

**Results of Proficiency Test  
Free and Released  
Formaldehyde in textile  
October 2013**

**Organised by:** Institute for Interlaboratory Studies  
Spijkenisse, the Netherlands

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**Report:** iis13A04T

December 2013

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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 2013/2014. In the 2013/2014 program it was decided to extend the Free Formaldehyde proficiency test with a scheme for Released Formaldehyde.

In this interlaboratory study 178 laboratories in 38 different countries participated. See appendix 2 for the number of participating laboratories per country.

In this report, the results of the 2013 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 (#13190 and #13191, each approx. 3 grams) which were treated to find two different concentration levels of Free Formaldehyde. Both samples are also suitable to determine Released 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 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](http://www.iisnl.com).

## 2.3 CONFIDENTIALITY STATEMENT

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

## 2.4 SAMPLES

The two different hosiery fabric textile samples (labelled sample #13190 and #13191) were divided over 180 subsamples of approx. 3 grams.

The homogeneity of both samples was checked on respective 8 and 15 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 #13190-1	245	Sample #13191-1	46
Sample #13190-2	299	Sample #13191-2	49
Sample #13190-3	288	Sample #13191-3	48
Sample #13190-4	282	Sample #13191-4	50
Sample #13190-5	267	Sample #13191-5	48
Sample #13190-6	282	Sample #13191-6	44
Sample #13190-7	297	Sample #13191-7	46
Sample #13190-8	292	Sample #13191-8	45
	--	Sample #13191-9	49
	--	Sample #13191-10	45
	--	Sample #13191-11	48
	--	Sample #13191-12	47
	--	Sample #13191-13	45
	--	Sample #13191-14	46
	--	Sample #13191-15	47

Table 1: homogeneity test results of subsamples #13190 and #13191

From the above test results, the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility 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 #13190	Free Formaldehyde in mg/kg Sample#13191
r	5	1
Reference test method	Horwitz	Horwitz
0.3*R <sub>(reference test method)</sub>	16	4

Table 2: repeatabilities of subsamples #13190 and #13191

From the above results of the homogeneity tests, the repeatabilities were calculated. The calculated repeatabilities for samples #13190 and #13191 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, #13190 and #13191 were sent to the participating laboratories on October 9, 2013.

## 2.5 ANALYSES

The participants were asked to determine on samples #13190 and #13191 the Free Formaldehyde content and the Released Formaldehyde content with the analytical procedures that are 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 content and Released Formaldehyde, 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 3.

### 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. Five laboratories did not report any test results and twenty-one other laboratories reported results after the final reporting date.

Finally, the 173 reporting laboratories send in total 378 numerical results. Observed were 13 statistical outlying results, which is 3.4% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

For Free Formaldehyde on sample #13190, a not normal distribution was found. Therefore the statistical evaluation should be used with due care for this determination.

In ISO14184-1:11 some information on precision data is given. In table B.1 "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 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 samples #13190 and #13191 are discussed. All statistical results reported on the textile samples are summarised in appendix 1.

##### **Sample #13190:**

**Free Formaldehyde content:** This determination was 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 problematic. Only one statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the estimated reproducibility calculated using the Horwitz equation.

#### **Sample #13191:**

**Free Formaldehyde content:** This determination was problematic. Five 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 problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers 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 #13190	mg/kg	169	308	84	58
Released Formaldehyde #13190	mg/kg	11	823	211	134
Free Formaldehyde #13191	mg/kg	167	83.3	29.1	19.2
Released Formaldehyde #13191	mg/kg	15	198	62	40

Table 3: reproducibilities of textile samples #13190 and #13191

From the above tables it can be concluded that, without statistical calculations, the group of participating laboratories does have 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 2013	October 2012	October 2011	October 2010	October 2009	October 2008
Free Formaldehyde	27-35%	23-25%	31-41%	21-24%	24-33%	19-42%
Released Formalde.	26-27%	n.e.	n.e.	n.e.	n.e.	n.e.

Table 4: Development of relative reproducibilities 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 was 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 extractable (mg/kg)	<16	300	75	300
Released Formaldehyde (mg/m <sup>3</sup> )	0.1	0.1	0.1	0.1

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.

### Extractable free formaldehyde:

For sample #13190, Sixty-one laboratories would accept the sample for the categories "Öko-Tex 103 no direct skin contact" and "Decoration material" (<300 mg/kg). The other laboratories would reject this sample for all categories.

For sample #13191, Forty-one laboratories would accept the sample for all categories except for Class 1 (Baby, <16mg/kg). The other laboratories would accept the sample for the categories "Öko-Tex 103 no direct skin contact" and "Decoration material" (<300 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/m<sup>3</sup> vs mg/kg).

### **General**

The Free Formaldehyde content and the Released Formaldehyde were determined at two different levels. The spreads observed for the Free Formaldehyde content 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 content on sample #13190; results in mg/kg

lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110	ISO14184-1	381.11		3.51	2379	ISO14184-1	295.21		-0.62
213	ISO14184-1	317.2		0.44	2380	ISO14184-1	268.00		-1.92
230	ISO14184-1	309.03		0.05	2381	ISO14184-1	265.60		-2.04
357	ISO14184-1	337.2		1.40	2385	ISO14184-1	229.5		-3.77
551	ISO14184-1	338.36	C	1.46	2390	ISO14184-1	243.7		-3.09
2102	INH-443	240.9		-3.23	2410	ISO14184-1	278.0		-1.44
2115	ISO14184-1	287		-1.01	2413	ISO14184-1	837.91	ex	25.47
2120	ISO14184-1	333.6		1.23	2420	ISO14184-1	287.2		-1.00
2121	ISO14184-1	312.1		0.20	2425	ISO14184-1	309.72		0.08
2127	LFGB B82.02.1	319		0.53	2426	ISO14184-1	320.45		0.60
2129	ISO14184-1	256.5		-2.48	2428	ISO14184-1	303.90		-0.20
2132	ISO14184-1	288.17		-0.95	2429	ISO14184-1	323.06		0.72
2135	ISO14184-1	298.9		-0.44	2432	ISO14184-1	283.10		-1.20
2138	ISO14184-1	293.48		-0.70	2433	ISO14184-1	321.20		0.63
2139	ISO14184-1	288.83		-0.92	2437	ISO14184-1	310		0.09
2146	ISO14184-1	335.5		1.32	2442	ISO14184-1	280.17		-1.34
2165	ISO14184-1	322.44		0.69	2452	ISO14184-1	320.20		0.59
2170	ISO14184-1	309.5		0.07	2453	ISO14184-1	329		1.01
2172	ISO14184-1	329.48		1.03	2454	ISO14184-1	314.59		0.32
2184	ISO14184-1	328.80		1.00	2456	ISO14184-1	312	C	0.19
2186	ISO14184-1	308.40		0.02	2459	ISO14184-1	348.9		1.96
2190	ISO14184-1	322		0.67	2460	ISO14184-1	293.65		-0.69
2196	ISO14184-1	309.7		0.08	2467		-----		-----
2197	ISO14184-1	373.5		3.15	2468	ISO14184-1	311.5		0.17
2201	ISO14184-1	335.1		1.30	2472	ISO14184-1	328.6		0.99
2212	JIS L1041-B	332.2		1.16	2474	ISO14184-1	274.50		-1.61
2213		-----		-----	2475	ISO14184-1	291.7		-0.78
2216	Jap.Law 112	340.5		1.56	2476	ISO14184-1	294.7		-0.64
2219	ISO14184-1	374.23		3.18	2481	ISO17226-1	342.72		1.67
2225	ISO14184-1	304.71		-0.16	2482	ISO14184-1	306.85		-0.06
2226	ISO14184	352.4	C	2.13	2483	ISO14184-1	334.72		1.28
2228	ISO14184-1	342.2239		1.64	2489	ISO14184-1	283.7		-1.17
2229	ISO14184-1	312.19		0.20	2492	ISO14184-1	345.2		1.79
2230	ISO14184-1	371.45		3.05	2493	ISO14184-1	401		4.47
2232	ISO14184-1	311.58		0.17	2495	ISO14184-1	258.9		-2.36
2235	ISO14184-1	330.95		1.10	2501	ISO14184-1	317.6		0.46
2236	Jap.Law 112	358.95		2.45	2506	ISO14184-1	290.00	C	-0.87
2238	ISO14184-1	305.7		-0.11	2511	ISO14184-1	281.8		-1.26
2241	ISO14184-1	305.69		-0.11	2514	ISO14184-1	311.26		0.16
2245	ISO14184-1	329.43		1.03	2515		-----		-----
2246	ISO14184-1	283.32		-1.19	2517	ISO14184-1	319.4		0.55
2247	ISO14184-1	304.30		-0.18	2518	ISO14184-1	325.484		0.84
2255	ISO14184-1	310.0		0.09	2519	ISO14184-1	323.5		0.74
2256	ISO14184-1	330.1		1.06	2525	ISO14184-1	299.34		-0.42
2261	GB/T2912.1	278.778		-1.41	2527	ISO14184-1	335.3	C	1.31
2265	ISO14184-1	269.04		-1.87	2532	ISO14184-1	323.9		0.76
2266	ISO14184-1	265.95		-2.02	2534	ISO14184-1	347.8		1.91
2269	ISO14184-1	312.772		0.23	2535	ISO14184-1	315.95		0.38
2273	ISO14184-1	304.50		-0.17	2542	GB/T2912.1	330.11508		1.06
2275	ISO14184-1	317.68		0.46	2546	ISO14184-1	306.815		-0.06
2277	ISO14184-1	305.22166		-0.13	2549	ISO14184-1	291.9		-0.78
2284	ISO14184-1	327.2		0.92	2553	JIS L1041-B	423.57	G(0.05)	5.55
2289	ISO14184-1	309.8		0.09	2555	ISO14184-1	282		-1.25
2290	ISO14184-1	310.92		0.14	2559	ISO14184	261.85		-2.22
2293	JIS L1041	319.8		0.57	2561	ISO14184-1	146	G(0.01)	-7.79
2294	ISO14184-1	319.43		0.55	2562	ISO14184-1	312.04		0.19
2295	ISO14184-1	336	C	1.34	2563		-----		-----
2296	ISO14184-1	277.79		-1.45	2566	ISO14184-1	317.44		0.45
2298	JIS L1041	311.7		0.18	2567	ISO14184-1	318.7		0.51
2303	ISO14184-1	333.2		1.21	2573	ISO14184-1	315.33		0.35
2310	ISO14184-1	270		-1.83	2576	ISO14184-1	353.33		2.18
2311	ISO14184-1	267.8		-1.93	2577	GB/T2912.1	257.8		-2.41
2313	ISO14184-1	274.4		-1.62	2580	ISO14184-1	295		-0.63
2314	ISO14184-1	265.67		-2.04	2582	ISO14184-1	290.001		-0.87
2351	ISO14184-1	298.77		-0.44	2584	ISO14184-1	311.01		0.14
2356	ISO14184-1	273.15		-1.68	2587	ISO14184-1	294.22		-0.66
2359	ISO14184-1	310.19		0.10	2589	ISO14184-1	312.50		0.21
2360	ISO14184-1	319.68		0.56	2590	ISO14184-1	248.1		-2.88
2361	INH-2012-3	260.55		-2.28	2591	ISO14184-1	285.9		-1.06
2363	ISO14184-1	302.75		-0.25	2598		-----		-----
2364	ISO14184-1	320		0.58	3100	ISO14184-1	304.67		-0.16
2366	ISO14184-1	303.4		-0.22	3110	ISO14184-1	259.88		-2.31
2367	ISO14184-1	304.50		-0.17	3116	ISO14184-1	336.34		1.36
2368	ISO14184-1	278.42		-1.42	3118	ISO14184-1	320.97		0.62
2372	ISO14184-1	401.78		4.51	3146	ISO14184-1	323.76		0.76
2375	ISO14184-1	260.5		-2.28	3150	ISO14184-1	302		-0.29

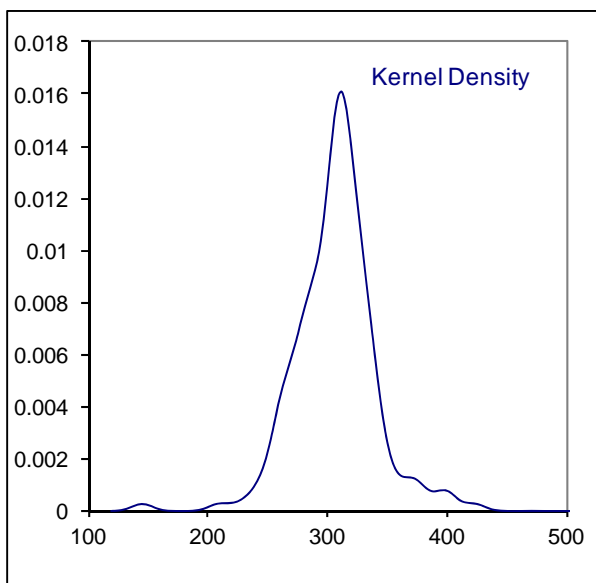
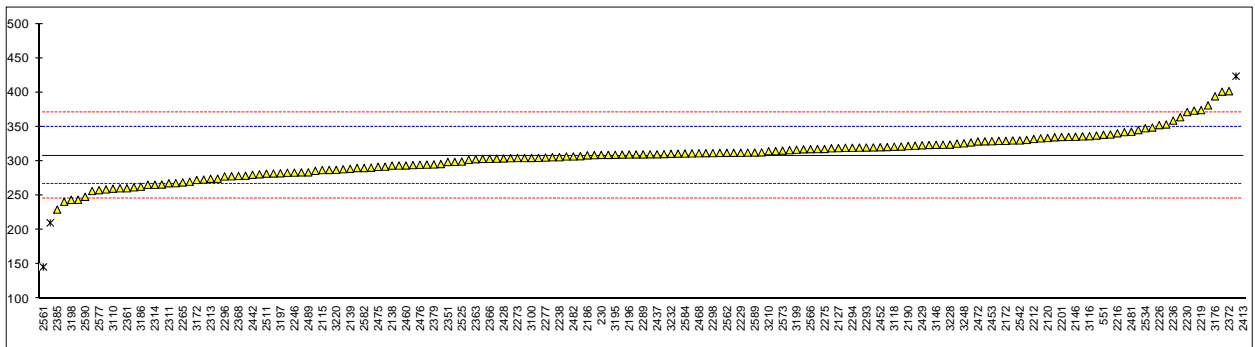
3153	ISO14184-1	293.60	-0.69
3154	ISO14184-1	290.45	-0.84
3167	ISO14184-1	303.3	-0.23
3172	ISO14184-1	272.58	-1.70
3176	ISO14184-1	394.3	4.15
3180	ISO14184-1	210.26	-4.70
3182	ISO14184-1	312.4	0.21
3185	ISO14184-1	280.9	-1.30
3186	ISO14184-1	262.80	-2.17
3190	ISO14184-1	308.74	0.03
3192	LFG B82.02.1	338.8	1.48
3195	ISO14184-1	309.3	0.06
3197	ISO14184-1	282.3	-1.24
3198	ISO14184-1	243.68	-3.09
3199	ISO14184-1	316.61	0.41
3207	JIS L1041	303.6	-0.21
3210	ISO14184-1	314.4	0.31
3214	ISO14184-1	295.8	-0.59
3220	ISO14184-1	287.23	-1.00
3222	ISO14184-1	307.25	-0.04
3226	ISO14184-1	363.88	2.68
3228	ISO14184-1	324	0.77
3232	ISO14184-1	310.65	0.13
3237	ISO14184-1	283.67	-1.17
3242	ISO14184-1	309.10	0.05
3248	ISO14184	326	0.86

G(0.05)

normality	not OK
n	169
outliers	3 +1 excl.
mean (n)	308.03
st.dev. (n)	30.029
R(calc.)	84.08
R(Horwitz)	58.25

Lab 551 first reported 676.73  
 Lab 2226 first reported 393.57  
 Lab 2295 first reported 418  
 Lab 2456 first reported 412  
 Lab 2506 first reported 427.97  
 Lab 2527 first reported 85.27

Lab 2413 result excluded as free formaldehyde is same as released formaldehyde result



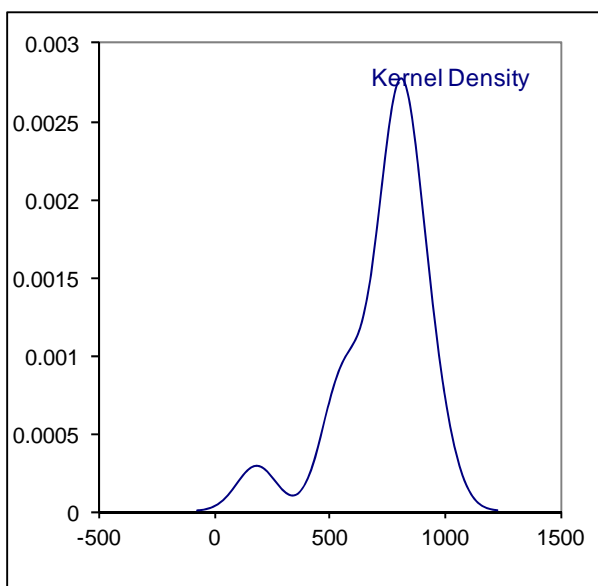
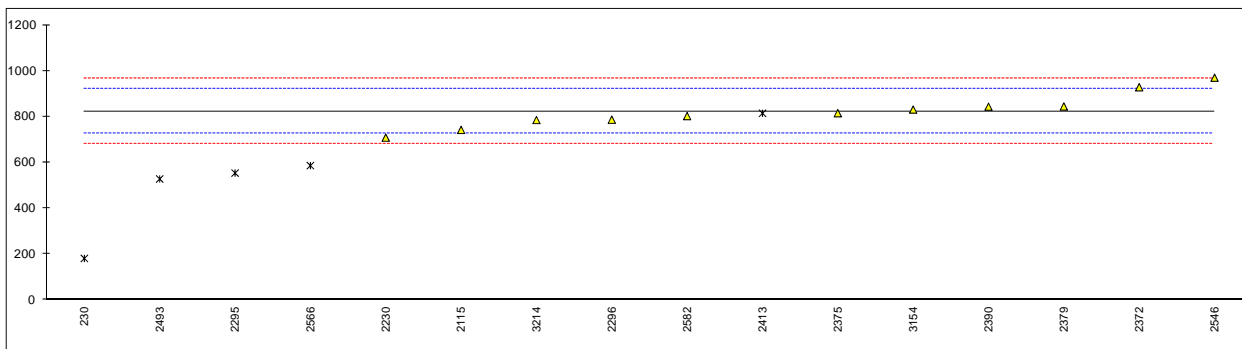
## Determination of Released Formaldehyde content on sample #13190; results in mg/kg

lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110		----		----	2379	ISO14184-2	844.58		0.44
213		----		----	2380		----		----
230	ISO14184-2	181.16	G(0.01)	-13.39	2381		----		----
357		----		----	2385		----		----
551		----		----	2390	ISO14184-2	843.7		0.42
2102		----		----	2410		----		----
2115	ISO14184-2	742		-1.70	2413	ISO14184-1	814.62	ex	-0.18
2120		----		----	2420		----		----
2121		----		----	2425		----		----
2127		----		----	2426		----		----
2129		----		----	2428		----		----
2132		----		----	2429		----		----
2135		----		----	2432		----		----
2138		----		----	2433		----		----
2139		----		----	2437		----		----
2146		----		----	2442		----		----
2165		----		----	2452		----		----
2170		----		----	2453		----		----
2172		----		----	2454		----		----
2184		----		----	2456		----		----
2186		----		----	2459		----		----
2190		----		----	2460		----		----
2196		----		----	2467		----		----
2197		----		----	2468		----		----
2201		----		----	2472		----		----
2212		----		----	2474		----		----
2213		----		----	2475		----		----
2216		----		----	2476		----		----
2219		----		----	2481		----		----
2225		----		----	2482		----		----
2226		----		----	2483		----		----
2228		----		----	2489		----		----
2229		----		----	2492		----		----
2230	ISO14184-2	708.49		-2.40	2493	ISO14184-2	528	G(0.05)	-6.16
2232		----		----	2495		----		----
2235		----		----	2501		----		----
2236		----		----	2506		----		----
2238		----		----	2511		----		----
2241		----		----	2514		----		----
2245		----		----	2515		----		----
2246		----		----	2517		----		----
2247		----		----	2518		----		----
2255		----		----	2519		----		----
2256		----		----	2525		----		----
2261		----		----	2527		----		----
2265		----		----	2532		----		----
2266		----		----	2534		----		----
2269		----		----	2535		----		----
2273		----		----	2542		----		----
2275		----		----	2546	ISO14184-2	970.03		3.06
2277		----		----	2549		----		----
2284		----		----	2553		----		----
2289		----		----	2555		----		----
2290		----		----	2559		----		----
2293		----		----	2561		----		----
2294		----		----	2562		----		----
2295	ISO14184-2	554	G(0.05)	-5.62	2563		----		----
2296	ISO14184-2	786.79		-0.76	2566	ISO14184-2	586.21	G(0.05)	-4.95
2298		----		----	2567		----		----
2303		----		----	2573		----		----
2310		----		----	2576		----		----
2311		----		----	2577		----		----
2313		----		----	2580		----		----
2314		----		----	2582	ISO14184-2	802.60		-0.43
2351		----		----	2584		----		----
2356		----		----	2587		----		----
2359		----		----	2589		----		----
2360		----		----	2590		----		----
2361		----		----	2591		----		----
2363		----		----	2598		----		----
2364		----		----	3100		----		----
2366		----		----	3110		----		----
2367		----		----	3116		----		----
2368		----		----	3118		----		----
2372	ISO14184-2	928.40		2.19	3146		----		----
2375	ISO14184-2	815.2		-0.17	3150		----		----
3153		----		----					

3154	ISO14184-2	831.01	0.16
3167		-----	-----
3172		-----	-----
3176		-----	-----
3180		-----	-----
3182		-----	-----
3185		-----	-----
3186		-----	-----
3190		-----	-----
3192		-----	-----
3195		-----	-----
3197		-----	-----
3198		-----	-----
3199		-----	-----
3207		-----	-----
3210		-----	-----
3214	ISO14184-2	785.3	-0.80
3220		-----	-----
3222		-----	-----
3226		-----	-----
3228		-----	-----
3232		-----	-----
3237		-----	-----
3242		-----	-----
3248		-----	-----

normality OK  
n 11  
outliers 4 +1 excl  
mean (n) 823.46  
st.dev. (n) 75.210  
R(calc.) 210.59  
R(Horwitz) 134.30

Lab 2413 result excluded as free formaldehyde is same as released formaldehyde result



## Determination of Free Formaldehyde content on sample #13191; results in mg/kg

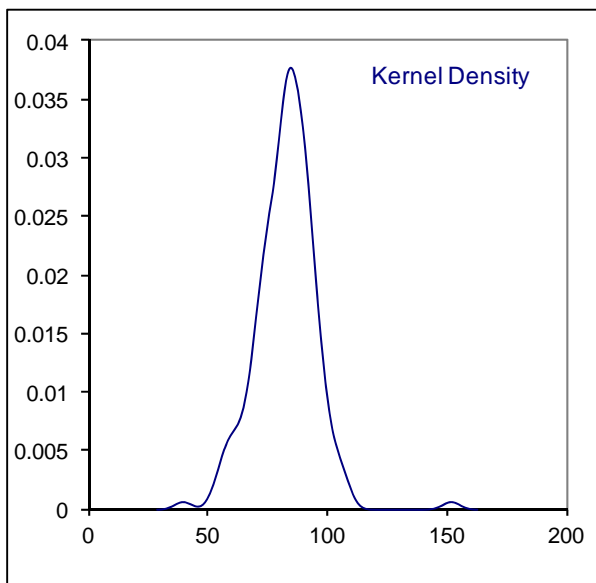
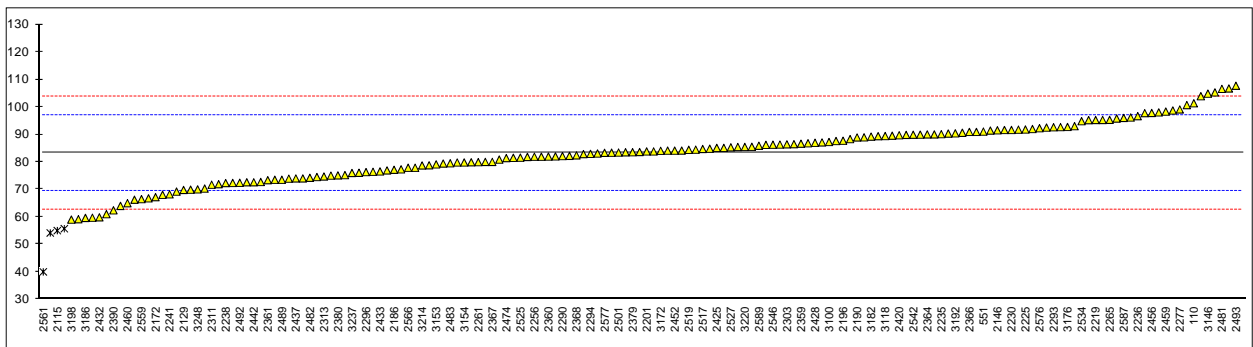
lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110	ISO14184-1	101.32		2.63	2379	ISO14184-1	83.53		0.04
213	ISO14184-1	84.1		0.12	2380	ISO14184-1	75.10		-1.20
230	ISO14184-1	68.04	C	-2.23	2381	ISO14184-1	72.60		-1.56
357	ISO14184-1	90.9		1.11	2385	ISO14184-1	69.9		-1.95
551	ISO14184-1	91.02	C	1.13	2390	ISO14184-1	62.4		-3.05
2102	INH-443	70.3		-1.90	2410	ISO14184-1	83.5		0.03
2115	ISO14184-1	55	G(0.01)	-4.13	2413	ISO14184-1	151.77	ex	10.00
2120	ISO14184-1	54.15	G(0.01)	-4.25	2420	ISO14184-1	89.7	C	0.94
2121		----		----	2425	ISO14184-1	85.10		0.27
2127	LFGB B82.02.1.	80		-0.48	2426	ISO14184-1	80.85		-0.36
2129	ISO14184-1	69.8		-1.97	2428	ISO14184-1	86.95		0.54
2132	ISO14184-1	83.60		0.05	2429	ISO14184-1	84.46		0.17
2135	ISO14184-1	77.3		-0.87	2432	ISO14184-1	59.84		-3.42
2138	ISO14184-1	86.79		0.51	2433	ISO14184-1	76.54		-0.98
2139	ISO14184-1	77.88		-0.79	2437	ISO14184-1	74		-1.36
2146	ISO14184-1	91.5		1.20	2442	ISO14184-1	72.60		-1.56
2165	ISO14184-1	91.73		1.23	2452	ISO14184-1	84.1	C	0.12
2170	ISO14184-1	83.8		0.08	2453	ISO14184-1	93		1.42
2172	ISO14184-1	67.18		-2.35	2454	ISO14184-1	83.34		0.01
2184	ISO14184-1	92.48		1.34	2456	ISO14184-1	97.8		2.12
2186	ISO14184-1	77.14		-0.90	2459	ISO14184-1	98.3		2.19
2190	ISO14184-1	88.9		0.82	2460	ISO14184-1	64.99		-2.67
2196	ISO14184-1	87.7		0.64	2467		----		----
2197	ISO14184-1	103.9		3.01	2468	ISO14184-1	74.0		-1.36
2201	ISO14184-1	83.8		0.08	2472	ISO14184-1	87.1		0.56
2212	JIS L1041-B	91.6		1.21	2474	ISO14184-1	81.38		-0.28
2213		----		----	2475	ISO14184-1	86.2		0.43
2216	Jap.Law 112	90.6		1.07	2476	ISO14184-1	87.6		0.63
2219	ISO14184-1	95.25		1.75	2481	ISO17226-1	106.56		3.40
2225	ISO14184-1	91.78		1.24	2482	ISO14184-1	74.223		-1.32
2226	ISO14184	106.7		3.42	2483	ISO14184-1	79.5		-0.55
2228	ISO14184-1	89.5403		0.91	2489	ISO14184-1	73.5		-1.43
2229	ISO14184-1	89.37		0.89	2492	ISO14184-1	72.4		-1.59
2230	ISO14184-1	91.61		1.22	2493	ISO14184-1	107.7		3.56
2232	ISO14184-1	78.77		-0.66	2495	ISO14184-1	76.9		-0.93
2235	ISO14184-1	90.08		0.99	2501	ISO14184-1	83.4		0.02
2236	Jap.Law 112	96.65		1.95	2506	ISO14184-1	59.70	C	-3.44
2238	ISO14184-1	72.3		-1.60	2511	ISO14184-1	75.2		-1.18
2241	ISO14184-1	68.21		-2.20	2514	ISO14184-1	90.31		1.03
2245	ISO14184-1	96.15		1.88	2515		----		----
2246	ISO14184-1	83.06		-0.03	2517	ISO14184-1	84.7		0.21
2247	ISO14184-1	69.20		-2.06	2518	ISO14184-1	81.4835		-0.26
2255	ISO14184-1	82.0		-0.19	2519	ISO14184-1	84.4		0.16
2256	ISO14184-1	81.9		-0.20	2525	ISO14184-1	81.55		-0.25
2261	GB/T2912.1	79.9		-0.49	2527	ISO14184-1	85.3	C	0.29
2265	ISO14184-1	95.30		1.75	2532	ISO14184-1	55.7	G(0.05)	-4.03
2266	ISO14184-1	76.05		-1.06	2534	ISO14184-1	94.8		1.68
2269	ISO14184-1	95.780		1.82	2535	ISO14184-1	95.26		1.75
2273	ISO14184-1	73.50		-1.43	2542	GB/T2912.1	89.88054		0.96
2275	ISO14184-1	82.84		-0.06	2546	ISO14184-1	86.26		0.43
2277	ISO14184-1	99.089		2.31	2549	ISO14184-1	84.8		0.22
2284	ISO14184-1	88.3		0.73	2553	JIS L1041-B	105.28		3.21
2289	ISO14184-1	85.5		0.32	2555	ISO14184-1	61		-3.25
2290	ISO14184-1	82.11		-0.17	2559	ISO14184	66.45		-2.46
2293	JIS L1041	92.6		1.36	2561	ISO14184-1	40	G(0.05)	-6.32
2294	ISO14184-1	82.92		-0.05	2562	ISO14184-1	89.82		0.95
2295	ISO14184-1	98		2.15	2563		----		----
2296	ISO14184-1	76.29		-1.02	2566	ISO14184-1	77.87		-0.79
2298	JIS L1041	91.4		1.18	2567	ISO14184-1	98.7		2.25
2303	ISO14184-1	86.4		0.45	2573	ISO14184-1	89.91		0.97
2310	ISO14184-1	73.9		-1.37	2576	ISO14184-1	92.26		1.31
2311	ISO14184-1	71.7		-1.69	2577	GB/T2912.1	83.3		0.00
2313	ISO14184-1	74.7		-1.25	2580	ISO14184-1	81.81		-0.22
2314	ISO14184-1	71.90		-1.66	2582	ISO14184-1	75.003		-1.21
2351	ISO14184-1	88.93		0.82	2584	ISO14184-1	79.78		-0.51
2356	ISO14184-1	95.20		1.74	2587	ISO14184-1	96.00		1.86
2359	ISO14184-1	86.62		0.49	2589	ISO14184-1	85.88		0.38
2360	ISO14184-1	81.94		-0.20	2590	ISO14184-1	66.74		-2.42
2361	INH-2012-3	73.35		-1.45	2591	ISO14184-1	74.5		-1.28
2363	ISO14184-1	91.95		1.27	2598		----		----
2364	ISO14184-1	90		0.98	3100	ISO14184-1	87.23		0.58
2366	ISO14184-1	90.9		1.11	3110	ISO14184-1	79.87		-0.50
2367	ISO14184-1	80.00		-0.48	3116	ISO14184-1	76.40		-1.01
2368	ISO14184-1	82.37		-0.13	3118	ISO14184-1	89.40		0.89
2372	ISO14184-1	97.75		2.11	3146	ISO14184-1	104.83		3.15
2375	ISO14184-1	72.7		-1.55	3150	ISO14184-1	64.0		-2.82

3153	ISO14184-1	79.09	-0.61
3154	ISO14184-1	79.80	-0.51
3167	ISO14184-1	92.67	1.37
3172	ISO14184-1	84.03	0.11
3176	ISO14184-1	92.7	1.37
3180	ISO14184-1	59.16	-3.52
3182	ISO14184-1	89.2	0.86
3185	ISO14184-1	79.4	-0.57
3186	ISO14184-1	59.63	-3.45
3190	ISO14184-1	86.45	0.46
3192	LFG B82.02.1	90.4	1.04
3195	ISO14184-1	85.1	0.27
3197	ISO14184-1	82.2	-0.16
3198	ISO14184-1	59.02	-3.54
3199	ISO14184-1	72.34	-1.60
3207	JIS L1041	86.3	0.44
3210	ISO14184-1	81.9	-0.20
3214	ISO14184-1	78.7	-0.67
3220	ISO14184-1	85.44	0.31
3222	ISO14184-1	66.25	-2.49
3226	ISO14184-1	100.67	2.54
3228	ISO14184-1	90	0.98
3232	ISO14184-1	84.13	0.12
3237	ISO14184-1	75.97	-1.07
3242	ISO14184-1	85.42	0.31
3248	ISO14184	70	-1.94

normality OK  
 n 167  
 outliers 4 +1 excl.  
 mean (n) 83.28  
 st.dev. (n) 10.409  
 R(calc.) 29.14  
 R(Horwitz) 19.18

Lab 230 first reported 279.91  
 Lab 551 first reported 182.05  
 Lab 2420 first reported 31.6  
 Lab 2452 first reported 41.73  
 Lab 2506 first reported 126.33  
 Lab 2527 first reported 335.3

Lab 2413 result excluded as free formaldehyde is same as released formaldehyde result



## Determination of Released Formaldehyde content on sample #13191; results in mg/kg

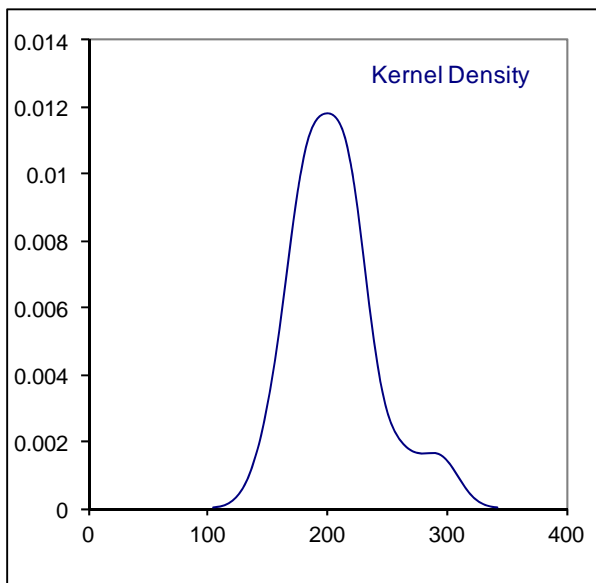
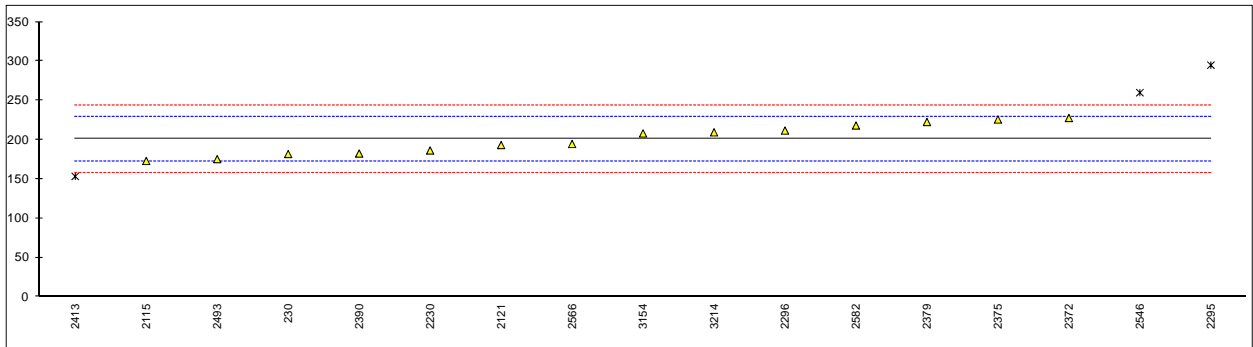
lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110		----		----	2379	ISO14184-2	222.69		1.51
213		----		----	2380		----		----
230	ISO14184-2	181.91		-1.30	2381		----		----
357		----		----	2385		----		----
551		----		----	2390	ISO14184-2	182.5		-1.26
2102		----		----	2410		----		----
2115	ISO14184-2	173		-1.92	2413	ISO14184-1	153.42	ex	-3.27
2120		----		----	2420		----		----
2121	ISO14184-1	193.3		-0.52	2425		----		----
2127		----		----	2426		----		----
2129		----		----	2428		----		----
2132		----		----	2429		----		----
2135		----		----	2432		----		----
2138		----		----	2433		----		----
2139		----		----	2437		----		----
2146		----		----	2442		----		----
2165		----		----	2452		----		----
2170		----		----	2453		----		----
2172		----		----	2454		----		----
2184		----		----	2456		----		----
2186		----		----	2459		----		----
2190		----		----	2460		----		----
2196		----		----	2467		----		----
2197		----		----	2468		----		----
2201		----		----	2472		----		----
2212		----		----	2474		----		----
2213		----		----	2475		----		----
2216		----		----	2476		----		----
2219		----		----	2481		----		----
2225		----		----	2482		----		----
2226		----		----	2483		----		----
2228		----		----	2489		----		----
2229		----		----	2492		----		----
2230	ISO14184-2	186.4		-0.99	2493	ISO14184-2	175.4		-1.75
2232		----		----	2495		----		----
2235		----		----	2501		----		----
2236		----		----	2506		----		----
2238		----		----	2511		----		----
2241		----		----	2514		----		----
2245		----		----	2515		----		----
2246		----		----	2517		----		----
2247		----		----	2518		----		----
2255		----		----	2519		----		----
2256		----		----	2525		----		----
2261		----		----	2527		----		----
2265		----		----	2532		----		----
2266		----		----	2534		----		----
2269		----		----	2535		----		----
2273		----		----	2542		----		----
2275		----		----	2546	ISO14184-2	259.95	DG(0.05)	4.09
2277		----		----	2549		----		----
2284		----		----	2553		----		----
2289		----		----	2555		----		----
2290		----		----	2559		----		----
2293		----		----	2561		----		----
2294		----		----	2562		----		----
2295	ISO14184-2	295	DG(0.05)	6.51	2563		----		----
2296	ISO14184-2	211.72		0.76	2566	ISO14184-2	194.64		-0.42
2298		----		----	2567		----		----
2303		----		----	2573		----		----
2310		----		----	2576		----		----
2311		----		----	2577		----		----
2313		----		----	2580		----		----
2314		----		----	2582	ISO14184-2	218.20		1.20
2351		----		----	2584		----		----
2356		----		----	2587		----		----
2359		----		----	2589		----		----
2360		----		----	2590		----		----
2361		----		----	2591		----		----
2363		----		----	2598		----		----
2364		----		----	3100		----		----
2366		----		----	3110		----		----
2367		----		----	3116		----		----
2368		----		----	3118		----		----
2372	ISO14184-2	227.72		1.86	3146		----		----
2375	ISO14184-2	225.8		1.73	3150		----		----



3153		----	----
3154	ISO14184-2	208.13	0.51
3167		----	----
3172		----	----
3176		----	----
3180		----	----
3182		----	----
3185		----	----
3186		----	----
3190		----	----
3192		----	----
3195		----	----
3197		----	----
3198		----	----
3199		----	----
3207		----	----
3210		----	----
3214	ISO14184-2	209.5	0.60
3220		----	----
3222		----	----
3226		----	----
3228		----	----
3232		----	----
3237		----	----
3242		----	----
3248		----	----

normality	OK	
n	14	
outliers	2	+ 1 excl
mean (n)	200.78	
st.dev. (n)	19.198	
R(calc.)	53.76	
R(Horwitz)	40.50	

Lab 2413 result excluded as free formaldehyde is same as released formaldehyde result



## APPENDIX 2

### Number of participants per country

7 labs in BANGLADESH  
1 lab in BRAZIL  
1 lab in CAMBODIA, Kingdom of  
1 lab in CROATIA  
1 lab in CZECH REPUBLIC  
1 lab in EGYPT  
3 labs in FINLAND  
6 labs in FRANCE  
15 labs in GERMANY  
1 lab in GREECE  
1 lab in GUATEMALA  
13 labs in HONG KONG  
1 lab in HUNGARY  
14 labs in INDIA  
2 labs in INDONESIA  
10 labs in ITALY  
4 labs in KOREA  
2 labs in MAURITIUS  
3 labs in MEXICO  
1 lab in MOROCCO  
1 lab in NICARAGUA  
46 labs in P.R. of CHINA  
3 labs in PAKISTAN  
1 lab in PHILIPPINES  
2 labs in PORTUGAL  
1 lab in ROMANIA  
2 labs in SINGAPORE  
1 lab in SLOVENIA  
1 lab in SPAIN  
2 labs in SRI LANKA  
1 lab in SWITZERLAND  
4 labs in TAIWAN R.O.C.  
3 labs in THAILAND  
1 lab in THE NETHERLANDS  
2 labs in TUNISIA  
5 labs in TURKEY  
7 labs in U.S.A.  
4 lab in UNITED KINGDOM  
2 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

### Literature:

1. iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, January 2010
2. Öko-Tex Standard 100; January 2008.
3. Thai Green label. TGL-16. July 2002.
4. Impacts of Environmental Standards and requirements in EU Countries. Aug 99.
5. Horwitz. Journal of AOAC International Vol. 79 No.3. 1996.
6. P.L. Davies. Fr Z. Anal. Chem. 351. 513 (1988).
7. W.J. Conover. Practical; Nonparametric Statistics. J. Wiley&Sons. NY. p.302 (1971).
8. ISO 5725. (1986).
9. ISO 5725. parts 1-6 (1994).
10. ISO105 E4: 1994.
11. ISO14184-1: 1994.
12. ISO13528-05.
13. M. Thompson and R. Wood. J. AOAC Int. 76. 926. (1993).
14. Analytical Methods Committee Technical brief, No4 January 2001.
15. The Royal Society of Chemistry 2002, Analyst 2002, 127 page 1359-1364, P.J. Lowthian and M. Thompson.(see <http://www.rsc.org/suppdata/an/b2/b205600n/>)
16. Official Journal of the European Communities L133/29: May 2002.