Results of Proficiency Test
Overall migration (fcm)
October 2015

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. C.M. Nijssen-Wester & dr. R.G. Visser
Correctors: ing. R.J. Starink & ing. Y. Akin

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CONTENTS

1 INTRODUCTION

On request of a number of participants in the iis PT program it was decided to start PTs on food contact materials in 2012. Since 2012 iis has organised a PT on Overall Migration every year. During the contact of the food contact materials with the food, molecules can migrate from the food contact material to the food. Because of this, in many countries regulations are made to ensure food safety. The framework Regulation (EC) No. 10/2011 (lit. 18 and lit. 19) applies to all food contact materials and describes a large number of requirements, e.g. limits for overall migration and specific limits for certain constituents. Article 12 of this regulation describes the overall migration limit, expressed in mg/dm^2 to be 10. Only when determined for food contact intended for infants and children, the overall migration is expressed in mg/kg food simulant with a limit of 60 mg/kg food simulant. The determination of specific migration requires additional analytical testing following the migration step, while the determination of the overall (also called global, or total) migration requires weighing as only quantitative analytical technique.
In the iis PT on Overall Migration conducted in September 2015, 63 laboratories from 22 different countries participated (See appendix 4).
In this report, the results of the 2015 proficiency test are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET-UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. It was decided to send one sample (three identical bowls), that gave a positive test result, labelled #15180, and to prescribe a number of test conditions (migration method, type of simulant, exposure time and temperature) to be used. Participants were also requested to report some of the test conditions that the laboratory used.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in accordance with ISO/IEC 17043:2010, (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie, see also www.RVA.nl). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially 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 was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol can be downloaded from 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 salad bowls for single use in the food industry that gave positive test results for Overall Migration was selected.
The homogeneity of the batch was checked by determination of the Overall Migration (48 hrs at 70°C and 3% acetic acid as simulant) on three sets of three stratified randomly selected samples.

3 sets of 3 samples	Overall Migration in mg/dm^2 #15180	Overall Migration in mg/dm^2 average per set #15180
Sample 1	14.50	13.7
Sample 2	13.10	
Sample 3	13.50	
Sample 4	12.40	13.7
Sample 5	14.00	
Sample 6	14.60	
Sample 7	14.00	13.5
Sample 8	14.10	
Sample 9	12.40	

Table 1: results of the homogeneity test on the subsamples #15180

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

	Overall Migration in mg/dm^2 #15180
r(observed)	0.3
reference method	EN1186-9:2002
0.3xR(reference method)	0.9
R(reference method)	2.9

Table 2: evaluation of the repeatability of the migration results on subsamples #15180

The calculated repeatability for Overall Migration on the three sets of three samples #15180 is in good agreement with the estimated target, calculated using EN1186-9 precision data, therefore homogeneity of the samples #15180 was assumed.

To each of the participating laboratories one set of samples #15180 (three identical bowls) was sent on September 9, 2015.

2.5 ANALYSIS

The participants were requested to determine Overall Migration on the sample using the prescribed test conditions (article filling, 48 hrs at 70°C and 3% acetic acid as simulant). It was requested not to report 'less than' results, which are above the detection limit, because such results cannot be used for meaningful statistical calculations.
To get comparable results a detailed report form, on which the units were prescribed as well as the required standards and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. A form to confirm receipt of the samples and a letter of instructions were added to the samples.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were received. The original data are tabulated per sample in the appendix 1 of this report.
The laboratories are represented by the code numbers.
Directly after the deadline, a reminder fax was sent to those laboratories that did not report results at that moment.
Shortly after the deadline, the available results were screened for suspect data. A 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 results. Additional or corrected results are used for the data analysis and the original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

The statistical calculations were performed as described in the procedures in the report 'iis Interlaboratory Studies, Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3).

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

In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon, Grubbs and or Rosner General ESD outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test and by R(0.01) for the Rosner General ESD test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test and by R(0.05) for the Rosner General ESD test (ref. 17). Both outliers and stragglers were not included in the calculations of averages and standard deviations. Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

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 significant consequences for the evaluation of the test results.

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 analysis results are plotted. The corresponding laboratory numbers are under 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 standard. 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 (see appendix 5; nr.14 and 15). 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, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of this interlaboratory study.

The target standard deviation was calculated from the target reproducibility (preferably taken from a standardized test method) by division with 2.8.
The z-scores were calculated in accordance with:

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

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

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 should be done in order to evaluate whether the reported test results are fit-for-purpose.

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 due to an official holiday period in China. Three participants reported test results after the final reporting date and three other participants did not report any test results at all. Finally, 60 of the 63 participants submitted analysis results. These 60 laboratories reported 204 numerical test results. From these results 106 numerical results were used in the statistical evaluations. Observed were 7 statistically outlying results, which is 6.6%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

For the determination of Overall Migration (identical to Global migration or Total Migration) using article filling, the EN1186 method series parts 9 is considered to be the official EC test method. In this PT, as mentioned in the letter of instructions, 3% acetic acid was used as simulant for 48 hrs at 70°C.

Nearly all of the participants reported to have used part 9 of the EN1186 test method (see lit. 4). The reported details of the methods that were used by the participants are listed in appendix 2.

4.1 EVALUATION OF THE REPORTED TEST RESULTS

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

Residue in mg: These intermediate results were not evaluated as they are in principle dependent of the size of the volume of simulant used. Although it was requested to report the total residue after all simulant was evaporated, thirteen participants reported the residue of the part of simulant that was evaporated (residue based on 100 or 200 ml).

Migration in mg/dm^2: This determination was problematic. Four statistical outliers were observed and 16 test results were excluded, see the discussion in paragraph 5. The results were excluded for using a too high or low volume and/or a too high or low surface area. After rejection of the suspect data, the calculated reproducibility is not in agreement with the target reproducibility estimated from EN1186-9:02.

Migration in mg/kg: The determination of migration in mg/kg food simulant was problematic. Three statistical outliers were observed and nineteen results were excluded, see the discussion in paragraph 5. The calculated reproducibility, after rejection of the suspect data was not in agreement with the target reproducibility estimated from EN1186-9.
Three participants did not report migration in mg/kg food simulant, but only a test result in mg/L food simulant. These three test results have been included in this evaluation because the specific gravity may be considered to be one, as stated in method EN1186-9.
Ten laboratories used a factor of 6 to calculate the migration in mg/kg from the migration in mg/dm^2. These ten test results were excluded from the

statistical evaluation because the deviating calculation leads to test results that are not in line with the EN1186-9 test results.
Furthermore, test results reported by participants that used a very low or a very high volume of simulant, were excluded from the evaluation. See the discussion in paragraph 5.

Migration in mg/L: These test results were not evaluated because EN1186-9 test results should be reported either in mg/dm^2 or in mg/kg food simulant.
An overview of the reported test results in mg/L can be found in appendix 3.

4.2 PERFORMANCE EVALUATION OF THE GROUP OF LABORATORIES

The calculated reproducibilities and the target reproducibilities derived from the literature standard method, here EN1186-9 are compared in the next table.

	unit	n	Average	2.8 * sd	R (target)
Overall migration	mg/dm^2	35	12.63	4.90	2.71
Overall migration	mg/kg	29	52.51	20.27	11.29

Table 3: performance overview for samples #15180

4.3 COMPARISON WITH PREVIOUS PROFICIENCY TESTS

The number of participants increased from 46 in 2012 to 63 in this round.

The evolution of the uncertainty for Overall Migration in mg/dm^2 as observed in this proficiency scheme and the comparison with the findings in previous rounds is visualized in table 4.

	article filling	total immersion	EN1186
2012	18%	----	17% (part 8)
2013	----	25-30%	13% (part 3)
2014	18%	----	17% (part 8)
2015	14%	-----	8% (part 9)

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

5 DISCUSSION

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

Sample ID	**#15180**
Simulant	3% acetic acid
Exposure time	48 hrs
Exposure temperature	70.0 °C
Migration method	Article filling
Simulant volume	as per method used

Table 5: preset test conditions used in this PT

Three migration results (mg/dm^2, mg/kg and mg/L) were requested to be reported. Method EN1186-9 only describes reporting in mg/dm^2 or in mg/kg food simulant. In previous PTs it sometimes was unclear, whether a test result was reported in mg/kg or in mg/L. Therefore it was allowed to report a migration result in mg/L as well as in mg/kg food simulant. The participants were requested to report additional details regarding preparation, surface area, simulant volume (all and used for evaporation) and details about the evaporation step (see appendix 2).

Using these intermediate test results and the reported test details, it was possible to check the calculations done by the laboratories. This revealed that several calculation errors were present. A number of laboratories corrected the calculation errors; see the original and the revised test results in appendix 1.

Preparation

Surprisingly twelve participants reported to have used water and/or detergent and some also drying as a preparation. Method EN1186-9 states in paragraph 6.1: “under no circumstances wash the sample with water or solvent”.

Determination of volume of simulant used of #15180 – a salad bowl

The amount of simulant used by each participant varied from 100 – 1450 ml, see appendix 2.
In method EN1186-9 is mentioned that a specimen should be filled to within 0.5 cm from the top. This should lead to a large volume of simulant and consequently also a large contact surface. Looking at the test item, a salad bowl, with a relatively large round bottom, rounded corners and only near the top almost square with an distinctive rim, it is obvious that using a lower simulant volume will result in a much different volume to surface ratio than using a large simulant volume. Based on this, the test results of the participants, which used a simulant volume smaller than 1000 ml, were excluded from the statistical evaluation.
The test item has an edge about 0.5 cm below the top, which according to the method, would be the maximum level to fill the test item. iis measured the maximum volume to 0.5 cm below the top for this sample as 1350 ml. When the bowl was filled to the top edge, a volume of 1500 ml was found. In order to cover the bowl during the test (to avoid dust particles falling in and to prevent evaporation of the simulant), the test item should not be filled to the top edge. Therefore the test results of participants that reported a simulant volume above 1420 ml were excluded.
Thus the test results based on a volume lower than 1000 ml and above 1420 ml were excluded.

Determination of the contact surface used

iis sent three bowls to every participant for the overall migration test. The surface area of the bowls could be determined by using less samples for the test, but the area could also be determined on the bowls after performing the migration test. The contact surface used as reported by the participants varies from 1 – 6.17 dm^2, with one extreme of 8.4387dm^2, see appendix 2. A large spread was found when the reported surface area was compared to the used simulant volume (see figure 1). For the same used volumes, very different surface areas were reported.

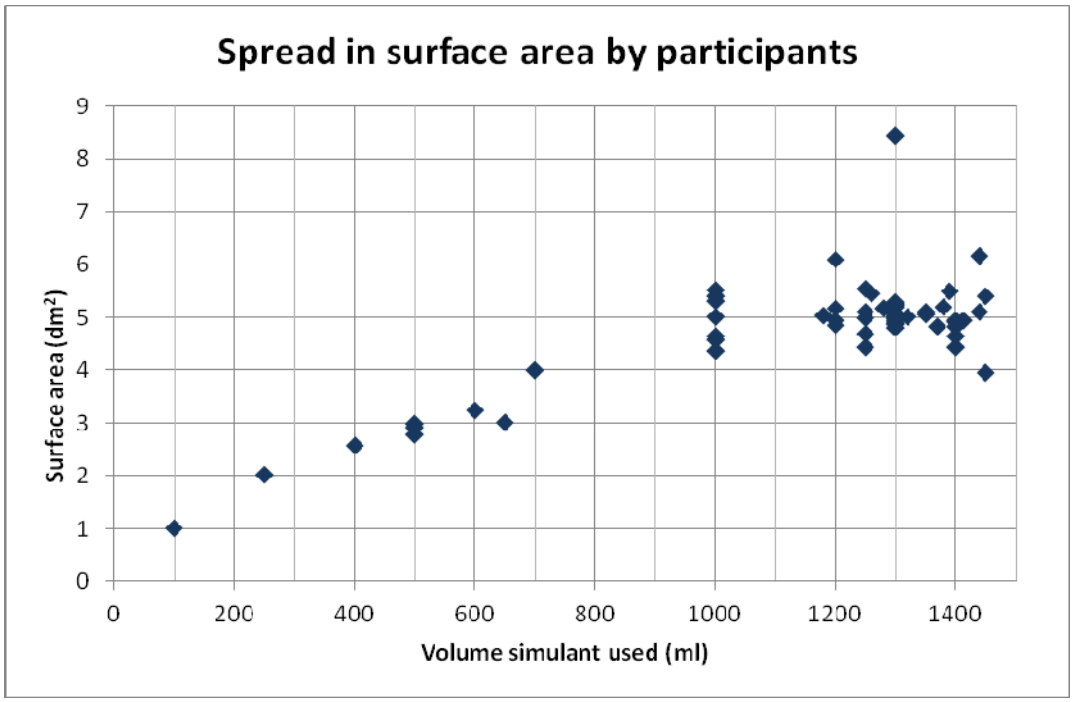


Figure 1: reported surface area versus volume of simulant used by participants

The maximum surface area (to 0.5 cm below the top) for sample #15180 was determined by iis to be approx. 5.85 dm^2. This was done in two ways. First, by measurement with a ruler and the approximation that some rounded parts are squares/triangles. The second way was cutting of the 0.5 cm top edge of the bowl, weighing the remaining bowl, cutting a square sample out of the bowl and determination of the weight/surface ratio. From this, the total surface area of the maximum volume of the bowl could be estimated to be 5.85 dm^2 using 1350 ml of simulant.
Using this information, the test results for the Overall Migration in mg/dm^2 based on a surface area below 4.5 and above 6.2 dm^2 were excluded.

Calculation of Overall Migration in mg/dm^2

According to method EN1186-9, the Overall Migration in mg/dm^2 should be calculated taking the mass residue after evaporation of all simulant and corrected for a blank sample mass in mg by dividing it by the surface area in dm^2. Thirteen participants reported the sample mass in mg for a part of the evaporated simulant, but the result of all residue could be calculated by iis using the volume of simulant used and the volume of the simulant evaporated.

Calculation of Overall Migration in mg/kg food simulant

Ten laboratories used a factor of 6 in the calculation from mg/dm^2 to mg/kg food simulant. This method of calculation is not mentioned in method EN1186-9. Therefore the test results from this calculation method were excluded from the statistical evaluation. However z-scores were

calculated for these test results. Method EN1186-1 does mention using a factor of 6 dm^2 to 1 kg (see paragraph 12.1.2 of the method), but only for unknown surface to volume ratios. Furthermore EN1186-1 states in paragraph 9.7 that the method for article filling is given in EN1186-9.

In EN1186-9, there are two ways of calculating the Overall Migration in mg/kg food simulant. The first, as per formula 2 in paragraph 8.1.1 of EN1186-9, describes a division of the mass of all residue in milligrams by the volume in litres and reporting this value as mg/kg (assuming the specific gravity of the simulant by convention to be one). The second way, as per formula 4 in paragraph 8.1.3 of EN1186-9, describes a division of the mass in milligrams of the residue of 200 ml of simulant times the factor of 5, again reporting the value in mg/kg. When grams are used instead of milligrams also a factor of 1000 is applied.
The majority of participants used either the first or second formula to determine the Overall Migration in mg/kg food simulant.
In the calculation of the test results in mg/kg food simulant the contact surface is irrelevant. Therefore one would expect to find a smaller dispersion in the test results in mg/kg food simulant than in the test results in dm^2, that do contain the uncertainty in the contact surface estimation. Surprisingly there is no significant difference between the two dispersions: 13.9% *vs* 13.7%.

Calculation of Overall Migration in mg/L food simulant
EN1186-9 does not describe reporting migration in mg/L, but in paragraph 8.1.1 under formula 2 is noted that the specific gravity of the simulant is considered to be one. Thus the amount of mg/kg will be the same as in mg/L. The majority of participants reported the same result for both the migration in mg/kg and mg/L.

Limits for overall migration from EU regulation No 10/2011
This EU regulation describes in article 12 that the limit for overall migration is 10 mg/dm2. Using this limit and all reported test results, 93% of the participants would reject this salad bowl.
Should this salad bowl be used in applications that will allow infants and children's food to come in contact, then a overall migration limit of 60 mg/kg is applied. Using this limit and all reported test results, 43% would have rejected the salad bowl, while 57% would have allowed it.
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.

Final remark
It is to be expected that the spread of the migration 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.

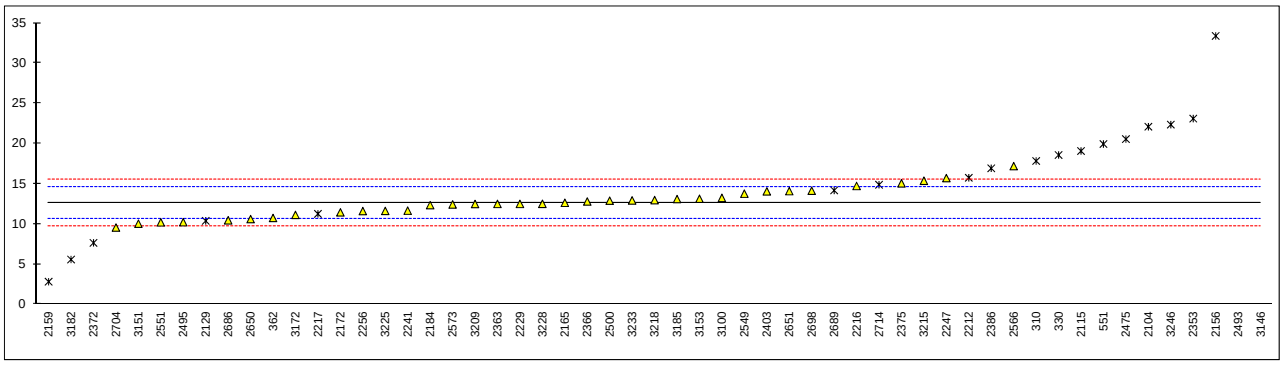
The high spread of amount of simulant volume and/or the determined surface area will also have a large negative effect on the spread of this test.

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

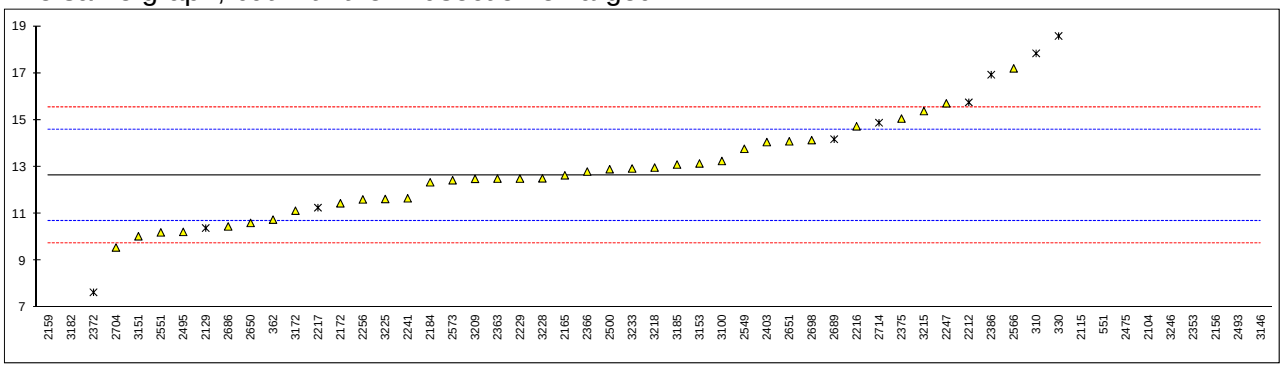
APPENDIX 1:

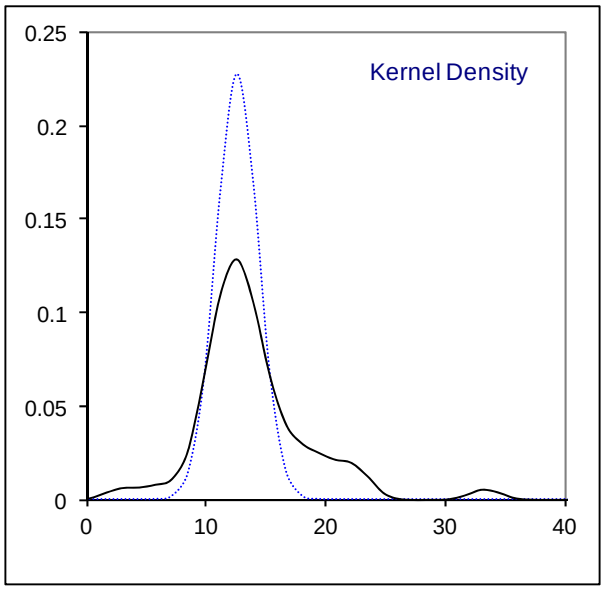
Determination of Overall Migration (per contact surface) on sample #15180; results in mg/dm^2

lab	method	value	mark	-----	remarks
310	EN1186-9	17.84	ex	5.38	excluded due to low volume, see §4.1
330	EN1186-9	18.58	ex	6.14	excluded due to low volume, see §4.1
362	EN1186-9	10.75		-1.94	
452		-----		-----	
551	EN1186-9	19.95	R(0.05)	7.55	
2104	EN1186-9	22.1	ex	9.77	excluded due to low volume, see §4.1
2115	EN1186-9	19.079	R(0.05)	6.65	
2129	EN1186-9	10.39	ex,C	-2.31	excluded due to low volume, see §4.1, first reported: 9.867
2146		-----		-----	
2156	in house	33.39	ex	21.42	excluded due to low volume, see §4.1
2159	EN1186-9	2.82	R(0.01)	-10.12	
2165	EN1186-9	12.64		0.01	
2172	EN1186-9	11.446		-1.22	
2184	EN1186-9	12.343		-0.29	
2186		-----		-----	
2212	CFR175.300	15.75	ex	3.22	excluded due to high volume, see §4.1
2216	CFR175.300	14.73		2.17	
2217	EN1186-9	11.26	ex	-1.41	excluded due to low surface area, see §4.1
2229	EN1186-9	12.5		-0.13	
2241	EN1186-9	11.66		-1.00	
2247	EN1186-9	15.71		3.18	
2256	EN1186-9	11.612		-1.05	
2353	EN1186-9	23.11	ex	10.81	excluded due to high volume, see §4.1
2363	EN1186-9	12.5		-0.13	
2366	EN1186-9	12.8		0.18	
2372	EN1186-9	7.6513	ex	-5.13	excluded due to high surface area, see §4.1
2375	EN1186-9	15.06		2.51	
2386	EN1186-9	16.93	ex	4.44	excluded due to low volume, see §4.1
2403	EN1186-9	14.056		1.47	
2433		-----		-----	
2475	EN1186-9	20.56	ex	8.18	excluded due to high volume and low surface area, see §4.1
2493	EN1186-9	64.3	ex	53.30	excluded due to low volume, see §4.1
2495	EN1186-9	10.227		-2.48	
2500	EN1186-9	12.90		0.28	
2549	EN1186-9	13.77		1.18	
2551	EN1186-9	10.206		-2.50	
2566	EN1186-9	17.2		4.72	
2573	EN1186-9	12.43		-0.20	
2650	EN1186-9	10.610		-2.08	
2651	EN1186-9	14.09		1.51	
2686	EN1186-9	10.46		-2.24	
2689	EN1186-9	14.178	ex	1.60	excluded due to low volume, see §4.1
2698	EN1186-9	14.14		1.56	
2704	EN1186-9	9.56		-3.16	
2707		-----		-----	
2714	EN1186-9	14.881769	ex,C	2.32	excluded due to low surface area, see §4.1, first reported: 1.27511633
3100	EN1186-9	13.25		0.64	
3113		-----		-----	
3146	EN1186-9	343.5	R(0.01)	341.31	
3151	EN1186-9	10.04		-2.67	
3153	EN1186-9	13.145		0.53	
3154		-----		-----	
3172	EN1186-9	11.13		-1.55	
3182	EN1186-9	5.58	ex	-7.27	excluded due to low surface area, see §4.1
3185	EN1186-9	13.1		0.49	
3209	EN1186-9	12.49		-0.14	
3215	EN1186-9	15.38		2.84	
3218	EN1186-9	12.97		0.35	
3220		-----		-----	
3225	EN1186-9	11.63		-1.03	
3228	EN1186-9	12.51		-0.12	
3233	EN1186-9	12.93		0.31	
3246	EN1186-9	22.3606	ex	10.04	excluded due to low volume, see §4.1
	normality	OK			
	n	35			
	outliers	4 (+16 ex)			
	mean (n)	12.628			
	st.dev. (n)	1.7498			
	R(calc.)	4.899			
	R(EN1186-9:02)	2.714			



The same graph, but with the midsection enlarged:



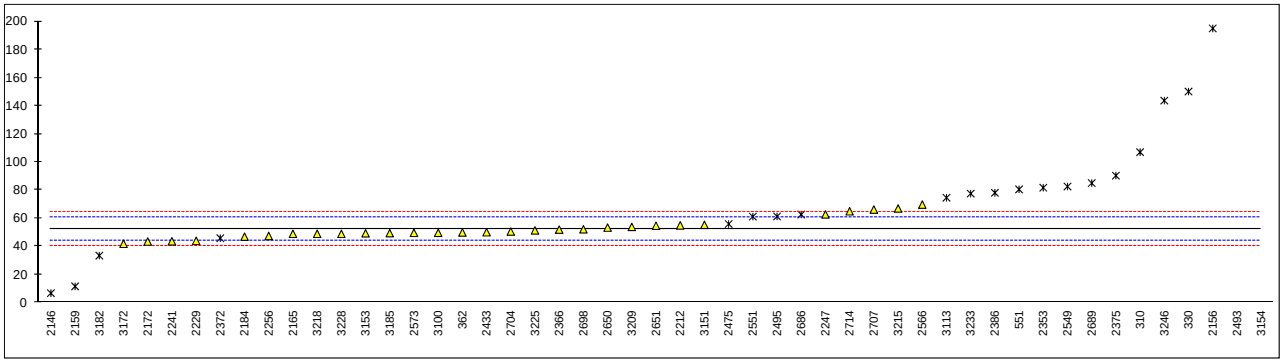


Determination of Overall Migration (per kg food stimulant) on sample #15180; results in mg/kg

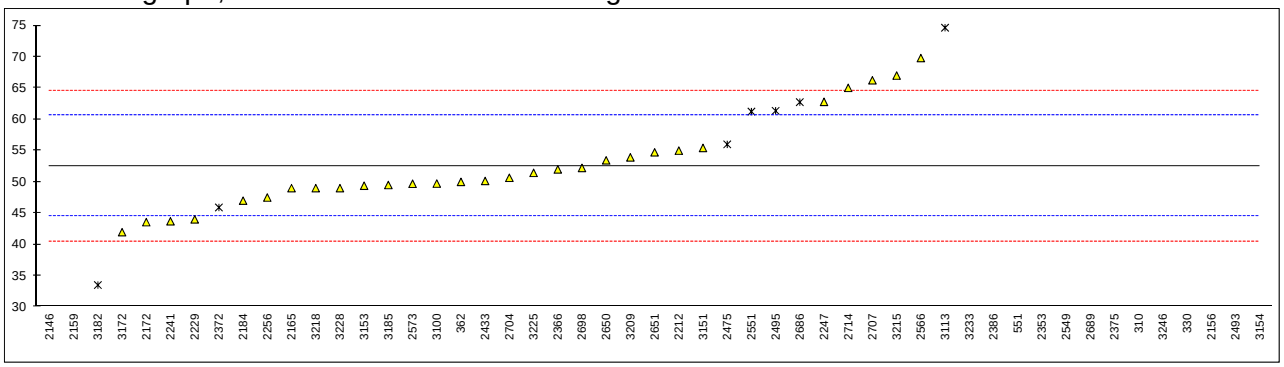
lab	method	value	mark	z(targ)	calc.	volume	remarks
310	EN1186-9	107.04	ex	13.53	factor 6	low	excluded, see §4.1
330	EN1189-9	150.13	ex	24.21	EN1186-9	low	excluded, see §4.1, reported as 150.13 mg/L
362	EN1186-9	50.0		-0.62	EN1186-9		
452		-----		-----			
551	EN1186-9	80.6	C,R(0.05)	6.97	EN1186-9		first reported: 119.7 mg/kg
2104		-----		-----			
2115		-----		-----			
2129		-----		-----			
2146	EN1186-9	6.83	ex, E	-11.33	EN1186-9	high	excluded, see §4.1, iis calculated 68.3 mg/kg
2156	in house	195	ex	35.34	EN1186-9	low	excluded, see §4.1
2159	EN1186-9	11.67	C,R(0.01)	-10.13	EN1186-9		first reported: 35 mg/kg
2165	EN1186-9	49.00		-0.87	EN1186-9		
2172	EN1186-9	43.56		-2.22	EN1186-9		
2184	EN1186-9	47.0		-1.37	EN1186-9		
2186		-----		-----			
2212	CFR175.300	55.00		0.62	EN1186-9		
2216		-----		-----			
2217		-----		-----			
2229	EN1186-9	44		-2.11	EN1186-9		
2241	EN1186-9	43.7		-2.19	EN1186-9		
2247	EN1186-9	62.8	C	2.55	EN1186-9		first rep. 0.0628 mg/kg, probably a unit error?
2256	EN1186-9	47.50		-1.24	EN1186-9		
2353	EN1186-9	81.75	ex	7.25	EN1186-9	high	excluded, see §4.1
2363		-----		-----			
2366	EN1186-9	52.0		-0.13	EN1186-9		
2372	EN1186-9	45.908	ex	-1.64	factor 6		excluded, see §4.1
2375	EN1186-9	90.36	ex	9.39	factor 6		excluded, see §4.1
2386	EN1186-9	78.17	ex	6.36	EN1186-9	low	excluded, see §4.1
2403		-----		-----			
2433	EN1186-9	50.15		-0.59	EN1186-9		reported as 50.15 mg/L
2475	EN1186-9	56.00	ex	0.86	EN1186-9	high	excluded, see §4.1
2493	EN1186-9	537.2	ex, E	120.23		low	excl. see §4.1,results could not be calc. by iis
2495	EN1186-9	61.364	ex	2.20	factor 6		excluded, see §4.1
2500		-----		-----			
2549	EN1186-9	82.62	ex	7.47	factor 6		excluded, see §4.1
2551	EN1186-9	61.24	ex	2.16	factor 6		excluded, see §4.1
2566	EN1186-9	69.8		4.29	EN1186-9		
2573	EN1186-9	49.70		-0.70	EN1186-9		
2650	EN1186-9	53.445	E	0.23	EN1186-9		iis calculated: 53.484 mg/kg
2651	EN1186-9	54.73	E	0.55	EN1186-9		iis calculated: 54.31 mg/kg
2686	EN1186-9	62.75	ex	2.54	factor 6		excluded, see §4.1
2689	EN1186-9	85.067	ex,C	8.07	factor 6	low	excluded, see §4.1, rep. also 84.5 mg/L *)
2698	EN1186-9	52.23		-0.07	EN1189-9		
2704	EN1186-9	50.65		-0.46	EN1186-9		
2707	EN1186-9	66.25		3.41	EN1186-9		
2714	EN1186-9	65.03		3.10	EN1186-9		
3100	EN1186-9	49.73		-0.69	EN1186-9		
3113	EN1186-9	74.65	ex	5.49	EN1189-9	high	excluded, see §4.1
3146		-----		-----			
3151	EN1186-9	55.43		0.72	EN1189-9		reported as 55.43 mg/L
3153	EN1186-9	49.385		-0.78	EN1186-9		
3154	EN1186-9	1887.6	R(0.01)	455.19	EN1186-9		
3172	EN1186-9	41.96		-2.62	EN1189-9		
3182	EN1186-9	33.50	ex	-4.72	factor 6		excluded, see §4.1, rep. also 24.73 mg/L **)
3185	EN1186-9	49.5		-0.75	EN1186-9		
3209	EN1186-9	53.92		0.35	EN1189-9		
3215	EN1186-9	67.0	E	3.59	EN1189-9		iis calculated: 70.3 mg/kg, also rep. 70.3 mg/L
3218	EN1186-9	49.0		-0.87	EN1189-9		
3220		-----		-----			
3225	EN1186-9	51.44		-0.27	EN1189-9		
3228	EN1186-9	49.0		-0.87	EN1189-9		
3233	EN1186-9	77.59	ex	6.22	factor 6		excluded, see §4.1
3246	EN1186-9	143.667	ex	22.61	EN1189-9	low	excluded, see §4.1

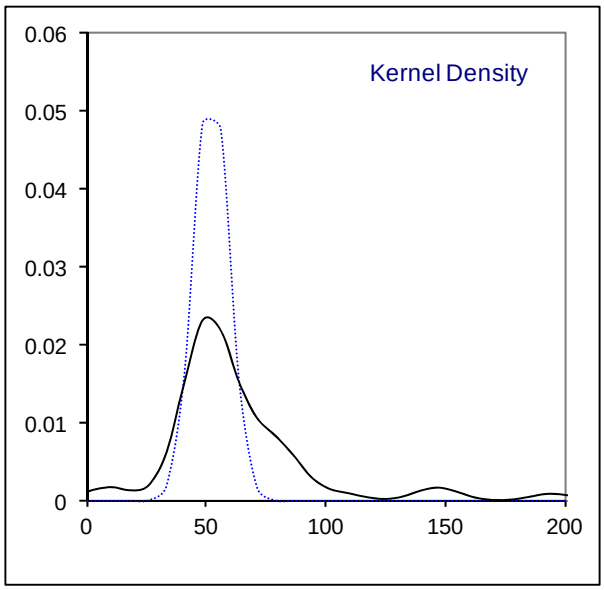
normality	suspect
n	29
outliers	3 (+19 ex)
mean (n)	52.514
st.dev. (n)	7.2407
R(calc.)	20.274
R(EN1186-9:02)	11.288

*) result calculated according to EN1186-9
**) result could not be calculated by iis



The same graph, but with the midsection enlarged:





APPENDIX 2:

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

lab	reported residue (mg)	residue after evap. of all simulant (mg)	volume simulant (ml)	volume simulant evap. (ml)	surface area (dm^2)	volume to area ratio (ml/dm^2)	remarks
310	9.63	57.78	**600**	100	3.24	185.19	
330	37.53	37.53	**250**	0	2.02	123.76	
362	50.0	50.0	1000	1000	4.65	215.05	
452	-----	-----	-----	-----	-----	-----	
551	104.76	104.76	1300	1300	5.25	247.62	
2104	61.25	61.25	**500**	100	2.77	180.51	
2115	-----	-----	1300	50	5.24	248.09	
2129	41.58	41.58	**700**	50	4	175.00	
2146	99.0	99.0	**1450**	200	5.41	268.02	
2156	97.5	97.5	**500**	200	2.92	171.23	
2159	14	14	1200	300	4.95	242.42	
2165	63.7	63.7	1300	200	5.04	257.94	
2172	57.46	57.46	1320	1320	5.02	262.95	
2184	61.1	61.1	1300	200	4.95	262.63	
2186	-----	-----	-----	-----	-----	-----	
2212	77.77	77.77	1413	200	4.94	286.03	
2216	75.87	75.87	1000	1000	5.01	199.60	
2217	4.0	50	1250	100	**4.44**	281.53	
2229	8.8	60.28	1370	200	4.82	284.23	
2241	8.8	59.4	1350	200	5.06	266.80	
2247	81.7	81.7	1300	1300	5.2	250.00	
2256	59.375	59.375	1250	200	5.1133	244.46	
2353	117.72	117.72	**1440**	200	5.0941	282.68	
2363	-----	-----	1400	200	4.95	282.83	
2366	62	62	1200	200	4.86	246.91	
2372	64.567	64.567	1300	300	**8.4387**	154.05	
2375	10	75.4	1300	200	5	260.00	
2386	15.63	50.7975	**650**	200	3	216.67	
2403	70.0	70.0	1300	10	4.98	261.04	
2433	10.03	10.03	1350	200	-----	-----	
2475	11.2	81.2	**1450**	200	**3.95**	367.09	
2493	64.3	64.3	**100**	120	1.0	100.00	
2495	51.423	51.423	1180	1180	5.028	234.69	
2500	62.2	62.2	1400	200	4.82	290.46	
2549	67.6	67.6	1400	1400	4.91	285.13	
2551	55.12	55.12	1000	1000	5.4	185.19	
2566	90.8	90.8	1300	1300	5.28	246.21	
2573	62.13	62.13	1250	1250	5	250.00	
2650	64.617	64.617	1200	200	6.09	197.04	
2651	70.6	70.6	1300	10	5.01	259.48	
2686	57	57	1260	1260	5.45	231.19	
2689	42.25	42.25	**500**	500	2.98	167.79	
2698	67.9	67.9	1300	1300	4.8	270.83	
2704	10.13	50.65	1000	200	5.3	188.68	
2707	13.25	-----	1000	5	-----	-----	
2714	65.03	65.03	1000	1000	**4.37**	228.83	
3100	9.96	62.25	1250	200	4.69	266.52	
3113	------	------	**1440**	200	6.17	233.39	
3146	1885.9	1885.9	1390	0	5.49	253.19	
3151	55.43	55.43	1000	200	5.523	181.06	
3153	64.200	64.200	1300	1300	4.88	266.39	
3154	377.52	377.52	1280	1280	5.18	247.10	
3172	57.9	57.9	1380	1380	5.2	265.38	
3182	8.83	24.724	1400	500	**4.43**	316.03	
3185	9.9	64.35	1300	200	4.9	265.31	
3209	64.7	64.7	1200	1200	5.18	231.66	
3215	70.3	70.3	1000	200	4.57	218.82	
3218	9.80	66.15	1350	200	5.1	264.71	
3220	------	------	-----	-----	-----	-----	
3225	64.3	64.3	1250	1250	5.53	226.04	
3228	63.7	63.7	1300	200	5.09	255.40	
3233	4.29	60.06	1400	100	4.64	301.72	
3246	57.47	57.47	**400**	400	2.57	155.64	

Details reported by the participating laboratories on preparation and evaporation

lab	Was sample cleaned during preparation?	How was the evaporation done?
310	No	Evaporation of simulant in one step
330	No	Other
362	No	Evaporation of simulant in small parts after each other
452	---	---
551	Yes, with a lint-free cloth	Evaporation of simulant in small parts after each other
2104	No	Evaporation of simulant in one step
2115	No	Evaporation of simulant in one step
2129	**Yes, with dist. water**	Evaporation of simulant in one step
2146	No	Evaporation of simulant in small parts after each other
2156	Yes, during preparation with lint cloth.	Evaporation of simulant in small parts after each other
2159	No	Other
2165	No	Evaporation of simulant in small parts after each other
2172	No	Evaporation of simulant in one step
2184	No	Evaporation of simulant in one step
2186	---	---
2212	Yes, with Kimwipes tissue	Evaporation of simulant in one step
2216	No	Evaporation of simulant in one step
2217	**Yes, with distilled water**	Evaporation of simulant in one step
2229	No	Evaporation of simulant in small parts after each other
2241	No	Evaporation of simulant in small parts after each other
2247	**Yes, with milli-Q water**	Evaporation of simulant in one step
2256	No	Evaporation of simulant in one step
2353	Yes, with soft brush to remove surface contaminations.	Evaporation of simulant in small parts after each other
2363	No	Evaporation of simulant in one step
2366	**Yes, with D.I. water**	Evaporation of simulant in small parts after each other
2372	**Yes, with D.I. Water**	Evaporation of simulant in one step
2375	No	Evaporation of simulant in small parts after each other
2386	No	Evaporation of simulant in one step
2403	Yes, with a lint-free cloth	Evaporation of simulant in small parts after each other
2433	No	Evaporation of simulant in one step
2475	No	Evaporation of simulant in one step
2493	No	Evaporation of simulant in one step
2495	Yes	Evaporation of simulant in one step
2500	No	Evaporation of simulant in small parts after each other
2549	Yes, by wiping the sample with a lint free cloth	Evaporation of simulant in small parts after each other
2551	No	Evaporation of simulant in small parts after each other
2566	Yes, with lint free cloth	Evaporation of simulant in small parts after each other
2573	Yes	Evaporation of simulant in small parts after each other
2650	No	Evaporation of simulant in small parts after each other
2651	**Yes, with distilled water during preparation**	Evaporation of simulant in one step
2686	No	Evaporation of simulant in small parts after each other
2689	No	Evaporation of simulant in one step
2698	**Yes, wash with Deionized Water**	Evaporation of simulant in small parts after each other
2704	No	Evaporation of simulant in small parts after each other
2707	**Yes, with with distilled water during preparation**	Evaporation of simulant in one step
2714	**Yes, with distilled water and dried before use.**	Evaporation of simulant in small parts after each other
3100	No	Evaporation of simulant in small parts after each other
3113	Yes, with lint free cloth	Evaporation of simulant in small parts after each other
3146	No	Evaporation of simulant in small parts after each other
3151	**Yes, with tap water and detergent followed by drying**	Evaporation of simulant in one step
3153	Yes, by wiping it with lint-free cloth	Evaporation of simulant in small parts after each other
3154	No	Evaporation of simulant in one step
3172	Yes, lightly rubbing with a cloth	Evaporation of simulant in small parts after each other
3182	No	Evaporation of simulant in one step
3185	**Yes, with warm water during preparation.**	Evaporation of simulant in small parts after each other
3209	**Yes, with distilled water**	Evaporation of simulant in one step
3215	No	Evaporation of simulant in one step
3218	No	Evaporation of simulant in small parts after each other
3220	---	---
3225	No	Other
3228	No	Evaporation of simulant in one step
3233	No	Evaporation of simulant in one step
3246	No	Evaporation of simulant in one step

APPENDIX 3:

Results reported by participating laboratories on Overall Migration on sample #15180 in mg/kg and mg/L

lab	method of calculation	reported result migration (mg/kg)	method of calculation	reported result migration (mg/L)	remarks
310	factor 6	107.04	-----	107.04	mg/kg = mg/L
330	-----	-----	EN1186-9	150.13	
362	EN1186-9	50.0	-----	50.5	difference = density
452	-----	-----	-----	-----	
551	EN1186-9	80.6	-----	80.6	mg/kg = mg/L
2104	-----	-----	-----	-----	
2115	-----	-----	-----	-----	
2129	-----	-----	-----	-----	
2146	-----	6.83	-----	-----	calculation error: iis calc. EN1186-9: 68.3 mg/kg
2156	EN1186-9	195	-----	195	mg/kg = mg/L
2159	EN1186-9	11.67	-----	2.82	reported value for mg/L is the same as result in mg/dm^2
2165	EN1186-9	49.00	-----	49.00	mg/kg = mg/L
2172	EN1186-9	43.56	-----	43.56	mg/kg = mg/L
2184	EN1186-9	47.0	-----	47.0	mg/kg = mg/L
2186		-----	-----	-----	
2212	EN1186-9	55.00	-----	55.00	mg/kg = mg/L
2216	-----	-----	-----	-----	
2217	-----	-----	-----	-----	
2229	EN1186-9	44	-----	44	mg/kg = mg/L
2241	EN1186-9	43.7	-----	43.7	mg/kg = mg/L
2247	EN1186-9	62.8	-----	62.8	mg/kg = mg/L
2256	EN1186-9	47.50	-----	47.50	mg/kg = mg/L
2353	EN1186-9	81.75	-----	-----	
2363	-----	-----	-----	-----	
2366	EN1186-9	52.0	-----	52.0	mg/kg = mg/L
2372	factor 6	45.908	-----	45.908	mg/kg = mg/L
2375	factor 6	90.36	-----	90.36	mg/kg = mg/L
2386	EN1186-9	78.17	-----	78.17	mg/kg = mg/L
2403	-----	-----	-----	-----	
2433	-----	-----	EN1186-9	50.15	
2475	EN1186-9	56.00	-----	56.00	mg/kg = mg/L
2493	-----	537.2	-----	535.8	results could not be calculated by iis
2495	factor 6	61.364	EN1186-9	43.579	calculated mg/kg with factor 6 and mg/L with EN1186-9
2500	-----	-----	-----	-----	
2549	factor 6	82.62	-----	82.62	mg/kg = mg/L
2551	factor 6	61.24	-----	61.24	mg/kg = mg/L
2566	EN1186-9	69.8	-----	69.8	mg/kg = mg/L
2573	EN1186-9	49.70	-----	49.70	mg/kg = mg/L
2650	-----	53.445	EN1186-9	53.848	difference = density
2651	-----	54.73	-----	-----	iis calc. EN1186-9 as 54.31 mg/kg or mg/L, reported result diff. = density
2686	factor 6	62.75	-----	62.75	mg/kg = mg/L
2689	factor 6	85.067	EN1186-9	84.5	calculated mg/kg with factor 6 and mg/L with EN1186-9
2698	EN1186-9	52.23	------	52.23	mg/kg = mg/L
2704	EN1186-9	50.65	-----	50.65	mg/kg = mg/L
2707	EN1186-9	66.25	-----	-----	
2714	EN1186-9	65.03	-----	65.03	mg/kg = mg/L
3100	EN1186-9	49.73	-----	49.73	mg/kg = mg/L
3113	EN1186-9	74.65	-----	-----	
3146	-----	-----	-----	-----	
3151	-----	-----	EN1186-9	55.43	
3153	EN1186-9	49.385	-----	49.385	mg/kg = mg/L
3154	EN1186-9	1887.6	-----	-----	
3172	EN1186-9	41.96	-----	41.96	mg/kg = mg/L
3182	factor 6	33.50	-----	24.73	result in mg/L could not be calculated by iis
3185	EN1186-9	49.5	-----	49.5	mg/kg = mg/L
3209	EN1186-9	53.92	-----	53.92	mg/kg = mg/L
3215	-----	67.0	EN1186-9	70.3	difference = density
3218	EN1186-9	49.0	-----	49.0	mg/kg = mg/L
3220	-----	-----	-----	-----	
3225	EN1186-9	51.44	-----	51.44	mg/kg = mg/L
3228	EN1186-9	49.0	-----	49.0	mg/kg = mg/L
3233	factor 6	77.59	-----	77.59	mg/kg = mg/L
3246	EN1186-9	143.667	-----	143.667	mg/kg = mg/L

APPENDIX 4

Number of participating laboratories per country

2 labs in BRAZIL

1 lab in BULGARIA

1 lab in DENMARK

1 lab in FINLAND

3 labs in FRANCE

6 labs in GERMANY

6 labs in HONG KONG

2 labs in HUNGARY

4 labs in INDIA

4 labs in ITALY

1 lab in MALAYSIA

20 labs in P.R. of CHINA

1 lab in PHILIPPINES

2 labs in SAUDI ARABIA

1 lab in SPAIN

1 lab in TAIWAN R.O.C.

1 lab in THAILAND

1 lab in THE NETHERLANDS

2 labs in TURKEY

1 lab in U.S.A.

1 lab in UNITED KINGDOM

1 lab in VIETNAM

APPENDIX 5

Abbreviations:

C = final result after checking of first reported suspect 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 outlier test
R(0.05) = straggler in Rosner outlier test
n.a. = not applicable
E = possible calculation error
W = result was withdrawn

Literature:

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2 EN 1186-1:02 - Guide to the selection of conditions and test methods for overall migration
3 EN 1186-8:02 - Test methods for overall migration into olive oil by article filling
4 EN 1186-9:02 - Test methods for overall migration into aqueous simulants by article filling
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9 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
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12 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
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17 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), pp. 165-172, (1983)
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
19 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