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

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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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 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 scheme for food contact materials every year. During the annual proficiency testing program 2021/2022 it was decided to continue the proficiency test for the determination of Specific Migration on Food Contact Materials.

In this interlaboratory study 21 laboratories in 14 different countries registered for participation. See appendix 5 for the number of participants per country. In this report the results of the Specific Migration proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send two different samples. The first sample was a cup labelled #21721 which was positive on 4,4'-Diaminodiphenylmethane or 4,4'-Methylenedianiline (MDA). The second sample was a plate labelled #21722 which was positive on some metals.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

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

2.2 PROTOCOL

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

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

For the first sample a batch of beige colored Acrylonitrile Butadiene Styrene (ABS) cups positive on MDA was prepared by a third party. The subsamples were labelled #21721. The homogeneity of the subsamples was checked by determination of the Specific Migration of MDA by an inhouse test method on 8 stratified randomly selected subsamples. Migration conditions: 1st step, 20% Ethanol, 200 mL, 2 hours at 70°C.

	MDA in mg/dm^2
sample #21721-1	0.017
sample #21721-2	0.015
sample #21721-3	0.014
sample #21721-4	0.016
sample #21721-5	0.017
sample #21721-6	0.016
sample #21721-7	0.015
sample #21721-8	0.016

Table 1: homogeneity test results of subsamples #21721

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.

	MDA in mg/dm^2
r(observed)	0.002
reference method	Horwitz
0.3 x R (reference method)	0.003

Table 2: evaluation of the repeatability of subsamples #21721

The calculated repeatability is 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 polypropylene plates positive on heavy metals as Cobalt, Copper and Zinc was selected. The subsamples were labelled #21722.

The batch for sample #21722 was used in a previous proficiency test on Specific Migration as sample #16620 in iis16P11SM. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one cup labelled #21721 and one plate labelled #21722 were sent on September 9, 2021.

2.5 ANALYZES

The participants were requested to determine MDA on sample #21721 using the prescribed test conditions (article filling, repeated use, 2 hours at 70°C and 20% Ethanol as simulant). For sample #21722 it was requested to determine: Aluminum, Barium, Cobalt, Copper, Iron, Lithium, Manganese, Nickel and Zinc using the prescribed conditions (total immersion, single use, 2 hours 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 test results cannot be used for meaningful statistical evaluations.

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

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per sample and per determination in appendices 1, 2 and 3 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 data analysis and the original test results are placed under 'Remarks' in the result tables in appendices 1 and 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.

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

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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

3.2 GRAPHICS

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

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

Furthermore, Kernel Density Graphs were made. This 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

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

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-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 in appendix 1.

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

	|z|	< 1	good
1 <	|z|	< 2	satisfactory
2 <	|z|	< 3	questionable
3 <	|z|		unsatisfactory

4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples. For sample #21721 twelve participants did not report any test results and for sample #21722 two participants did not report any test results.
Finally, in total 19 participants reported 117 numerical test results in mg/dm^2. Observed were 6 outlying test results, which is 5.1%.
In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Not all data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section the reported test 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 original data. The abbreviations, used in these tables, are explained in appendix 6.

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 observed that the Overall and Specific Migration methods, limits and calculations are mixed up 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. 14) to help participants understand the differences between the two migration 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. Method EN13130-1 describes how the Specific Migration test should be performed. Regretfully no reference test method is available with precision requirements for the migration of MDA 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 #21721

MDA: This determination may be problematic for all three steps. In total three statistical outliers were observed over three migration steps. The calculated reproducibility of each migration step after rejection of statistical outliers is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

Sample #21722

Aluminum: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is full in agreement with the estimated reproducibility calculated with the Horwitz equation.

Barium: This determination may be very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the estimated reproducibility calculated with the Horwitz equation.

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

Copper: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the estimated reproducibility calculated with the Horwitz equation.

Zinc: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the estimated reproducibility calculated with the Horwitz equation.

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 FOR THE GROUP OF LABORATORIES

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

Component	unit	n	average	2.8 * sd	R(target)
MDA – step 1	mg/dm^2	8	0.008	0.015	0.007
MDA – step 2	mg/dm^2	8	0.004	0.010	0.005
MDA – step 3	mg/dm^2	8	0.004	0.009	0.004

Table 3: Reproducibilities of components on sample #21721

Component	unit	n	average	2.8 * sd	R(target)
Aluminum	mg/dm^2	18	0.059	0.038	0.041
Barium	mg/dm^2	17	0.323	0.395	0.172
Cobalt	mg/dm^2	17	0.043	0.029	0.031
Copper	mg/dm^2	18	0.316	0.179	0.168
Zinc	mg/dm^2	17	0.617	0.313	0.297

Table 4: Reproducibilities of components on sample #21722

Without further statistical calculations it can be concluded that for MDA 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 the metals present in sample #21722 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 2021 WITH PREVIOUS PTS

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	20 – 140	3 – 0.2
2013	Formaldehyde	article filling	41 – 61	14 – 20	3 – 0.2
2014	Bisphenol-A	total immersion	44 – 52	14 – 20	3 – 0.2
2015	DEHP	total immersion	34 – 40	14 – 20	3 – 0.2
2016	Metals	total immersion	29 – 30	14 – 20	3 – 0.2
2017	Bisphenol-A	article filling	33 – 50	20 – 33	0.2 – 0.009
2018	Metals	article filling	21 – 35	17 – 38	0.6 – 0.003
2019	DEHP/DAP	article filling	24 – 34	19 – 20	0.34 – 0.20
2020	Formaldehyde	article filling	28 – 51	17 – 19	0.62 – 0.32
	Metals	article filling	17 – 20	19 – 20	0.32 – 0.22
2021	MDA	article filling	70 – 84	41 – 82	0.008 – 0.004
	Metals	total immersion	18 – 44	17 – 26	0.6 – 0.04

Table 5: 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 4. Seventeen of the reporting laboratories are accredited for the determination of the Specific Migration for both MDA and Metals.

Twelve of the reporting participants mentioned to have used test method EN13130-1 for the Specific Migration of MDA and Metals, the others used in house methods.
For sample #21721 three participants reported to clean the sample before the determination of the Specific Migration. Two participants reported to clean the cup with lint-free cloth. One participant reported to clean the cup with warm water, which is not in line with test method EN13130-1 paragraph 15.5. All the reporting participants preheated the simulant solution to 70°C.

For sample #21722 six participants reported to clean the sample for the determination of Specific Migration. All of these participants reported to clean the cup with lint-free cloth. All but one of the participants preheated the simulant solution to 70°C.
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 above details have shown an apparent influence on the final results in this PT.

5 DISCUSSION

The limits for specific migration for Metals are mentioned in mg/kg food or food simulant. 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 tables.

Component	Specific Migration Detection Limit in mg/kg food or food simulant	Specific Migration Detection Limit in mg/dm^2
MDA	0.01	0.0017

Table 6: Specific Migration maximum limits according to 10/2011/EU

Component	Specific Migration Limit in mg/kg food or food simulant	Specific Migration Limit in mg/dm^2
Aluminum	1	0.167
Barium	1	0.167
Cobalt	0.05	0.008
Copper	5	0.833
Zinc	5	0.833

Table 7: Specific Migration maximum limits according to 10/2011/EU

Six reporting laboratories would reject sample #21721 for containing too much MDA in the third step, while three would accept the sample.
All reporting laboratories would reject sample #21722 for containing too much Barium and/or Cobalt.
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 #21721, only one participant reported higher results in step 3 than the step before. The other eight 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.

Sample #21722 was used earlier as sample #16620 in iis16P11SM (2016). In table 8 a comparison is given.

	Sample #21722				Sample #16620			
	unit	n	average	R(calc)	unit	n	average	R(calc)
Barium	mg/dm^2	17	0.32	0.40	mg/dm^2	24	0.29	0.29
Cobalt	mg/dm^2	17	0.04	0.03	mg/dm^2	23	0.05	0.03
Copper	mg/dm^2	18	0.32	0.18	mg/dm^2	24	0.33	0.26
Zinc	mg/dm^2	17	0.62	0.31	mg/dm^2	24	0.58	0.48

Table 8: comparison of sample #21722 with #16620

It is observed that the group in this PT performed in line with the previous determination of these metals with smaller variation for Copper and Zinc.

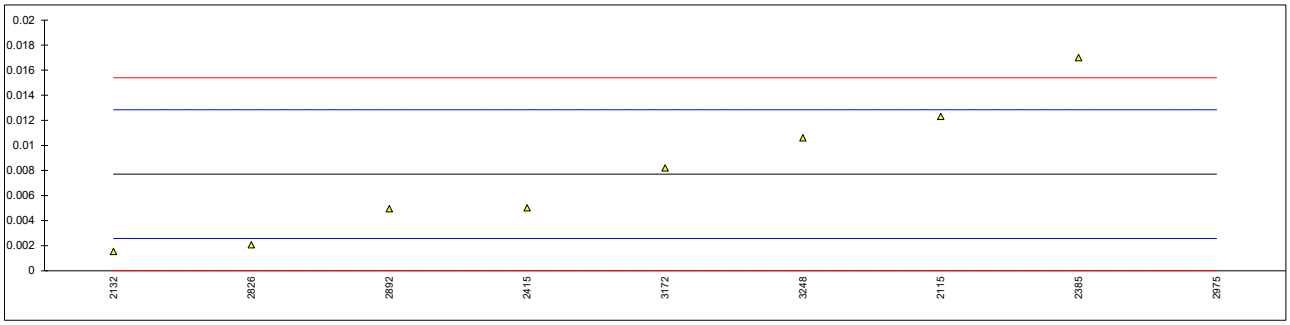
6 CONCLUSION

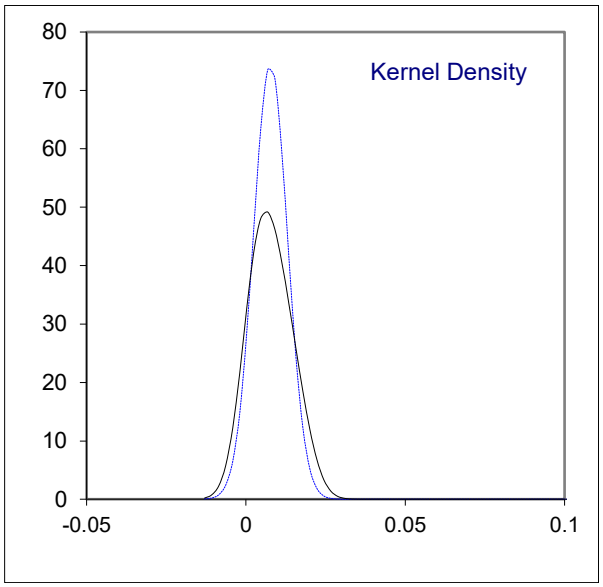
Each laboratory should evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and the quality of the analytical results.

APPENDIX 1

Determination of 1st Specific Migration of MDA on sample #21721; results in mg/dm^2 per contact surface

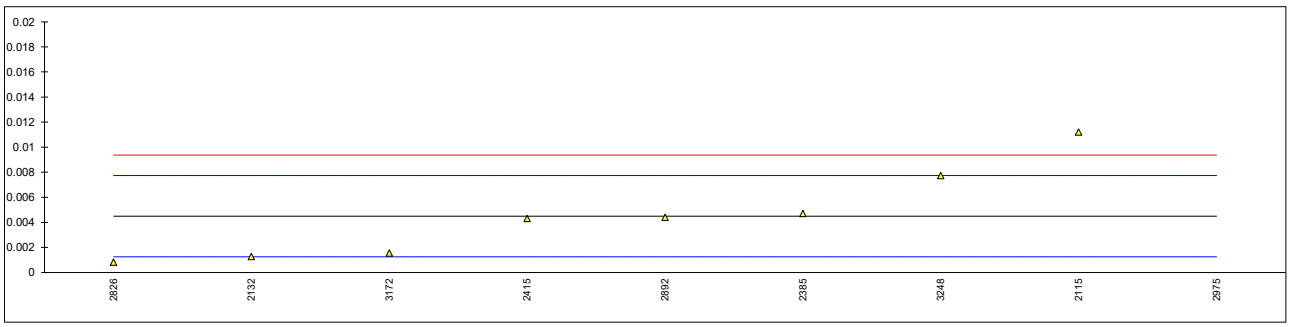
lab	method	value	mark	z(targ)	remarks
310		-----		-----	
2115	EN13130-1	0.0123		1.79	
2132	In house	0.00153		-2.41	
2366		-----		-----	
2375		-----		-----	
2379		-----		-----	
2384		-----		-----	
2385	In house	0.017		3.62	
2415	EN13130-1	0.00502		-1.05	
2425		-----		-----	
2475		-----		-----	
2525		-----		-----	
2549		-----		-----	
2826	EN13130-1	0.0020712		-2.20	
2892	EN13130-1	0.004937		-1.08	
2896		-----		-----	
2936		-----		-----	
2958		-----		-----	
2975	EN13130-1	0.534	C,G(0.01), E	205.22	first reported: 0.1886, calculation difference, iis calc: 0.424
3172	EUR24815 EN2011	0.00819		0.19	
3248	EN13130-1	0.0106	C	1.13	first reported: 0.0462
	normality	OK			
	n	8			
	outliers	1			
	mean (n)	0.007706			
	st.dev. (n)	0.0053568	RSD = 70%		
	R(calc.)	0.014999			
	st.dev.(Horwitz)	0.0025646			
	R(Horwitz)	0.007181			

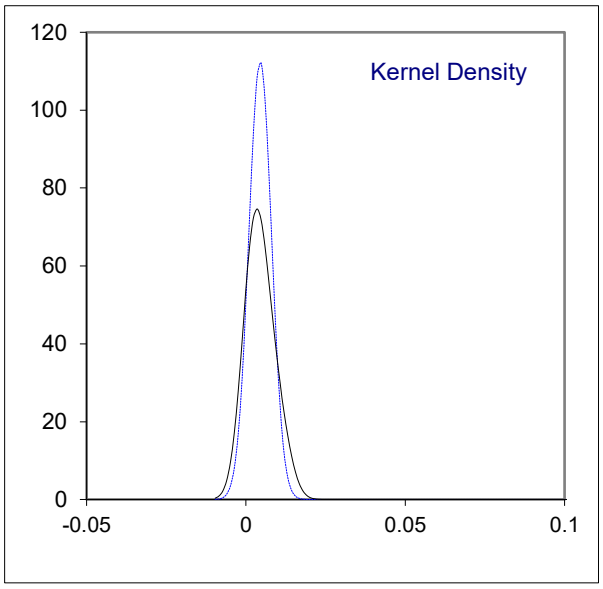




Determination of 2nd Specific Migration of MDA on sample #21721; results in mg/dm^2 per contact surface

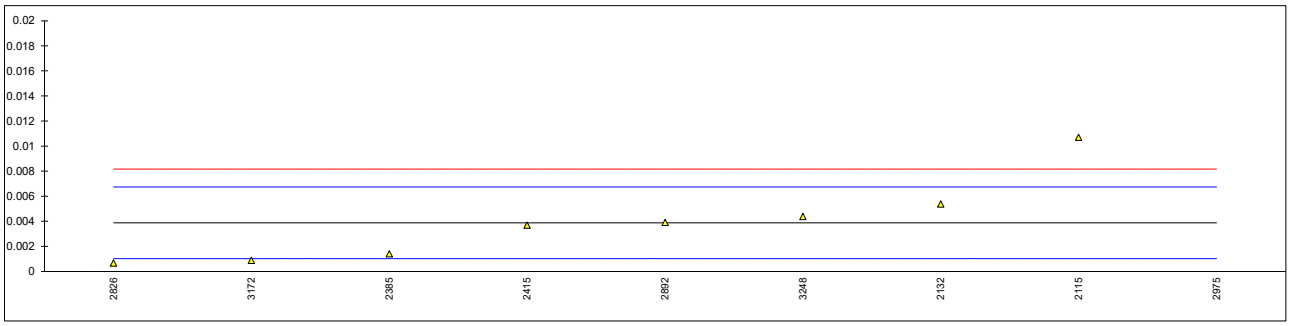
lab	method	value	mark	z(targ)	remarks
310		-----		-----	
2115	EN13130-1	0.0112		4.13	
2132	In house	0.00127		-1.99	
2366		-----		-----	
2375		-----		-----	
2379		-----		-----	
2384		-----		-----	
2385	In house	0.0047		0.13	
2415	EN13130-1	0.00430		-0.12	
2425		-----		-----	
2475		-----		-----	
2525		-----		-----	
2549		-----		-----	
2826	EN13130-1	0.00082335		-2.26	
2892	EN13130-1	0.0044	C	-0.06	first reported: 0.0035
2896		-----		-----	
2936		-----		-----	
2958		-----		-----	
2975	EN13130-1	0.286	C,G(0.01), E	173.50	first reported: 0.1011, calculation difference, iis calc: 0.080
3172	EUR24815 EN2011	0.00155		-1.82	
3248	EN13130-1	0.00772		1.99	
	normality	OK			
	n	8			
	outliers	1			
	mean (n)	0.004495			
	st.dev. (n)	0.0035457	RSD = 79%		
	R(calc.)	0.009928			
	st.dev.(Horwitz)	0.0016225			
	R(Horwitz)	0.004543			

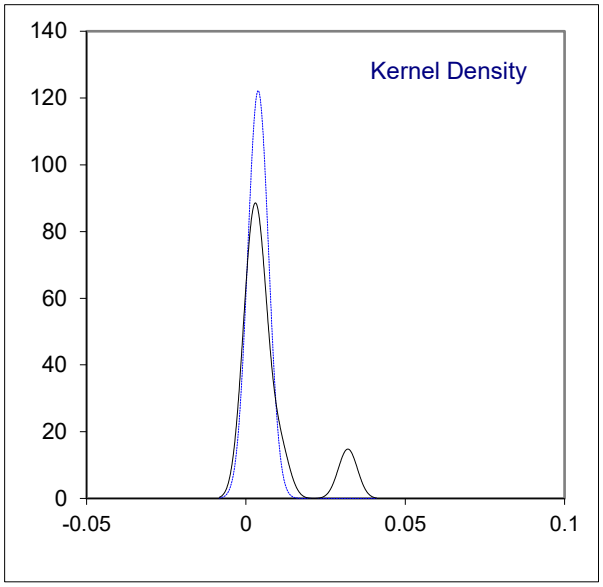




Determination of 3rd Specific Migration of MDA on sample #21721; results in mg/dm^2 per contact surface

lab	method	value	mark	z(targ)	remarks
310		-----		-----	
2115	EN13130-1	0.0107		4.77	
2132	In house	0.00538		1.05	test result of step 3 is higher than the test result of step 2
2366		-----		-----	
2375		-----		-----	
2379		-----		-----	
2384		-----		-----	
2385	In house	0.0014		-1.73	
2415	EN13130-1	0.00369		-0.13	
2425		-----		-----	
2475		-----		-----	
2525		-----		-----	
2549		-----		-----	
2826	EN13130-1	0.00066322		-2.25	
2892	EN13130-1	0.0039	C	0.02	first reported: 0.00309
2896		-----		-----	
2936		-----		-----	
2958		-----		-----	
2975	EN13130-1	0.032	C,G(0.01), E	19.67	first reported: 0.0112, calculation difference, iis calc. 0.025
3172	EUR24815 EN2011	0.000876		-2.10	
3248	EN13130-1	0.00439		0.36	
	normality	not OK			
	n	8			
	outliers	1			
	mean (n)	0.003875			
	st.dev. (n)	0.0032641	RSD = 84%		
	R(calc.)	0.009140			
	st.dev.(Horwitz)	0.0014302			
	R(Horwitz)	0.004004			



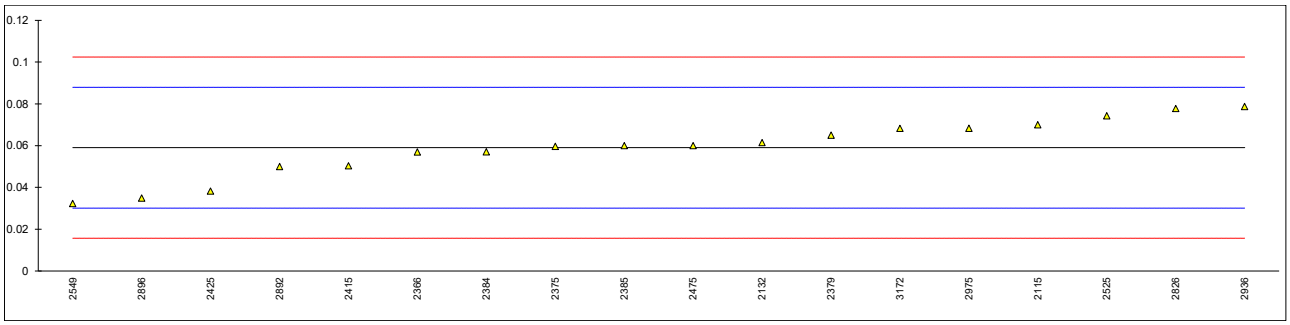


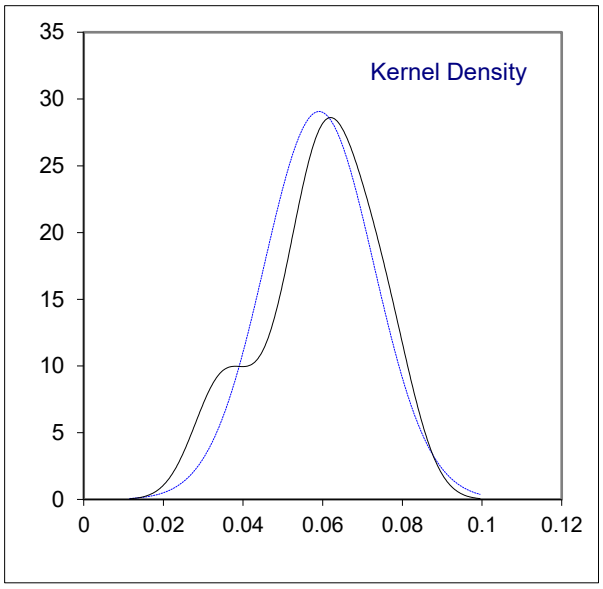
Determination of Specific Migration of Aluminum as Al on sample #21722; results in mg/dm^2 per contact surface

lab	method	value	mark	z(targ)	remarks
310		-----		-----	
2115	EN13130-1	0.07		0.76	
2132	In house	0.06141		0.16	
2366	EN13130-1	0.0569		-0.15	
2375	EN13130-1	0.0596		0.04	
2379		0.065		0.41	
2384	EN13130-1	0.0570		-0.14	
2385		0.0600		0.07	
2415	EN13130-1	0.0503	C	-0.60	first reported: 50.33
2425	EN13130-1	0.0382		-1.44	
2475	EN13130-1	0.060		0.07	
2525	EN13130-1	0.07429		1.05	
2549	EN13130-1	0.0323		-1.85	
2826	EN13130-1	0.077745		1.29	
2892	EN13130-1	0.050	C	-0.63	first reported: 0.01007
2896	In house	0.0349		-1.67	
2936	In house	0.07869		1.36	
2958		-----		-----	
2975	EN13130-1	0.0683	E	0.64	calculation difference, iis calculated: 0.0409
3172		0.06825		0.64	
3248		-----		-----	

normality	OK
n	18
outliers	0
mean (n)	0.05905
st.dev. (n)	0.013723
R(calc.)	0.03842
st.dev.(Horwitz)	0.014464
R(Horwitz)	0.04050

RSD = 23%



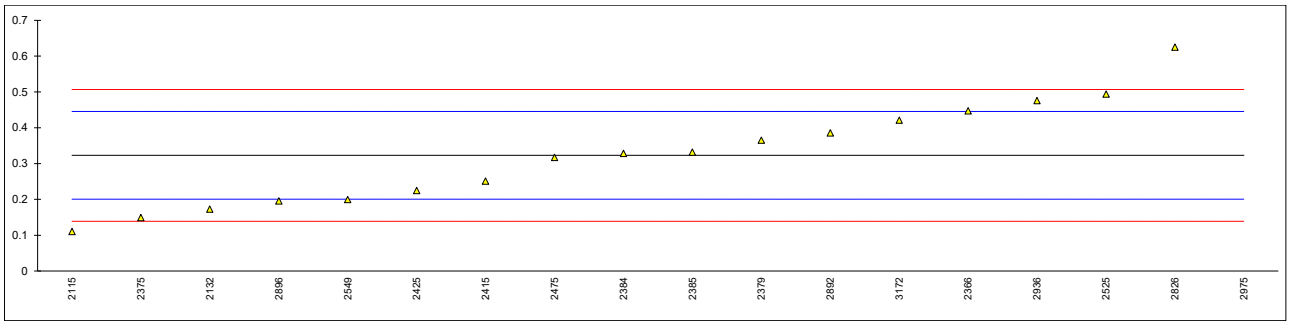


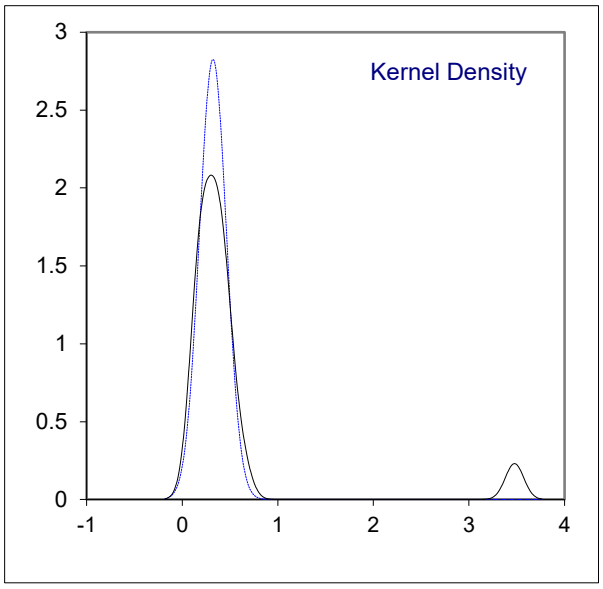
Determination of Specific Migration of Barium as Ba on sample #21722; results in mg/dm^2 per contact surface

lab	method	value	mark	z(targ)	remarks
310		-----		-----	
2115	EN13130-1	0.11		-3.48	
2132	In house	0.1725	C	-2.46	first reported: 0.003451
2366	EN13130-1	0.447		2.02	
2375	EN13130-1	0.149		-2.84	
2379		0.365		0.69	
2384	EN13130-1	0.3282		0.08	
2385		0.3317		0.14	
2415	EN13130-1	0.2510	C	-1.18	first reported: 250.95
2425	EN13130-1	0.2245		-1.61	
2475	EN13130-1	0.317		-0.10	
2525	EN13130-1	0.49393		2.79	
2549	EN13130-1	0.1994		-2.02	
2826	EN13130-1	0.62497		4.93	
2892	EN13130-1	0.38527		1.02	
2896	In house	0.19524		-2.09	
2936	In house	0.4755		2.49	
2958		-----		-----	
2975	EN13130-1	3.476	G(0.01),E	51.47	calculation difference, iis calculated: 2.080
3172		0.4209		1.60	
3248		-----		-----	

normality	OK
n	17
outliers	1
mean (n)	0.32301
st.dev. (n)	0.141240
R(calc.)	0.39547
st.dev.(Horwitz)	0.061264
R(Horwitz)	0.17154

RSD = 44%



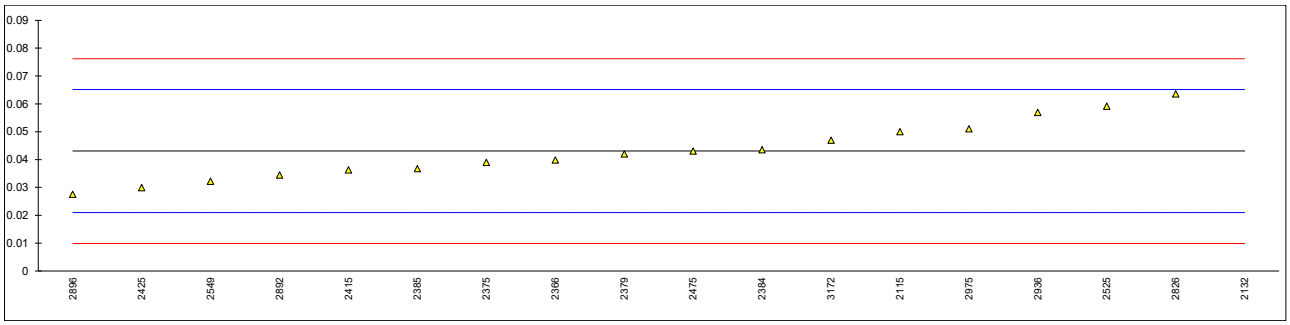


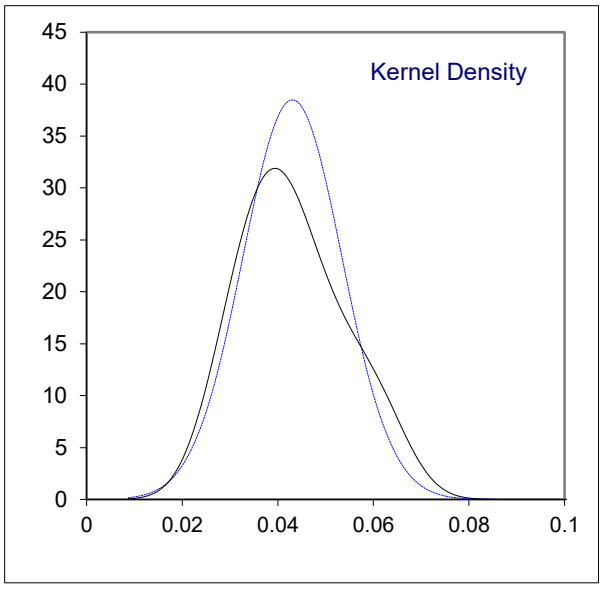
Determination of Specific Migration of Cobalt as Co on sample #21722; results in mg/dm^2 per contact surface

lab	method	value	mark	z(targ)	remarks
310		-----		-----	
2115	EN13130-1	0.05		0.63	
2132	In house	0.2979	C,G(0.01)	23.04	first reported: 0.005959
2366	EN13130-1	0.0398		-0.29	
2375	EN13130-1	0.039		-0.37	
2379		0.042		-0.10	
2384	EN13130-1	0.0435		0.04	
2385		0.0367		-0.57	
2415	EN13130-1	0.0363	C	-0.61	first reported: 36.33
2425	EN13130-1	0.0299		-1.19	
2475	EN13130-1	0.043		0.00	
2525	EN13130-1	0.05911		1.45	
2549	EN13130-1	0.0322		-0.98	
2826	EN13130-1	0.063614		1.86	
2892	EN13130-1	0.03443		-0.78	
2896	In house	0.02750		-1.41	
2936	In house	0.0569		1.25	
2958		-----		-----	
2975	EN13130-1	0.0510	E	0.72	calculation difference, iis calculated: 0.0305
3172		0.04696		0.35	
3248		-----		-----	

normality	OK
n	17
outliers	1
mean (n)	0.04305
st.dev. (n)	0.010369
R(calc.)	0.02903
st.dev.(Horwitz)	0.011059
R(Horwitz)	0.03097

RSD = 24%



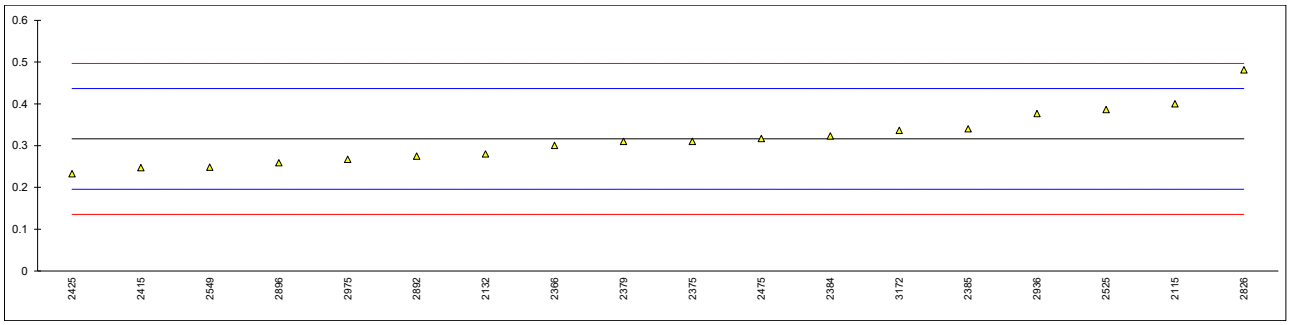


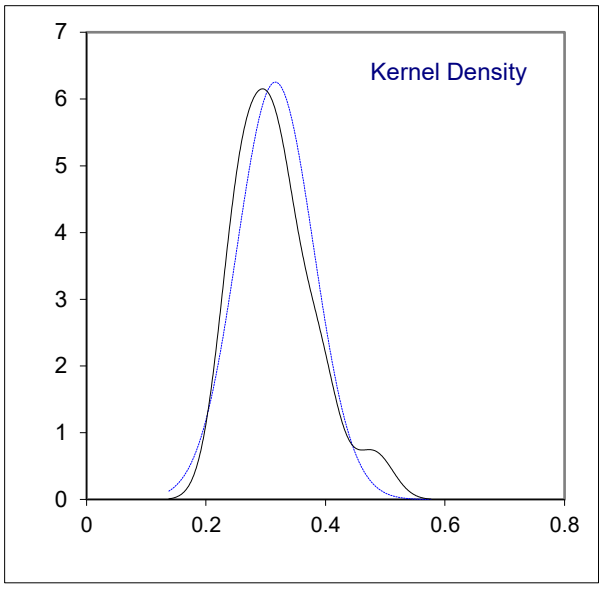
Determination of Specific Migration of Copper as Cu on sample #21722; results in mg/dm^2 per contact surface

lab	method	value	mark	z(targ)	remarks
310		-----		-----	
2115	EN13130-1	0.4		1.39	
2132	In house	0.2799	C	-0.60	first reported: 0.005599
2366	EN13130-1	0.3007		-0.26	
2375	EN13130-1	0.310		-0.10	
2379		0.310		-0.10	
2384	EN13130-1	0.3232		0.12	
2385		0.3400		0.40	
2415	EN13130-1	0.2472	C	-1.15	first reported: 247.20
2425	EN13130-1	0.2328		-1.39	
2475	EN13130-1	0.317		0.01	
2525	EN13130-1	0.38629		1.17	
2549	EN13130-1	0.2483		-1.13	
2826	EN13130-1	0.48137		2.75	
2892	EN13130-1	0.27477		-0.69	
2896	In house	0.25908		-0.95	
2936	In house	0.3767		1.01	
2958		-----		-----	
2975	EN13130-1	0.2672	E	-0.81	calculation difference, iis calculated:0.1599
3172		0.3366		0.34	
3248		-----		-----	

	normality	not OK
	n	18
	outliers	0
	mean (n)	0.31617
	st.dev. (n)	0.063824
	R(calc.)	0.17871
	st.dev.(Horwitz)	0.060161
	R(Horwitz)	0.16845

RSD = 20%



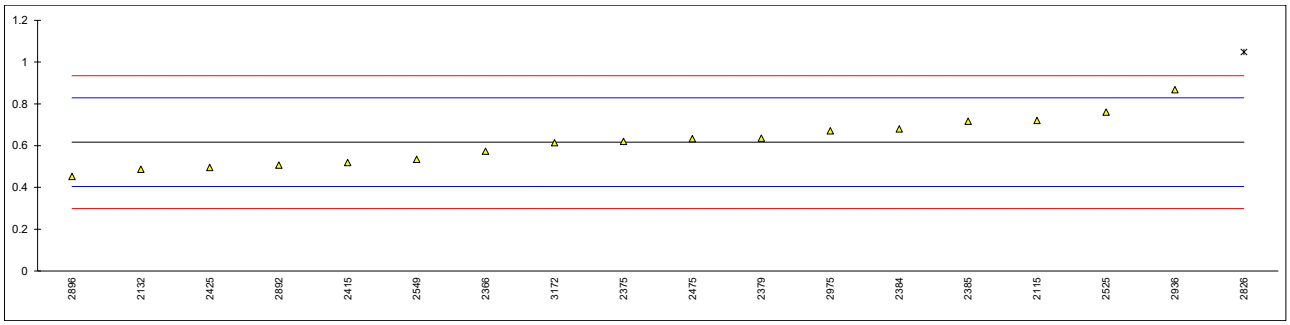


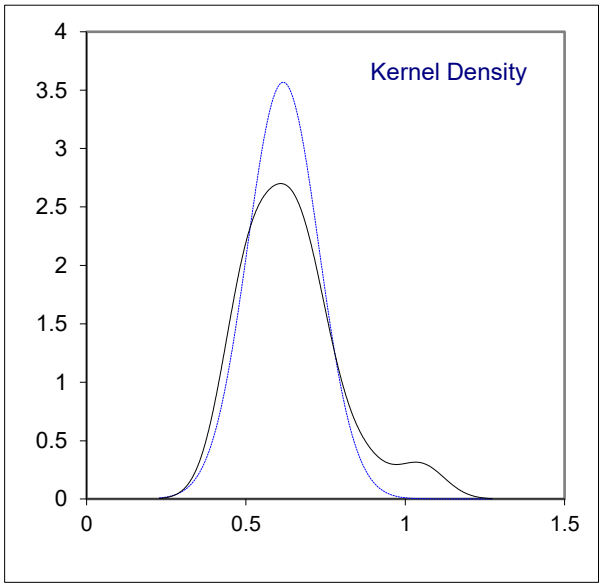
Determination of Specific Migration of Zinc as Zn on sample #21722; results in mg/dm^2 per contact surface

lab	method	value	mark	z(targ)	remarks
310		-----		-----	
2115	EN13130-1	0.72		0.97	
2132	In house	0.4867	C	-1.23	first reported: 0.009733
2366	EN13130-1	0.573		-0.41	
2375	EN13130-1	0.62	C	0.03	first reported: 0.062
2379		0.635		0.17	
2384	EN13130-1	0.6795		0.59	
2385		0.7167		0.94	
2415	EN13130-1	0.5193	C	-0.92	first reported: 519.62
2425	EN13130-1	0.4955		-1.14	
2475	EN13130-1	0.633		0.15	
2525	EN13130-1	0.76006		1.35	
2549	EN13130-1	0.5346		-0.77	
2826	EN13130-1	1.0481	G(0.05)	4.06	
2892	EN13130-1	0.5071		-1.03	
2896	In house	0.45273		-1.55	
2936	In house	0.8672		2.36	
2958		-----		-----	
2975	EN13130-1	0.6712	E	0.51	calculation difference, iis calculated: 0.4016
3172		0.6138		-0.03	
3248		-----		-----	

	normality	OK
	n	17
	outliers	1
	mean (n)	0.61679
	st.dev. (n)	0.111840
	R(calc.)	0.31315
	st.dev.(Horwitz)	0.106131
	R(Horwitz)	0.29717

RSD = 18%





APPENDIX 2

Determination of Specific Migration of other elements on sample #21722; results in mg/dm^2

lab	Iron (Fe)	Lithium (Li)	Manganese (Mn)	Nickel (Ni)
310	-----	-----	-----	-----
2115	-----	-----	-----	-----
2132	0.006889	0.000051	0.000523	0.001326
2366	<0.25	<0.5	<0.25	<0.01
2375	0.149	ND	ND	ND
2379	Not detected	Not detected	Not detected	Not detected
2384	not detected	not detected	not detected	not detected
2385	0.009	<0.005	<0.005	<0.005
2415	-----	-----	-----	-----
2425	Not Detected	Not Detected	Not Detected	Not Detected
2475	<0.01	<0.01	<0.01	<0.01
2525	0.0631	0.0020	not detected	not detected
2549	Not Detected	Not Detected	Not Detected	Not Detected
2826	0.010678	Not detected	Not detected	Not detected
2892	Not detected	Not detected	Not detected	Not detected
2896	NOT ANALYZED	NOT ANALYZED	NOT ANALYZED	NOT ANALYZED
2936	<0.5	<0.1	<0.1	-----
2958	-----	-----	-----	-----
2975	BLQ	BLQ	BLQ	BLQ
3172	< 10	< 0.1	< 0.1	< 0.01
3248	0.132	ND	ND	ND

APPENDIX 3 Details on reported intermediate test results

sample #21721 - surface area, volume of simulant and final concentration reported per step

	surface area (dm^2)	volume simulant (mL)	Final conc. (mg/L)	surface area (dm^2)	volume simulant (mL)	Final conc. (mg/L)	surface area (dm^2)	volume simulant (mL)	Final conc. (mg/L)
lab	1st step			2nd step			3rd step		
310	-----	-----	-----	-----	-----	-----	-----	-----	-----
2115	1.56	240	0.0798	1.56	240	0.0728	1.56	240	0.0697
2132	1.60	250	0.009758	1.60	250	0.0081399	1.60	250	0.034459
2366	-----	-----	-----	-----	-----	-----	-----	-----	-----
2375	-----	-----	-----	-----	-----	-----	-----	-----	-----
2379	-----	-----	-----	-----	-----	-----	-----	-----	-----
2384	-----	-----	-----	-----	-----	-----	-----	-----	-----
2385	1.78	230	0.134	1.78	230	0.036	1.78	230	0.011
2415	1.426	200	0.0358	1.426	200	0.0307	1.426	200	0.0263
2425	-----	-----	-----	-----	-----	-----	-----	-----	-----
2475	-----	-----	-----	-----	-----	-----	-----	-----	-----
2525	-----	-----	-----	-----	-----	-----	-----	-----	-----
2549	-----	-----	-----	-----	-----	-----	-----	-----	-----
2826	1.6932	240	0.0014612	1.6932	240	0.0058087	1.6932	240	0.0046790
2892	1.6	250	0.0316	1.6	250	0.028 C	1.6 C	250	0.025 C
2896	-----	-----	-----	-----	-----	-----	-----	-----	-----
2936	-----	-----	-----	-----	-----	-----	-----	-----	-----
2958	-----	-----	-----	-----	-----	-----	-----	-----	-----
2975	1.89 C	250	3.206 C	5.36	250	1.719 C	1.89 C	250	0.191 C
3172	1.7672	250	0.05759	1.7672	250	0.01129	1.7672	250	0.00617
3248	1.68	230	0.07763	1.68	230	0.01297	1.68	230	0.00737

Lab 2892 first reported for final concentration 2nd step: 0.0044, for surface area 3rd step: 1.2 and for final concentration 3rd step: 0.0198
Lab 2975 first reported for surface area 1st step: 5.36, for final concentration 1st step: 1.131, for final concentration 2nd step: 0.6067, for surface area 3rd step: 5.36 and for final concentration 3rd step: 0.0673

sample #21722 - surface area, volume of simulant, final concentration, single or double sided and thickness

lab	surface area (dm^2)	volume simulant (mL)	Final conc. (mg/L)	single or double surface	thickness Yes/No
310	-----	-----	-----	-----	-----
2115	1.16	100	1.28	double surface	yes
2132	4.52	752	1.0371	single surface	yes
2366	4.41	740	2.664	double surface	yes
2375	4.51	400	1.681	double surface	yes
2379	0.6	100	2.191	double surface	no
2384	0.6016	100	1.969	double surface	yes
2385	4.50	750	1.99	single surface	-----
2415	0.6533	50	3.279	double surface	yes
2425	4.51	750	1.35	double surface	yes
2475	4.49	748	1.9	double surface	yes
2525	1	166.7	2.9636	double surface	no
2549	4.638	750	1.1961	double surface	yes
2826	0.6419	100	4.0117	double surface	yes
2892	1.2	200	2.3116	double surface	yes
2896	4.5237	400	2.208	double surface	no
2936	2.2126	368.7666	2.84	single surface	no
2958	-----	-----	-----	-----	-----
2975	4.512	450	20.856	double surface	yes
3172	4.5508	758	2.5268	double surface	yes
3248	0.60	100	1.255	double surface	no

APPENDIX 4 Analytical details

Sample #21721

lab	accredited ISO/IEC 17025	sample cleaned prior to the migration step	simulant preheated	Equipment used	Sample sealed during test
310	---	---	---	---	---
2115	Yes	No	Yes	Incubator	Yes, tested in an airtight container
2132	Yes	Yes, with a lint-free cloth	Yes	Oven	Yes, covered with a glass plate and the whole container wrapped with plastic wrap.
2366	---	---	---	---	---
2375	---	---	---	---	---
2379	---	---	---	---	---
2384	---	---	---	---	---
2385	Yes	Yes, cleaned with warm water	Yes	Oven	Yes, tested in an airtight container
2415	Yes	No	Yes	Oven	Yes, tested in an airtight container
2425	---	---	---	---	---
2475	---	---	---	---	---
2525	---	---	---	---	---
2549	---	---	---	---	---
2826	Yes	No	Yes	Oven	Yes, with microwave wrap seal
2892	Yes	Yes, with a lint-free cloth	Yes	Incubator	Yes, Use glass surface for cover
2896	---	---	---	---	---
2936	---	---	---	---	---
2958	---	---	---	---	---
2975	Yes	No	Yes	Incubator	Yes, tested in an airtight container
3172	Yes	No	Yes	Oven	Yes, covered with an MDA free tested plastic food grade film
3248	Yes	No	Yes	Oven	Yes, with aluminum seal

Sample #21722

lab	accredited ISO/IEC 17025	sample cleaned prior to the migration step	simulant preheated	Equipment used	Sample sealed during test
310	---	---	---	---	---
2115	Yes	No	Yes	Incubator	Yes, tested in an airtight container
2132	Yes	Yes, with lint-free cloth	Yes	Oven	Yes, covered with a glass plate and the whole container wrapped with plastic wrap.
2366	---	No	Yes	Oven	Yes, tested in an airtight container
2375	Yes	No	Yes	Oven	Yes, with glass plate
2379	Yes	No	Yes	Oven	Yes, tested in an airtight container
2384	Yes	Yes, with lint free cloth	Yes	Oven	Yes, tested in an airtight container
2385	Yes	No	Yes	Oven	Yes, tested in an airtight container
2415	Yes	Yes, with lint free cloth	Yes	Oven	Yes, tested in an airtight container
2425	Yes	Yes, with lint free cloth	Yes	Oven	No
2475	No	No	Yes	Oven	Yes, with film plastic
2525	Yes	No	Yes	Oven	Yes, tested in an airtight container
2549	Yes	Yes, with lint free cloth	Yes	Oven	No
2826	Yes	No	Yes	Oven	Yes, with microwave wrap seal
2892	Yes	Yes, with lint free cloth	Yes	Oven	Yest, glass surface to cover
2896	Yes	No	Yes	Oven	Yes, covered with another glass container
2936	Yes	No	No	Incubator	Yes, with aluminum seal
2958	---	---	---	---	---
2975	Yes	No	Yes	Oven	Yes, tested in an airtight container
3172	Yes	No	Yes	Oven	Yes, covered with a Metal free tested plastic food grade film
3248	Yes	No	Yes	Incubator	Yes, with aluminum seal

APPENDIX 5

Number of participants per country

1 lab in	BANGLADESH
1 lab in	FRANCE
2 labs in	GERMANY
3 labs in	HONG KONG
2 labs in	INDIA
3 labs in	ITALY
1 lab in	MALAYSIA
1 lab in	P.R. of CHINA
1 lab in	SERBIA
1 lab in	SRI LANKA
1 lab in	THAILAND
1 lab in	THE NETHERLANDS
1 lab in	TURKEY
2 labs in	VIETNAM

APPENDIX 6

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 = calculation difference between reported test result and result calculated by iis
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

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