Results of Proficiency Test Free and Released Formaldehyde in textile November 2016

Organised by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

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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), Thai Green Label (Thailand) and Bluesign® (Switzerland), which has created a Bluesign® system substances list (BSSL). Since 2008, the Institute for Interlaboratory Studies (iis) organises a proficiency test (PT) for Free Formaldehyde in textile, which was extended in 2013/2014 with a PT for Released Formaldehyde. During the annual proficiency testing program 2016/2017, it was decided to continue the PT for the analysis of Free and Released Formaldehyde. In this interlaboratory study 194 laboratories in 41 different countries registered for participation. See appendix 3 for the number of participating laboratories per country. In this report, the results of the 2016 Free and Released Formaldehyde in Textile proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies in Spijkenisse was the organiser of this proficiency test (PT). Sample preparation and analyses of fit-for-use and homogeneity were subcontracted to an ISO17025 accredited laboratory. It was decided to send in this Proficiency Test two positive samples. Sample #16330 is approx. 3 grams and sample #16331 is approx. 1.7 grams. The participants were requested to report rounded and unrounded test results. These unrounded test results were preferably used for the statistical evaluations.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC 17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol can be downloaded from the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

Two batches, each positive on (Free) Formaldehyde were selected. From the first batch, a pink coloured cotton, 200 subsamples of approx. 3 gram were prepared and labelled #16330. 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 an accredited laboratory. See the following tables for the test results.

<u> </u>	1
	Free Formaldehyde in mg/kg
Sample #16330-1	99.5
Sample #16330-2	95.3
Sample #16330-3	98.1
Sample #16330-4	102.0
Sample #16330-5	99.8
Sample #16330-6	102.0
Sample #16330-7	98.3

Table 1: homogeneity test results of subsamples #16330

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)	6.6
reference	Horwitz
0.3 x R (reference)	6.7

Table 2: evaluation of the repeatability of subsamples #16330

From the second batch, a black coloured cotton, 200 subsamples of approx. 1.7 gram were prepared and labelled #16331. Each sample was packed in aluminium foil. The homogeneity of the subsamples was checked on 15 stratified randomly selected samples. The homogeneity testing was performed by a subcontracted ISO17025 accredited laboratory. See the following tables for the test results.

	Free Formaldehyde in mg/kg
Sample #16331-1	213.1
Sample #16331-2	208.4
Sample #16331-3	212.4
Sample #16331-4	207.6
Sample #16331-5	208.9
Sample #16331-6	206.4
Sample #16331-7	210.1
Sample #16331-8	204.2
Sample #16331-9	211.4
Sample #16331-10	210.1
Sample #16331-11	209.7
Sample #16331-12	211.2
Sample #16331-13	208.4
Sample #16331-14	205.4
Sample #16331-15	209.4

Table 3: homogeneity test results of subsamples #16331

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)	7.0
reference	Horwitz
0.3 x R (reference)	12.6

Table 4: evaluation of the repeatability of subsamples #16331

The calculated repeatabilities of subsamples #16330 and #16331 are in good agreement with the target, estimated from the Horwitz equation. Therefore, homogeneity of the subsamples #16330 and #16331 was assumed.

To the participants, a set of samples (1 sample labelled #16330 and 1 sample labelled #16331) was sent on October 12, 2016.

2.5 ANALYSIS

The participants were asked to determine on both samples (#16330 and #16331) the Free Formaldehyde content and the Released Formaldehyde content with the analytical procedures that are routinely used in the laboratory.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the results more, but report as much significant figures as possible. It was also requested not to report 'less than' results, which are above the detection limit, because such results cannot be used for meaningful statistical calculations.

To get comparable results a detailed report form, on which the units were prescribed as well as the reference test methods and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The laboratories were also requested to confirm the sample receipt on the same data entry portal together with some details of the test methods used. A letter of instructions was added to the sample package.

3 RESULTS

During five weeks after sample dispatch, the results of the individual laboratories were gathered via the data entry portal www.kmpd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in the appendix 1 of this report. The laboratories are represented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that did not report test results at that moment.

Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results. Additional or corrected test results are used for the data analysis and the original results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies, Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<..." or '>..." were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. 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 the original test results per determination were submitted subsequently to Dixon's, Grubbs' and or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or

DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's. 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 on the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected 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. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the 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. In case no literature reproducibility was available, other target values were used.

The z-scores were calculated according to:

z_(target) = (test result - average of PT) / target standard deviation

The z (target) scores are listed in the 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. The usual interpretation of z-scores is as follows:

```
|z| < 1 good</li>
1 < |z| < 2 satisfactory</li>
2 < |z| < 3 questionable</li>
3 < |z| unsatisfactory</li>
```

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 452 numerical test results. Observed were 26 outlying test results, which is 5.8% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

In ISO14184-1:11 and ISO14184-2:11 some information on precision data is given. In table B.1 of ISO14184-1 and table C.2 of ISO14184-2 precision values are mentioned, but they were obtained using slightly different methods for calibration or performance than the ISO14184 methods. Therefore, it was concluded that reliable reproducibility data cannot be estimated and therefore target reproducibilities estimated from the Horwitz equation were used for evaluation.

One laboratory (2449) reported to have used ISO14184-1 for both Free and Released Formaldehyde. The test results of this laboratory for Released Formaldehyde were excluded from the statistical evaluations.

4.1 EVALUATION ANALYSIS DETAILS

For this PT some analysis details were requested (see appendix 2). Questions like: was the laboratory accredited, how long before use was the Acetylacetone prepared and for the Released Formaldehyde only: what was the intake amount of the sample and what was the volume of the water in the jar and at which temperature and how long took the development of the colour?

Looking at the answers given by the participants the following can be summarized:

About 90% of the participants are accredited according to ISO/IEC 17025.

Surprisingly 11% of the participants used the acetylacetone directly after preparation or on the same day instead of after 12 hours as is stated in method ISO14184-1. The other participants used it from 12 hours up to six weeks after preparation.

Roughly half of the participants that reported the volume of water used, used an intake of 50 ml water per gram of sample and the other half used 100 ml water per gram of sample. When evaluating these two above differences in the execution of the test, no relation was found between these test conditions and the reported test results.

However, the temperature, which was used to develop the colour, did appear to have an influence on the test result. From the 158 participants that reported this temperature, 6 participants reported to have used 58-65°C instead of 40°C to develop the colour. The test results of these six laboratories (2228, 2536, 2629, 2700, 3220, 3238) were excluded from the statistical evaluation.

4.2 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.

Sample #16330:

Free Formaldehyde content: This determination may be problematic for a number of laboratories. Nine 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 may be problematic. Two statistical outliers were observed and six test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the estimated reproducibility calculated using the Horwitz equation.

Sample #16331:

Free Formaldehyde content: This determination may be problematic. Seven 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 may be problematic. Four statistical outliers were observed and six test results were excluded from the statistical evaluation. The calculated reproducibility after rejection of the suspect data is not in agreement with the estimated reproducibility calculated using the Horwitz equation.

4.3 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	178	93.4	20.2	21.1
Released Formaldehyde	mg/kg	33	147.2	42.2	31.1

Table 5: reproducibilities of tests on sample #16330

Parameter	unit	n	average	2.8 * sd	R (target)
Free Formaldehyde	mg/kg	180	318.6	76.4	60.2
Released Formaldehyde	mg/kg	27	915	234	147

Table 6: reproducibilities of tests on sample #16331

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. However, the analysis of Released Formaldehyde gives more difficulties when the calculated target results are compared with the Horwitz equation. See also the discussions in paragraphs 4.1 and 6.

4.4 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2016 WITH PREVIOUS PTS

	November	November	October	October	October
	2016	2015	2014	2013	2012
Number of reporting labs	192	192	176	173	169
Number of results reported	452	415	193	378	338
Number of statistical outliers	26	14	8	13	12
Percentage outliers	5.8%	3.4%	4.1%	3.4%	3.6%

Table 7: Comparison with previous PTs

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

Doromotor	November	November	October	October	October
Parameter	2016	2015	2014	2013	2009 - 2012
Free Formaldehyde	8-9%	9-10%	8%	10-13%	7-15%
Released Formaldehyde	9-10%	17-22%	10%	9-10%	n.e.

Table 8: Comparison of relative uncertainties over the years

5 DISCUSSION

When the results of this interlaboratory study were compared to the Ecolabelling Standards and Requirements for Textiles in EU and with the similar Bluesign® BSSL (table 9), it was noticed that not all participants would make identical decisions about the acceptability of the textiles for the determined parameters.

Coolabal	haby alathaa	Öko-Tex 103	Öko-Tex 103	
Ecolabel	baby clothes	in direct skin contact	no direct skin contact	
Bluesign® BSSL	next to skin use	occasional skin contact	no skin contact	
Free Formaldehyde	.40	75	200	
extractable (mg/kg)	<16	75	300	
Released Formaldehyde	0.4	0.4	0.4	
(mg/m ³)	0.1	0.1	0.1	

Table 9: Bluesign® BSSL and 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 #16330, all reporting laboratories, except one, would accept the sample for the category "no (direct) skin contact" (<300 mg/kg). Only four laboratories would accept the sample for the category "(occasional) skin contact" (<75 mg/kg). All reporting laboratories, would reject the sample for the categories "Baby clothes" and "next to skin use" (<16 mg/kg).

For sample #16331, about 50% of the laboratories would reject and the other 50% of the laboratories would accept the sample for the category "no (direct) skin contact" (<300 mg/kg), since the mean of the formaldehyde for this sample is close to 300 mg/kg. All reporting laboratories would reject the sample for the categories "Baby clothes" and "next to skin use" (<16 mg/kg) and all laboratories, except one, for the category "(occasional) 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).

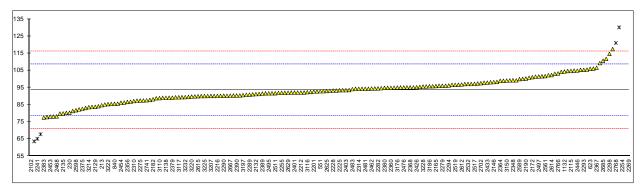
In this proficiency test, the Free Formaldehyde content and the Released Formaldehyde were determined. The variations observed for the Free Formaldehyde content and the Released Formaldehyde in this interlaboratory study are in line with observations in the previous proficiency tests. The variations 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.

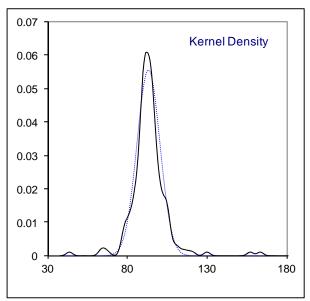
Determination of Free Formaldehyde content on sample #16630; results in mg/kg

Deterr	mination of Free I	Formaide	enyae con	itent on	sample	#16630;	results in mg/kg		
lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
213	ISO14184-1	84.6		-1.16	2379	ISO14184-1	89.028		-0.57
230		80.25		-1.74	2380	ISO14184-1			0.18
348	In house	94.894		0.20	2381	ISO14184-1			-0.40
362	ISO14184-1	97.31		0.52	2383	ISO14184-1			-2.11
551	ISO14184-1	92.78		-0.08	2385	ISO14184-1			-0.18
622	ISO14184-1	88.95		-0.58	2389	ISO14184-1			-0.26
623	ISO14184-1	105.9		1.66	2390	ISO14184-1			-0.40
840	ISO14184-1	85.4		-1.05	2401	GB/T2912	92		-0.18
1132	In house	104.284		1.45	2403	ISO14184-1	93.4		0.01
2102	In house	43.76	R(0.01)	-6.57	2404	ISO14184-1			-0.18
2115	ISO14184-1	104.7	11(0.01)	1.50	2423	ISO14184-1			-1.04
2129	ISO14184-1	83.7		-1.28	2426	ISO14184-1			0.23
2132	ISO14184-1	91.10	_	-0.30	2429	ISO14184-1			-1.61
2135	ISO14184-1	79.8	С	-1.80	2432	ISO14184-1			-0.81
2138	ISO14184-1	88.995	_	-0.58	2433	ISO14184-1			0.59
2139	ISO14184-1	91.00	С	-0.31	2439	ISO14184-1			0.29
2146	ISO14184-1	98.11		0.63	2442	ISO14184-1	95.72		0.31
2159	In house	93.18		-0.02	2446		104.84		1.52
2165	ISO14184-1	95.80		0.32	2449	ISO14184-1	104.053		1.42
2170	ISO14184-1	98.018		0.62	2453	ISO14184-1	78.0		-2.03
2172	ISO14184-1	101		1.01	2454	ISO14184-1			-0.97
2182	Japan Std 2016	88.08		-0.70	2456	ISO14184-1			-0.79
2184	ISO14184-1	94.2		0.70	2462	ISO14184-1	94.4		0.14
								D(0.04)	
2190	ISO14184-1	100.2		0.91	2467	ISO14184-1		R(0.01)	4.87
2201	ISO14184-1	92.64		-0.09	2468	ISO14184-1			-2.02
2212	JIS L1041	92.0		-0.18	2474	ISO14184-1	83.00		-1.37
2213	ISO14184-1	90.7		-0.35	2475	ISO14184-1	89.782		-0.47
2215	ISO14184-1	94.22		0.11	2476	ISO14184-1	95.0		0.22
2216	JISL1041/ISO14184-1	90.03		-0.44	2481	ISO14184-1	94.2		0.11
2225	ISO14184-1	93.38		0.00	2483	ISO14184-1			0.09
2226	ISO14184-1	104.835		1.52	2489	ISO14184-1			1.49
2228	ISO14184-1	93.1580		-0.03	2495	ISO14184-1			-0.24
								_	
2236	ISO14184-1	95.28		0.26	2497	ISO14184-1		С	1.07
2238					2506				
2241	ISO14184-1	65	C,R(0.05)	-3.76	2511	ISO14184-1			-0.23
2245	ISO14184-1	92.0		-0.18	2514	ISO14184-1			0.48
2246	ISO14184-1	92.35		-0.13	2517	ISO14184-1	97.135		0.50
2247	ISO14184-1	101.5		1.08	2519	ISO14184-1	96.5		0.42
2254	ISO14184-1	157.204	R(0.01)	8.46	2520				
2255	ISO14184-1	91.81	, ,	-0.20	2527	ISO14184-1	106		1.68
2256	ISO14184-1	77.68		-2.08	2532	ISO14184-1			0.48
2269	ISO14184-1	392.311	R(0.01)	39.62	2534	ISO14184-1			0.97
2275	ISO14184-1	87.40	11(0.01)	-0.79	2536	ISO14184-1			1.57
2277	ISO14184-1	85.33		-1.06	2546	ISO14184-1			1.16
2279	ISO14184-1	96		0.35	2553	ISO14184-1			-1.75
2280	ISO14184-1	96.48		0.41	2561	ISO14184-1			1.09
2282	ISO14184-1	94.46		0.15	2563	ISO14184-1			-0.21
2289	GB/T2912	90.80		-0.34	2567	ISO14184-1	94.56		0.16
2290	ISO14184-1	90.2		-0.42	2569	ISO14184-1	103		1.28
2293	ISO14184-1	105.247		1.58	2572	ISO14184-1	89.23		-0.55
2294	JIS L1041	96.11		0.37	2582	ISO14184-1	67.66	R(0.05)	-3.41
2295	ISO14184-1	79.7		-1.81	2589	ISO14184-1	99.87	(/	0.86
2296					2590	ISO14184-1	95.02		0.22
2298	JIS L1041	114.6		2.82	2591	In house	109.23		2.10
2301	010 L1041	114.0		2.02	2596	GB/T2912	111.7		2.10
	100444044								
2303	ISO14184-1	86.61		-0.89	2598	ISO14184-1	81.685		-1.55
2310	ISO14184-1	87.03		-0.84	2606	ISO14184-1	94.4		0.14
2311	ISO14184-1	91.56		-0.24	2612	ISO14184-1	96.71		0.44
2313	ISO14184-1	89.95		-0.45	2614	ISO14184-1	102.2		1.17
2314	ISO14184-1	94.2		0.11	2615	ISO14184-1	89.88		-0.46
2330	ISO14184-1	96.60		0.43	2625	ISO14184-1	92.91		-0.06
2347	ISO14184-1	96.97		0.48	2629	ISO14184-1	91.946		-0.19
2348	ISO14184-1	99.15		0.77	2638	ISO14184-1	87.67		-0.75
2351	ISO14184-1	96		0.35	2644	ISO14184-1	99.11		0.76
2356	ISO14184-1	86.5		-0.91	2646	ISO14184-1	89		-0.58
2358	ISO14184-1	93.4		0.01	2667	ISO14184-1	90.21		-0.42
2360	ISO14184-1	94.81		0.19	2670	ISO14184-1	117.4		3.19
2363	ISO14184-1	94.20		0.11	2674	ISO14184-1	92.8		-0.07
2364	ISO14184-1	98.88		0.73	2678	ISO14184-1	84	_	-1.24
2365	ISO14184-1	95.0		0.22	2685	ISO14184-1	110.58	С	2.28
2366	ISO14184-1	97.77		0.59	2689	ISO14184-1	105.37		1.59
2367	ISO14184-1	106.5		1.74	2700	ISO14184-1	82.19		-1.48
2368	ISO14184-1	95.02		0.22	2702	ISO14184-1	97.48		0.55
2370	ISO14184-1	91.15		-0.29	2719	ISO14184-1	90.29		-0.41
2373	ISO14184-1	86.17		-0.95	2728	ISO14184-1	90.08		-0.43
2375	ISO14184-1	82.5	С	-1.44	2730	ISO14184-1	101.29		1.05
2010	10014104-1	02.0	J		2100	.5017104-1	101.29		1.00

2741 2749 2755 2759 2761 2763 2766 2767 2768 3110 3116 3149 3150 3153 3154 3167 3172 3176	ISO14184-1	87.5 95 91.587 85.03 78 103.2 63.5 120.97 88.733 92.22 89.25 98.90 88.7 163.5 99 89.28 83.58 99.15 89.42 94.9	C,R(0.05) R(0.05)	-0.78 0.22 -0.23 -1.10 -2.03 1.30 -3.96 3.66 -0.61 -0.15 -0.54 0.73 -0.62 9.30 0.75 -0.54 -1.30 0.77 -0.52 0.20	3182 3186 3190 3191 3195 3197 3200 3207 3210 3212 3214 3222 3225 3228 3237 3238 3248 8005 8008	ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1	96.00 95.7 90 91.8658 100 90.5 90.2 90 93.475 94.78 83.45 89.7 85.25 89.95 95.5 89.35 98.23 90 92.68 93.032	С	0.35 0.31 -0.44 -0.20 0.88 -0.38 -0.42 -0.44 0.02 0.19 -1.31 -0.48 -1.07 -0.45 0.28 -0.53 0.65 -0.44 -0.09 -0.04
	normality n outliers mean (n) st.dev. (n) R(calc.) R(Horwitz)	OK 178 9 93.355 7.2026 20.167 21.129	RSD = 8%		Compare	e estimated R(ISO	14184-1:11) = 6.5	535	

Lab 2135 first reported: 61.60 Lab 2139 first reported: 59.67 Lab 2241 first reported: 62.02 Lab 2375 first reported: 62.5 Lab 2497 first reported: 135.42 Lab 2685 first reported: 120.035 Lab 2767 first reported: 127 Lab 3220 first reported: 139.8





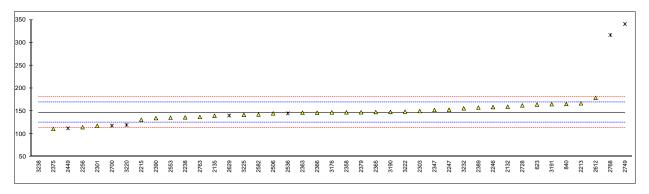
Determination of Released Formaldehyde content on sample #16630; results in mg/kg

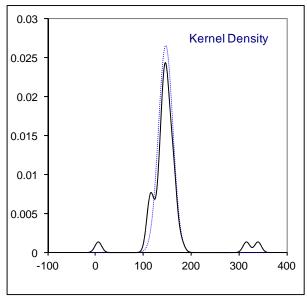
lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
213					2379	ISO14184-2	147.344		0.02
230					2380				
348					2381				
362					2383				
551					2385	10044404.0	457.7		
622	10044404.0	404.0		4.54	2389	ISO14184-2	157.7		0.95
623 840	ISO14184-2	164.3		1.54	2390 2401	ISO14184-2	134.6		-1.13
1132	ISO14184-2	165.5 		1.65 	2401				
2102					2403				
2115					2423				
2129					2426				
2132	ISO14184-2	159.50		1.11	2429				
2135	ISO14184-2	139.72		-0.67	2432				
2138					2433				
2139					2439				
2146					2442				
2159					2446	100444044	440.450		0.45
2165 2170					2449 2453	ISO14181-1	112.152	ex	-3.15
2170					2453				
2182					2456				
2184					2462				
2190					2467				
2201					2468				
2212					2474				
2213	ISO14184-2	167		1.79	2475				
2215	ISO14184-2	131.18		-1.44	2476				
2216					2481				
2225					2483				
2226 2228					2489				
2226					2495 2497				
2238	ISO14184-2	136.26		-0.98	2506	ISO14184-2	144.91		-0.20
2241	100141042				2511	100141042			
2245					2514				
2246	ISO14184-2	158.7		1.04	2517				
2247	ISO14184-2	153.0		0.53	2519				
2254					2520				
2255					2527				
2256	ISO14184-2	114.55		-2.94	2532				
2269					2534	A A TO C 4 4 0	444 0000		0.00
2275 2277					2536 2546	AATCC112	144.9232	ex	-0.20
2279					2553	ISO14184-2	135.456		-1.05
2280					2561	100141042			
2282					2563				
2289					2567				
2290					2569				
2293					2572				
2294					2582	ISO14184-2	142.54		-0.42
2295					2589				
2296 2298					2590 2591				
2301	ISO14184-2	117.765		-2.65	2596				
2303	ISO14184-2	149.98		0.25	2598				
2310	100111012				2606				
2311					2612	ISO14184-2	179.19		2.88
2313					2614				
2314					2615				
2330					2625				
2347	ISO14184-2	152.21		0.46	2629	AATCC TM112	140.236	ex	-0.62
2348					2638				
2351					2644				
2356 2358	ISO14184-2	147.2		0.00	2646 2667				
2360	13014104-2	147.2			2670				
2363	ISO14184-2	146.35		-0.07	2674				
2364					2678				
2365	ISO14184-2	147.8		0.06	2685				
2366	ISO14184-2	146.40		-0.07	2689				
2367					2700	AATCC112	118.00	ex	-2.62
2368					2702				
2370					2719	10044404.0	400.40	0	4.07
2373	10014104.0	110.05		2.26	2728	ISO14184-2	162.40	С	1.37
2375	ISO14184-2	110.95		-3.26	2730				

					i				
2741					3182				
2749	ISO14184-2	340.7	R(0.01)	17.42	3186				
2755					3190	ISO14184-2	148		0.08
2759					3191	GB/T2912	165.0888	С	1.61
2761					3195				
2763	ISO14184-2	137		-0.91	3197				
2766					3200				
2767					3207				
2768	ISO14184-2	316.51	R(0.01)	15.25	3210				
3110			,		3212				
3116					3214				
3117					3220	ISO14184-2	119.6	ex	-2.48
3118					3222	ISO14184-2	148.64		0.13
3146					3225	ISO14184-2	141.87		-0.48
3149					3228				
3150					3232	ISO14184-2	156.0		0.80
3153					3237	100111012			
3154					3238	In house	5.79	ex	-12.73
3167					3248	mmodoc		OX.	12.70
3172					8005				
3176	ISO14184-2	147.04		-0.01	8008				
3170	13014104-2	147.04		-0.01	0000				
	normality n outliers mean (n) st.dev. (n) R(calc.)	OK 33 2 (+6ex) 147.156 15.0735 42.206	RSD = 10%						
	R(Horwitz)	31.101			Compare	e estimated R (ISC	114104-2:11) = 80	.3	

Lab 2728 first reported: 3721.5

Test result of lab 2449 was excluded as ISO14184-1 was performed, which determines Free Formaldehyde and not released. Test result of lab 2536, 2629, 2700, 3220 and 3238 were excluded as a higher temperature than 40°C was used for the development of the color (see §4).





Determination of Free Formaldehyde content on sample #16631; results in mg/kg

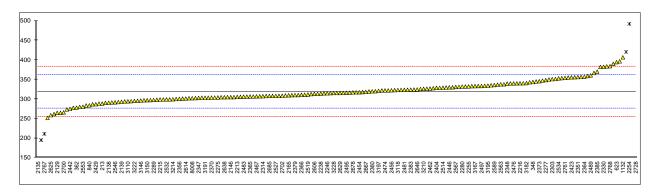
lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
213	ISO14184-1	288.4		-1.41	2379	ISO14184-1	310.029		-0.40
230		358.66		1.87	2380	ISO14184-1	298.82		-0.92
348	In house	343.337		1.16	2381	ISO14184-1	301.11		-0.82
362	ISO14184-1	277.72		-1.91	2383	ISO14184-1	324		0.25
551	ISO14184-1	277.33		-1.93	2385	ISO14184-1	370		2.40
622	ISO14184-1	317.26		-0.06	2389	ISO14184-1	317.7		-0.04
623	ISO14184-1	394.2		3.53	2390	ISO14184-1	264.7		-2.52
840	ISO14184-1	283.9		-1.62	2401	GB/T2912	322		0.16
1132 2102	In house In house	406.490 331.25		4.10 0.59	2403 2404	ISO14184-1 ISO14184-1	328.8 328.67		0.48 0.47
2115	ISO14184-1	326.2		0.35	2423	ISO14184-1	355.39		1.72
2129	ISO14184-1	264.7		-2.52	2426	ISO14184-1	324.1		0.26
2132	ISO14184-1	316.2		-0.11	2429	ISO14184-1	286.74		-1.49
2135	ISO14184-1	58.2	C,R(0.01)	-12.16	2432	ISO14184-1	286.525		-1.50
2138	ISO14184-1	290.575		-1.31	2433	ISO14184-1	332.04		0.63
2139	ISO14184-1	293.00		-1.20	2439	ISO14184-1	347.046		1.33
2146	ISO14184-1	304.77		-0.65	2442	ISO14184-1	275.06		-2.03
2159 2165	ISO14184-1	309.50		-0.43	2446 2449	ISO14184-1	329.93 322.008		0.53 0.16
2170	ISO14184-1	310.708		-0.43	2449	ISO14184-1	261.5		-2.67
2170	ISO14184-1	319.89		0.06	2454	ISO14184-1	317.26		-0.06
2182	ISO14184-1	292.23		-1.23	2456	ISO14184-1	322.25		0.17
2184	ISO14184-1	310.1		-0.40	2462	ISO14184-1	326.9		0.39
2190	ISO14184-1	355.1		1.70	2467	ISO14184-1	306.863		-0.55
2201	ISO14184-1	312.58		-0.28	2468	ISO14184-1	251.5		-3.13
2212	JIS L1041	294.6		-1.12	2474	ISO14184-1	322.00		0.16
2213	ISO14184-1	305.3		-0.62	2475	ISO14184-1	291.327		-1.27
2215	ISO14184-1	298.75		-0.93	2476	ISO14184-1	340.0		1.00
2216 2225	JIS L1041/ISO14184-1 ISO14184-1	340.27 323.70		1.01 0.24	2481 2483	ISO14184-1 ISO14184-1	323.5 306		0.23 -0.59
2226	ISO14184-1	344.767		1.22	2489	ISO14184-1	360.4		1.95
2228	15014104-1				2495	ISO14184-1	316.1		-0.12
2236	ISO14184-1	420.41	R(0.05)	4.76	2497	ISO14184-1	333.26		0.68
2238	ISO14184-1	313.78	()	-0.23	2506	ISO14184-1	332.83		0.66
2241	ISO14184-1	306.52		-0.56	2511	GB/T2912	303.7		-0.70
2245	ISO14184-1	325.5		0.32	2514	ISO14184-1	329.0165		0.49
2246	ISO14184-1	314.75		-0.18	2517	ISO14184-1	333.20		0.68
2247	ISO14184-1	342.9	D(0.04)	1.13	2519	ISO14184-1	311.6		-0.33
2254 2255	ISO14184-1 ISO14184-1	492.017 332.13	R(0.01)	8.10 0.63	2520 2527	ISO14184-1	308		 -0.50
2256	ISO14184-1	315.36		-0.15	2532	ISO14184-1	299		-0.50
2269	ISO14184-1	333.700		0.70	2534	GB/T2912	352.78		1.60
2275	ISO14184-1	304.00		-0.68	2536	ISO14184-1	329.4734		0.51
2277	ISO14184-1	348.4		1.39	2546	ISO14184-1	291.5606		-1.26
2279	ISO14184-1	340		1.00	2553	ISO14184-1	280.213		-1.79
2280	ISO14184-1	331.91		0.62	2561	ISO14184-1	309.0		-0.45
2282	ISO14184-1	308.03		-0.49	2563	ISO14184-1	337.6		0.89
2289 2290	ISO14184-1	297.50 298.3		-0.99 -0.95	2567	ISO14184-1	330.75		0.57 -0.92
2290	ISO14184-1 ISO14184-1	296.3 381.97		2.96	2569 2572	ISO14184-1 ISO14184-1	299 307.8		-0.92
2294	JIS L1041	340.30		1.01	2582	ISO14184-1	355.77		1.74
2295	ISO14184-1	309.9		-0.41	2589	ISO14184-1	335.65		0.80
2296					2590	ISO14184-1	350.05		1.47
2298	JIS L1041	389.9		3.33	2591	In house	356.65		1.78
2301					2596	GB/T2912	273.4		-2.11
2303	ISO14184-1	350.76		1.50	2598	ISO14184-1	336.63		0.84
2310	ISO14184-1	302		-0.78	2606	ISO14184-1	313.18		-0.25
2311 2313	ISO14184-1 ISO14184-1	295.3 306.2		-1.09 -0.58	2612 2614	ISO14184-1 ISO14184-1	351.09 301.2		1.52 -0.81
2313	ISO14184-1	307.2		-0.53	2615	ISO14184-1	323.41		0.22
2330	ISO14184-1	382.26		2.97	2625	ISO14184-1	258.7		-2.80
2347	ISO14184-1	302.73		-0.74	2629	ISO14184-1	315.363		-0.15
2348	ISO14184-1	339.55		0.98	2638	ISO14184-1	304.57		-0.66
2351	ISO14184-1	356		1.75	2644	ISO14184-1	283.58		-1.64
2356	ISO14184-1	300.4		-0.85	2646	ISO14184-1	324.5		0.28
2358	ISO14184-1	337.7		0.89	2667	ISO14184-1	318.21		-0.02
2360	ISO14184-1	319.79		0.06	2670	ISO14184-1	303.3		-0.72
2363 2364	ISO14184-1 ISO14184-1	313.32 357.00		-0.25 1.79	2674 2678	ISO14184-1 ISO14184-1	307.6 317		-0.51 -0.08
2365	ISO14184-1	306.4		-0.57	2685	ISO14184-1	307.759		-0.08
2366	ISO14184-1	310.68		-0.37	2689	ISO14184-1	383.45		3.03
2367	ISO14184-1	353.5		1.63	2700	ISO14184-1	265.99		-2.46
2368	ISO14184-1	335.16		0.77	2702	ISO14184-1	308.37		-0.48
2370	ISO14184-1	303.69		-0.70	2719	ISO14184-1	303.38		-0.71
2373	ISO14184-1	345.42		1.25	2728	ISO14184-1	1678.00	R(0.01)	63.50
2375	ISO14184-1	279.8		-1.81	2730	ISO14184-1	304.56		-0.66

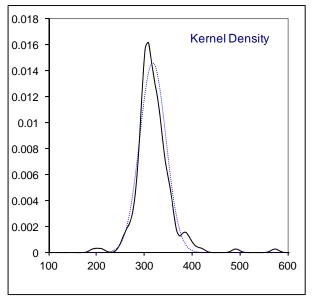
2749 2755 2759 2761 2763 2766 2767	ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1	304.7 290 319.304 354.02 195 297.2 211	C,R(0.01) C,R(0.05)	-0.65 -1.34 0.03 1.65 -5.77 -1.00 -5.03	3186 3190 3191 3195 3197 3200 3207	ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 JIS L1041	340.2 315 303.3166 334 320.9 307.0 305.4	C C	1.03 1.01 -0.17 -0.71 0.72 0.11 -0.54 -0.62
2768 3110 3116 3117 3118 3146	ISO14184-1 ST part 3 2016 ISO14184-1 ISO14184-1 ISO14184-1	383.79 294.135 329.96 288.30 323.07 296.1	0,11(0.00)	3.04 -1.14 0.53 -1.42 0.21 -1.05	3210 3212 3214 3220 3222 3225	ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1	325.96 302.24 299.45 396.3 295.04 366.53		0.34 -0.76 -0.89 3.63 -1.10 2.24
3149 3150 3153 3154 3167 3172 3176	ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1	574.1 297 293.89 322.68 332.94 315.57 314.6	R(0.01)	11.93 -1.01 -1.15 0.19 0.67 -0.14 -0.19	3228 3232 3237 3238 3248 8005 8008	ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 GB/T2912 JIS L1041-B ISO14184-1	315.3 296.76 304.78 300 327.67 302.153		-0.15 -1.02 -0.65 -0.87 0.42 -0.77
	normality n outliers mean (n) st.dev. (n) R(calc.) R(Horwitz)	OK 180 7 318.609 27.2686 76.352 60.187	RSD = 9%		Compare	e estimated R(ISO)14184-1:11) = 17.	842	

Lab 2135 first reported: 45.11 Lab 2763 first reported: 226 Lab 2767 first reported: 422

Lab 2767 first reported: 422 Lab 3190 first reported result as Released Formaldehyde

Lab 3191 first reported: 998.3207





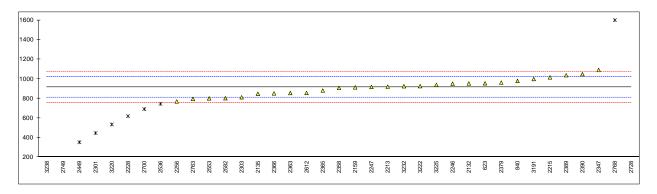
Determination of Released Formaldehyde content on sample #16631; results in mg/kg

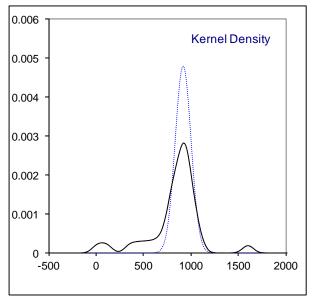
233	lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
388	213						ISO14184-2			
Section										
561										
BOS BOS										
SOI SOI										
1132							ISO14184-2			
2102		15014184-2								
2115										
1972 1972										
1.28 SO14184-2 SO15 SO14184-2 SO14184-2 SO15 SO14184-2 SO15 SO14184-2 SO15 SO14184-2 SO15 SO14184-2 SO15 SO14184-2 SO14184-2 SO15 SO14184-2 SO15 SO14184-2 SO15 SO14184-2 SO15 SO14184-2 SO15 SO14184-2 SO15 SO14184-2 SO14184-2 SO15 SO14		100								
2138										
2439		13014104-2								
2165										
2170										
2465		In house					100444044		۵۷	
2456							15014164-1		ex	
2190										
2290										
2201										
2212										
2215 SO14184-2										
2216 — 2481 — — 2226 — 2483 — — 2228 AATCC112 618.303 ex -5.66 2495 — — 2238 — 2497 — — — — 2241 — 2511 — — — — 2244 — 2514 — — — — 2246 ISO14184-2 950.5 0.68 2517 — — — 2254 ISO14184-2 917.5 0.05 2519 — — — 2256 ISO14184-2 976.86 — 2527 — — — — 2256 ISO14184-2 768.86 — — 2534 — <td>2213</td> <td></td> <td>920.1</td> <td></td> <td>0.10</td> <td>2475</td> <td></td> <td></td> <td></td> <td></td>	2213		920.1		0.10	2475				
2225 — 2483 — </td <td></td> <td>ISO14184-2</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>		ISO14184-2								
2226										
2228 AATCC112										
2238		AATCC112	618.303	ex	-5.66					
2241 — 2511 — — 2514 — — 2246 ISO14184-2 950.5 0.68 2517 — <										
2245 — 2514 — — 2514 —										
2246 SO14184-2 950.5 0.68 2517										
2254		ISO14184-2	950.5		0.68					
2255		ISO14184-2								
2256 ISO14184-2 768.86 -2.79 2534										
2269 2534 AATCC112 744.2557 ex -3.25 2277 2546 2279 2553 ISO14184-2 800.584 -2.18 2280 2563 2282 2563 2289 2567 2290 2569 2293 2572 2294 2582 ISO14184-2 801.93 -2.16 2295 2589 2296 2589 2296 2590 2301 ISO14184-2 445.790 R(0.01) -8.94 2596 2301 ISO14184-2		ISO14184-2								
2277										
2279 2553 ISO14184-2 800.584 -2.18 2280 2563 2289 2567 2290 2569 2293 2572 2294 2582 ISO14184-2 801.93 -2.16 2296 2589 2296 2590 2298 2590 2301 ISO14184-2 445.790 R(0.01) -8.94 2596 2301 ISO14184-2 811.99 -1.96 2598							AATCC112		ex	
2280 2561							ISO14184-2			
2282							100141042			
2290 2569	2282					2563				
2293 2572										
2294 2582 ISO14184-2 801.93 -2.16 2295 2589 2298 2590 2301 ISO14184-2 445.790 R(0.01) -8.94 2596 2303 ISO14184-2 811.99 -1.96 2598 2311 2606 2311 2612 ISO14184-2 856.02 -1.12 2313 2615 2314 2615 2330 2625 2347 ISO14184-2 1090.55 3.35 2629 2348 2638 2356 2644										
2295 2589 2590							ISO14184-2			-2.16
2298 2591 2301 ISO14184-2 445.790 R(0.01) -8.94 2596 2310 2606 2311 2612 ISO14184-2 856.02 -1.12 2313 2615 2314 2615 2330 2625 2347 ISO14184-2 1090.55 3.35 2629 2348 2638 2351 2644 2356 2646 2360 2670 2363 ISO14184-2 855.31 -1.14 2674 2366 ISO14184-2 881.0 -0.65 2685	2295					2589				
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2367 2700 AATCC112 690.67 ex -4.28 2368 2702 2370 2719 2373 2728 ISO14184-2 3721.15 R(0.01) 53.49										
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,							ISO14184-2		R(0.01)	
							- · -		(- 5-)	

2741					3182				
2749	ISO14184-2	108.4	R(0.01)	-15.38	3186				
2755					3190				
2759					3191	ISO14184-2	998.3207	С	1.59
2761					3195				
2763	ISO14184-2	795		-2.29	3197				
2766					3200				
2767					3207				
2768	ISO14184-2	1599.95	R(0.01)	13.06	3210				
3110			, ,		3212				
3116					3214				
3117					3220	ISO14184-2	533.8	ex	-7.27
3118					3222	ISO14184-2	925.69		0.20
3146					3225	ISO14184-2	938.91		0.46
3149					3228				
3150					3232	ISO14184-2	924.5		0.18
3153					3237				
3154					3238	In house	10.21	ex	-17.25
3167					3248	111110000		O/C	
3172					8005				
3176					8008				
3170					0000				
	normality n outliers mean (n) st.dev. (n) R(calc.) R(Horwitz)	OK 27 4 (+6ex) 914.991 83.3973 233.512 146.878	RSD = 9%		Compor	e estimated R (ISC	MAAQA 2:44\ _ 00	. 2	
	T(TIOTWILE)	170.070			Jonipan	c commated it (100	71 + 10 + 2.11 = 00		

Lab 3191 first reported: 165.0888

Test result of lab 2449 was excluded as ISO14184-1 was performed, which determines Free Formaldehyde and not released. Test result of lab 2228, 2536, 2700, 3220 and 3238 were excluded as a higher temperature than 40°C was used for the development of the color (see §4).





labnrs	ISO/IEC17025 accredited?	Acetylacetone preparation time to test	Released Formald.: Intake of sample in grams	Released Formald.: volume of water in mls	Released Formald.: temp. and time for colour to develop
213	Yes	ONE HOUR	J. J		
230		0.12.1.00.1			
348	Yes	4 days maximun			
362	Yes	24 h			
551	Yes	12h	1,25 g		30 min / 40°C
622	Yes	fresh before use	, , ,		40°C, 30 minute
623	Yes	Freshly prepared	1 g		40 C for 30 mins
840	Yes	12h	1g		40oC, 30 minnutes
1132	Yes	-			
2102					
2115	Yes	immediately before			
2129	Yes	same day	1 g		40°C
	.,		#16630 - 1 g;		
2132	Yes	12 hr before use	#16631 - 1 g		40C for 30 min
2135	Yes	2 days	0,8		40°C 30min
2138	Yes	within 24 hours			
2139	Yes	12 hours	16630: 1,3 g and		
2146	Yes	One day before	16631: 0,8 g		30 minutes in 40 °C
2159	Yes	30 minutes	1 gram		at 40 °C for 30 minutes
2165	Yes	24 hours	NA		NA
2170	Yes	a day before use.			
2172	Yes	The same day			
2182	Yes	24-48 hours before			
2184	Yes	1 day			
2190	No	the day analysis	2.5g for #16630 1g for #16631		40°C
2201	No	>12h	N/A		N/A
2212	Yes	One day before use	1,471		
2213	Yes	1 Week	0.5 g		40 °C temp and 30 min
2215	Yes	15 hours	- 0.0 g		40°C 30min
2216	Yes	4 Weeks and 4 Days before analysis	1.0115 (16630) 1.0025 (16631)		40°C+/-2°C
2225	Yes	over 12 hours			
2226	Yes	24 hrs before use			
2228	Yes	12 hours before use	1.0004 g		58±1°C and 6 min
2236	Yes	2 weeks prior to use			
2238	Yes	about 20 hours	One 1.0024g ,another 0.9994g		40°C,30min
2241	Yes	12h			
2245	Yes	Three days			
2246	Yes	12 hours before use	1g		40°Cfor 30 minutes
2247	Yes	7 days	0.5257 g		30 mins at 40
2254	Yes	18 hours			
2255	Yes	12 hrs			
2256	Yes	24 Hours	0.5 gram		40°C for 30 min
2269	Yes				
2275	Yes				
2277	Yes				
2279	Yes	more than 12 hours			
2280	Yes	two days			
2282	Yes	About 15 to 18 hours			
2289	Yes	5days.			

	ISO/IEC17025 accredited?	Acetylacetone preparation	Released Formald.: Intake of sample	Released Formald.: volume of water	Released Formald.: temp. and time for
labnrs		time to test	in grams	in mls	colour to develop
2290					
2293	No	in the same day			
2294	Yes	24 hours before the test			
2295	Yes	12 hours			
2296					
2298	Yes	One week			
2301	Yes	1 day	1 gram #16630 - 0.5168g,		40 °C and 30 min
2303	No	Two weeks	#16631 - 0.4192g		40 °C for 30 min
2310	Yes	2 days			
2311	Yes	3days			
2313	Yes	one day before			
2314	Yes	One month			
2330	Yes	12hrs	-		-
2347	Yes	24hrs	0.5 gram		40°C:30 minutes
2348	Yes	36H			
2351	Yes	after 12hours			
2356	Yes	One week	NA		NA
2358	Yes	12 hours	1 g		40°C for 30 minutes
2360	Yes	24 hour above	/		/
2363	Yes	12h	0.5g		at 40°C for 30 min
2364	Yes	12hours			
2365	Yes	12 hours	0.5grams		40°C 30mins
2366	Yes	20¡À0.25H	0.5g		40°C,30min
2367	Yes	8 days			
2368	Yes				
2370	Yes	12hr			
2373	Yes	4days			
2375	Yes	before 12 hours	0.5020 g		49 C - 20 hours
2379	No	12 hour	0.5g		40 +/- 2°C, 30 minute
2380	Yes	12 Hours	N/A		N/A
2381	Yes	2 DAYS AGO	N/A		N/A
2383	Yes	12h	1g		40°C,30min
2385	Yes	1 week			40 °C; 30 minutes
2389	Yes	07 days	1 gram		40°C, for 30 minutes
2390	Yes	3 days before	1.0 g		40C and 30 minutes
2401	Yes				
2403	Yes	2 days	/		/
2404	Yes	One day.			
2423	No	same day			
2426	Yes	Before 02 days of test			
2429	Yes	5 days			
2432	No	12h			
2433	Yes	12 hours			
2439	Yes	24 hours			40°C and 30 minutes
2442	Yes	3 Days			
2446	No	1 h before			
2449	Yes	one week	1.007 gram		40°C: 30 minutes
2453	Yes	5 DAYS	1.5g		40°C/30 min
2454	Yes	held for 12h before use			
2456	Yes	twelve days before			
2462	Yes	24 hours			
2467	No	7 days			

	ISO/IEC17025 accredited?	Acetylacetone preparation	Released Formald.: Intake of sample	Released Formald.:	Released Formald.: temp. and time for
labnrs	accredited?	time to test	in grams	in mls	colour to develop
2468	Yes	3 weeks	N/A		N/A
2474	Yes	24 hours before use.	147.		1,471
2475	Yes	Min 12H			
2476	Yes	24 hours			
2481	No	One our before use			
2483	Yes	0.00 00.000.000			
2489	Yes	6 weeks			
2495	Yes	1 day before			
2497	Yes	one week			
2506	Yes	2 Weeks	1.0091g & 1.0088g		50°C for 20 hrs
2511	Yes	Z WCCR3	1.003 1g & 1.0000g		30 0 101 20 1113
2514	Yes	12 hours			
2517		12 110015			
2517	No	on the day.	1.0 g		40°C 30min
2520		on the day.	1.0 g		40 C 30IIIII
2527	Yes	2 days later			
		3 days later			
2532	Yes	24 hrs	-		-
2534	Yes	7 days	For 16630 (0.5001 g) &		
2536	Yes	24 hrs	For 16631 (1.0010 g)		58 (°C) & 6 (minutes)
2546	Yes	12 h	1g		40°C 40 minutes
2553	Yes	12 days	1.0039 g		40 °C for 30 min.
2561	Yes				
2563	Yes	one day before use			
0567	Voc	mara than 12 haura	#16630: 0.6067 grams ;		NC
2567	Yes	more than 12 hours	#16631: 0.5121 grams		NC
2569	 V	041			
2572	Yes	24 hours	0.0040		(40 0)00 (00 5)
2582	Yes	5 days	0.8043		$(40 \pm 2)^{\circ}$ C, (30 ± 5) min.
2589	Yes				
2590	Yes	just before use			
2591	Yes	2 days			
2596	No				
2598	Yes	before one week ago.	It was 1.004 gram.		40°Cfor 30 min
2606	Yes	1 Day			
2612	Yes	10 days	0,5g		40 °C for 15 min
2614	Yes	4 weeks			
2615	Yes	Around one week	/		/
2625	Yes	6 weeks			40C, 30 minutes
2629	Yes	24 hours	1.0 gram		65°C in 4 hours
2638	No	2 days before			
2644	Yes	30 minutes			
2646	Yes	on day of analysis			
2667				50 ml	
2670	Yes	eight days			
2674	Yes	24h		50 ml	
2678	Yes	One day	1 gram	1L	40°C 30 minutes
2685	No	4 days			
2689	Yes	24 hours			
2700	Yes	At least 12 hours	1.0 gram		65°C, 4hours
2702	No	2 Days		100 ml	
2719	Yes			#16630 - 50 ml; #16631 - 50 ml	
		20 hours	1 gram		40°C 20 hours
2728	Yes	20 hours	1 gram	50	49°C, 20 hours

sed Formald.: and time for	ten	Released Formald.: volume of water in mls	Released Formald.: Intake of sample in grams	Acetylacetone preparation time to test	ISO/IEC17025 accredited?	lohoro
r to develop	COI	III IIIIS	III grains	48 hours before used	No	labnrs
			1	Held for 12hrs	No Yes	2730 2741
	40°	100 ml	0.5 and 1.0 a	six months	Yes	
			0.5 and 1.0 g			2749
	40°	50 ml	1 gram	1day	Yes	2755
		NA		2 days	Yes	2759
				3 days	Yes	2761
and 30 minutes	40°		0.5394 gm	12 Hours	Yes	2763
				Two days	No	2766
				1 day	No	2767
		100 ml				2768
		N/A			Yes	3110
				4 days	Yes	3116
		25 ml		Not less than 12 hours.	Yes	3117
		50ml		1 day before	Yes	3118
		100mL		12 h	Yes	3146
			0,5 g	3 days	Yes	3149
				24 h	Yes	3150
	a jar NA	50 ml of water into a ja	NA	at least 12 hours	Yes	3153
					Yes	3154
		50ml		24h	Yes	3167
					Yes	3172
0 min.	400		0,78 g	2 days	Yes	3176
		50mL		24 hours	Yes	3182
		50 ml			Yes	3186
 20h	49°		1g	6 weeks	No	3190
30min	40°		#16630:0.5194 gram; #16631:0.2686 gram	Free form.18h; Released form.:34h	Yes	3191
	/	25 mL	1	24 hour	Yes	3195
				17 hours	Yes	3197
			1g	The day before test	Yes	3200
			1 gram	Within 1 week	Yes	3207
				2 mois	Yes	3210
				1 day	Yes	3212
				•	Yes	3214
or 6 minutes	58°		5ml	Prepared on 2/11/2016 and test was conducted on 3/11/2016	Yes	3220
30 min	40°		0.5 g	2 week	Yes	3222
1200minutes			~0.5g/per sample	24 hours	Yes	3225
	NA		NA	> 12hours	Yes	3228
				Fresh	Yes	3232
			-			
10 mn	60°		1,7268 g			
		50 mL				
				, -		
30 min	40° 50° NA	50 mL 50ml	0.5 g ~0.5g/per sample	2 week 24 hours > 12hours	Yes Yes Yes	3222 3225 3228

Number of participants per country

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

Abbreviations:

C = final result after checking of first reported suspect result

= straggler in Double Grubbs' outlier test

 $\begin{array}{ll} D(0.01) &= \text{outlier in Dixon's outlier test} \\ D(0.05) &= \text{straggler in Dixon's outlier test} \\ G(0.01) &= \text{outlier in Grubbs' outlier test} \\ G(0.05) &= \text{straggler in Grubbs' outlier test} \\ DG(0.01) &= \text{outlier in Double Grubbs' outlier test} \\ \end{array}$

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

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

Literature:

DG(0.05)

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