Results of Proficiency Test Formaldehyde in Leather/Footwear November 2021

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

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 2021/2022 it was decided to continue the proficiency test for the determination of Formaldehyde in Leather/Footwear.

In this interlaboratory study 101 laboratories in 27 different 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 labelled #21765 positive on Formaldehyde.
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 grey leather positive for Formaldehyde was obtained from a leather supplier. The batch was grinded and homogenized. After homogenization 150 bags were filled with approximately 6 grams each and labelled #21765. 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 #21765-1	93.9
sample #21765-2	96.3
sample #21765-3	96.5
sample #21765-4	95.9
sample #21765-5	95.8
sample #21765-6	96.0
sample #21765-7	94.3
sample #21765-8	95.4

Table 1: homogeneity test results of subsamples #21765

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)	2.6
reference test method	ISO17226-1:21
0.3 x R (reference test method)	17.7

Table 2: evaluation of the repeatability of subsamples #21765

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 #21765 was sent on October 20, 2021.

2.5 ANALYZES

The participants were requested to determine Formaldehyde according to ISO17226-1 (HPLC method) and/or ISO17226-2 (Colorimetric method).
It was also requested to report if the laboratory was accredited for the determined components 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 method (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 test 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) test 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 samples. Eight participants reported the test results after the final reporting date and four other participants were not able to report any test results. Not all participants were able to report all parameters requested.
In total 97 participants reported 140 numerical test results. Observed were 14 outlying test results, which is 10% of the numerical test results. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Not all 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, see also paragraph 3.1.

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. Therefore, it was decided to request the test method version of the participants and to evaluate the participants that performed ISO17226-1 version 2018 or 2008 separately from ISO17226-1 version 2021, see also discussion in paragraph 5.
The precision data for ISO17226-1 version 2008 and 2018 are the same and have been updated in the 2021 version. For previous iis PTs the precision data of the 2008/2018 versions were estimated from an equation based on the given data in ISO17226-1 version 2008/2018. 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 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.

Formaldehyde content (HPLC): It was decided to evaluate the test results of ISO17226-1 version 2021 separately from the older versions. The test results of laboratories that did not report the test method version were kept in the data set of the 2021 version but were excluded in the statistical evaluation. This determination may be problematic for some laboratories.
For the group that performed ISO17226-1 version 2021: eight statistical outliers were observed and twenty other test results were excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the estimated requirements of ISO17226-1:21.
For the group that performed ISO17226-1 version 2008 or 2018: one statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated requirements of IS17226-1:18.

Formaldehyde content (Colorimetric): This determination was not problematic. Five statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is 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 test methods (in casu ISO test methods) are presented in the next table.

Component	unit	n	average	2.8 * sd	R(lit)
Formaldehyde (HPLC) ISO17226-1:21	mg/kg	42	39.4	15.2	24.3
Formaldehyde (HPLC) ISO17226-1:08/18	mg/kg	7	68.1	7.9	41.0
Formaldehyde (Colorimetric)	mg/kg	57	41.6	9.8	11.3

Table 3: reproducibilities of tests on sample #21765

Without further statistical calculations, it can be concluded that for all 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 2021 WITH PREVIOUS PTS

	November 2021	November 2020	November 2019	November 2018	November 2017
Number of reporting laboratories	97	106	136	114	102
Number of test results	140	356	441	396	378
Number of statistical outliers	14	14	17	12	16
Percentage of statistical outliers	10%	3.9%	3.9%	3.0%	4.2%

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 expressed as relative standard deviation (RSD) of the PTs, see next table.

Component	November 2021	November 2020	November 2019	2013-2018	Target
Formaldehyde (HPLC) 2021	14%	n.a.	n.a.	n.a.	22%
Formaldehyde (HPLC) 2018/2008	4%	12%	12%	9-30%	13-25%
Formaldehyde (Colorimetric)	8%	8%	8%	17-39%	11-22%

Table 5: development of the uncertainties over the years

The uncertainties for the 2021 PT are equal or smaller to the uncertainties of the PT of 2020 for the HPLC and Colorimetric determinations of Formaldehyde in Leather/Footwear. Both determinations meet again the estimated targets from the reference test methods.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

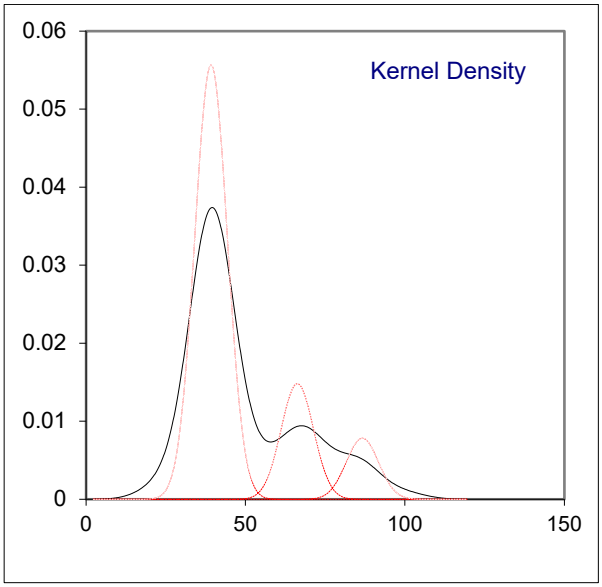
The reported details of the analytical test methods are listed in appendix 2.
About 90% of the reporting laboratories reported to be accredited for the determination of Formaldehyde in Leather/Footwear. And approximately 70% of the reporting laboratories used 2 grams as intake as prescribed in ISO17226.
As the majority of the group follow the same analytical procedures no separate statistical analysis has been performed.

5 DISCUSSION

Difference of test method version for ISO17226-1 (HPLC) and impact on evaluation

The version of ISO17226-2 (Colorimetric) did not change, but in 2021 a new version of ISO17226-1 (HPLC) was published. The procedure in this version has an added step in the derivatization with Dinitrophenylhydrazine (DNPH). After adding the leather and reagents in the 10 mL volumetric flask it should be placed in a water bath at 50°C for 180 minutes before filing the flask to the mark with water and follow the procedure as it was in the earlier version. This additional step appeared to influence the final test result and may deviate from the test results that are obtained when using the 2008/2018 version.

After the PT was closed and a first assessment was made of the reported test results, one large group and two smaller groups of test results appeared to be present (see black line in the graph below).



In general in iis PTs request the test method used and not the method version. Only a few laboratories reported the version of the test method as a remark. Therefore, iis decided to request the version of ISO17226-1 by email from all participants after closure of the PT. In total 59 participants reported the version of the test method that was used. Eight participants reported to have used ISO17226-1:08 or ISO17226-1:18 and fifty-one participants reported to have used ISO17226-1:21. Twenty participants did not answer the request.
It appeared that the test results based on ISO17226-1 version 2018 or 2008 were all in the group with the higher test results. From the test results based on ISO17226-1 version 2021 the majority was in the group with the lower test results. It was decided by iis to evaluate the group that reported ISO17226-1 version 2018 and 2008 separately and to evaluate the other test results as one group. The test results of the participants that did not answered which test method version was used were put in the 2021 group but excluded from the statistical evaluation.
Although only 8 laboratories reported to have used the older versions of ISO17226-1, it appears that the new version of this test method with the amended procedures gives lower test results than the older versions.

Difference in test results for ISO17226-1 and ISO17226-2

The Formaldehyde test method ISO17226 part 1 and part 2 describe both the determination of the Formaldehyde content by extraction of the Formaldehyde from Leather with a detergent solution. The difference between both parts is the method of quantification. Quantification of the Formaldehyde in part 1 is done by HPLC and in part 2 by Colorimetric analysis. Part 1 is specific for Formaldehyde alone and part 2 measures a color solution and is more sensitive for interferences of other substances. Therefore, in theory, the test results from part 2 could be higher on average than the test results from part 1. Remarkably, this is not observed in sample #21765 for the version of part one of 2008/2018. However, the group using ISO17226-1:21 give an average result which is in line to the average result of the group performing ISO17226-2:18.

Sample #21765 compared to Formaldehyde limits

When the test 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 may be noticed that not all participants would make identical decisions about the acceptability of the Leather.

	Category A Products for babies: underclothes, bedding, etc	Category B Products with **Direct** skin contact	Category C Products 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 all reporting laboratories would reject this sample for category A except one. For category B not all reporting laboratories would make the same decision about accepting or rejecting this sample. All of the reporting laboratories would accept this sample for category C.

6 CONCLUSION

The variation observed for the determinations in this interlaboratory study can be caused by the pretreatment by the laboratories of the sample and/or by the performance of the analysis. Consequently, the reproducibility cannot be improved by only one change in the analysis. Each laboratory has to evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1

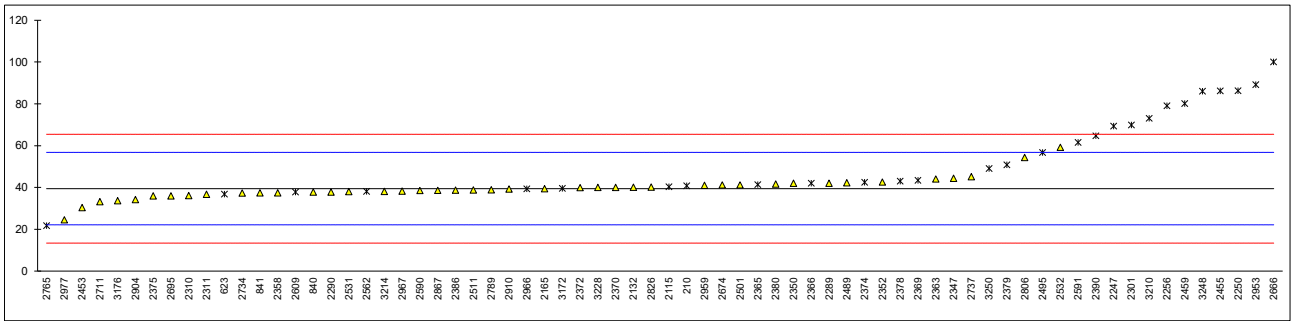
Determination of Formaldehyde (HPLC) ISO17226-1:21 on sample #21765; results in mg/kg

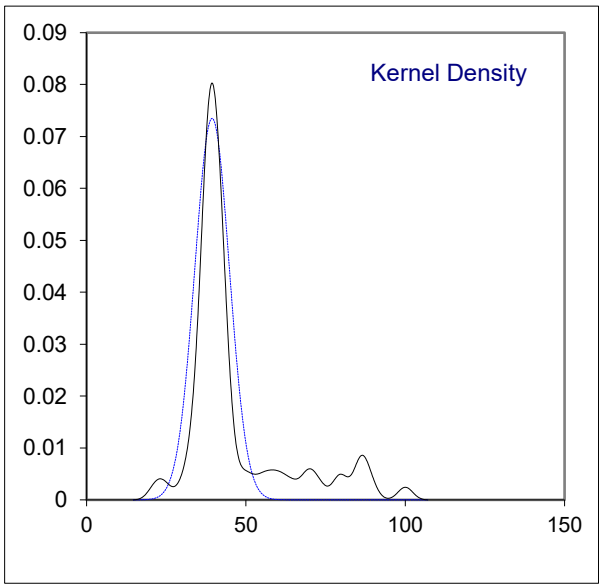
lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210	ISO17226-1	40.75	Ex	0.15	
339		-----		-----	
362		-----		-----	
623	ISO17226-1	36.80	Ex	-0.30	
840	ISO17226-1:2021	37.8		-0.19	
841	ISO17226-1:2021	37.4		-0.23	
2115	ISO17226-1	40.3	Ex	0.10	
2129		-----		-----	
2132	ISO17226-1:2021	40.0722		0.07	
2139		-----		-----	
2165	ISO17226-1:2021	39.4		0.00	
2166		-----		-----	
2247	ISO17226-1:2021	69.30	R(0.01)	3.44	
2250	ISO17226-1:2021	86.2	R(0.01)	5.39	
2256	ISO17226-1:2021	79.07	R(0.01)	4.57	
2265		-----		-----	
2273		-----		-----	
2289	ISO17226-1:2021	42		0.30	
2290	ISO17226-1:2021	37.8		-0.19	
2293		-----		-----	
2301	ISO17226-1:2021	69.82	R(0.01)	3.50	
2310	ISO17226-1:2021	36.1		-0.38	
2311	ISO17226-1:2021	36.70		-0.31	
2330		-----		-----	
2347	ISO17226-1:2021	44.4		0.57	
2350	ISO17226-1:2021	41.97		0.29	
2352	ISO17226-1:2021	42.6		0.37	
2357		-----		-----	
2358	ISO17226-1:2021	37.45		-0.23	
2363	ISO17226-1:2021	44		0.53	
2365	ISO17226-1	41.30	ex	0.22	
2366	ISO17226-1	42	ex	0.30	
2369	ISO17226-1	43.4	ex	0.46	
2370	ISO17226-1:2021	40.02		0.07	
2372	ISO17226-1:2021	39.849		0.05	
2374	ISO17226-1	42.43	ex	0.35	
2375	ISO17226-1:2021	36.0		-0.39	
2378	ISO17226-1	43.0	ex	0.41	
2379	ISO17226-1	50.74	ex	1.30	
2380	ISO17226-1:2021	41.6		0.25	
2381	ISO17226-1	Out Cap		-----	
2386	ISO17226-1:2021	38.73		-0.08	
2390	ISO17226-1:2021	64.69	R(0.01)	2.91	
2410		-----		-----	
2449		-----		-----	
2453	ISO17226-1:2021	30.39		-1.04	
2455	ISO17226	86.13	ex	5.39	
2459	ISO17226-1:2021	80.15	R(0.01)	4.70	
2460		-----		-----	
2489	ISO17226-1:2021	42.17		0.32	
2495	ISO17226-1	56.70	ex	1.99	
2501	ISO17226-1:2021	41.21		0.21	
2511	ISO17226-1:2021	38.8		-0.07	
2531	ISO17226-1:2021	38.03		-0.16	
2532	ISO17226-1:2021	59.2		2.28	
2553		-----		-----	
2561		-----		-----	
2562	In house	38.06	ex	-0.16	
2573		-----		-----	
2582		-----		-----	
2590	ISO17226-1:2021	38.472		-0.11	
2591	ISO17226-1	61.50	ex	2.55	
2609	ISO17226-1	37.75	ex	-0.19	
2612		-----		-----	
2639		-----		-----	
2650		-----		-----	
2666	ISO17226-1	100.02	ex	6.99	
2674	ISO17226-1:2021	41.2		0.20	
2695	ISO17226-1:2021	36.000		-0.39	
2703		-----		-----	
2711	ISO17226-1:2021	33.2		-0.72	
2727		-----		-----	
2734	ISO17226-1:2021	37.32		-0.24	

lab	method	value	mark	z(targ)	remarks
2737	ISO17226-1:2021	45.12		0.66	
2765	ISO17226-1:2021	21.7	R(0.01)	-2.04	
2789	ISO17226-1:2021	38.87		-0.06	
2806	ISO17226-1:2021	54.3		1.72	
2826	ISO17226-1:2021	40.2		0.09	
2849		-----		-----	
2867	ISO17226-1:2021	38.6		-0.10	
2904	ISO17226-1:2021	34.13		-0.61	
2910	ISO17226-1:2021	39.24		-0.02	
2953	ISO17226-1:2021	89.12	R(0.01)	5.73	
2959	ISO17226-1:2021	41		0.18	
2966	ISO17226-1	39.3	ex	-0.01	
2967	ISO17226-1:2021	38.20		-0.14	
2977	ISO17226-1:2021	24.54		-1.72	
2985		-----		-----	
3116		-----		-----	
3146		-----		-----	
3154		-----		-----	
3160		-----		-----	
3172	ISO17226-1	39.513	ex	0.01	
3176	ISO17226-1:2021	33.63		-0.67	
3210	In house	73.03	ex	3.87	
3214	ISO17226-1:2021	38.10		-0.15	
3228	ISO17226-1:2021	40		0.07	
3230		-----		-----	
3248	ISO17226-1	86.0227	ex	5.37	
3250	ISO17226-1	49.0	ex	1.10	

	normality	not OK
	n	42
	outliers	8 (+20ex)
	mean (n)	39.424
	st.dev. (n)	5.4276 RSD = 14%
	R(calc.)	15.197
	st.dev.(ISO17226-1:21)	8.6733
	R(ISO17226-1:21)	24.285

ex = test result excluded for statistical calculations, as the year of the test method was not reported, see paragraph 4.1 and discussion





Determination of Formaldehyde (HPLC) ISO17226-1:18 on sample #21765; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210		-----		-----	
339		-----		-----	
362		-----		-----	
623		-----		-----	
840		-----		-----	
841		-----		-----	
2115		-----		-----	
2129	ISO17226-1:2008	65.125		-0.21	
2132		-----		-----	
2139	ISO17226-1:2018	65.4		-0.19	
2165		-----		-----	
2166		-----		-----	
2247		-----		-----	
2250		-----		-----	
2256		-----		-----	
2265		-----		-----	
2273		-----		-----	
2289		-----		-----	
2290		-----		-----	
2293		-----		-----	
2301		-----		-----	
2310		-----		-----	
2311		-----		-----	
2330	ISO17226-1:2018	73.43		0.36	
2347		-----		-----	
2350		-----		-----	
2352		-----		-----	
2357		-----		-----	
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2365		-----		-----	
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2372		-----		-----	
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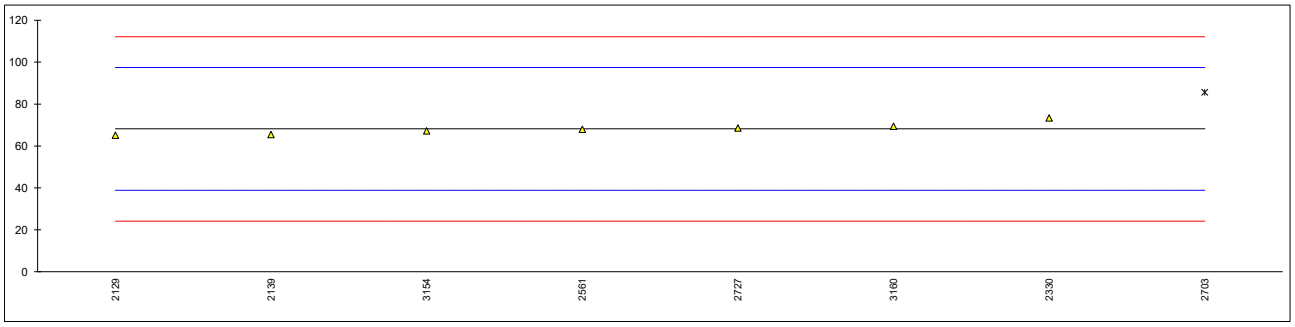
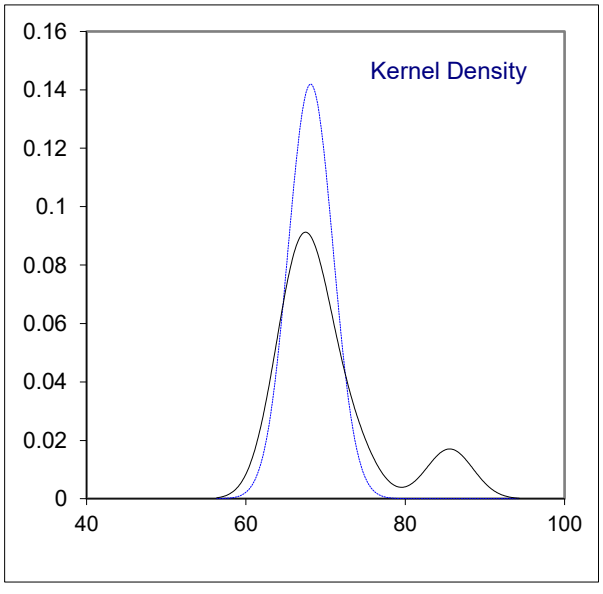
2379		-----		-----	
2380		-----		-----	
2381		-----		-----	
2386		-----		-----	
2390		-----		-----	
2410		-----		-----	
2449		-----		-----	
2453		-----		-----	
2455		-----		-----	
2459		-----		-----	
2460		-----		-----	
2489		-----		-----	
2495		-----		-----	
2501		-----		-----	
2511		-----		-----	
2531		-----		-----	
2532		-----		-----	
2553		-----		-----	
2561	ISO17226-1:2019	67.95		-0.01	
2562		-----		-----	
2573		-----		-----	
2582		-----		-----	
2590		-----		-----	
2591		-----		-----	
2609		-----		-----	
2612		-----		-----	
2639		-----		-----	
2650		-----		-----	
2666		-----		-----	
2674		-----		-----	
2695		-----		-----	
2703	ISO17226-1:2019	85.6	G(0.01)	1.19	
2711		-----		-----	
2727	ISO17226-1:2018	68.54		0.03	
2734		-----		-----	

lab	method	value	mark	z(targ)	remarks
2737		-----		-----	
2765		-----		-----	
2789		-----		-----	
2806		-----		-----	
2826		-----		-----	
2849		-----		-----	
2867		-----		-----	
2904		-----		-----	
2910		-----		-----	
2953		-----		-----	
2959		-----		-----	
2966		-----		-----	
2967		-----		-----	
2977		-----		-----	
2985		-----		-----	
3116		-----		-----	
3146		-----		-----	
3154	ISO17226-1:2019	67.14		-0.07	
3160	ISO17226-1:2018	69.41		0.09	
3172		-----		-----	
3176		-----		-----	
3210		-----		-----	
3214		-----		-----	
3228		-----		-----	
3230		-----		-----	
3248		-----		-----	
3250		-----		-----	

normality	unknown	
n	7	
outliers	1	
mean (n)	68.142	
st.dev. (n)	2.8104	RSD = 4%
R(calc.)	7.869	
st.dev.(ISO17226-1:18)	14.6547	
R(ISO17226-1:18)	41.033	



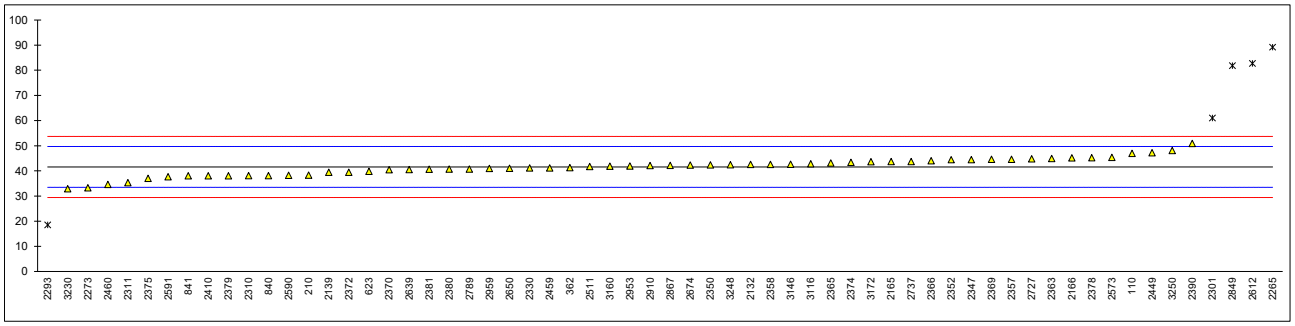


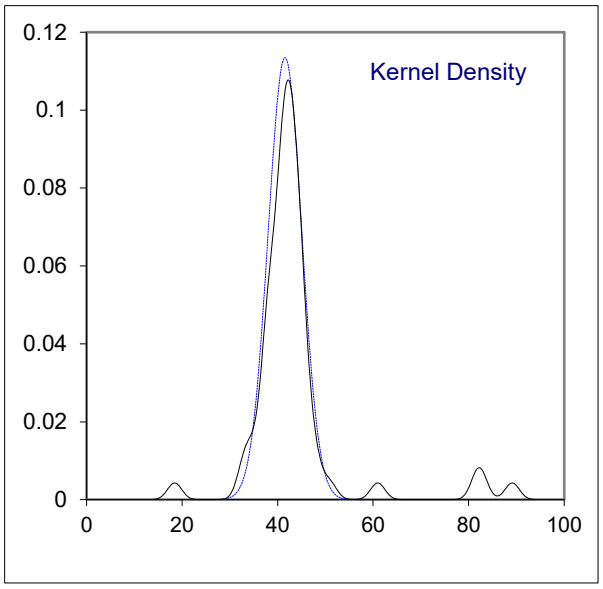
Determination of Formaldehyde content (Colorimetric) on sample #21765; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO17226-2	46.976		1.34	
210	ISO17226-2	38.26		-0.82	
339		-----		-----	
362	ISO17226-2	41.29	C	-0.07	first reported: 82.85
623	ISO17226-2	39.80		-0.44	
840	ISO17226-2	38.1		-0.86	
841	ISO17226-2	38.0		-0.89	
2115		-----		-----	
2129		-----		-----	
2132	ISO17226-2	42.55		0.24	
2139	ISO17226-2	39.4		-0.54	
2165	ISO17226-2	43.7		0.53	
2166	In house	45.16		0.89	
2247		-----		-----	
2250		-----		-----	
2256		-----		-----	
2265	ISO17226-2	89.15	R(0.01)	11.77	
2273	ISO17226-2	33.22		-2.07	
2289		-----		-----	
2290		-----		-----	
2293	ISO17226-2	18.47	R(0.01)	-5.72	
2301	ISO17226-2	60.99	C,R(0.01)	4.80	first reported: 68.18
2310	ISO17226-2	38.05		-0.87	
2311	ISO17226-2	35.30		-1.55	
2330	ISO17226-2	41.18		-0.10	
2347	ISO17226-2	44.5		0.72	
2350	ISO17226-2	42.35		0.19	
2352	ISO17226-2	44.4		0.70	
2357	ISO17226-2	44.6		0.75	
2358	ISO17226-2	42.55		0.24	
2363	ISO17226-2	44.9		0.82	
2365	ISO17226-2	43.11		0.38	
2366	ISO17226-2	44		0.60	
2369	ISO17226-2	44.58		0.74	
2370	ISO17226-2	40.43		-0.28	
2372	ISO17226-2	39.4		-0.54	
2374	ISO17226-2	43.39		0.45	
2375	ISO17226-2	37.0		-1.13	
2378	ISO17226-2	45.2		0.90	
2379	ISO17226-2	38.01		-0.88	
2380	ISO17226-2	40.7		-0.22	
2381	ISO17226-2	40.60		-0.24	
2386		-----		-----	
2390	ISO17226-2	50.903		2.31	
2410	ISO17226-2	38		-0.89	
2449	ISO17226-2	47.18		1.39	
2453		-----		-----	
2455		-----		-----	
2459	ISO17226-2	41.18		-0.10	
2460	ISO17226-2	34.58		-1.73	
2489		-----		-----	
2495		-----		-----	
2501		-----		-----	
2511	ISO17226-2	41.7		0.03	
2531		-----		-----	
2532		-----		-----	
2553		-----		-----	
2561		-----		-----	
2562		-----		-----	
2573	ISO17226-2	45.34		0.93	
2582		-----		-----	
2590	ISO17226-2	38.17		-0.84	
2591	ISO17226-2	37.63		-0.98	
2609		-----		-----	
2612	ISO17226-2	82.6745	R(0.01)	10.17	
2639	GB/T19941.2	40.50		-0.27	
2650	ISO17226-2	41.05		-0.13	
2666		-----		-----	
2674	ISO17226-2	42.33		0.19	
2695		-----		-----	
2703		-----		-----	
2711		-----		-----	
2727	ISO17226-2	44.81		0.80	
2734		-----		-----	

lab	method	value	mark	z(targ)	remarks
2737	ISO17226-2	43.71		0.53	
2765		-----		-----	
2789	ISO17226-2	40.70		-0.22	
2806		-----		-----	
2826		-----		-----	
2849	ISO17226-2	81.8	C,R(0.01)	9.95	first reported: 64.735
2867	ISO17226-2	42.2		0.15	
2904		-----		-----	
2910	ISO17226-2	42.14		0.14	
2953	ISO17226-2	41.91		0.08	
2959	ISO17226-2	41		-0.14	
2966		-----		-----	
2967		-----		-----	
2977		-----		-----	
2985		-----		-----	
3116	ISO17226-2	42.80		0.30	
3146	ISO17226-2	42.58		0.25	
3154		-----		-----	
3160	ISO17226-2	41.84		0.06	
3172	ISO17226-2	43.607		0.50	
3176		-----		-----	
3210		-----		-----	
3214		-----		-----	
3228		-----		-----	
3230	ISO17226-2	32.893		-2.15	
3248	ISO17226-2	42.4107		0.21	
3250	ISO17226-2	48.1		1.61	

	normality	OK	
	n	57	
	outliers	5	
	mean (n)	41.578	
	st.dev. (n)	3.5163	RSD = 8%
	R(calc.)	9.846	
	st.dev.(ISO17226-2:18)	4.0406	
	R(ISO17226-2:18)	11.314	





APPENDIX 2 Analytical details

lab	ISO/IEC17025 accredited	Sample intake (in grams)
110	Yes	1 gram
210	Yes	
339	---	
362	Yes	2g
623	Yes	1 gram
840	Yes	2 grams
841	Yes	2 grams
2115	Yes	2 g
2129	Yes	1.0 g
2132	Yes	2 grams
2139	Yes	2.020 g
2165	Yes	2g
2166	Yes	1 g
2247	Yes	Approx six grams
2250	Yes	2 g
2256	Yes	1 gram
2265	No	2,0
2273	Yes	2
2289	Yes	2g
2290	Yes	
2293	Yes	2.0
2301	Yes	2.0017
2310	Yes	2g
2311	Yes	2
2330	Yes	2 g
2347	Yes	1g
2350	Yes	2 g
2352	Yes	ISO 17226-1 : 1g ISO 17226-2 : 1g
2357	---	
2358	Yes	1.0 gram
2363	Yes	1g
2365	Yes	1g
2366	Yes	2g
2369	Yes	
2370	Yes	2 g
2372	No	2g
2374	Yes	1g
2375	Yes	1g
2378	Yes	2g
2379	Yes	1 g
2380	Yes	4.00 g
2381	Yes	2 gm
2386	Yes	1 g / 25ml
2390	Yes	2.0067 g
2410	Yes	1 g
2449	Yes	2.0
2453	No	±1g
2455	Yes	2.0552 grams of sample used
2459	Yes	2.0 gram
2460	Yes	2 g
2489	Yes	1.0026 g
2495	Yes	1.00
2501	Yes	2g
2511	---	
2531	Yes	2 grams
2532	Yes	1gram
2553	---	
2561	Yes	2g
2562	No	2.0
2573	Yes	2g
2582	---	
2590	Yes	1g
2591	Yes	2.0 grams
2609	Yes	2.00
2612	Yes	2 gramms
2639	No	2.0029g.
2650	Yes	2 grams
2666	Yes	2,0
2674	No	2.0g
2695	Yes	1g
2703	Yes	2g
2711	No	1,991
2727	Yes	2.00g
2734	Yes	6 g
2737	Yes	1 grams
2765	Yes	1.5 g
2789	Yes	2
2806	No	
2826	Yes	2grams
2849	No	Two samples of about 2.0 grams.
2867	Yes	2g
2904	No	2 grams
2910	Yes	2.0g
2953	Yes	2
2959	Yes	2.000g
2966	Yes	2.000
2967	No	2 grams
2977	Yes	4
2985	---	
3116	Yes	2 grams
3146	Yes	2.00g
3154	Yes	
3160	Yes	2 g
3172	Yes	
3176	Yes	2
3210	Yes	2g
3214	Yes	2 g
3228	Yes	2
3230	Yes	4grams(2 x 2grams)
3248	Yes	2.0049
3250	Yes	1g

APPENDIX 3

Number of participants per country

2 labs in	BANGLADESH
1 lab in	BULGARIA
2 labs in	CAMBODIA
3 labs in	FRANCE
8 labs in	GERMANY
1 lab in	GUATEMALA
6 labs in	HONG KONG
5 labs in	INDIA
2 labs in	INDONESIA
15 labs in	ITALY
1 lab in	MAURITIUS
1 lab in	MEXICO
2 labs in	MOROCCO
23 labs in	P.R. of CHINA
3 labs in	PAKISTAN
1 lab in	POLAND
2 labs in	PORTUGAL
3 labs in	SOUTH KOREA
4 labs in	SPAIN
2 labs in	SRI LANKA
3 labs in	TAIWAN
1 lab in	THAILAND
1 lab in	TUNISIA
2 labs in	TURKEY
2 labs in	U.S.A.
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
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7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
8 J.N. Miller, Analyst, 118, 455, (1993)
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