Results of Proficiency Test Free Formaldehyde in textile October 2011

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iis11A04

December 2011

Report:

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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 2011/2012.

In this interlaboratory study 144 laboratories in 29 different countries participated. See appendix 2 for the number of participating laboratories per country.

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

# 2 SET UP

The Institute for Interlaboratory Studies in Spijkenisse was the organiser of this proficiency test. Sample preparation and analyses of fit for use and homogeneity were subcontracted. In this Proficiency Test, it was decided to use two different samples (#11087 and #11089, 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 Spijkenisse, the Netherlands, has implemented a quality system based on ISO guide 43, ILAC-G13:2007 and ISO 17043:2010. This ensures 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 present 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 textile samples, a blue hosiery fabric (sample #11087) and a purple hosiery Fabric (sample #11089) were divided over 150 subsamples of approx. 3 grams. The homogeneity was checked on respectively 15 and 8 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 #11087-1	46
Sample #11087-2	49
Sample #11087-3	48
Sample #11087-4	48
Sample #11087-5	44
Sample #11087-6	46
Sample #11087-7	45
Sample #11087-8	49
Sample #11087-9	45
Sample #11087-10	50
Sample #11087-11	49
Sample #11087-12	46
Sample #11087-13	48
Sample #11087-14	45
Sample #11087-15	47

Table 1: homogeneity test results of subsamples #11087

	Free Formaldehyde in mg/kg
Sample #11089-1	246
Sample #11089-2	246
Sample #11089-3	252
Sample #11089-4	251
Sample #11089-5	254
Sample #11089-6	254
Sample #11089-7	249
Sample #11089-8	248

Table 2: homogeneity test results of subsamples #11089

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 in the next table:

	Free Formaldehyde in mg/kg Sample #11087	Free Formaldehyde in mg/kg Sample#11089		
r	4.9	9.1		
Reference test	Horwitz	Horwitz		
0.3*R <sub>(reference test)</sub>	4.7	10.5		

table 3: repeatabilities of subsamples #11087 and 11089

From the above results of the homogeneity tests, the repeatabilities were calculated. The calculated repeatabilities for samples #11087 and #11089 are both in good agreement with 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, #11087 and #11089 were sent to the participating laboratories on October 12, 2011.

# 2.5 ANALYSES

The participants were asked to determine on samples #11087 and #11089 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 form 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 anormal 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  $z_{(target)}$ -scores were calculated according to:

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

The  $z_{(target)}$ -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 some problems occurred with the delivery of the samples. Two laboratories received the samples late. Two other laboratories did not report any test results and 6 laboratories reported results after the final reporting date.

Finally, the 144 reporting laboratories send in total 284 numerical results. Observed were 5 statistical outlying results, which is 1.8% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

For sample #11089, 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 #11087 and #11089 are discussed. All statistical results reported on the textile samples are summarised in appendix 1.

- Sample #11087: This determination was problematic at a concentration level of 79 mg/kg. Four statistical outliers were observed. The calculated reproducibility is, after rejection of the statistical outliers not in agreement with the estimated reproducibility calculated using the Horwitz equation.
- Sample #11089: This determination was very problematic at a concentration level of 170 mg/kg. 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.

#### 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 #11087	mg/kg	138	78.7	24.7	18.3
Free Formaldehyde #11089	mg/kg	141	169.6	69.3	35.1

Table 4: reproducibilities of textile samples #11087 and #11089

From the above tables it can be concluded that, without statistical calculations, the group of participating laboratories has difficulties with the analysis when compared with the strict 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, that were found in the results of the samples Free Formaldehyde during the present round, are slightly higher than the spreads as observed in previous rounds (see next table).

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

Table 5: Development of relative reproducibilities of Free Formaldehyde over the last 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 some participants would make different decisions about the acceptability of the textiles for the determined parameters, to the majority of the group.

Ecolabel	EU-adult clothes	EU-baby clothes	Öko-Tex 103 non skin contact	Öko-Tex 103 direct skin contact	Öko-Tex 106 baby clothes
Free Formaldehyde in mg/kg	75	30	300	75	20

Table 6: Ecolabelling Standards and Requirements for Textiles in EU

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

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

All laboratories would accept both samples #11087 and #11089 for the category "Öko-Tex 103 non skin contact" (<300 mg/kg), except lab 2455 which reported 381.6 mg/kg on sample #11089.

For sample #11087 is also true that 42 laboratories would accept this sample for the category "EU-adult clothes" or "Öko-Tex 103 direct skin contact" (<75 mg/kg) and 2 laboratories ( lab 2301 and lab 2277) would accept this sample even for the category "EU-baby clothes".

# General

The Free Formaldehyde content was determined at two different levels. 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 raise of the quality of the analytical results.

# **APPENDIX 1**

Determination of Free Formaldehyde on sample #11087; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO14184-1	79.3		0.10	
357	ISO14184-1	79.4		0.11	
551	ISO14184-1	76.2		-0.38	
1213	ISO14184-1	83.1		0.68	
1245	ISO14184-1	90.7		1.85	
1649	ISO14184	84.3		0.86	
2102	INH-443	68.3		-1.59	
2120	ISO14184-1	35.3	G(0.01)	-6.65	
2127	LFGB B82.02-1	86.3		1.17	
2129	ISO14184	71.6		-1.08	
2132	ISO14184-1	80.5		0.28	
2137	ISO14184-1	75.9		-0.42	
2139	15014184-1	70.0		-1.33	
2140	15014184	75.Z	C	-0.53	first reported: 172 0560
2160	13014104-1	77.0092	C	-0.17	llist reported. 172.0009
2105	ISO14184-1	83.1		0.90	
2172	ISO14184-1	81.6		0.00	
2186	Jap, Jaw 112	51.24		-4.20	
2190	ISO14184	76		-0.41	
2196	ISO14184-1	80		0.21	
2197	ISO14184-1	86.14		1.15	
2201	ISO14184-1	78.5		-0.02	
2212	JIS L1041	75.5		-0.48	
2216	Jap. law 112	75.3		-0.52	
2217	ISO14184-1	57.0		-3.32	
2218	ISO14184	55.9		-3.49	
2219	ISO14184-1	79.0		0.05	
2225	ISO14184	80.4		0.27	
2226	ISO14184	84.8		0.94	
2227	Jap. law 112	87.4	C	1.34	first reported: 123.7
2228	Jap. law 112	91.4		1.95	
2229	15014184	72		-1.0Z	
2230	ISO14184	94.0 81.2		2.33	
2230	ISO14184-1	56.2		-3 44	
2247	ISO14184-1	80.77		0.32	
2255	ISO14184-1	81.0		0.36	
2256	ISO14184-1	72.1		-1.01	
2260	ISO14184-1	90.2		1.77	
2261	INH2912	86.319		1.17	
2265	LFGB B82.02-1	83.6		0.76	
2269	ISO14184-1	91.279		1.93	
2277	ISO14184-1	27.2	G(0.01)	-7.89	
2279	ISO14184-1	74.6		-0.62	
2280	ISO14184	72.8		-0.90	
2282	ISO14184-1	83.1		0.68	
2284	15014184-1	72.2		-0.99	
2285	15014184-1	74.85		-0.58	
2209	ISO14104-1	00 88 78		1.55	
2292	10014104-1	67.9		-1.65	
2296	ISO14184-1	64.19		-2.22	
2298	JIS L1041	77.98		-0.10	
2301	ISO14184-1	16.4	C,G(0.01)	-9.54	first reported:31.8
2303	ISO14184-1	80.6		0.30	
2310	ISO14184	83.6		0.76	
2311	ISO14184-1	83.0		0.66	
2313	ISO14184-1	82.0		0.51	
2314	ISO14184-1	84.65		0.92	
2315					
2350	ISO14184	86.1		1.14	
2352	15014184-1	79.4		0.11	
2356	15014184-1	70.4 70.4		-0.35	
2320 2380	13014104-1	19.4 73 G		0.11	
2362	ISO14104-1	13.0 80.6		-0.70	
2364	ISO14184-1	73		-0.30 -0.87	
2365	ISO14184-1	64.8		0.60	
2366	ISO14184	77.4		-0.19	
2367	ISO14184-1	74.1		-0.70	
2368	ISO14184-1	75		-0.56	
2369	ISO14184	77.7		-0.15	
2370	ISO14184-1	77.4		-0.19	

2372	ISO14184-1	86.8		1.25	
2375	ISO14184-1	85.35		1.03	
2379	ISO14184-1	74.7		-0.61	
2380	ISO14184-1	67.00		-1.79	
2385	In house	98		2.96	
2390		73.3		-0.62	
2401	INT-2912	04 75 25		0.62	
2403	15014104-1	75.25		-0.52	
2410	ISO14104-1	053		-0.14	
2415	ISO14184-1	30.0 72 3		-0.98	
2413	ISO14184-1	95.4		2 57	
2426	ISO14184-1	69.5		-1 40	
2427	ISO14184-1	69		-1.48	
2428	ISO14184-1	76.8		-0.29	
2429	ISO14184-1	80.5		0.28	
2432	ISO14184-1	69.5		-1.40	
2433	Jap. law 112	100.9		3.41	
2436	ISO14184	80.8		0.33	
2437	ISO14184-1	75.5		-0.48	
2440	INH-2912	82.5		0.59	
2442	ISO14184-1	64.51		-2.17	
2443	ISO14184	76.1		-0.39	
2446	LFGB B82.02-1	75.5		-0.48	
2449	15014184-1	01		-2.71	
2452	13014104-1	90.0		2.75	
2404	ISO14104-1	180.5	C G(0,01)	15.61	first reported:10/0/
2456	ISO1/220-1	85.8	0,0(0.01)	1 00	mst reported. 1049.4
2450	ISO14184-1	67 76		-1.67	
2458	ISO14184	69.5		-1.40	
2459	ISO14184-1	76.5		-0.33	
3100	ISO14184-1	75.2		-0.53	
3101	ISO14184-1	86.0		1.12	
3104	ISO14184-1	83.1		0.68	
3110	ISO14184-1	73.39		-0.81	
3116	ISO14184-1	76.5		-0.33	
3117	ISO14184-1	72.7		-0.91	
3118	ISO14184-1	88.49		1.51	
3134	ISO14184	87.0		1.28	
3145		90.5 74 7		2.73	
3150	LFGD D02.02-1	74.7 66.6		-0.01	
3154	ISO14184-1	87.9		1 42	
3159	ISO14184-1	84 18		0.85	
3166	ISO14184-1	86.9	С	1.26	first reported: 85.8
3167	ISO14184-1	87.7	C	1.39	
3172	ISO14184-1	76.3		-0.36	
3174	ISO14184-1	79.0		0.05	
3176	ISO14184-1	71.4	С	-1.11	first reported: 51.1
3182	ISO14184-1	86.3		1.17	
3185	ISO14184-1	77.5		-0.18	
3190	ISO14184-1	81.0		0.36	
3191	ISO14184-1	76		-0.41	
3192	LFGB B82.02-1	76.4		-0.35	
3195	ISO14184-1	90.0		1.74	
3197	15014184-1	85.6		1.06	
3200	ISO14104-1	93.2 77.5		2.23 -0.18	
3200		74		-0.10	
3208	010 21041				
3210	ISO14184-1	75.5		-0.48	
3218	ISO14184-1	68.4		-1.57	
3220				0 50	
3222	ISO14184-1	101.5		3.50	
0222	ISO14184-1 ISO14184-1	101.5 69.4		3.50 -1.42	
3226	ISO14184-1 ISO14184-1 ISO14184	101.5 69.4 76.3		3.50 -1.42 -0.36	
3226 3228	ISO14184-1 ISO14184-1 ISO14184 ISO14184-1	101.5 69.4 76.3 77.6		3.50 -1.42 -0.36 -0.16	
3226 3228 3232	ISO14184-1 ISO14184-1 ISO14184 ISO14184-1 ISO14184-1	101.5 69.4 76.3 77.6 75.9		3.50 -1.42 -0.36 -0.16 -0.42	
3226 3228 3232 3237	ISO14184-1 ISO14184-1 ISO14184 ISO14184-1 ISO14184-1 ISO14184-1	101.5 69.4 76.3 77.6 75.9 67		3.50 -1.42 -0.36 -0.16 -0.42 -1.79	
3226 3228 3232 3237 3248	ISO14184-1 ISO14184-1 ISO14184 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1	101.5 69.4 76.3 77.6 75.9 67 63		3.50 -1.42 -0.36 -0.16 -0.42 -1.79 -2.40	
3226 3228 3232 3237 3248	ISO14184-1 ISO14184-1 ISO14184 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1	101.5 69.4 76.3 77.6 75.9 67 63		3.50 -1.42 -0.36 -0.16 -0.42 -1.79 -2.40	
3226 3228 3232 3237 3248	ISO14184-1 ISO14184-1 ISO14184 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 normality	101.5 69.4 76.3 77.6 75.9 67 63 OK 138		3.50 -1.42 -0.36 -0.16 -0.42 -1.79 -2.40	
3226 3228 3232 3237 3248	ISO14184-1 ISO14184-1 ISO14184 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 normality n outliers	101.5 69.4 76.3 77.6 75.9 67 63 OK 138 4		3.50 -1.42 -0.36 -0.16 -0.42 -1.79 -2.40	
3226 3228 3232 3237 3248	ISO14184-1 ISO14184-1 ISO14184 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 normality n outliers mean (n)	101.5 69.4 76.3 77.6 75.9 67 63 OK 138 4 78.66		3.50 -1.42 -0.36 -0.16 -0.42 -1.79 -2.40	
3226 3228 3232 3237 3248	ISO14184-1 ISO14184-1 ISO14184 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 normality n outliers mean (n) st.dev. (n)	101.5 69.4 76.3 77.6 75.9 67 63 OK 138 4 78.66 8.827		3.50 -1.42 -0.36 -0.16 -0.42 -1.79 -2.40	
3226 3228 3232 3237 3248	ISO14184-1 ISO14184-1 ISO14184 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 normality n outliers mean (n) st.dev. (n) R(calc.)	101.5 69.4 76.3 77.6 75.9 67 63 OK 138 4 78.66 8.827 24.72		3.50 -1.42 -0.36 -0.16 -0.42 -1.79 -2.40	





# Determination of Free Formaldehyde on sample #11089; results in mg/kg

lak	us a the a st			no no na luc
lab	method	value	mark z(targ)	remarks
110	ISO14184-1	169.9	0.03	
357	ISO14184-1	194.8	2.01	
551	ISO14184-1	164.1	-0.44	
1213	ISO14184-1	117.9	-4.12	
1245	ISO14184-1	217.3	3.81	
1649	ISO14184	197.3	2.21	
2102	INH-443	114.4	-4.40	
2120	ISO14184-1	153.2	-1.31	
2127	LEGB B82 02-1	107.8	2.25	
2120	ISO14184	135.8	-2.69	
2120	ISO14184-1	176.0	0.51	
2122	19014194 1	170.0	1.56	
2137	10014104-1	100.0	-1.50	
2139	15014164-1	160.0	-0.76	
2146	15014184	180.9	0.90	
2160	ISO14184-1	229.2995	4.77	
2165	ISO14184-1	172.0	0.19	
2172	ISO14184	185.4	1.26	
2184	ISO14184-1	171.1	0.12	
2186	Jap. law 112	114.20	-4.42	
2190	ISO14184	162	-0.60	
2196	ISO14184-1	179	0.75	
2197	ISO14184-1	197.8	2.25	
2201	ISO14184-1	179.6	0.80	
2212	JIS L1041	173.8	0.34	
2216	Jap. law 112	175.8	0.50	
2217	ISO14184-1	140.5	-2.32	
2218	ISO14184	141 1	-2 27	
2210	ISO14184-1	175.7	0.49	
2225	19014184	182.3	1.02	
2220	19014194	170.9	0.92	
2220	13014104	179.0	0.02	
2227	Jap. law 112	100.0	1.23	
2220		174.3	0.38	
2229	15014184	161	-0.68	
2236	ISO14184	215.2	3.64	
2238	ISO14184	194.8	2.01	
2241	ISO14184-1	145.2	-1.94	
2247	ISO14184-1	166.43	-0.25	
2255	ISO14184-1	145.0	-1.96	
2256	ISO14184-1	166	-0.28	
2260	ISO14184-1	192.1	1.80	
2261	INH2912	177.395	0.63	
2265	LFGB B82.02-1	179.6	0.80	
2269	ISO14184-1	150.533	-1.52	
2277	ISO14184-1	118.6	-4.07	
2279	ISO14184-1	172.9	0.27	
2280	ISO14184	175.8	0.50	
2282	ISO14184-1	139.6	-2 39	
2284	ISO14184-1	164.9	-0.37	
2285	ISO14184-1	173 04	0.07	
2200	10014104 1	106	2.11	
2209	10014104-1	190	2.11	
2292	13014104-1	190.14	2.12	
2295	10014104 4	144.17	-2.03	
2290	15014164-1	137.69	-2.53	
2298	JIS L1041	173.28	0.30	
2301	ISO14184-1	126.2	-3.46	
2303	ISO14184-1	183.0	1.07	
2310	ISO14184	186.6	1.36	
2311	ISO14184-1	183.8	1.14	
2313	ISO14184-1	177.0	0.59	
2314	ISO14184-1	181.74	0.97	
2315				
2350	ISO14184	204.4	2.78	
2352	ISO14184-1	179.8	0.82	
2356	ISO14184-1	170.3	0.06	
2358	ISO14184-1	180.4	0.86	
2360	ISO14184-1	155.2	-1.15	
2363	ISO14184	183.4	1.10	
2364	ISO14184-1	169	-0.05	
2365	ISO14184-1	136.8	0.55	
2366	ISO14184	175 5	0.00	
2367	ISO14184-1	164 5	_0.47	
2001	10014104-1	171	-0.40	
2300	10014104-1	177 4	0.11	
2009 2270	10014104	170	0.60	
2310	10014104-1	204 7	0.67	
2312	15014184-1	201.7	2.56	
2315	13014184-1	124.19	-3.62	

2401 2403 2410 2413 2415 2420 2427 2428 2427 2428 2429 2432 2433 2436 2437 2440 2442 2443 2446 2449 2452 2454	INH-2912 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184 ISO14184 ISO14184 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1	184 177.26 183.04 207.1 114.3 192.8 141 162 177.2 194.6 156.5 198.5 180.0 180.0 174.5 130.00 160.9 159.0 136 201.3 160.3 381.6	C C,G(0.01)	$\begin{array}{c} 1.15\\ 0.61\\ 1.08\\ 3.00\\ -4.41\\ 1.85\\ -2.28\\ -0.60\\ 0.61\\ 2.00\\ -1.04\\ 2.31\\ 0.83\\ 0.39\\ -3.16\\ -0.69\\ -0.84\\ -2.68\\ 2.53\\ -0.74\\ 16.92\\ \end{array}$	first reported: 109.2 first reported: 2322,4
2456 2457 2458 2459 3100 3101 3104 3110 3116 3117 3118 3134 3145 3155 3155 3154 3159	ISO14184-1 Jap. law 112 ISO14184 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	222.6 149.3 161.6 104.1 176.3 164.0 158.1 153.23 164.8 178.0 131.72 153.9 214.7 164.0 137.9 185.7 177.60		4.23 -1.62 -0.64 -5.23 0.54 -0.92 -1.30 -0.38 0.67 -3.02 -1.25 3.60 -0.44 -2.53 1.29 0.64	
3166 3167 3172 3174 3176 3182 3185 3190 3191 3192 3195 3197 3199 3200 3207 3208	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 JIS L1041	204.6 212.1 160.3 137.4 146.8 119.2 172.5 174.6 174 174.9 194.0 159.7 204.7 192.8 141	С	2.80 3.40 -0.74 -2.57 -1.82 -4.02 0.23 0.40 0.35 0.43 1.95 -0.79 2.80 1.85 -2.28	first reported:296.0
3208 3210 3218 3220 3222 3226 3228 3232 3237 3248	ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184 ISO14184-1 ISO14184-1 ISO14184-1 ISO14184-1	166 164.9 187.4 211.1 132.1 165.8 180.6 137 146		-0.28 -0.37 1.42 3.32 -2.99 -0.30 0.88 -2.60 -1.88	
	normality n outliers mean (n) st.dev. (n) R(calc.) R(Horwitz)	not OK 141 1 169.56 24.740 69.27 35.08			





# **APPENDIX 2**

#### Number of participants per country

1 lab in AUSTRIA

4 labs in BANGLADESH

1 lab in BRAZIL

3 labs in FINLAND

2 labs in FRANCE

13 labs in GERMANY

1 lab in GREECE

13 labs in HONG KONG

1 lab in HRVATSKA - CROATIA

1 lab in HUNGARY

8 labs in INDIA

2 labs in INDONESIA

5 labs in ITALY

4 labs in KOREA

3 labs in MEXICO

1 lab in NEW ZEALAND

46 labs in P.R. of CHINA

5 labs in PAKISTAN

1 lab in PHILIPPINES

1 lab in PORTUGAL

1 lab in SLOVENIA

2 labs in TAIWAN R.O.C.

4 labs in THAILAND

1 lab in THE NETHERLANDS

1 lab in TUNISIA

6 labs in TURKEY

9 labs in U.S.A.

2 labs in UNITED KINGDOM

2 labs in VIETNAM

# **APPENDIX 3**

### Abbreviations:

С	= 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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