

Results of Proficiency Test Free Formaldehyde in textile October 2012

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) and Thai Green Label (Thailand).

Since several years, the Institute for Interlaboratory Studies (iis) organises a proficiency scheme for Free Formaldehyde in textile. Also, this year this scheme is part of the proficiency testing program 2012/2013.

In this interlaboratory study 175 laboratories in 38 different countries participated. See appendix 2 for the number of participating laboratories per country.

In this report, the results of this 2012 proficiency test are presented and discussed.

2 SET UP

The Institute for Interlaboratory Studies in Spijkensisse was the organiser of this proficiency test. Sample preparation and analyses of fit for use and homogeneity were subcontracted. In this Proficiency Test, it was decided to use two different samples (#12122 and #12123, each approx. 3 grams) which were treated to find two different concentration levels of Free Formaldehyde. Participants were requested to report results with one extra figure. These unrounded results were preferably used for the statistical evaluations.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkensisse, the Netherlands, has implemented a quality system based on ISO guide 43, ILAC-G13:2007 and ISO 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 January 2010 (iis-protocol, version 3.2) which can be downloaded from www.iisnl.com.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

The two different hosiery fabric textile samples (labelled sample #12122 and #12123) were divided over 190 subsamples of approx. 3 grams.

The homogeneity of both samples was checked on respective 15 and 13 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		Free Formaldehyde in mg/kg
Sample #12122-1	137	Sample #12123-1	40.9
Sample #12122-2	135	Sample #12123-2	40.6
Sample #12122-3	133	Sample #12123-3	41.1
Sample #12122-4	132	Sample #12123-4	42.3
Sample #12122-5	134	Sample #12123-5	40.7
Sample #12122-6	136	Sample #12123-6	41.5
Sample #12122-7	140	Sample #12123-7	41.8
Sample #12122-8	136	Sample #12123-8	42.1
Sample #12122-9	135	Sample #12123-9	41.2
Sample #12122-10	138	Sample #12123-10	41.5
Sample #12122-11	135	Sample #12123-11	42.5
Sample #12122-12	137	Sample #12123-12	40.8
Sample #12122-13	135	Sample #12123-13	40.6
Sample #12122-14	137	Sample #12123-14	40.5
Sample #12122-15	138	Sample #12123-15	42.4

Table 1: homogeneity test results of subsamples #12122 and #12123

From the above test results, the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities in agreement with the procedure of ISO 13528 (Annex B2) or with the repeatability of the reference method, in the next table:

	Free Formaldehyde in mg/kg Sample #12122	Free Formaldehyde in mg/kg Sample#12123
r	5.8	2.0
Reference test	Horwitz	Horwitz
0.3*R _(reference test)	8.7	3.2

Table 2: repeatabilities of subsamples #12122 and 12123

From the above results of the homogeneity tests, the repeatabilities were calculated. The calculated repeatabilities for samples #12122 and #12123 are both in good agreement with 0.3 times the estimated target reproducibilities, calculated using the Horwitz equation. Therefore, homogeneity of all subsamples was assumed.

In total approx. 3 grams of each of the samples, #12122 and #12123 were sent to the participating laboratories on October 10, 2012.

2.5 ANALYSES

The participants were asked to determine on samples #12122 and #12123 the concentrations of Free Formaldehyde with the analytical procedure that is routinely used in the laboratory. To get comparable results, detailed report forms were sent together with each set of samples. On the report from the requested Free Formaldehyde concentration, including the units was pre-printed. Also a letter of instructions was sent along.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original data are tabulated in the appendices of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder fax was sent to those laboratories that had not yet reported. 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, see lit.5) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. Additional or corrected data are placed under 'Remarks' in the result tables in appendix 1. A list of abbreviations used in the tables can be found in appendix 4.

3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2).

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

Before further calculations, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test. In the case of an abnormal distribution, the statistical evaluation should be used with care.

According to ISO 5725 (1986 and 1994, lit.7 and 8) the original results per determination were submitted subsequently to Dixon's and Grubbs' 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. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test. Both outliers and stragglers were not included in the calculations of averages and standard deviations. Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have 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 method is producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nos.14 and 15).

3.3 Z-SCORES

To evaluate the performance of the individual participating laboratories the z-scores were calculated.

In order to be able to have an objective evaluation of the performance of the individual participants, it was decided to evaluate this performance against the literature requirements. Therefore, the z-scores were calculated using a target standard deviation. This target standard deviation was calculated from the literature reproducibility by division with 2.8.

The $z_{(\text{target})}$ -scores were calculated according to:

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

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

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

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

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
$1 < z < 2$	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. Six laboratories did not report any test results and eleven other laboratories reported results after the final reporting date.

Finally, the 169 reporting laboratories send in total 338 numerical results. Observed were 12 statistical outlying results, which is 3.6% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

For sample #12123, a not normal distribution was found. Therefore the statistical evaluation for this sample should be used with due care.

In ISO14184:11 (Table B.1) precision data are given. In this table B.1 "approximate accuracy" values are mentioned. These values are probably the calculated repeatability standard deviations. Note also that is mentioned under table B.1 "that the method in this part of ISO14184:11 uses a different calibration graph from that used in the determination of the above-mentioned results".

Due to the lack of the reproducibility data in this test method the reproducibilities estimated by the Horwitz equation were used for evaluation.

4.1 EVALUATION PER SAMPLE

In this section, the samples #12122 and #12123 are discussed. All statistical results reported on the textile samples are summarised in appendix 1.

Sample #12122: No analytical problems were found for this determination. Six statistical outliers were observed. The calculated reproducibility is, after rejection of the statistical outliers in full agreement with the estimated reproducibility calculated using the Horwitz equation.

Sample #12123: No analytical problems were found for this determination. Six statistical outliers were observed. The calculated reproducibility is, after rejection of the statistical outliers in full 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 #12122	mg/kg	163	166.1	38.5	34.5
Free Formaldehyde #12123	mg/kg	163	103.7	25.8	23.1

Table 3: reproducibilities of textile samples #12122 and #12123

From the above tables it can be concluded that, without statistical calculations, the group of participating laboratories does not have any difficulties with the analysis when compared with the target results calculated 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 iis PTs (see below table).

Parameter	October 2012	October 2011	October 2010	October 2009	October 2008	November 2007
Free Formaldehyde	23-25%	31-41%	21-24%	24-33%	19-42%	24-25%

Table 4: Development of relative reproducibilities of Free Formaldehyde 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 5), it could be noticed that not all participants would make identical decisions about the acceptability of the textiles for the determined parameters when evaluating the test results against the Ecolabel requirements.

Ecolabel	baby clothes	Öko-Tex 103 no direct skin contact	Öko-Tex 103 in direct skin contact	Decoration material
Free Formaldehyde in mg/kg	<16	300	75	300

Table 5: 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.

All laboratories would accept both samples #12122 and #12123 for the categories “Öko-Tex 103 no direct skin contact” and “Decoration material” (<300 mg/kg)

Also, all laboratories would reject both samples #12122 and #12123 for the category “baby clothes”.

However, for the category “Öko-Tex 103 in direct skin contact” two laboratories (2186 and 2411) would have accepted sample #12123. All other laboratories would have rejected both samples for this category.

General

The Free Formaldehyde content was determined at two different levels. The spreads observed in this interlaboratory study are in line with the previous round robins. The spreads observed in this interlaboratory study are not caused by just one critical point in the analysis. Consequently, the reproducibilities 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

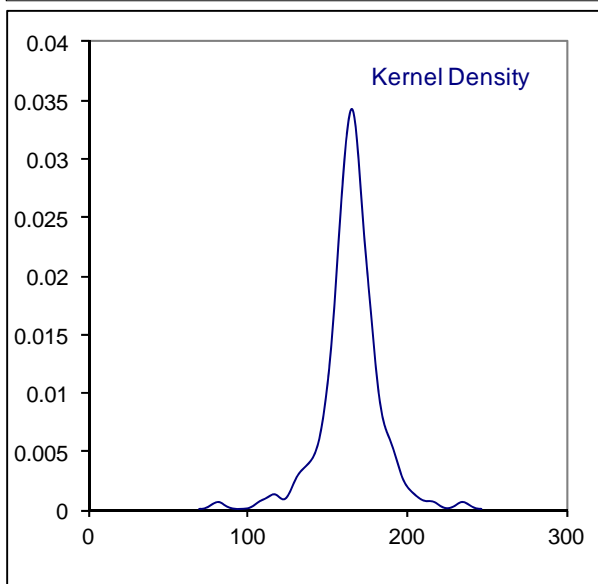
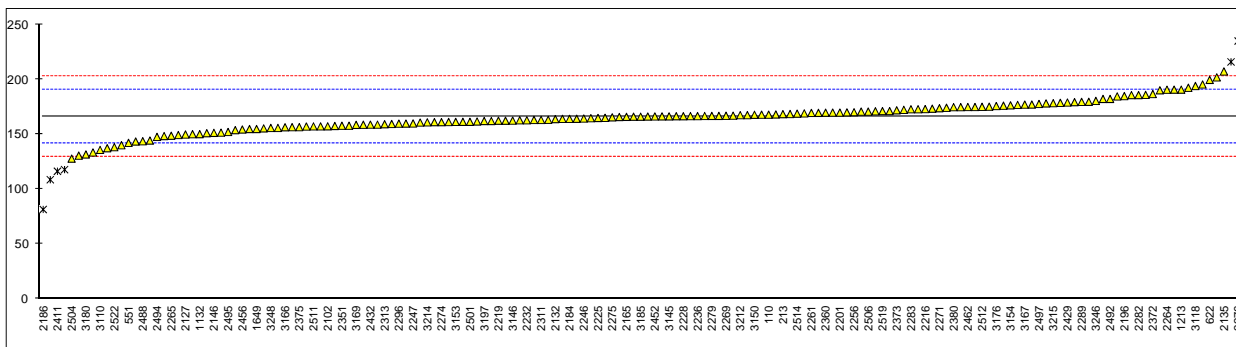
Determination of Free Formaldehyde on sample #12122; results in mg/kg

lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110	JIS L1041B	168.1		0.17	2356	ISO14184-1	166.4		0.03
213	ISO14184-1	168.6		0.21	2358	ISO14184	170.0		0.32
339	ISO14184-1	160		-0.49	2359	ISO14184-1	172.5		0.52
348	ISO14184-1	158.0		-0.66	2360	ISO14184-1	169.9		0.31
357	ISO14184-1	179.6		1.10	2361	INH-2002	170.9		0.39
361	ISO14184-1	170.2		0.34	2364	ISO14184-1	151.7		-1.17
551	ISO14184-1	142.36		-1.93	2366	ISO14184-1	175.1		0.73
622	ISO14184	199.9		2.75	2367	ISO14184-1	156.7		-0.76
826	ISO14184-1	178.4		1.00	2368	ISO14184-1	156		-0.82
840	ISO14184-1	182.5		1.33	2372	ISO14184-1	187.0		1.70
1132	in house	150.32	C	-1.28	2373	ISO14184-1	172.1		0.49
1213	TCVN 7421-1	191.0		2.03	2375	ISO14184-1	156.73		-0.76
1649	LAW112	155		-0.90	2379	ISO14184-1	216.3	G(0.01)	4.08
2102	ISO14184	157.5		-0.70	2380	ISO14184-1	175		0.73
2127	64LFGB82-02.1	150.0		-1.31	2385	ISO14184-1	158.9		-0.58
2129	ISO14184-1	166.54		0.04	2390	ISO14184-1	164.9		-0.09
2132	ISO14184-1	164.1		-0.16	2403	ISO14184	179.9		1.12
2135	ISO14184-1	207.6		3.37	2404	ISO14184	190.4		1.98
2137	ISO14184-1	137.5		-2.32	2410	ISO14184-1	186.2		1.64
2139	ISO14184-1	173.6		0.61	2411	ISO14184-1	116.4	G(0.01)	-4.04
2146	ISO14184	151.5		-1.18	2413	ISO14184	117.9	G(0.01)	-3.91
2165	ISO14184	166.1		0.00	2415	ISO14184-1	175.4		0.76
2172	ISO14184-1	150.3		-1.28	2426	ISO14184	192.7		2.16
2182	ISO14184-1	168.1		0.17	2428	ISO14184-1	154.0		-0.98
2184	ISO14184-1	164.3		-0.14	2429	ISO14184-1	179.2		1.07
2186	Jap.law 112	81.5	G(0.01)	-6.87	2432	ISO14184-1	159.0		-0.57
2190	ISO14184-1	160.9		-0.42	2433	Jap.Law 112	195.7		2.41
2196	ISO14184	185.1		1.55	2440	ISO14184-1	186.0		1.62
2197	ISO14184-1	191.0		2.03	2442	ISO14184-1	157.29		-0.71
2201	ISO14184-1	170.0		0.32	2452	ISO14184-1	166.5		0.04
2212	-----	-----		-----	2454	ISO14184-1	168.7		0.21
2216	INH-B	173.2		0.58	2456	ISO14184-1	154.6		-0.93
2217	ISO14184-1	177.00		0.89	2462	ISO14184-1	175.1		0.73
2219	ISO14184-1	162.6		-0.28	2467	ISO14184-1	144.5		-1.75
2225	ISO14184	165.1		-0.08	2472	ISO14184-1	173.1		0.57
2226	ISO14184	169.8		0.30	2474	ISO14184-1	177.4		0.92
2228	ISO14184-1	166.74		0.05	2475	ISO14184-1	143.6		-1.83
2229	ISO14184-1	166.3		0.02	2476	ISO14184-1	130.7		-2.87
2232	ISO14184	163.0		-0.25	2482	ISO14184-1	174.4		0.68
2236	ISO14184	166.8		0.06	2488	ISO14184-1	143.86		-1.80
2238	ISO14184	155.6		-0.85	2489	ISO14184-1	166.7		0.05
2241	ISO14184-1	171.4		0.43	2492	Jap.Law 112	182.5		1.33
2245	ISO14184-1	163.0		-0.25	2493	ISO14184-1	161.3		-0.39
2246	ISO14184-1	164.6		-0.12	2494	ISO14184-1	147.8		-1.48
2247	ISO14184-1	160.10		-0.48	2495	ISO14184	152.3		-1.12
2255	ISO14184-1	176.30		0.83	2496	ISO14184-1	155.0		-0.90
2256	ISO14184-1	170.4		0.35	2497	ISO14184	177.9		0.96
2261	GB/T2912.1	169.7		0.30	2500	ISO14184	161.6		-0.36
2264	JIS L112 L1041	190.9		2.02	2501	ISO14184-1	161.6		-0.36
2265	LFGB B82-02.1	148.9		-1.39	2504	ISO14184-1	128.0		-3.09
2269	ISO14184-1	167.103		0.08	2506	ISO14184-1	170.9		0.39
2271	ISO14184-1	174		0.64	2511	ISO14184-1	157.4		-0.70
2273	ISO14184-1	169.3		0.26	2512	ISO14184-1	175.3		0.75
2274	ISO14184-1	161.3		-0.39	2514	ISO14184-1	169.02		0.24
2275	ISO14184-1	165.7		-0.03	2517	ISO14184-1	167.2		0.09
2276	ISO14184	235.4	G(0.01)	5.63	2518	ISO14184-1	164.3		-0.14
2277	ISO14184	162.6		-0.28	2519	ISO14184-1	171.4		0.43
2279	ISO14184-1	167.0		0.08	2520	ISO14184-1	162.3		-0.31
2282	ISO14184-1	186.0		1.62	2522	ISO14184-1	138.4		-2.25
2283	ISO14184-1	173.05		0.57	3100	ISO14184-1	164.4		-0.14
2284	ISO14184-1	166.0		-0.01	3110	ISO14184-1	135.9		-2.45
2289	ISO14184-1	179.8		1.12	3116	ISO14184-1	166.8		0.06
2290	ISO14184-1	178.9		1.04	3117	ISO14184-1	151.2		-1.21
2292	-----	-----		-----	3118	ISO14184	194.3		2.29
2293	JIS L1041B	140.21	C	-2.10	3134	-----	-----		-----
2294	ISO14184	168.3		0.18	3145	Jap.Law 112	166.6		0.04
2295	ISO14184-1	108.7	C,G(0.01)	-4.66	3146	ISO14184-1	162.7		-0.27
2296	ISO14184	159.9		-0.50	3150	LFGB B82-02.1	168.0		0.16
2298	JIS L1041	166.9		0.07	3153	ISO14184-1	161.5		-0.37
2301	-----	-----		-----	3154	ISO14184-1	176.66		0.86
2310	ISO14184	165.2		-0.07	3166	ISO14184-1	156.6		-0.77
2311	ISO14184-1	163.3		-0.22	3167	ISO14184-1	177.3		0.91
2313	ISO14184-1	159.4		-0.54	3169	ISO14184	158.8		-0.59
2314	ISO14184-1	159.6		-0.53	3172	ISO14184-1	163.4		-0.22
2350	ISO14184	171.2		0.42	3176	ISO14184-1	176		0.81
2351	ISO14184-1	158		-0.66	3180	ISO14184	131.8		-2.78

3182	ISO14184-1	184.8	C	1.52
3185	ISO14184-1	166.3		0.02
3190	ISO14184-1	157.8		-0.67
3192	LFG B82-02.1	161.9		-0.34
3195	ISO14184	133.6		-2.64
3197	ISO14184-1	162.3		-0.31
3198		-----		-----
3199	Jap.Law 112	167.7		0.13
3207	JIS L1041	163.3		-0.22
3208		-----		-----
3210	ISO14184-1	149.5		-1.35
3212	ISO14184-1	167.67		0.13
3214	ISO14184-1	161.1		-0.40
3215	ISO14184-1	178.6		1.02
3218	ISO14184-1	159.0		-0.57
3220	ISO14184-1	202.2		2.94
3225	ISO14184-1	167		0.08
3226	ISO14184-1	175.0		0.73
3228	ISO14184-1	161.4		-0.38
3237	ISO14184-1	148.60		-1.42
3243	ISO14184-1	157.4		-0.70
3246	ISO14184-1	180.70		1.19
3248	ISO14184	156		-0.82

normality OK
n 163
outliers 6
mean (n) 166.07
st.dev. (n) 13.742
R(calc.) 38.48
R(Horwitz) 34.47

Lab 1132: first reported 40.92
Lab 2293: first reported 0.578
Lab 2295: first reported 100.83
Lab 3182: first reported 207.8

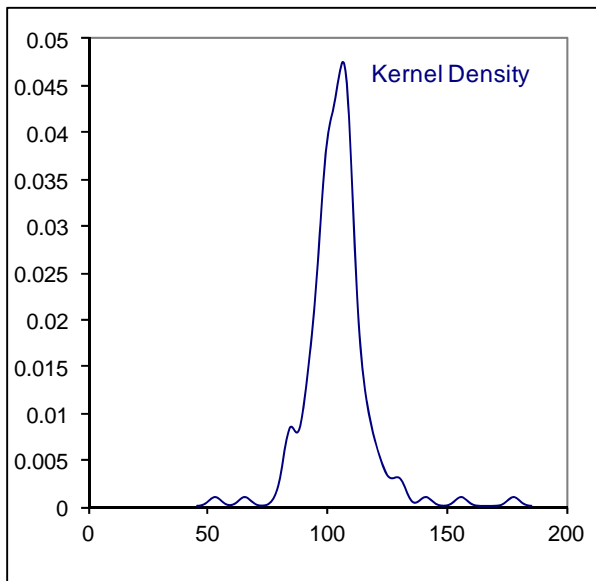
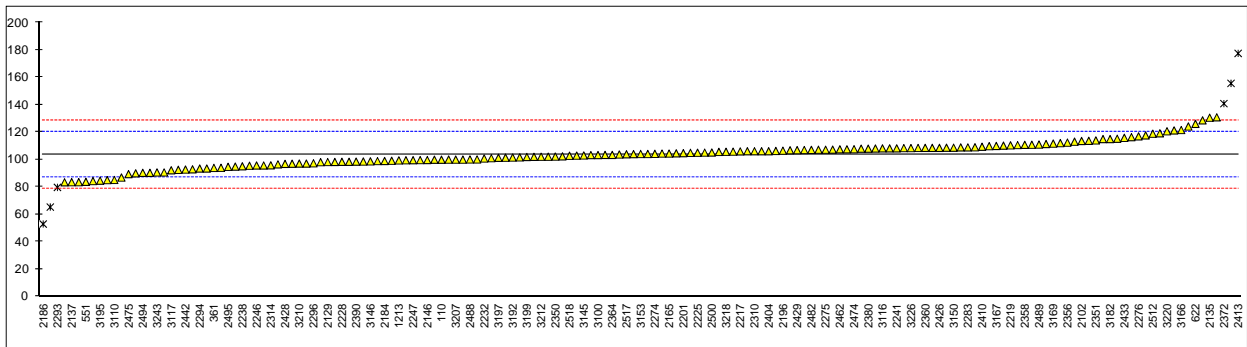


Determination of Free Formaldehyde on sample #12123; results in mg/kg

lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110	JIS L1041B	99.9		-0.46	2356	ISO14184-1	112.4		1.05
213	ISO14184-1	93.5		-1.24	2358	ISO14184	110.8		0.86
339	ISO14184-1	90		-1.66	2359	ISO14184-1	102.9		-0.10
348	ISO14184-1	83.4		-2.46	2360	ISO14184-1	108.6		0.59
357	ISO14184-1	108.2		0.54	2361	INH-2002	109.8		0.74
361	ISO14184-1	93.9		-1.19	2364	ISO14184-1	103.4		-0.04
551	ISO14184-1	83.75		-2.42	2366	ISO14184-1	105.1		0.17
622	ISO14184	126.1		2.71	2367	ISO14184-1	98.3		-0.66
826	ISO14184-1	104.1		0.04	2368	ISO14184-1	102		-0.21
840	ISO14184-1	106.1		0.29	2372	ISO14184-1	140.9	G(0.01)	4.50
1132	in house	90.62	C	-1.59	2373	ISO14184-1	115.0		1.37
1213	TCVN 7421-1	99.4		-0.52	2375	ISO14184-1	99.14		-0.56
1649	LAW112	100		-0.45	2379	ISO14184-1	128.7		3.03
2102	ISO14184	113.6		1.20	2380	ISO14184-1	108		0.52
2127	64LFGB82-02.1	95.7		-0.97	2385	ISO14184-1	104.4		0.08
2129	ISO14184-1	98.2		-0.67	2390	ISO14184-1	98.5		-0.63
2132	ISO14184-1	98.6		-0.62	2403	ISO14184	99.5		-0.51
2135	ISO14184-1	130.6		3.26	2404	ISO14184	106.1		0.29
2137	ISO14184-1	83.5		-2.45	2410	ISO14184-1	109.4		0.69
2139	ISO14184-1	102.4		-0.16	2411	ISO14184-1	65.3	G(0.01)	-4.66
2146	ISO14184	99.7		-0.49	2413	ISO14184	177.6	G(0.01)	8.95
2165	ISO14184	104.4		0.08	2415	ISO14184-1	110.6		0.83
2172	ISO14184-1	108.7		0.60	2426	ISO14184	108.6		0.59
2182	ISO14184-1	101.6		-0.26	2428	ISO14184-1	96.9		-0.83
2184	ISO14184-1	99.0		-0.57	2429	ISO14184-1	106.9		0.38
2186	Jap.law 112	52.9	G(0.01)	-6.16	2432	ISO14184-1	94.0		-1.18
2190	ISO14184-1	108.6		0.59	2433	Jap.Law 112	115.9		1.47
2196	ISO14184	106.5		0.34	2440	ISO14184-1	104.0		0.03
2197	ISO14184-1	116.4		1.53	2442	ISO14184-1	92.71		-1.34
2201	ISO14184-1	104.7		0.12	2452	ISO14184-1	107.3		0.43
2212		----		----	2454	ISO14184-1	104.9		0.14
2216	INH-B	106.1		0.29	2456	ISO14184-1	92.5		-1.36
2217	ISO14184-1	106.00		0.27	2462	ISO14184-1	107.5		0.46
2219	ISO14184-1	110.4		0.81	2467	ISO14184-1	90.3		-1.63
2225	ISO14184	105.0		0.15	2472	ISO14184-1	101.0		-0.33
2226	ISO14184	110.2		0.78	2474	ISO14184-1	107.6		0.47
2228	ISO14184-1	98.37		-0.65	2475	ISO14184-1	89.4		-1.74
2229	ISO14184-1	108.9		0.63	2476	ISO14184-1	86.9		-2.04
2232	ISO14184	100.8		-0.36	2482	ISO14184-1	107.1		0.41
2236	ISO14184	103.3		-0.05	2488	ISO14184-1	100.09		-0.44
2238	ISO14184	95.1		-1.05	2489	ISO14184-1	111.0		0.88
2241	ISO14184-1	108.2		0.54	2492	Jap.Law 112	105.6		0.23
2245	ISO14184-1	113.7		1.21	2493	ISO14184-1	92.9		-1.31
2246	ISO14184-1	95.7		-0.97	2494	ISO14184-1	90.3		-1.63
2247	ISO14184-1	99.58		-0.50	2495	ISO14184	94.7		-1.09
2255	ISO14184-1	115.26		1.40	2496	ISO14184-1	98.9		-0.59
2256	ISO14184-1	108.5		0.58	2497	ISO14184	113.1		1.14
2261	GB/T2912.1	124.2		2.48	2500	ISO14184	105.2		0.18
2264	JIS L112 L1041	111.4		0.93	2501	ISO14184-1	103.7		0.00
2265	LFGB 82-02.1	94.9		-1.07	2504	ISO14184-1	83.5		-2.45
2269	ISO14184-1	106.300		0.31	2506	ISO14184-1	130.9		3.29
2271	ISO14184-1	108		0.52	2511	ISO14184-1	98.4		-0.65
2273	ISO14184-1	107.0		0.40	2512	ISO14184-1	118.9		1.84
2274	ISO14184-1	104.2		0.06	2514	ISO14184-1	117.67		1.69
2275	ISO14184-1	107.2		0.42	2517	ISO14184-1	103.9		0.02
2276	ISO14184	117.0		1.61	2518	ISO14184-1	102.8		-0.11
2277	ISO14184	103.4		-0.04	2519	ISO14184-1	108.4		0.57
2279	ISO14184-1	107.5		0.46	2520	ISO14184-1	95.51		-1.00
2282	ISO14184-1	106.8		0.37	2522	ISO14184-1	84.3		-2.35
2283	ISO14184-1	108.94		0.63	3100	ISO14184-1	103.3		-0.05
2284	ISO14184-1	100.0		-0.45	3110	ISO14184-1	85.1		-2.26
2289	ISO14184-1	105.8		0.25	3116	ISO14184-1	108.2		0.54
2290	ISO14184-1	108.1		0.53	3117	ISO14184-1	92.2		-1.40
2292		----		----	3118	ISO14184	119.2		1.87
2293	JIS L1041B	79.68	C,G(0.01)	-2.91	3134		----		----
2294	ISO14184	93.5		-1.24	3145	Jap.Law 112	103.0		-0.09
2295	ISO14184-1	155.7	C,G(0.01)	6.30	3146	ISO14184-1	98.7		-0.61
2296	ISO14184	97.3		-0.78	3150	LFGB82-02.1	108.7		0.60
2298	JIS L1041	102.1		-0.20	3153	ISO14184-1	104.1		0.04
2301		----		----	3154	ISO14184-1	112.04		1.01
2310	ISO14184	106.1		0.29	3166	ISO14184-1	121.6		2.17
2311	ISO14184-1	107.2		0.42	3167	ISO14184-1	110.0		0.76
2313	ISO14184-1	99.9		-0.46	3169	ISO14184	111.7		0.97
2314	ISO14184-1	95.8		-0.96	3172	ISO14184-1	96.5		-0.88
2350	ISO14184	102.2		-0.19	3176	ISO14184-1	121.3		2.13
2351	ISO14184-1	114		1.24	3180	ISO14184	85.0		-2.27

3182	ISO14184-1	115	C	1.37
3185	ISO14184-1	104.6		0.11
3190	ISO14184-1	101.4		-0.28
3192	LFG B82-02.1	101.5		-0.27
3195	ISO14184	84.6		-2.32
3197	ISO14184-1	101.3		-0.29
3198		-----		-----
3199	Jap.Law 112	101.9		-0.22
3207	JIS L1041	100		-0.45
3208		-----		-----
3210	ISO14184-1	97.1		-0.80
3212	ISO14184-1	102.07		-0.20
3214	ISO14184-1	98.1		-0.68
3215	ISO14184-1	99.6		-0.50
3218	ISO14184-1	105.7		0.24
3220	ISO14184-1	120.7		2.06
3225	ISO14184-1	109		0.64
3226	ISO14184-1	108.4		0.57
3228	ISO14184-1	100.1		-0.44
3237	ISO14184-1	97.10		-0.80
3243	ISO14184-1	90.6		-1.59
3246	ISO14184-1	110.85		0.86
3248	ISO14184	97		-0.82
normality		not OK		
n		163		
outliers		6		
mean (n)		103.73		
st.dev. (n)		9.206		
R(calc.)		25.78		
R(Horwitz)		23.11		

Lab 1132: first reported 24.13
 Lab 2293: first reported 0.328
 Lab 2295: first reported 146.64
 Lab 3182: first reported 127.4



APPENDIX 2

Number of participants per country

1 lab in AUSTRIA
5 labs in BANGLADESH
1 lab in BELGIUM
1 lab in BRAZIL
1 lab in BULGARIA
1 lab in CZECH REPUBLIC
2 labs in EGYPT
3 labs in FINLAND
4 labs in FRANCE
15 labs in GERMANY
1 lab in GREECE
1 lab in GUATEMALA
15 labs in HONG KONG
2 labs in HUNGARY
7 labs in INDIA
4 labs in INDONESIA
6 labs in ITALY
5 labs in KOREA
1 lab in MAURITIUS
4 labs in MEXICO
1 lab in MOROCCO
55 labs in P.R. of CHINA
2 labs in PAKISTAN
1 lab in PERU
1 lab in PHILIPPINES
1 lab in ROMANIA
1 lab in SINGAPORE
1 lab in SLOVENIA
1 lab in SPAIN
1 lab in SWITZERLAND
2 labs in TAIWAN R.O.C.
5 labs in THAILAND
1 lab in THE NETHERLANDS
2 labs in TUNISIA
6 labs in TURKEY
7 labs in U.S.A.
1 lab in UNITED KINGDOM
5 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
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
n.a.	= not applicable
n.d.	= not detected
W	= withdrawn

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