Results of Proficiency Test
Specific migration (fcm)
October 2012

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

1 INTRODUCTION

On request of a number of participants in the iis PT program it was decided to start PTs on food contact materials in 2012.
During the contact of materials, like kitchenware, with food, 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 (EC) No. 1935/2004 applies to all food contact materials and describes a large number of requirements, e.g. limits for overall migration and specific limits for certain constituents. The determination of specific migration requires additional analytical testing following the migration step, while the determination of the overall migration requires weighing as only quantitative analytical technique. This makes the specific migration of formaldehyde from melamine kitchenware more difficult than determination of the overall migration.
In the interlaboratory study of October 2012, 38 laboratories from 14 different countries participated (See appendix 3). In this report, the results of the proficiency test on Specific Migration are presented and discussed.

2 SET-UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted. It was decided to send one sample, that was known to give a measurable test result, labelled #12112, and to prescribe a number of test conditions (type of simulant, amount of simulant, exposure time and temperature) to be used. Participants were also requested to report the test conditions that the laboratory would have used in case these were not prescribed by iis.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO guide 43, ILAC-G13:2007 and ISO/IEC 17043:2010. This ensures 100% confidentially of participant's data. Also, customer's satisfaction is measured on a regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2). This protocol can be downloaded from the iis website http://www.iisnl.com.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

A batch of melamine bowls for repetitive use in the household that gave positive test results for specific migration of formaldehyde was selected.
The homogeneity of the batch was checked by determination of the Specific Migration of formaldehyde on seven stratified randomly selected bowls.

	Specific Migration in mg/kg #12112
Sample 1	3.3
Sample 2	4.0
Sample 3	2.7
Sample 4	2.5
Sample 5	3.3
Sample 6	2.7
Sample 7	3.5

Table 1: results of the homogeneity test on the subsamples #12112

The repeatability for Specific Migration on the seven samples #12112 is in agreement with the repeatability of the laboratory performing the tests.
Therefore, homogeneity of the samples #12112 was assumed.

To each of the participating laboratories one sample #12112 was sent on September 26, 2012.

2.5 ANALYSIS

The participants were requested to determine the Specific Migration of formaldehyde on the sample using the prescribed test conditions. It was requested to report the analytical results using the indicated units on the report form and to use a minimum number of digits and not to round the results more. It was also requested not to report ‘less than’ results, which are above the detection limit, because such results cannot be used for meaningful statistical calculations.
To get comparable results a detailed report form, on which the units were prescribed, was sent together with the sample. Also, a letter of instructions was added to the package.
The laboratories were also requested to report the test conditions that the laboratory would have used in case these were not prescribed by iis.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were received.
The original data are tabulated per sample in the appendix 1 of this report.
The laboratories are represented by the code numbers.

Directly after the deadline, a reminder fax was sent to those laboratories that did not report results at that moment.
Shortly after the deadline, the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier.

The laboratories that produced these suspect data were asked to check the results. Additional or corrected results are used for the data analysis and the original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

The statistical calculations were performed as described in the procedures in the report ‘iis Interlaboratory Studies, Protocol for the Organisation, Statistics and Evaluation’ of January 2010 (iis-protocol, version 3.2).

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

In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have significant consequences for the evaluation of the test results.

3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are under the X-axis.
The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 4; refs.14 and 15).

3.3 Z-SCORES

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

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

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

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

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used. This, in order to evaluate the fit-for-useness of the reported test result. See also appendix 3; ref. 16.

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

$\lvert z \rvert < 1$	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. Three participants reported test results after the final reporting date and four other participants did not report any test results at all. Finally, 34 of the 38 participants submitted analysis results. These 34 laboratories reported 303 numerical test results. Observed were 12 outlying test results, which is 3.8%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

A not-normal distribution was found for the reported specific migration results in mg/kg for the second contact. Therefore this statistical evaluation should be used with due care.

For the determination of Specific Migration, no standardised test method does exist. The most relevant literature is the JRC report EUR 24815 EN 2011 (ref. 17). These guidelines describe the migration test in detail, for example that for formaldehyde migration from polyamide and melamine kitchenware three successive migration tests should be performed and that 3% acetic acid should be used as simulant. The guidelines mention repeatability data for formaldehyde in 3% acetic acid (equal to the data mentioned in CEN/TS13130-23:2005). However, this repeatability appears not to be realistic as it is much smaller than the corresponding Horwitz value (r=0.25 mg/kg vs. r(Horwitz) = 1.49 mg/kg (4.47/3), both at a level of 15 mg/kg formaldehyde). Therefore it was decided to estimate the target reproducibility from the Horwitz equation.

Three laboratories appeared to have reported inconsistent test results. For laboratory 2115, both the formaldehyde concentration and the specific migration result in mg/kg are 10-fold lower as expected, while the result in mg/dm^2 is 100-fold lower as expected. For laboratories 2372 and 2375, the migration results in mg/dm^2 are not in line with the formaldehyde concentrations in the simulant. And because all test results of one laboratory are correlated, all test results of the laboratories 2115, 2372 and 2375 were excluded manually prior to the statistical analysis.

4.1 PERFORMANCE EVALUATION OF THE GROUP OF LABORATORIES

The calculated reproducibilities and the target reproducibilities are compared in the next table.

	unit	n	Average	2.8 * sd	R (target)
Formaldehyde in simulant, 1st c.	mg/L	29	4.90	6.26	1.73
Specific migration, 1st contact	mg/dm^2	29	0.65	0.83	0.31
Specific migration, 1st contact	mg/kg	26	3.83	4.66	1.40
Formaldehyde in simulant, 2nd c.	mg/L	30	4.34	4.96	1.56
Specific migration, 2nd contact	mg/dm^2	30	0.58	0.66	0.28
Specific migration, 2nd contact	mg/kg	29	3.77	5.32	1.38
Formaldehyde in simulant, 3rd c.	mg/L	31	4.38	5.74	1.57
Specific migration, 3rd contact	mg/dm^2	31	0.58	0.77	0.28
Specific migration, 3rd contact	mg/kg	29	3.55	5.14	1.31

Table 2: performance overview for sample #12112

4.2 EVALUATION

In this section the results are discussed.
No significant differences were observed between the results of the 1st, the 2nd and 3rd contact.

Formaldehyde in simulant in mg/L:

This determination was very problematic. A wide range of test results was reported, e.g. for the 3rd contact: 0.332 – 9.42 mg/kg. In total only two statistical outliers were detected. However, the calculated reproducibilities, after rejection of the statistical outliers, are not at all in agreement with the target reproducibilities estimated from the Horwitz equation.

Specific migration of formaldehyde in mg/dm^2:

This determination was very problematic. A wide range of test results was reported, e.g. for the 3rd contact: 0.0061 – 1.26 mg/dm^2. In total five statistical outliers were detected. The calculated reproducibilities, after rejection of the statistical outliers, are not at all in agreement with the target reproducibilities estimated from the Horwitz equation.

Specific migration of formaldehyde in mg/kg:

The reporting in mg/kg was very problematic. A wide range of test results was reported, e.g. for the 3rd contact: 0.12 – 9.41 mg/kg. In total five statistical outliers were detected. The calculated reproducibilities, after rejection of the statistical outliers, are not at all in agreement with the target reproducibilities estimated from the Horwitz equation. Apparently the majority of the participants did report test results in mg/L instead of the requested mg/kg. See also paragraph 5. Therefore no z-scores were calculated.

4.3 EVALUATION OF THE TEST METHODS USED

Most participants reported to have used as test method EUR 24815 EN 2011 or EN13130 (part 1 or 23). These methods describe identical procedures and therefore no differences in the test results are expected.

5 CONCLUSIONS

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 preset conditions were:

Sample ID	**#12112**
Simulant	3% acetic acid in water
Bowl volume	600 cm^3 (*fictive figure*)
Simulant amount	200 ml (preheated)
Exposure time	2.0 hrs
Exposure temperature	70.0 °C
Contact surface	1.50 dm^2 (*fictive figure*)
Migration method	Article filling

Table 3: preset test conditions used in this PT

Not only a migration result was to be reported, but the participants were requested to report also the intermediate formaldehyde concentration in the simulant. Using these intermediate test results it would be possible to check all calculations and corrections done by the laboratories. Thus it was found that three participating laboratories possibly made calculation errors.

The spread in the intermediate test results (the formaldehyde concentrations in mg/L) is equal to the spread in the migration results mg/dm^2. However, the Specific Migration results in mg/kg show a larger spread. This is rather surprising as the calculation is not difficult:

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

Upon investigation for the reason of the increase in spread, it was found that a number of laboratories did not use the conventional surface to volume ratio of 6 dm^2/kg cfr. EN13130-1:2004, paragraphs 10.2 and 13.1.1. The factor between the residue and the migration result was not 6, but varied from 0.2 – 130.6, see appendix 1. Apparently the majority of the participants reported test results in mg/L simulant (!) instead of the requested Specific Migration in mg/kg food. See also Annex 1 of 2002/72/EC (L220/22) for this requirement.

During the PT the participants were requested to report which test conditions they would have selected in case these were not prescribed as in this PT. From the responses (appendix 2) it became clear that the test conditions as set were quite realistic:

- All participating laboratories would have selected article filling.
- The vast majority of the participants (>90%) would have used an exposure of 2 hours, an exposure temperature of 70°C and would have reported the migration in mg/kg only.
- The amount of simulant to be used varies from 200 mL up to 600 mL (a 100% filled bowl)
- The ratio amount of simulant per dm^2 contact surface varies from 80 - 343 (iis gave 133)

A number of methods to determine the contact surface were mentioned:

- measure by caliper and calculate using mathematical equations
- graphic millimeter paper method cfr JRC guidelines EUR24815 EN 2011
- by contour / measurement of projected area
- by dividing surface into polygons
- by cutting & measurement of rectangles
- by cutting and weighing

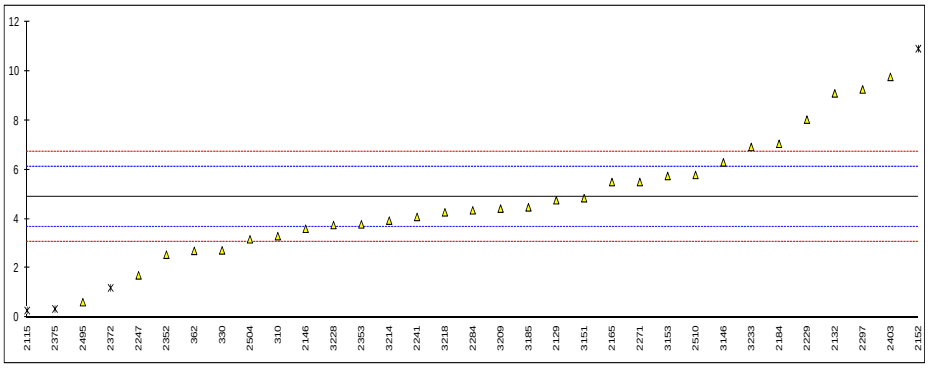
Although all mentioned methods may be sufficient to estimate the contact surface, a large spread in the estimated surfaces is observed, where the largest estimate is about 4 times the smallest estimate (see above and also appendix 2).

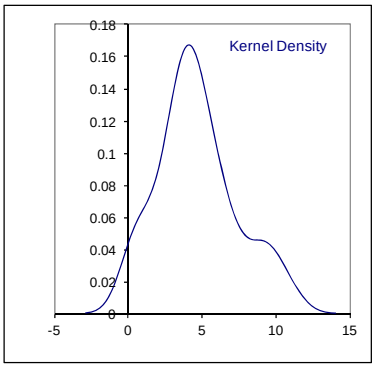
Each laboratory has to evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and the quality of the analytical results.

APPENDIX 1

Concentration of formaldehyde in simulant 1st contact on sample #12112; results in mg/L

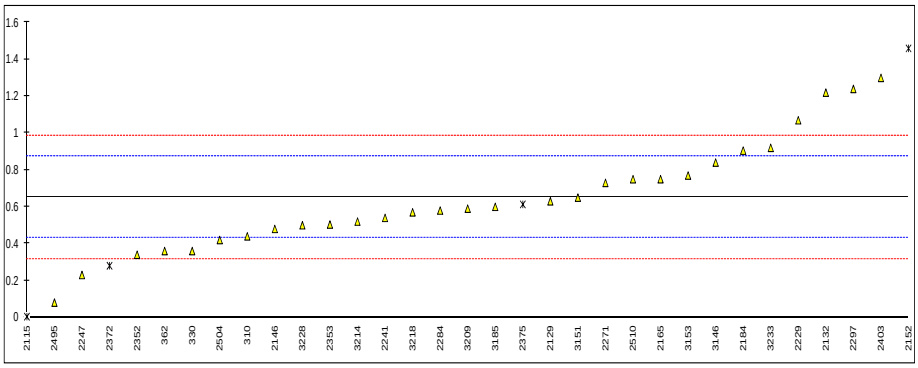
lab	method	value	mark	z(targ)	remarks
310	EN1186-1	3.30		-2.59	
330		2.72		-3.53	
362		2.70		-3.56	
2115		0.27	ex	-7.50	See § 4.1
2129	ISO14184-1	4.76		-0.22	
2132	CEN/TS13130-23	9.11		6.83	
2146	Acetyl/Acetone	3.6		-2.10	
2152	CEN/TS13130-23	10.93	D(0.05)	9.78	
2156		-----		-----	
2165	EN13130-23	5.50		0.98	
2184	EN13130-23	7.06		3.51	
2190		-----		-----	
2229	EUR24815 EN2011	8.04		5.09	
2241		4.08		-1.32	
2247	UV Vis Spec.	1.70		-5.18	
2271		5.50		0.98	
2284	EU10/2011, EN13130-1	4.35		-0.89	
2297		9.27		7.09	
2352	EUR24815 EN2011, EN13130-1	2.54		-3.82	
2353	EN13130-32	3.7775		-1.81	
2357		-----		-----	
2372	EUR24815 EN2011, EN13130-1	1.19	ex	-6.01	See § 4.1
2375	CEN/TS13130-23, EN13130-1	0.333	ex	-7.40	See § 4.1
2403		9.78		7.92	
2475		-----		-----	
2488		-----		-----	
2495	ISO4614	0.61		-6.95	
2504		3.17		-2.80	
2510	EN13130-1	5.79		1.45	
3146	EN13130	6.30		2.27	
3151	EN13130-1	4.84		-0.09	
3153	EN13130-1	5.75		1.38	
3185	EU10/2011, EN13130-1, EN13130-23	4.47		-0.69	
3209	EN13130-23	4.42		-0.77	
3214	CEN/TS13130	3.93		-1.57	
3218	EN13130-23	4.27		-1.02	
3228	ISO14184-1	3.75		-1.86	
3233	EUR24815 EN2011, EU10/2011	6.93		3.30	
	normality	OK			
	n	29			
	outliers	1			
	mean (n)	4.897			
	st.dev. (n)	2.2349			
	R(calc.)	6.258			
	R(Horwitz)	1.727			

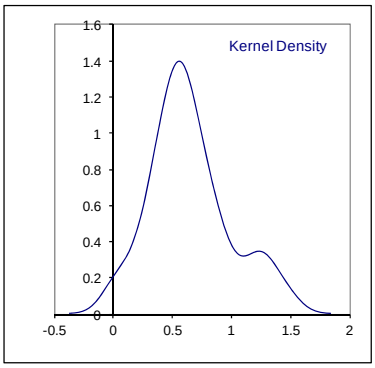




Specific Migration of formaldehyde 1st contact on sample #12112; results in mg/dm^2

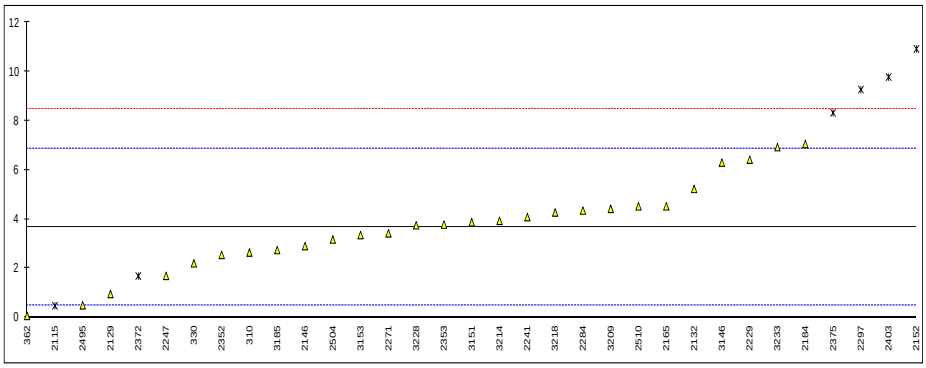
lab	method	value	mark	z(targ)	remarks
310	EN1186-1	0.44		-1.90	
330		0.36		-2.62	
362		0.36	C	-2.62	first reported 3.60
2115		0.0036	ex	-5.83	See § 4.1
2129	ISO14184-1	0.63		-0.19	
2132	CEN/TS13130-23	1.22		5.12	
2146	Acetyl/Acetone	0.48		-1.54	
2152	CEN/TS13130-23	1.46	D(0.05)	7.28	
2156		-----		-----	
2165	EN13130-23	0.75		0.89	
2184	EN13130-23	0.905		2.29	
2190		-----		-----	
2229	EUR24815 EN2011	1.07		3.77	
2241		0.54		-1.00	
2247	UV Vis Spec.	0.23		-3.79	
2271		0.73		0.71	
2284	EU10/2011, EN13130-1	0.58		-0.64	
2297		1.24		5.30	
2352	EUR24815 EN2011, EN13130-1	0.34		-2.80	
2353	EN13130-32	0.5037		-1.33	
2357		-----		-----	
2372	EUR24815 EN2011, EN13130-1	0.28	ex	-3.34	See § 4.1
2375	CEN/TS13130-23, EN13130-1	0.613	ex	-0.34	See § 4.1
2403		1.30		5.84	
2475		-----		-----	
2488		-----		-----	
2495	ISO4614	0.08		-5.14	
2504		0.42		-2.08	
2510	EN13130-1	0.75		0.89	
3146	EN13130	0.84		1.70	
3151	EN13130-1	0.65		-0.01	
3153	EN13130-1	0.77		1.07	
3185	EU10/2011, EN13130-1, EN13130-23	0.60		-0.46	
3209	EN13130-23	0.59		-0.55	
3214	CEN/TS13130	0.52		-1.18	
3218	EN13130-23	0.57		-0.73	
3228	ISO14184-1	0.50		-1.36	
3233	EUR24815 EN2011, EU10/2011	0.92		2.42	
	normality	OK			
	n	29			
	Outliers	1			
	mean (n)	0.651			
	st.dev. (n)	0.2969			
	R(calc.)	0.831			
	R(Horwitz)	0.311			

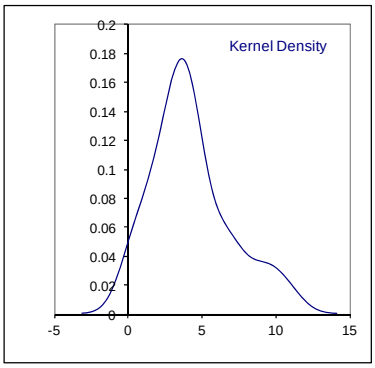




Specific Migration of formaldehyde 1st contact on sample #12112; results in mg/kg

lab	method	value	mark	z(targ)	remarks	
310	EN1186-1	2.64		-----		6.0
330		2.2		-----		6.3
362		0.06	ex, C	-----	See § 4.1, fr. 0.60	0.2
2115		0.47	ex	-----	See § 4.1	130.6
2129	ISO14184-1	0.95		-----		1.5
2132	CEN/TS13130-23	5.23		-----		4.3
2146	Acetyl/Acetone	2.9		-----		6.0
2152	CEN/TS13130-23	10.93	D(0.05)	-----	reported in mg/L?	7.5
2156		-----		-----		
2165	EN13130-23	4.52		-----		6.0
2184	EN13130-23	7.06		-----	reported in mg/L?	7.8
2190		-----		-----		
2229	EUR24815 EN2011	6.42		-----		6.0
2241		4.08		-----	reported in mg/L?	7.6
2247	UV Vis Spec.	1.69		-----	reported in mg/L?	7.3
2271		3.42		-----		4.7
2284	EU10/2011, EN13130-1	4.35		-----	reported in mg/L?	7.5
2297		9.27	DG(0.05)	-----	reported in mg/L?	7.5
2352	EUR24815 EN2011, EN13130-1	2.54		-----	reported in mg/L?	7.5
2353	EN13130-32	3.7775		-----	reported in mg/L?	7.5
2357		-----		-----		
2372	EUR24815 EN2011, EN13130-1	1.68	ex	-----	See § 4.1	6.0
2375	CEN/TS13130-23, EN13130-1	8.325	ex	-----	See § 4.1	13.6
2403		9.78	DG(0.05)	-----	reported in mg/L?	7.5
2475		-----		-----		
2488		-----		-----		
2495	ISO4614	0.49		-----		6.1
2504		3.17		-----	reported in mg/L?	7.5
2510	EN13130-1	4.52		-----		6.0
3146	EN13130	6.30		-----	reported in mg/L?	7.5
3151	EN13130-1	3.87		-----		6.0
3153	EN13130-1	3.35		-----		4.4
3185	EU10/2011, EN13130-1, EN13130-23	2.74		-----		4.6
3209	EN13130-23	4.42		-----	reported in mg/L?	7.5
3214	CEN/TS13130	3.93		-----	reported in mg/L?	7.6
3218	EN13130-23	4.27		-----	reported in mg/L?	7.5
3228	ISO14184-1	3.75		-----	reported in mg/L?	7.5
3233	EUR24815 EN2011, EU10/2011	6.93		-----	reported in mg/L?	7.5
					theoretical results using 6 as conversion factor:	
	normality	OK			OK	
	n	26			26	
	Outliers	3			3	
	mean (n)	3.828			3.701	
	st.dev. (n)	1.6651			1.5192	
	R(calc.)	4.662			4.254	
	R(Horwitz)	1.401			1.362	

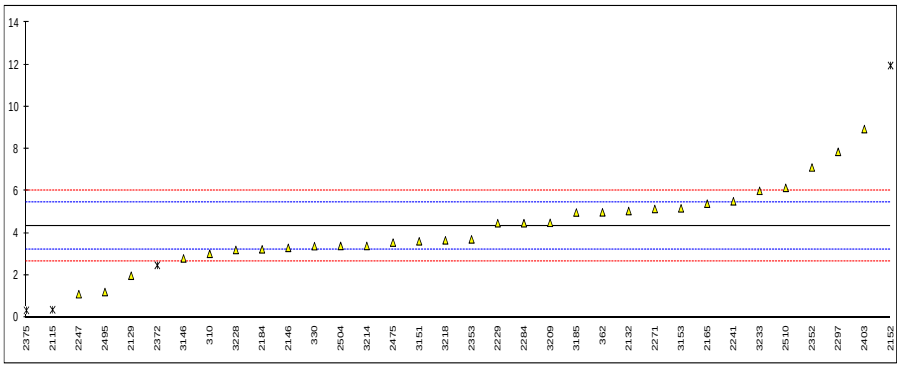


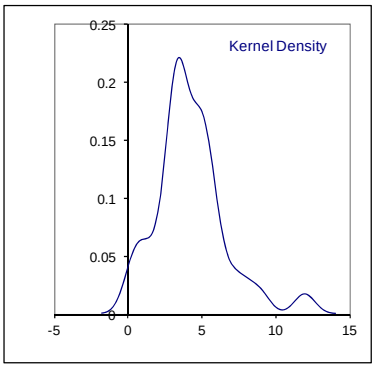


Concentration of formaldehyde in simulant 2nd contact on sample #12112; results in mg/L

lab	method	value	mark	z(targ)	remarks
310	EN1186-1	3.02		-2.37	
330		3.38		-1.73	
362		4.99		1.16	
2115		0.36	ex	-7.15	See § 4.1
2129	ISO14184-1	1.98		-4.24	
2132	CEN/TS13130-23	5.05		1.27	
2146	Acetyl/Acetone	3.3		-1.87	
2152	CEN/TS13130-23	11.96	D(0.05)	13.67	
2156		-----		-----	
2165	EN13130-23	5.40		1.90	
2184	EN13130-23	3.23		-2.00	
2190		-----		-----	
2229	EUR24815 EN2011	4.47		0.23	
2241		5.51		2.10	
2247	UV Vis Spec.	1.10		-5.82	
2271		5.15		1.45	
2284	EU10/2011, EN13130-1	4.47		0.23	
2297		7.86		6.31	
2352	EUR24815 EN2011, EN13130-1	7.12		4.99	
2353	EN13130-32	3.7011		-1.15	
2357		-----		-----	
2372	EUR24815 EN2011, EN13130-1	2.47	ex	-3.36	See § 4.1
2375	CEN/TS13130-23, EN13130-1	0.331	ex	-7.20	See § 4.1
2403		8.94		8.25	
2475		3.55		-1.42	
2488		-----		-----	
2495	ISO4614	1.20		-5.64	
2504		3.39		-1.71	
2510	EN13130-1	6.15		3.24	
3146	EN13130	2.80		-2.77	
3151	EN13130-1	3.61		-1.32	
3153	EN13130-1	5.18		1.50	
3185	EU10/2011, EN13130-1, EN13130-23	4.98		1.14	
3209	EN13130-23	4.49		0.26	
3214	CEN/TS13130	3.39		-1.71	
3218	EN13130-23	3.66		-1.23	
3228	ISO14184-1	3.20		-2.05	
3233	EUR24815 EN2011, EU10/2011	6.01		2.99	

normality	OK
n	30
outliers	1
mean (n)	4.343
st.dev. (n)	1.7714
R(calc.)	4.9599
R(Horwitz)	1.560

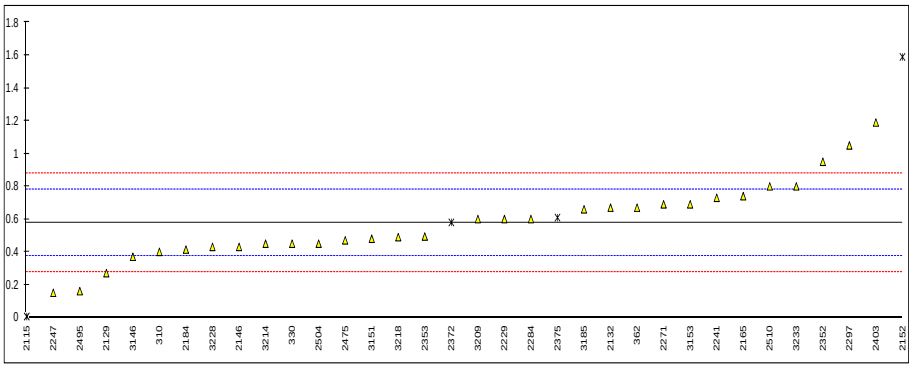


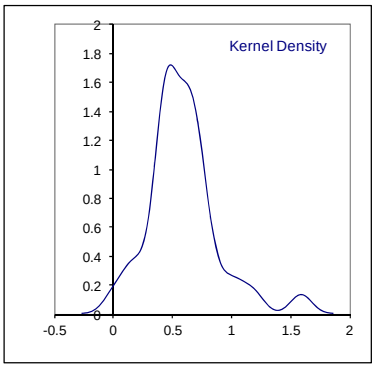


Specific Migration of formaldehyde 2nd contact on sample #12112; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
310	EN1186-1	0.40		-1.77	
330		0.45		-1.28	
362		0.67	C	0.91	first reported 6.65
2115		0.0048	ex	-5.71	See § 4.1
2129	ISO14184-1	0.27		-3.07	
2132	CEN/TS13130-23	0.67		0.91	
2146	Acetyl/Acetone	0.43		-1.48	
2152	CEN/TS13130-23	1.59	D(0.05)	10.07	
2156		-----		-----	
2165	EN13130-23	0.74		1.61	
2184	EN13130-23	0.414		-1.63	
2190		-----		-----	
2229	EUR24815 EN2011	0.60		0.22	
2241		0.73		1.51	
2247	UV Vis Spec.	0.15		-4.26	
2271		0.69		1.11	
2284	EU10/2011, EN13130-1	0.60		0.22	
2297		1.05		4.70	
2352	EUR24815 EN2011, EN13130-1	0.95		3.70	
2353	EN13130-32	0.4935		-0.84	
2357		-----		-----	
2372	EUR24815 EN2011, EN13130-1	0.58	ex	0.02	See § 4.1
2375	CEN/TS13130-23, EN13130-1	0.609	ex	0.31	See § 4.1
2403		1.19		6.09	
2475		0.47		-1.08	
2488		-----		-----	
2495	ISO4614	0.16		-4.16	
2504		0.45		-1.28	
2510	EN13130-1	0.80		2.21	
3146	EN13130	0.37		-2.07	
3151	EN13130-1	0.48		-0.98	
3153	EN13130-1	0.69		1.11	
3185	EU10/2011, EN13130-1, EN13130-23	0.66		0.81	
3209	EN13130-23	0.60		0.22	
3214	CEN/TS13130	0.45		-1.28	
3218	EN13130-23	0.49		-0.88	
3228	ISO14184-1	0.43		-1.48	
3233	EUR24815 EN2011, EU10/2011	0.80		2.21	

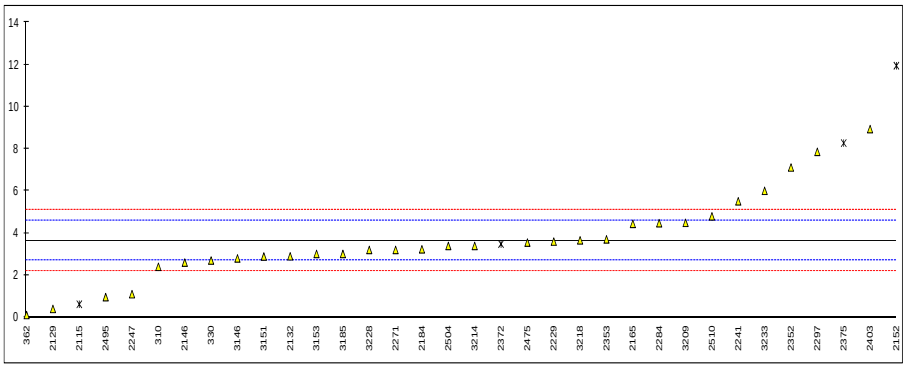
normality	OK
n	30
outliers	1
mean (n)	0.578
st.dev. (n)	0.2362
R(calc.)	0.662
R(Horwitz)	0.281

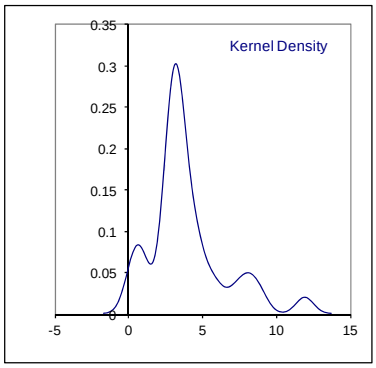




Specific Migration of formaldehyde 2nd contact on sample #12112; results in mg/kg

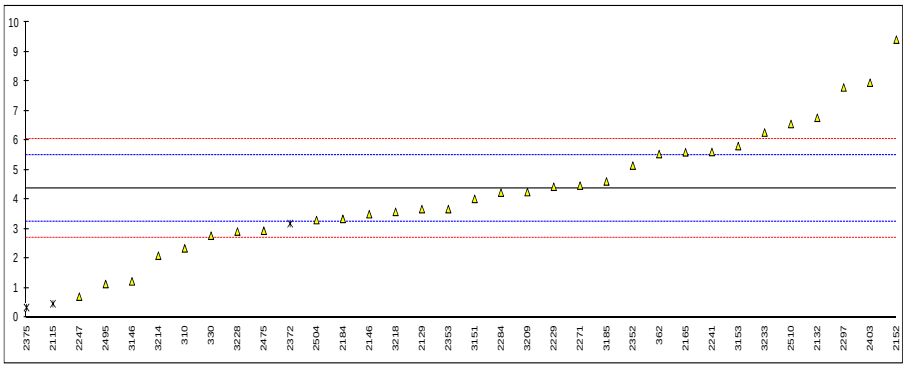
lab	method	value	mark	z(targ)	remarks	conversion factor used
310	EN1186-1	2.40		-----		6.0
330		2.7		-----		6.0
362		0.11	ex, C	-----	See § 4.1, fr. 1.11	0.2
2115		0.62	ex	-----	See § 4.1	129.2
2129	ISO14184-1	0.40		-----		1.5
2132	CEN/TS13130-23	2.90		-----		4.3
2146	Acetyl/Acetone	2.6		-----		6.0
2152	CEN/TS13130-23	11.95	G(0.01)	-----	reported in mg/L?	7.5
2156		-----		-----		
2165	EN13130-23	4.44		-----		6.0
2184	EN13130-23	3.23		-----	reported in mg/L?	7.8
2190		-----		-----		
2229	EUR24815 EN2011	3.60		-----		6.0
2241		5.51		-----	reported in mg/L?	7.5
2247	UV Vis Spec.	1.10		-----	reported in mg/L?	7.3
2271		3.20		-----		4.6
2284	EU10/2011, EN13130-1	4.47		-----	reported in mg/L?	7.5
2297		7.86		-----	reported in mg/L?	7.5
2352	EUR24815 EN2011, EN13130-1	7.12		-----	reported in mg/L?	7.5
2353	EN13130-32	3.7011		-----	reported in mg/L?	7.5
2357		-----		-----		
2372	EUR24815 EN2011, EN13130-1	3.48	ex	-----	See § 4.1	6.0
2375	CEN/TS13130-23, EN13130-1	8.275	ex	-----	See § 4.1	13.6
2403		8.94		-----	reported in mg/L?	7.5
2475		3.55		-----	reported in mg/L?	7.6
2488		-----		-----		
2495	ISO4614	0.96		-----		6.0
2504		3.39		-----	reported in mg/L?	7.5
2510	EN13130-1	4.80		-----		6.0
3146	EN13130	2.80		-----	reported in mg/L?	7.6
3151	EN13130-1	2.89		-----		6.0
3153	EN13130-1	3.01		-----		4.4
3185	EU10/2011, EN13130-1, EN13130-23	3.01		-----		4.6
3209	EN13130-23	4.49		-----	reported in mg/L?	7.5
3214	CEN/TS13130	3.39		-----	reported in mg/L?	7.5
3218	EN13130-23	3.66		-----	reported in mg/L?	7.5
3228	ISO14184-1	3.20		-----	reported in mg/L?	7.4
3233	EUR24815 EN2011, EU10/2011	6.01		-----	reported in mg/L?	7.5
					theoretical results using 6 as conversion factor:	
	normality	not OK			OK	
	n	29			29	
	outliers	1			1	
	mean (n)	3.770			3.456	
	st.dev. (n)	1.8989			1.4388	
	R(calc.)	5.317			4.029	
	R(Horwitz)	1.383			1.285	

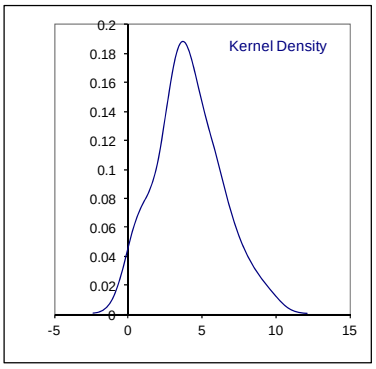




Concentration of formaldehyde in simulant 3rd contact on sample #12112; results in mg/L

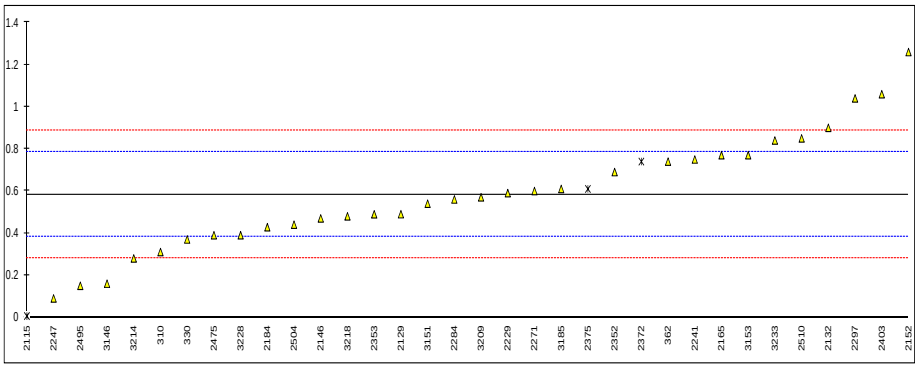
lab	method	Value	mark	z(targ)	remarks
310	EN1186-1	2.34		-3.63	
330		2.77		-2.87	
362		5.54		2.07	
2115		0.46	ex	-6.98	See § 4.1
2129	ISO14184-1	3.67		-1.26	
2132	CEN/TS13130-23	6.77		4.27	
2146	Acetyl/Acetone	3.5		-1.56	
2152	CEN/TS13130-23	9.42		8.99	
2156		-----		-----	
2165	EN13130-23	5.60		2.18	
2184	EN13130-23	3.34		-1.85	
2190		-----		-----	
2229	EUR24815 EN2011	4.43		0.09	
2241		5.61		2.20	
2247	UV Vis Spec.	0.70		-6.56	
2271		4.47		0.16	
2284	EU10/2011, EN13130-1	4.23		-0.26	
2297		7.80		6.10	
2352	EUR24815 EN2011, EN13130-1	5.15		1.38	
2353	EN13130-32	3.6702		-1.26	
2357		-----		-----	
2372	EUR24815 EN2011, EN13130-1	3.18	ex	-2.14	See § 4.1
2375	CEN/TS13130-23, EN13130-1	0.332	ex	-7.21	See § 4.1
2403		7.96		6.39	
2475		2.94		-2.56	
2488		-----		-----	
2495	ISO4614	1.13		-5.79	
2504		3.30		-1.92	
2510	EN13130-1	6.56		3.89	
3146	EN13130	1.22		-5.63	
3151	EN13130-1	4.02		-0.64	
3153	EN13130-1	5.81		2.55	
3185	EU10/2011, EN13130-1, EN13130-23	4.61		0.41	
3209	EN13130-23	4.25		-0.23	
3214	CEN/TS13130	2.09		-4.08	
3218	EN13130-23	3.58		-1.42	
3228	ISO14184-1	2.91		-2.62	
3233	EUR24815 EN2011, EU10/2011	6.27		3.37	
	normality	OK			
	n	31			
	outliers	0			
	mean (n)	4.376			
	st.dev. (n)	2.0507			
	R(calc.)	5.742			
	R(Horwitz)	1.570			

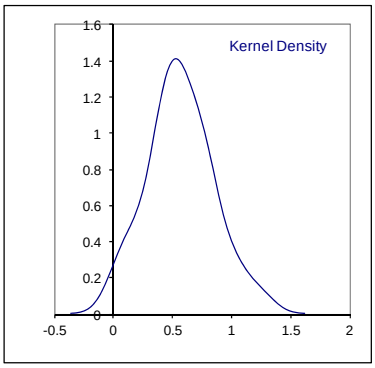




Specific Migration of formaldehyde 3rd contact on sample #12112; results in mg/dm^2

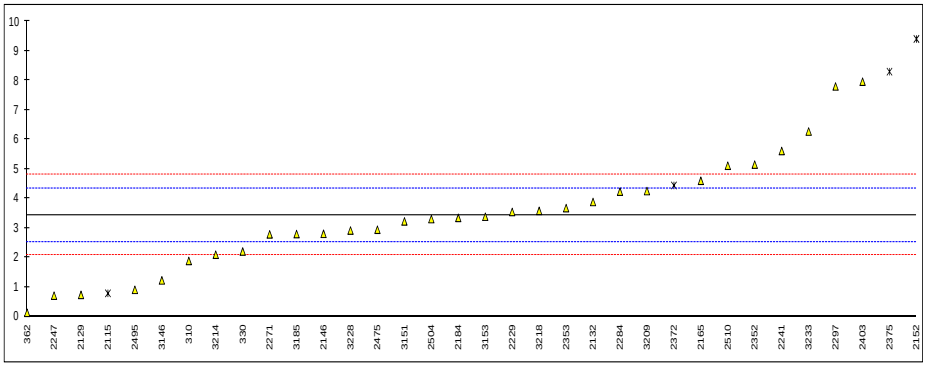
lab	method	value	mark	z(targ)	remarks
310	EN1186-1	0.31		-2.70	
330		0.37		-2.11	
362		0.74	C	1.55	first reported 7.39
2115		0.0061	ex	-5.70	See § 4.1
2129	ISO14184-1	0.49		-0.92	
2132	CEN/TS13130-23	0.90		3.13	
2146	Acetyl/Acetone	0.47		-1.12	
2152	CEN/TS13130-23	1.26		6.69	
2156		-----		-----	
2165	EN13130-23	0.77		1.85	
2184	EN13130-23	0.428		-1.53	
2190		-----		-----	
2229	EUR24815 EN2011	0.59		0.07	
2241		0.75		1.65	
2247	UV Vis Spec.	0.09		-4.87	
2271		0.60		0.17	
2284	EU10/2011, EN13130-1	0.56		-0.23	
2297		1.04		4.51	
2352	EUR24815 EN2011, EN13130-1	0.69		1.06	
2353	EN13130-32	0.4894		-0.93	
2357		-----		-----	
2372	EUR24815 EN2011, EN13130-1	0.74	ex	1.55	See § 4.1
2375	CEN/TS13130-23, EN13130-1	0.610	ex	0.27	See § 4.1
2403		1.06		4.71	
2475		0.39		-1.91	
2488		-----		-----	
2495	ISO4614	0.15		-4.28	
2504		0.44		-1.41	
2510	EN13130-1	0.85		2.64	
3146	EN13130	0.16		-4.18	
3151	EN13130-1	0.54		-0.43	
3153	EN13130-1	0.77		1.85	
3185	EU10/2011, EN13130-1, EN13130-23	0.61		0.27	
3209	EN13130-23	0.57		-0.13	
3214	CEN/TS13130	0.28		-3.00	
3218	EN13130-23	0.48		-1.02	
3228	ISO14184-1	0.39		-1.91	
3233	EUR24815 EN2011, EU10/2011	0.84		2.54	
	normality	OK			
	n	31			
	outliers	3			
	mean (n)	0.583			
	st.dev. (n)	0.2739			
	R(calc.)	0.767			
	R(Horwitz)	0.283			

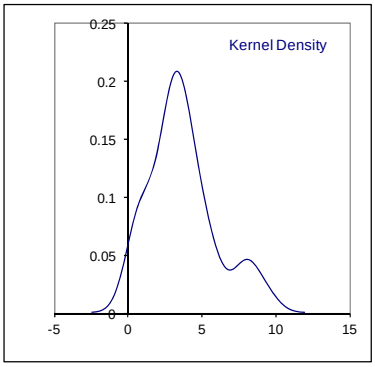




Specific Migration of formaldehyde 3rd contact on sample #12112; results in mg/kg

lab	method	value	mark	z(targ)	remarks	conversion factor used
310	EN1186-1	1.88		-----		6.1
330		2.2		-----		5.9
362		0.12	ex, C	-----	See § 4.1, fr. 1.23	0.2
2115		0.78	ex	-----	See § 4.1	127.9
2129	ISO14184-1	0.73		-----		1.5
2132	CEN/TS13130-23	3.88		-----		4.3
2146	Acetyl/Acetone	2.8		-----		6.0
2152	CEN/TS13130-23	9.41	D(0.05)	-----	reported in mg/L?	7.5
2156		-----		-----		
2165	EN13130-23	4.60		-----		6.0
2184	EN13130-23	3.34		-----	reported in mg/L?	7.8
2190		-----		-----		
2229	EUR24815 EN2011	3.54		-----		6.0
2241		5.61		-----	reported in mg/L?	7.5
2247	UV Vis Spec.	0.70		-----	reported in mg/L?	7.8
2271		2.78		-----		4.6
2284	EU10/2011, EN13130-1	4.23		-----	reported in mg/L?	7.6
2297		7.80		-----	reported in mg/L?	7.5
2352	EUR24815 EN2011, EN13130-1	5.15		-----	reported in mg/L?	7.5
2353	EN13130-32	3.6702		-----	reported in mg/L?	7.5
2357		-----		-----		
2372	EUR24815 EN2011, EN13130-1	4.44	ex	-----	See § 4.1	6.0
2375	CEN/TS13130-23, EN13130-1	8.300	ex	-----	See § 4.1	13.6
2403		7.96		-----	reported in mg/L?	7.5
2475		2.94		-----	reported in mg/L?	7.5
2488		-----		-----		
2495	ISO4614	0.90		-----		6.0
2504		3.30		-----	reported in mg/L?	7.5
2510	EN13130-1	5.11		-----		6.0
3146	EN13130	1.22		-----	reported in mg/L?	7.6
3151	EN13130-1	3.22		-----		6.0
3153	EN13130-1	3.38		-----		4.4
3185	EU10/2011, EN13130-1, EN13130-23	2.79		-----		4.6
3209	EN13130-23	4.25		-----	reported in mg/L?	7.5
3214	CEN/TS13130	2.09		-----	reported in mg/L?	7.5
3218	EN13130-23	3.58		-----	reported in mg/L?	7.5
3228	ISO14184-1	2.91		-----	reported in mg/L?	7.5
3233	EUR24815 EN2011, EU10/2011	6.27		-----	reported in mg/L?	7.5
					theoretical results using 6 as conversion factor:	
	normality	OK			OK	
	n	29			29	
	outliers	1			1	
	mean (n)	3.546			3.331	
	st.dev. (n)	1.8357			1.4965	
	R(calc.)	5.140			4.190	
	R(Horwitz)	1.313			1.245	





APPENDIX 2

Test conditions when selected by participants

lab	type of simulant	amount of simulant in ml	exposure time in hrs	exposure temperature in°C	contact surface in dm^2	reporting unit
310	3% HAc	300	2	70	1.9	mg/kg
330	----	----	----	----	----	----
362	----	----	----	----	----	----
2115	----	----	----	----	----	----
2129	3% HAc	500	2	70	3.0	mg/kg
2132	3% HAc	200	0.5 - 4	70 - 40	1.5	mg/kg
2146	3% HAc	500	2	70	2.75	mg/kg
2152	----	----	----	----	----	----
2165	3% HAc	200	2	70	1.5	mg/kg, mg/dm^2
2184	3% HAc	200	2	70	1.56	mg/kg
2190	----	----	----	----	----	----
2229	3% HAc	200	2.0	70.0	1.48	mg/kg
2241	----	----	----	----	----	----
2247	3% HAc	200	2.0	70	1.5	mg/L, mg/kg, mg/dm^2
2271	3% HAc	200	2.0	70.0	1.53	mg/kg
2284	3% HAc	500	2	70	4.25	mg/kg
2297	----	----	----	----	----	----
2352	----	----	----	----	----	----
2353	3% HAc	200	2	70	2.5-3.0	mg/kg, mg/dm^2
2357	----	----	----	----	----	----
2372	3% HAc	500	2	70	2.1377	mg/kg
2375	3% HAc	200	2	70	1.5	mg/kg
2403	----	----	----	----	----	----
2475	3% HAc, olive oil, EtOH	490	2	70	1.43	mg/kg
2495	3% HAc	400	6	70	1.29	mg/kg
2504	----	----	----	----	----	----
2510	3% HAc	480	3x2	70	2.37	mg/kg
3146	3% HAc	600	2	70	3.4	mg/kg
3151	3% HAc	200	2	70	1.5	mg/kg
3153	3% HAc	500	2	70	2.4	mg/kg
3185	3% HAc	500	2	70	2.5	mg/kg
3209	3% HAc	450	2	70	2.5	mg/kg
3214	3% HAc	200	2.0	70.0	1.5	mg/kg
3218	3% HAc	360	2.0	70.0	1.9	mg/kg
3228	----	----	----	----	----	----
3233	3% HAc	500	2.00	70.00	2.40	mg/kg

APPENDIX 3

Number of participating laboratories per country

1 lab in BULGARIA

1 lab in FINLAND

4 labs in FRANCE

3 labs in GERMANY

5 labs in HONG KONG

1 lab in INDIA

1 lab in IRELAND

2 labs in ITALY

1 lab in MALAYSIA

13 labs in P.R. of CHINA

2 labs in TAIWAN R.O.C.

1 lab in THAILAND

1 lab in THE NETHERLANDS

2 labs in TURKEY

APPENDIX 4

Abbreviations:

C = final result after checking of first reported suspect result
D(0.01) = outlier in Dixon's outlier test
D(0.05) = straggler in Dixon's outlier test
G(0.01) = outlier in Grubbs' outlier test
G(0.05) = straggler in Grubbs' outlier test
DG(0.01) = outlier in Double Grubbs' outlier test
DG(0.05) = straggler in Double Grubbs' outlier test
n.a. = not applicable
f.r. = first reported

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, January 2010
2 EN13130-1 Materials and articles in contact with foodstuffs – Plastics substances subject to limitation - Part 1: Guide to test methods for the specific migration of substances from plastics to foods and food simulants and the determination of substances in plastics and the selection of conditions of exposure to food simulants
3 CEN/TS 13130-23 Materials and articles in contact with foodstuffs – Plastics substances subject to limitation - Part 23: Determination of formaldehyde and hexamethylenetetramine in food simulants
4 ASTM E178-02
5 ASTM E1301-03
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