# Results of Proficiency Test
## pH and Formaldehyde
## in leather
## October 2015


Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. R.J. Starink
Correctors: dr. R. Visser & ing. C Nijssen-Wester


Report: iis15A05L

January 2016

**CONTENTS**



Appendices:

# 1 INTRODUCTION

Worldwide, many consumer products are produced from leather. During the production of leather products, many different types of auxiliary agents and dyes are used to process leather. Neither in the U.S. nor in the European Union there is general legislation that limits the presence of formaldehyde in leather. However, some individual countries have restricting limits on the concentration of free formaldehyde in leather that may vary from 20 mg/kg for leather used for young children to 100 mg/kg when the leather is in contact with the skin, 150 mg/kg for shoe uppers and 400 mg/kg for leather without permanent contact with the skin. In 2006, The China Leather Industry Standard Committee Organization established the Limit of Harmful Matters in Leather: GB20400-2006. This national mandatory standard was approved by the General Administration of P.R. of China for Quality Supervision and Inspection and Quarantine and implemented in December 2007.
Since several years, the Institute for Interlaboratory Studies (iis) organises a proficiency scheme for Formaldehyde in textile. The institute decided to organize also a proficiency test for Formaldehyde and pH in Leather in 2013. It was decided to continue this scheme as part of the proficiency testing program 2015/2016.
In this interlaboratory study, 119 laboratories in 27 different countries participated. See appendix 3 for the number of participating laboratories per country.
In this report, the results of this 2015 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 to an ISO17025 accredited laboratory. In practice it appears to be hard to find or to prepare large amounts of suitable PT material. Therefore it was decided to use in this proficiency test only one positive sample. Sample #15209 is approx. 8 grams. 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, from the FAQ page.

## 2.3 CONFIDENTIALITY STATEMENT

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

## 2.4 SAMPLES

A black leather sample was shreddered and after homogenisation divided over 140 subsamples of approx. 8 gram and labelled sample #15209.
The homogeneity of the subsamples was checked on Formaldehyde according to ISO17226-1 on 8 stratified randomly selected samples. The homogeneity testing was performed by a subcontracted ISO17025 accredited laboratory. See the following tables for the test results.

| | Formaldehyde in mg/kg |
|---|---|
| Sample #15209-1 | 68.5 |
| Sample #15209-2 | 73.7 |
| Sample #15209-3 | 84.2 |
| Sample #15209-4 | 78.3 |
| Sample #15209-5 | 80.7 |
| Sample #15209-6 | 80.5 |
| Sample #15209-7 | 81.0 |
| Sample #15209-8 | 73.9 |

Table 1: homogeneity test results of subsamples #15209

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

| | Formaldehyde in mg/kg |
|---|---|
| r | 14.4 |
| Reference test | ISO17226-1:08 |
| $0.3*R_{(reference\ test)}$ | 14.2 |

Table 2: repeatability of subsamples #15209

The calculated repeatability for sample #15209 is in good agreement with 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of all subsamples was assumed.

One sample of approx. 8 grams (labelled #15209) was sent to the participating laboratories on October 14, 2015.

## 2.5 ANALYSES

The participants were asked to determine on sample #15209, the content of Formaldehyde (HPLC), the content of Formaldehyde (colorimetric) and pH 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 as well as the required standards and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. A form to confirm receipt of the samples and a letter of instructions were added to the samples.

# 3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered via the data entry portal www.kmpd.co.uk/sgs-iis-cts/. 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 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 4; 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:

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

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
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. Three laboratories did not report any test results and twenty other laboratories reported results after the final reporting date.

Finally, the 116 reporting laboratories did send in total 239 numerical results. Observed were 7 statistical outlying results, which is 2.9% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

A number of participants reported that the amount of material was not sufficient for testing the pH and/or to perform the test in duplicate as required.

For the determination of Formaldehyde in Leather the test methods ISO17226-1 and ISO17226-2 are considered to be the official test methods. Therefore, the target reproducibilities were estimated from the reproducibility data as mentioned in the annexes of ISO17226-1 and ISO17226-2.

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.

### 4.1 EVALUATION PER TEST

In this section, the results on sample #15209 are discussed. All statistical results reported on the leather sample are summarised in appendix 1.

Formaldehyde content (HPLC): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility after rejection of suspect data is in agreement with the requirements of ISO17226-1:2008.

Formaldehyde content (colorimetric): This determination was very problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ISO17226-2:2008.

pH: This determination was very problematic. Three statistical outliers were observed and one test result was excluded from the statistical evaluation as the reported test method is for textiles only. The calculated reproducibility after rejection of the suspect data is not at all in agreement with the requirements of ASTM D2810:2013.

Regretfully, ISO4045 does not provide precision data.
Therefore, the reproducibility of ASTM D2810 was taken to estimate the target reproducibility. This appears to be very strict. In general the reproducibility of a method is three times the repeatability. However, in ASTM D2810, the repeatability is 0.03 pH units and the reproducibility is 0.06 pH units (factor of 2 instead of 3). Also the repeatability and reproducibility are based on the values of duplicate tests. Therefore in this report the reproducibility for this test is calculated by three times the repeatability times the square root of two (0.127 pH units), assuming that the sample material was not sufficient for most participants to perform the determination in duplicate.

The majority of the laboratories reported according to either ASTM D2810 or ISO4045. Both methods were also evaluated separately. The group of 16 laboratories performing ASTM D2810 showed slightly better precision than the group of 80 laboratories performing ISO4045, however still the calculated reproducibilities of both groups after rejection of the statistical outliers are not at all in agreement with the estimated requirements of ASTM D2810:2013.

### 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the calculated reproducibilities estimated from ISO17226 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 (ISO17226 and ASTM D2810), are compared in the next table.

| Parameter | unit | n | average | 2.8 * sd | R (target) |
|---|---|---|---|---|---|
| Formaldehyde (HPLC) | mg/kg | 79 | 108.1 | 69.9 | 67.6 |
| Formaldehyde (colorimetric) | mg/kg | 60 | 72.4 | 44.5 | 18.1 |
| pH | | 92 | 4.80 | 0.35 | 0.13 |

Table 3: observed reproducibilities of leather sample #15209

From the above tables it can be concluded that, without statistical calculations, the group of participating laboratories has severe difficulties with the determination of formaldehyde (colorimetric) and pH, but have no problems with the HPLC analysis, when compared with the requirements of the target test methods for this sample.

See also the discussions in paragraphs 4.1 and 6.

## 5 COMPARISON OF THE PROFICIENCY TEST OF OCTOBER 2015 WITH PREVIOUS PTS

The uncertainty in the test result of determined Formaldehyde in leather (HPLC) in the iis15A05 PT is in line with the uncertainty of the target test method. However, the uncertainty in the test result of the colorimetric determination of Formaldehyde in leather is not in line with the uncertainty of the target test method. Some improvement is visible in comparison with the results in previous PTs (see below table).

| Parameter | October 2015 | October 2014 | October 2013 | Estimated from target test method |
|---|---|---|---|---|
| Formaldehyde (HPLC) | 23% | 30% | 22% | 22% (17226-1) |
| Formaldehyde (colorimetric) | 22% | 33% | 25% | 9% (17226-2) |
| pH | 2.6% | 3.2% | n.e. | 0.9% (D2810) |

Table 4: Development of relative uncertainties over the years

## 6 DISCUSSION

The standard test method for formaldehyde content is ISO17226. Part 1 and part 2 describe the determination of the formaldehyde content by extraction of the free formaldehyde from the leather with a detergent solution. The difference between both parts of ISO17226 is the method of quantification. Quantification of the formaldehyde is done by HPLC in part 1 and by colorimetric analysis in part 2. Therefore part 2 is not selective for formaldehyde, whereas part 1 is selective. The test results from part 2 will in general be higher than the test results from part 1. Surprisingly, this is not the case with the leather sample in this PT. In the case of dispute part 1 shall be used in preference. Looking at the reproducibility statements of both methods, it is remarkable that the reproducibility of the colorimetric method is smaller than the reproducibility of the HPLC method. Maybe the precision data for the colorimetric method were obtained with samples and/or conditions that did not influence the test (as the method describes that the test could for example be influenced by absorbances from the leather colouring).

Analytical Details Colorimetric method

In this PT several analytical details were asked on the report form for test method ISO17226-2 (colorimetric). Especially about corrections for absorbances found in reagents and acetyl acetone colouring components (see Appendix 2 for the analytical details).

In total 62 participants completed this section of the report form. Regretfully, the reported details are inconsistent and therefore it was impossible to draw significant conclusions.

Sample #15209 in comparison to formaldehyde limits

When the results of this interlaboratory study were compared to the Standard “Limit of Harmful Matters in Leather” of the Chinese Leather Industry Committee Organization: GB20400-2006 (table 5), it may be noticed that not all participants would make identical decisions about the acceptability of the leather.

| GB20400 | Category A Products for babies: underclothes, bedding, etc | Category B Products with **Direct** skin contact | Category C Products Without direct skin contact |
|---|---|---|---|
| Free Formaldehyde in mg/kg | <20 | <75 | <300 |

Table 5: Summary of limits from Standard GB20400:2006

When using ISO17226 part 1, all reporting laboratories would reject this sample for category A. For category B, six laboratories would accept this sample, while all other reporting laboratories would reject this sample. None of the reporting laboratories would reject this sample for category C.
When using ISO17226 part 2, all reporting laboratories would reject this sample for category A. Twenty-four laboratories would reject this sample also for category B, while 40 other laboratories would accept this sample for category B. None of the reporting laboratories would reject this sample for category C.

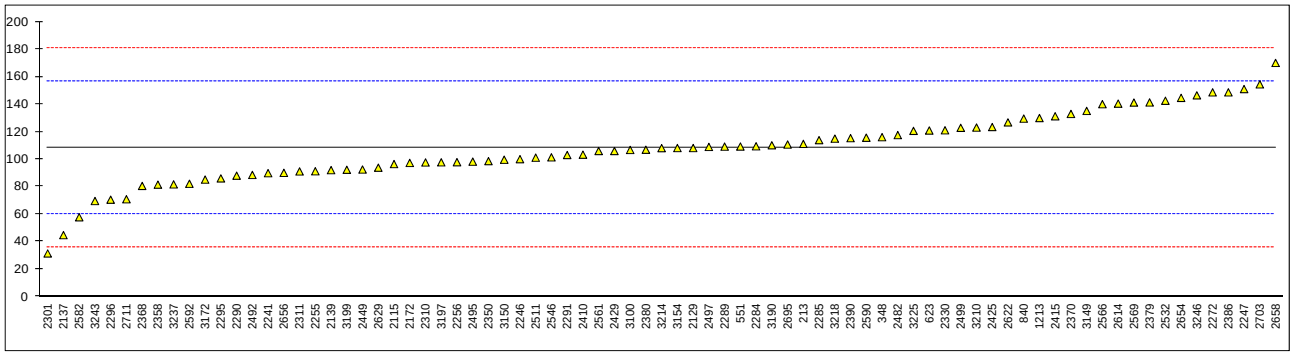
**APPENDIX 1**

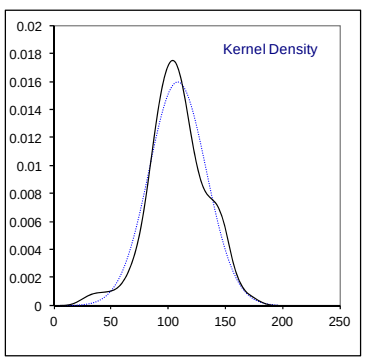
Determination of Formaldehyde content (HPLC) on sample #15209; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 110 | | ----- | | ----- | |
| 213 | ISO17226-1 | 111.1 | | 0.12 | |
| 230 | | ----- | | ----- | |
| 348 | In house | 116.0 | | 0.33 | |
| 361 | | ----- | | ----- | |
| 551 | ISO17226-1 | 109.2 | | 0.05 | |
| 622 | | ----- | | ----- | |
| 623 | ISO17226-1 | 120.85 | | 0.53 | |
| 840 | ISO17226-1 | 129.5 | | 0.89 | |
| 1213 | ISO17226-1 | 129.93 | | 0.90 | |
| 2115 | ISO17226-1 | 96.43 | | -0.48 | |
| 2129 | ISO17226-1 | 108.1 | | 0.00 | |
| 2132 | | ----- | | ----- | |
| 2137 | KS M ISO17226-1 | 44.75 | | -2.63 | |
| 2139 | ISO17226-1 | 92 | | -0.67 | |
| 2159 | | ----- | | ----- | |
| 2172 | ISO17226-1 | 97.25 | | -0.45 | |
| 2182 | | ----- | | ----- | |
| 2238 | | ----- | | ----- | |
| 2241 | ISO17226-1 | 89.8 | | -0.76 | |
| 2246 | ISO17226-1 | 100 | | -0.34 | |
| 2247 | ISO17226-1 | 151.0 | C | 1.78 | First reported 180.00 |
| 2255 | ISO17226-1 | 91.2 | | -0.70 | |
| 2256 | ISO17226-1 | 97.8 | | -0.43 | |
| 2272 | ISO17226-1 | 148.6 | | 1.68 | |
| 2284 | ISO17226-1 | 109.36 | | 0.05 | |
| 2285 | ISO17226-1 | 113.84 | | 0.24 | |
| 2289 | ISO17226-1 | 109.13 | | 0.04 | |
| 2290 | ISO17226-1 | 87.98 | | -0.83 | |
| 2291 | ISO17226-1 | 103 | | -0.21 | |
| 2292 | | ----- | | ----- | |
| 2293 | | ----- | | ----- | |
| 2295 | ISO17226-1 | 86 | | -0.92 | |
| 2296 | ISO17226-1 | 70.53 | | -1.56 | |
| 2301 | ISO17226-1 | 31.42 | | -3.18 | |
| 2310 | ISO17226-1 | 97.6 | | -0.44 | |
| 2311 | ISO17226-1 | 91.1 | | -0.70 | |
| 2330 | ISO17226-1 | 121.01 | | 0.53 | |
| 2348 | | ----- | | ----- | |
| 2350 | ISO17226-1 | 98.57 | | -0.39 | |
| 2358 | ISO17226-1 | 81.47 | | -1.10 | |
| 2367 | | ----- | | ----- | |
| 2368 | ISO17226-1 | 80.55 | | -1.14 | |
| 2370 | ISO17226-1 | 132.9 | | 1.03 | |
| 2373 | | ----- | | ----- | |
| 2379 | ISO17226-1 | 141.25 | | 1.37 | |
| 2380 | ISO17226-1 | 106.84 | | -0.05 | |
| 2381 | | ----- | | ----- | |
| 2386 | ISO17226-1 | 148.6 | | 1.68 | |
| 2389 | | ----- | | ----- | |
| 2390 | ISO17226-1 | 115.29 | | 0.30 | |
| 2403 | | ----- | | ----- | |
| 2410 | ISO17226-1 | 103.26 | | -0.20 | |
| 2415 | ISO17226-1 | 131.2 | | 0.96 | |
| 2425 | ISO17226-1 | 123.38 | | 0.63 | |
| 2426 | | ----- | | ----- | |
| 2429 | ISO17226-1 | 105.93 | | -0.09 | |
| 2446 | | ----- | | ----- | |
| 2449 | ISO17226-1 | 92.45 | | -0.65 | |
| 2453 | | ----- | | ----- | |
| 2459 | | ----- | | ----- | |
| 2460 | | ----- | | ----- | |
| 2462 | | ----- | | ----- | |
| 2477 | | ----- | | ----- | |
| 2482 | ISO17226-1 | 117.50 | | 0.39 | |
| 2488 | | ----- | | ----- | |
| 2492 | ISO17226-1 | 88.54 | | -0.81 | |
| 2495 | ISO17226-1 | 98.2 | | -0.41 | |
| 2497 | ISO17226-1 | 108.9 | | 0.03 | |
| 2499 | GB/T 19941 | 122.78 | | 0.61 | |
| 2501 | | ----- | | ----- | |
| 2511 | ISO17226-1 | 101.1 | | -0.29 | |
| 2515 | | ----- | | ----- | |
| 2519 | | ----- | | ----- | |
| 2522 | | ----- | | ----- | |
| 2532 | ISO17226-1 | 142.5 | | 1.43 | |
| 2534 | | ----- | | ----- | |

| | | | |
|---|---|---|---|
| 2546 | ISO17226-1 | 101.319 | -0.28 |
| 2561 | ISO17226-1 | 105.91 | -0.09 |
| 2563 | | ----- | ----- |
| 2566 | ISO17226-1 | 140 | 1.32 |
| 2569 | ISO17226-1 | 141.2 | 1.37 |
| 2572 | | ----- | ----- |
| 2582 | ISO17226-1 | 57.74 | -2.09 |
| 2586 | | ----- | ----- |
| 2590 | ISO17226-1 | 115.61 | 0.31 |
| 2592 | ISO17226-1 | 82.101 | -1.08 |
| 2602 | | ----- | ----- |
| 2614 | ISO17226-1 | 140.4 | 1.34 |
| 2622 | In house | 126.75 | 0.77 |
| 2629 | ISO17226-1 | 93.80 | -0.59 |
| 2654 | ISO17226-1 | 144.59 | 1.51 |
| 2656 | ISO17226-1 | 90.048294 | -0.75 |
| 2658 | ISO17226-1 | 170 | 2.57 |
| 2682 | | ----- | ----- |
| 2695 | ISO17226-1 | 110.65 | 0.11 |
| 2700 | | ----- | ----- |
| 2702 | | ----- | ----- |
| 2703 | ISO17226-1 | 154.4 | 1.92 |
| 2711 | ISO17226-1 | 70.838 | -1.54 |
| 3100 | ISO17226-1 | 106.68 | -0.06 |
| 3146 | | ----- | ----- |
| 3149 | ISO17226-1 | 135.1 | 1.12 |
| 3150 | ISO17226-1 | 99.6 | -0.35 |
| 3154 | ISO17226-1 | 108.075 | 0.00 |
| 3172 | ISO17226-1 | 85.145 | -0.95 |
| 3176 | | ----- | ----- |
| 3190 | ISO17226-1 | 110.09 | 0.08 |
| 3197 | ISO17226-1 | 97.7 | -0.43 |
| 3199 | ISO17226-1 | 92.2897 | -0.66 |
| 3210 | In house | 123 | 0.62 |
| 3214 | ISO17226-1 | 108.03 | 0.00 |
| 3218 | ISO17226-1 | 114.93 | 0.28 |
| 3220 | | ----- | ----- |
| 3225 | ISO17226-1 | 120.60 | 0.52 |
| 3237 | ISO17226-1 | 81.65 | -1.10 |
| 3243 | ISO17226-1 | 69.57 | -1.60 |
| 3246 | ISO17226-1 | 146.43 | 1.59 |
| 3248 | | ----- | ----- |

| | |
|---|---|
| normality | OK |
| n | 79 |
| outliers | 0 |
| mean (n) | 108.100 |
| st.dev. (n) | 24.9696 |
| R(calc.) | 69.915 |
| R(ISO17226-1:08) | 67.566 |



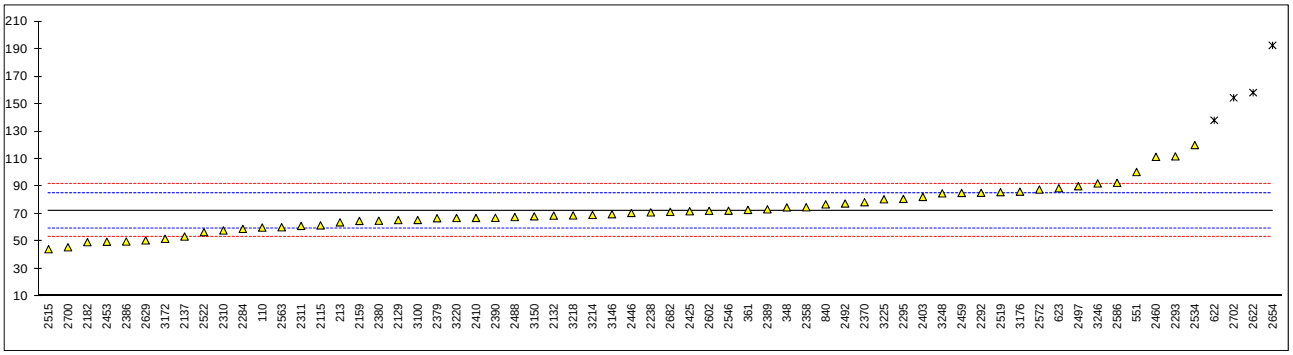



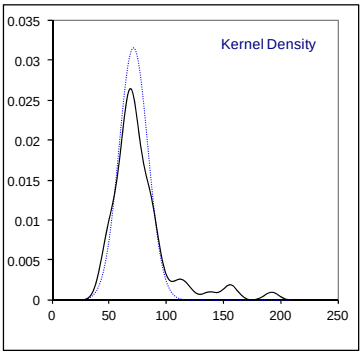
Determination of Formaldehyde content (colorimetric) on sample #15209; results in mg/kg

| lab | method | value | mark | z(targ) | Remarks |
|---|---|---|---|---|---|
| 110 | ISO17226-2 | 60.110 | C | -1.91 | First reported 218.598 |
| 213 | ISO17226-2 | 63.9 | | -1.32 | |
| 230 | | ----- | | ----- | |
| 348 | In house | 74.81 | | 0.37 | |
| 361 | ISO17226-2 | 72.99 | | 0.08 | |
| 551 | ISO17226-2 | 100.5 | | 4.34 | |
| 622 | DIN 53315A | 138.18 | R(0.01) | 10.16 | |
| 623 | ISO17226-2 | 88.72 | | 2.52 | |
| 840 | ISO17226-2 | 77 | | 0.70 | |
| 1213 | | ----- | | ----- | |
| 2115 | ISO17226-2 | 61.7 | | -1.66 | |
| 2129 | ISO17226-2 | 65.56 | | -1.06 | |
| 2132 | ISO17226-2 | 68.82 | | -0.56 | |
| 2137 | KS M ISO17226-2 | 53.60 | | -2.91 | |
| 2139 | | ----- | | ----- | |
| 2159 | ISO17226-2 | 64.96 | | -1.16 | |
| 2172 | | ----- | | ----- | |
| 2182 | ISO17226-2 | 49.5 | | -3.55 | |
| 2238 | ISO17226-2 | 71.18 | | -0.20 | |
| 2241 | | ----- | | ----- | |
| 2246 | | ----- | | ----- | |
| 2247 | | ----- | | ----- | |
| 2255 | | ----- | | ----- | |
| 2256 | | ----- | | ----- | |
| 2272 | | ----- | | ----- | |
| 2284 | ISO17226-2 | 59.15 | | -2.06 | |
| 2285 | | ----- | | ----- | |
| 2289 | | ----- | | ----- | |
| 2290 | | ----- | | ----- | |
| 2291 | | ----- | | ----- | |
| 2292 | ISO17226-2 | 85.411 | | 2.00 | |
| 2293 | ISO17226-2 | 111.885 | | 6.10 | |
| 2295 | ISO17226-2 | 81 | | 1.32 | |
| 2296 | | ----- | | ----- | |
| 2301 | | ----- | | ----- | |
| 2310 | ISO17226-2 | 58 | | -2.23 | |
| 2311 | ISO17226-2 | 61.3 | | -1.72 | |
| 2330 | | ----- | | ----- | |
| 2348 | | ----- | | ----- | |
| 2350 | | ----- | | ----- | |
| 2358 | ISO17226-2 | 74.89 | | 0.38 | |
| 2367 | | ----- | | ----- | |
| 2368 | | ----- | | ----- | |
| 2370 | ISO17226-2 | 78.57 | | 0.95 | |
| 2373 | | ----- | | ----- | |
| 2379 | ISO17226-2 | 66.93 | | -0.85 | |
| 2380 | ISO17226-2 | 65.09 | | -1.14 | |
| 2381 | | ----- | | ----- | |
| 2386 | ISO17226-2 | 50 | | -3.47 | |
| 2389 | ISO17226-2 | 73.4 | | 0.15 | |
| 2390 | ISO17226-2 | 67.21 | | -0.81 | |
| 2403 | ISO17226-2 | 82.42 | | 1.54 | |
| 2410 | ISO17226-2 | 67.13 | | -0.82 | |
| 2415 | | ----- | | ----- | |
| 2425 | ISO17226-2 | 72.00 | | -0.07 | |
| 2426 | | ----- | | ----- | |
| 2429 | | ----- | | ----- | |
| 2446 | In house | 70.74 | | -0.26 | |
| 2449 | | ----- | | ----- | |
| 2453 | ISO17226-2 | 49.81 | | -3.50 | |
| 2459 | ISO17226-2 | 85.311 | | 1.99 | |
| 2460 | ISO17226-2 | 111.57 | | 6.05 | |
| 2462 | | ----- | | ----- | |
| 2477 | | ----- | | ----- | |
| 2482 | | ----- | | ----- | |
| 2488 | ISO17226-2 | 67.803 | C | -0.72 | First reported 135.606. |
| 2492 | ISO17226-2 | 77.60 | | 0.80 | |
| 2495 | | ----- | | ----- | |
| 2497 | ISO17226-2 | 90.22 | | 2.75 | |
| 2499 | | ----- | | ----- | |
| 2501 | | ----- | | ----- | |
| 2511 | | ----- | | ----- | |
| 2515 | ISO17226-2 | 44.387 | | -4.34 | |
| 2519 | ISO17226-2 | 85.88 | | 2.08 | |
| 2522 | ISO17226-2 | 56.77 | | -2.42 | |
| 2532 | | ----- | | ----- | |
| 2534 | ISO17226-2 | 120.1 | C | 7.37 | First reported 135.1 |
| 2546 | ISO17226-2 | 72.3876 | | -0.01 | |

| | | | | | |
|---|---|---|---|---|---|
| 2561 | | ----- | | ----- | |
| 2563 | ISO17226-2 | 60.35 | | -1.87 | |
| 2566 | | ----- | | ----- | |
| 2569 | | ----- | | ----- | |
| 2572 | ISO17226-2 | 87.8 | | 2.37 | |
| 2582 | | ----- | | ----- | |
| 2586 | ISO17226-2 | 92.68 | C | 3.13 | First reported 158.81. |
| 2590 | | ----- | | ----- | |
| 2592 | | ----- | | ----- | |
| 2602 | LFBG B82.02-1 | 72.3 | | -0.02 | |
| 2614 | | ----- | | ----- | |
| 2622 | ISO17226-2 | 158.29 | R(0.01) | 13.27 | |
| 2629 | ISO17226-2 | 50.76 | | -3.35 | |
| 2654 | ISO17226-2 | 192.65 | C,R(0.01) | 18.59 | First reported 131.87 |
| 2656 | | ----- | | ----- | |
| 2658 | | ----- | | ----- | |
| 2682 | ISO17226-2 | 71.5 | | -0.15 | |
| 2695 | | ----- | | ----- | |
| 2700 | ISO17226-2 | 45.81 | | -4.12 | |
| 2702 | ISO17226-2 | 154.53 | C,R(0.01) | 12.69 | First reported 143.74 |
| 2703 | | ----- | | ----- | |
| 2711 | | ----- | | ----- | |
| 3100 | ISO17226-2 | 65.63 | | -1.05 | |
| 3146 | ISO17226-2 | 69.8 | | -0.41 | |
| 3149 | | ----- | | ----- | |
| 3150 | ISO17226-2 | 68.3 | | -0.64 | |
| 3154 | | ----- | | ----- | |
| 3172 | ISO17226-2 | 51.97 | | -3.17 | |
| 3176 | ISO17226-2 | 86.21 | | 2.13 | |
| 3190 | | ----- | | ----- | |
| 3197 | | ----- | W | ----- | Result withdrawn, reported 129.2 |
| 3199 | | ----- | | ----- | |
| 3210 | | ----- | | ----- | |
| 3214 | ISO17226-2 | 69.32 | | -0.48 | |
| 3218 | ISO17226-2 | 68.97 | | -0.54 | |
| 3220 | ISO17226-2 | 67.1 | | -0.83 | |
| 3225 | ISO17226-2 | 80.72 | | 1.28 | |
| 3237 | | ----- | | ----- | |
| 3243 | | ----- | | ----- | |
| 3246 | ISO17226-2 | 92.24 | | 3.06 | |
| 3248 | ISO17226-2 | 85 | | 1.94 | |

| | |
|---|---|
| normality | OK |
| n | 60 |
| outliers | 4 |
| mean (n) | 72.446 |
| st.dev. (n) | 15.8880 |
| R(calc.) | 44.486 |
| R(ISO17226-2:08) | 18.108 |



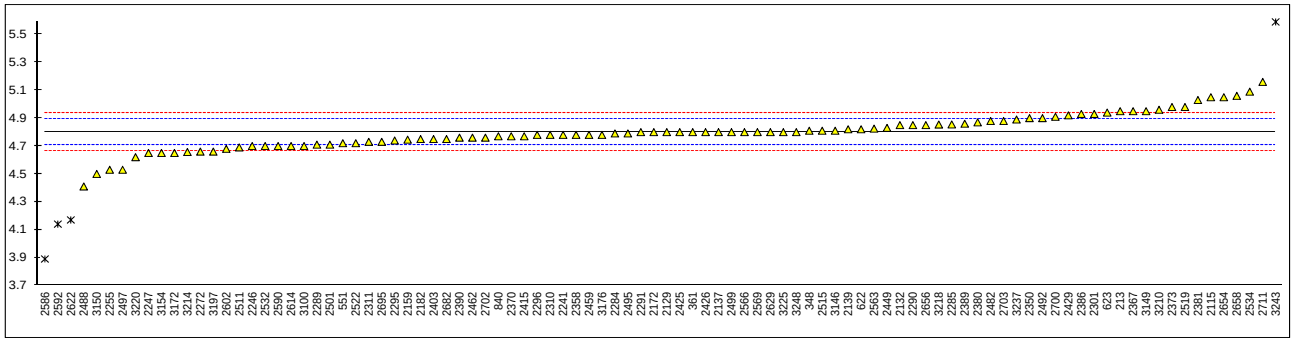
## Determination of pH on sample #15209; unitless results

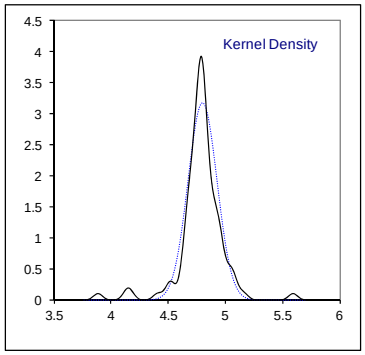
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 110 | | ----- | | ----- | |
| 213 | ISO4045 | 4.95 | | 3.28 | |
| 230 | | ----- | | ----- | |
| 348 | ISO4045 | 4.81 | | 0.20 | |
| 361 | ISO4045 | 4.80 | | -0.02 | |
| 551 | ISO4045 | 4.72 | | -1.78 | |
| 622 | ISO4045 | 4.82 | | 0.42 | |
| 623 | ISO4045 | 4.94 | | 3.06 | |
| 840 | ISO4045 | 4.77 | | -0.68 | |
| 1213 | | ----- | | ----- | |
| 2115 | ISO4045 | 5.05 | | 5.48 | |
| 2129 | ISO4045 | 4.8 | | -0.02 | |
| 2132 | ASTM D2810 | 4.85 | | 1.08 | |
| 2137 | ISO4045 | 4.80 | | -0.02 | |
| 2139 | ASTM D2810 | 4.82 | | 0.42 | |
| 2159 | ISO4045 | 4.746 | | -1.21 | |
| 2172 | ASTM D2810 | 4.80 | | -0.02 | |
| 2182 | ASTM D2810 | 4.75 | | -1.12 | |
| 2238 | | ----- | | ----- | |
| 2241 | ISO4045 | 4.78 | | -0.46 | |
| 2246 | ISO4045 | 4.7 | | -2.22 | |
| 2247 | ISO4045 | 4.65 | | -3.32 | |
| 2255 | ISO4045 | 4.53 | | -5.96 | |
| 2256 | | ----- | | ----- | |
| 2272 | ISO4045 | 4.66 | | -3.10 | |
| 2284 | ISO4045 | 4.79 | | -0.24 | |
| 2285 | ASTM D2810 | 4.855 | | 1.19 | |
| 2289 | ASTM D2810 | 4.71 | | -2.00 | |
| 2290 | ASTM D2810 | 4.85 | | 1.08 | |
| 2291 | ISO4045 | 4.8 | | -0.02 | |
| 2292 | | ----- | | ----- | |
| 2293 | | ----- | | ----- | |
| 2295 | ISO4045 | 4.74 | | -1.34 | |
| 2296 | ISO4045 | 4.78 | | -0.46 | |
| 2301 | ASTM D2810 | 4.93 | | 2.84 | |
| 2310 | ISO4045 | 4.78 | | -0.46 | |
| 2311 | ISO4045 | 4.73 | | -1.56 | |
| 2330 | | ----- | | ----- | |
| 2348 | | ----- | | ----- | |
| 2350 | ISO4045 | 4.90 | | 2.18 | |
| 2358 | ISO4045 | 4.78 | | -0.46 | |
| 2367 | ISO4045 | 4.95 | | 3.28 | Measured on undiluted sample |
| 2368 | | ----- | | ----- | |
| 2370 | ISO4045 | 4.77 | | -0.68 | |
| 2373 | ISO4045 | 4.98 | | 3.94 | Measured on undiluted sample |
| 2379 | | ----- | | ----- | |
| 2380 | ISO4045 | 4.87 | | 1.52 | |
| 2381 | ISO4045 | 5.03 | | 5.04 | |
| 2386 | ISO4045 | 4.93 | | 2.84 | |
| 2389 | ISO4045 | 4.86 | | 1.30 | |
| 2390 | ISO4045 | 4.76 | | -0.90 | |
| 2403 | ASTM D2810 | 4.75 | | -1.12 | |
| 2410 | | ----- | | ----- | |
| 2415 | ISO4045 | 4.77 | | -0.68 | |
| 2425 | ISO4045 | 4.8 | | -0.02 | |
| 2426 | ISO4045 | 4.8 | | -0.02 | |
| 2429 | ASTM D2810 | 4.92 | | 2.62 | |
| 2446 | | ----- | W | ----- | Result withdrawn, reported 6.96 |
| 2449 | ASTM D2810 | 4.83 | | 0.64 | |
| 2453 | | ----- | | ----- | |
| 2459 | ISO4045 | 4.78 | | -0.46 | |
| 2460 | | ----- | | ----- | |
| 2462 | ISO4045 | 4.76 | | -0.90 | |
| 2477 | | ----- | | ----- | |
| 2482 | ISO4045 | 4.88 | | 1.74 | |
| 2488 | ISO4045 | 4.41 | | -8.60 | |
| 2492 | ISO4045 | 4.90 | | 2.18 | |
| 2495 | ISO4045 | 4.79 | | -0.24 | |
| 2497 | ISO4045 | 4.53 | | -5.96 | |
| 2499 | ISO4045 | 4.80 | | -0.02 | |
| 2501 | ISO4045 | 4.71 | | -2.00 | |
| 2511 | ISO4045 | 4.69 | | -2.44 | |
| 2515 | ISO4045 | 4.81 | | 0.20 | |
| 2519 | ASTM D2810 | 4.98 | | 3.94 | |
| 2522 | ASTM D2810 | 4.72 | | -1.78 | |
| 2532 | ISO4045 | 4.7 | | -2.22 | |
| 2534 | ISO4045 | 5.09 | | 6.36 | |
| 2546 | | ----- | | ----- | |

| | | | | | |
|---|---|---|---|---|---|
| 2561 | | ----- | W | ----- | Result withdrawn, reported 5.52 |
| 2563 | ISO4045 | 4.825 | | 0.53 | |
| 2566 | ISO4045 | 4.8 | | -0.02 | |
| 2569 | ISO4045 | 4.8 | | -0.02 | |
| 2572 | | ----- | | ----- | |
| 2582 | | ----- | | ----- | |
| 2586 | ISO4045 | 3.89 | C,R(0.01) | -20.04 | First reported 4.36 |
| 2590 | ISO4045 | 4.70 | C | -2.22 | First reported 3.90 |
| 2592 | ISO4045 | 4.14 | C,R(0.01) | -14.54 | First reported 3.86 |
| 2602 | ISO4045 | 4.68 | | -2.66 | |
| 2614 | ISO4045 | 4.7 | | -2.22 | |
| 2622 | ISO4045 | 4.17 | R(0.01) | -13.88 | |
| 2629 | ISO4045 | 4.80 | | -0.02 | |
| 2654 | ISO4045 | 5.05 | | 5.48 | |
| 2656 | ISO4045 | 4.85 | | 1.08 | |
| 2658 | ISO4045 | 5.06 | | 5.70 | |
| 2682 | ISO4045 | 4.75 | | -1.12 | |
| 2695 | ISO4045 | 4.73 | | -1.56 | |
| 2700 | ISO4045 | 4.91 | | 2.40 | |
| 2702 | ISO4045 | 4.76 | | -0.90 | |
| 2703 | ISO4045 | 4.88 | | 1.74 | |
| 2711 | ISO4045 | 5.16 | | 7.90 | |
| 3100 | ISO4045 | 4.70 | | -2.22 | |
| 3146 | ISO4045 | 4.81 | | 0.20 | |
| 3149 | ISO4045 | 4.95 | | 3.28 | |
| 3150 | ISO4045 | 4.50 | C | -6.62 | First reported 5.91 |
| 3154 | ISO4045 | 4.65 | | -3.32 | |
| 3172 | ISO4045 | 4.65 | C | -3.32 | First reported 4.25 |
| 3176 | ISO4045 | 4.78 | | -0.46 | |
| 3190 | | ----- | | ----- | |
| 3197 | ISO4045 | 4.66 | | -3.10 | |
| 3199 | | ----- | | ----- | |
| 3210 | ISO4045 | 4.96 | | 3.50 | |
| 3214 | ISO4045 | 4.657 | | -3.17 | |
| 3218 | ASTM D2810 | 4.854 | | 1.16 | |
| 3220 | ISO4045 | 4.62 | | -3.98 | |
| 3225 | ISO4045 | 4.80 | | -0.02 | |
| 3237 | ISO4045 | 4.89 | | 1.96 | |
| 3243 | DIN EN ISO3071 | 5.590 | C, ex | 17.36 | First reported 5.965; Result excluded, test method for textile only |
| 3246 | | ----- | | ----- | |
| 3248 | ASTM D2810 | 4.8 | | -0.02 | |

| | | Only ASTM D2810 | Only ISO4045 data |
|---|---|---|---|
| normality | suspect | OK | OK |
| n | 92 | 16 | 77 |
| outliers | 3 (+1 excl) | 0 | 3 |
| mean (n) | 4.801 | 4.836 | 4.796 |
| st.dev. (n) | 0.1262 | 0.0807 | 0.1334 |
| R(calc.) | 0.353 | 0.226 | 0.374 |
| R(D2810:13)* | 0.127 | 0.127 | n.a. |

*) Calculation of R(D2810:13) see §4.1



## APPENDIX 2
## Analytical Details ISO17226-2

| lab | Reagents checked for absence of formaldehyde | If yes, please give absorbance of reagents | Tested for other compounds which cause a coloring with acetylacetone? | If yes, please give absorbance | Was the absorbance of the solution corrected? | If yes, please give absorbance of the sample solution before and after correction | Was the pH measured in a 10 times diluted extract solution as per 8.3 of ISO4045? | If yes, please give the pH of the undiluted extract solution below |
|---|---|---|---|---|---|---|---|---|
| 110 | YES | 0.0254 | YES | 0.0271 | YES | before 0.34, after 0.0633 | NO | |
| 213 | *YES?* | *not reported* | *NO?* | 0.0082 | YES | measurement done with substraction 0.0082 only | NO | |
| 230 | --- | --- | --- | --- | --- | --- | --- | --- |
| 348 | *YES?* | *not reported* | *NO?* | 0.034 (a cell of 50 mm was used) | NO | | NOT APPLICABLE | |
| 361 | YES | N/A | NO | N/A | NO | N/A | NOT APPLICABLE | N/A, the pH value is not below 4 & is not over 10 |
| 551 | *YES?* | *not reported* | *NO?* | 0.0199 | YES | before 0.2875, after 0.1396 | NOT APPLICABLE | |
| 622 | *NO?* | 0.054 | *YES?* | *not reported* | YES | before 0.331, after 0.277 | NO | |
| 623 | --- | --- | --- | --- | --- | --- | --- | --- |
| 840 | *YES?* | *not reported* | *NO?* | 0.045 | YES | before 0.476, after 0.431 | NO | |
| 1213 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2115 | NO | --- | NO | --- | NO | | NO | |
| 2129 | YES | <1 | YES | <1 | NO | | NO | |
| 2132 | *YES?* | not reported | *NO?* | 0.009 | YES | before 0.441, after 0.389 | NO | |
| 2137 | YES, automatic correction in instrument | | YES, automatic correction in instrument | | YES, automatic correction in instrument | | NO | |
| 2139 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2159 | YES | 0.0736 | YES | 0.0004 | YES | before 0.4231, after 0.3495 | NO | N/A |
| 2172 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2182 | YES | 0.3503 | YES | -0.0018 | YES | before 0.3503, after 0.2707 | NOT APPLICABLE | NA |
| 2238 | YES | 0.01 | YES | 0.002 | NO | NOT APPLICABLE | NOT APPLICABLE | NOT APPLICABLE |
| 2241 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2246 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2247 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2255 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2256 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2272 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2284 | *YES?* | *not reported* | NO | --- | NO | --- | YES | 4.79 |
| 2285 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2289 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2290 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2291 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2292 | YES | 0.05 | YES | 0.011 | YES | before 0.8505, after 0.4920 | NOT APPLICABLE | not applicable |
| 2293 | YES | 0.169 | YES | 0 | NO | | NOT APPLICABLE | |
| 2295 | YES | Dimedone absorbance autosubtracted | YES | 0.025436 | YES | Software calculates automatically | NO | |
| 2296 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2301 | --- | --- | --- | --- | --- | --- | --- | --- |

| lab | Reagents checked for absence of formaldehyde | If yes, please give absorbance of reagents | Tested for other compounds which cause a coloring with acetylacetone? | If yes, please give absorbance | Was the absorbance of the solution corrected? | If yes, please give absorbance of the sample solution before and after correction | Was the pH measured in a 10 times diluted extract solution as per 8.3 of ISO4045? | If yes, please give the pH of the undiluted extract solution below |
|---|---|---|---|---|---|---|---|---|
| 2310 | YES | Sample reading after DIMEDONE correction | YES | 0.0037 | YES | before 0.3185, after 0.3148 | NOT APPLICABLE | |
| 2311 | YES | 0.0269 | YES | 0.005 | YES | before 0.344, after 0.3171 | NOT APPLICABLE | |
| 2330 | -- | -- | -- | -- | -- | -- | -- | -- |
| 2348 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2350 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2358 | YES | 0.066 | YES | 0.001 | NO | --- | NOT APPLICABLE | |
| 2367 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2368 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2370 | NO | --- | NO | --- | NO | --- | NO | |
| 2373 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2379 | YES | 0.1375 | YES | 0.0264 | NO | --- | NOT APPLICABLE | |
| 2380 | NO | | NO | | NO | --- | NO | |
| 2381 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2386 | *YES?* | *not reported* | NO | | NO | | NO | |
| 2389 | --- | --- | --- | --- | --- | --- | --- | |
| 2390 | *YES?* | *not reported* | NO | 0.001 | NO | --- | NO | |
| 2403 | *YES?* | *not reported* | NO | 0.0157 | YES | before:0.5101, after:0.4944 | NOT APPLICABLE | |
| 2410 | YES | 0.0047 | YES | 0.001 | NO | --- | NOT APPLICABLE | |
| 2415 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2425 | *NO?* | 0.061 | *YES?* | *not reported* | YES | before 0.2586, after 0.1976 | NOT APPLICABLE | |
| 2426 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2429 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2446 | *YES?* | *not reported* | NO | 0 | NO | --- | NO | - |
| 2449 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2453 | *YES?* | 0 | *YES?* | 0 | *YES?* | 0 | NOT APPLICABLE | - |
| 2459 | NO | | NO | | NO | --- | NOT APPLICABLE | |
| 2460 | YES | 0.11 | YES | 0.019 | YES | 0.1 | NOT APPLICABLE | |
| 2462 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2477 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2482 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2488 | YES | 0.067 | YES | 0.0043 | NO | | NO | |
| 2492 | YES | 0.0248 & 0.0282 | YES | 0 | NO | | NO | |
| 2495 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2497 | YES | <0.001 | YES | <0.001 | NO | | NOT APPLICABLE | |
| 2499 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2501 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2511 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2515 | YES | 0.0331 | YES | 0.0106 | YES | before 0.3003, after 0.2368 | NOT APPLICABLE | |
| 2519 | NO | | NO | | NO | | NO | |
| 2522 | YES | 0.0002 | YES | 0.0002 | YES | before 0.3285, after 0.3281 | NOT APPLICABLE | NO |
| 2532 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2534 | *YES?* | *not reported* | *NO?* | 0.009 | NO | - | NOT APPLICABLE | - |

| lab | Reagents checked for absence of formaldehyde | If yes, please give absorbance of reagents | Tested for other compounds which cause a coloring with acetylacetone? | If yes, please give absorbance | Was the absorbance of the solution corrected? | If yes, please give absorbance of the sample solution before and after correction | Was the pH measured in a 10 times diluted extract solution as per 8.3 of ISO4045? | If yes, please give the pH of the undiluted extract solution below |
|---|---|---|---|---|---|---|---|---|
| 2546 | *YES?* | *not reported* | *NO?* | 0.0001 | YES | 0.0364 Abs blank | NOT APPLICABLE | insufficient sample |
| 2561 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2563 | *YES?* | no test | *NO?* | 0.2542 | NO | no | NO | not measured |
| 2566 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2569 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2572 | YES | 0 | YES | 0 | YES | | NOT APPLICABLE | |
| 2582 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2586 | YES | 0.032 | YES | 0.018 | YES | before 0.769, after 0.719 | NOT APPLICABLE | 4,36 |
| 2590 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2592 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2602 | NO | --- | NO | --- | NO | --- | NO | |
| 2614 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2622 | YES | 0.0997 | YES | 0.02 | YES | before 0.2917, after 0.1920 | NO | |
| 2629 | *YES?* | *not reported* | *NO?* | 0.002 | NO | --- | NO | |
| 2654 | YES | 0.023 | YES | 0.002 | NO | --- | NOT APPLICABLE | |
| 2656 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2658 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2682 | NO | --- | NO | --- | NO | --- | NOT APPLICABLE | |
| 2695 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2700 | YES | 0.0363 | YES | 0.0116 | YES | before 0.3108, after 0.2629 | NO | |
| 2702 | *YES?* | *not reported* | *NO?* | 0.002 | NO | --- | NO | |
| 2703 | --- | --- | --- | --- | --- | --- | --- | --- |
| 2711 | --- | --- | --- | --- | --- | --- | --- | --- |
| 3100 | YES | 0.03 | YES | 0 | YES | before 0.3730, after 0.3430 | NO | |
| 3146 | NO | --- | NO | --- | YES | --- | NOT APPLICABLE | |
| 3149 | --- | --- | --- | --- | --- | --- | --- | --- |
| 3150 | YES | 0.02 | YES | 0.014 | YES | before: 0.160 after: 0.126 | NO | didn't dillute |
| 3154 | --- | --- | --- | --- | --- | --- | --- | --- |
| 3172 | NO | --- | NO | --- | NO | --- | YES | |
| 3176 | YES | -0.008 | YES | -0.015 | NO | no | NO | no |
| 3190 | --- | --- | --- | --- | --- | --- | --- | --- |
| 3197 | --- | --- | --- | --- | --- | --- | --- | --- |
| 3199 | --- | --- | --- | --- | --- | --- | --- | --- |
| 3210 | --- | --- | --- | --- | --- | --- | --- | --- |
| 3214 | NO | --- | NO | --- | NO | --- | NO | |
| 3218 | *YES?* | *not reported* | *NO?* | 0.0008 | NO | --- | NOT APPLICABLE | |
| 3220 | NO | 0.705 | *YES?* | *not reported* | YES | 0.049 | NO | ---- |
| 3225 | *YES?* | *not reported* | NO | --- | NO | --- | NO | |
| 3237 | --- | --- | --- | --- | --- | --- | --- | --- |
| 3243 | --- | --- | --- | --- | --- | --- | --- | --- |
| 3246 | *YES?* | *not reported* | *NO?* | 0.0066 | YES | before 0.2541, after 0.2535 | NOT APPLICABLE | |
| 3248 | *YES?* | *not reported* | *YES?* | *not reported* | YES | | NO | |

## APPENDIX 3

### Number of participants per country

4 labs in BANGLADESH
1 lab in BRAZIL
2 labs in BULGARIA
1 lab in CAMBODIA
2 labs in FRANCE
11 labs in GERMANY
1 lab in GUATEMALA
7 labs in HONG KONG
9 labs in INDIA
3 labs in INDONESIA
13 labs in ITALY
4 labs in KOREA
1 lab in MAURITIUS
2 labs in MEXICO
2 labs in MOROCCO
22 labs in P.R. of CHINA
6 labs in PAKISTAN
1 lab in PORTUGAL
1 lab in SPAIN
1 lab in SRI LANKA
2 labs in TAIWAN R.O.C.
1 lab in THAILAND
1 lab in TUNISIA
7 labs in TURKEY
2 labs in U.S.A.
3 labs in UNITED KINGDOM
9 labs in VIETNAM

## APPENDIX 4

**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
R(0.01) = outlier in Rosner outlier test
R(0.05) = straggler in Rosner outlier test
n.a. = not applicable
n.d. = not detected
W = withdrawn

**Literature**:

1. iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, April 2014
2. Öko-Tex Standard 100; January 2014.
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.
16. Official Journal of the European Communities L133/29: May 2002.
17. Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)