Results of Proficiency Test
Overall migration (fcm)
September 2013

CONTENTS

Appendices:

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. This PT was repeated in 2013.

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. 1935/2004 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. 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 October 2013, 56 laboratories from 19 different countries participated (See appendix 3).

In this report, the results of the 2013 proficiency test are presented and discussed.

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 (2 identical PTFE plates), that gave a positive test result, labelled #13183, and to prescribe a number of test conditions (type of simulant, exposure time and temperature) to be used. Participants were also requested to report the test conditions that the laboratory would have used in case these were not prescribed by iis.

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 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 January 2010 (iis-protocol, version 3.2). This protocol can be downloaded from the iis website http://www.iisnl.com.

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 PTFE plates (10 x 10 x 0.2 cm each) for repetitive 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 (2hrs @70°C and water as simulant) on 7 stratified randomly selected samples.

	Overall Migration 3rd step in mg/dm^2 #13183
Sample 1	18
Sample 2	19
Sample 3	17
Sample 4	19
Sample 5	18
Sample 6	19
Sample 7	20
Sample 8	19

Table 1: results of the homogeneity test on the subsamples #13183

From the above results of the homogeneity test, the between sample standard deviation r was calculated and compared with 0.3 times the relative proficiency target standard deviations RSD_R in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Overall Migration 3rd step in mg/dm^2 #13183
r(observed)	2.6
reference method	EN1186-3:2002
0.3xR(reference method)	2.1
r(reference method)	3.5

Table 2: evaluation of the repeatability of the migration results on subsamples #13183

The calculated repeatability for Overall Migration on the eight samples #13183 is not in good agreement with the estimated target, calculated using EN1186-3 precision data, but as it is smaller than the repeatability of EN1186-3, homogeneity of the samples #13183 was assumed.

To each of the participating laboratories one set of samples #13183, (2 identical PTFE plates) was sent on September 25, 2013.

2.5 ANALYSIS

The participants were requested to determine Overall Migration on the sample using the prescribed test conditions (total immersion, 2hrs @70°C and water as simulant). It was requested to report the analytical results using the indicated units on the report form and to use a minimum number of digits and not to round the results more. It was also 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, was sent together with each set of samples. Also, a letter of instructions was added to the package.
The laboratories were also requested to report the test conditions that the laboratory would have used in case these were not prescribed by iis.

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 January 2010 (iis-protocol, version 3.2).

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test. After removal of outliers this check was repeated.

In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs 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. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test. 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 4; nr.14 and 15).

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:

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

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 in order to evaluate the fit-for-useness of the reported test result. See also: appendix 3, ref. 16.

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, no problems were encountered with the dispatch of the samples. Four participants reported test results after the final reporting date and two other participants did not report any test results at all. Finally, 54 of the 56 participants submitted analysis results. These 54 laboratories reported 304 numerical test results. Observed were 15 statistically outlying results, which is 4.9%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

A not-normal distribution was found for the four of the six reported data sets. Therefore this statistical evaluation should be used with due care.

For the determination of Overall Migration (identical to Global migration or Total Migration), the EN1186 method series (parts 1 – 15) is considered to be the official EC test method.

In this PT, given the use as mentioned in the letter of instructions, water was used as simulant, cfr. EN1186 parts 1 and 3. The target reproducibility was estimated from the reproducibility as mentioned in EN1186 part 3, annex A.

4.1   PERFORMANCE EVALUATION OF THE GROUP OF LABORATORIES

The calculated reproducibilities and the target reproducibilities derived from the literature standard method, here EN1186-3:02, are compared in the next table.

	unit	n	Average	2.8 * sd	R (target)
Overall migration 1st step	mg/dm^2	53	26.5	27.1	10.0
Overall migration 1st step	mg/kg	44	186.9	233.9	70.4
Overall migration 2nd step	mg/dm^2	54	18.7	13.4	7.1
Overall migration 2nd step	mg/kg	44	132.6	161.9	50.0
Overall migration 3rd step	mg/dm^2	52	12.2	10.3	4.6
Overall migration 3rd step	mg/kg	42	81.0	76.3	30.5

Table 3: performance overview for samples #13183

4.2   EVALUATION OF THE REPORTED TEST RESULTS

In this section the results are discussed.

residue in mg: These test results were not evaluated as they are in principle dependent of the size of the contact surface used. Use of a large contact surface will of course give a higher residue than use of a small contact surface.

migration in mg/dm^2: This determination was problematic. In total only two statistical outliers were detected. However, the calculated reproducibilities, after rejection of the statistical outliers, are both not in agreement with the target reproducibility requirement of EN1186-3:02

migration in mg/kg: The reporting in mg/kg was very problematic. In total 13 statistical outliers were detected. The calculated reproducibilities, after rejection of the statistical outliers, are not at all in agreement with the target reproducibility requirements of EN1186-3:02. See also paragraph 5.

4.3 EVALUATION OF THE TEST METHODS USED

The majority of the participants reported to have used a part of the EN1186 test method. Besides the general part 1 of this test method, also parts 3 and 6 were mentioned.

The reported details of the methods that were used by the participants are listed in appendix 2.

5   CONCLUSIONS

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 was given together with the instructions to all participants. These preset conditions were:

Sample ID	**#13183**
Simulant	Distilled water
Exposure time	2.0 hrs
Exposure temperature	70.0 °C
Migration method	Total immersion

Table 4: preset test conditions used in this PT

Not only a migration result was to be reported, but the participants were requested to report also the intermediate amount of residue after removal of the simulant. Using these intermediate test results it was possible to check the calculations done by the laboratories.

This revealed that initially indeed some calculation errors were present. Several laboratories corrected these calculation errors; see the original and the revised test results in appendix 1.

The spread in the Overall Migration results in mg/kg show a larger spread than the spread in the Overall Migration results in mg/dm^2. Upon investigation for the reason of the increase in spread, it was found that a number of laboratories did not use the conventional surface to volume ratio of 6 dm^2/kg *cfr*. EN1186-1, paragraph 12.1.2. The factor between the residue and the migration result was not 6 as expected, but varied from 2 - 70, see appendix 2. Apparently a number of the participants reported test results in mg/kg PTFE or mg/kg water instead of the requested Migration in mg/kg. The calculation to use is as follows:

Overall Migration results in mg/kg = (Overall Migration results in mg/dm^2) * 6 dm^2/kg

It was remarkable to see that the amount of simulant used per dm^2 contact surface varied from 48-500 ml/dm^2 (see appendix 2). This is remarkable because in EN1186-3 is mentioned that a specimen of approx 1 dm^2 is to be immersed into 100 ml of simulant (= 100 ml/dm^2). Therefore it was investigated whether this factor may be of significant influence on the spread of Migration test results. In appendix 1, the evaluations of the test results based on a migration with 100 ml/dm^2 and based on a migration with 50 ml/dm^2 are presented. From these separate evaluations it became clear that the influence of the different treatments of the contact surface determination is not significant in this PT.

From the reported contact surfaces it was clear that a number of laboratories (21 labs = 40%) did take the side surfaces of the plates into account, while other laboratories (30 labs = 60%) did not do so (see appendix 2). Therefore it was investigated whether this factor may be of significant

influence on the spread of Migration test results. The influence appeared to not significant. The results are presented in the next table:

	with contact surfaces as reported	recalculated with contact surfaces without sides
Overall migration 1st step in mg/dm^2	26.5	27.2
Overall migration 2nd step in mg/dm^2	18.7	18.9
Overall migration 3rd step in mg/dm^2	12.2	12.2

Table 5: influence of plate thickness on contact surface used

Other details may also be of influence on the test result. During the preparation of the final report some feedback was received from a number of laboratories. Thus it became clear that a number of laboratories immersed a whole PTFE plate into a Petri dish to use only 100 ml of simulant. Other laboratories did cut the plates in 2 parts, or in 4 parts or even in 8 parts, see appendix 3. Many laboratories reported to have evaporated all simulant and only few reported to have evaporated part of the simulant and calculated the total amount of residue. It is unknown whether evaporation of the simulant was performed in the migration container or in another container. In case of low soluble components this may give different results.

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.
See appendix 2 for the test conditions that the laboratories would have selected in case these were not prescribed as in this PT.
From the responses it became clear that the test conditions as set in this PT were quite realistic. However, the majority of the laboratories would have taken a different simulant:
- 77% of the responding laboratories would have selected total immersion (remarkably 14% = 5 laboratories responded to select article filling).
- Almost all responding participants (97%) would have used an exposure time of 2 hours.
- 69% of the responding laboratories would have selected an exposure temperature of 70°C and 26% would have selected an exposure temperature of 40°C.
- Only 11% of the responding participants (53%) would have selected water as simulant, while 63% would have preferred 3% Acetic Acid as simulant.

A number of methods to determine the contact surface were mentioned:
- measure by caliper and calculate using mathematical equations
- graphic millimeter paper method cfr JRC guidelines EUR24815 EN 2011
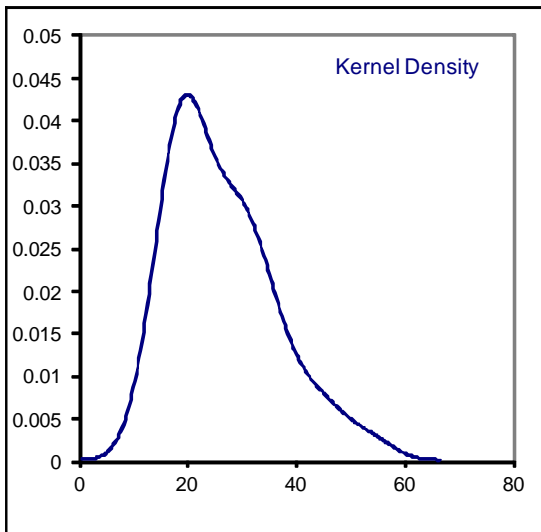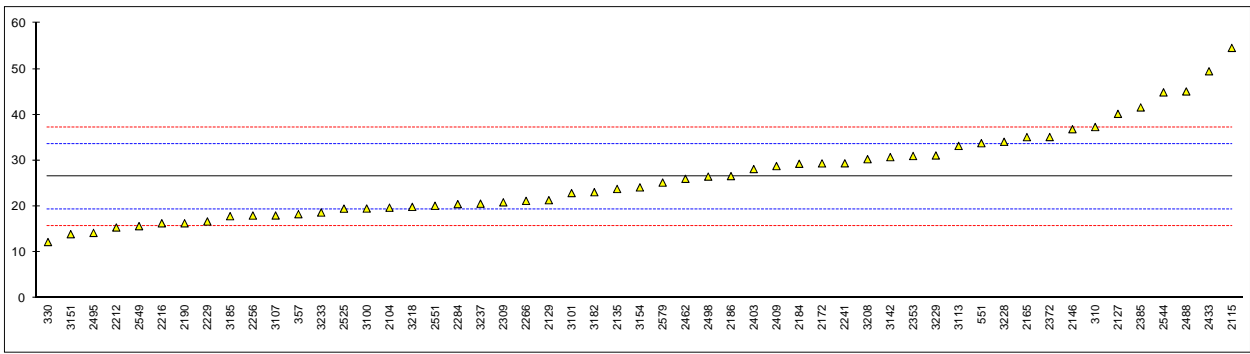- by cutting & measurement or weighing

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 on sample #13183; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
310	EN1186-3	37.33		3.05	
330	EN1186-3	12.2		-4.01	
357	EN1186-3	18.29		-2.29	
551	EN1186-3	33.80		2.06	
2104	EN1186	19.71		-1.90	
2115		54.61		7.90	
2127		40.2		3.86	
2129	EN1186	21.34		-1.44	
2135	EN1186-3	23.8		-0.75	
2146		36.85		2.92	
2165	EN1186-3	35.12		2.43	
2172	EN1186	29.38		0.82	
2184	EN1186 & EC 10/2011	29.3		0.80	
2186		26.6		0.04	
2190		16.3		-2.85	
2212	CFR 175.300, EN1186-1&3	15.38		-3.11	
2216	21CFR175.300	16.30		-2.85	
2229	EN1186	16.70		-2.74	
2241	EN1186-1&3	29.40	C	0.82	first reported 58.81
2256	EN1186-1&3	17.99		-2.38	
2266	EN1186-3	21.2	C	-1.48	first reported 42.4
2284	EN1186	20.49		-1.68	
2309	EN1186-3	20.88		-1.57	
2353	EN1186-3	30.99		1.27	
2372	EN1186 & EC 10/2011	35.14		2.44	
2385	EN1186-3	41.61		4.25	
2386		-----		-----	
2403	EN1186-3	28.14		0.47	
2409		28.80		0.66	
2433	EN1186-3	49.50	C	6.47	first reported 99.00
2462	EN1186-3	26.01		-0.13	
2488		45.10		5.23	
2495	EN1186	14.2		-3.44	
2498		26.50		0.01	
2525		19.50		-1.96	
2544	EN1186-3	44.90		5.18	
2549	EN1186-3	15.67		-3.03	
2551		20.13		-1.78	
2579		25.19		-0.36	
3100		19.52		-1.95	
3101	EN1186-3	22.9		-1.00	
3107	EN1186-3	18.0	C	-2.38	first reported 35.9
3113	EN1186	33.20		1.89	
3142	EN1186-3	30.75		1.20	
3146	EN1186	-----		-----	
3151	EN1186-1	13.92		-3.52	
3154	EN1186-6	24.14		-0.65	
3172		-----		-----	
3182	EN1186-3	23.09		-0.95	
3185	EN1186 & EC 10/2011	17.85		-2.42	
3208	EU-10/2011	30.30		1.08	
3218	EN1186	19.88		-1.85	
3228	EN1186-3	34.10		2.14	
3229	EN1186-3	31.11		1.30	
3233	EN1186-3	18.65		-2.19	
3237	EN1186-3	20.58		-1.65	

			only 100 ml/dm^2:	only 50 ml/dm^2:
normality	not OK		OK	not OK
n	53		19	8
outliers	0		0	0
mean (n)	26.463		27.460	23.715
st.dev. (n)	9.6954		9.475	6.8042
R(calc.)	27.147		26.530	19.052
R(EN1186-3:02)	9.972		10.347	8.936

Determination of 1st Overall Migration on sample #13183; results in mg/dm^2, continued

Determination of 1st Overall Migration on sample #13183; results in mg/kg

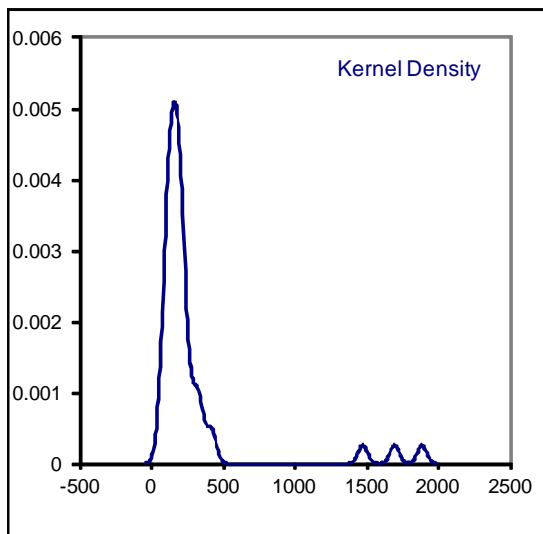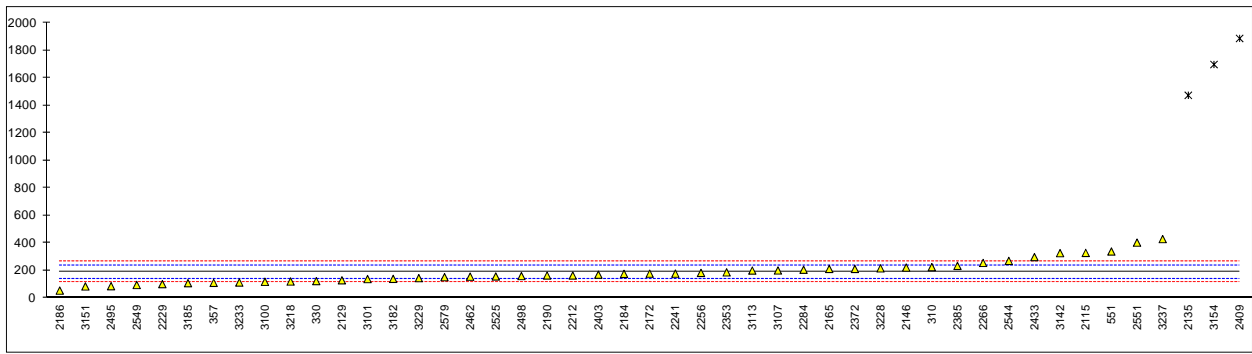| lab	method	value	mark	z(targ)	remarks
310	EN1186-3	224.00		1.47	
330	EN1186-3	122.3		-2.57	
357	EN1186-3	109.71		-3.07	
551	EN1186-3	338.00		6.01	
2104	EN1186	-----		-----	
2115		327.66		5.60	
2127		-----		-----	
2129	EN1186	128.03		-2.34	
2135	EN1186-3	1475	G(0.01)	51.21	
2146		221.1		1.36	
2165	EN1186-3	210.72		0.95	
2172	EN1186	176.28		-0.42	
2184	EN1186 & EC 10/2011	175		-0.47	
2186		53.3		-5.31	
2190		163		-0.95	
2212	CFR 175.300, EN1186-1&3	163.00		-0.95	
2216	21CFR175.300	-----		-----	
2229	EN1186	100.20		-3.45	
2241	EN1186-1&3	176.43	C	-0.42	first reported 352.86
2256	EN1186-1&3	182.00		-0.20	
2266	EN1186-3	254.4		2.68	
2284	EN1186	204.90		0.72	
2309	EN1186-3	-----		-----	
2353	EN1186-3	185.91		-0.04	
2372	EN1186 & EC 10/2011	210.84		0.95	
2385	EN1186-3	233.0		1.83	
2386		-----		-----	
2403	EN1186-3	168.84		-0.72	
2409		1889.25	G(0.01)	67.68	
2433	EN1186-3	297.00	C	4.38	first reported 594.00
2462	EN1186-3	154.57		-1.29	
2488		-----		-----	
2495	EN1186	85.2		-4.04	
2498		159.00		-1.11	
2525		156.00		-1.23	
2544	EN1186-3	269.40		3.28	
2549	EN1186-3	94.02		-3.69	
2551		402.50	C	8.57	first reported 201.25
2579		151.14		-1.42	
3100		117.12		-2.77	
3101	EN1186-3	137		-1.98	
3107	EN1186-3	199.7		0.51	
3113	EN1186	199.23	C	0.49	first reported 332.05
3142	EN1186-3	326.00		5.53	
3146	EN1186	-----		-----	
3151	EN1186-1	83.52		-4.11	
3154	EN1186-6	1699.46	G(0.01)	60.13	
3172		-----		-----	
3182	EN1186-3	138.53		-1.92	
3185	EN1186 & EC 10/2011	107.10		-3.17	
3208	EU-10/2011	-----	W	-----	
3218	EN1186	119.40		-2.68	
3228	EN1186-3	214.93		1.11	
3229	EN1186-3	143.78		-1.71	
3233	EN1186-3	112.00		-2.98	
3237	EN1186-3	428.10	C	9.59	first reported 1361.42
	normality	OK			
	n	44			
	outliers	3			
	mean (n)	186.906			
	st.dev. (n)	83.5274			
	R(calc.)	233.877			
	R(EN1186-3:02)	70.428			

Determination of 1st Overall Migration on sample #13183; results in mg/kg, continued

Determination of 2nd Overall Migration on sample #13183; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
310	EN1186-3	18.57		-0.06	
330	EN1186-3	6.4		-4.89	
357	EN1186-3	13.57		-2.05	
551	EN1186-3	27.20		3.36	
2104	EN1186	25.85		2.83	
2115		20.44		0.68	
2127		30.9		4.83	
2129	EN1186	14.44		-1.70	
2135	EN1186-3	16.8		-0.76	
2146		15.3		-1.36	
2165	EN1186-3	23.19		1.77	
2172	EN1186	21.94		1.28	
2184	EN1186 & EC 10/2011	20.6		0.75	
2186		18.0		-0.29	
2190		19.0		0.11	
2212	CFR 175.300, EN1186-1&3	14.72		-1.59	
2216	21CFR175.300	12.63		-2.42	
2229	EN1186	17.13		-0.63	
2241	EN1186-1&3	21.46	C	1.09	first reported 42.91
2256	EN1186-1&3	22.83		1.63	
2266	EN1186-3	22.05	C	1.32	first reported 44.1
2284	EN1186	16.56		-0.86	
2309	EN1186-3	15.90		-1.12	
2353	EN1186-3	19.92		0.48	
2372	EN1186 & EC 10/2011	26.04		2.90	
2385	EN1186-3	26.87		3.23	
2386		-----		-----	
2403	EN1186-3	16.33		-0.95	
2409		16.11		-1.04	
2433	EN1186-3	30.70	C	4.75	first reported 61.40
2462	EN1186-3	18.42		-0.12	
2488		14.35		-1.74	
2495	EN1186	14.7		-1.60	
2498		21.85		1.24	
2525		13.00		-2.27	
2544	EN1186-3	23.39		1.85	
2549	EN1186-3	13.51		-2.07	
2551		19.52		0.32	
2579		17.29		-0.57	
3100		14.75		-1.58	
3101	EN1186-3	17.4		-0.52	
3107	EN1186-3	19.6	C	0.35	first reported 39.2
3113	EN1186	21.58	C	1.13	first reported 33.54
3142	EN1186-3	17.26		-0.58	
3146	EN1186	24.33		2.23	
3151	EN1186-1	14.49		-1.68	
3154	EN1186-6	14.42		-1.71	
3172		-----		-----	
3182	EN1186-3	14.14		-1.82	
3185	EN1186 & EC 10/2011	16.13		-1.03	
3208	EU-10/2011	21.20		0.98	
3218	EN1186	16.52		-0.87	
3228	EN1186-3	23.03		1.71	
3229	EN1186-3	17.16		-0.62	
3233	EN1186-3	16.32		-0.95	
3237	EN1186-3	15.22		-1.39	

			only 100 ml/dm^2:	only 50 m/dm^2:
normality	not OK		OK	OK
n	54		20	8
outliers	0		0	0
mean (n)	18.723		18.870	18.576
st.dev. (n)	4.7690		4.7171	4.2705
R(calc.)	13.353		13.208	11.957
R(EN1186-3:02)	7.055		7.110	7.000

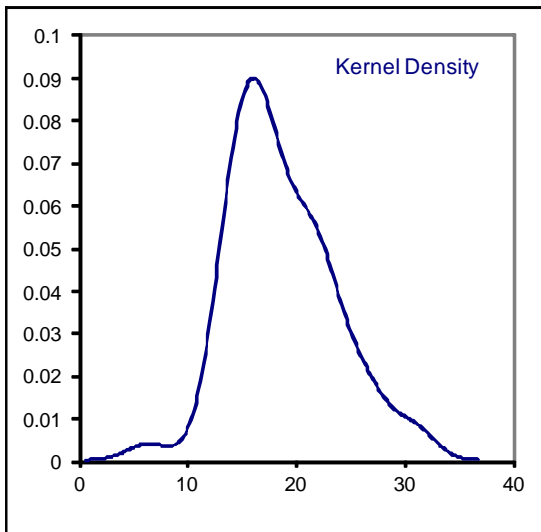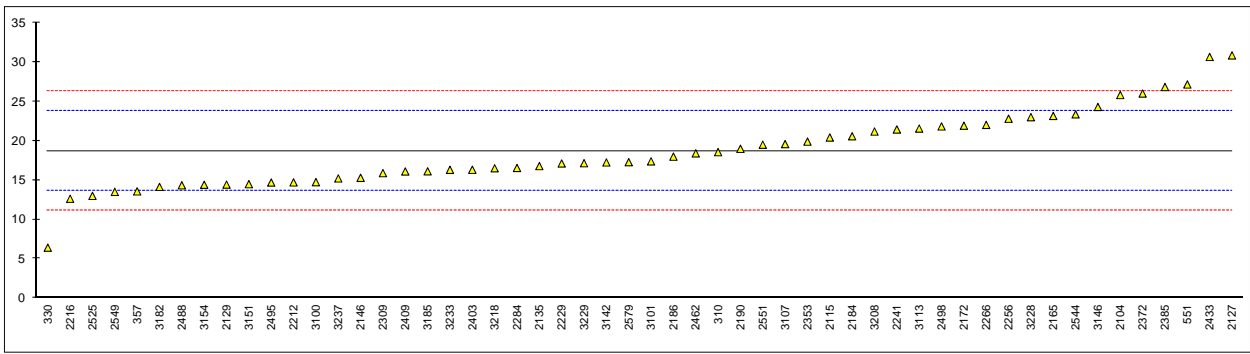Determination of 2nd Overall Total Migration on sample #13183; results in mg/dm^2, continued

Determination of 2nd Overall Migration on sample #13183; results in mg/kg

Determination of 2nd Overall Migration on sample #13183; results in mg/kg

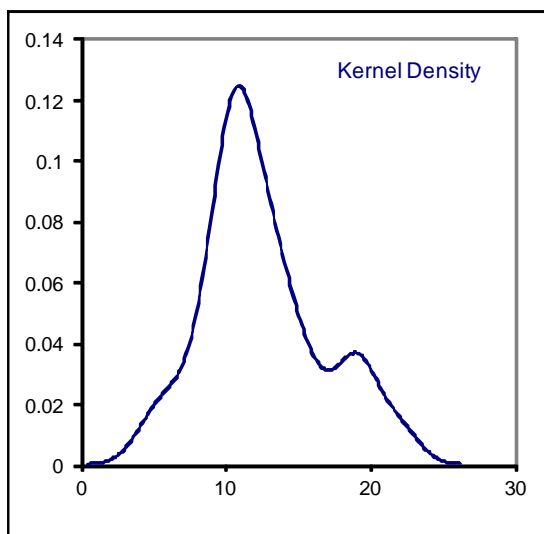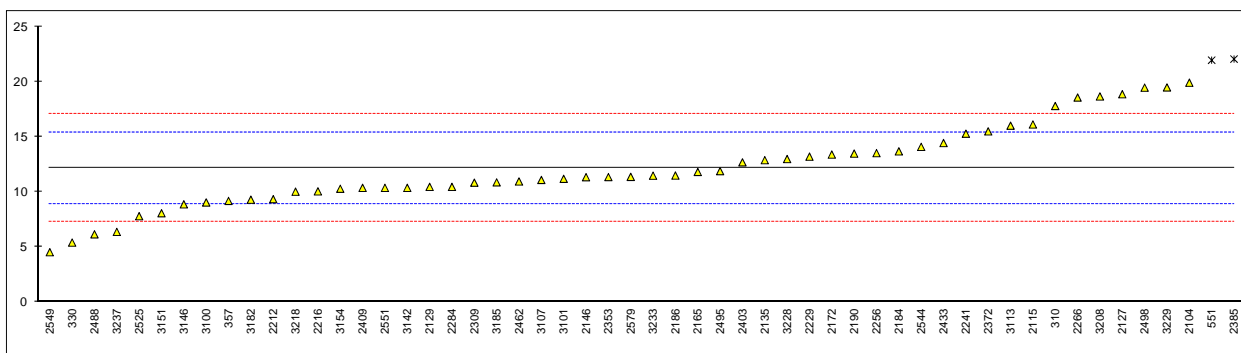| lab	method	value	mark	z(targ)	remarks
310	EN1186-3	111.40		-1.19	
330	EN1186-3	63.7		-3.86	
357	EN1186-3	81.43		-2.87	
551	EN1186-3	272.00		7.81	
2104	EN1186	-----		-----	
2115		122.64		-0.56	
2127		-----		-----	
2129	EN1186	86.64		-2.58	
2135	EN1186-3	1037	G(0.01)	50.68	
2146		91.8		-2.29	
2165	EN1186-3	139.14		0.37	
2172	EN1186	131.64		-0.05	
2184	EN1186 & EC 10/2011	123		-0.54	
2186		36.0		-5.41	
2190		190		3.22	
2212	CFR 175.300, EN1186-1&3	159.00		1.48	
2216	21CFR175.300	-----		-----	
2229	EN1186	102.78		-1.67	
2241	EN1186-1&3	128.73	C	-0.22	first reported 257.46
2256	EN1186-1&3	231.00		5.51	
2266	EN1186-3	264.6		7.40	
2284	EN1186	165.60		1.85	
2309	EN1186-3	-----		-----	
2353	EN1186-3	119.52		-0.73	
2372	EN1186 & EC 10/2011	156.24		1.32	
2385	EN1186-3	150.45		1.00	
2386		-----		-----	
2403	EN1186-3	97.98		-1.94	
2409		1056.59	G(0.01)	51.78	
2433	EN1186-3	184.20	C	2.89	first reported 368.40
2462	EN1186-3	109.46		-1.30	
2488		-----		-----	
2495	EN1186	88.4		-2.48	
2498		131.10		-0.08	
2525		103.60		-1.63	
2544	EN1186-3	140.34		0.43	
2549	EN1186-3	81.06		-2.89	
2551		390.5	C,G(0.01)	14.45	first reported 195.25
2579		103.74		-1.62	
3100		80.50		-2.92	
3101	EN1186-3	104		-1.60	
3107	EN1186-3	217.5		4.76	
3113	EN1186	129.50	C	-0.17	first reported 335.38
3142	EN1186-3	183.00		2.82	
3146	EN1186	145.98		0.75	
3151	EN1186-1	86.94		-2.56	
3154	EN1186-6	1011.18	G(0.01)	49.23	
3172		-----		-----	
3182	EN1186-3	84.84		-2.68	
3185	EN1186 & EC 10/2011	96.78		-2.01	
3208	EU-10/2011	-----	W	-----	
3218	EN1186	99.22		-1.87	
3228	EN1186-3	145.16		0.70	
3229	EN1186-3	79.33		-2.99	
3233	EN1186-3	98.00		-1.94	
3237	EN1186-3	316.50	C	10.31	first reported 1006.75
	normality	not OK			
	n	44			
	outliers	4			
	mean (n)	132.601			
	st.dev. (n)	57.8124			
	R(calc.)	161.875			
	R(EN1186-3:02)	49.966			

Determination of 2nd Overall Migration on sample #13183; results in mg/kg, continued
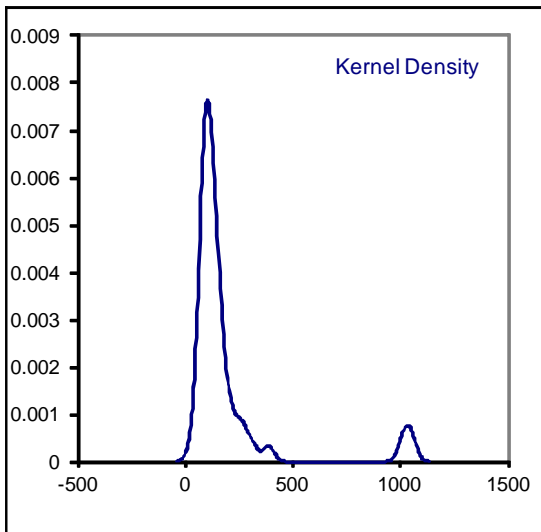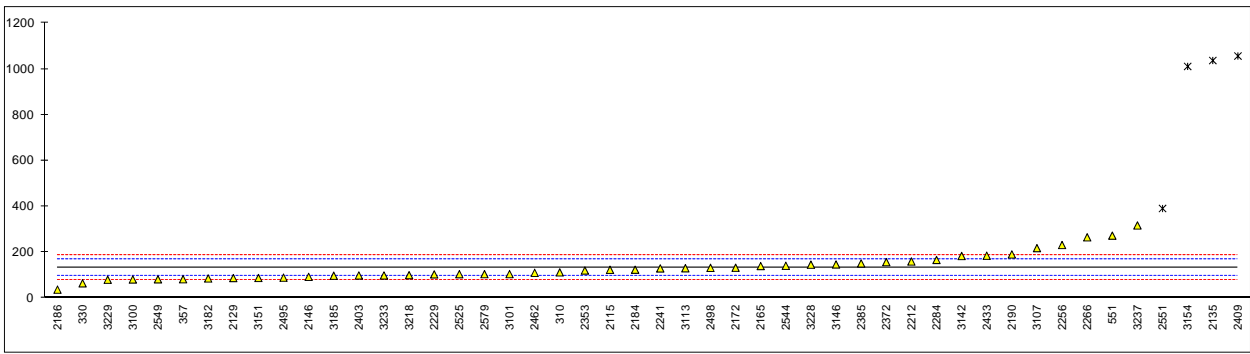
Determination of 3rd Overall Migration on sample #13183; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
310	EN1186-3	17.83		3.45	
330	EN1186-3	5.4		-4.13	
357	EN1186-3	9.19		-1.82	
551	EN1186-3	22.00	DG(0.05)	6.00	
2104	EN1186	19.95		4.75	
2115		16.15		2.43	
2127		18.9		4.11	
2129	EN1186	10.47		-1.04	
2135	EN1186-3	12.9		0.44	
2146		11.35		-0.50	
2165	EN1186-3	11.83		-0.21	
2172	EN1186	13.41		0.76	
2184	EN1186 & EC 10/2011	13.7		0.93	
2186		11.5		-0.41	
2190		13.5		0.81	
2212	CFR 175.300, EN1186-1&3	9.35		-1.72	
2216	21CFR175.300	10.06		-1.29	
2229	EN1186	13.22		0.64	
2241	EN1186-1&3	15.31	C	1.92	first reported 3062
2256	EN1186-1&3	13.54		0.84	
2266	EN1186-3	18.6	C	3.92	first reported 37.2
2284	EN1186	10.47		-1.04	
2309	EN1186-3	10.85		-0.81	
2353	EN1186-3	11.36		-0.50	
2372	EN1186 & EC 10/2011	15.52		2.04	
2385	EN1186-3	22.10	DG(0.05)	6.06	
2386		-----		-----	
2403	EN1186-3	12.70		0.32	
2409		10.38		-1.09	
2433	EN1186-3	14.46	C	1.40	first reported 28.92
2462	EN1186-3	10.96		-0.74	
2488		6.15		-3.68	
2495	EN1186	11.9		-0.17	
2498		19.50		4.47	
2525		7.80		-2.67	
2544	EN1186-3	14.11		1.18	
2549	EN1186-3	4.52		-4.67	
2551		10.38		-1.09	
2579		11.38		-0.48	
3100		9.05		-1.91	
3101	EN1186-3	11.2		-0.59	
3107	EN1186-3	11.1	C	-0.65	first reported 22.1
3113	EN1186	16.04	C	2.36	first reported 21.58
3142	EN1186-3	10.38		-1.09	
3146	EN1186	8.88		-2.01	
3151	EN1186-1	8.07		-2.50	
3154	EN1186-6	10.30		-1.14	
3172		-----		-----	
3182	EN1186-3	9.30		-1.75	
3185	EN1186 & EC 10/2011	10.88		-0.79	
3208	EU-10/2011	18.70		3.99	
3218	EN1186	10.03		-1.31	
3228	EN1186-3	13.00		0.51	
3229	EN1186-3	19.52		4.49	
3233	EN1186-3	11.49		-0.42	
3237	EN1186-3	6.37		-3.54	

|				only 100 ml/dm^2:	only 50 m/dm^2:
	normality	not OK		OK	OK
	n	52		23	8
	outliers	2		1	0
	mean (n)	12.171		12.226	12.279
	st.dev. (n)	3.6897		3.0079	5.6291
	R(calc.)	10.331		8.422	15.761
	R(EN1186-3:02)	4.586		4.607	4.627

Determination of 3rd Overall Migration on sample #13183; results in mg/dm^2, continued

Determination of 3rd Overall Migration on sample #13183; results in mg/kg

lab	method	value	mark	z(targ)	remarks
310	EN1186-3	107.00		2.39	
330	EN1186-3	54.0		-2.47	
357	EN1186-3	55.14		-2.37	
551	EN1186-3	220.00	DG(0.01)	12.76	
2104	EN1186	-----		-----	
2115		96.90		1.46	
2127		-----		-----	
2129	EN1186	62.82		-1.66	
2135	EN1186-3	831	G(0.01)	68.84	
2146		68.1		-1.18	
2165	EN1186-3	70.98		-0.92	
2172	EN1186	80.46		-0.05	
2184	EN1186 & EC 10/2011	82		0.10	
2186		23.0		-5.32	
2190		135		4.96	
2212	CFR 175.300, EN1186-1&3	101.00		1.84	
2216	21CFR175.300	-----		-----	
2229	EN1186	79.32		-0.15	
2241	EN1186-1&3	91.86	C	1.00	first reported 183.72
2256	EN1186-1&3	137.00		5.14	
2266	EN1186-3	223.2	DG(0.01)	13.06	
2284	EN1186	104.70		2.18	
2309	EN1186-3	-----		-----	
2353	EN1186-3	68.13		-1.18	
2372	EN1186 & EC 10/2011	93.12		1.12	
2385	EN1186-3	123.75		3.93	
2386		-----		-----	
2403	EN1186-3	76.20		-0.44	
2409		681.26	G(0.01)	55.10	
2433	EN1186-3	86.76	C	0.53	first reported 175.52
2462	EN1186-3	65.14		-1.45	
2488		-----		-----	
2495	EN1186	71.5		-0.87	
2498		117.00		3.31	
2525		62.00		-1.74	
2544	EN1186-3	84.66		0.34	
2549	EN1186-3	27.12		-4.94	
2551		207.50	C,G(0.01)	11.62	first reported 103.75
2579		68.28		-1.16	
3100		54.30		-2.45	
3101	EN1186-3	67.2		-1.26	
3107	EN1186-3	122.8		3.84	
3113	EN1186	96.22	C	1.40	first reported 215.82
3142	EN1186-3	110.00		2.67	
3146	EN1186	53.28		-2.54	
3151	EN1186-1	48.42		-2.99	
3154	EN1186-6	725.40	G(0.01)	59.15	
3172		-----		-----	
3182	EN1186-3	55.81		-2.31	
3185	EN1186 & EC 10/2011	65.28		-1.44	
3208	EU-10/2011	-----	W	-----	
3218	EN1186	60.24		-1.90	
3228	EN1186-3	81.94		0.09	
3229	EN1186-3	90.22		0.85	
3233	EN1186-3	69.00		-1.10	
3237	EN1186-3	132.50	C	4.73	first reported 421.47
	normality	OK			
	n	42			
	outliers	6			
	mean (n)	80.956			
	st.dev. (n)	27.2499			
	R(calc.)	76.300			
	R(EN1186-3:02)	30.505			

Determination of 3rd Overall Migration on sample #13183; results in mg/kg, continued
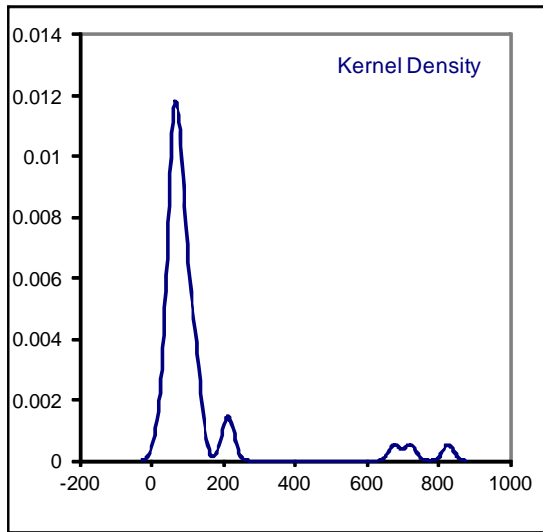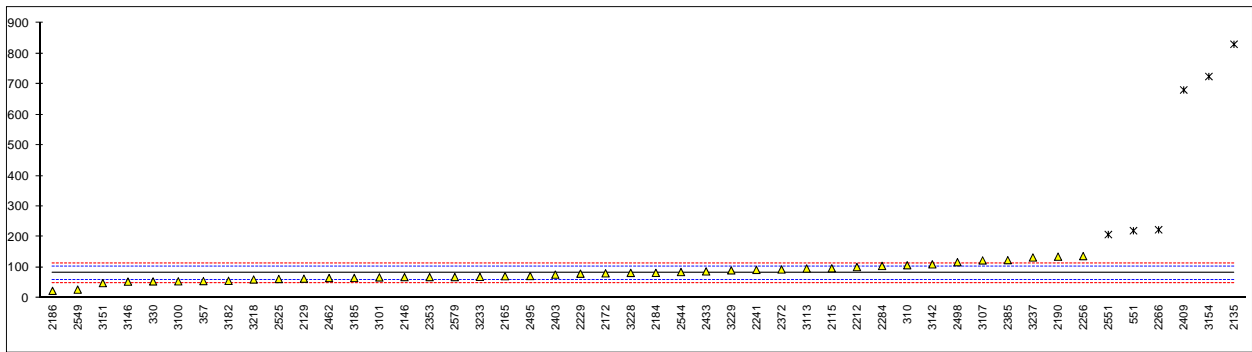
APPENDIX 2

Actual amount of simulant for each migration step, amount of residue after evaporation and actual contact surface used on sample #13183; results in ml, mg and dm^2

lab	ml simulant 1st Migration	mg residue 1st Migration	ml simulant 2nd Migration	mg residue 2nd Migration	ml simulant 3rd Migration	mg residue 3rd Migration	dm^2 contact surface
310	100	37.33	100	18.57	100	17.83	1
330	100	12.23	100	6.37	100	5.40	1
357	120	38.40	120	28.80	120	19.20	2.1
551	100	33.80	100	27.20	100	22.00	1
2104	100	40.65	100	52.85	100	41.15	2
2115	200	54.61	200	20.44	200	16.15	1
2127	-----	-----	-----	-----	-----	-----	-----
2129	150	42.68	150	28.88	150	20.93	2.0
2135	100	22.9	100	16.1	100	12.4	0.96
2146	100	73.7	100	30.6	100	22.7	2
2165	350	73.05	350	48.24	350	24.61	2.08
2172	200	58.76	200	43.88	200	26.82	2.0
2184	350	61.2	350	43.0	350	28.6	2.09
2186	1000	53.5	1000	36	1000	23	2
2190	100	16.3	100	19.0	100	13.5	1
2212	100	16.30	100	15.90	100	10.10	1.08
2216	500	33.98	500	26.33	500	20.98	2.085
2229	210	35.10	210	36.00	210	27.80	2.1
2241	100	58.81	100	42.91	100	30.62	2
2256	100	18.20	100	23.1	-----	13.70	1.0119
2266	100	42.4	100	44.1	100	37.2	1
2284	200	40.98	200	33.12	200	20.94	2.00
2309	100	41.75	100	31.80	100	21.70	2
2353	100	30.99	100	19.92	100	11.36	1
2372	212	74.50	212	55.20	212	32.90	2.12
2385	200	46.60	200	30.09	200	24.75	1.12
2386	-----	-----	-----	-----	-----	-----	-----
2403	100	31.80	100	18.45	100	14.35	1.13
2409	200	59.90	200	33.50	200	21.60	2.08
2433	100	24.75	100	15.35	100	7.23	0.50
2462	350	54.10	350	38.31	350	22.80	2.08
2488	200	90.20	200	28.70	200	12.30	2
2495	150	29.6	150	30.7	150	24.8	2.08
2498	100	53.00	100	43.70	100	39.00	2
2525	250	39.00	250	25.90	250	15.50	2
2544	100	44.90	100	23.39	100	14.11	1.0
2549	100	32.26	100	28.11	100	9.41	2.08
2551	100	40.65	100	39.85	100	20.95	2
2579	210	52.90	210	36.31	210	23.90	2.10
3100	200	39.05	200	29.50	200	18.10	2.0
3101	210	48.1	210	36.6	210	23.6	2.10
3107	180	35.9	180	39.2	180	22.1	2.0
3113	200	66.41	200	43.16	200	32.08	2.00
3142	100	32.55	100	18.35	100	11	1.06
3146	200	-----	200	48.70	200	17.75	2
3151	350	29.05	350	30.25	350	16.85	2.087
3154	170	20.75	170	12.40	170	8.85	0.86
3172	-----	-----	-----	-----	-----	-----	-----
3182	300	48.9	300	29.95	300	19.70	2.12
3185	200	35.70	200	32.25	200	21.75	2.00
3208	100	60.60	90	42.40	90	37.40	2
3218	333	39.76	333	33.04	333	20.06	2.0
3228	330	70.93	330	47.90	330	27.04	2.08
3229	450	64.70	450	35.70	450	40.60	2.08
3233	333	37.30	333	32.63	333	22.98	2
3237	100	42.81	100	31.65	100	13.25	2.08

APPENDIX 3

Test conditions when selected by participants

lab	type of simulant	exposure time in hrs	exposure temperature in °C	reporting unit	migration method	method used to determine contact surface	sample pretreatment
310	3% acetic acid	2	70	mg/dm^2	immersion	EN1186-1	plate cut in two
330	distilled water	2	70	mg			plate cut in two
357	various	various	various	mg/kg	many various	measurement & calc.	none
551							pl. cut in 4 parts
2104	3% acetic acid	2	40	mg/dm^2	total immersion		pl. cut in 8 parts
2115							
2127							
2129	3% acetic acid	2	40	mg/dm^2	total immersion		
2135	3% acetic acid	2	70	mg/dm^2	total immersion	ruler	plate cut in two
2146	3% acetic acid	2	70	mg/dm^2	total immersion	calculation	
2165							
2172	3% acetic acid	2	40	mg/dm^2	total immersion	calculation	
2184	varies per use	2	70	g	total immersion		
2186							
2190							
2212	3% acetic acid	2	40	per use	article filling	counter and caliper	plate cut in two
2216	water	2	65.6 (150°F)	mg/dm^2	total immersion	caliper	
2229	3% acetic acid	2.0	70	mg/dm^2	total immersion		
2241						GB/T5009.156-2003	pl. cut in 4 parts
2256		2	40			EN1186-1&3:2002	plate cut in two
2266	water	2	70	mg/dm^2	total immersion	measurement	
2284	water	2.0	70	mg/dm^2	total immersion	calculation	
2309	distilled water	2	70	mg/dm^2	total immersion	EN1186-1	
2353	3% acetic acid	2	40	mg/kg	article filling	gravimetric / graph paper	plate cut in two
2372	water	2	70	mg/dm^2	total immersion		
2385	3% acetic acid	2	70	mg/dm^2	total immersion	EN1186	plate cut in two
2386							
2403							
2409	95% Ethanol	2.0	70.0	mg/dm^2	total immersion		
2433	3% acetic acid	2	40	mg/dm^2	total immersion	measured by ruler	pl. cut in 4 parts
2462	3% acetic acid	2	40	mg/dm^2	total immersion	micrometer	none
2488	water	2	70	mg/dm^2	total immersion		
2495	3% acetic acid	2	70	mg/dm^2	total immersion	measure with ruler	
2498							
2525	3% acetic acid	2	70	mg/kg	article filling	ruler caliper	
2544	3% acetic acid	2	70	mg/kg	cell	EUR24815 clause7.3	
2549							
2551	3% acetic acid	2	70	mg/dm^2	total immersion	ruler	
2579							
3100							
3101	distilled water	2.0	70	mg/dm^2	total immersion	EN1186-1:2002	
3107	3% acetic acid	2	70	mg/dm^2	total immersion	calculation	none
3113							
3142							pl. cut in 4 parts
3146	3% acetic acid	2	70	mg/dm^2	total immersion	EN1186	
3151	distilled water	2	70	mg/kg	immersion	meas./calc.	
3154							used 0.43dm^2
3172							
3182	3% acetic acid	2	70		article filling	vernier	
3185	per actual use	per use	per use	per use	total immersion	calculation	
3208							none
3218	3% acetic acid	2.0	70.0	mg/dm^2	total immersion	calculation	
3228							
3229	3% acetic acid	2	40	mg/dm^2	total immersion	ruler	
3233	3% acetic acid	2	70	mg/dm^2	filling		
3237	3% acetic acid						none

APPENDIX 4

Number of participating laboratories per country

     1 lab in   BRAZIL

     1 lab in   DENMARK

     2 labs in FINLAND

     4 labs in FRANCE

    12 labs in GERMANY

     4 labs in HONG KONG

     2 labs in INDIA

     3 labs in ITALY

     1 lab in   MALAYSIA

    15 labs in P.R. of CHINA

     1 lab in   PHILIPPINES

     1 lab in   SAUDI ARABIA

     1 lab in   SPAIN

     1 lab in   TAIWAN R.O.C.

     2 labs in THAILAND

     1 lab in   THE NETHERLANDS

     2 labs in TURKEY

     1 lab in   U.S.A.

     1 lab in   UNITED KINGDOM

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
n.a.   = not applicable
E   = possible calculation error
W   = result was withdrawn

Literature:

1   iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, January 2010
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 1186-14:02 - Test methods for 'substitute tests' for overall migration from plastics intended to come into contact with fatty foodstuffs using test media iso-octane and 95 % ethanol
5   ASTM E1301-03
6   ISO 5725-86
7   ISO 5725, parts 1-6, 1994
8   M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
9   W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
10   IP 367/96
11   DIN 38402 T41/42
12   P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
13   J.N. Miller, Analyst, 118, 455, (1993)
14   Analytical Methods Committee Technical Brief, No4 January 2001
15   The Royal Society of Chemistry 2002, Analyst 2002, 127 pages 1359-1364, P.J. Lowthian and M. Thompson. (see http://www.rsc.org/suppdata/an/b2/b205600n/)
16   R.G. Visser, Reliability of proficiency test results for metals and phthalates in plastics, Accred Qual Assur, 14:29-34 (2009)