)	**0.0257** *)
2375	13.653		13.6789	9.831		9.8548	7.360		7.3726
2379	14.221	14.2209		11.429	11.4294		10.135	10.1350	
2384	19.6875	**7.8642**	**9.8302**	11.5625	**4.6186**	**5.7733**	8.90625	**3.5576**	**4.4470**
2385	16.28	15.8427		10.32	10.1685		7.67	7.6404	
2429	13.94	13.9429		10.34	10.3429		6.91	6.9143	
2446	16.44	16.4407		8.53	8.5311		6.05	6.0452	
2475	13.192		13.7684	8.362		8.9387	5.911	5.5215	
2504	17.582	17.5816		13.133	13.1324		9.114	9.5443	
2525	17.84	17.8378		8.29	8.2883		5.59	5.5856	
2703	-----			-----			-----		
2729	35.6595 C	35.6595		12.8270	12.8270		6.6270	6.6270	
2799	8.7565	**0.0087**	**0.0087**	5.1295	**0.0051**	**0.0051**	3.7824	**0.0038**	**0.0038**
2826	17.058		17.0579	10.829		10.8293	5.5916		5.5916
2850	7.3	7.2937		11.3	11.2604		4.7	4.6705	
2867	14.430	14.4304		11.582	11.5823		9.747	9.7468	
2892	14.188	14.3750		11.062	11.1250		7.625	7.8125	
2901	17.688	17.6369		10.462	10.4323		-----		
2910	14.329	14.3293		11.890	11.8902		9.817	9.8171	
2958	-----			-----			-----		
2976	6.788	6.7870		6.641	6.6411		6.493	6.4953	
3100	14.742		14.7419	11.550		11.5503	9.727		9.7266
3116	15.10	15.1000		13.30	13.3000		10.15	10.1500	
3122	11.7	11.6667		9.6	9.5714		8.0	8.0476	
3153	16.189	16.1890		11.245	11.2451		8.504	8.5039	
3172	18.1514	18.1504		8.7012	8.7007		7.8944	7.8940	
3182	14.36	15.0920		9.14	9.8773		7.67	8.4049	
3185	21.484		21.7969	13.281		13.7500	8.359		8.9063
3190	15.77	16.0652		10.88	11.1758		8.32	8.6147	
3218	15.756	15.7558		12.267	12.2674		8.547	8.5465	
3225	18.4	**7.6687**	**9.2025**	13.1	**5.4601**	**6.5521**	10.6	**4.4172**	**5.3006**
3228	14.2	14.1762		12.7	12.7287		9.9	9.9085	
3246	14.759	14.7598		11.500	11.5000		8.034	8.0349	
3248	12.7976	12.7976		7.3214	7.3214		6.726	6.7262	

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 2369 possibly reported total residue in grams?

Lab 2729 first reported 32.9514

Details for calculation of Overall Migration on sample #21720

| lab	1st step Total residue (mg)	surface area (dm²)	volume simulant (mL)	2nd step Total residue (mg)	surface area (dm²)	volume simulant (mL)	3rd step Total residue (mg)	surface area (dm²)	volume simulant (mL)	iis calc. ratio area/simulant (dm²/100 mL)
310	-----	-----	-----	-----	-----	-----	-----	-----	-----	
339	24.7	1.65	250	19.9	1.65	250	14.8	1.65	250	0.6600
551	23.1	1.227	230	15.1	1.227	230	16.9	1.227	230	0.5335
840	9.3	3.01	250	7.2	3.01	250	5.3	3.01	250	1.2040 R1
1648	30.9	1.609	240	17.5	1.609	240	12.3	1.609	240	0.6704
2115	25.1	1.56	240	19.6	1.56	240	15.20	1.56	240	0.6500
2132	26.56	1.60	250	20.288	1.60	250	16.784	1.60	250	0.6400
2165	22.9	1.61	245	19.3	1.61	245	15.8	1.61	245	0.6571
2184	22.3	1.58	240	18.7	1.58	240	15.6	1.58	240	0.6583
2241	29.62	1.70	235	19.60	1.70	235	15.03	1.70	235	0.7234
2247	31.70	1.73	240.00	18.80	1.73	240.00	14.70	1.73	240.00	0.7208
2256	19.1	1.4397	200	13.7	1.4397	200	9.2	1.4397	200	0.7199
2353	23.52	1.5921	250	18.08	1.5921	250	13.30	1.5921	250	0.6368
2369	0.0133 *)	0.74	250	0.0297 *)	0.74	250	0.0152 *)	0.74	250	0.2960 R1
2375	18.461	1.687	250	13.300	1.687	250	9.95	1.687	250	0.6748
2379	23.18	1.63	250	18.63	1.63	250	16.52	1.63	250	0.6520
2384	12.6	1.6022	250	7.4	1.6022	250	5.7	1.6022	250	0.6409
2385	28.2	1.78	210	18.1	1.78	210	13.6	1.78	210	0.8476
2429	24.4	1.75	240.0	18.1	1.75	240.0	12.1	1.75	240.0	0.7292
2446	29.1	1.77	224	15.1	1.77	226	10.7	1.77	224	0.7902
2475	19.1	1.63	235	12.4	1.63	235	9.0	1.63	235	0.6936
2504	24.5	1.3935	200	18.3	1.3935	200	13.3	1.3935	200	0.6968
2525	29.7	1.665	250	13.8	1.665	250	9.3	1.665	250	0.6660
2703	-----	-----	-----	-----	-----	-----	-----	-----	-----	
2729	65.97 C	1.85	260	23.73	1.85	260	12.26	1.85	260	0.7115
2799	0.0169	1.9345	200	0.0099	1.9345	200	0.0073	1.9345	200	0.9673 R1
2826	24.1	1.6954	240	15.3	1.6954	240	7.9	1.6954	240	0.7064
2850	11.4	1.563	210	17.6	1.563	210	7.3	1.563	210	0.7443
2867	22.8	1.58	250	18.3	1.58	250	15.4	1.58	250	0.6320
2892	23.0	1.6	250	17.8	1.6	250	12.5	1.6	250	0.6400
2901	30.6	1.735	200	18.1	1.735	240	-----	-----	-----	0.8675
2910	23.5	1.640	250	19.5	1.640	250	16.1	1.640	250	0.6560
2958	-----	-----	-----	-----	-----	-----	-----	-----	-----	
2976	11.17 C	1.6458	242.91	10.93 C	1.6458	242.91	10.69 C	1.6458	242.91	0.6775
3100	19.4	1.612076	245	15.2	1.612076	245	12.8	1.612076	245	0.6580
3116	30.2	2.00	244	26.6	2.00	244	20.3	2.00	244	0.8197
3122	24.5	2.1	190	20.1	2.1	190	16.9	2.1	190	1.1053 R1
3153	26.966	1.6657	240	18.731	1.6657	240	14.165	1.6657	240	0.6940
3172	31.5	1.7355	250	15.1	1.7355	250	13.7	1.7355	250	0.6942
3182	24.6	1.63	250.0	16.1	1.63	250.0	13.7	1.63	250.0	0.6520
3185	27.9	1.60	250	17.6	1.60	250	11.4	1.60	250	0.6400
3190	27.6	1.718	250	19.2	1.718	250	14.8	1.718	250	0.6872
3218	27.1	1.72	250	21.1	1.72	250	14.7	1.72	250	0.6880
3225	12.5	1.63	240	8.9	1.63	240	7.2	1.63	240	0.6792
3228	23.249	1.64	250	20.875	1.64	250	16.250	1.64	250	0.6560
3246	20.280	1.374	200	15.801	1.374	200	11.040	1.374	200	0.6870
3248	21.50	1.68	230	12.30	1.68	230	11.30	1.68	230	0.7304

Lab 551   the overall migration (OM) in the third step is higher than OM in the second step (OM1 > OM2 < OM3)
Lab 2369 the overall migration (OM) in the second step is higher than OM in the first step (OM1 < OM2 > OM3),
          possibly reported total residue in grams?
Lab 2850 the overall migration (OM) in the second step is higher than OM in the first step (OM1 < OM2 > OM3)
Lab 2729 first reported 60.96
Lab 2729 first reported 9.2, 9.0 and 8.8 respectively
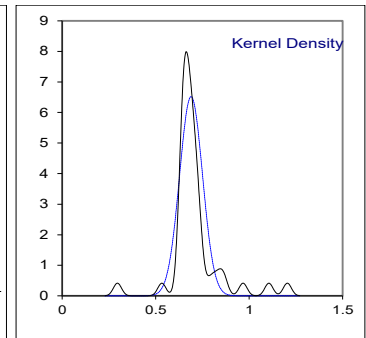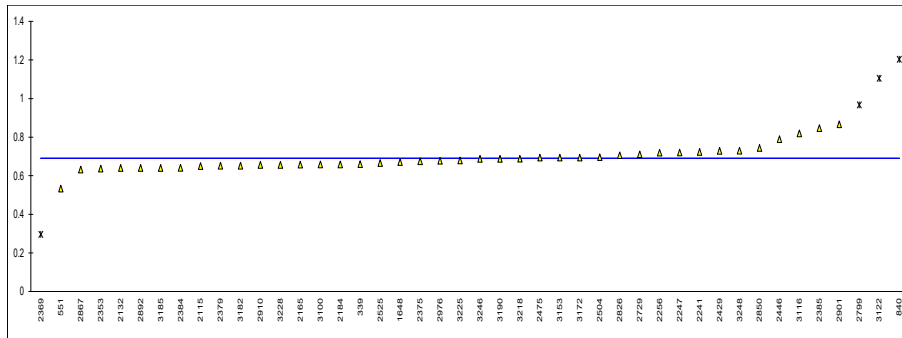
APPENDIX 3　　Analytical details

lab	ISO 17025 accr.	Cleaned prior to migration step	simulant heated to 100°C	ratio surface/volume (dm2/mL)
310	---	---	---	
339	Yes	No	Yes	1.65dm² / 250ml
551	Yes	No	Yes	0.00534
840	Yes	No	Yes	3.01 dm2/250 mL
1648	No	No	Yes	The surface-to-volume ratio was 0.007dm2/mL. The cup was filled with the simulant to 5mm from the top.
2115	Yes	No	Yes	1.56/240
2132	Yes	Yes, lint-free cloth	Yes	0.006 dm2/mL. It is because the sample is container.
2165	Yes	Yes, lint-free cloth	Yes	1.61 dm2/245mL = 0.00657 dm2/mL
2184	Yes	Yes, lint-free cloth	Yes	1.58dm2/240ml
2241	Yes	No	Yes	0.0068
2247	Yes	Yes, lint-free tissue paper	Yes	0.72
2256	Yes	No	Yes	0.7198dm2/100mL
2353	Yes	Yes	Yes	0.006384
2369	Yes	Yes	Yes	0.74/250=0.00296dm2/mL
2375	Yes	No	Yes	1.479
2379	Yes	No	Yes	0.007
2384	Yes	Yes, lint-free tissue	Yes	0.0064
2385	Yes	Yes, warm water	Yes	1,78 dm2/210 ml
2429	Yes	Yes, soft cloth	Yes	S/V=1.75dm2/240.0mL
2446	Yes	Yes, dest. water	Yes	0,0078 and 0,0079
2475	Yes	No	Yes	1.63/235. We fill with the simulant up to 5mm from the RIM.
2504	Yes	Yes, tissue paper	Yes	1.3935 dm2 / 200mL
2525	Yes	No	Yes	0,00666
2703	---	---	---	
2729	No	Yes, once with distilled water	Yes	0.0071
2799	Yes	Yes, cloth	Yes	0.0096
2826	Yes	No	Yes	0.006, for container less than 500ml
2850	Yes	No	Yes	0,0074
2867	Yes	No	Yes	1.58 dm2/250 mL
2892	Yes	Yes, lint-free cloth	Yes	For articles that are fillable and have a volume below 500 mL or greater than 10 L, the analytical result needs to be corrected for the difference in surface-to-volume ratio between the experiment and the food contact conditions of 6 dm2/kg.
2901	Yes	No	Yes	1.73/240= 0.0072
2910	Yes	Yes	Yes	1.64dm2/250mL
2958	---	---	---	
2976	No	Yes, lint-free cloth	Yes	0.006776
3100	Yes	Yes, rinsing several times by distilled water	Yes	contact surface area:1.61dm2 volume of simulant:245mL surface-to-volume:0.00657dm2/mL
3116	No	No	Yes	1dm2 to 122mL
3122	Yes	No	Yes	0.01
3153	Yes	Yes, lint-free cloth	Yes	0.00666 dm2/mL
3172	Yes	No	Yes	0.0069416
3182	Yes	No	Yes	1.63/250
3185	Yes	Yes, rinsed by water	Yes	1.60dm2/250mL
3190	Yes	Yes, soft cloth	Yes	1.718dm2 / 250mL
3218	Yes	Yes	Yes	1.72dm2/250mL
3225	Yes	Yes, remove the dirt on surface	Yes	By article filling: 1.63dm2/240mL = 0.0068dm2/ml
3228	Yes	Yes, lint-free cloth	Yes	0.00656
3246	Yes	Yes, distilled water	Yes	0.7
3248	Yes	No	Yes	1

Summary of reported analytical details - continued

lab	simulant total evaporated in a dish of first a part distilled	evaporation time of the simulant to low volume	evaporation Temp. to low volume (°C)
310	---		
339	only 200ml from 250ml evaporated.	-	95°C
551	Evaporation method		
840	Evaporation method	3 hours	100°C
1648	Evaporation method	1st step: 435min / 2nd step: 425min / 3rd step: 395min / Blank: 345min	250°C - 350°C
2115	Evaporation method	180 min	170°C
2132	First distilled before further evaporation	120 minutes	100°C for evaporation
2165	Evaporation method	over night	105°C
2184	Evaporation method	over night	105°C
2241	First distilled before further evaporation	about 180 minutes	on a hot plate at 220°C
2247	Evaporation method	150°C via hot plate up to 10ml than 10ml left in oven overnight @100°C	100°C
2256	Evaporation method	120min	around 100°C
2353	Evaporation method	180	105
2369	Evaporation method	about 120min	about 200°C
2375	Evaporation method	-	-
2379	Evaporation method	2 hours	100 °C
2384	Evaporation method	240-360 Min	220
2385	---	120	100
2429	Evaporation method	About 2.5 hours.	270 °C
2446	Evaporation method	480 min	ca. 103°C
2475	Evaporation method	1080	105°C
2504	Evaporation method	90 mins	105°C
2525	Evaporation method	75	250
2703	---		
2729	Evaporation method	2880	110°C
2799	Evaporation method	120 minutes	100
2826	Evaporation method	40 minutes	106°C
2850	Evaporation method	4200	100°C
2867	Evaporation method	4 hours	105 °C
2892	Evaporation method	about 2 hours	about 250°C
2901	Evaporation method	300	240
2910	First distilled before further evaporation	120minuts	105 °C
2958	---		
2976	Evaporation method	120	hot plate, increase temperature slowly
3100	Evaporation method	40mins	105℃
3116	First distilled before further evaporation	90 minutes	100°C
3122	Evaporation method	About 24 h	Not measured
3153	Evaporation method	180mins	100°C
3172	Evaporation method	600	100
3182	Evaporation method	1st step = 1 hr 29 min 31 sec 2nd step = 1 hr 57 min 48 sec 3rd step = 1 hr 50 min 27 sec	98 °C
3185	First distilled before further evaporation	6 hours	105℃
3190	Evaporation method	About 180 minutes.	About 200℃
3218	First distilled before further evaporation	3hours	105°C
3225	Evaporation method	120	105
3228	Evaporation method	more than 12h	105
3246	Evaporation method	240 minutes	110°C
3248	Evaporation method	30 minutes	

APPENDIX 4

Number of participants per country

  2 labs in BRAZIL

  2 labs in FRANCE

  3 labs in GERMANY

   1 lab in GREECE

  8 labs in HONG KONG

   1 lab in INDIA

  3 labs in ITALY

   1 lab in MALAYSIA

12 labs in P.R. of CHINA

   1 lab in SOUTH KOREA

   1 lab in SPAIN

   1 lab in SRI LANKA

  3 labs in THAILAND

   1 lab in THE NETHERLANDS

   1 lab in TURKEY

   1 lab in UNITED ARAB EMIRATES

   1 lab in UNITED KINGDOM

  4 labs in VIETNAM

APPENDIX 5

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01), R1	= outlier in Rosner's outlier test
R(0.05), R5	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported

Literature

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14   Union Guidelines on Regulation (EU) No 10/2011 on plastic materials and articles intended to come into contact with food, 21st of February 2014, website: ec.europa.eu
15   15th amendment to EU 10/2011 of September 2020, published as 'Commission Regulation (EU) 2020/1245 amending Regulation (EU) No 10/2011 on plastic materials and articles intended to come into contact with food'
16   iis memo 1801 White paper on the determinations of Overall and Specific migration on food contact materials