Results of Proficiency Test
Specific migration (fcm)
September 2013

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. This PT was repeated in 2013.
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 2013, 39 laboratories from 12 different countries participated (See appendix 4).
In this report, the results of the 2013 proficiency test are presented and discussed.

2 SET-UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. 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 #13184, and to prescribe a number of test conditions (type of simulant, bowl volume, exposure time and temperature) to be used. Participants were also requested to report the test conditions that the laboratory would have used in case these were not prescribed by iis.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in accordance with ISO/IEC 17043:2010, (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie, see also www.RVA.nl). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

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

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

A batch of 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 7 stratified randomly selected bowls.

	Specific Migration in mg/kg #13184
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 #13184

The repeatability for Specific Migration on the seven samples #13184 is in agreement with the repeatability of the laboratory performing the tests.

Therefore, homogeneity of the samples #13184 was assumed.

To each of the participating laboratories one sample #13184 was sent on September 25, 2013.

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.

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.

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 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 5; 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

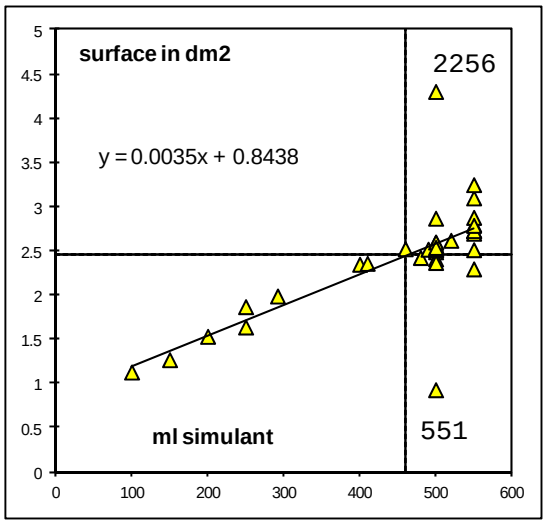
4 EVALUATION

In this interlaboratory study, no problems were encountered with the dispatch of the samples. None of the participants reported test results after the final reporting date. Two participants did not report any test results at all. Thus, 37 of the 39 participants submitted analysis results. These 37 laboratories reported 212 numerical test results. Observed were 10 outlying test results, which is 4.7%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

A non Gaussian distribution was only observed for the reported specific migration results in mg/dm^2 for the third contact. Therefore this statistical evaluation should be used with due care.

For the determination of Specific Migration, several standardised test methods 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 reproducibilities from the Horwitz equation.

Two laboratories (551 and 2256) appeared to have made an error in either the contact surface determination or in the volume determination of the simulant used. The ratio volume of simulant per contact surface in ml/dm^2 is significantly deviating from the other reported data (see below graph).



Furthermore the migration results of laboratory 2190 were deviating due to an unresolved error. The result in mg/dm^2 is deviating with a factor two from the theoretical value. Either the contact surface was wrongly reported or the simulant volume was wrongly reported or a calculation error was made.
Therefore the reported test results of the three laboratories 551, 2190 and 2256 were excluded from the data 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)
Specific migration, 1st contact	mg/dm^2	33	0.5	0.8	0.2
Specific migration, 1st contact	mg/kg	32	2.6	4.2	1.0
Specific migration, 2nd contact	mg/dm^2	31	0.4	0.4	0.2
Specific migration, 2nd contact	mg/kg	30	2.0	2.2	0.8
Specific migration, 3rd contact	mg/dm^2	31	0.3	0.4	0.2
Specific migration, 3rd contact	mg/kg	29	1.9	2.9	0.8

Table 2: performance overview for sample #13184

4.2 EVALUATION

No significant differences were observed between the results of the 1st, the 2nd and 3rd contact.

Specific migration of formaldehyde in mg/dm^2:

This determination may be very problematic. A wide range of test results was reported, e.g. for the 3rd contact: 0.04 – 1.50 mg/dm^2. In total five statistical outliers were detected. The three calculated reproducibilities, after rejection of the statistical outliers, are all 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 may be very problematic. A wide range of test results was reported, e.g. for the 3rd contact: 0.1 – 7.56 mg/kg. In total five statistical outliers were detected. The factor used for the conversion from mg/dm^2 to mg/kg varies from 1.9 – 8.6. Only 13 laboratories appeared to have used 6 as a conversion factor. Another 18 laboratories reported equal results for mg/kg and for results in mg/L (!) and consequently did not do a conversion from mg/dm^2 to mg/kg. 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). Also EN1186-1 and EN1186-9 were reported. These methods all describe identical procedures and therefore no differences in the test results are expected.
The reported details that were used by the participants (volume of simulant and contact surface) as well as the actual formaldehyde concentrations measured in the simulant for each of the three migration steps are listed in appendix 2.

5 CONCLUSIONS

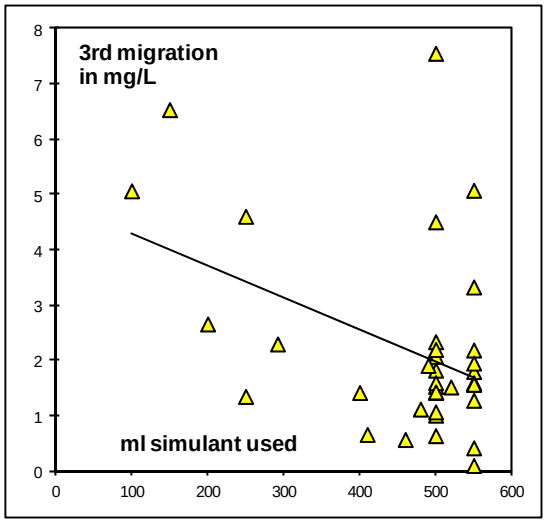
Before the start of this PT it was assumed that a wide range of test results would be reported when the choice of the test conditions would have been left to the participating laboratories. Therefore a set of predetermined test conditions was given together with the instructions to all participants. These preset conditions were:

Sample ID	**#13184**
Simulant	3% acetic acid in water
Bowl volume	550 ml
Exposure time	2.0 hrs
Exposure temperature	70.0 °C
Migration method	Article filling

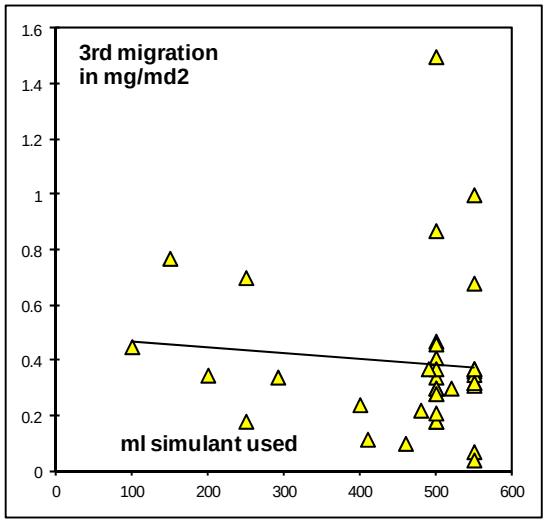
Table 3: preset test conditions used in this PT

Not only a migration result was to be reported, but the participants were requested to report also the intermediate formaldehyde concentration in the simulant. The reported formaldehyde concentrations are listed in appendix 2. Using these intermediate test results it was possible to check all calculations and corrections done by the laboratories.
This revealed that initially indeed some calculation errors were present. Several laboratories corrected these calculation errors; see the original and the revised test results in appendix 1.

The intermediate test results (the formaldehyde concentrations in mg/L) cannot be evaluated in terms of z-scores because the volume of simulant used is not a fixed value, but the volume varies per laboratory and there is a correlation between the amount of simulant used and the formaldehyde concentration measured:



correlation between the formaldehyde concentration and volume of simulant used



correlation between the specific migration and volume of simulant used

When the two above correlation graphs are compared (between the intermediate formaldehyde concentration and the volume of simulant used and between the migration in mg/dm^2 and the volume of simulant used, both for the third migration step), the decrease in correlation is clearly visible. This was to be expected. However still some correlation may be present.
The amount of simulant used varied from 100 - 550 ml. This is unexpected because, when strictly following the reported test methods, 500 – 550 ml would be used:

Paragraphs 6.2, 6.3 and 7.1 of EN1186-9 mention that “a specimen should be filled to the nominal volume, if known, or to 5 mm from the top”. No nominal volume was given in this PT and therefore it was expected that the laboratories using EN1186-9 would use approx 500 ml of simulant. Paragraph 19.2 of EN13130-1 mentions that “Test specimens are filled with the food simulant or test medium, with the minimum of headspace”. Therefore it was expected that the laboratories using EN13130-1 would use approx 550 ml of simulant.

It may be interesting to know whether the large variance in simulant volume may be of influence on the spread of Specific Migration test results, in other words whether the residual correlation may be significant. In below table 5, the evaluations of the test results based on a migration with 500 ml are presented and compared with all reported test results.

	with simulant volumes as reported	only for results with 500 ml of simulant used
Specific migration 1st step in mg/dm^2	0.45	0.53
Specific migration 2nd step in mg/dm^2	0.36	0.39
Specific migration 3rd step in mg/dm^2	0.33	0.33

Table 4: influence of volume of simulant used on Specific Migration

From this evaluation it may be clear that the influence of the use of different volumes of simulant may be significant only during the first two migration steps.

The Specific Migration results in mg/kg show a larger spread than the results for Specific Migration in mg/dm^2. Upon investigation for the reason of the increase in spread, it was found that a large number of the laboratories (approx 50%) did not calculate the specific migration in mg/kg from the migration in mg/dm^2 using the conventional factor of 6 dm^2/kg cfr. EN13130-1:2004, paragraphs 4.7, 10.2 and 13.1.1. No less than 19 laboratories reported equal results for mg/kg and for results in mg/L, which is statistically not possible. See also Annex 1 of 2002/72/EC (L220/22) for this requirement.

It is remarkable to see that about 30% of the laboratories the results for 1st migration > 2nd migration > 3rd migration, while for another 20% the results for 1st migration < 2nd migration < 3rd migration. Only 55% of the laboratories reported 3rd migration < 1st migration, see appendix 2. No explanation is available to explain this phenomenon. It is unknown whether details like for example (not) cleaning the bowl before use and the (not) preheating of the simulant before use, may explain this.

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 used 3% acetic acid, except one laboratory.
- All participants would have used an exposure of 2 hrs and an exposure temperature of 70°C
- 74 % of the participants would have reported the migration in mg/kg only.

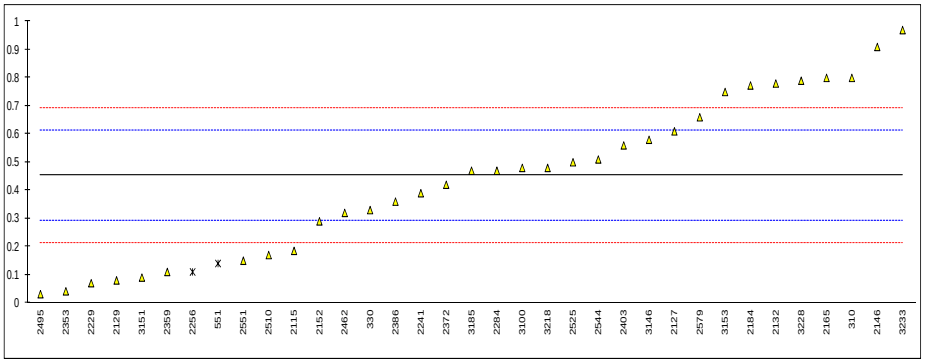
Each laboratory has to evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and the quality of the analytical results.

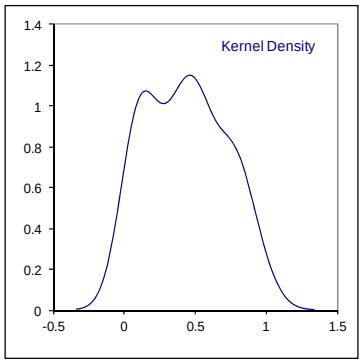
APPENDIX 1

Specific Migration of formaldehyde 1st contact on sample #13184; results in mg/dm^2

lab	method	value	mark	z(targ)	remarks
310	EN13130-1	0.80		4.26	
330	EN13130-1	0.33		-1.50	
551		0.14	ex	-3.83	see §4.0
2115		0.185		-3.28	
2127		0.61		1.93	
2129		0.08	C	-4.57	first reported:0.15
2132	EN13130-1, JRC EUR24815 EN 2011	0.78		4.02	
2146	EUR24815 EN 2011	0.91	C	5.61	first reported:0.96
2152	CEN/TS131310-23	0.29		-1.99	
2165		0.80		4.26	
2184	EN13130-23	0.773		3.93	
2190		<0.04		<-5.06	false negative?
2229	EUR24815 EN 2011	0.07		-4.69	
2241	CEN/TS131310-23, EUR24815 EN 2011	0.39		-0.77	
2256	EN1186-1	0.11	ex	-4.20	see §4.0
2284	EN13130-1, EN1186-9	0.47		0.22	
2309		-----		-----	
2353	EN13130-1	0.0412		-5.04	
2359		0.11	C	-4.20	first reported:109.00
2372	EU 10/2011	0.42		-0.40	
2386	EN13130-1	0.36		-1.13	
2403	EN13130-1	0.56		1.32	
2462	EN13130-1	0.32		-1.62	
2495	ISO4614	0.031		-5.17	
2510	EN13130-1	0.17		-3.46	
2525	EN1541	0.50		0.58	
2544	EN1186-9	0.51		0.71	
2551	in house	0.15		-3.71	
2579	EN13130-1	0.66		2.54	
3100		0.48		0.34	
3146	EN13130-1	0.58		1.56	
3151	EN13130-1	0.09		-4.44	
3153	EN13130-1	0.75		3.65	
3154		-----		-----	
3172		-----		-----	
3185	EU10/2011, EU284/2011, EN13130-1	0.47		0.22	
3218	EN1186-9	0.48		0.34	
3228		0.79		4.14	
3233	EUR24815 EN 2011, EU10/2011	0.97		6.35	

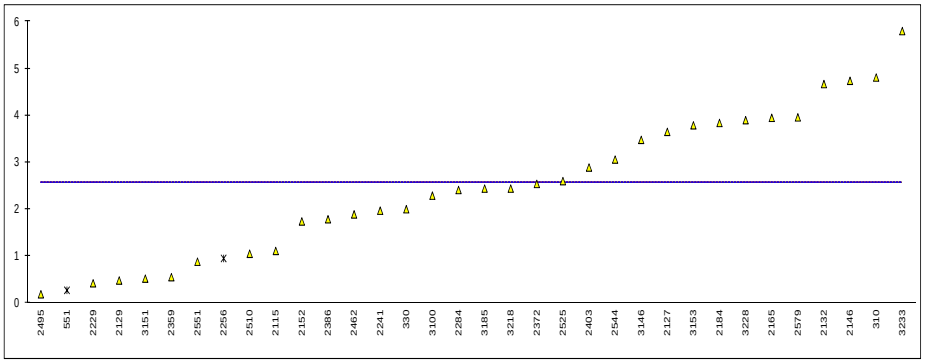
normality	OK
n	33
outliers	0 +2excl.
mean (n)	0.452
st.dev. (n)	0.2756
R(calc.)	0.772
R(Horwitz)	0.228

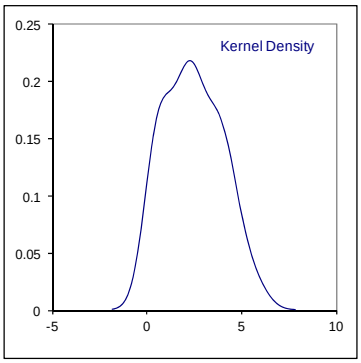




Specific Migration of formaldehyde 1st contact on sample #13184; results in mg/kg

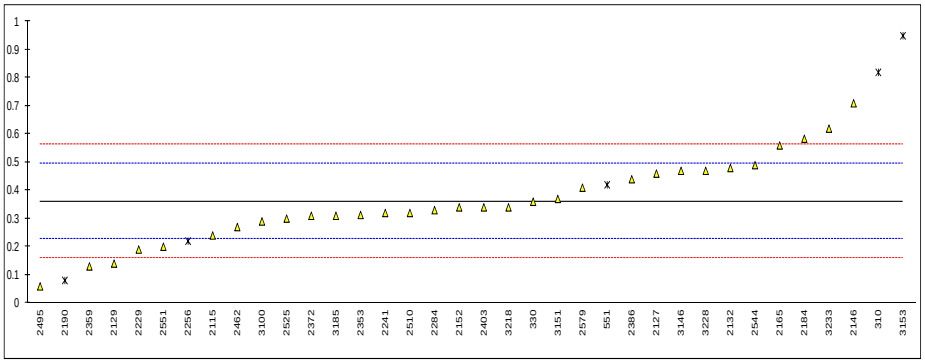
lab	method	value	mark	z(targ)	remarks	factor kg/dm^2 used
310	EN13130-1	4.81		-----		6.0
330	EN13130-1	2.0		-----		6.1
551		0.27	ex	-----	see §4.0	1.9
2115		1.11		-----		6.0
2127		3.65		-----		6.0
2129		0.48	C	-----	first reported:0.90	6.0
2132	EN13130-1, JRC EUR24815 EN 2011	4.67		-----		6.0
2146	EUR24815 EN 2011	4.74		-----	identical to mg/L	5.2
2152	CEN/TS131310-23	1.74		-----		6.0
2165		3.95		-----	identical to mg/L	4.9
2184	EN13130-23	3.84		-----	identical to mg/L	5.0
2190		<0.1		-----	false negative?	2.9
2229	EUR24815 EN 2011	0.42		-----		6.0
2241	CEN/TS131310-23, EUR24815 EN 2011	1.97		-----	identical to mg/L	5.1
2256	EN1186-1	0.95	ex	-----	see §4.0	8.6
2284	EN13130-1, EN1186-9	2.41		-----	identical to mg/L	5.1
2309		-----		-----		----
2353		-----		-----		----
2359		0.55		-----	identical to mg/L	5.0
2372	EU 10/2011	2.54		-----		6.0
2386	EN13130-1	1.786		-----	identical to mg/L	5.0
2403	EN13130-1	2.89		-----	identical to mg/L	5.2
2462	EN13130-1	1.89		-----	identical to mg/L	5.9
2495	ISO4614	0.186		-----		6.0
2510	EN13130-1	1.05		-----		6.2
2525	EN1541	2.60		-----	identical to mg/L	5.2
2544	EN1186-9	3.06		-----		6.0
2551	in house	0.88		-----	identical to mg/L	5.9
2579	EN13130-1	3.96		-----		6.0
3100		2.29		-----	identical to mg/L	4.8
3146	EN13130-1	3.48		-----		6.0
3151	EN13130-1	0.52		-----		5.8
3153	EN13130-1	3.79		-----	identical to mg/L	5.1
3154		-----		-----		----
3172		-----		-----		----
3185	EU10/2011, EU284/2011, EN13130-1	2.44		-----	identical to mg/L	5.2
3218	EN1186-9	2.44		-----	identical to mg/L	5.1
3228		3.90		-----	identical to mg/L	4.9
3233	EUR24815 EN 2011, EU10/2011	5.80		-----		6.0
					only with 6.0 used as conversion factor:	
	normality	OK			OK	
	n	32			16	
	outliers	0	+2 excl.		0	
	mean (n)	2.558			2.467	
	st.dev. (n)	1.4860			1.7924	
	R(calc.)	4.161			5.019	
	R(Horwitz)	0.995			0.965	

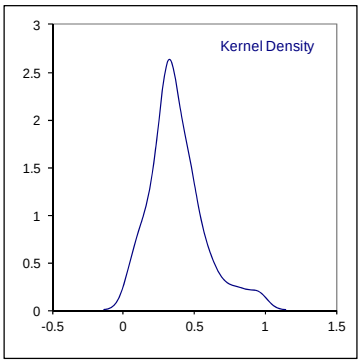




Specific Migration of formaldehyde 2nd contact on sample #13184; results in mg/dm^2

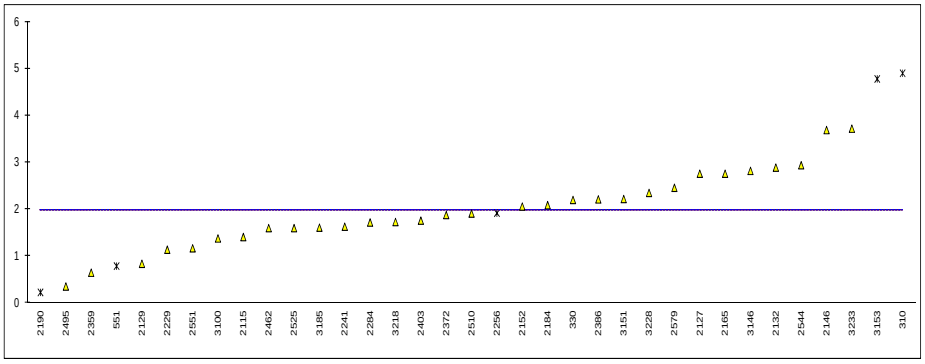
lab	method	value	mark	z(targ)	remarks
310	EN13130-1	0.82	DG(0.05)	6.84	
330	EN13130-1	0.36		0.00	
551		0.42	ex	0.89	see §4.0
2115		0.24		-1.79	
2127		0.46		1.49	
2129		0.14	C	-3.28	first reported: 0.25
2132	EN13130-1, JRC EUR24815 EN 2011	0.48		1.78	
2146	EUR24815 EN 2011	0.71	C	5.21	first reported: 0.75
2152	CEN/TS131310-23	0.34		-0.30	
2165		0.56		2.97	
2184	EN13130-23	0.584		3.33	
2190		0.08	ex	-4.17	see § 4.0
2229	EUR24815 EN 2011	0.19		-2.53	
2241	CEN/TS131310-23, EUR24815 EN 2011	0.32		-0.60	
2256	EN1186-1	0.22	ex	-2.09	see § 4.0
2284	EN13130-1, EN1186-9	0.33		-0.45	
2309		-----		-----	
2353	EN13130-1	0.3128		-0.71	
2359		0.13	C	-3.43	first reported:128.14
2372	EU 10/2011	0.31		-0.75	
2386	EN13130-1	0.44		1.19	
2403	EN13130-1	0.34		-0.30	
2462	EN13130-1	0.27		-1.34	
2495	ISO4614	0.059		-4.48	
2510	EN13130-1	0.32		-0.60	
2525	EN1541	0.30		-0.90	
2544	EN1186-9	0.49	C	1.93	
2551	in house	0.20		-2.38	
2579	EN13130-1	0.41		0.74	
3100		0.29		-1.04	
3146	EN13130-1	0.47		1.63	
3151	EN13130-1	0.37		0.15	
3153	EN13130-1	0.95	DG(0.05)	8.78	
3154		-----		-----	
3172		-----		-----	
3185	EU10/2011, EU284/2011, EN13130-1	0.31		-0.75	
3218	EN1186-9	0.34		-0.30	
3228		0.47		1.63	
3233	EUR24815 EN 2011, EU10/2011	0.62		3.87	
	normality	OK			
	n	31			
	outliers	2	+3 excl		
	mean (n)	0.360			
	st.dev. (n)	0.1466			
	R(calc.)	0.410			
	R(Horwitz)	0.188			

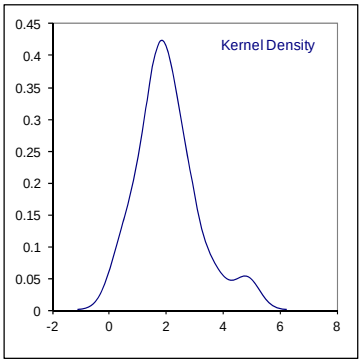




Specific Migration of formaldehyde 2nd contact on sample #13184; results in mg/kg

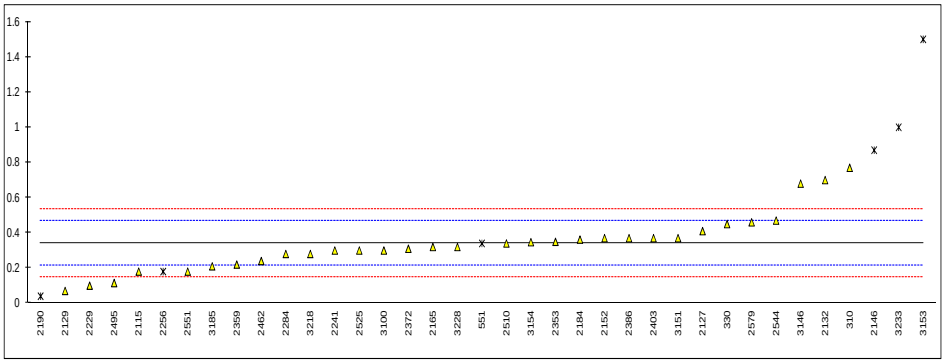
lab	method	value	mark	z(targ)	remarks	factor kg/dm^2 used
310	EN13130-1	4.90	DG(0.05)	-----		6.0
330	EN13130-1	2.2		-----		6.1
551		0.79	ex	-----	see § 4.0	1.9
2115		1.41		-----		6.0
2127		2.76		-----		6.0
2129		0.84	C	-----	first reported: 1.50	6.0
2132	EN13130-1, JRC EUR24815 EN 2011	2.89		-----		6.0
2146	EUR24815 EN 2011	3.69		-----	identical to mg/L	5.2
2152	CEN/TS131310-23	2.06		-----		6.0
2165		2.76		-----	identical to mg/L	4.9
2184	EN13130-23	2.09		-----	identical to mg/L	5.0
2190		0.23	ex	-----	see § 4.0	2.9
2229	EUR24815 EN 2011	1.14		-----		6.0
2241	CEN/TS131310-23, EUR24815 EN 2011	1.63		-----	identical to mg/L	5.1
2256	EN1186-1	1.92	ex	-----	see § 4.0	8.6
2284	EN13130-1, EN1186-9	1.72		-----	identical to mg/L	5.1
2309		-----		-----		----
2353		-----		-----		----
2359		0.65		-----	identical to mg/L	5.0
2372	EU 10/2011	1.88		-----		6.0
2386	EN13130-1	2.212		-----	identical to mg/L	5.0
2403	EN13130-1	1.76		-----	identical to mg/L	5.2
2462	EN13130-1	1.60		-----	identical to mg/L	5.9
2495	ISO4614	0.356		-----		6.0
2510	EN13130-1	1.91		-----		6.2
2525	EN1541	1.60		-----	identical to mg/L	5.2
2544	EN1186-9	2.94		-----		6.0
2551	in house	1.17		-----	identical to mg/L	5.9
2579	EN13130-1	2.46		-----		6.0
3100		1.38		-----	identical to mg/L	4.8
3146	EN13130-1	2.82		-----		6.0
3151	EN13130-1	2.22		-----		5.8
3153	EN13130-1	4.78	DG(0.05)	-----	identical to mg/L	5.1
3154		-----		-----		----
3172		-----		-----		----
3185	EU10/2011, EU284/2011, EN13130-1	1.61		-----	identical to mg/L	5.2
3218	EN1186-9	1.73		-----	identical to mg/L	5.1
3228		2.35		-----	identical to mg/L	4.9
3233	EUR24815 EN 2011, EU10/2011	3.72		-----		6.0
					only with 6.0 used as conversion factor:	
	normality	OK			OK	
	n	30			15	
	outliers	2	+ 3 excl.		1	
	mean (n)	1.985			2.107	
	st.dev. (n)	0.8022			0.8934	
	R(calc.)	2.246			2.502	
	R(Horwitz)	0.802			0.844	

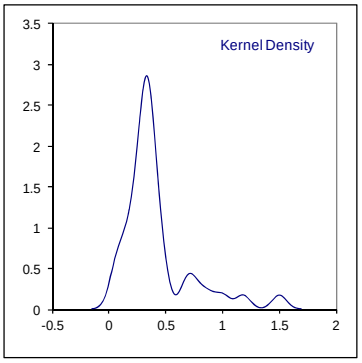




Specific Migration of formaldehyde 3rd contact on sample #13184; results in mg/dm^2

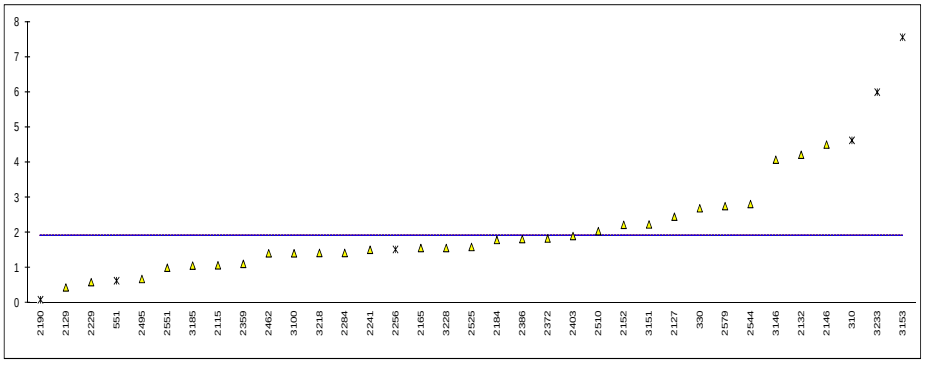
lab	method	Value	mark	z(targ)	remarks
310	EN13130-1	0.77		6.72	
330	EN13130-1	0.45		1.72	
551		0.34	ex	0.00	see §4.0
2115		0.18		-2.50	
2127		0.41		1.09	
2129		0.07	C	-4.22	first reported: 0.14
2132	EN13130-1, JRC EUR24815 EN 2011	0.70		5.62	
2146	EUR24815 EN 2011	0.87	DG(0.05)	8.28	first reported:0.92
2152	CEN/TS131310-23	0.37		0.47	
2165		0.32		-0.31	
2184	EN13130-23	0.362		0.34	
2190		0.04	ex	-4.69	see §4.0
2229	EUR24815 EN 2011	0.10		-3.75	
2241	CEN/TS131310-23, EUR24815 EN 2011	0.30		-0.63	
2256	EN1186-1	0.18	ex	-2.50	see §4.0
2284	EN13130-1, EN1186-9	0.28		-0.94	
2309		-----		-----	
2353	EN13130-1	0.3492		0.14	
2359		0.22	C	-1.88	first reported:220.86
2372	EU 10/2011	0.31		-0.47	
2386	EN13130-1	0.37		0.47	
2403	EN13130-1	0.37		0.47	
2462	EN13130-1	0.24		-1.56	
2495	ISO4614	0.115		-3.52	
2510	EN13130-1	0.34		0.00	
2525	EN1541	0.30		-0.63	
2544	EN1186-9	0.47		2.03	
2551	in house	0.18		-2.50	
2579	EN13130-1	0.46		1.87	
3100		0.30		-0.63	
3146	EN13130-1	0.68		5.31	
3151	EN13130-1	0.37		0.47	
3153	EN13130-1	1.50	G(0.01)	18.12	
3154		0.347	C	0.11	first reported:1.734
3172		-----		-----	
3185	EU10/2011, EU284/2011, EN13130-1	0.21		-2.03	
3218	EN1186-9	0.28		-0.94	
3228		0.32		-0.31	
3233	EUR24815 EN 2011, EU10/2011	1.00	DG(0.05)	10.31	
	normality	not OK			
	n	31			
	outliers	3	+3 excl.		
	mean (n)	0.340			
	st.dev. (n)	0.1602			
	R(calc.)	0.449			
	R(Horwitz)	0.179			

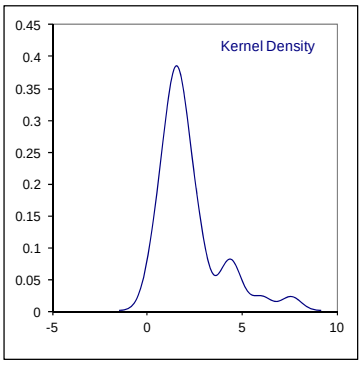




Specific Migration of formaldehyde 3rd contact on sample #13184; results in mg/kg

lab	method	Value	Mark	z(targ)	remarks	factor kg/dm^2 used
310	EN13130-1	4.63	DG(0.05)	-----		6.0
330	EN13130-1	2.7		-----		6.1
551		0.64	ex	-----	see §4.0	1.9
2115		1.08		-----		6.0
2127		2.46		-----		6.0
2129		0.45	C	-----	First reported: 0.84	6.0
2132	EN13130-1, JRC EUR24815 EN 2011	4.22		-----		6.0
2146	EUR24815 EN 2011	4.51		-----	identical to mg/L	5.2
2152	CEN/TS131310-23	2.23		-----		6.0
2165		1.57		-----	identical to mg/L	4.9
2184	EN13130-23	1.80		-----	identical to mg/L	5.0
2190		0.1	ex	-----	see §4.0	2.9
2229	EUR24815 EN 2011	0.60		-----		6.0
2241	CEN/TS131310-23, EUR24815 EN 2011	1.52		-----		5.1
2256	EN1186-1	1.53	ex	-----	see §4.0	8.6
2284	EN13130-1, EN1186-9	1.43		-----	identical to mg/L	5.1
2309		-----		-----		----
2353		-----		-----		----
2359		1.12		-----	identical to mg/L	5.0
2372	EU 10/2011	1.84		-----		6.0
2386	EN13130-1	1.824		-----	identical to mg/L	5.0
2403	EN13130-1	1.91		-----	identical to mg/L	5.2
2462	EN13130-1	1.42		-----	identical to mg/L	5.9
2495	ISO4614	0.689		-----		6.0
2510	EN13130-1	2.05		-----		6.2
2525	EN1541	1.60		-----	identical to mg/L	5.2
2544	EN1186-9	2.82		-----		6.0
2551	in house	1.01		-----	identical to mg/L	5.9
2579	EN13130-1	2.76		-----		6.0
3100		1.42		-----	identical to mg/L	4.8
3146	EN13130-1	4.08		-----		6.0
3151	EN13130-1	2.24		-----		5.8
3153	EN13130-1	7.56	G(0.05)	-----	identical to mg/L	5.1
3154		-----		-----		----
3172		-----		-----		----
3185	EU10/2011, EU284/2011, EN13130-1	1.07		-----	identical to mg/L	5.2
3218	EN1186-9	1.43		-----	identical to mg/L	5.1
3228		1.57		-----	identical to mg/L	4.9
3233	EUR24815 EN 2011, EU10/2011	6.00	DG(0.05)	-----		6.0
					only with 6.0 used as conversion factor:	
	normality	OK			OK	
	n	29			15	
	outliers	3	+3 excl.		1	
	mean (n)	1.911			2.323	
	st.dev. (n)	1.0199			1.2990	
	R(calc.)	2.856			3.637	
	R(Horwitz)	0.777			0.917	





APPENDIX 2

Actual amount of simulant for each migration step, actual contact surface used and measured formaldehyde concentrations on sample #13184; results in ml, dm^2 and mg/l

lab	1st ml	1st surf	1st formal.	2nd ml	2nd surf	2nd formal.	3rd ml	3rd surf	3rd formal.	remarks
310	150	1.27	6.79	150	1.27	6.91	150	1.27	6.54	
330	100	1.13	3.76	100	1.13	4.10	100	1.13	5.07	
551	500	0.93	0.27	500	0.93	0.79	500	0.93	0.64	
2115	250	1.87	1.380	250	1.87	1.76	250	1.87	1.35	
2127	500	2.53	3.075	500	2.53	2.325	500	2.53	2.075	
2129	550	3.1	0.45	550	3.1	0.79	550	3.1	0.42	
2132	250	1.64	5.10	250	1.64	3.16	250	1.64	4.61	
2146	500	2.6	4.74	500	2.6	3.69	500	2.6	4.51	
2152	550	3.25	1.71	550	3.25	2.03	550	3.25	2.19	
2165	550	2.73	3.95	550	2.73	2.76	550	2.73	1.57	
2184	550	2.73	3.84	550	2.73	2.09	550	2.73	1.80	
2190	550	2.8	<0.1	550	2.8	0.23	550	2.8	0.1	
2229	460	2.525	0.39	460	2.525	1.03	460	2.525	0.57	
2241	520	2.62	1.97	520	2.62	1.63	520	2.62	1.52	
2256	500	4.304	0.95	500	4.304	1.92	500	4.304	1.53	
2284	500	2.57	2.41	500	2.57	1.72	500	2.57	1.43	
2309	-----	-----	-----	-----	-----	-----	-----	-----	-----	
2353	550	2.5148	0.1886	550	2.5148	1.4301	550	2.5148	1.5968	
2359	480	2.426	0.55	480	2.426	0.65	480	2.426	1.12	
2372	550	2.2973	1.77	550	2.2973	1.31	550	2.2973	1.28	
2386	500	2.49	1.786	500	2.49	2.212	500	2.49	1.824	
2403	490	2.52	2.89	490	2.52	1.76	490	2.52	1.91	
2462	400	2.35	1.89	400	2.35	1.60	400	2.35	1.42	
2495	410	2.36	0.178	410	2.36	0.341	410	2.36	0.661	
2510	292	1.99	1.19	292	1.99	2.16	296	1.99	2.30	
2525	500	2.52	2.60	500	2.52	1.60	500	2.52	1.60	
2544	500	2.51	2.56	500	2.51	2.45	500	2.51	2.34	
2551	500	2.87	0.88	500	2.87	1.17	500	2.87	1.01	
2579	500	2.41	3.20	500	2.41	2.00	500	2.41	2.20	
3100	500	2.37	2.29	500	2.37	1.38	500	2.37	1.42	
3146	550	2.70	2.84	550	2.70	2.31	550	2.70	3.33	
3151	550	2.88	0.4581	550	2.88	1.934	550	2.88	1.9527	
3153	500	2.52	3.79	500	2.52	4.78	500	2.52	7.56	
3154	200	1.534	-----	200	1.534	-----	200	1.534	2.661	
3172	-----	-----	-----	-----	-----	-----	-----	-----	-----	
3185	500	2.60	2.44	500	2.60	1.61	500	2.60	1.07	
3218	500	2.54	2.44	500	2.54	1.73	500	2.54	1.43	
3228	550	2.73	3.90	550	2.73	2.35	550	2.73	1.57	
3233	550	2.79	4.91	550	2.79	3.12	550	2.79	5.08	

The abbreviations used in above table are as follows:

1st ml = simulant used in the first migration step in millilitres

1st surf = contact surface used in the first migration step in dm^2

1st form. = formaldehyde concentration measured after the first migration step in mg/l

2nd ml = ml of simulant used in the second migration step

2nd surf = contact surface used in the second migration step in dm^2

2nd form. = formaldehyde concentration measured after the second migration step in mg/l

3rd ml = ml of simulant used in the third migration step

3rd surf = contact surface used in the third migration step in dm^2

3rd form. = formaldehyde concentration measured after the third migration step in mg/l

APPENDIX 3

Test conditions when selected by participants

lab	type of simulant	Estimated bowl volume in cm^3	exposure time in hrs	exposure temp in °C	reporting unit	migration method
310	3% acetic acid	127	2	70	mg/kg	filling
330	3% acetic acid		2	70	mg/l	article filling
551						
2115						
2127						
2129	3% acetic acid		2	70	mg/kg	article filling
2132	3% acetic acid	570	2	70	mg/kg	article filling
2146	3% acetic acid	500	2	70	mg/kg	article filling
2152						
2165						
2184	dist.H_2O, isooctane, 95%EtOH, 3% HAc		2	70	mg/kg	article filling
2190						
2229	3% acetic acid	460	2.0	70	mg/l, mg/dm^2, mg/kg	article filling
2241						
2256		500	2	70		
2284	3% acetic acid	500	2	70	mg/kg	article filling
2309						
2353	3% acetic acid	550	2	70	mg/dm^2	article filling
2359	3% acetic acid	480	2	70		article filling
2372	3% acetic acid	229.7381	2	70	mg/kg	article filling
2386	3% acetic acid	500	2	70	mg/dm^2	article filling
2403						
2462	3% acetic acid	550	2	70	mg/kg	article filling
2495	3% acetic acid	470	2	70	mg/dm^2	filling
2510	3% acetic acid	500	2*3 times	70	mg/kg	article filling
2525	3% acetic acid	545.46	2	70	mg/kg	article filling
2544	3% acetic acid	550	2	70	mg/kg	article filling
2551						
2579	3% acetic acid	500	2.0	70.0	mg/kg	article filling
3100						
3146	3% acetic acid	550	2	70	mg/kg	article filling
3151	3% acetic acid	550	2	70	mg/kg, mg/dm^2	filling
3153						
3154						
3172						
3185	3% acetic acid	570	2	70	mg/kg	article filling
3218	3% acetic acid	550	2.0	70.0	mg/kg	article filling
3228						
3233	3% acetic acid	550	2	70	mg/kg	filling

APPENDIX 4

Number of participating laboratories per country

1 lab in	BRAZIL
1 lab in	FINLAND
3 labs in	FRANCE
7 labs in	GERMANY
5 labs in	HONG KONG
1 lab in	INDIA
1 lab in	IRELAND
3 labs in	ITALY
14 labs in	P.R. of CHINA
1 lab in	SAUDI ARABIA
1 lab in	TAIWAN R.O.C.
1 lab in	THE NETHERLANDS

APPENDIX 5

Abbreviations:

C = final result after checking of first reported suspect result
D(0.01) = outlier in Dixon's outlier test
D(0.05) = straggler in Dixon's outlier test
G(0.01) = outlier in Grubbs' outlier test
G(0.05) = straggler in Grubbs' outlier test
DG(0.01) = outlier in Double Grubbs' outlier test
DG(0.05) = straggler in Double Grubbs' outlier test
n.a. = not applicable
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
6 ISO 5725-86
7 ISO 5725, parts 1-6, 1994
8 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
9 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
10 IP 367/96
11 DIN 38402 T41/42
12 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
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14 Analytical Methods Committee Technical Brief, No4 January 2001
15 The Royal Society of Chemistry 2002, Analyst 2002, 127 page1359-1364, P.J. Lowthian and M. Thompson. (see http://www.rsc.org/suppdata/an/b2/b205600n/)
16 R.G. Visser, Reliability of proficiency test results for metals and phthalates in plastics, Accred Qual Assur, 14:29-34 (2009)
17 EUR 24815 EN 2011 - Technical guidelines on testing the migration of primary aromatic amines from polyamide kitchenware and of formaldehyde from melamine kitchenware, JRC (2011)