# Results of Proficiency Test Free and Released Formaldehyde in textile November 2015

Organised by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

Author: ing. R.J. Starink

Correctors: dr. R. Visser & ing. A.S. Noordman-de Neef

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#### 1 INTRODUCTION

Since the 1990's, many countries have adopted environmental standards and requirements restricting the use of harmful chemicals in the production of textiles and clothing. Laws and regulations impose some of these standards and requirements. In addition to mandatory environmental standards and requirements for textiles, there are some Ecolabelling schemes imposing environmental requirements for textile products on a voluntary basis. Well known programs are for instance Milieukeur (the Netherlands), Öko-Tex Standard 100 (Germany) and Thai Green Label (Thailand).

Since 2008, the Institute for Interlaboratory Studies (iis) organises a proficiency scheme for Free Formaldehyde in textile, which was extended in 2013/2014 with a scheme for Released Formaldehyde. In the 2015/2016 program it was decided to organize both schemes again.

In the 2015/2016 interlaboratory study 194 laboratories in 42 different countries participated. See appendix 2 for the number of participating laboratories per country. In this report, the results of the 2015 proficiency test are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

#### 2 SET UP

The Institute for Interlaboratory Studies in Spijkenisse was the organiser of this proficiency test. Sample preparation and analyses of fit for use and homogeneity were subcontracted. The analyses for fit-for-use testing were subcontracted to an ISO17025 accredited laboratory. It was decided to use in this Proficiency Test two positive samples. Sample #15207 is approx. 3 grams and sample #15208 is approx. 2 gram. The participants were requested to report rounded and unrounded results. These unrounded results were preferably used for the statistical evaluations.

#### 2.1 QUALITY SYSTEM

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 April 2014 (iis-protocol, version 3.3). This protocol can be downloaded from the iis website <a href="https://www.iisnl.com">www.iisnl.com</a>, 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

Two batches each positive on (Free) Formaldehyde were selected. From the first batch, an uncoloured cotton, 200 subsamples of approx. 3 gram were prepared and labelled #15207. Each sample was packed in aluminium foil.

The homogeneity of the subsamples was checked on 7 stratified randomly selected samples. The homogeneity testing was performed by a subcontracted ISO17027 accredited laboratory. See the following tables for the test results.

	Free Formaldehyde in mg/kg
Sample #15207-1	28.5
Sample #15207-2	28.9
Sample #15207-3	30.0
Sample #15207-4	29.4
Sample #15207-5	30.0
Sample #15207-6	30.5
Sample #15207-7	30.8

Table 1: homogeneity test results of subsamples #15207

From the above results of the homogeneity test, the observed repeatability was calculated and compared with 0.3 times the proficiency target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Free Formaldehyde in mg/kg
r(observed)	2.3
reference	Horwitz
0.3 x R (reference)	2.4

Table 2: evaluation of the repeatability of the homogeneity test results

The calculated repeatability of subsamples #15207 is in good agreement with the target, estimated from the Horwitz equation. Therefore, homogeneity of the subsamples #15207 was assumed.

From the second batch, a blue coloured cotton, 200 subsamples of approx. 2 gram were prepared and labelled #15208. Each sample was packed in aluminium foil.

This sample was used in a previous proficiency test (iis12A04). The statistical evaluation of the group performance in the previous proficiency test proved the homogeneity of the sample. Therefore the homogeneity of the subsamples #15208 was assumed.

To the participants, a set of samples (1 sample labelled #15207 containing approx. 3 grams textile and 1 sample labelled #15208 containing approx. 2 grams textile) was sent on October 14, 2015.

#### 2.5 ANALYSIS

The participants were asked to determine on both samples (#15207 and #15208) the Free Formaldehyde content and the Released Formaldehyde content with the analytical procedures that are routinely used in the laboratory.

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 as well as the required standards and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. A form to confirm receipt of the samples and a letter of instructions were added to the samples.

#### 3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered via the data entry portal www.kmpd.co.uk/sgs-iis-cts/. 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 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 April 2014 (iis-protocol, version 3.3).

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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon, Grubbs and or Rosner General ESD 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 and by R(0.01) for the Rosner General ESD test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test and by R(0.05) for the Rosner General ESD test (ref. 17). 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 5; nr.14 and 15). 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 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) = (result - average of PT) / 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 should be done in order to evaluate whether the reported test results are fit-for-purpose.

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

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

# 4 **EVALUATION**

During the execution of this proficiency test no problems occurred with the delivery of the samples. Two laboratories did not report any test results and five laboratories reported results after the final reporting date.

Finally, the 192 reporting laboratories sent in total 415 numerical results. Observed were 14 statistical outlying results, which is 3.4% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

In ISO14184-1:11 some information on precision data is given. In table B.1 of this method "approximate accuracy" values are mentioned. These values are probably the calculated repeatability standard deviations. Note also that under table B.1 is mentioned "that the method in this part of ISO14184 uses a different calibration graph from that used in the determination of the above-mentioned results". Therefore it was concluded that reliable reproducibility data cannot be estimated and therefore target reproducibilities estimated from the Horwitz equation were used for evaluation.

Also in ISO14184-2:11 some information on precision data is given. In tables C.1 and C.2 of this method critical differences are given for zero-formaldehyde fabrics and for low-level-formaldehyde fabrics (100 – 400 mg/kg). The given reproducibilities are divided between 'single fabric' and 'multiple fabric'.". Therefore it was concluded that reliable reproducibility data cannot be estimated and therefore target reproducibilities estimated from the Horwitz equation were used for evaluation.

### 4.1 EVALUATION PER SAMPLE

In this section, the reported results are discussed per test and per sample. All statistical results reported on the textile samples are summarised in appendix 1.

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.

#### #15207:

<u>Free Formaldehyde content:</u> This determination was problematic for a number of laboratories. Eight statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the estimated reproducibility calculated using the Horwitz equation.

Released Formaldehyde: This determination was very problematic. No statistical outliers were observed. Three results were excluded from statistical evaluation, as the reported test method is for the determination of Free Formaldehyde.

The calculated reproducibility after rejection of the suspect data is not at all in agreement with the estimated reproducibility calculated using the Horwitz equation.

#### #15208:

<u>Free Formaldehyde content:</u> This determination may be problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility calculated using the Horwitz equation.

Released Formaldehyde: This determination was very problematic. Two statistical outliers were observed and two results were excluded from statistical evaluation, as the reported test method is for the determination of Free Formaldehyde. The calculated reproducibility after rejection of the suspect data is not at all in agreement with the estimated reproducibility calculated using the Horwitz equation.

#### 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the calculated reproducibilities using the Horwitz equation and the reproducibilities as found for the group of participating laboratories. The number of significant results, the average results, the calculated reproducibilities (standard deviation\*2.8) and the target reproducibilities (Horwitz equation), are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R (target)
Free Formaldehyde	mg/kg	182	46.1	11.0	11.6
Released Formaldehyde	mg/kg	19	73.9	45.2	17.3

Table 3: reproducibilities of textile sample #15207

Parameter	unit	n	average	2.8 * sd	R (target)
Free Formaldehyde	mg/kg	181	109.3	29.8	24.2
Released Formaldehyde	mg/kg	14	319	151	60

Table 4: reproducibilities of textile sample #15208

From the above tables it can be concluded that, without further statistical calculations, the group of participating laboratories do not have much difficulties with the analysis of Free Formaldehyde but with the analysis of released formaldehyde the laboratories do have large difficulties when the calculated target results are compared with the Horwitz equation. See also the discussions in paragraphs 4.1 and 6.

# 5 COMPARISON WITH THE PREVIOUS PROFICIENCY TESTS

The spreads, present in the results for the two samples with Free Formaldehyde during the present PT, are fully in line with the spreads as observed in previous its PTs (see below table).

Parameter	October	October	October	October	October
Farameter	2015	2014	2013	2012	2009 - 2011
Free Formaldehyde	9-10%	8%	10-13%	8-9%	7-15%
Released Formaldehyde	17-22%	10%	9-10%	n.e.	n.e.

Table 5: Development of relative uncertainties over the years

#### 6 DISCUSSION

When the results of this interlaboratory study were compared to the Ecolabelling Standards and Requirements for Textiles in EU (table 6), it was noticed that not all participants would make identical decisions about the acceptability of the textiles for the determined parameters.

Ecolabel	baby clothes	Öko-Tex 103 no direct skin contact	Öko-Tex 103 in direct skin contact	Decoration material
Free Formaldehyde extractable (mg/kg)	<16	300	75	300
Released Formaldehyde (mg/m³)	0.1	0.1	0.1	0.1

Table 6: Ecolabelling Standards and Requirements for Textiles in EU

The method for determination of the Free Formaldehyde is specified in the Standards of the Ecolabelling Institutes.

It should be noticed that ISO14184-1 corresponds to the Japanese method specified in the Japanese Law 112 and is described in the Japanese Standard JIS L1096.

# Extractable free formaldehyde:

For sample #15207, all reporting laboratories would accept the sample for the categories "Öko-Tex 103 no direct skin contact" and "Decoration material" (<300 mg/kg). All reporting laboratories, except three, would accept the sample for the category "Öko-Tex 103 direct skin contact" (<75 mg/kg). All reporting laboratories, except one, would reject the sample for the category "Baby clothes" (<16 mg/kg).

For sample #15208, all reporting laboratories, except one, would accept the sample for the categories "Öko-Tex 103 no direct skin contact" and "Decoration material" (<300 mg/kg). All reporting laboratories, except two, would reject the sample for the categories "Baby clothes" (<16 mg/kg) and "Öko-Tex 103 direct skin contact" (<75 mg/kg).

### Released Formaldehyde:

No conclusions can be drawn, as the limits mentioned in the Ecolabel Standard have a different unit compared with test method ISO14184-2:11 (mg/m³ vs mg/kg).

#### 6.1 GENERAL CONCLUSION

In this proficiency test the Free Formaldehyde content and the Released Formaldehyde were determined. The spread observed for the Free Formaldehyde content and the Released Formaldehyde in this interlaboratory study are in line with the previous proficiency tests. The spread observed in this interlaboratory study can be caused by the preparation or the conditioning of the sample and/or by the performance of the analysis. Consequently, the reproducibility cannot be improved by only one change in the analysis. 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 thus increase of the quality of the analytical results.

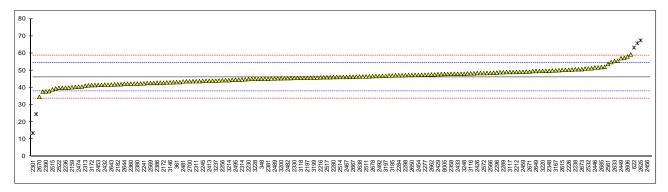
**APPENDIX 1** 

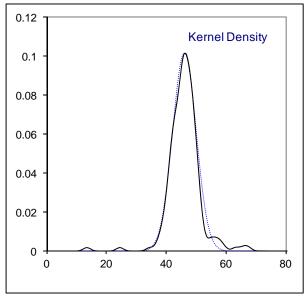
Determination of Free Formaldehyde content on sample #15207; results in mg/kg

Deterr	nination of Fre	e Formaide	enyae cor	itent on	samp	ie #15207; i	esuits in mg/kg		
lab	Method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110	JIS L1041B	44.117		-0.48	2368	ISO14184-1	46.37		0.06
213	ISO14184-1	99.1	R(0.01)	12.78	2373	ISO14184-1	48.89		0.67
			K(0.01)						
230	ISO14184-1	46.7		0.14	2379	ISO14184-1	44.47		-0.40
348	In house	45.06		-0.26	2380	ISO14184-1	42.33		-0.91
361	ISO14184-1	43.15		-0.72	2381	ISO14184-1	45.12		-0.24
551	ISO14184-1	42.81		-0.80	2386	ISO14184-1	42.8	С	-0.80
622	ISO14184-1	63.24	R(0.01)	4.13	2389	ISO14184-1	45.6	Ü	-0.13
632	ISO14184-1	167.8958	R(0.01)	29.38	2390	ISO14184-1	37.77		-2.01
826	ISO14184-1	47.3		0.28	2401	ISO14184-1	50.9995		1.18
1051	GB/T2912	47.92		0.43	2403	ISO14184-1	51.6820		1.34
2102					2404	ISO14184-1	48.29		0.52
2115	ISO14184-1	43.08		-0.73	2410	ISO14184-1	45.02		-0.27
2129	ISO14184-1	40.30		-1.40	2413	ISO14184-1	43.92		-0.53
2132	ISO14184-1	47.32		0.29	2415	ISO14184-1	45.4		-0.17
2137	ISO14184-1	43.64		-0.60	2426	ISO14184-1	48.4		0.55
2138	ISO14184-1	41.66		-1.08	2429	ISO14184-1	47.8		0.41
2139	ISO14184-1	50.66		1.10	2432	ISO14184-1	41.64		-1.08
2146	ISO14184-1	43.98		-0.52	2433	ISO14184-1	48.00		0.45
2156	ISO14184-1	47.12		0.24	2442	ISO14184-1	47.15		0.25
2159	ISO14184-1	40.17		-1.44	2446	In house	51.65		1.33
2165	ISO14184-1	45.0		-0.27	2449	ISO14184-1	57.06		2.64
2170	ISO14184-1	55.5		2.26	2453	ISO14184-1	41.50		-1.11
2172	ISO14184-1	42.83		-0.79	2454	ISO14184-1	47.36		0.30
					_			C D(0 04)	
2182	ISO14184-1	41.9		-1.02	2456	ISO14184-1	133.4	C,R(0.01)	21.05
2184	ISO14184-1	47.6		0.36	2459	ISO14184-1	49.085		0.72
2186					2460	ISO14184-1	40.54	С	-1.35
2190	ISO14184-1	51.2		1.23	2467	ISO14184-1	46.14		0.00
2197	ISO14184-1	45.6		-0.13	2474	ISO14184-1	40.50		-1.36
2201	ISO14184-1						43.15		
		46.44		0.08	2475	ISO14184-1			-0.72
2212	JIS L1041B	49.02		0.70	2476	ISO14184-1	46.12		0.00
2216	JIS L1041B	45.91		-0.05	2481	ISO14184-1	43.56	С	-0.62
2217	ISO14184-1	45.41		-0.17	2482	ISO14184-1	45.43		-0.17
2225	ISO14184-1	48.0		0.45	2483	ISO14184-1	49		0.69
2226	ISO14184-1	50.30		1.01	2489	ISO14184-1	45.3		-0.20
	ISO14184-1								
2229		45.48		-0.15	2492	In house	46.7		0.14
2230	ISO14184-1	44.9		-0.29	2495	ISO14184-1	44.6		-0.37
2236	ISO14184-1	39.90		-1.50	2497	ISO14184-1	59.27		3.17
2238	ISO14184-1	50.64		1.09	2500	ISO14184-1	48.4		0.55
2241	ISO14184-1	42.45		-0.89	2506	ISO14184-1	41.82		-1.04
2245	ISO14184-1	43.87		-0.54	2511	GB/T2912	46.45		0.08
2246	ISO14184-1	46		-0.03	2514	ISO14184-1	46.11		0.00
2255	ISO14184-1	47.8		0.41	2515	ISO14184-1	38.896		-1.74
2256	ISO14184-1	44.2		-0.46	2517	ISO14184-1	46.0		-0.03
2264	GB/T2912	41.9		-1.02	2519	ISO14184-1	43.7		-0.58
2269	ISO14184-1	49.076		0.71	2522	ISO14184-1	39.77		-1.53
2272	ISO14184-1	40.0		-1.48	2532	ISO14184-1	51.15		1.21
2275	ISO14184-1	48.2		0.50	2534	ISO14184-1	49.2		0.74
2277	ISO14184-1	47.45		0.32	2535	ISO14184-1	52		1.42
2279	ISO14184-1	48		0.45	2540	ISO14184-1	49.62		0.84
								C D(0 04)	
2280	ISO14184-1	46.07		-0.01	2559	ISO14184-1	65.8	C,R(0.01)	4.75
2282	ISO14184-1	46.1		0.00	2561	ISO14184-1	53.68		1.82
2284	ISO14184-1	47.14		0.25	2563				
2289	ISO14184-1	48.92		0.68	2566	JIS B	48.5	С	0.57
2290	ISO14184-1	49.73		0.87	2567				
2292	ISO14184-1	45.8075		-0.08	2569	ISO14184-1	42.6		-0.85
2293	ISO14184-1	49.564		0.83	2572	ISO14184-1	48.4		0.55
2294				-1.98	2582		39.80		
	ISO14184-1	37.9	0			ISO14184-1			-1.52
2295	ISO14184-1	46.5	С	0.09	2589	ISO14184-1	47.01		0.21
2296	ISO14184-1	48.70		0.62	2590	ISO14184-1	45.1		-0.25
2298	JIS L1041B	47.2		0.26	2591	In house	39.53		-1.59
2301	ISO14184-1	13.51	R(0.01)	-7.87	2602	LFGB B 82.02	-1 44.8		-0.32
2310	ISO14184-1	42.3	, ,	-0.92	2606	ISO14184-1	58	С	2.87
2311	ISO14184-1	43.7		-0.58	2615	ISO14184-1	50.20	-	0.98
2313	ISO14184-1	41.0		-1.24	2625	ISO14184-1	67.43	R(0.01)	5.14
								13(0.01)	
2314	ISO14184-1	44.69		-0.35	2629	ISO14184-1	50.40		1.03
2330	ISO14184-1	45.55		-0.14	2633	GB/T2912	55.4		2.24
2348	ISO14184-1	49.77		0.88	2638	ISO14184-1	46.391		0.07
2351	ISO14184-1	45.28		-0.20	2643	ISO14184-1	41.7		-1.07
2356	ISO14184-1	42.6		-0.85	2644	ISO14184-1	42.2	С	-0.95
2358	ISO14184-1	47.94		0.44	2649	ISO14184-1	49.60		0.84
2360	ISO14184-1	42.25		-0.93	2650	ISO14184-1	47.3		0.28
2361	Japan ST2012	46.8		0.16	2654	ISO14184-1	41.28		-1.17
	•								
2363	ISO14184-1	43		-0.75	2662	ISO14184-1	47.58		0.35
2364	ISO14184-1	43.89		-0.54	2667	ISO14184-1	46.23		0.03
2366	ISO14184-1	49.9		0.91	2670	ISO14184-1	34.56	_	-2.79
2367	ISO14184-1	42.64		-0.84	2671	ISO14184-1	49.2	С	0.74

2672	ISO14184-1	45.70	-0.10	3176	ISO14184-1	42.23		-0.94
2673	ISO14184-1	50.67	1.10	3182	ISO14184-1	49.0		0.69
2678	ISO14184-1	46.6	0.12	3185	ISO14184-1	47.02		0.22
2685	ISO14184-1	51.88	1.39	3186	ISO14184-1	41.4		-1.14
2700	ISO14184-1	43.65	-0.60	3190	ISO14184-1	50.08		0.96
2702	ISO14184-1	48.45	0.56	3191	ISO14184-1	45.55		-0.14
2703	ISO14184-1	54.8	2.09	3195	ISO14184-1	46		-0.03
2712	ISO14184-1	44.6	-0.37	3197	ISO14184-1	46.8		0.16
3110	ISO14184-1	43.69	-0.59	3199	ISO14184-1	45.78		-0.08
3116	ISO14184-1	48.24	0.51	3200	ISO14184-1	45.4		-0.17
3117	ISO14184-1	49.0	0.69	3207	JIS L1041B	46.2		0.02
3118	ISO14184-1	45.59	-0.13	3210	ISO14184-1	47.5		0.33
3145	LFGB B 82.02-1	50.22	0.99	3214	ISO14184-1	44.26		-0.45
3146	ISO14184-1	43.0	-0.75	3216	ISO14184-1	24.53	R(0.01)	-5.21
3149	ISO14184-1	57.2	2.67	3218	ISO14184-1	48.62		0.60
3150	ISO14184-1	37.6	-2.06	3220	ISO14184-1	49.66		0.85
3153	ISO14184-1	44.26	-0.45	3225	ISO14184-1	47.41		0.31
3154	ISO14184-1	41.5735	-1.10	3228	ISO14184-1	45		-0.27
3166	ISO14184-1	42.35	-0.91	3237	ISO14184-1	43.99	С	-0.51
3167	ISO14184-1	50	0.94	3248	GB/T2912	48		0.45
3172	ISO14184-1	41.39	-1.14	8005	JIS L1041B	47.86		0.42
	normality	suspect						
	n	182						
	outliers	8						
	mean (n)	46.120						
	st.dev. (n)	3.9372						
	R(calc.)	11.024						
	R(Horwitz)	11.607						
	( - I-)							

Lab 2295: first reported 33.3 Lab 2386: first reported 42.8 Lab 2456: first reported 66.7 Lab 2460: first reported 32.59 Lab 2481: first reported 42.17 Lab 2559: first reported 60.2 Lab 2566: first reported 105.6 Lab 2606: first reported 85 Lab 2644: first reported 21.1 Lab 2671: first reported 104.5 Lab 3237: first reported 37.05





# Determination of Released Formaldehyde content on sample #15207; results in mg/kg

213 ISO14184-2 72.3 -0.26 2373 2379 ISO14184-2 70.87 -0 348 2380 2381 2381 2386 2386 2389 ISO14184-2 81.4 1 632 ISO14184-1 443.0532 ex 59.64 2390 ISO14184-2 70.8 -0 826 2401 2401 2403 2403 2402 2403 2403 2402 2403 2403 2402 2402 2403 2403 2402 2403 2403 2402 2403 2403 2402 2403 2403 2403 2402 2403 2403 2402 2403 2403 2403 2403 2403 2403 2403 2403 2403 2403 2403 2403 2403 2403 2403 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404 2404	-0.49  1.21 -0.50 
230      2379     ISO14184-2     70.87     -0       348      2380         361      2381         551      2386         622      2389     ISO14184-2     81.4     1       632     ISO14184-1     443.0532     ex     59.64     2390     ISO14184-2     70.8     -0       826      2401        2403         1051      2403          2102     In house     44.68     -4.72     2404         2115      2410	-0.49  1.21 -0.50 
348      2380        361      2381        551      2386        622      2389     ISO14184-2     81.4     1       632     ISO14184-1     443.0532     ex     59.64     2390     ISO14184-2     70.8     -0       826      2401       2403         2102     In house     44.68     -4.72     2404          2115       2410	1.21 -0.50 
551      2386        622      2389     ISO14184-2     81.4     1       632     ISO14184-1     443.0532     ex     59.64     2390     ISO14184-2     70.8     -0       826       2401          1051      2403          2102     In house     44.68     -4.72     2404         2115      2410	1.21 -0.50 
622       2389     ISO14184-2     81.4     1       632     ISO14184-1     443.0532     ex     59.64     2390     ISO14184-2     70.8     -0       826       2401           1051       2403           2102     In house     44.68     -4.72     2404          2115      2410	1.21 -0.50  
632 ISO14184-1 443.0532 ex 59.64 2390 ISO14184-2 70.8 -0 826 2401 2403 2102 In house 44.68 -4.72 2404 2115 2410 2410	-0.50   
1051 2403 2102 In house 44.68 -4.72 2404 2115 2410	 
2102 In house 44.68 -4.72 2404	
2445	  
2137 ISO14184-2 69.25 -0.75 2426	
2138 2429	
2139 2432 2436 2433	
2156 2442	
2159 ISO14184-2 72.91 -0.16 2446 2165 2449	
2170 2453	
2112	
2102	5.08
0.400	
2107	
2197 2474 2201 2475	
2212 2476	
210	
2217 2482 2225 2483	
2226 2489	
2229 W 2492 2230 2495	
2236 2497	
2238 2500	
==	5.21
2246 2514	
0050	-1.55
0004 11014044 40.04	
2269 2522	
2277 2535	
2279 2540	
2280 2559 2282 2561	
2284 2563 ISO14184-1 89.8 C,ex 2	2.57
2200	
0000	
2293 2572	
2294 2582 2295 ISO14184-2 73.8 -0.02 2589	
2296 ISO14184-2 80.41 1.05 2590	
2298 2591 2301 2602	
2301   2602	
2011	
2313 2625 2314 2629	
2330 2633	
2348 2638	
2007	
2358 ISO14184-2 76.80 0.47 2649 ISO14184-2 65.2 -1	-1.41
2000	
2363 2662	
2364 ISO14184-1 106.03 ex 5.19 2667	
2366 2670 2367 2671	
2672 3176	

2673					3182			
2678					3185			
2685					3186			
2700	AATCC112	74.80		0.14	3190			
2702					3191	ISO14184-2	60.54	-2.16
2703					3195			
2712					3197			
3110					3199			
3116					3200			
3117					3207			
3118					3210			
3145					3214			
3146					3216			
3149					3218			
3150					3220			
3153					3225	ISO14184-2	82.45	1.38
3154					3228			
3166	ISO14184-1	39	ex	-5.64	3237			
3167					3248			
3172					8005			
					•			
	normality	OK						
	•	4.0						

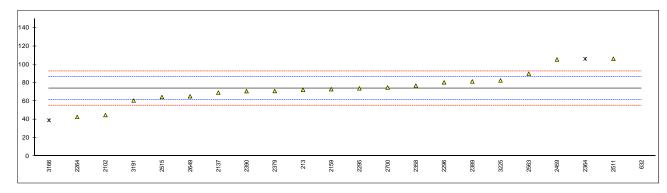
n 19
outliers 0 (+3 excl)
mean (n) 73.921
st.dev. (n) 16.1328
R(calc.) 45.172
R(Horwitz) 17.329

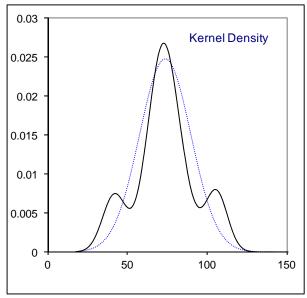
Lab 2229: results withdrawn: reported 45.48 Lab 2454: results withdrawn: reported 47.73

Lab 2563: first reported 43.4, result excluded as test method is for free formaldehyde

Lab 2650: results withdrawn: reported 45.8

Lab 632: result excluded as test method is for free formaldehyde Lab 3166: result excluded as test method is for free formaldehyde



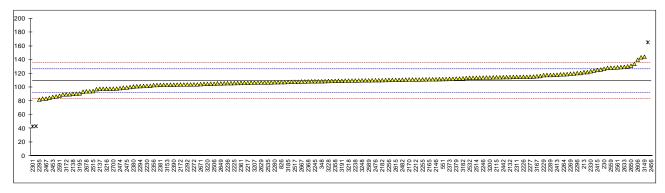


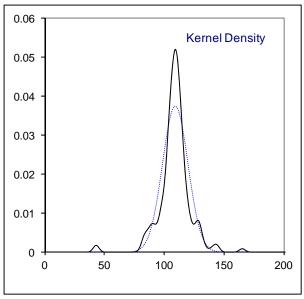
# Determination of Free Formaldehyde content on sample #15208; results in mg/kg

lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110	JIS L1041B	103.196	mark	-0.71	2368	ISO14184-1	108.54	man	-0.09
213	ISO14184-1	121.9		1.46	2373	ISO14184-1	112.26		0.34
230	ISO14184-1	126.9		2.04	2379	ISO14184-1	112.56		0.38
348	In house	108.72		-0.07	2380	ISO14184-1	101.01		-0.96
361	ISO14184-1	105.93		-0.39	2381	ISO14184-1	103.39		-0.69
551	ISO14184-1	111.87		0.30	2386	ISO14184-1	83.4		-3.00
622	ISO14184-1	43.31	R(0.01)	-7.65	2389	ISO14184-1	111.5		0.25
632					2390	ISO14184-1	103.69		-0.65
826	ISO14184-1	107.4		-0.22	2401	ISO14184-1	104.9990		-0.50
1051	GB/T2912	119.02		1.12	2403	ISO14184-1	112.5620		0.38
2102	100444044	 114.49		0.60	2404	ISO14184-1	109.10 103.45		-0.03
2115 2129	ISO14184-1 ISO14184-1	128.05		0.60 2.17	2410 2413	ISO14184-1 ISO14184-1	103.45		-0.68 1.02
2132	ISO14184-1	114.79		0.63	2415	ISO14184-1	125.1		1.83
2137	ISO14184-1	97.70		-1.35	2426	ISO14184-1	108.0		-0.15
2138	ISO14184-1	90.47		-2.18	2429	ISO14184-1	115.0		0.66
2139	ISO14184-1	108.83		-0.06	2432	ISO14184-1	97.87		-1.33
2146	ISO14184-1	111.62		0.27	2433	ISO14184-1	110.40		0.13
2156	ISO14184-1	119.84		1.22	2442	ISO14184-1	114.66		0.62
2159	ISO14184-1	94.13		-1.76	2446	In house	115.0		0.66
2165	ISO14184-1	111.6		0.26	2449	ISO14184-1	113.78		0.52
2170	ISO14184-1	111.2		0.22	2453	ISO14184-1	85.84		-2.72
2172	ISO14184-1	103.9		-0.63	2454	ISO14184-1	106.80	C D(0.04)	-0.29
2182 2184	Japan ST2012 ISO14184-1	110.3 112.0		0.11 0.31	2456 2459	ISO14184-1	308.4	C,R(0.01)	23.07
2186	13014104-1	112.0		0.51	2459	ISO14184-1	97.73		-1.34
2190	ISO14184-1	130.4		2.44	2467	ISO14184-1	83.43		-3.00
2197	ISO14184-1	122.0		1.47	2474	ISO14184-1	98.63		-1.24
2201	ISO14184-2	109.69		0.04	2475	ISO14184-1	99.49		-1.14
2212	JIS L1041B	111.30		0.23	2476	ISO14184-1	110.25		0.11
2216	JIS L1041B	117.73		0.97	2481	ISO14184-1	109.29	С	0.00
2217	ISO14184-1	106.52		-0.32	2482	ISO14184-1	110.94		0.19
2225	ISO14184-1	106.0		-0.38	2483	ISO14184-1	115.5		0.72
2226	ISO14184-1	115.17		0.68	2489	ISO14184-1	114.4		0.59
2229	ISO14184-1	117.52		0.95	2492	In house	114.7		0.62
2230 2236	ISO14184-1 ISO14184-1	102.0 105.84		-0.85 -0.40	2495 2497	ISO14184-1 ISO14184-1	102.1 143.55	С	-0.84 3.97
2238	ISO14184-1	103.84		0.05	2500	ISO14184-1	111.3	C	0.23
2241	ISO14184-1	107.30		-0.23	2506	ISO14184-1	105.03		-0.50
2245	ISO14184-1	108.63		-0.08	2511				
2246	ISO14184-1	114		0.54	2514	ISO14184-1	113.83		0.52
2255	ISO14184-1	111.4		0.24	2515	ISO14184-1	94.398		-1.73
2256	ISO14184-1	110.6		0.15	2517	ISO14184-1	107.97		-0.16
2264	GB/T2912	118.8		1.10	2519	ISO14184-1	120.7		1.32
2269	ISO14184-1	119.609		1.19	2522	ISO14184-1	106.47		-0.33
2272 2275	ISO14184-1 ISO14184-1	104.1 110.1		-0.60 0.09	2532 2534	ISO14184-1	113.7 114.5		0.51 0.60
2277	ISO14184-1	115.36		0.09	2535	ISO14184-1 ISO14184-1	107		-0.27
2279	ISO14184-1	107		-0.27	2540	ISO14184-1	111.77		0.28
2280	ISO14184-1	107.28		-0.24	2559	ISO14184-1	128.4		2.21
2282	ISO14184-1	107.8		-0.18	2561	ISO14184-1	128.87		2.27
2284	ISO14184-1	108.65		-0.08	2563				
2289	ISO14184-1	117.75		0.98	2566	JIS B	105.6	С	-0.43
2290	ISO14184-1	113.41		0.47	2567				
2292	ISO14184-1	104.029		-0.61	2569	ISO14184-1	106.7		-0.30
2293	ISO14184-1	116.273		0.81	2572	ISO14184-1	112.3	0	0.35
2294	ISO14184-1	101.6		-0.89	2582	ISO14184-1	89.76	С	-2.27
2295 2296	ISO14184-1 ISO14184-1	81.9 120.52		-3.18 1.30	2589 2590	ISO14184-1 ISO14184-1	110.11 97.1		0.09 -1.42
2298	JIS L1041B	110.7		0.16	2590	In house	87.65		-2.51
2301	ISO14184-1	43.05	R(0.01)	-7.68	2602	LFGB B 82.02-1	104.1		-0.60
2310	ISO14184-1	111.6	11(0.01)	0.26	2606	ISO14184-1	140		3.56
2311	ISO14184-1	115		0.66	2615	ISO14184-1	110.64		0.15
2313	ISO14184-1	108.4		-0.11	2625	ISO14184-1	165.37	R(0.01)	6.50
2314	ISO14184-1	111.27		0.23	2629	ISO14184-1	106.90		-0.28
2330	ISO14184-1	123.19		1.61	2633	GB/T2912	129.6		2.35
2348	ISO14184-1	124.23		1.73	2638	ISO14184-1	97.969		-1.32
2351	ISO14184-1	125.74		1.90	2643	ISO14184-1	90.6	C	-2.17
2356 2358	ISO14184-1 ISO14184-1	103.1 109.21		-0.72 -0.01	2644 2649	GB/T2912 ISO14184-1	107.4 105.60	С	-0.22 -0.43
2360	ISO14184-1	109.21		-0.01 -0.91	2650	ISO14184-1	131.3		-0.43 2.55
2361	Japan ST2012	106.4		-0.34	2654	ISO14184-1	93.46		-1.84
2363	ISO14184-1	110.2		0.10	2662	ISO14184-1	118.66		1.08
2364	- •				2667	ISO14184-1	108.32		-0.12
2366	ISO14184-1	104.8		-0.52	2670	ISO14184-1	89.46	_	-2.30
2367	ISO14184-1	115.21		0.68	2671	ISO14184-1	104.5	С	-0.56
2672	ISO14184-1	106.1		-0.37	3176	ISO14184-1	110.63		0.15

2673	ISO14184-1	117.90		0.99	3182	ISO14184-1	113.0	0.43
2678	ISO14184-1	94.1		-1.76	3185	ISO14184-1	107.59	-0.20
2685	ISO14184-1	103.79		-0.64	3186	ISO14184-1	105.8	-0.41
2700	ISO14184-1	97.91		-1.32	3190	ISO14184-1	114.12	0.56
2702	ISO14184-1	110.26		0.11	3191	ISO14184-1	109.29	0.00
2703	ISO14184-1	134.0		2.86	3195	ISO14184-1	91	-2.12
2712					3197	ISO14184-1	103.6	-0.66
3110	ISO14184-1	99.42		-1.15	3199	ISO14184-1	108.6	-0.08
3116	ISO14184-1	111.17		0.21	3200	ISO14184-1	114.2	0.57
3117	ISO14184-1	109.9		0.07	3207	JIS L1041B	106.7	-0.30
3118	ISO14184-1	100.27		-1.05	3210	ISO14184-1	107	-0.27
3145	LFGB B 82.02-1	129.28		2.31	3214	ISO14184-1	104.01	-0.62
3146	ISO14184-1	101.8		-0.87	3216	ISO14184-1	97.77	-1.34
3149	ISO14184-1	144.3	С	4.05	3218	ISO14184-1	109.68	0.04
3150	ISO14184-1	84.6		-2.86	3220	ISO14184-1	104.89	-0.51
3153	ISO14184-1	103.45		-0.68	3225	ISO14184-1	104.08	-0.61
3154	ISO14184-1	113.9375		0.54	3228	ISO14184-1	109	-0.04
3166	ISO14184-1	128.5		2.22	3237	ISO14184-1	86.57	-2.64
3167	ISO14184-1	116		0.77	3248	GB/T2912	110	0.08
3172	ISO14184-1	89.63		-2.28	8005	JIS L1041B	109.61	0.03
	normality	suspect						
	n	181						
	outliers	4						
	mean (n)	109.318						
	st.dev. (n)	10.6475						
	R(calc.)	29.813						
	R(Horwitz)	24.161						
	11(11011112)	2 5 !						

Lab 2456: first reported 154.2 Lab 2481: first reported 101.56 Lab 2497: first reported 173.31 Lab 2566: first reported 48.5 Lab 2582: first reported 37.63 Lab 2644: first reported 53.7 Lab 2671: first reported 49.2 Lab 3149: first reported 157.7





# Determination of Released Formaldehyde content on sample #15208; results in mg/kg

lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110	10044404.0				2368				
213 230	ISO14184-2	295.8		-1.09 	2373 2379	ISO14184-2	317.07		-0.10
348					2380				
361 551					2381 2386				
622					2389	ISO14184-2	315.2		-0.19
632					2390	ISO14184-2	268.30		-2.37
826 1051					2401 2403				
2102	In house	114.06	DG(0.05)	-9.57	2404				
2115 2129					2410 2413				
2129					2415				
2137	ISO14184-2	255.95		-2.95	2426				
2138 2139					2429 2432				
2146					2433				
2156	10044404.0			0.70	2442				
2159 2165	ISO14184-2	335.42		0.76	2446 2449				
2170					2453				
2172 2182					2454 2456			W	
2184					2459				
2186					2460				
2190 2197					2467 2474				
2201					2475				
2212 2216					2476 2481				
2217					2482				
2225					2483				
2226 2229			W		2489 2492				
2230			••		2495				
2236					2497				
2238 2241					2500 2506				
2245					2511				
2246 2255					2514 2515	AATCC112	331.47		0.57
2256					2517	701100112			
2264 2269	JIS L1041B	124.78	DG(0.05)	-9.07 	2519 2522				
2272					2532				
2275					2534				
2277 2279					2535 2540				
2280					2559				
2282 2284					2561 2563	ISO14184-1	204.3	Cov	5 26
2289					2566	13014104-1	204.3	C,ex	-5.36 
2290					2567				
2292 2293					2569 2572				
2294					2582				
2295 2296	ISO14184-2 ISO14181-2	369.5 368.37		2.35 2.29	2589 2590				
2298	13014161-2	300.37		2.29	2590				
2301					2602				
2310 2311					2606 2615				
2313					2625				
2314 2330					2629 2633				
2348					2638				
2351					2643				
2356 2358	ISO14184-2	394.78		3.53	2644 2649	ISO14184-2	264.3		-2.56
2360					2650			W	
2361					2654				
2363 2364					2662 2667				
2366					2670				
2367					2671				

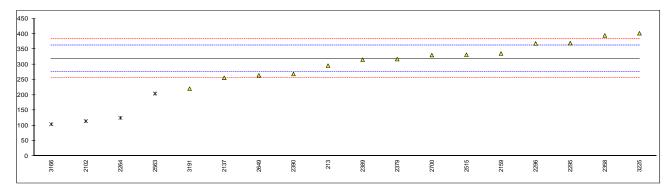
2672					3176			-	
2673					3182			-	
2678					3185			=	
2685					3186			=	
2700	AATCC112	330.32		0.52	3190			-	
2702					3191	ISO14184-2	220	0.40	-4.61
2703					3195			-	
2712					3197			-	
3110					3199			-	
3116					3200			=	
3117					3207			=	
3118					3210			=	
3145					3214			=	
3146					3216			=	
3149					3218			=	
3150					3220			=	
3153					3225	ISO14184-2	40	1.80	3.85
3154					3228			=	
3166	ISO14184-1	104	ex	-10.04	3237			=	
3167					3248			=	
3172					8005			-	
	normality	OK							
	n	14							
	outliers mean (n) st.dev. (n) R(calc.) R(Horwitz)	2 (+2 excl) 319.191 54.0335 151.294 60.039							

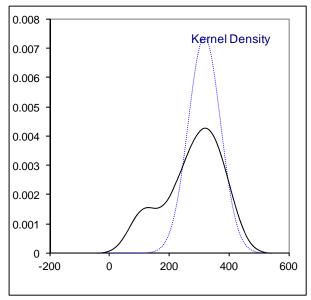
Lab 2229: results withdrawn: reported 117.52

Lab 2454: results withdrawn: reported 106.87

Lab 2563: first reported 103.9, result excluded as test method is for free formaldehyde Lab 2650: results withdrawn: reported 121.1

Lab 3166: result excluded as test method is for free formaldehyde





### **APPENDIX 2**

# Number of participants per country

- 7 labs in BANGLADESH
- 1 lab in BRAZIL
- 1 lab in BULGARIA
- 2 labs in CAMBODIA, Kingdom of
- 1 lab in CROATIA
- 1 lab in EGYPT
- 1 lab in FINLAND
- 4 labs in FRANCE
- 14 labs in GERMANY
  - 1 lab in GREECE
  - 1 lab in GUATEMALA
- 16 labs in HONG KONG
  - 1 lab in HUNGARY
- 12 labs in INDIA
- 4 labs in INDONESIA
- 9 labs in ITALY
- 7 labs in KOREA
- 1 lab in MALAYSIA
- 2 labs in MAURITIUS
- 4 labs in MEXICO
- 2 labs in MOROCCO
- 1 lab in NICARAGUA
- 49 labs in P.R. of CHINA
- 6 labs in PAKISTAN
- 1 lab in PERU
- 2 labs in PHILIPPINES
- 1 lab in PORTUGAL
- 1 lab in ROMANIA
- 1 lab in SERBIA
- 1 lab in SINGAPORE
- 1 lab in SLOVENIA
- 5 labs in SPAIN
- 1 lab in SRI LANKA
- 4 labs in TAIWAN R.O.C.
- 3 labs in THAILAND
- 1 lab in THE NETHERLANDS
- 2 labs in TUNISIA
- 5 labs in TURKEY
- 1 lab in U.A.E.
- 7 labs in U.S.A.
- 3 labs in UNITED KINGDOM
- 6 labs in VIETNAM

## **APPENDIX 3**

#### 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 D(0.01) = outlier in Grubbs' outlier test D(0.05) = straggler in Grubbs' outlier test D(0.05) = outlier in Double Grubbs' outlier test

DG(0.05) = straggler in Double Grubbs' outlier test

R(0.01) = outlier in Rosner outlier test R(0.05) = straggler in Rosner outlier test

n.a. = not applicablen.d. = not detectedW = withdrawn

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