Results of Proficiency Test
Specific migration (fcm)
September 2018

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis18P10SM

December 2018

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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 (lit. 3) applies to all food contact materials and describes a large number of requirements, e.g. limits for overall migration and specific limits for certain constituents. Article 11 (and Annex II) of this regulation describes the specific migration limit, expressed in mg/kg food or food simulant. It has been recently amended with regulation 1416/2016/EU in which a limit for Aluminum and a lower limit for Zinc is published (lit. 4/5). The limits are summarized in appendix 4.

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 of metals 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. 20) to help participants understand the differences between the two methods, the units used for reporting and the regulated limits.

Since 2012, the Institute of Interlaboratory Studies (iis) organizes a proficiency test scheme for food contact materials. During the annual proficiency testing program 2018/2019, it was decided to continue the proficiency test for the determination of Specific Migration.
In the interlaboratory study of September 2018, 26 laboratories from 15 different countries participated (see appendix 5). In this report, the results of the 2018 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 organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one sample, a cup, labelled #18620, artificially fortified with different metals, and to prescribe a number of test conditions (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/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls in 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 organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

A batch of 40 colourless Polycarbonate (food) cups containing a relevant concentration of different metals was prepared by a third party and labelled #18620. The homogeneity was checked by determination of the specific migration of Cobalt, Copper and Zinc on 8 stratified randomly selected plates.

migration in food simulant: 200 ml, 3% Acetic acid, 120 min at 100°C	Cobalt in mg/dm^2	Copper in mg/dm^2	Zinc in mg/dm^2
Sample #18620-1	0.2245	0.6043	0.4417
Sample #18620-2	0.1942	0.5468	0.4259
Sample #18620-3	0.2101	0.5942	0.4014
Sample #18620-4	0.2216	0.5942	0.4288
Sample #18620-5	0.2273	0.6072	0.4504
Sample #18620-6	0.2259	0.5957	0.4403
Sample #18620-7	0.2029	0.5496	0.4360
Sample #18620-8	0.2273	0.5266	0.4115

Table 1: homogeneity test results on the subsamples #18620

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 ISO 13528, Annex B2 in the next table:

migration in food simulant: 200 ml, 3% acetic Acid, 120 min at 100°C	Cobalt in mg/dm^2	Copper in mg/dm^2	Zinc in mg/dm^2
r(observed)	0.036	0.087	0.046
reference method	Horwitz	Horwitz	Horwitz
0.3 x R (reference method)	0.037	0.084	0.066

Table 2: evaluation of the repeatabilities of subsamples #18620

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

To each of the participating laboratories one sample #18620 was sent on September 5, 2018.

2.5 ANALYSES

The participants were requested to determine the metals Barium as Ba, Cobalt as Co, Copper as Cu, Iron as Fe, Lithium as Li, Manganese as Mn and Zinc as Zn on sample #18620 using the prescribed test conditions (article filling, single use, 2 hrs at 100°C and 3% Acetic acid as simulant).
It was also requested to report some analytical details and if the laboratory was accredited for the components that were determined.

It was explicitly requested to treat the sample as a routine sample and to report the test results using the indicated units on the report form in the data entry portal and not to round the results, but report as much significant figures as possible. 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 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 appropriate reference test methods 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 were 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 component in the appendix 1 of this report. The laboratories are represented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that did not report 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 reanalyses). 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 appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organisation 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.

In accordance to ISO 5725 the original test results per determination were submitted subsequently to Dixon’s, Grubbs’ and 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 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 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. 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 general, when no literature reproducibility is available, another target may be 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 should be done in order to evaluate whether the reported test results are fit-for-purpose.

The z-scores were calculated in accordance with:

$z_{(target)}$ = (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. The usual interpretation of z-scores is as follows:

	$\lvert z \rvert$	< 1	good
1 <	$\lvert z \rvert$	< 2	satisfactory
2 <	$\lvert z \rvert$	< 3	questionable
3 <	$\lvert z \rvert$		unsatisfactory

4 EVALUATION

In this interlaboratory study, no problems were encountered with the dispatch of the samples. No participants reported test results after the final reporting date, but two participants did not report any test results at all. Thus, 24 of the 26 participants submitted test results.
In total over 275 (intermediate) results were reported, of which 137 test results in in mg/dm^2. Three statistical outliers were observed, which is 2.2% of the 137 test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

For the determination of Specific Migration, several standardised 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 metals from food contact materials. Therefore, it was decided to estimate the target reproducibilities from the Horwitz equation.

Not all original 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.

4.1 EVALUATION PER COMPONENT

The test results of the specific migration reported in mg/dm^2 were used for the statistical evaluation. The test results of one laboratory were excluded for three of the six reported test results were outliers.

Barium as Ba: This determination may not be problematic. No statistical outliers were observed, but one test result was excluded. However, the calculated reproducibility after rejection of the suspect data is in agreement with the estimated reproducibility using the Horwitz equation.

Cobalt as Co: This determination may not be problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility using the Horwitz equation.

Copper as Cu: This determination may be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility using the Horwitz equation.

Iron as Fe: This determination may not be problematic. No statistical outliers were observed, but one test result was excluded. However, the calculated reproducibility after rejection of the suspect data is in agreement with the estimated reproducibility using the Horwitz equation.

Lithium as Li: The majority of the participants did not detect this metal and reported a "smaller than" value or "not detected".

Manganese as Mn: This determination may not be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility using the Horwitz equation.

Zinc as Zn: This determination may be problematic. No statistical outliers were observed, but one test result was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the estimated reproducibility using the Horwitz equation.

4.2 PERFORMANCE EVALUATION OF THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant test method and the reproducibility as found for the group of participating laboratories. The target reproducibilities derived from literature standards are compared in the next tables.

Specific Migration	unit	n	average	2.8 * sd	R (Horwitz)
Barium	mg/dm^2	20	0.019	0.014	0.015
Cobalt	mg/dm^2	22	0.18	0.11	0.11
Copper	mg/dm^2	22	0.53	0.34	0.26
Iron	mg/dm^2	21	0.041	0.024	0.030
Lithium	mg/dm^2	17	<0.1	n.a.	n.a.
Manganese	mg/dm^2	17	0.003	0.002	0.003
Zinc	mg/dm^2	22	0.35	0.34	0.18

Table 3: Reproducibilities of tests on sample #18620

Without further statistical calculations, it can be concluded that for many tests there is a good compliance of the group of laboratories with the relevant target reproducibility (see for discussion paragraph 4.1 and 5).

4.3 COMPARISON OF PROFICIENCY TEST OF SEPTEMBER 2018 TO PREVIOUS PROFICIENCY TESTS

The evolution of the uncertainty for Specific Migration in mg/dm^2 as observed in this proficiency scheme and the comparison with the findings in previous rounds is listed in table 5.

	BPA via total immersion	BPA via article filling	Metals via total immersion	Metals via article filling	DEHP via total immersion	Formalde-hyde via article filling	Target (Hor-witz)	Conc range
2012	----	----	----	----	----	41 - 47%	14-20%	0.2 - 3
2013	----	----	----	----	----	41 - 61%	14-20%	0.2 - 3
2014	44 - 52%	----	----	----	----	----	14-20%	0.2 - 3
2015	----	----	----	----	34 - 40%	----	14-20%	0.2 - 3
2016	----	----	29 - 30%	----	----	----	14-20%	0.2 - 3
2017	----	33 - 50%	----	----	----	----	20-33%	0.009 - 0.2
2018	----	----	----	21 - 35%	----	----	17-38%	0.003 - 0.6

Table 4: comparison of the uncertainties in % for Specific Migration in the previous and present PT

From the above table, it is clear that the performance of this PT shows improvement compared to the PTs of the last years. It also shows that the strict requirements, estimated from the Horwitz equation are met.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

Before the start of this PT, it was assumed that a wide range of test results would be reported when the choice of the 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 pre-set conditions were:

Simulant	3% Acetic acid
Exposure time	2 hours (120 min)
Exposure temperature	100°C
Migration method	Article filling, single use

Table 5: test conditions described for this PT

About 63% of the participants reported to have used test method EN13130-1 for the specific migration of metals and 25% of the participants reported to have used an ‘in house’ method. One participant reported to have used EN1186 (which is an Overall migration method) and another two participants did not report a test method at all.

Using the intermediate results in mg/L, the reported surface and used volume of simulant, iis could calculate the test results in mg/dm^2. iis calculated the results for all metals, an example of the calculated results for Copper can be found in appendix 2. One laboratory reported a very small surface area (0.00648 dm^2), probably in a different unit. For two other laboratories, the values that were calculated by iis were different from the reported test results.

One laboratory only reported results in mg/L, with the comment “no results expressed in mg/dm^2 because of not regulated ratio [surface / simulant volume] = 1,63 dm^2 / 250ml by filling” and “usually, we express specific migration results in mg/kg simulant with the regulated ratio 6 dm^2/kg”. This is remarkable, since method EN13130-1, which this laboratory uses for the determination, calculates the Specific migration in mg/dm^2. Also, when using article filling (variable surface to volume ratio), the result in mg/L simulant is not the same as the result in mg/kg food (see White paper on the determination of Overall and Specific migration on food contact materials, lit. 20).

The reported analytical details that were used by the participants are listed in appendix 3. About 42% reported to be accredited for the determination of the specific migration of metals.

About 63% reported not to clean the sample before the determination of the specific migration of metals and 33% reported to clean the cup. Two participants reported to clean the cup with lint-free cloth. Three participants reported to clean the cup with water, which is not in line with test method EN13130-1 paragraph 15.5. One participant reported to clean the cup with water and soap. The results of this participant were excluded, for three out of six reported results were outliers. All test results of this participant were lower than the group. This may be why in method EN13130-1 paragraph 15.5 it is stated that the specimen should not be cleaned with water.

The majority of the participants (71%) preheated the simulant solution.

5 DISCUSSION

After investigating the methods, limits and calculations for the determination of Specific Migration (see White paper, lit.20), iis decided to only request the test results for the concentration of the metals in mg/L simulant and the migration of the metals in mg/dm^2. All, except one, reporting laboratories reported a test result in mg/dm^2.

The limits for specific migration (see appendix 4) 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 table 6.

Metal	Specific Migration Limit (mg/dm^2) 10/2011/EU	Specific Migration Limit (mg/dm^2) amendment 1416/2016/EU
Aluminum	not restricted	0.17
Barium	0.17	0.17
Cobalt	0.008	0.008
Copper	0.83	0.83
Iron	8.00	8.00
Lithium	0.10	0.10
Manganese	0.10	0.10
Zinc	4.17	0.83

Table 6: specific migration maximum limits

As can be seen from the table above, all laboratories, except one, would reject the sample for being above the maximum limit for Cobalt.

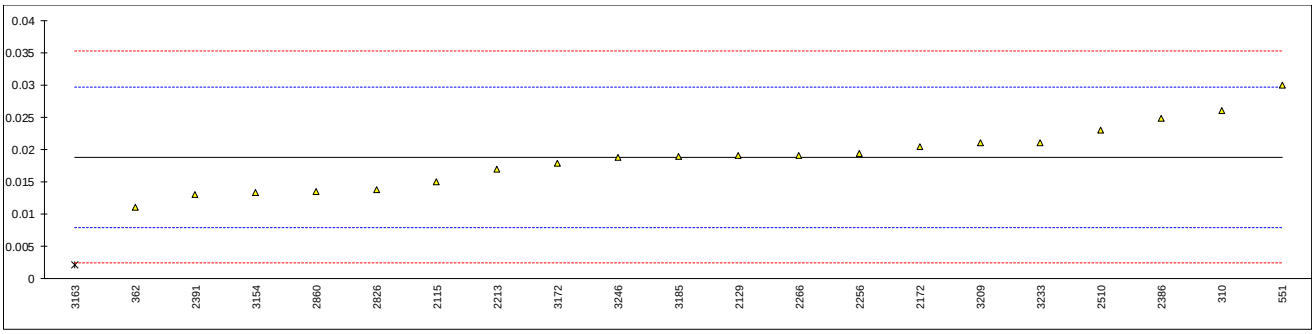
Each laboratory should evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and the quality of the analytical results.

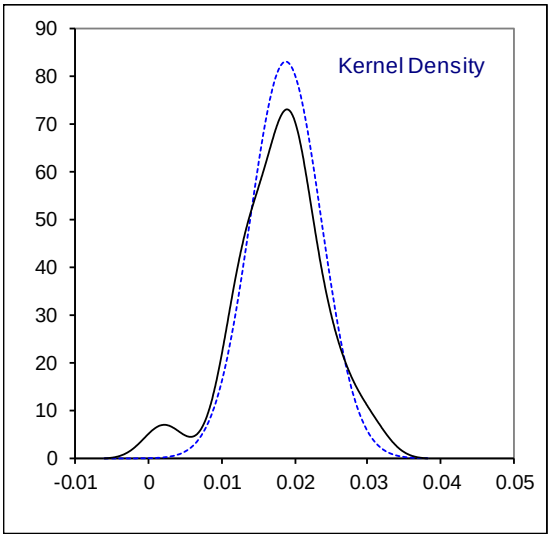
APPENDIX 1

Determination of Specific Migration of Barium as Ba on sample #18620; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
310	EN13130-1	0.026		1.32	
330	EN13130-1	-----		-----	reported only in mg/L, no results in mg/dm^2 (see par. 4.4)
362	In house	0.011	C	-1.42	first reported: 11.3
551	EN13130-1	0.03	C	2.05	first reported: 0.120
827	EN13130-1	ND		-----	
2115	EN13130-1	0.015		-0.69	
2129	In house	0.019		0.04	
2172	EN13130-1	0.0204		0.30	
2213	EN13130-1	0.0170		-0.33	
2256	EN13130-1	0.01932		0.10	
2266	In house	0.019		0.04	
2386	In house	0.0248		1.10	
2391	EN13130-1	0.013		-1.06	
2495		-----		-----	
2510	In house	0.0230		0.77	
2826	EN1186	0.013717		-0.93	
2860	In house	0.0135		-0.97	
3100	EN13130-1	<0.03		-----	
3154	EN13130-1	0.0134		-0.99	
3163		0.0022	ex	-3.03	excluded, for 3 out of 6 reported test results were outliers
3172	EN13130-1	0.0179		-0.16	
3185	EN13130-1	0.0189	C	0.02	first reported: 0.1135
3209	EN13130-1	0.021		0.40	
3233	EN13130-1	0.021		0.40	
3246		0.0188		0.00	

normality	OK	
n	20	
outliers	0 (+1ex)	
mean (n)	0.0188	
st.dev. (n)	0.00481	RSD = 26%
R(calc.)	0.0135	
st.dev.(Horwitz)	0.00547	
R(Horwitz)	0.0153	



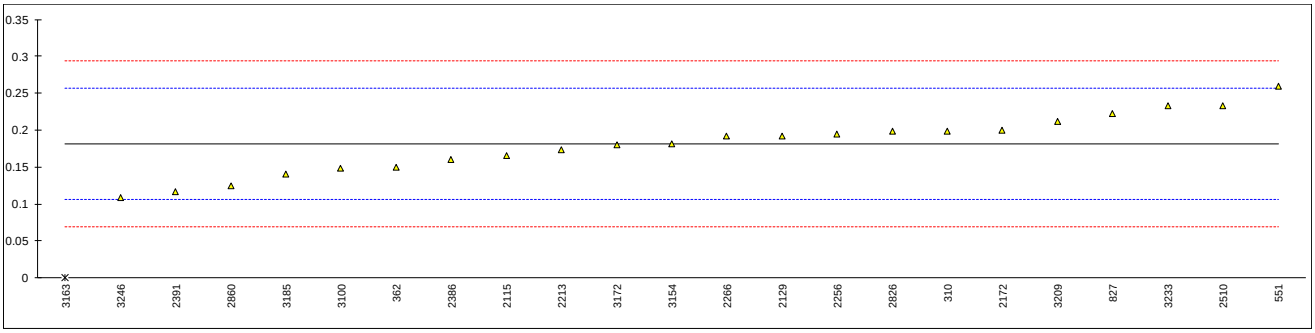


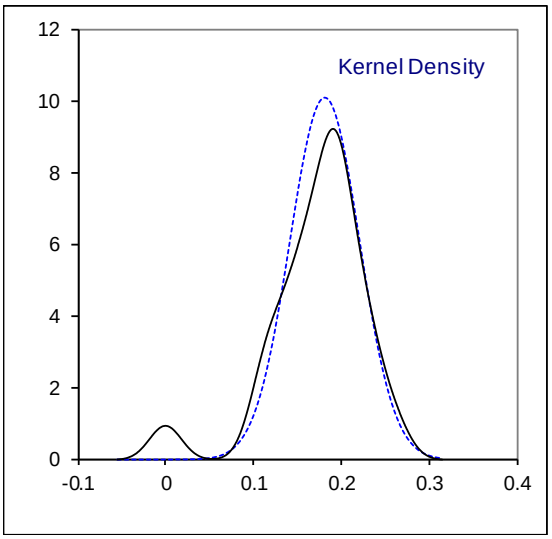
Determination of Specific Migration of Cobalt as Co on sample #18620; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
310	EN13130-1	0.199		0.47	
330	EN13130-1	-----		-----	reported only in mg/L, no results in mg/dm^2 (see par. 4.4)
362	In house	0.15	C	-0.83	first reported: 146
551	EN13130-1	0.26	C	2.10	first reported: 0.959
827	EN13130-1	0.222		1.09	
2115	EN13130-1	0.166		-0.41	
2129	In house	0.192		0.29	
2172	EN13130-1	0.1999		0.50	
2213	EN13130-1	0.173		-0.22	
2256	EN13130-1	0.1946		0.36	
2266	In house	0.192		0.29	
2386	In house	0.1609		-0.54	
2391	EN13130-1	0.117		-1.71	
2495		-----		-----	
2510	In house	0.2332		1.39	
2826	EN1186	0.198938		0.47	
2860	In house	0.1244		-1.52	
3100	EN13130-1	0.149		-0.86	
3154	EN13130-1	0.1816		0.01	
3163		0.000175	R(0.01)	-4.83	
3172	EN13130-1	0.1798		-0.04	
3185	EN13130-1	0.1399	C	-1.10	first reported: 0.8394
3209	EN13130-1	0.212		0.82	
3233	EN13130-1	0.233		1.38	
3246		0.1083		-1.94	

normality	OK
n	22
outliers	1
mean (n)	0.1812
st.dev. (n)	0.03946
R(calc.)	0.1105
st.dev.(Horwitz)	0.03749
R(Horwitz)	0.1050

RSD = 22%

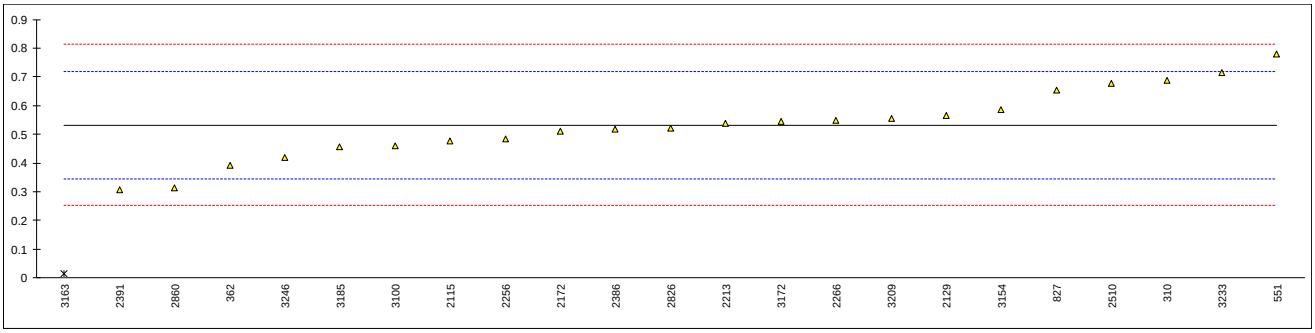


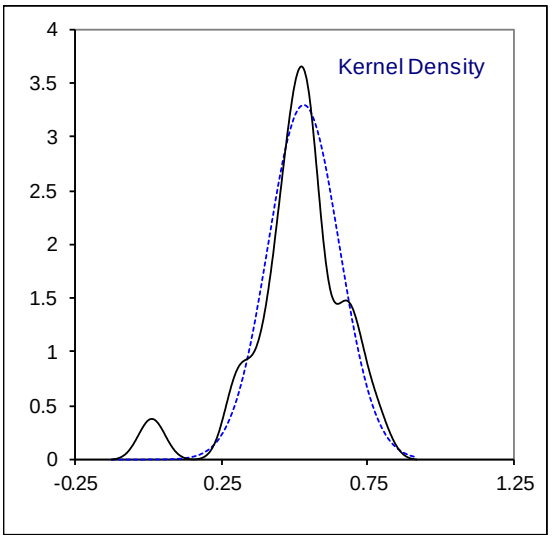


Determination of Specific Migration of Copper as Cu on sample #18620; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
310	EN13130-1	0.688		1.67	
330	EN13130-1	-----		-----	reported only in mg/L, no results in mg/dm^2 (see par. 4.4)
362	In house	0.39	C	-1.51	first reported: 390
551	EN13130-1	0.78	C	2.65	first reported: 2.840
827	EN13130-1	0.653		1.30	
2115	EN13130-1	0.475		-0.61	
2129	In house	0.564		0.35	
2172	EN13130-1	0.5108		-0.22	
2213	EN13130-1	0.539		0.08	
2256	EN13130-1	0.4818		-0.53	
2266	In house	0.548		0.17	
2386	In house	0.5166		-0.16	
2391	EN13130-1	0.305		-2.42	
2495		-----		-----	
2510	In house	0.6771		1.55	
2826	EN1186	0.519766		-0.13	
2860	In house	0.3146		-2.32	
3100	EN13130-1	0.461		-0.76	
3154	EN13130-1	0.5838		0.56	
3163		0.0138	R(0.01)	-5.54	
3172	EN13130-1	0.5454		0.15	
3185	EN13130-1	0.4569	C	-0.80	first reported: 2.7417
3209	EN13130-1	0.555		0.25	
3233	EN13130-1	0.714		1.95	
3246		0.4183		-1.21	

normality	OK	
n	22	
outliers	1	
mean (n)	0.5317	
st.dev. (n)	0.12121	RSD = 23%
R(calc.)	0.3394	
st.dev.(Horwitz)	0.09356	
R(Horwitz)	0.2620	

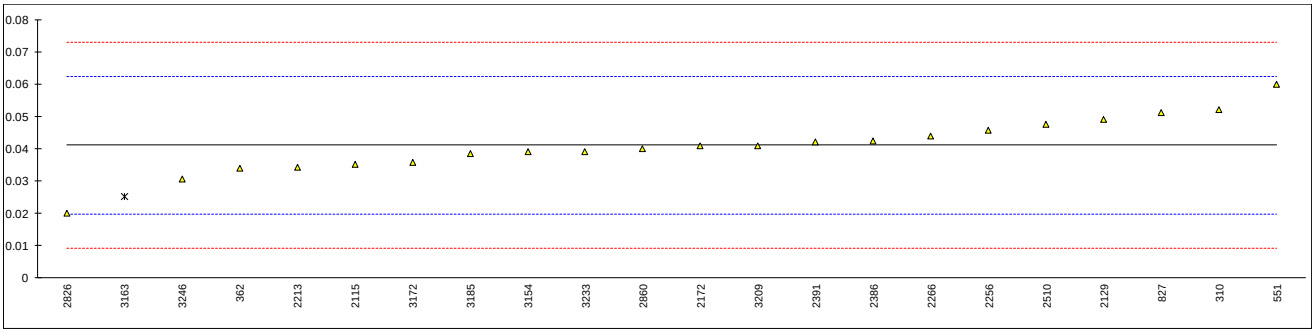


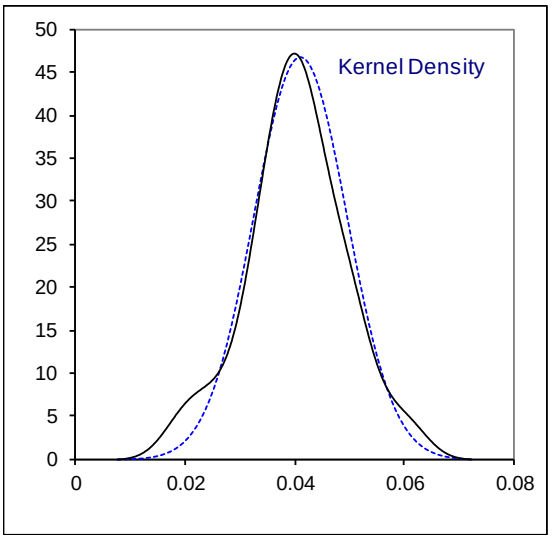


Determination of Specific Migration of Iron as Fe on sample #18620; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
310	EN13130-1	0.052		1.03	
330	EN13130-1	-----		-----	reported only in mg/L, no results in mg/dm^2 (see par. 4.4)
362	In house	0.034	C	-0.66	first reported: 34.0
551	EN13130-1	0.06	C	1.79	first reported: 0.214
827	EN13130-1	0.0511		0.95	
2115	EN13130-1	0.035		-0.57	
2129	In house	0.049		0.75	
2172	EN13130-1	0.0408		-0.02	
2213	EN13130-1	0.0342		-0.64	
2256	EN13130-1	0.04576		0.45	
2266	In house	0.044		0.28	
2386	In house	0.0424		0.13	
2391	EN13130-1	0.042		0.09	
2495		-----		-----	
2510	In house	0.0475		0.61	
2826	EN1186	0.019872		-1.99	
2860	In house	0.0401		-0.09	
3100	EN13130-1	<0.85		-----	
3154	EN13130-1	0.0389		-0.20	
3163		0.0251	ex	-1.50	excluded, for 3 out of 6 reported test results were outliers
3172	EN13130-1	0.0358		-0.49	
3185	EN13130-1	0.0384	C	-0.25	first reported: 0.2306
3209	EN13130-1	0.041		0.00	
3233	EN13130-1	0.039		-0.19	
3246		0.0307		-0.97	

	normality	suspect
	n	21
	outliers	0 (+1ex)
	mean (n)	0.0410
	st.dev. (n)	0.00852 RSD = 21%
	R(calc.)	0.0239
	st.dev.(Horwitz)	0.01062
	R(Horwitz)	0.0297





Determination of Specific Migration of Lithium as Li on sample #18620; results in mg/dm^2

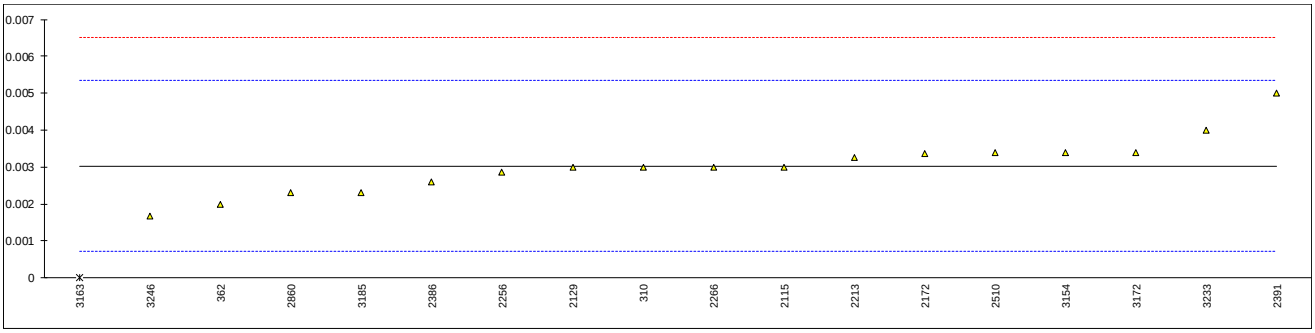
lab	method	value	mark	z(targ)	remarks
110		-----		-----	
310	EN13130-1	<0.001		-----	
330	EN13130-1	-----		-----	reported only in mg/L, no results in mg/dm^2 (see par. 4.4)
362	In house	< 7.75		-----	
551	EN13130-1	<0.05		-----	
827	EN13130-1	ND		-----	
2115		-----		-----	
2129	In house	<0,01		-----	
2172	EN13130-1	0		-----	
2213	EN13130-1	0.000752		-----	
2256	EN13130-1	ND		-----	
2266	In house	0		-----	
2386	In house	<0,001		-----	
2391	EN13130-1	0.001		-----	
2495		-----		-----	
2510	In house	0.000017		-----	
2826	EN1186	<0.0057		-----	
2860	In house	ND		-----	
3100	EN13130-1	<0.03		-----	
3154		-----		-----	
3163		0		-----	
3172	EN13130-1	< 0.001		-----	
3185	EN13130-1	<0.0100		-----	
3209	EN13130-1	<0.01		-----	
3233	EN13130-1	< 0.01		-----	
3246		0		-----	
	n	17			
	mean (n)	<0.1			

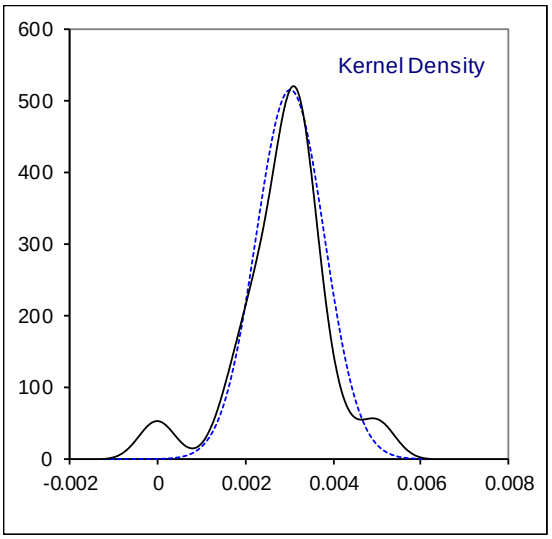
Determination of Specific Migration of Manganese as Mn on sample #18620; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
310	EN13130-1	0.003		-0.03	
330	EN13130-1	-----		-----	reported only in mg/L, no results in mg/dm^2 (see par. 4.4)
362	In house	0.002	C	-0.89	first reported: 2.33
551	EN13130-1	<0.05		-----	
827	EN13130-1	ND		-----	
2115	EN13130-1	0.003		-0.03	
2129	In house	0.0030		-0.03	
2172	EN13130-1	0.00336		0.28	
2213	EN13130-1	0.00326		0.20	
2256	EN13130-1	0.002873		-0.14	
2266	In house	0.003		-0.03	
2386	In house	0.0026		-0.37	
2391	EN13130-1	0.005		1.69	
2495		-----		-----	
2510	In house	0.0034		0.32	
2826	EN1186	<0.0057		-----	
2860	In house	0.0023		-0.63	
3100	EN13130-1	<0.03		-----	
3154	EN13130-1	0.0034		0.32	
3163		0	G(0.05)	-2.61	
3172	EN13130-1	0.0034		0.32	
3185	EN13130-1	0.0023	C	-0.63	first reported: 0.0140
3209	EN13130-1	<0.01		-----	
3233	EN13130-1	0.004		0.83	
3246		0.00166		-1.18	

	normality	suspect
	n	17
	outliers	1
	mean (n)	0.0030
	st.dev. (n)	0.00078
	R(calc.)	0.0022
	st.dev.(Horwitz)	0.00116
	R(Horwitz)	0.0033

RSD = 26%

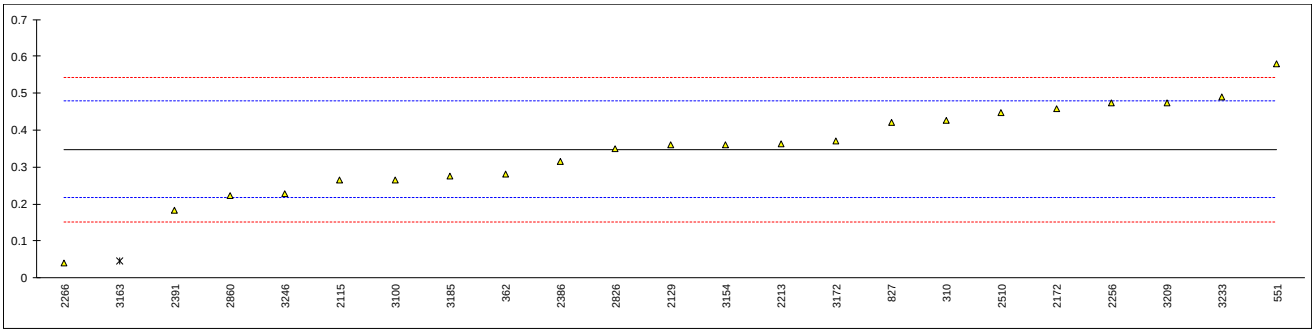


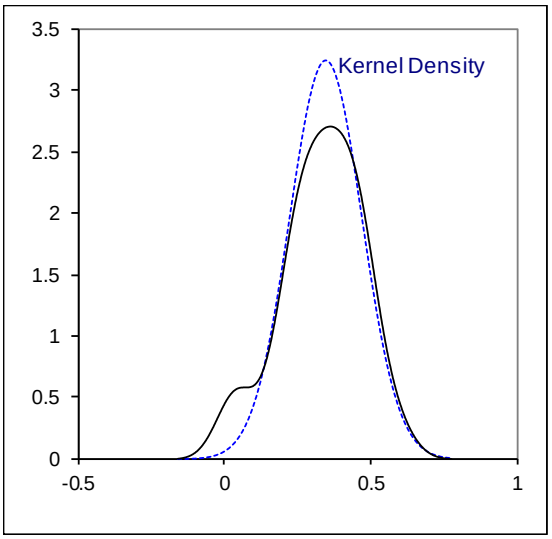


Determination of Specific Migration of Zinc as Zn on sample #18620; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
310	EN13130-1	0.425		1.18	
330	EN13130-1	-----		-----	reported only in mg/L, no results in mg/dm^2 (see par. 4.4)
362	In house	0.28	C	-1.04	first reported: 280
551	EN13130-1	0.58	C	3.56	first reported: 2.122
827	EN13130-1	0.420		1.11	
2115	EN13130-1	0.265		-1.27	
2129	In house	0.359		0.17	
2172	EN13130-1	0.4592		1.71	
2213	EN13130-1	0.364		0.25	
2256	EN13130-1	0.4742		1.94	
2266	In house	0.04		-4.72	
2386	In house	0.3161		-0.49	
2391	EN13130-1	0.183		-2.53	
2495		-----		-----	
2510	In house	0.4482		1.54	
2826	EN1186	0.349676		0.03	
2860	In house	0.2224		-1.92	
3100	EN13130-1	0.266		-1.25	
3154	EN13130-1	0.3593		0.18	
3163		0.0448	ex	-4.64	excluded, for 3 out of 6 reported test results were outliers
3172	EN13130-1	0.3713		0.36	
3185	EN13130-1	0.2761	C	-1.10	first reported: 1.6569
3209	EN13130-1	0.475		1.95	
3233	EN13130-1	0.490		2.18	
3246		0.229		-1.82	

normality	OK
n	22
outliers	0 (+1ex)
mean (n)	0.3478
st.dev. (n)	0.12287 RSD = 35%
R(calc.)	0.3440
st.dev.(Horwitz)	0.06524
R(Horwitz)	0.1827





APPENDIX 2

Details on final concentration, surface area and volume of simulant reported for one metal (Copper)

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
110	-----	-----	-----	-----	-----	-----	-----
310	1.56	220	0.71	4.127	0.688	0.5820	-0.11
330	1.63	250	0.65	4.29	-----	0.6580	-----
362	1.61	250	0.64	2.51	0.39	0.3898	0.00
551	1.65	240	0.69	4.657	0.78	0.6774	-0.10
827	1.63	250	0.65	4.2553	0.653	0.6527	0.00
2115	1.638	236	0.69	3.3	0.475	0.4755	0.00
2129	1.2	200	0.60	3.39	0.564	0.5650	0.00
2172	0.00648	250	0.00	3.3103	0.5108	127.7122	127.20
2213	1.61	230	0.70	3.773	0.539	0.5390	0.00
2256	1.62	250	0.65	3.122	0.4818	0.4818	0.00
2266	1.67	235	0.71	3.894	0.548	0.5480	0.00
2386	1.64	240	0.68	3.53	0.5166	0.5166	0.00
2391	1.7554	260	0.68	2.061	0.305	0.3053	0.00
2495	-----	-----	-----	-----	-----	-----	-----
2510	1.494	204	0.73	4.9588	0.6771	0.6771	0.00
2826	1.73514	247.8	0.70	3.6395	0.519766	0.5198	0.00
2860	1.74113	250	0.70	2.191	0.3146	0.3146	0.00
3100	1.756	250	0.70	3.239	0.461	0.4611	0.00
3154	-----	-----	-----	6.007	0.5838	-----	-----
3163	1.713	230	0.74	0.0236	0.0138	0.0032	-0.01
3172	1.565	230	0.68	3.7113	0.5454	0.5454	0.00
3185	1.58	230	0.69	3.139	0.4569	0.4569	0.00
3209	1.6	250	0.64	3.556	0.555	0.5556	0.00
3233	1.67	241	0.69	4.284	0.714	0.6182	-0.10
3246	1.332	222	0.60	2.51	0.4183	0.4183	0.00

APPENDIX 3

Analytical Details

lab	accredited acc. ISO/IEC17025	sample cleaned prior to the migration step	Cleaned with	simulant preheated to 100°C	Migration time (min)	Migration temp. (°C)
110	---	---		---		
310	No	No		Yes	120	100
330	No	Yes		Yes	120 min	100°C
362	Yes	No		No	120	100
551	Yes	Yes	a lint-free cloth	Yes	120	100
827	No	No		---	120 min	100
2115	Yes	No		Yes	120 min	100°C
2129	Yes	No		Yes	120	100
2172	Yes	No		Yes	120 minutes	100
2213	No	Yes	Distilled water	Yes	120	100.1
2256	Yes	Yes		Yes	120	100
2266	No	No		Yes	120	100
2386	Yes	No		Yes	120	100
2391	No	No		No	120	100
2495	---	---		---		
2510	No	No		Yes	120	100
2826	No	No		Yes	60	100
2860	No	No		No	120	100.0
3100	Yes	Yes	distilled water	Yes	120	100
3154	---	---		---		
3163	No	Yes	water and soap	Yes	120	100
3172	No	No		No	120	100
3185	Yes	No	NA	Yes	120 minutes	100°C
3209	Yes	No		Yes	120	100
3233	No	Yes	a lint-free cloth	Yes	120 min	100°C
3246	No	Yes	Distilled water	No	120 minutes	100°C

APPENDIX 4

Specific migration maximum limits by regulation for metals:

Metal	Specific Migration Limit (mg/kg food simulant) 10/2011/EU	Specific Migration Limit (mg/kg food simulant) amendment 1416/2016/EU
Aluminum	not restricted	1
Barium	1	1
Cobalt	0.05	0.05
Copper	5	5
Iron	48	48
Lithium	0.6	0.6
Manganese	0.6	0.6
Zinc	25	5

APPENDIX 5

Number of participating laboratories per country

1 lab in BRAZIL
1 lab in BULGARIA
3 labs in FRANCE
3 labs in GERMANY
1 lab in HONG KONG
1 lab in INDIA
1 lab in IRELAND
3 labs in ITALY
1 lab in KOREA
1 lab in MALAYSIA
5 labs in P.R. of CHINA
1 lab in SERBIA
2 labs in THE NETHERLANDS
1 lab in U.S.A.
1 lab 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 = probably an error in calculation
U = test result probably reported in a different unit
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 EN13130-13 Materials and articles in contact with foodstuffs – Plastics substances subject to limitation - Determination of 2,2-bis(4-hydroxyphenyl)propane (Bisphenol A) in food simulants
4 Commission regulation (EU) No 1935/2004 of 27 October 2004, on materials and articles intended to come into contact with food
5 Commission regulation (EU) No 10/2011 of 14 January 2011 on plastic materials and articles intended to come into contact with food
6 ASTM E178:02
7 ASTM E1301:03
8 ISO 5725:86
9 ISO 5725, parts 1-6, 1994
10 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
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20 White paper on the determinations of Overall and Specific migration on food contact materials, February 2018, http://www.iisnl.com/pdf/WhitePaper.pdf (or www.iisnl.com on News and Report page in News)