Institute for
Interlaboratory Studies

Results of Proficiency Test
Formaldehyde in Leather/Footwear
November 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

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 2022/2023 it was decided to continue the proficiency test for the determination of Formaldehyde in Leather/Footwear.

In this interlaboratory study 94 laboratories in 29 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 an ISO/IEC17025 accredited laboratory.
It was decided to send one leather sample of approximately 6 grams labelled #22765.
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 shredded and after homogenization 120 small plastic bags were filled with approximately 6 grams each and labelled #22765. 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 #22765-1	31
sample #22765-2	30
sample #22765-3	31
sample #22765-4	31
sample #22765-5	32
sample #22765-6	31
sample #22765-7	28
sample #22765-8	31

Table 1: homogeneity test results of subsamples #22765

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)	3.3
reference test method	ISO17226-1:21
0.3 x R (reference test method)	5.7

Table 2: evaluation of the repeatability of subsamples #22765

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 #22765 was sent on October 19, 2022.

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 test methods), the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of 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:

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

The $z_{(target)}$ 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 some problems were encountered with the dispatch of the samples. Six 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 92 participants reported 140 numerical test results. There were no outlying test results observed. 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. Therefore, the target reproducibilities were estimated from the reproducibility data as mentioned in the annexes of ISO17226-1 and ISO17226-2.

In 2021 a new version of ISO17226-1 was published in which the execution of the test is different from earlier versions. The precision data for ISO17226-1 have been updated in the 2021 version. The precision data given in the 2021 version the given standard deviations do not show a strong dependency to the concentration but is not entirely neglectable. Therefore, it was decided that the given standard deviations mentioned in Annex A of ISO17226-1:21 was divided by the corresponding Formaldehyde concentration to give a relative standard deviation (RSD). The average RSD is 22% and multiplied by 2.8 to obtain an estimation for the reproducibility of ISO17226-1:21.

In 2021 iis PT it was observed that the test results of ISO17226-1 from the older versions were biased to the 2021 test results. This year most participants reported to have used the latest version of ISO17226-1 and no separate groups appeared to be present in the test results. Therefore, it was decided to evaluate the data as one group.

Formaldehyde content (HPLC): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated requirements of ISO17226-1:21.

Formaldehyde content (Colorimetric): This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated 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 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	80	26.7	14.0	16.5
Formaldehyde (Colorimetric)	mg/kg	60	24.7	12.7	7.6

Table 3: reproducibilities of tests on sample #22765

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2022 WITH PREVIOUS PTS

	November 2022	November 2021	November 2020	November 2019	November 2018
Number of reporting laboratories	92	97	106	136	114
Number of test results	140	140	356	441	396
Number of statistical outliers	0	14	14	17	12
Percentage of statistical outliers	0.0%	10%	3.9%	3.9%	3.0%

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 2022	November 2021	November 2020	2013-2019	Target
Formaldehyde (HPLC)	19%	14%	12%	9-30%	22%
Formaldehyde (Colorimetric)	18%	8%	8%	8-39%	11-22%

Table 5: development of the uncertainties over the years

The uncertainties for the 2022 PT meet the estimated targets from the reference test methods. The reproducibility in the determination with colorimetric method is much higher compared to last years.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

For this PT some analytical details were requested which are listed in appendix 2. Based on the answers given by the participants it can be summarized that about 80% of the participants mentioned that they are ISO/IEC17025 accredited to determine the reported component(s). And approximately 70% used 2 grams as sample intake as prescribed in ISO17226.
As the majority of the group follow the same analytical procedures no separate statistical analysis has been performed.

5 DISCUSSION

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 and Oeko-Tex® Standard 100 (see table 6), it was noticed that not all participants would make identical decisions about the acceptability of the leather, see next table.

	Category A babies: underclothes, bedding, etc	Category B with direct skin contact	Category C without direct skin contact
Formaldehyde in mg/kg	<20	<75	<300

Table 6: summary of limits from Standard GB20400:2006 and Oeko-Tex® 100

When comparing the test results to the limits mentioned in table 6, seventy-two of the eighty reporting laboratories would have rejected the sample for category A based on the HPLC method. Based on the Colorimetric method forty-eight of the sixty reporting laboratories would have rejected the sample for category A.
All participants would have accepted this sample for category B and C.

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.

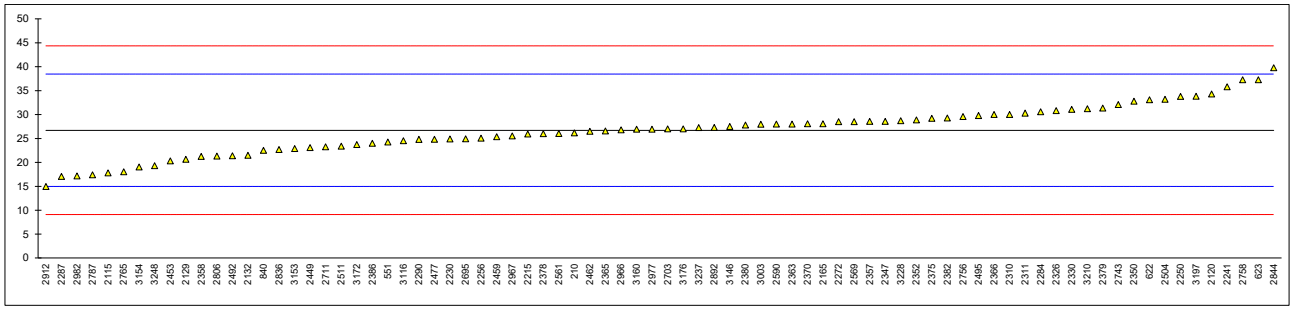
APPENDIX 1

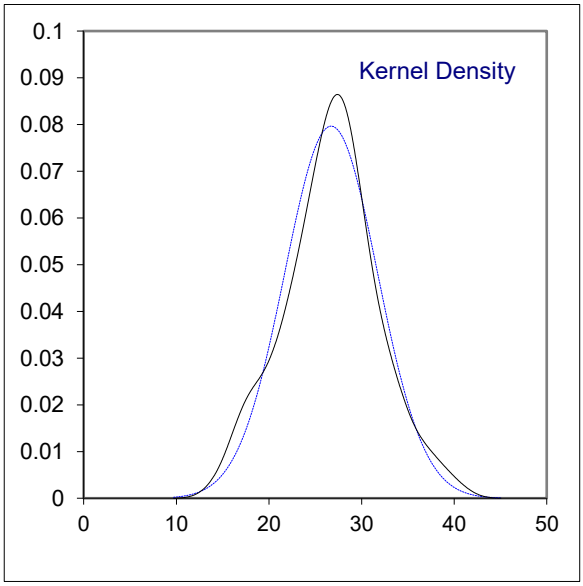
Determination of Formaldehyde content (HPLC) on sample #22765; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	ISO17226-1:2021	26.18		-0.09	
362		-----		-----	
523		Not determined		-----	
551	ISO17226-1	24.26		-0.42	
622	ISO17226-1:2021	33.09		1.09	
623	ISO17226-1:2021	37.29		1.80	
840	ISO17226-1:2021	22.5		-0.72	
2115	ISO17226-1:2021	17.79		-1.52	
2120	ISO17226-1:2021	34.3		1.29	
2129	ISO17226-1:2018	20.66		-1.03	
2132	ISO17226-1:2021	21.4746		-0.89	
2165	ISO17226-1:2021	28.1		0.24	
2215	ISO17226-1:2018	25.94		-0.13	
2230	ISO17226-1:2021	24.9		-0.31	
2241	ISO17226-1:2021	35.81		1.55	
2250	ISO17226-1:2021	33.8		1.21	
2256	ISO17226-1:2021	25.04		-0.28	
2265		-----		-----	
2272	ISO17226-1:2021	28.5		0.30	
2284	ISO17226-1:2021	30.6		0.66	
2287	ISO17226-1:2021	17.05		-1.64	
2290	ISO17226-1:2021	24.8		-0.33	
2310	ISO17226-1:2021	30		0.56	
2311	ISO17226-1:2021	30.3		0.61	
2326	ISO17226-1:2021	30.84		0.70	
2330	ISO17226-1:2021	31.10		0.75	
2347	ISO17226-1:2021	28.6		0.32	
2350	ISO17226-1:2021	32.78		1.03	
2352	ISO17226-1:2021	28.88		0.37	
2357	ISO17226-1	28.6		0.32	
2358	ISO17226-1:2021	21.253		-0.93	
2363	ISO17226-1:2021	28		0.22	
2365	ISO17226-1:2021	26.57		-0.02	
2366	ISO17226-1:2018	30		0.56	
2370	ISO17226-1:2021	28.08		0.23	
2372		-----		-----	
2373		not applicable		-----	
2375	ISO17226-1:2021	29.2		0.42	
2378	ISO17226-1:2021	26.0		-0.12	
2379	ISO17226-1:2021	31.37		0.79	
2380	ISO17226-1:2021	27.81		0.19	
2381		-----		-----	
2382	ISO17226-1:2021	29.27		0.44	
2386	ISO17226-1:2021	24.0		-0.46	
2449	ISO17266-1	23.1		-0.61	
2453	ISO17226-1:2008	20.3		-1.09	
2459	ISO17226-1:2021	25.38		-0.23	
2460		-----		-----	
2462	ISO17226-1:2021	26.5		-0.04	
2477	ISO17226-1:2021	24.8559		-0.32	
2492	ISO17226-1:2021	21.4		-0.90	
2495	ISO17226-1:2021	29.79		0.52	
2504	ISO17226-1:2021	33.2020		1.10	
2511	ISO17226-1	23.4		-0.56	
2561	ISO17226-1:2021	26.01		-0.12	
2569	ISO17226-1:2021	28.5		0.30	
2582		-----		-----	
2590	ISO17226-1:2021	27.992		0.22	
2639		-----		-----	
2643		-----		-----	
2649		-----		-----	
2695	ISO17226-1:2021	24.92		-0.31	
2703	ISO17226-1:2018	27.0		0.05	
2711	ISO17226-1:2021	23.25		-0.59	
2743	ISO17226-1:2021	32.11		0.92	
2756	ISO17226-1:2017	29.58		0.49	
2758	ISO17226-1:2021	37.27		1.80	
2765	ISO17226-1:2021	18.03		-1.48	
2787	ISO17226-1:2021	17.419		-1.58	
2806	ISO17226-1:2021	21.3		-0.92	
2836	ISO17226-1:2021	22.69		-0.68	
2844	ISO17226-1:2021	39.80		2.23	
2892	ISO17226-1:2021	27.31		0.10	
2912	ISO17226-1:2021	14.95		-2.00	

lab	method	value	mark	z(targ)	remarks
2966	ISO17226-1:2021	26.8		0.01	
2967	ISO17226-1:2021	25.52		-0.20	
2977	ISO17226-1:2021	26.93		0.04	
2982	ISO17226-1:2021	17.18		-1.62	
2989		-----		-----	
3003	ISO17226-1:2021	27.96		0.21	
3116	ISO17226-1:2021	24.53		-0.37	
3146	ISO17226-1:2021	27.5		0.13	
3153	ISO17226-1:2008	22.88		-0.65	
3154	ISO17226-1:2021	19.025		-1.31	
3160	ISO17226-1:2021	26.925		0.04	
3172	ISO17226-1:2021	23.707		-0.51	
3176	ISO17226-1:2021	27.00		0.05	
3197	ISO17226-1:2021	33.85		1.21	
3210	ISO17226-1:2021	31.23		0.77	
3216		Not analyzed		-----	
3228	ISO17226-1:2021	28.7		0.34	
3230		-----		-----	
3237	ISO17226-1:2021	27.25		0.09	
3248	GB/T19941	19.28		-1.26	

normality	OK	
n	80	
outliers	0	
mean (n)	26.713	
st.dev. (n)	5.0076	RSD = 19%
R(calc.)	14.021	
st.dev.(ISO17226-1:21)	5.8769	
R(ISO17226-1:21)	16.455	



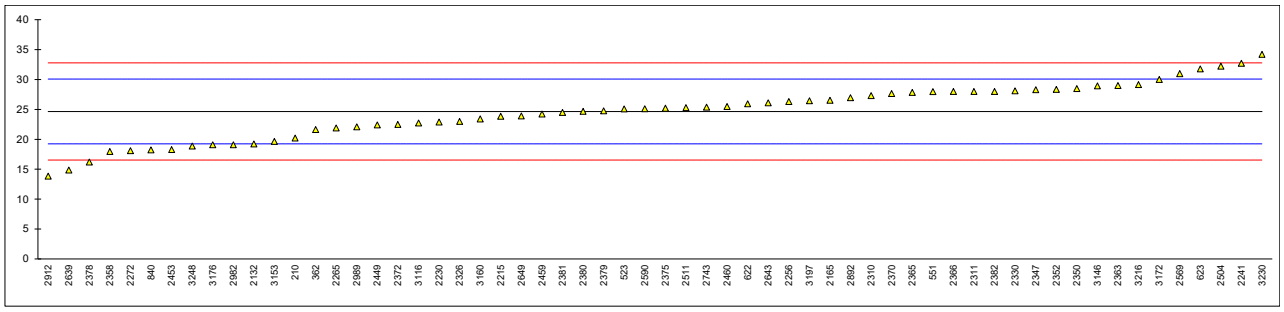


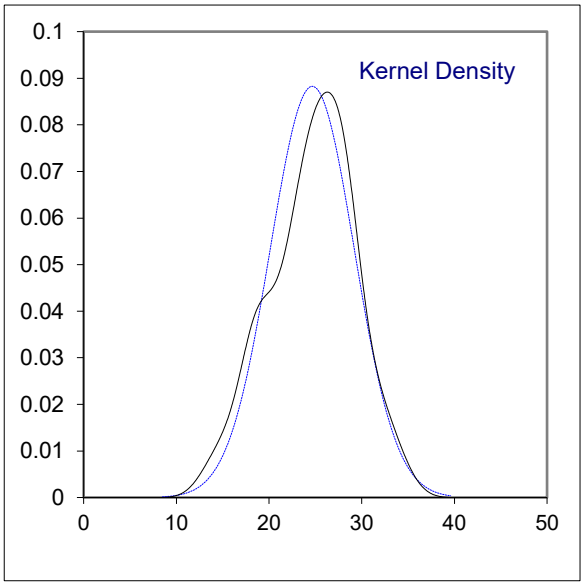
Determination of Formaldehyde content (Colorimetric) on sample #22765; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	ISO17226-2	20.23		-1.63	
362	ISO17226-2	21.64		-1.11	
523	ISO17226-2	25.09		0.16	
551	ISO17226-2	27.98		1.23	
622	ISO17226-2	25.94		0.47	
623	ISO17226-2	31.8		2.64	
840	ISO17226-2	18.23		-2.37	
2115		-----		-----	
2120		-----		-----	
2129		-----		-----	
2132	ISO17226-2	19.2450		-2.00	
2165	ISO17226-2	26.537		0.69	
2215	ISO17226-2	23.87		-0.29	
2230	ISO17226-2	22.9		-0.65	
2241	ISO17226-2	32.71		2.97	
2250		-----		-----	
2256	ISO17226-2	26.31		0.61	
2265	ISO17226-2	21.907		-1.01	
2272	ISO17226-2	18.1		-2.42	
2284		-----		-----	
2287		-----		-----	
2290		-----		-----	
2310	ISO17226-2	27.3		0.98	
2311	ISO17226-2	28.0		1.23	
2326	ISO17226-2	22.98		-0.62	
2330	ISO17226-2	28.11		1.28	
2347	ISO17226-2	28.3		1.35	
2350	ISO17226-2	28.48		1.41	
2352	ISO17226-2	28.34		1.36	
2357		-----		-----	
2358	ISO17226-2	17.956		-2.47	
2363	ISO17226-2	29		1.60	
2365	ISO17226-2	27.85		1.18	
2366	ISO17226-2	28		1.23	
2370	ISO17226-2	27.67		1.11	
2372	ISO17226-2	22.5		-0.79	
2373		not applicable		-----	
2375	ISO17226-2	25.2		0.20	
2378	ISO17226-2	16.2		-3.12	
2379	ISO17226-2	24.76		0.04	
2380	ISO17226-2	24.68		0.01	
2381	ISO17226-2	24.5		-0.06	
2382	ISO17226-2	28.00		1.23	
2386		-----		-----	
2449	ISO17226-2	22.4		-0.83	
2453	ISO17226-2	18.3		-2.34	
2459	ISO17226-2	24.25		-0.15	
2460	ISO17226-2	25.5011		0.31	
2462		-----		-----	
2477		-----		-----	
2492		-----		-----	
2495		-----		-----	
2504	ISO17226-2	32.2449		2.80	
2511	ISO17226-2	25.3		0.24	
2561		-----		-----	
2569	ISO17226-2	31		2.34	
2582		-----		-----	
2590	ISO17226-2	25.125		0.17	
2639	GB/T19941.2	14.86		-3.61	
2643	ISO17226-2	26.10		0.53	
2649	ISO17226-2	23.9		-0.28	
2695		-----		-----	
2703		-----		-----	
2711		-----		-----	
2743	ISO17226-2	25.38		0.27	
2756		-----		-----	
2758		-----		-----	
2765		-----		-----	
2787		-----		-----	
2806		-----		-----	
2836		-----		-----	
2844		-----		-----	
2892	ISO17226-2	26.95		0.85	
2912	ISO17226-2	13.85		-3.99	

lab	method	value	mark	z(targ)	remarks
2966		-----		-----	
2967		-----		-----	
2977		-----		-----	
2982	ISO17226-2	19.10		-2.05	
2989	ISO17226-2	22.0961		-0.94	
3003		-----		-----	
3116	ISO17226-2	22.72		-0.71	
3146	ISO17226-2	28.94		1.58	
3153	ISO17226-2	19.65		-1.85	
3154		-----		-----	
3160	ISO17226-2	23.420		-0.46	
3172	ISO17226-2	30.022		1.98	
3176	ISO17226-2	19.09		-2.05	
3197	ISO17226-2	26.45		0.66	
3210		-----		-----	
3216	ISO17226-2	29.14		1.66	
3228		-----		-----	
3230	In house	34.22		3.53	
3237		-----		-----	
3248	ISO17226-2	18.89		-2.13	

normality	OK	
n	60	
outliers	0	
mean (n)	24.654	
st.dev. (n)	4.5189	RSD = 18%
R(calc.)	12.653	
st.dev.(ISO17226-2:18)	2.7102	
R(ISO17226-2:18)	7.589	





APPENDIX 2 Analytical details

lab	ISO/IEC17025 accredited	Sample intake (in grams)
210	Yes	
362	Yes	2g
523	No	4 g
551	Yes	0.5042g
622	Yes	2 grams
623	Yes	2 grams
840	Yes	1 gram
2115	Yes	2 grams
2120	No	1 g
2129	Yes	1 g
2132	Yes	2g
2165	Yes	2.0g
2215	Yes	2g
2230	Yes	1.0051g
2241	Yes	0.5g
2250	Yes	2g
2256	No	2.00g
2265	Yes	2
2272	---	
2284	Yes	1g
2287	No	1.0 g
2290	Yes	
2310	Yes	2
2311	Yes	2
2326	Yes	2.0049g
2330	Yes	2 g
2347	Yes	1g
2350	Yes	2g
2352	Yes	ISO 17226-1:1g ISO 17226-2:1g
2357	---	
2358	Yes	2g
2363	Yes	2g
2365	Yes	2g
2366	Yes	2g
2370	Yes	2 g
2372	Yes	2g
2373	---	not applicable
2375	Yes	1 gram
2378	Yes	2g
2379	Yes	1 gram
2380	Yes	2.0 g
2381	Yes	2 gm
2382	Yes	2g
2386	Yes	1.0 g
2449	No	2.0 gram
2453	No	±2g
2459	Yes	2gm
2460	Yes	2 g
2462	Yes	0.5g
2477	Yes	2 g
2492	Yes	0.5g
2495	Yes	1
2504	Yes	2g
2511	---	
2561	No	1g
2569	Yes	2 gm
2582	---	
2590	Yes	1g
2639	No	about 2 grams
2643	Yes	2 g
2649	Yes	2g
2682	---	
2695	Yes	2.0026
2703	No	1.99g
2711	No	2,0
2743	Yes	1g
2756	Yes	2.0g
2758	No	1g
2765	No	1.5 g
2787	Yes	2g
2806	Yes	
2836	Yes	About 2.0000g.
2844	No	2.0604 2.0592
2892	Yes	2.0146
2912	Yes	2 g
2966	Yes	2.000
2967	No	2 grams
2977	Yes	1g
2982	Yes	2.0g
2989	No	2g
3003	Yes	3 grms
3116	Yes	2
3146	Yes	2.00g
3153	No	2 grams
3154	Yes	2 g
3160	Yes	2g
3172	Yes	
3176	Yes	0,5 gram
3197	Yes	2 g
3210	Yes	2 grams
3216	Yes	Sample1: 2,0579g Sample2: 2,0362g
3228	Yes	1
3230	Yes	2 grams
3237	No	2
3248	Yes	2.0000

APPENDIX 3

Number of participants per country

5 labs in	BANGLADESH
2 labs in	BRAZIL
1 lab in	BULGARIA
1 lab in	CAMBODIA
1 lab in	ETHIOPIA
2 labs in	FRANCE
6 labs in	GERMANY
6 labs in	HONG KONG
3 labs in	INDIA
2 labs in	INDONESIA
12 labs in	ITALY
1 lab in	JAPAN
2 labs in	KOREA, Republic of
1 lab in	MAURITIUS
3 labs in	MEXICO
1 lab in	MOROCCO
18 labs in	P.R. of CHINA
4 labs in	PAKISTAN
2 labs in	PORTUGAL
1 lab in	SERBIA
3 labs in	SPAIN
1 lab in	SRI LANKA
1 lab in	SWITZERLAND
3 labs in	TAIWAN
2 labs in	THAILAND
1 lab in	TUNISIA
4 labs in	TURKEY
2 labs in	UNITED KINGDOM
3 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

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 ISO5725:86
3 ISO5725 parts 1-6:94
4 ISO13528:05
5 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
8 J.N. Miller, Analyst, 118, 455, (1993)
9 Analytical Methods Committee, Technical Brief, No 4, January 2001
10 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)