Results of Proficiency Test
Specific Migration (fcm)
October 2020

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. R.J. Starink
Correctors: ing. A.S. Noordman-de Neef & ing. C.M. Nijssen-Wester

Report: iis20P10SM

January 2021

CONTENTS

1 INTRODUCTION 3

2 SET UP 3

2.1 ACCREDITATION 3

2.2 PROTOCOL 3

2.3 CONFIDENTIALITY STATEMENT 4

2.4 SAMPLES 4

2.5 ANALYZES 5

3 RESULTS 6

3.1 STATISTICS 6

3.2 GRAPHICS 7

3.3 Z-SCORES 7

4 EVALUATION 8

4.1 EVALUATION PER SAMPLE AND PER COMPONENT 8

4.2 PERFORMANCE EVALUATION OF THE GROUP OF LABORATORIES 9

4.3 COMPARISON OF PROFICIENCY TEST OF OCTOBER 2020 TO PREVIOUS PTs 10

4.4 EVALUATION OF THE ANALYTICAL DETAILS 10

5 DISCUSSION 11

6 CONCLUSION 11

Appendices:

1. Data, statistical and graphic results sample #20680 12
2. Data, statistical and graphic results sample #20681 16
3. Analytical Details 20
4. Number of participants per country 22
5. Abbreviations and literature 23

1 INTRODUCTION

During the contact of food with materials (like kitchenware) molecules can migrate from the 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. 4) applies to all food contact materials and describes a large number of requirements, e.g. limits for Overall Migration and Specific Migration limits for certain constituents. Article 11 (and Annex II) of this regulation describes the Specific Migration limit, expressed in mg/kg food or food simulant.

Since 2012 the Institute of Interlaboratory Studies (iis) organizes a proficiency test scheme for food contact materials every year. During the annual proficiency testing program 2020/2021 it was decided to continue the proficiency test for the determination of Specific Migration on food contact materials. In this interlaboratory study 26 laboratories from 14 different countries registered for participation. See appendix 4 for the number of participants per country. In this report the results of this proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send two samples. The first sample was a bowl labelled #20680 which was positive on Formaldehyde. The second sample was a cup labelled #20681 which was positive on some metals. For both samples a number of test conditions were prescribed (migration method, type of simulant, exposure time and temperature). Participants were also requested to report some intermediate test results and 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

For the first sample a batch of red colored Melamine Bowls containing a relevant concentration of Formaldehyde was obtained from the local market. The subsamples were labelled #20680.
The homogeneity of the subsamples was checked by determination of the Specific Migration of Formaldehyde by an inhouse test method on 7 stratified randomly selected subsamples. Migration conditions: 1st step, 3% Acetic Acid, 400 mL, 120 min at 70°C.

	Formaldehyde in mg/dm^2
Sample #20680-1	0.8186
Sample #20680-2	0.7786
Sample #20680-3	0.8369
Sample #20680-4	0.8520
Sample #20680-5	0.8117
Sample #20680-6	0.7402
Sample #20680-7	0.8781

Table 1: homogeneity test results of subsamples #20680

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

	Formaldehyde in mg/dm^2
r(observed)	0.1291
reference method	Horwitz
0.3 x R (reference method)	0.1131

Table 2: evaluation of the repeatabilities of subsamples #20680

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

For the second sample a batch of beige colored Polypropylene cups containing a relevant concentration of the metals Cobalt and Lithium was prepared by a third party. The subsamples were labelled #20681.
The homogeneity of the subsamples was checked by determination of the Specific Migration of Cobalt and Lithium by an inhouse test method on 8 stratified randomly selected subsamples. Migration conditions: single use, 3% Acetic Acid, 200 mL, 120 min at 100°C.

	Cobalt in mg/dm^2	Lithium in mg/dm^2
Sample #20681-1	0.1110	0.1785
Sample #20681-2	0.1173	0.1911
Sample #20681-3	0.1143	0.1848
Sample #20681-4	0.1168	0.1797
Sample #20681-5	0.1100	0.1886
Sample #20681-6	0.1082	0.1797
Sample #20681-7	0.1189	0.1873
Sample #20681-8	0.1068	0.1810

Table 3: homogeneity test results of subsamples #20681

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

	Cobalt in mg/dm^2	Lithium in mg/dm^2
r(observed)	0.0127	0.0134
reference method	Horwitz	Horwitz
0.3 x R (reference method)	0.0211	0.0319

Table 4: evaluation of the repeatabilities of subsamples #20681

The calculated repeatabilities were in agreement with 0.3 times the reproducibility of the corresponding reference method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample #20680 and one sample #20681 were sent on September 9, 2020.

2.5 ANALYZES

The participants were requested to determine Formaldehyde on sample #20680 using the prescribed test conditions (article filling, repeated use, 120 minutes at 70°C and 3% Acetic Acid as simulant). For sample #20681 it was requested to determine: Barium, Cobalt, Copper, Iron, Lithium, Manganese and Zinc using the prescribed conditions (article filling, single use, 120 minutes at 100°C and 3% Acetic Acid as simulant)
It was also requested to report for both samples if the laboratory was accredited for the requested components that were determined and to report a few analytical details.

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

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

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per sample and per determination in appendices 1 and 2 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 re-analysis). Additional or corrected test results are used for the data analysis and the original test results are placed under 'Remarks' in the test result tables in appendices 1 or 2. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as ‘<...’ or ‘>...’ were not used in the statistical evaluation.

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

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

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

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1 and 2). 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 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. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, 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.

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-purpose.

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 of 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, no problems were encountered with the dispatch of the samples. For sample #20680 four participants did not report any test results and for sample #20681 three participants did not report any test results.
Finally, in total were reported 104 test results in mg/dm^2. Observed were 11 outlying test results, which is 10.6% of the statistically evaluated numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

All original datasets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section the results are discussed per sample and per component. 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 reported test results in appendix 1 and 2. The abbreviations, used in these tables, are explained in appendix 5.

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. This makes the Specific Migration from food contact materials more difficult than determination of the Overall Migration.

In the past, iis has found that the Overall and Specific Migration methods, limits and calculations are mixed up and used inappropriately by participants. So iis issued a White Paper on this subject in February 2018 (White Paper on the determination of Overall and Specific Migration on food contact materials, lit. 19) to help participants understand the differences between the two methods, the units used for reporting and the regulated limits. The test results of the Specific Migration reported in mg/dm^2 were used for the statistical evaluation.

For the determination of Specific Migration, several test methods exist. The most relevant literature is test method EN13130 part 1 and part 23. Method EN13130-1 and EN13130-23 describes how the Specific Migration test should be performed. Regretfully no reference test method is available with precision requirements for the migration of Formaldehyde and for Metals from food contact materials in mg/dm^2. Therefore, it was decided to estimate the target reproducibilities calculated from the Horwitz equation.

Sample #20680

Formaldehyde: This determination may be problematic for all three steps. In total seven statistical outliers were observed over three migration steps and one other test result was excluded. The calculated reproducibility of each migration step after rejection of the suspect data is not in agreement with the estimated reproducibility calculated from the Horwitz equation.

Sample #20681

Cobalt: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the estimated reproducibility calculated from the Horwitz equation.

Lithium: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the estimated reproducibility calculated from the Horwitz equation.

Other Metals: The majority of participants agreed on a concentration near or below the limit of detection for the other requested Metals. Therefore, no z-scores are calculated. See appendix 2 for the reported test results.

4.2 PERFORMANCE EVALUATION OF THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the estimated target reproducibility using the Horwitz equation 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 target reproducibility derived from the estimated target reproducibility are presented in the next tables.

Component	unit	n	average	2.8 * sd	R(target)
Formaldehyde – step 1	mg/dm^2	18	0.622	0.887	0.299
Formaldehyde – step 2	mg/dm^2	18	0.470	0.507	0.236
Formaldehyde – step 3	mg/dm^2	17	0.321	0.249	0.171

Table 5: Reproducibilities of components on sample #20680

Component	unit	n	average	2.8 * sd	R(target)
Cobalt	mg/dm^2	20	0.217	0.102	0.123
Lithium	mg/dm^2	19	0.315	0.174	0.168

Table 6: Reproducibilities of components on sample #20681

Without further statistical calculations it can be concluded that for Formaldehyde present in the sample there is not a good compliance of the group of laboratories with the relevant target reproducibility.
There was a good compliance for the determination of both metals Cobalt and Lithium of the group of laboratories with the relevant target reproducibilities. See for discussion also paragraph 4.1 and 5.

4.3 COMPARISON OF PROFICIENCY TEST OF OCTOBER 2020 TO PREVIOUS PROFICIENCY TESTS

The evolution of the uncertainty for Specific Migration in mg/dm^2 as observed in this PT and the comparison with the findings in previous rounds are listed in below table.

Year	Components	Type of migration	Observed RSD%	Target RSD%	Concentration range mg/dm^2
2012	Formaldehyde	article filling	41 – 47	14 – 20	0.2 – 3
2013	Formaldehyde	article filling	41 – 61	14 – 20	0.2 – 3
2014	Bisphenol-A	total immersion	44 – 52	14 – 20	0.2 – 3
2015	DEHP	total immersion	34 – 40	14 – 20	0.2 – 3
2016	Metals	total immersion	29 – 30	14 – 20	0.2 – 3
2017	Bisphenol-A	article filling	33 – 50	20 – 33	0.009 – 0.2
2018	Metals	article filling	21 – 35	17 – 38	0.003 – 0.6
2019	DEHP/DAP	article filling	24 – 34	19 – 20	0.20 – 0.34
2020	Formaldehyde	article filling	28 – 51	17 – 19	0.32 – 0.62
	Metals	article filling	17 - 20	19 - 20	0.22 – 0.32

Table 7: comparison of the uncertainties in % for Specific Migration in the present and previous PTs

From the above table, it is clear that the performance of this PT is in line with the previous PTs.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

The reported analytical details that were used by the participants are listed in appendix 3. About 60% of the reporting laboratories are accredited for the determination of the Specific Migration for both Formaldehyde and Metals.

About 70% of the reporting participants mentioned to have used test method EN13130 for the Specific Migration of Formaldehyde (part1 and 23) and for Metals (part1).
For sample #20680 about 50% of the participants reported to clean the sample before the determination of the Specific Migration. One of these participants reported to clean the cup with lint-free cloth. Eight other participants reported to clean the cup with water, which is not in line with test method EN13130-1 paragraph 15.5. About 80% of the participants preheated the simulant solution to 70°C.

For sample # 20681 about 30% of the participants reported to clean the sample for the determination of Specific Migration. One of these participants reported to clean the cup with lint-free cloth. Five other participants reported to clean the cup with water. About 80% of the participants preheated the simulant solution to 70°C.

For both samples about 80% of participants used an oven for the test. Two participants reported to have used an incubator and one used a water bath.

The seal used during testing differs much. For both samples four different types of seals were used by the participants, e.g. a plastic film/foil, an aluminum film, a glass plate or an airtight container.

None of the details have shown a parent influence on the final results in this PT.

5 DISCUSSION

The limits for specific migration for Formaldehyde and Metals are mentioned in mg/kg food. As it is mentioned in EN13130-1, the limits expressed in mg/kg shall be divided by the conventional conversion factor of 6 in order to express them in mg/dm^2, see next table.

Component	Specific Migration Limit in mg/kg	Specific Migration Limit in mg/dm^2
Formaldehyde	0.01	0.002
Cobalt	0.05	0.008
Lithium	0.6	0.1

Table 8: Specific Migration maximum limits according to 10/2011/EU

All reporting laboratories would reject sample #20680 for containing too much Formaldehyde and sample #20681 for containing too much Cobalt and Lithium.

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 (SM1>SM2>SM3). The third step will be leading for the end result of approval of the sample.

When evaluating the test results of sample #20680, nine participants reported higher results in step 2 and/or 3 than the step before. Twelve participants find the Specific Migration steps correctly SM1>SM2>SM3. Since the average PT results are also lower with each step the laboratories reporting higher results than the step before may reject a sample for this under the new regulation.

6 CONCLUSION

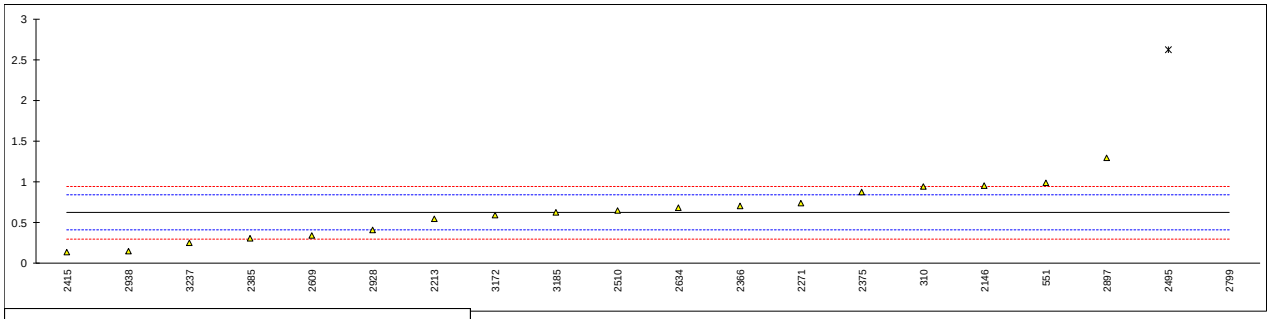
Each laboratory should evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and the quality of the analytical results.

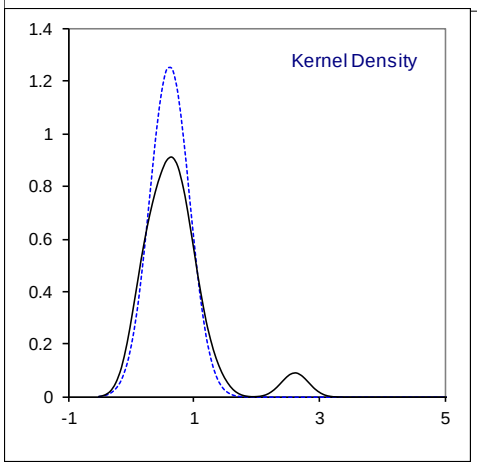
APPENDIX 1

Determination of 1st Specific Migration of Formaldehyde on sample #20680; results in mg/dm^2 per contact surface

lab	Method	value	mark	z(targ)	remarks
310	EN13130-1	0.9383		2.96	
551	EN13130-1	0.9926		3.46	
2129		-----		-----	
2146		0.9501		3.07	
2159	EN13130-23	< D.L		-----	
2213	EN13130-23	0.549		-0.68	
2271	EN13130-1	0.7381		1.08	
2366	EN13130-1	0.71		0.82	
2375	EN13130-23	0.878		2.39	
2385		0.311		-2.91	
2415	EN13130-23	0.142		-4.49	
2495	EN13130-1	2.6204	G(0.01)	18.69	
2510	In house	0.651		0.27	
2609	EN13130-23	0.3446		-2.60	
2634	In house	0.68		0.54	
2749		-----		-----	
2799	EN13130-23	21.8196	C,G(0.01)	198.24	First reported 6.5004
2840		-----		-----	
2897	EN13130-23	1.293		6.27	
2901		-----		-----	
2928	EN13130-23	0.412		-1.97	
2938	In house	0.1476		-4.44	
3172	EN13130-23	0.59	C	-0.30	First reported 1.17
3185	EN13130-23	0.623		0.01	
3218		-----		-----	
3237	EN13130-23	0.25		-3.48	

	normality	OK
	n	18
	outliers	2
	mean (n)	0.62224
	st.dev. (n)	0.316619
	R(calc.)	0.88653
	st.dev.(Horwitz)	0.106928
	R(Horwitz)	0.29940

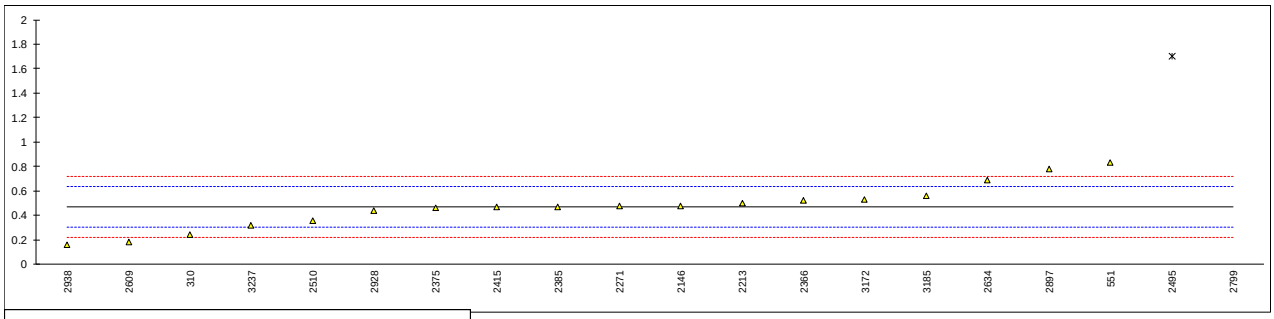


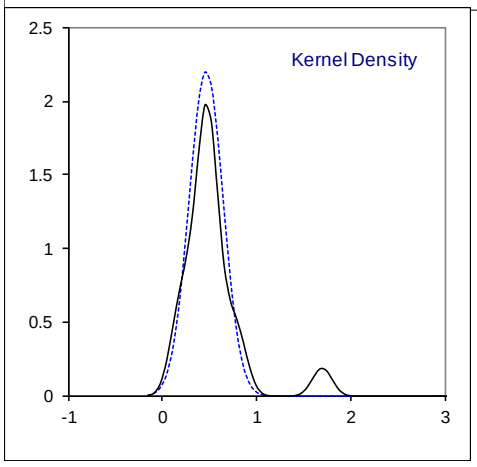


Determination of 2nd Specific Migration of Formaldehyde on sample #20680; results in mg/dm^2 per contact surface

lab	method	value	mark	z(targ)	remarks
310	EN13130-1	0.23992		-2.73	
551	EN13130-1	0.8317		4.29	
2129		-----		-----	
2146		0.4783		0.10	
2159	EN13130-23	< D.L		-----	
2213	EN13130-23	0.498		0.33	
2271	EN13130-1	0.4778		0.09	
2366	EN13130-1	0.52		0.59	
2375	EN13130-23	0.462		-0.09	
2385		0.469		-0.01	
2415	EN13130-23	0.468		-0.02	
2495	EN13130-1	1.7021	G(0.01)	14.62	
2510	In house	0.353		-1.39	
2609	EN13130-23	0.1816		-3.42	
2634	In house	0.69		2.61	
2749		-----		-----	
2799	EN13130-23	16.8017	C,G(0.01)	193.85	First reported 5.0055
2840		-----		-----	
2897	EN13130-23	0.776		3.63	
2901		-----		-----	
2928	EN13130-23	0.439		-0.37	
2938	In house	0.16256		-3.65	
3172	EN13130-23	0.53	C	0.71	First reported 1.06
3185	EN13130-23	0.563		1.10	
3218		-----		-----	
3237	EN13130-23	0.32		-1.78	

	normality	OK
	n	18
	outliers	2
	mean (n)	0.46999
	st.dev. (n)	0.180909
RSD=38%		
	R(calc.)	0.50654
	st.dev.(Horwitz)	0.084249
	R(Horwitz)	0.23590

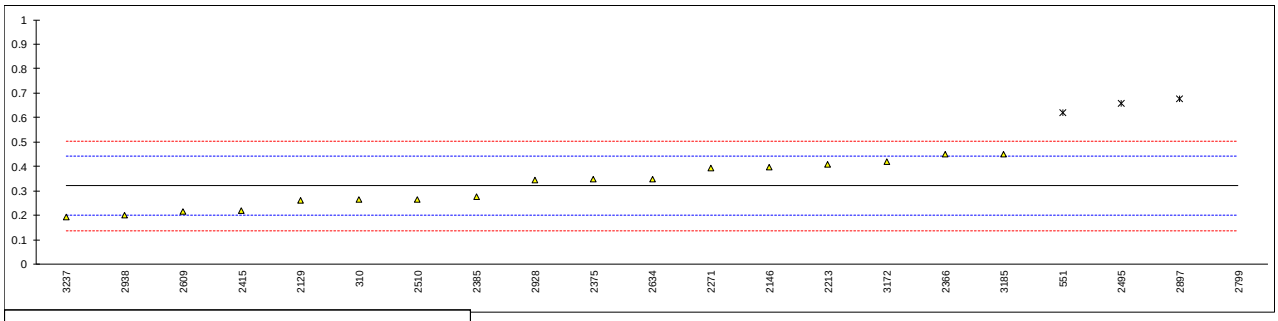


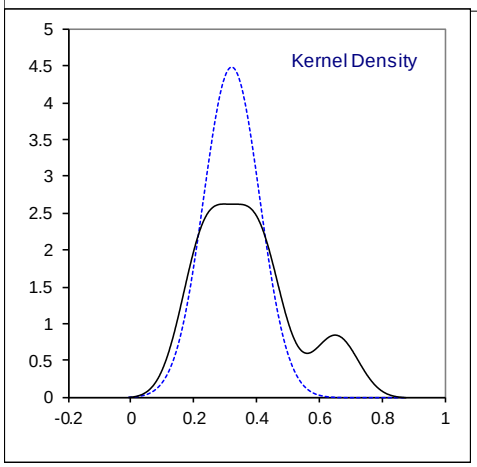


Determination of 3rd Specific Migration of Formaldehyde on sample #20680; results in mg/dm^2 per contact surface

lab	method	value	mark	z(targ)	remarks
310	EN13130-1	0.26508		-0.92	
551	EN13130-1	0.6213	DG(0.05)	4.92	
2129	EN13130-1	0.26		-1.01	
2146		0.3975		1.25	
2159	EN13130-23	< D.L		-----	
2213	EN13130-23	0.408		1.42	
2271	EN13130-1	0.3925		1.17	
2366	EN13130-1	0.45		2.11	
2375	EN13130-23	0.348		0.44	
2385		0.275		-0.76	
2415	EN13130-23	0.221		-1.64	
2495	EN13130-1	0.6598	ex	5.55	Test result excluded as first step and second step are outliers
2510	In house	0.267		-0.89	
2609	EN13130-23	0.2168		-1.71	
2634	In house	0.35		0.47	
2749		-----		-----	
2799	EN13130-23	15.9050	C,G(0.01)	255.52	First reported 4.7384
2840		-----		-----	
2897	EN13130-23	0.676	DG(0.05)	5.82	
2901		-----		-----	
2928	EN13130-23	0.344		0.37	
2938	In house	0.2021		-1.95	
3172	EN13130-23	0.42	C	1.62	First reported 0.83
3185	EN13130-23	0.451		2.13	
3218		-----		-----	
3237	EN13130-23	0.194		-2.09	

	normality	OK
	n	17
	outliers	3 (+1ex)
	mean (n)	0.32129
	st.dev. (n)	0.089085
	R(calc.)	0.24944
	st.dev.(Horwitz)	0.060987
	R(Horwitz)	0.17076





Details on final concentration, surface area and volume of simulant reported per step on sample #20680

lab	1st Final conc. (mg/L)	1st surface area (dm²)	1st volume simulant (mL)	2nd Final conc. (mg/L)	2nd surface area (dm²)	2nd volume simulant (mL)	3rd Final conc. (mg/L)	3rd surface area (dm²)	3rd volume simulant (mL)
310	4.6917	2.49982	500	1.1996	2.49982	500	1.3254	2.49982	500
551	2.91630	2.938	518.0	2.4436	2.938	518.0	1.8253	2.938	518.0
2129	-----	-----	-----	-----	-----	-----	1.58	2.4	400
2146	4.8455	2.545	500	2.4395	2.545	500	2.0270	2.545	500
2159	< D.L	2.47	500	< D.L	2.47	500	< D.L	2.47	500
2213	2.81	2.56	500	2.55	2.56	500	2.09	2.56	500
2271	3.7055	2.51	500	2.3985	2.51	500	1.9703	2.51	500
2366	3.53	2.41	485	2.56	2.41	485	2.25	2.41	485
2375	5.269	2.55	500	2.776	2.55	500	2.091	2.55	500
2385	1.645	2.64	500	2.48	2.64	500	1.455	2.64	500
2415	0.569	2.0041	500	1.877	2.0041	500	0.884	2.0041	500
2495	15.30	2.044	350	9.94	2.044	350	3.85	2.044	350
2510	3.765	2.650	470	2.022	2.650	475	1.514	2.650	480
2609	1.7929	2.6015	500.0	0.9451	2.6015	500.0	1.1281	2.6015	500.0
2634	6.49	0.96	100	6.62	0.96	100	3.35	0.96	100
2749	-----	-----	-----	-----	-----	-----	-----	-----	-----
2799	730.52	5.619	50	562.52	5.619	50	532.5	5.619	50
2840	-----	-----	-----	-----	-----	-----	-----	-----	-----
2897	6.7	2.585	500	4.02	2.585	500	3.50	2.585	500
2901	-----	-----	-----	-----	-----	-----	-----	-----	-----
2928	2.281	2.769	500	2.434	2.769	500	1.909	2.769	500
2938	0.84	2.276	400	0.925	2.276	400	1.15	2.276	400
3172	3.1	2.64	500	2.8	2.64	500	2.2	2.64	500
3185	3.014	2.42	500	2.725	2.42	500	2.185	2.42	500
3218	-----	-----	-----	-----	-----	-----	-----	-----	-----
3237	1.671	3.23	500	2.069	3.23	500	1.259	3.23	500

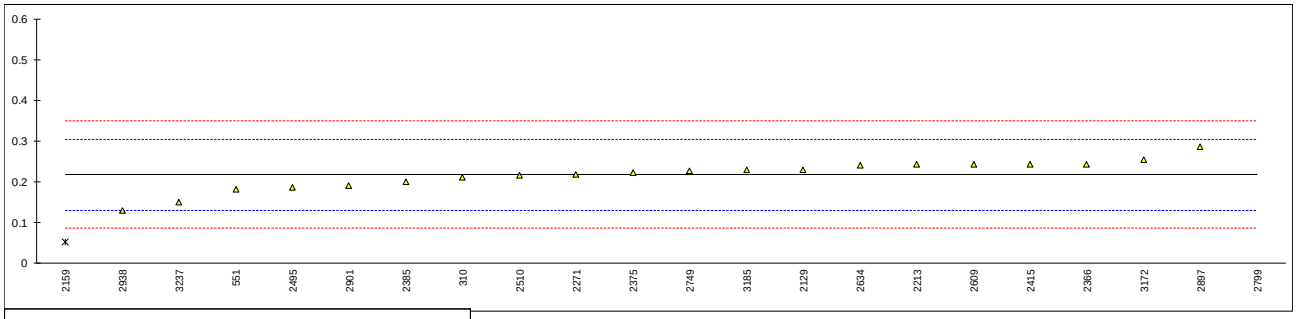
lab	1st calculated Specific Migration in mg/dm²	1st iis reported Specific Migration in mg/dm²	Difference absolute	2nd calculated Specific Migration in mg/dm²	2nd iis reported Specific Migration in mg/dm²	Difference absolute	3th calculated Specific Migration in mg/dm²	3thiis reported Specific Migration in mg/dm²	Difference absolute
310	0.9384	0.9383	0.00	0.2399	0.23992	0.00	0.2651	0.26508	0.00
551	0.5142	0.9926	-0.48	0.4308	0.8317	-0.40	0.3218	0.6213	-0.30
2129	---	---	---	---	---	---	0.2633	0.26	0.00
2146	0.9520	0.9501	0.00	0.4793	0.4783	0.00	0.3982	0.3975	0.00
2159	---	---	---	---	---	---	---	---	---
2213	0.5488	0.549	0.00	0.4980	0.498	0.00	0.4082	0.408	0.00
2271	0.7381	0.7381	0.00	0.4778	0.4778	0.00	0.3925	0.3925	0.00
2366	0.7104	0.71	0.00	0.5152	0.52	0.00	0.4528	0.45	0.00
2375	1.0331	0.878	0.16	0.5443	0.462	0.08	0.4100	0.348	0.06
2385	0.3116	0.311	0.00	0.4697	0.469	0.00	0.2756	0.275	0.00
2415	0.1420	0.142	0.00	0.4683	0.468	0.00	0.2205	0.221	0.00
2495	2.6199	2.6204	0.00	1.7021	1.7021	0.00	0.6592	0.6598	0.00
2510	0.6678	0.651	0.02	0.3624	0.353	0.01	0.2742	0.267	0.01
2609	0.3446	0.3446	0.00	0.1816	0.1816	0.00	0.2168	0.2168	0.00
2634	0.6760	0.68	0.00	0.6896	0.69	0.00	0.3490	0.35	0.00
2749	---	---	---	---	---	---	---	---	---
2799	---	---	---	---	---	---	---	---	---
2840	---	---	---	---	---	---	---	---	---
2897	1.2959	1.293	0.00	0.7776	0.776	0.00	0.6770	0.676	0.00
2901	---	---	---	---	---	---	---	---	---
2928	0.4119	0.412	0.00	0.4395	0.439	0.00	0.3447	0.344	0.00
2938	0.1476	0.1476	0.00	0.1626	0.16256	0.00	0.2021	0.2021	0.00
3172	0.5871	0.59	0.00	0.5303	0.53	0.00	0.4167	0.42	0.00
3185	0.6227	0.623	0.00	0.5630	0.563	0.00	0.4514	0.451	0.00
3218	---	---	---	---	---	---	---	---	---
3237	0.2587	0.25	0.01	0.3203	0.32	0.00	0.1949	0.194	0.00

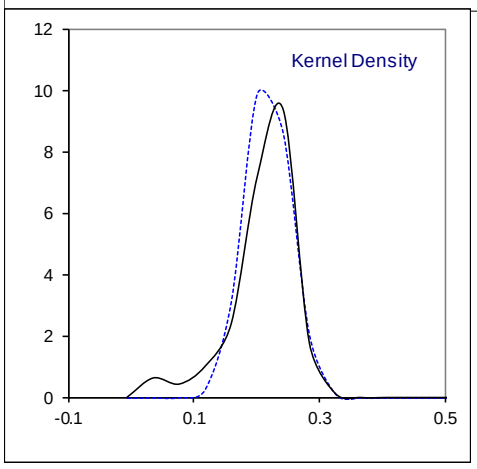
APPENDIX 2

Determination of Specific Migration of Cobalt as Co on sample #20681; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
310	EN13130-1	0.2122		-0.12	
551	EN13130-1	0.1818	E	-0.81	Calculation difference, iis calculated 0.04462
2129	EN13130-1	0.23		0.29	
2146		-----		-----	
2159	EN13130-1	0.052	G(0.01)	-3.78	
2213	EN13130-1	0.243		0.59	
2271	EN13130-1	0.2178		0.01	
2366	EN13130-1	0.244		0.61	
2375	EN13130-1	0.222	E	0.11	Calculation difference, iis calculated 0.20221
2385		0.200		-0.40	
2415	EN13130-1	0.244		0.61	
2495	EN13130-1	0.18543451		-0.73	
2510	In house	0.2151		-0.05	
2609	EN13130-1	0.243		0.59	
2634	EN13130-1	0.24		0.52	
2749	In house	0.226720		0.22	
2799	In house	10.513	C,G(0.01)	235.33	First reported 10.119
2840		-----		-----	
2897	EPA6010	0.287		1.59	
2901	EN13130-1	0.1908		-0.61	
2928		-----		-----	
2938	In house	0.13007	E	-1.99	Calculation difference, iis calculated 0.12778
3172	EN13130-1	0.254	C	0.84	First reported 1.015
3185	EN13130-1	0.2293		0.27	
3218		-----		-----	
3237	EN13130-1	0.15	E	-1.54	Calculation difference, iis calculated 0.0754

	normality	OK
	n	20
	outliers	2
	mean (n)	0.21731
	st.dev. (n)	0.036512
	R(calc.)	0.10223
	st.dev.(Horwitz)	0.043750
	R(Horwitz)	0.12250

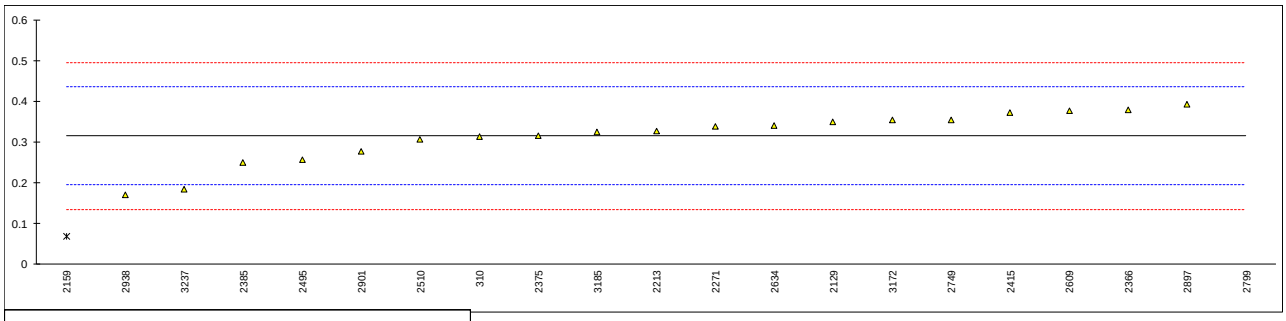


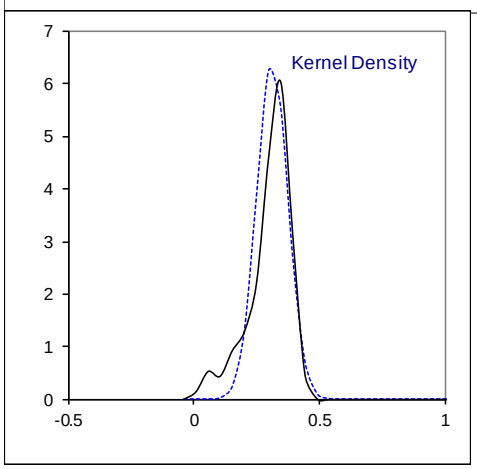


Determination of Specific Migration of Lithium as Li on sample #20681; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
310	EN13130-1	0.3135		-0.02	
551		-----		-----	
2129	EN13130-1	0.35		0.59	
2146		-----		-----	
2159	EN13130-1	0.068	G(0.05)	-4.12	
2213	EN13130-1	0.326		0.19	
2271	EN13130-1	0.3372		0.37	
2366	EN13130-1	0.378		1.05	
2375	EN13130-1	0.316	E	0.02	Calculation difference, iis calculated 0.28788
2385		0.2507		-1.07	
2415	EN13130-1	0.373		0.97	
2495	EN13130-1	0.25629		-0.98	
2510	In house	0.3073		-0.13	
2609	EN13130-1	0.376		1.02	
2634	EN13130-1	0.34	C	0.42	First reported not detected
2749	In house	0.353358		0.64	
2799	In house	12.154	C,G(0.01)	197.52	First reported 11.6983
2840		-----		-----	
2897	EPA6010	0.393		1.30	
2901	EN13130-1	0.2781		-0.61	
2928		-----		-----	
2938	In house	0.170658	E	-2.40	Calculation difference, iis calculated 0.16765
3172	EN13130-1	0.353	C	0.64	First reported 1.41
3185	EN13130-1	0.3244		0.16	
3218		-----		-----	
3237	EN13130-1	0.1848	E	-2.17	Calculation difference, iis calculated 0.00917

normality	OK
n	19
outliers	2
mean (n)	0.31481
st.dev. (n)	0.062092
R(calc.)	0.17386
st.dev.(Horwitz)	0.059940
R(Horwitz)	0.16783
	RSD=20%





Determination of Specific Migration of other elements on sample #20681; results in mg/dm^2

lab	Barium (Ba)	Copper (Cu)	Iron (Fe)	Manganese (Mn)	Zinc (Zn)
310	below reporting limit	below reporting limit	below reporting limit	below reporting limit	0.0033015
551	n	-----	-----	0.00266	0.000969
2129	n	-----	-----	-----	-----
2146	n	-----	-----	-----	-----
2159	< D.L	< D.L	< D.L	< D.L	< D.L
2213	<0.2	<1	<10	<0.1	<0.5
2271	<0.015	<0.015	<0.015	<0.015	<0.015
2366	<0.038	<0.038	<0.038	<0.038	<0.077
2375	ND	ND	ND	ND	ND
2385	<0,005	0.0007	0.00128	<0,005	0.00179
2415	not detected	not detected	not detected	not detected	not detected
2495	<0.001	<0.001	<0.001	<0.001	<0.001
2510	n	-----	-----	-----	-----
2609	〈0.00005〉	<0.00002	0.00275	〈0.00003	0.00172
2634	not detected	not detected	not detected	not detected	not detected
2749	0.000126	0.000113	0.004972	0.000955	0.001517
2799	<0.0005	1.326	<0.0005	<0.0005	0.545
2840	n	-----	-----	-----	-----
2897	0	0	0.001	0	0.001
2901	not detected	not detected	not detected	not detected	not detected
2928	n	-----	-----	-----	-----
2938	<0.05	0.02016	0.07917	0.004158	0.0568
3172	< 0.2	< 1	< 10	< 0.1	< 0.5
3185	<0.010	<0.010	<0.010	<0.010	<0.010
3218	n	-----	-----	-----	-----
3237	n	-----	-----	-----	-----

Details on final concentration, surface area and volume of simulant reported for Cobalt on sample #20681

lab	surface area in dm^2	volume simulant in ml	surface to volume ratio in dm^2/100 ml	final conc. in simulant in mg/l	reported Specific Migration in mg/dm^2	iis calculated Specific Migration in mg/dm^2	Difference absolute
310	1.6038	241	0.665477	1.4144	0.2122	0.21254	0.00034
551	1.654	246	0.672358	0.3	0.1818	0.04462	-0.13718
2129	1.7	250	0.68	1.59	0.23	0.23382	0.00382
2146	-----	-----	-----	-----	-----	-----	-----
2159	1.63	250	0.652	0.336	0.052	0.05153	-0.00047
2213	1.82	250	0.728	1.769	0.243	0.24299	-0.00001
2271	1.61	240	0.670833	1.461	0.2178	0.21779	-0.00001
2366	1.56	240	0.65	1.586	0.244	0.24400	0.00000
2375	1.65	250	0.66	1.3346	0.222	0.20221	-0.00198
2385	1.64	235	0.697872	1.4	0.2	0.20061	0.00061
2415	1.263	234	0.539744	1.319	0.244	0.24438	0.00038
2495	1.53	200	0.765	1.418574	0.185435	0.18543	0.00000
2510	1.653	210	0.787143	1.693	0.2151	0.21508	-0.00002
2609	1.6126	250	0.64504	1.566	0.243	0.24278	-0.00022
2634	0.84	100	0.84	2.01	0.24	0.23929	-0.00071
2749	1.6286	236.766	0.687852	1.5595	0.22672	0.22672	0.00000
2799	1.029	50	2.058	208.25	-----	-----	-----
2840	-----	220	-----	-----	-----	-----	-----
2897	1.75	250	0.7	2.011	0.287	0.28729	0.00029
2901	1.679	240	0.699583	1.3346	0.1908	0.19077	-0.00003
2928	-----	-----	-----	-----	-----	-----	-----
2938	1.53	200	0.765	0.9775	0.13007	0.12778	-0.00229
3172	1.71	250	0.684	1.736	0.254	0.25380	-0.00020
3185	1.57	250	0.628	1.44	0.2293	0.22930	0.00000
3218	-----	-----	-----	-----	-----	-----	-----
3237	1.99	250	0.796	0.06	0.15	0.00754	-0.14246

Details on final concentration, surface area and volume of simulant reported for Lithium on sample #20681

lab	surface area in dm^2	volume simulant in ml	surface to volume ratio in dm^2/100 ml	final conc. in simulant in mg/l	reported Specific Migration in mg/dm^2	iis calculated Specific Migration in mg/dm^2	Difference absolute
310	1.6038	241	0.665477	2.0900	0.3135	0.31406	0.00056
551	1.654	246	0.672358	-----	-----	-----	-----
2129	1.7	250	0.68	2.3800	0.35	0.35000	0.00000
2146	-----	-----	-----	-----	-----	-----	-----
2159	1.63	250	0.652	0.4460	0.068	0.06840	0.00040
2213	1.82	250	0.728	2.3730	0.326	0.32596	-0.00004
2271	1.61	240	0.670833	2.2620	0.3372	0.33719	-0.00001
2366	1.56	240	0.65	2.4580	0.378	0.37815	0.00015
2375	1.65	250	0.66	1.9000	0.316	0.28788	-0.02812
2385	1.64	235	0.697872	1.7500	0.2507	0.25076	0.00006
2415	1.263	234	0.539744	2.0110	0.373	0.37258	-0.00042
2495	1.53	200	0.765	1.9606	0.25629	0.25629	0.00000
2510	1.653	210	0.787143	2.4191	0.3073	0.30733	0.00003
2609	1.6126	250	0.64504	2.4270	0.376	0.37626	0.00026
2634	0.84	100	0.84	2.8400	0.34	0.33810	-0.00190
2749	1.6286	236.766	0.687852	2.4306	0.353358	0.35336	0.00000
2799	1.029	50	2.058	-----	-----	-----	-----
2840	-----	220	-----	-----	-----	-----	-----
2897	1.75	250	0.7	2.7510	0.393	0.39300	0.00000
2901	1.679	240	0.699583	1.9458	0.2781	0.27814	0.00004
2928	-----	-----	-----	-----	-----	-----	-----
2938	1.53	200	0.765	1.2825	0.170658	0.16765	-0.00301
3172	1.71	250	0.684	2.4170	0.353	0.35336	0.00036
3185	1.57	250	0.628	2.0370	0.3244	0.32436	-0.00004
3218	-----	-----	-----	-----	-----	-----	-----
3237	1.99	250	0.796	0.0730	0.1848	0.00917	-0.17563

APPENDIX 3 Analytical details

Sample #20680

lab	accredited acc. ISO/IEC 17025	sample cleaned prior to the migration step	simulant preheated to 70°C	Equipment used	Sample sealed during test
310	No	No	Yes	Oven	Yes, plastic foil
551	Yes	No	Yes	Oven	Yes, with aluminum seal
2129	---	---	---	---	---
2146	Yes	Yes, with brush	Yes	Incubator	Yes, with glass plate
2159	No	No	Yes	Oven	Yes, with glass plate
2213	No	Yes, with dest. water	Yes	Oven	No
2271	Yes	Yes, with lint free cloth	Yes	Oven	Yes, tested in an airtight container
2366	No	No	Yes	Oven	Yes, with film
2375	Yes	No	Yes	Oven	Yes, with aluminum seal
2385	Yes	Yes, with water	Yes	Oven	Yes, with glass plate
2415	Yes	No	Yes	Oven	Yes, with film
2495	Yes	Yes, with cold water and soap	Yes	Oven	Yes, with glass plate
2510	Yes	No	Yes	Oven	Yes, with aluminum seal
2609	Yes	No	Yes	Water bath	Yes, with aluminum seal
2634	Yes	Yes, with water	No	Oven	Yes, with glass plate
2749	---	---	---	---	---
2799	No	No	No	Oven	Yes, tested in an airtight container
2840	---	---	---	---	---
2897	Yes	Yes, with water	Yes	Oven	Yes, with glass
2901	---	---	---	---	---
2928	No	Yes, with dest. water	Yes	Incubator	Yes, with watch glass
2938	No	No	No	Oven	No
3172	---	---	---	---	---
3185	Yes	Yes, with dest. water	Yes	Oven	Yes, with glass board
3218	---	---	---	---	---
3237	Yes	Yes, with dest. water	Yes	Oven	Yes, with film

Sample #20681

lab	accredited acc. ISO/IEC 17025	sample cleaned prior to the migration step	simulant preheated to 70°C	Equipment used	Sample sealed during test
310	No	No	Yes	Oven	Yes, plastic foil
551	Yes	No	Yes	Oven	Yes, plastic film
2129	Yes	No	Yes	Oven	Yes, with aluminum seal
2146	---	---	---	---	---
2159	No	No	Yes	Oven	Yes, with glass plate
2213	Yes	Yes, with water	Yes	Oven	No
2271	Yes	Yes, with lint free cloth	Yes	Oven	Yes, tested in an airtight container
2366	Yes	No	Yes	Oven	Yes, plastic film
2375	Yes	No	Yes	Oven	Yes, with aluminum seal
2385	Yes	Yes, with water	Yes	Oven	Yes, with glass plate
2415	Yes	No	Yes	Oven	Yes, with film
2495	---	---	---	---	---
2510	Yes	No	Yes	Oven	Yes, with aluminum seal
2609	Yes	No	Yes	Incubator	Yes, with aluminum seal
2634	Yes	Yes, with water	No	Oven	Yes, with glass plate
2749	No	No	Yes	Water bath	Yes, with watch glass
2799	No	No	No	Oven	Yes, tested in an airtight container
2840	No	Yes, with water	Yes	Oven	Yes, tested in an airtight container
2897	Yes	Yes,	Yes	Oven	Yes, with glass
2901	No	No	Yes	Oven	Yes, with glass
2928	---	---	---	---	---
2938	No	No	No	Oven	No
3172	---	---	---	---	---
3185	Yes	Yes, with dest. water	Yes	Oven	Yes, with glass board
3218	---	---	---	---	---
3237	Yes	No	Yes	Oven	Yes, with plastic wrap

APPENDIX 4

Number of participating laboratories per country

3 labs in	BRAZIL
1 lab in	FINLAND
2 labs in	GERMANY
1 lab in	INDIA
1 lab in	IRELAND
1 lab in	ISRAEL
3 labs in	ITALY
5 labs in	P.R. of CHINA
1 lab in	SOUTH KOREA
1 lab in	SWITZERLAND
1 lab in	THE NETHERLANDS
3 labs in	TURKEY
2 labs in	UNITED ARAB EMIRATES
1 lab in	VIETNAM

APPENDIX 5

Abbreviations

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

Literature

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 EN13130-1 Materials and articles in contact with foodstuffs – Plastics substances subject to limitation
3 Commission regulation (EU) No 1935/2004 of 27 October 2004, on materials and articles intended to come into contact with food
4 Commission regulation (EU) No 10/2011 of 14 January 2011 on plastic materials and articles intended to come into contact with food
5 ASTM E178:02
6 ASTM E1301:03
7 ISO13528:05
8 ISO5725:86
9 ISO5725, parts 1-6, 1994
10 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
11 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
12 IP367/96
13 DIN38402 T41/42
14 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
15 J.N. Miller, Analyst, 118, 455, (1993)
16 Analytical Methods Committee Technical Brief, No 4, January 2001
17 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364 (2002)
18 R.G. Visser, Accred Qual Assur, 14,29-34, (2009)
19 Bernard Rosner, Technometrics, 25(2), 165-172, (1983)
20 White Paper on the determinations of Overall and Specific Migration on food contact materials, February 2018, www.iisnl.com