# Results of Proficiency Test Free and Released Formaldehyde in textile October 2014

Organised by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

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Report: iis14A04

January 2015

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#### 1 INTRODUCTION

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

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

In the 2014/2015 interlaboratory study 178 laboratories in 39 different countries participated. See appendix 2 for the number of participating laboratories per country. In this report, the results of the 2014 proficiency test are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

#### 2 SET UP

The Institute for Interlaboratory Studies in Spijkenisse was the organiser of this proficiency test. Sample preparation and analyses of fit for use and homogeneity were subcontracted. It was decided to use in this Proficiency Test one positive sample of approx. 2.5 grams, labelled #14207. The analyses for fit-for-use testing were subcontracted to an ISO17025 accredited laboratory. The participants were requested to report rounded and unrounded results. These unrounded results were preferably used for the statistical evaluations.

#### 2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in accordance with ISO/IEC 17043:2010, (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie, see also www.RVA.nl). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

#### 2.2 PROTOCOL

The protocol followed in the organisation was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol can be downloaded from the iis website 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 cotton sample (labelled, #14207) was divided over 190 subsamples of approx. 2.5 grams. The homogeneity of the sample was checked on 8 stratified randomly selected samples. The homogeneity testing was performed by a subcontracted ISO17027 accredited laboratory. See the following tables for the test results.

|                 | Free Formaldehyde in mg/kg |
|-----------------|----------------------------|
| Sample #14207-1 | 112                        |
| Sample #14207-2 | 111                        |
| Sample #14207-3 | 115                        |
| Sample #14207-4 | 116                        |
| Sample #14207-5 | 114                        |
| Sample #14207-6 | 116                        |
| Sample #14207-7 | 113                        |
| Sample #14207-8 | 112                        |

Table 1: homogeneity test results of subsamples #14207

From the above test results, the repeatability was 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<br>Sample #14207 |  |  |  |  |
|------------------------------------------|---------------------------------------------|--|--|--|--|
| r                                        | 5.4                                         |  |  |  |  |
| Reference test method                    | Horwitz                                     |  |  |  |  |
| 0.3*R <sub>(reference test method)</sub> | 7.5                                         |  |  |  |  |

Table 2: repeatability of subsamples #14207

The calculated repeatability of subsamples #14207 is in agreement with 0.3 times the estimated target reproducibility, calculated using the Horwitz equation. Therefore, homogeneity of all subsamples was assumed.

To the participants, approx. 2.5 grams of sample, labelled #14207 was sent on October 15, 2014

# 2.5 ANALYSES

The participants were asked to determine on sample #14207 the Free Formaldehyde content and the Released Formaldehyde content with the analytical procedures that are routinely used in the laboratory.

It was requested to report the analytical results using the indicated units on the report form and to use a minimum number of digits and not to round the results more. It was also requested not to report 'less than' results, which are above the detection limit, because such results cannot be used for meaningful statistical calculations. To get comparable results a detailed report form, on which the units were prescribed, was sent together with each set of samples. Also, a letter of instructions was added to the package.

### 3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were received. The original data are tabulated per sample in the appendix 1 of this report. The laboratories are represented by the code numbers.

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

Shortly after the deadline, the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. Additional or corrected results are used for the data analysis and the original results are placed under 'Remarks' in the result tables in appendix 1.

# 3.1 STATISTICS

The statistical calculations were performed as described in the procedures in the report 'iis Interlaboratory Studies, Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3).

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon, Grubbs and or Rosner General ESD outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test and by R(0.01) for the Rosner General ESD test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test and by R(0.05) for the Rosner General ESD test (ref. 17). Both outliers and stragglers were not included in the calculations of averages and standard deviations.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have significant consequences for the evaluation of the test results.

#### 3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are under the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nr.14 and 15). Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

# 3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of this interlaboratory study.

The target standard deviation was calculated from the target reproducibility (preferably taken from a standardized test method) by division with 2.8.

The z-scores were calculated in accordance with:

z<sub>(target)</sub> = (result - average of PT) / target standard deviation

The z (target) scores are listed in the result tables in appendix 1.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used. This should be done in order to evaluate whether the reported test results are fit-for-purpose.

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

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

#### 4 EVALUATION

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

Finally, the 176 reporting laboratories sent in total 193 numerical results. Observed were 8 statistical outlying results, which is 4.1% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as "not OK" or "suspect". The statistical evaluation of these data sets should be used with due care.

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 sample #14207 is discussed. All statistical results reported on the textile samples are summarised in appendix 1.

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

Released Formaldehyde: This determination was problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outlier is not 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 #14207     | mg/kg | 170 | 158     | 37       | 33         |
| Released Formaldehyde #14207 | mg/kg | 15  | 225     | 61       | 45         |

Table 3: reproducibilities of textile sample #14207

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

#### 5 COMPARISON WITH THE PREVIOUS PROFICIENCY TESTS

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

| Darameter          | October | October | October | October | October | October |
|--------------------|---------|---------|---------|---------|---------|---------|
| Parameter          | 2014    | 2013    | 2012    | 2011    | 2010    | 2009    |
| Free Formaldehyde  | 8%      | 10-13%  | 8-9%    | 11-15%  | 7-9%    | 9-12%   |
| Released Formalde. | 10%     | 9-10%   | n.e.    | n.e.    | n.e.    | n.e.    |

Table 4: Development of uncertainties (in %) 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<br>no direct skin<br>contact | Öko-Tex 103<br>in direct skin<br>contact | Decoration<br>material |
|---------------------------------------|--------------|------------------------------------------|------------------------------------------|------------------------|
| Free Formaldehyde extractable (mg/kg) | <16          | 300                                      | 75                                       | 300                    |
| Released Formaldehyde (mg/m³)         | 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:

All reporting laboratories would accept sample, #14207 for the categories "Öko-Tex 103 no direct skin contact" and "Decoration material" (<300 mg/kg), but would reject the sample for the other 2 classes ("Baby clothes" and "in direct skin contact").

# Released Formaldehyde:

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

#### General

In this proficiency test the Free Formaldehyde content and the Released Formaldehyde were determined. The spread observed for the Free Formaldehyde content and the Released Formaldehyde in this interlaboratory study are in line with the previous proficiency tests. The spread observed in this interlaboratory study can be caused by the preparation or the conditioning of the sample and/or by the performance of the analysis. Consequently, the reproducibility cannot be improved by only one change in the analysis. Each laboratory has to evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1

Determination of Free Formaldehyde content on sample #14207; results in mg/kg

| lab          | method                   | value            | mark    | z(targ)        | lab          | method                   | value            | mark      | z(targ)        |
|--------------|--------------------------|------------------|---------|----------------|--------------|--------------------------|------------------|-----------|----------------|
| 110          | ISO14184-1               | 174.3            | mark    | 1.38           | 2456         | ISO14184-1               | 283.0            | R(0.01)   | 10.59          |
| 213          | ISO14184-1               | 138.75           |         | -1.63          | 2457         | JapLaw112                | 168.6            | 11(0.01)  | 0.90           |
| 348          | in house                 | 156.50           |         | -0.13          | 2458         | ISO14184-1               | 159.04           |           | 0.09           |
| 361          | ISO14184-1               | 188.72           |         | 2.60           | 2460         | ISO14184-1               | 157.89           |           | -0.01          |
| 551          | ISO14184-1               | 144.99           |         | -1.10          | 2462         | ISO14184-1               | 151.89           |           | -0.52          |
| 1132         | ISO14184-1               | 163.38           |         | 0.46           | 2468         | ISO14184-1               | 155.92           |           | -0.18          |
| 1213         | TCVN7421                 | 145.3            |         | -1.08          | 2472         | ISO14184-1               | 159.9            |           | 0.16           |
| 2102         | ISO14184-1               | 155.94           |         | -0.18          | 2476         | ISO14184-1               | 151.12           |           | -0.58          |
| 2104         | ISO14184-1               | 160.6            |         | 0.22           | 2481         | ISO17226-1               | 84.8             | R(0.01)   | -6.20          |
| 2108         | JapLaw112                | 157.20           |         | -0.07          | 2482         | ISO14184-1               | 157.93           | ` ,       | -0.01          |
| 2115         | ISO14184-1               | 165.00           |         | 0.59           | 2483         | ISO14184                 | 93               | C,R(0.01) | -5.51          |
| 2120         | ISO14184-1               | 135.98           |         | -1.87          | 2488         | ISO14184-1               | 169.97           |           | 1.01           |
| 2121         | ISO14184-1               | 126.78           |         | -2.65          | 2489         | ISO14184-1               | 157.8            |           | -0.02          |
| 2129         | ISO14184-2               | 141.4            |         | -1.41          | 2492         | in house                 | 138.8            |           | -1.63          |
| 2132         | ISO14184-1               | 165.71           |         | 0.65           | 2495         | ISO14184-1               | 139.8            |           | -1.54          |
| 2135         | ISO14184-1               | 132.6            |         | -2.15          | 2497         | ISO14184-1               | 173.4            |           | 1.30           |
| 2137         | 100444044                | 128.2            |         | -2.53          | 2500         | ISO14184-1               | 170.14           |           | 1.03           |
| 2139         | ISO14184-1               | 167.77           |         | 0.83           | 2501         | ISO14184-1               | 159.00           |           | 0.08           |
| 2146         | ISO14184-1               | 156.5            |         | -0.13          | 2504         | ISO14184-1               | 151.72           |           | -0.53          |
| 2170         | ISO14184-1               | 153.07           |         | -0.42          | 2506         | ISO14184-1               | 182.34           |           | 2.06           |
| 2172<br>2182 | ISO14184-1<br>ISO14184-1 | 162.07<br>149.06 |         | 0.34<br>-0.76  | 2511<br>2514 | ISO14184-1<br>ISO14184-1 | 146.8<br>153.33  |           | -0.95<br>-0.40 |
| 2184         | ISO14184-1               | 166.6            |         | 0.73           | 2514         | ISO14184-1               | 163.0            |           | 0.42           |
| 2186         | ISO14184-1               | 151.2            |         | -0.58          | 2517         | ISO14184-1               | 158.1            |           | 0.42           |
| 2190         | ISO14184-1               | 180.12           |         | 1.87           | 2519         | ISO14184-1               | 176.2            |           | 1.54           |
| 2196         | ISO14184-1               | 159.1            |         | 0.09           | 2522         | ISO14184-1               | 131.35           | С         | -2.26          |
| 2201         | ISO14184-1               | 162.62           |         | 0.39           | 2525         | ISO14184-1               | 167.97           | Ü         | 0.84           |
| 2212         | JISL1041                 | 151.83           |         | -0.52          | 2532         | ISO14184-1               | 162.8719         |           | 0.41           |
| 2216         | JapLaw112                | 159.53           |         | 0.13           | 2534         | ISO14184-1               | 152.0            |           | -0.51          |
| 2219         | ISO14184-1               | 169.19           |         | 0.95           | 2535         | ISO14184-1               | 156.848          |           | -0.10          |
| 2226         | ISO14184-1               | 142.0            | С       | -1.36          | 2538         | §35LMBG82.02.1           | 171.67           | С         | 1.16           |
| 2229         | ISO14184-1               | 147.61           |         | -0.88          | 2546         | ISO14184-1               | 190.8256         |           | 2.78           |
| 2230         | ISO14184-1               | 92.96            | R(0.01) | -5.51          | 2553         | ISO14184-1               | 167.09           |           | 0.77           |
| 2232         | ISO14184-1               | 174.14           |         | 1.37           | 2559         | ISO14184-1               | 180.40           |           | 1.90           |
| 2236         | JISL1041                 | 159.85           |         | 0.16           | 2566         | ISO14184-1               | 109.33           | R(0.05)   | -4.13          |
| 2238         | ISO14184-1               | 180.11           |         | 1.87           | 2567         | ISO14184-1               | 147.88           |           | -0.86          |
| 2241         | ISO14184-1               | 149.4            |         | -0.73          | 2571         | ISO14184-1               | 161.25<br>153.75 |           | 0.27           |
| 2245         | ISO14184-1               | 154.5            |         | -0.30<br>-0.99 | 2572         | ISO14184-1               |                  |           | -0.36          |
| 2246<br>2247 | ISO14184-1<br>ISO14184-1 | 146.3<br>162.65  |         | 0.39           | 2586<br>2590 | ISO14184-1<br>ISO14184-1 | 181.75<br>160.0  |           | 2.01<br>0.17   |
| 2255         | ISO14184-1               | 142.12           |         | -1.35          | 2590         | INH-58                   | 148.2            |           | -0.83          |
| 2256         | ISO14184-1               | 163.7            | С       | 0.48           | 2596         | GB/T2912                 | 163.33           |           | 0.45           |
| 2261         | GB/T2912                 | 176.5535         | · ·     | 1.57           | 2603         | ISO14184-1               | 170.14           |           | 1.03           |
| 2265         | ISO14184-1               | 169.88           |         | 1.01           | 2612         | ISO14184-1               | 169.4            |           | 0.97           |
| 2269         | ISO14184-1               | 165.63           |         | 0.65           | 2614         |                          |                  |           |                |
| 2275         | ISO14184-1               | 163.02           |         | 0.42           | 2623         | ISO14184-1               | 186.47           |           | 2.41           |
| 2277         | ISO14184-1               | 144.2            | С       | -1.17          | 2624         |                          |                  |           |                |
| 2289         | ISO14184-1               | 151.22           |         | -0.58          | 2625         | ISO14184-1               | 104.28           | C,R(0.05) | -4.55          |
| 2290         | ISO14184-1               | 158.2            |         | 0.02           | 2626         | ISO14184-1               | 156.3            |           | -0.14          |
| 2293         | ISO14184-1               | 152.976          |         | -0.43          | 2631         | ISO14184-1               | 176.00           |           | 1.53           |
| 2294         | ISO14184-1               | 161.10           |         | 0.26           | 2635         | ISO14184-1               | 162.93           |           | 0.42           |
| 2295<br>2296 | ISO14184-1<br>ISO14184-1 | 177<br>163.68    |         | 1.61<br>0.48   | 2638<br>2644 | ISO14184-1<br>ISO14184-1 | 136.8<br>141.5   |           | -1.80<br>-1.40 |
| 2298         | JISL1041                 | 139.3            |         | -1.59          | 2648         | 10017104-1               | 162.56           |           | 0.39           |
| 2301         | ISO14184-1               | 161.92           |         | 0.33           | 3100         | ISO14184-1               | 152.48           |           | -0.47          |
| 2310         | ISO14184-1               | 158              |         | 0.00           | 3110         | ISO14184-1               | 153.76           |           | -0.36          |
| 2311         | 100111011                | 154              |         | -0.34          | 3116         | ISO14184-1               | 156.43           |           | -0.13          |
| 2313         | ISO14184-1               | 153.4            |         | -0.39          | 3117         | ISO14184-1               | 174.7            |           | 1.41           |
| 2314         | ISO14184-1               | 160.8            |         | 0.24           | 3118         | ISO14184-1               | 152.52           |           | -0.46          |
| 2330         | ISO14184-1               | 153.82           |         | -0.35          | 3146         | ISO14184-1               | 159.4            | С         | 0.12           |
| 2351         | ISO14184-1               | 166.54           |         | 0.72           | 3150         | ISO14184-1               | 124.7            |           | -2.82          |
| 2356         | ISO14184-1               | 173.70           |         | 1.33           | 3153         | ISO14184-1               | 147.85           |           | -0.86          |
| 2360         | ISO14184-1               | 162              |         | 0.34           | 3154         | ISO14184-1               | 138.82           |           | -1.63          |
| 2362         | INH-2012                 | 150.01           |         | -0.68          | 3167         | ISO14184-1               | 163.40           |           | 0.46           |
| 2363         | ISO14184-1               | 160.25           |         | 0.19           | 3172         | ISO14184-1               | 145.45           |           | -1.06          |
| 2364         | ISO14184-1               | 158              |         | 0.00           | 3176         | ISO14184-1               | 174.35           |           | 1.39           |
| 2366         | ISO14184-1               | 150.03           |         | -0.68          | 3180         | ISO14184                 | 126.7            |           | -2.65          |
| 2367<br>2368 | ISO14184-1<br>ISO14184-1 | 153.96<br>151.04 |         | -0.34<br>-0.59 | 3182<br>3183 | ISO14184<br>JapLaw112    | 150.02<br>165.55 |           | -0.68<br>0.64  |
| 2372         | ISO14184-1               | 151.04<br>157.6  |         | -0.59          | 3185         | ISO14184-1               | 152.75           |           | -0.45          |
| 2373         | ISO14184-1               | 152.48           |         | -0.03          | 3190         | ISO14184-1               | 192.47           |           | 2.92           |
| 2375         | ISO14184-1               | 154.5            |         | -0.30          | 3191         | ISO14184-1               | 157              |           | -0.09          |
| 2379         | ISO14184-1               | 152.67           |         | -0.45          | 3192         | §64LFGB B82.02.1         | 159.62           |           | 0.14           |
| 2380         | ISO14184-1               | 141.50           |         | -1.40          | 3195         | ISO14184-1               | 139              |           | -1.61          |
|              |                          |                  |         |                |              |                          |                  |           |                |

| 2381 | ISO14184-1     | 142.16 | -1.34 | 3197 | ISO14184-1 | 154.10     | -0.33 |
|------|----------------|--------|-------|------|------------|------------|-------|
| 2386 | ISO14184-1     | 173    | 1.27  | 3199 | ISO14184-1 | 164.093626 | 0.52  |
| 2389 | ISO14184-1     | 179.87 | 1.85  | 3200 | ISO14184-1 | 156.03     | -0.17 |
| 2390 | ISO14184-1     | 190.67 | 2.77  | 3207 | JIS1041    | 153.6      | -0.37 |
| 2410 | ISO14184-1     | 148    | -0.85 | 3210 | ISO14184-1 | 157.2      | -0.07 |
| 2426 | ISO14184-1     | 154.58 | -0.29 | 3212 | ISO14184-1 | 158.26     | 0.02  |
| 2428 | ISO14184-1     | 166.7  | 0.74  | 3214 | ISO14184-1 | 149        | -0.76 |
| 2429 | ISO14184-1     | 158.78 | 0.07  | 3218 | ISO14184-1 | 149.75     | -0.70 |
| 2432 | ISO14184-1     | 145.76 | -1.04 | 3220 | ISO14184-1 | 169.3      | 0.96  |
| 2433 | ISO14184-1     | 157.80 | -0.02 | 3225 | ISO14184-1 | 160.14     | 0.18  |
| 2441 | ISO14184-1     | 156.2  | -0.15 | 3228 | ISO14184-1 | 162.7      | 0.40  |
| 2442 | ISO14184-1     | 150.23 | -0.66 | 3237 | ISO14184-1 | 137.7      | -1.72 |
| 2446 | §64LFGB82.02.1 | 180.10 | 1.87  | 3246 | ISO14184-1 | 140.23     | -1.51 |
| 2452 | ISO14184-1     | 154.94 | -0.26 | 3248 | GB/T2912   | 155        | -0.25 |
| 2453 | ISO14184-1     | 188.8  | 2.61  | 8005 | JISB       | 155.91     | -0.18 |
|      |                |        |       |      |            |            |       |

normality OK 170 outliers 158.01 mean (n) st.dev. (n) 13.075 R(calc.) R(Horwitz) 36.61 33.04

Lab 2226: test result was first reported as Released Formaldehyde

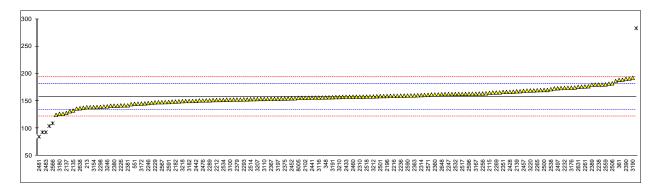
Lab 2256: test result was first reported as Released Formaldehyde Lab 2277: test result was first reported as Released Formaldehyde

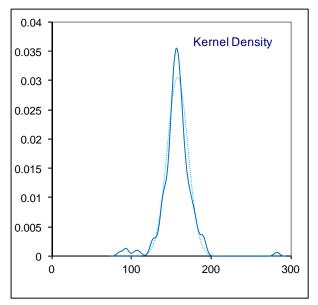
Lab 2483 first reported: 202

Lab 2522 first reported:113.35

Lab 2538: test result was first reported as Released Formaldehyde Lab 2625 first reported: 52.14

Lab 3146: test result was first reported as Released Formaldehyde



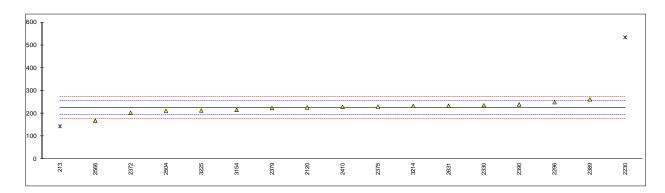


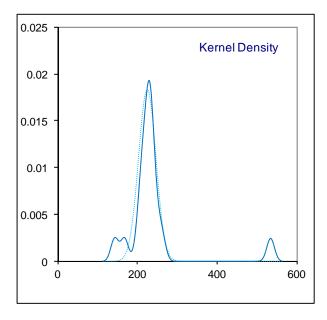
# Determination of Released Formaldehyde content on sample #14207; results in mg/kg

| 1-1-         |            |        |         | -(1)    | 1-1-            |            |        |      | -(1      |
|--------------|------------|--------|---------|---------|-----------------|------------|--------|------|----------|
| lab<br>110   | method     | value  | mark    | z(targ) | <b>lab</b> 2456 | method     | value  | mark | z(targ)  |
| 213          | ISO14184-2 | 143.61 | G(0.05) | -5.11   | 2457            |            |        |      |          |
| 348          |            |        | ,       |         | 2458            |            |        |      |          |
| 361          |            |        |         |         | 2460            |            |        |      |          |
| 551          |            |        |         |         | 2462            |            |        |      |          |
| 1132<br>1213 |            |        |         |         | 2468<br>2472    |            |        |      |          |
| 2102         |            |        |         |         | 2476            |            |        |      |          |
| 2104         |            |        |         |         | 2481            |            |        |      |          |
| 2108         |            |        |         |         | 2482            |            |        |      |          |
| 2115         | 100444040  |        |         |         | 2483            |            |        |      |          |
| 2120<br>2121 | ISO14184-2 | 226.15 |         | 0.06    | 2488<br>2489    |            |        |      |          |
| 2121         |            |        |         |         | 2492            |            |        |      |          |
| 2132         |            |        |         |         | 2495            |            |        |      |          |
| 2135         |            |        |         |         | 2497            |            |        |      |          |
| 2137         |            |        |         |         | 2500            |            |        |      |          |
| 2139<br>2146 |            |        |         |         | 2501<br>2504    | ISO14184-2 | 212.35 |      | -0.80    |
| 2170         |            |        |         |         | 2504            | 13014104-2 | 212.33 |      | -0.60    |
| 2172         |            |        |         |         | 2511            |            |        |      |          |
| 2182         |            |        |         |         | 2514            |            |        |      |          |
| 2184         |            |        |         |         | 2517            |            |        |      |          |
| 2186<br>2190 |            |        |         |         | 2518<br>2519    |            |        |      |          |
| 2196         |            |        |         |         | 2522            |            |        |      |          |
| 2201         |            |        |         |         | 2525            |            |        |      |          |
| 2212         |            |        |         |         | 2532            |            |        |      |          |
| 2216         |            |        |         |         | 2534            |            |        |      |          |
| 2219         |            |        | 0       |         | 2535            |            |        | 0    |          |
| 2226<br>2229 |            |        | С       |         | 2538<br>2546    |            |        | С    |          |
| 2230         | ISO14184-2 | 534.0  | G(0.01) | 19.38   | 2553            |            |        |      |          |
| 2232         |            |        | - ( ,   |         | 2559            |            |        |      |          |
| 2236         |            |        |         |         | 2566            | ISO14184-2 | 168.33 |      | -3.56    |
| 2238         |            |        |         |         | 2567            |            |        |      |          |
| 2241<br>2245 |            |        |         |         | 2571<br>2572    |            |        |      |          |
| 2245         |            |        |         |         | 2586            |            |        |      |          |
| 2247         |            |        |         |         | 2590            |            |        |      |          |
| 2255         |            |        |         |         | 2591            |            |        |      |          |
| 2256         |            |        | С       |         | 2596            |            |        |      |          |
| 2261<br>2265 |            |        |         |         | 2603<br>2612    |            |        |      |          |
| 2269         |            |        |         |         | 2614            |            |        |      |          |
| 2275         |            |        |         |         | 2623            |            |        |      |          |
| 2277         |            |        | С       |         | 2624            | ISO14184-2 | n.d.   |      |          |
| 2289         |            |        |         |         | 2625            |            |        |      |          |
| 2290<br>2293 |            |        |         |         | 2626            | ISO14194 2 | 233.95 |      | 0.55     |
| 2293         |            |        |         |         | 2631<br>2635    | ISO14184-2 | 233.93 |      | 0.55<br> |
| 2295         |            |        |         |         | 2638            |            |        |      |          |
| 2296         | ISO14184-2 | 249.60 |         | 1.54    | 2644            |            |        |      |          |
| 2298         |            |        |         |         | 2648            |            |        |      |          |
| 2301<br>2310 |            |        |         |         | 3100<br>3110    |            |        |      |          |
| 2310         |            |        |         |         | 3116            |            |        |      |          |
| 2313         |            |        |         |         | 3117            |            |        |      |          |
| 2314         |            |        |         |         | 3118            |            |        |      |          |
| 2330         | ISO14184-2 | 236.08 |         | 0.69    | 3146            |            |        | С    |          |
| 2351<br>2356 |            |        |         |         | 3150<br>3153    |            |        |      |          |
| 2360         |            |        |         |         | 3154            | ISO14184-2 | 215.78 |      | -0.59    |
| 2362         |            |        |         |         | 3167            | 100111012  |        |      |          |
| 2363         |            |        |         |         | 3172            |            |        |      |          |
| 2364         |            |        |         |         | 3176            |            |        |      |          |
| 2366<br>2367 |            |        |         |         | 3180            |            |        |      |          |
| 2367<br>2368 |            |        |         |         | 3182<br>3183    |            |        |      |          |
| 2372         | ISO14184-2 | 203.2  |         | -1.38   | 3185            |            |        |      |          |
| 2373         |            |        |         |         | 3190            |            |        |      |          |
| 2375         | ISO14184-1 | 230.3  |         | 0.32    | 3191            |            |        |      |          |
| 2379         | ISO14184-2 | 224.13 |         | -0.06   | 3192            |            |        |      |          |
| 2380<br>2381 |            |        |         |         | 3195<br>3197    |            |        |      |          |
| 2386         |            |        |         |         | 3199            |            |        |      |          |
| 2389         | ISO14184-2 | 262.13 |         | 2.32    | 3200            |            |        |      |          |
|              |            |        |         |         |                 |            |        |      |          |

| 2390 | ISO14184-2             | 239.91 | 0.93 | 3207 |            |     |       |
|------|------------------------|--------|------|------|------------|-----|-------|
| 2410 | ISO14184-2             | 229    | 0.24 | 3210 |            |     |       |
| 2426 |                        |        |      | 3212 |            |     |       |
| 2428 |                        |        |      | 3214 | ISO14184-2 | 233 | 0.49  |
| 2429 |                        |        |      | 3218 |            |     |       |
| 2432 |                        |        |      | 3220 |            |     |       |
| 2433 |                        |        |      | 3225 | ISO14184-2 | 213 | -0.76 |
| 2441 |                        |        |      | 3228 |            |     |       |
| 2442 |                        |        |      | 3237 |            |     |       |
| 2446 |                        |        |      | 3246 |            |     |       |
| 2452 |                        |        |      | 3248 |            |     |       |
| 2453 |                        |        |      | 8005 |            |     |       |
|      |                        |        |      |      |            |     |       |
|      | normality              | not OK |      |      |            |     |       |
|      | n                      | 15     |      |      |            |     |       |
|      | outliers               | 2      |      |      |            |     |       |
|      | mean (n)               | 225.13 |      |      |            |     |       |
|      | st.dev. (n)            | 21.704 |      |      |            |     |       |
|      | R(calc.)               | 60.77  |      |      |            |     |       |
|      | R(Gaic.)<br>R(Horwitz) | 44.63  |      |      |            |     |       |
|      | K(HUIWILZ)             | 44.03  |      |      |            |     |       |
|      |                        |        |      |      |            |     |       |

Lab 2226: test result should be free formaldehyde but was reported as released formaldehyde Lab 2256: test result should be free formaldehyde but was reported as released formaldehyde Lab 2277: test result should be free formaldehyde but was reported as released formaldehyde Lab 2538: test result should be free formaldehyde but was reported as released formaldehyde Lab 3146: test result should be free formaldehyde but was reported as released formaldehyde





# **APPENDIX 2**

# Number of participants per country

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

# **APPENDIX 3**

#### Abbreviations:

C = final result after checking of first reported suspect result

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

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

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

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