

Institute for
Interlaboratory Studies

Results of Proficiency Test Formaldehyde in Leather/Footwear November 2023

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: Mrs. G.A. Oosterlaken-Buijs, BSc

Correctors: Mrs. C.M. Nijssen-Wester, BSc & Mr. R.J. Starink, BSc

Approved by: Mr. R.J. Starink, BSc

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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 leather consumer products. Laws and regulations impose some of these standards and requirements. In addition to mandatory environmental standards and requirements for leather some Ecolabelling schemes are imposing environmental requirements for leather products on a voluntary basis e.g. EU Ecolabel for footwear regulation 2016/1349/EU, Oeko-Tex® Standard, bluesign®, Chinese Standard GB20400-2006 and the American Apparel and Footwear Association.

Since 2013 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Formaldehyde in Leather/Footwear every year. During the annual proficiency testing program of 2023 it was decided to continue the proficiency test for the determination of Formaldehyde in Leather/Footwear.

In this interlaboratory study 95 laboratories in 25 countries registered for participation, see appendix 3 for the number of participants per country. In this report the results of the Formaldehyde in Leather/Footwear proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to a laboratory that has performed the tests in accordance with for ISO/IEC17043 relevant requirements of ISO/IEC17025.

It was decided to send one leather sample of approximately 6 grams labelled #23760. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for the statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through 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 batch of brown leather pieces with a detectable level of Formaldehyde was obtained from a leather supplier. The leather was grinded and after homogenization 120 small plastic bags were filled with approximately 6 grams each and labelled #23760. Each subsample was wrapped in Aluminum foil and again packed in a bag.

The homogeneity of the subsamples was checked by the determination of Formaldehyde in accordance with ISO17226-1 on 8 stratified randomly selected subsamples.

	Formaldehyde in mg/kg
sample #23760-1	130.9
sample #23760-2	133.6
sample #23760-3	134.0
sample #23760-4	134.2
sample #23760-5	133.3
sample #23760-6	136.4
sample #23760-7	139.5
sample #23760-8	134.5

Table 1: homogeneity test results of subsamples #23760

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2, in the next table.

	Formaldehyde in mg/kg
r (observed)	7.0
reference test method	ISO17226-1:21
0.3 x R (reference test method)	24.9

Table 2: evaluation of the repeatability of subsamples #23760

The calculated repeatability is in agreement with 0.3 times the estimated reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one leather sample labelled #23760 was sent on October 25, 2023.

2.5 ANALYZES

The participants were requested to determine the Formaldehyde content with a HPLC method and/or a colorimetric method.

To ensure homogeneity it was requested not to use less than 0.5 gram per determination, and not to age or dry the sample. It was also requested to report if the laboratory was accredited for the determined component and to report some analytical details.

It was explicitly requested to treat the sample as if it was a routine sample and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test 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 reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).

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

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. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by $D(0.01)$ for the Dixon's test, by $G(0.01)$ or $DG(0.01)$ for the Grubbs' test and by $R(0.01)$ for the Rosner's test. Stragglers are marked by $D(0.05)$ for the Dixon's test, by $G(0.05)$ or $DG(0.05)$ for the Grubbs' test and by $R(0.05)$ for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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. In this PT the criterion of ISO13528, paragraph 9.2.1, was met for all evaluated tests. Therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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

3.2 GRAPHICS

In order to visualize 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 test results are plotted. The corresponding laboratory numbers are on 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 reference test method. 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. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

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 (derived from e.g. ISO or ASTM test methods), the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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

The z-scores were calculated according to:

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

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

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

In this proficiency test no problems were encountered with the dispatch of the samples. Two participants reported test results after the final reporting date and two other participants did not report any test results. Not all participants were able to report all tests requested. In total 93 participants reported 141 numerical test results. Observed were 10 outlying test results, which is 7.1%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Both data sets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER TEST

In this section the reported test results are discussed per test. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 4.

Test methods ISO17226-1 and ISO17226-2 are considered to be the official test methods for the determination of Formaldehyde in Leather/Footwear. In 2021 a new version of ISO17226-1 was published in which the execution of the test is different from earlier versions. In the 2021 version the given standard deviations do not show a strong dependency on the concentration as was the case in the older versions of the method. Therefore, every standard deviation in Annex A of ISO17226-1:21 was divided by the concentration and averaged. The calculated average RSD is 22%. To calculate the reproducibility of ISO17226-1:21 this relative standard deviation was multiplied by the concentration and then multiplied by 2.8.

Formaldehyde content (HPLC): The group of participants met the target requirements. Six statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated requirements of ISO17226-1:21.

Formaldehyde content (Colorimetric): The group of participants met the target requirements. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO17226-2:18.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from reference methods are presented in the next table.

Component	unit	n	average	2.8 * sd	R(lit)
Formaldehyde (HPLC)	mg/kg	76	85.8	22.2	52.9
Formaldehyde (Colorimetric)	mg/kg	55	82.1	19.9	20.2

Table 3: reproducibilities of tests on sample #23760

Without further statistical calculations it can be concluded that for both tests there is a good compliance of the group of participants with the reference test methods.

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2023 WITH PREVIOUS PTS

	November 2023	November 2022	November 2021	November 2020	November 2019
Number of reporting laboratories	93	92	97	106	136
Number of test results	141	140	140	356	441
Number of statistical outliers	10	0	14	14	17
Percentage of statistical outliers	7.1%	0.0%	10%	3.9%	3.9%

Table 4: comparison with previous proficiency tests

In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency test was compared to uncertainties observed in PTs over the years, expressed as relative standard deviation (RSD) of the PTs, see next table.

Component	November 2023	November 2022	November 2021	November 2020	2013-2019
Formaldehyde (HPLC)	9%	19%	14%	12%	9-30%
Formaldehyde (Colorimetric)	9%	18%	8%	8%	8-39%

Table 5: development of the uncertainties over the years

The uncertainties observed in this PT are comparable to the uncertainties observed in previous PTs.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

For this PT some analytical details were requested which are listed in appendix 3. Based on the answers given by the participants the following can be summarized:

- A majority (89%) of the participants mentioned that they are ISO/IEC17025 accredited to determine the reported component.
- The sample intake varied from 0.5 grams to 6 grams. About 21% of the participants used a sample intake of 1 gram and about 67% used a sample intake 2 grams.

The calculated reproducibilities are in agreement with the requirements of the corresponding target reproducibility, therefore no separate statistical analysis has been performed.

5 DISCUSSION

Limits for Formaldehyde in leather are specified in several standards e.g. EU Ecolabel footwear regulation 2016/1349/EU, Oeko-Tex® Leather Standard, bluesign® and the Chinese Standard GB20400-2006. When the results of this interlaboratory study were compared to these limits (see table 6), it was noticed that not all participants would make identical decisions about the acceptability of the leather.

Standard Limits	Formaldehyde in mg/kg
Eco-label 2016/1349/EU, linings and socks leather	75
OEKO-TEX® Leather, direct skin contact	75
bluesign®, next to skin use leather	15
GB20400-2006, direct skin contact leather	75

Table 6: summary of limits of some standards

Most of the participants would have rejected the sample. Based on the standard used, some participants would have accepted the sample.

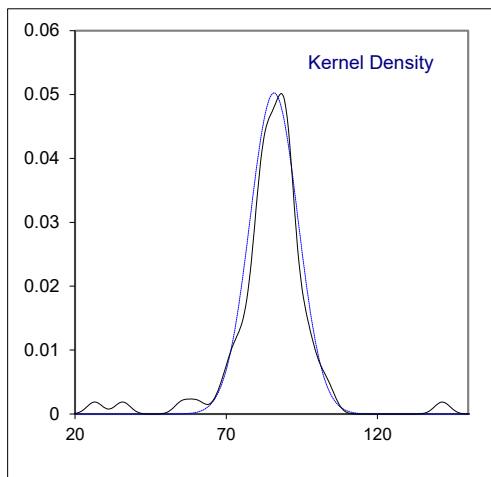
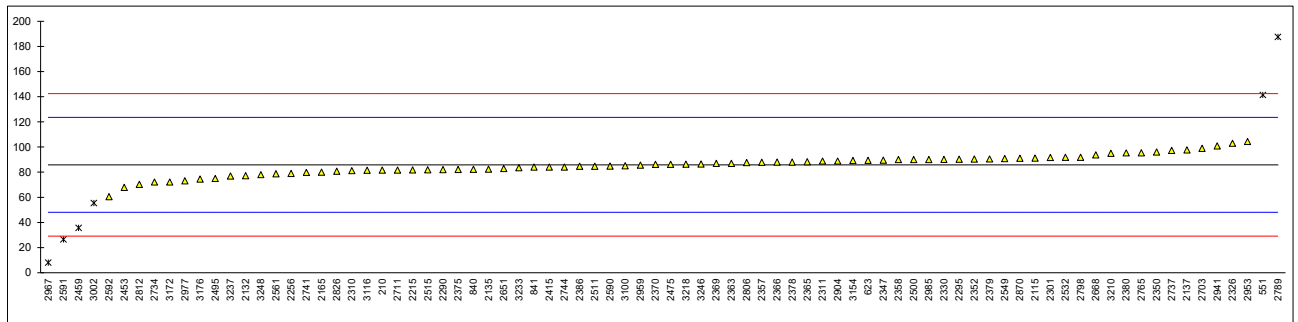
6 CONCLUSION

Although it can be concluded that most of the participants have no problem with the determination on Formaldehyde in this PT, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. 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 Formaldehyde content (HPLC) on sample #23760; results in mg/kg**

lab	method	value	mark	z(targ)	remarks
210	ISO17226-1:2021	81.71		-0.22	
362		----		----	
551	In house	141.4526	C,R(0.01)	2.95	first reported 28.29052
623	ISO17226-1:2008	89.48		0.19	
840	ISO17226-1:2021	82.31		-0.19	
841	ISO17226-1:2021	84		-0.10	
2115	ISO17226-1:2021	91.2	C	0.29	first reported 160.66
2132	ISO17226-1:2021	77.2		-0.46	
2135	ISO17226-1:2021	82.35		-0.18	
2137	ISO17226-1:2018	97.74		0.63	
2165	ISO17226-1:2021	80.1		-0.30	
2215	ISO17226-1:2021	81.78		-0.21	
2256	ISO17226-1:2021	79.01		-0.36	
2265		----		----	
2290	ISO17226-1:2021	82.1		-0.20	
2293		----		----	
2295	ISO17226-1:2021	90.31		0.24	
2300		----		----	
2301	ISO17226-1:2018	91.67		0.31	
2310	ISO17226-1:2021	81.2		-0.24	
2311	ISO17226-1:2021	88.871		0.16	
2326	ISO17226-1:2021	102.930		0.91	
2330	GB/T19941.1	90.17	C	0.23	first reported 128.96
2347	ISO17226-1:2021	89.6		0.20	
2350	ISO17226-1:2021	96.04		0.54	
2352	ISO17226-1:2021	90.4		0.24	
2357	ISO17226-1:2021	87.9		0.11	
2358	ISO17226-1:2021	90		0.22	
2363	ISO17226-1:2021	87		0.06	
2365	ISO17226-1:2021	88.30		0.13	
2366	ISO17226-1:2021	88		0.12	
2369	ISO17226-1:2021	87		0.06	
2370	ISO17226-1:2021	86.27		0.02	
2372		----		----	
2375	ISO17226-1:2021	82.2		-0.19	
2378	ISO17226-1:2021	88		0.12	
2379	ISO17226-1:2021	90.52		0.25	
2380	ISO17226-1:2021	95.30		0.50	
2386	ISO17226-1:2021	84.7		-0.06	
2410		----		----	
2415	ISO17226-1:2021	84.0		-0.10	
2453	ISO17226-1:2021	67.89		-0.95	
2459	ISO17226-1	35.73	R(0.01)	-2.65	
2460		----		----	
2475	ISO17226-1:2021	86.33		0.03	
2495	ISO17226-1:2021	75.02		-0.57	
2500	ISO17226-1:2021	90		0.22	
2511	ISO17226-1:2021	84.75		-0.06	
2515	ISO17226-1:2021	81.98		-0.20	
2532	ISO17226-1:2021	91.8		0.32	
2549	ISO17226-1:2021	90.84		0.27	
2561	ISO17226-1:2021	78.72		-0.38	
2590	ISO17226-1:2018	84.882		-0.05	
2591	ISO17226-1:2021	26.53	R(0.01)	-3.14	
2592	ISO17226-1:2021	60.53		-1.34	
2639		----		----	
2643		----		----	
2651	ISO17226-1:2021	83.01		-0.15	
2668	ISO17226-1:2021	93.84		0.42	
2703	ISO17226-1:2021	99.0		0.70	
2711	ISO17226-1:2021	81.75		-0.22	
2734	ISO17226-1:2021	72.19		-0.72	
2737	ISO17226-1:2021	97.364		0.61	
2741	ISO17226-1:2021	79.8		-0.32	
2744	ISO17226-1:2021	84.00		-0.10	
2765	ISO17226-1:2021	95.44		0.51	
2789	ISO17226-1:2021	187.5	R(0.01)	5.39	
2798	ISO17226-1:2021	91.8		0.32	
2806	ISO17226-1:2021	87.7		0.10	
2812	ISO17226-1:2021	70.30		-0.82	
2826	ISO17226-1:2021	80.8		-0.27	
2870	ISO17226-1:2021	91.1		0.28	
2904	ISO17226-1:2021	88.88		0.16	
2941	ISO17226-1:2021	100.865		0.80	
2952		----		----	

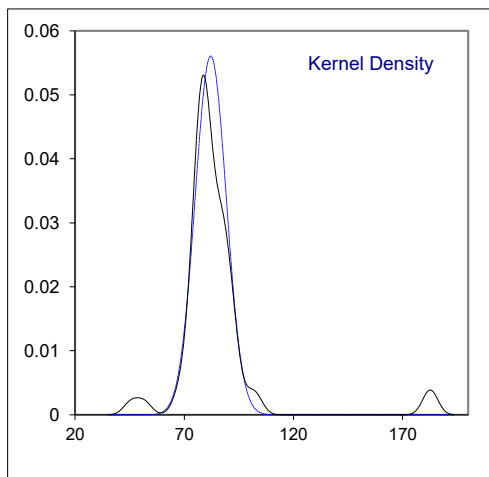
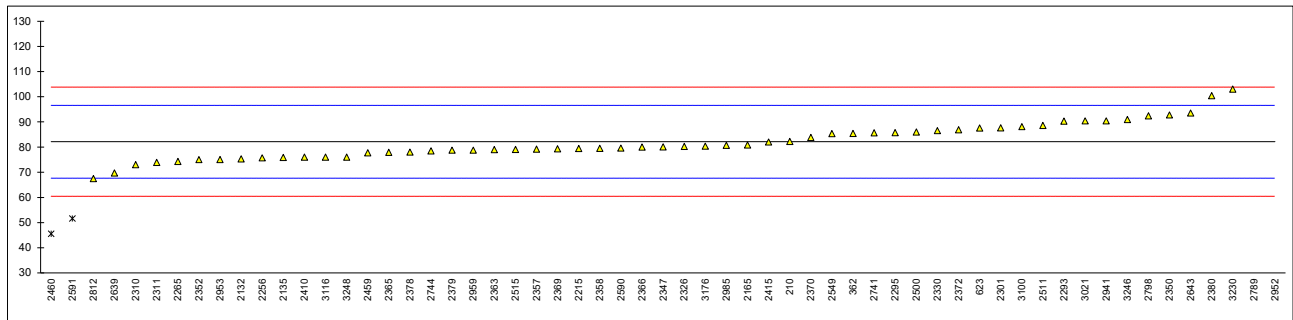
lab	method	value	mark	z(targ)	remarks
2953	ISO17226-1:2018	104.38		0.98	
2959	ISO17226-1:2021	85.6		-0.01	
2967	ISO17226-1:2021	8.13	R(0.01)	-4.11	
2977	ISO17226-1:2021	73.10		-0.67	
2985	ISO17226-1:2021	90.02		0.22	
2989		-----		-----	
3002	ISO17226-1:2021	55.5	R(0.05)	-1.61	
3021		-----		-----	
3100	ISO17226-1:2021	85.093		-0.04	
3116	ISO17226-1:2021	81.6		-0.22	
3154	ISO17226-1	89.34		0.19	
3172	ISO17226-1:2021	72.198		-0.72	
3176	ISO17226-1:2021	74.5		-0.60	
3210	In house	95.02		0.49	
3218	ISO17226-1:2021	86.37		0.03	
3230		-----		-----	
3233	ISO17226-1:2021	83.57		-0.12	
3237	ISO17226-1:2021	77.0		-0.47	
3246	ISO17226-1:2021	86.497		0.04	
3248	ISO17226-1:2021	78		-0.41	
normality		OK			
n		76			
outliers		6			
mean (n)		85.819			
st.dev. (n)		7.9356	RSD = 9%		
R(calc.)		22.220			
st.dev.(ISO17226-1:21)		18.8801			
R(ISO17226-1:21)		52.864			



Determination of Formaldehyde content (Colorimetric) on sample #23760; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	ISO17226-2	82.23		0.01	
362	ISO17226-2	85.45		0.46	
551		----		----	
623	ISO17226-2	87.6		0.76	
840		----		----	
841		----		----	
2115		----		----	
2132	ISO17226-2	75.3		-0.94	
2135	ISO17226-2	75.90	C	-0.86	first reported 52.06
2137		----		----	
2165	ISO17226-2	80.866		-0.17	
2215	ISO17226-2	79.44		-0.37	
2256	ISO17226-2	75.73		-0.88	
2265	ISO17226-2	74.29		-1.08	
2290		----		----	
2293	ISO17226-2	90.30	C	1.13	first reported 40.078
2295	ISO17226-2	85.75		0.50	
2300		----		----	
2301	ISO17226-2	87.68		0.77	
2310	ISO17226-2	73		-1.26	
2311	ISO17226-2	73.92		-1.14	
2326	ISO17226-2	80.239		-0.26	
2330	GB/T19941.1	86.54		0.61	
2347	GB/T19941.2	80.1		-0.28	
2350	ISO17226-2	92.77		1.47	
2352	GB/T19941.2	75.0		-0.99	
2357	ISO17226-2	79.2		-0.40	
2358	ISO17226-2	79.49		-0.36	
2363	ISO17226-2	79		-0.43	
2365	ISO17226-2	77.88		-0.59	
2366	ISO17226-2	80		-0.29	
2369	ISO17226-2	79.29		-0.39	
2370	ISO17226-2	83.83		0.24	
2372	ISO17226-2	86.9		0.66	
2375	ISO17226-2	N/A		----	
2378	ISO17226-2	78		-0.57	
2379	ISO17226-2	78.78		-0.46	
2380	ISO17226-2	100.40		2.53	
2386		----		----	
2410	ISO17226-2	76		-0.85	
2415	ISO17226-2	82.0		-0.02	
2453		----		----	
2459	ISO17226-2	77.74		-0.61	
2460	ISO17226-2	45.6	C,R(0.01)	-5.05	first reported 41.46
2475		----		----	
2495		----		----	
2500	ISO17226-2	86		0.54	
2511	ISO17226-2	88.61		0.90	
2515	ISO17226-2	79.1		-0.42	
2532		----		----	
2549	ISO17226-2	85.39		0.45	
2561		----		----	
2590	ISO17226-2	79.616		-0.35	
2591	ISO17226-2	51.64	R(0.01)	-4.22	
2592		----		----	
2639	GB/T19941.2	69.73		-1.71	
2643	ISO17226-2	93.55		1.58	
2651		----		----	
2668		----		----	
2703		----		----	
2711		----		----	
2734		----		----	
2737		----		----	
2741	ISO17226-2	85.66		0.49	
2744	ISO17226-2	78.50		-0.50	
2765		----		----	
2789	ISO17226-2	182.2	R(0.01)	13.85	
2798	ISO17226-2	92.4		1.42	
2806		----		----	
2812	ISO17226-2	67.5		-2.02	
2826		----		----	
2870		----		----	
2904		----		----	
2941	ISO17226-2	90.45		1.15	
2952	ISO17226-2	183.13	C,R(0.01)	13.97	first reported 160.49

lab	method	value	mark	z(targ)	remarks
2953	ISO17226-2	75.11		-0.97	
2959	ISO17226-2	78.8		-0.46	
2967		----		----	
2977		----		----	
2985	ISO17226-2	80.75		-0.19	
2989		----		----	
3002		----		----	
3021	ISO17226-2	90.44		1.15	
3100	ISO17226-2	88.127		0.83	
3116	ISO17226-2	76.0		-0.85	
3154		----		----	
3172		----		----	
3176	ISO17226-2	80.4		-0.24	
3210		----		----	
3218		----		----	
3230	ISO17226-2	103.09		2.90	
3233		----		----	
3237		----		----	
3246	ISO17226-2	90.98		1.23	
3248	ISO17226-2	76		-0.85	
normality		OK			
n		55			
outliers		4			
mean (n)		82.124			
st.dev. (n)		7.1175	RSD = 9%		
R(calc.)		19.929			
st.dev.(ISO17226-2:18)		7.2278			
R(ISO17226-2:18)		20.238			



APPENDIX 2 Analytical details

ISO/IEC17025			ISO/IEC17025		
lab	accredited	Sample intake (in grams)	lab	accredited	Sample intake (in grams)
210	Yes	2g	2515	Yes	2 grams
362	Yes	2g	2532	Yes	2 grams
551	Yes	2.0002g	2549	Yes	2 grams
623	Yes	2 grams	2561	No	2g
840	Yes	2g	2590	Yes	1g
841	Yes	2 grams	2591	Yes	2.0 grams
2115	Yes	0.7 g	2592	Yes	2.0100
2132	Yes	2g	2639	No	approximately 4 grams.
2135	Yes	2g	2643	Yes	2.000
2137	Yes	2	2651	Yes	
2165	Yes	2.0g	2668	Yes	0.5079
2215	Yes	1.0012g	2703	No	2
2256	Yes		2711	No	2.026
2265	Yes	2	2734	Yes	2g
2290	Yes		2737	Yes	1g
2293	Yes	2.0 grams	2741	Yes	2g
2295	Yes	2 grams	2744	Yes	2 g
2300	---		2765	Yes	2 g
2301	Yes	2 grams	2789	Yes	2
2310	Yes	2	2798	Yes	2g
2311	Yes	2	2806	Yes	2
2326	Yes	2.0060	2812	Yes	2.0
2330	Yes	2 grams	2826	Yes	0.5
2347	Yes		2870	Yes	2 gm
2350	Yes	2 g	2904	No	2 g x 2 replicates
2352	Yes	UV:2g HPLC:1g	2941	Yes	4 g
2357	Yes		2952	Yes	2.0004
2358	Yes	2.0	2953	Yes	1g
2363	Yes	2g	2959	Yes	1g
2365	Yes	2g	2967	No	2
2366	Yes	2g	2977	Yes	1g
2369	Yes		2985	Yes	6g
2370	Yes	2 g	2989	---	
2372	Yes	2g 50mL	3002	Yes	1g
2375	Yes	1 gram	3021	Yes	1.8174
2378	Yes	1g	3100	Yes	2g
2379	Yes	1 gram	3116	Yes	2
2380	Yes	2.0 g	3154	Yes	1 g
2386	Yes	For ISO 17226-1:2021: 1g	3172	Yes	
2410	Yes	1 g	3176	Yes	1
2415	Yes	1 gram	3210	Yes	2
2453	No	±1g	3218	Yes	2g
2459	Yes	2.0 grams	3230	Yes	4grams (2 x 2grams)
2460	Yes	2 g	3233	No	2g
2475	No	1g	3237	Yes	2g
2495	Yes	0.5	3246	Yes	2g
2500	No		3248	Yes	1
2511	Yes	4g			

APPENDIX 3

Number of participants per country

1 lab in BANGLADESH
1 lab in BRAZIL
2 labs in BULGARIA
1 lab in CAMBODIA
4 labs in FRANCE
4 labs in GERMANY
1 lab in GUATEMALA
5 labs in HONG KONG
7 labs in INDIA
2 labs in INDONESIA
13 labs in ITALY
4 labs in KOREA, Republic of
1 lab in MAURITIUS
2 labs in MEXICO
2 labs in MOROCCO
19 labs in P.R. of CHINA
4 labs in PAKISTAN
1 lab in PORTUGAL
2 labs in SPAIN
2 labs in TAIWAN
1 lab in THAILAND
1 lab in TUNISIA
6 labs in TURKEY
2 labs in UNITED KINGDOM
7 labs in VIETNAM

APPENDIX 4

Abbreviations

C	= final test result after checking of first reported suspect test 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's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?

Literature

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Address: Malledijk 18, P.O. Box 200, 3200 AE Spijkenisse, The Netherlands
Telephone number: +31 (0)88 214 45 41
Email address: nl.iis@sgs.com
Website: www.iisnl.com

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