Results of Proficiency Test pH and Formaldehyde in leather November 2017

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1 INTRODUCTION

Worldwide, many consumer products are produced from leather. During the production of leather products, many different types of auxiliary agents and dyes are used to process leather. Neither in the U.S. nor in the European Union there is general legislation that limits the presence of formaldehyde in leather. Many countries have adopted environmental standards and requirements restricting the use of harmful chemicals. Laws and regulations impose some of these standards and requirements. In addition to mandatory environmental standards and requiremental requirements for leather, there are some Ecolabelling schemes imposing environmental requirements for textile & leather products on a voluntary basis. Well-known organisations are Öko-Tex Standard 100 (Germany) and Bluesign® (Switzerland), which has created a Bluesign® system substances list (BSSL).

Since 2013, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Formaldehyde and pH in leather. During the annual proficiency testing program 2017/2018, it was decided to continue the round robin for the analysis of Formaldehyde content and pH.

In this interlaboratory study 103 laboratories in 28 different countries registered for participation. See appendix 3 for the number of participating laboratories per country. In this report, the results of the 2017 Formaldehyde and pH in leather 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 preparation and analyses of fit-for-use and homogeneity were subcontracted to an ISO17025 accredited laboratory. It was decided to use in this proficiency test one leather sample (labelled #17640) positive on Formaldehyde and one leather sample (labelled #17641) especially for pH determination. 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 (iis) in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC 17043:2010. 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 March 2017 (iis-protocol, version 3.4). 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

The first batch, brown leather pieces was obtained from a third party laboratory. The batch was used before in a previous interlaboratory study (iis13A04L). Therefore, the samples were considered to be homogeneous (see report iis13A04L). Each participant received a sample of approx. 6 grams packed in a polypropylene bag and wrapped in aluminium foil (labelled #17640).

The second batch was a black leather sample, which was shreddered into small pieces. After homogenisation, 130 subsamples of approx. 10 grams were prepared and labelled sample #17641. The homogeneity of the subsamples was checked on 8 stratified randomly selected samples. See the following table for the test results.

	рН
Sample #17641-1	3.28
Sample #17641-2	3.28
Sample #17641-3	3.27
Sample #17641-4	3.27
Sample #17641-5	3.26
Sample #17641-6	3.28
Sample #17641-7	3.26
Sample #17641-8	3.27

Table 1: homogeneity test results of subsamples #17641

From the above test results, the observed repeatability was calculated and compared with 0.3 times the target reproducibility (based on the repeatability) in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	рН
r (observed)	0.02
Reference test method	ASTM D2810:13
0.3*R (ref. test method)	0.04

Table 2: repeatability of subsamples #17641

The calculated repeatability was in agreement with 0.3 times the estimated reproducibility (based on the repeatability) of the reference test method. Therefore, homogeneity of the subsamples #17641 was assumed.

To each of the participants, 1 sample labelled #17640 and 1 sample labelled #17641 was sent on October 11, 2017.

2.5 ANALYSES

The participants were requested to determine on sample #17640, the content of Formaldehyde (HPLC) and/or the content of Formaldehyde (colorimetric). On sample #17641 was requested to determine the pH "undiluted", pH "ten times diluted extract" and/or the "difference between pH of extract and pH of ten times diluted extract"

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

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 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.com/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 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 the appendix 1 of this report. The laboratories are represented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that did not report 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. Additional or corrected test results are used for the data analysis and the original 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 organisation 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 March 2017 (iis-protocol, version 3.4).

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.

In accordance to ISO 5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. 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 Rosner's. 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. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation, it is mentioned in the report and it will have significant consequences for the evaluation of the test results.

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

3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported 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 standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. The Kernel Density Graph 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 was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

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

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-purpose.

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 result tables in appendix 1.

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

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

 $\begin{aligned} |z| &< 1 \text{ good} \\ 1 &< |z| &< 2 \text{ satisfactory} \\ 2 &< |z| &< 3 \text{ questionable} \\ 3 &< |z| & \text{ unsatisfactory} \end{aligned}$

4 EVALUATION

During the execution of this proficiency test, no problems were encountered with the delivery of the samples. Only one laboratory did not report any test results. Finally, the 102 reporting laboratories sent in total 378 numerical test results. Observed were 16 outlying test results, which is 4.2% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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

For the determination pH of Leather, the test methods ASTM D2810:13 and ISO4045:08 are considered to be the official test methods. Regretfully, ISO4045 does not provide precision data. Therefore, the reproducibility of ASTM D2810 was taken to estimate the target reproducibility. This appears to be very strict. In general, the reproducibility of a method is three times the repeatability. However, in ASTM D2810, the repeatability is 0.03 pH units and the reproducibility is 0.06 pH units (factor of 2 instead of 3). Also, the repeatability and reproducibility for this test is calculated by three times the repeatability times the square root of two (0.127 pH units), assuming that the sample material was not sufficient for most participants to perform the determination at least in duplicate.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as "not OK" or "suspect". The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER SAMPLE AND PER TEST

In this paragraph, the reported test results are discussed per sample and 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 methods are also in the table together with the original data. The abbreviations, used in these tables, are listed in appendix 4.

Sample #17640:

<u>Formaldehyde content (HPLC):</u> This determination was not problematic. Five statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the estimated requirements of ISO17226-1:08.

<u>Formaldehyde content (colorimetric)</u>: This determination was very problematic. No statistical outliers were observed. The reported test results appear to be trimodally distributed. Therefore, no significant conclusions were drawn.

Sample #17641:

- <u>pH of extract:</u> This determination was very problematic. Seven statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D2810:13.
- <u>pH of ten times diluted extract:</u> This determination was very problematic. Three statistical outliers were observed and two test results were excluded (see page 19 and 20). The calculated reproducibility after rejection of the suspect data is not at all in agreement with the requirements of ASTM D2810:13.

<u>Difference between pH of extract and pH ten times diluted extract:</u> One statistical outlier was observed and four test results were excluded, as the reported test result for "pH of extract" was a statistical outlier. Regretfully, no precision data are available for "the difference in pH", therefore no significant conclusions were drawn.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the calculated reproducibilities estimated from the target test methods and the reproducibilities as found for the group of participating laboratories.

The number of significant results, the average results, the calculated reproducibilities (standard deviation*2.8) and the target reproducibilities (ISO17226 and ASTM D2810), are compared in the next tables.

Parameter	unit	n	Average	2.8 * sd	R (target)
Formaldehyde (HPLC)	mg/kg	70	63.0	16.2	37.6
Formaldehyde (colorimetric)	mg/kg	53	191	206	(44)

Table 3: reproducibilities of tests on sample #17640

Reproducibility between brackets is estimated and should be used with due care.

Parameter	unit	n	Average	2.8 * sd	R (target)
pH of extract	-	87	3.40	0.27	0.13
pH of extract ten times diluted	-	73	4.09	0.35	0.13
Difference between pH's	-	73	0.69	0.27	n.a.

Table 4: reproducibilities of test on sample #17641

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

See also the discussions in paragraphs 4.1 and 5.

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2017 WITH PREVIOUS PTS

Deremeter	November	November	October	October	October
Parameter	2017	2016	2015	2014	2013
Number of reporting labs	102	106	116	108	48
Number of results reported	378	240	239	224	52
Number of statistical outliers	16	16	7	7	6
Percentage outliers	4.2%	6.7%	2.9%	3.1%	11.5%

Table 5: Comparison with previous proficiency tests

Surprisingly, the uncertainty of the 2017 PT on the HPLC determination of Formaldehyde in leather is much smaller than the uncertainty of the target test method. No improvement is visible for the colorimetric determination of Formaldehyde in leather and for the pH determination in comparison with the results in previous PTs. These targets are probably too strict to be met (see below table).

Deremeter	Nov	Nov	Oct	Oct	Oct	Est. from
Falameter	2017	2016	2015	2014	2013	target test method
Formaldehyde (HPLC)	9%	20%	23%	30%	22%	22% (17226-1)
Formaldehyde (colorimetric)	39%	26%	22%	33%	25%	9% (17226-2)
pH (undiluted)	2.8%	2.1%	2.6%	3.2%	n.e.	0.9% (D2810)
pH (10x diluted)	3.0%	2.3%	n.e.	n.e.	n.e.	0.9% (D2810)

Table 6: Development of relative uncertainties over the years

In this PT one of the samples from a previous PT (#13092 of iis13A04L) was re-used as sample #17640. An overview of the differences in results is given in below table:

		#	13092 in iis1	3A04L	#1	17640 in iis17	'A10
Parameter	unit	n	average	2.8 * sd	n	average	2.8 * sd
Formaldehyde (HPLC)	mg/kg	23	72.4	44.6	70	63.0	16.2
Formaldehyde (colorimetric)	mg/kg	19	165	115	53	191	206

Table 7: comparison of results of identical samples in iis13A04L and iis17A10

It is remarkable to see that the used testing material is stable for at least 4 years.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

The reported details of the analytical test methods that were used by the participants are listed in appendix 2. About 69% of the participating laboratories reported to be accredited for the determination of Formaldehyde and pH in leather.

For this PT, some analytical details of the determination: Formaldehyde (colorimetric) in leather were requested. A variety of analytical details was reported. Only a few laboratories reported to have corrected the absorbance measured for "Formaldehyde" with the absorbance measured for "interfering compounds". This is remarkable, as this is mentioned in the test method.

It appeared that from the reported answers no effect was observed on the reported test results for Formaldehyde (colorimetric) in sample #17641.

5 DISCUSSION

The standard test method for formaldehyde content is ISO17226. Part 1 and part 2 describe the determination of the formaldehyde content by extraction of the formaldehyde from the leather with a detergent solution. The difference between both parts of ISO17226 is the method of quantification. Quantification of the formaldehyde is done by HPLC in part 1 and by colorimetric analysis in part 2. Therefore part 2 is not selective for formaldehyde, whereas part 1 is selective. The test results from part 2 will in general be higher than the test results from part 1, which is the case with the leather sample in this PT. In the case of dispute part 1 shall be used in preference.

Looking at the reproducibility statements of both methods, it is remarkable that the reproducibility of the colorimetric method is smaller than the reproducibility of the HPLC method. Maybe the precision data for the colorimetric method were obtained with samples and/or conditions that did not influence the test (as the method describes that the test could for example be influenced by absorbances from the leather colouring).

Analytical Details Colorimetric method

In this PT several analytical details were asked on the report form for test method ISO17226-2 (colorimetric). Especially about corrections for absorbances found in reagents and acetyl acetone colouring components (see Appendix 2 for the analytical details). In total 72 participants completed this section of the report form. Regretfully, the reported details are inconsistent and therefore it was impossible to draw significant conclusions.

Sample #17640 in comparison to formaldehyde limits

When the results of this interlaboratory study were compared to the Standard "Limit of Harmful Matters in Leather" of the Chinese Leather Industry Committee Organization: GB20400-2006 and Öko-Tex Standard 100 (table 8), it may be noticed that not all participants would make identical decisions about the acceptability of the leather.

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

Table 8: Summary of limits from Standard GB20400:2006 and Öko-Tex 100

When using ISO17226 part 1, all reporting laboratories would reject this sample for category A. For category B, ten laboratories would reject this sample, while all other reporting laboratories would accept this sample. One of the reporting laboratories would also reject this sample for category C.

When using ISO17226 part 2, all reporting laboratories would reject this sample for category A and B (except one for category B only). Forty-eight laboratories would accept this sample for category C, while five of the reporting laboratories would reject this sample for category C.

Compared to other labelling standards different decisions would be made concerning the acceptance or rejection of the sample.

Sample #17641 was chosen to determine the pH only as the leather was not positive on formaldehyde. Two different test methods are available to determine the pH of leather, ASTM D2810 and ISO4045. The difference between the two test methods is the dilution of the extract (10 times) in ISO4045, in case the pH of the undiluted extract is not between 4.00 and 10.00. Three participants reported to have used ASTM D2810. These three participants measured only the pH of the extract. Ten participants reported to have used ISO4045 and reported a pH<4.00, but they did not report a test result for the difference between pH of extract and pH of a ten times diluted solution.

6 CONCLUSION

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

APPENDIX 1

Determination of Formaldehyde content (HPLC) on sample #17640; results in mg/kg

lab	method	value	mark	z(targ)	remarks
213	ISO17226-1	55.47		-0.56	
348	In house	58.52		-0.33	
362					
551	ISO17226-1	66.73		0.28	
2115	ISO17226-1	55.15		-0.58	
2120	15017226-1	61.42		-0.12	
2129	15017220-1	09.7	P(0.01)	199.22	
2131	ISO17226-1	60.0	1(0.01)	-0.22	
2137	ISO17226-1	62.86		-0.01	
2139					
2159					
2165	ISO17226-1	69.34		0.47	
2172	ISO17226-1	64.39		0.10	
2184	ISO17226-1	67.72		0.35	
2213	ISO17220-1 ISO17226-1	56 08		-0.50	
2235	ISO17226-1	91.0	R(0.01)	2.08	
2247	ISO17226-1	67.0	14(0.01)	0.30	
2255	ISO17226-1	75.3		0.92	
2256	ISO17226-1	65.08		0.15	
2273					
2290	ISO17226-1	62.63		-0.03	
2293	10017000 1			4.04	
2295	15017226-1	81 50.00		1.34	
2300	ISO17226-1	52.65		-0.76	
2330	ISO17226-1	60.44		-0.19	
2350	ISO17226-1	66.42		0.25	
2351					
2358	ISO17226-1	60.36		-0.20	
2360					
2364	100 17000 1				
2370	15017226-1	59.6		-0.25	
2378	ISO17226-1	62.42		-0.04	
2379	ISO17226-1	61.10		-0.14	
2380	ISO17226-1	60.6		-0.18	
2381					
2385	ISO17226-1	65		0.15	
2389	10017000 /				
2390	ISO17226-1	53.21		-0.73	
2449	15017226-1	60.93		-0.15	
2455	ISO17226-1	97 37	R(0.01)	2 56	
2459	ISO17226-1	59.690	14(0.01)	-0.25	
2460					
2477					
2488	100 (7000 /				
2489	ISO17226-1	63.04		0.00	
2492	ISO17226-1	58.2	P(0.01)	-0.36	
2495	ISO17226-1	54.068	K(0.01)	-0.67	
2497	ISO17226-1	78.421		1.15	
2501					
2504	ISO17226-1	61.8089		-0.09	
2511	ISO17226-1	64.438		0.11	
2519					
2532	ISO17226-1	62.75		-0.02	
2560	15017226-1	76.3 64.37		0.99	
2563	13017220-1				
2569	ISO17226-1	62.2		-0.06	
2572					
2587					
2590	ISO17226-1	55.01		-0.59	
2592	ISO17226-1	58.40		-0.34	
2039					
2043 2650					
2656	ISO17226-1	63.7	С	0.05	First reported 0.9
2666	ISO17226-1	64.3734		0.10	
2671	ISO17226-1	64.85		0.14	
2674	10.0 / = 0.0 /				
2695	15017226-1	62.21		-0.06	
2701 2711	ISO17220-1	58.4		-0.34	

lab	method	value	mark	z(targ)	remarks
2713	ISO17226-1	66.56		0.26	
2730	ISO17226-1	61.476		-0.11	
2737	ISO17226-1	63.15		0.01	
2741					
2743					
2756	ISO17226-1	61.7715		-0.09	
2769	ISO17226-1	60.3		-0.20	
2783					
2789	ISO17226-1	84.1	R(0.05)	1.57	
2792	ISO17226-1	63.76		0.06	
2806	ISO17226-1	61.0		-0.15	
3146					
3150	ISO17226-1	56.5		-0.48	
3154	ISO17226-1	62.62		-0.03	
3160	ISO17226-1	63.75		0.06	
3172	ISO17226-1	66.6		0.27	
3179	ISO17226-1	59.333		-0.27	
3197	ISO17226-1	74.2		0.83	
3209	ISO17226-1	75.03		0.90	
3210	In house	66.21		0.24	
3216					
3220	ISO17226-1	50.79		-0.91	
3228	ISO17226-1	67.13		0.31	
3237	ISO17226-1	58.30		-0.35	
3243	ISO17226-1	62.4		-0.04	
3248	ISO17226-1	60		-0.22	
	normality	suspect			
	n	70 [.]			
	outliers	5			
	mean (n)	63.004			
	st.dev. (n)	5.7952			
	R(calc.)	16.227			
	st.dev.(ISO17226-1:08)	13.4363			
	R(ISO17226-1:08)	37.622			





Determination of Formaldehyde content (colorimetric) on sample #17640; results in mg/kg

lah	method	valuo	mark	z(tara)	remarks
212	19017226.2	1/1 92	mark		TOTIGET OF
213	ISU17226-2	141.82			
362		200.04			
551	13017220-2	217.7			
2115					
2120					
2120	ISO17226-2	332.1			
2120	100 11 220 2				
2132	ISO17226-2	240			
2137	ISO17226-2	144.02			
2139	ISO17226-2	143.39			
2159	In house	212.75			
2165					
2172					
2184					
2213	ISO17226-2	231			
2235					
2241					
2247		NA			
2255	ISO17226-2	84.8			
2256	ISO17226-2	88.94			
2273	15017226-2	347.66			
2290	15017336 3	107 505			
2293	15017220-2	107.090			
2290	15017220-2	249			
2300	13017220-2	179.03			
2330	15017226-2	255 89			
2350	ISO17226-2	198.31			
2351					
2358	ISO17226-2	200.0			
2360					
2364	ISO17226-2	244			
2370	ISO17226-2	129.0			
2374					
2378					
2379	ISO17226-2	236.315			
2380	ISO17226-2	264.5			
2381					
2385					
2389	ISO17226-2	119.7			
2390	ISO17226-2	128.20			
2449	10017000 0				
2453	15017226-2	143.9			
2455	15017226.2	145 50			
2409	15017220-2	140.00			
2400	13017220-2	103.02			
2477	15017226-2	131			
2489	100 11 220 2				
2492					
2493					
2495					
2497	ISO17226-2	157.11			
2501	ISO17226-2	85.9	С		First reported 566.89
2504	ISO17226-2	153.6018			
2511					
2519	ISO17226-2	157.5			
2532					
2560	ISO17226-2	236.00			
2561	100 17000 0				
2563	ISO17226-2	386.2			
2569					
2012	15017226.2	206 14			
2507	13017220-2	300.14			
2590					
2092	GB/T10041	125 64			
2643	ISO17226-2	150 13			
2650	100 11 LL0-L				
2656					
2666					
2671	ISO17226-2	205.54			
2674					
2695					
2701	ISO17226-2	207.96			
2711					
2713	ISO17226-2	327.43			

lab	method	value	mark	z(targ)	remarks	
2730						
2737	ISO17226-2	249.40				
2741	ISO17226-2	160				
2743	ISO17226-2	150.7578				
2756						
2769	10017000 0					
2783	ISO17226-2	118.3980				
2789	15017226-2	134.3				
2792						
2000	19017226 2	225.0				
2150	13017220-2	225.0				
3154						
3160						
3172	ISO17226-2	197.2				
3179	ISO17226-2	133.58				
3197	ISO17226-2	80.4				
3209	ISO17226-2	225.52				
3210						
3216	ISO17226-2	22.1				
3220	ISO17226-2	256.10				
3228						
3237						
3243						
3248	ISO17226-2	240				
					Only accredited labs	Only accredited that follow test method
	normality	OK			0K	OK
	n	53			41	13
	outliers	0			0	0
	mean (n)	191.100			197.889	167.287
	st.dev. (n)	73.6659			72.6838	64.3375
	R(calc.)	206.264			203.515	180.145
	st.dev.(ISO17226-2:08)	(15.7941)			(16.3278)	(13.9222)
	R(ISO17226-2:08)	(44.223)			(45.718)	(38.982)





Determination of pH of extract on sample #17641; unitless results

lah	mothod	value	mark	z(tara)	romarks
242			IIIdi K	2(lary)	Tenidiks
213	1504045	3.37		-0.01	
340	1304045	3.43		1 40	
551	1304045	3.33		-1.49	
2115	ISO4045	3.5		2 24	
2120	ISO4045	3 375		-0.50	
2129	ISO4045	3.37		-0.61	
2131	ISO4045	3.45		1.14	
2132	ISO4045	3.61		4.66	
2137	ISO4045	3.50		2.24	
2139	ISO4045	3.345		-1.16	
2159	In house	3.29		-2.37	
2165	ISO4045	3.29		-2.37	
2172	ISO4045	3.35		-1.05	
2184	ISO4045	3.29		-2.37	
2213	ISO4045	3.40		0.05	
2235	1504045	3.58		4.00	
2241	1504045	3.30		-0.39	
2241	1904045	2 29		0.20	
2255	ISO4045	3 443		0.33	
2273	ISO4045	3.38		-0.39	
2290	ISO4045	3.407		0.20	
2293					
2295	ISO4045	3.38		-0.39	
2300	ISO4045	3.82	R(0.05)	9.28	
2301	ISO4045	3.20		-4.35	
2330	ISO4045	3.35		-1.05	
2350	ISO4045	3.39		-0.17	
2351	ISO4045	3.31		-1.93	
2358	ISO4045	3.44		0.92	
2360	1504045	3.30		-2.15	
2304	1504045	3.40		1 90	
2370	1304045	3.40		0.05	
2374	ISO4045	3 36		-0.83	
2379	ISO4045	3.36		-0.83	
2380	ISO4045	3.39		-0.17	
2381	ISO4045	3.40		0.05	
2385	ISO4045	3.325		-1.60	
2389	ISO4045	3.31		-1.93	
2390	ISO4045	3.4		0.05	
2449	ASTM D2810	3.37		-0.61	
2453					
2455	ISO4045	3.05	R(0.05)	-7.65	
2459	ISO4045	3.385		-0.28	
2460	1004045			0.20	
2477	1504045	3.30		-0.39	
2400	1304045	3.4		-1 /0	
2403	In house	3.40		0.05	
2493	III IIouse				
2495	ISO4045	3.43		0.71	
2497	ISO4045	3.59		4.22	
2501	ISO4045	3.25		-3.25	
2504	ISO4045	4.19	C,R(0.01)	17.42	First reported 3.89
2511	ISO4045	3.394		-0.09	
2519	ASTM D2810	3.36		-0.83	
2532	ISO4045	3.47		1.58	
2560	ISO4045	3.39		-0.17	
2561	ISO4045	3.71		6.86	
2003	1504045	3.20		-3.03	
2509	1304045	5.5		2.24	
2587					
2590	ISO4045	3.32		-1 71	
2592	ISO4045	3.35		-1.05	
2639	QB/T2724	3.38	С	-0.39	First reported 3.63
2643	ASTM D2810	3.33		-1.49	
2650	ISO4045	4.035	R(0.01)	14.01	
2656	ISO4045	3.43		0.71	
2666	1504045	3.40		0.05	
2671	1504045	3.34		-1.27	
2014	1304045 1904045	3.4U 2.415		0.05	
2090 27∩1	1504045	3.410 3.25		-1 05	
2711	ISO4045	3.40		0.05	
2713	ISO4045	3.845	R(0.05)	9.83	

lab	method	value	mark	z(targ)	remarks
2730	ISO4045	3.08	R(0.05)	-6.99	
2737	ISO4045	3.32		-1.71	
2741	ISO4045	3.3		-2.15	
2743	ISO4045	3.755	R(0.05)	7.85	
2756	INH-13	3.32		-1.71	
2769	ISO4045	3.577		3.94	
2783					
2789	ISO4045	3.65		5.54	
2792	ISO4045	3.27		-2.81	
2806	ISO4045	3.31		-1.93	
3146	ISO4045	3.34		-1.27	
3150	ISO4045	3.54		3.12	
3154					
3160	ISO4045	3.37		-0.61	
3172	ISO4045	3.51		2.46	
3179	ISO4045	3.28		-2.59	
3197	ISO4045	3.39		-0.17	
3209	ISO4045	3.353		-0.99	
3210	ISO4045	3.393		-0.11	
3216	ISO4045	3.35		-1.05	
3220	ISO4045	3.45		1.14	
3228	ISO4045	3.35		-1.05	
3237	ISO4045	3.66		5.76	
3243	ISO4045	3.45		1.14	
3248	ISO4045	3.6		4.44	
					Only ISO4045
	normality	not OK			not OK
	n	87			80
	outliers	7			7
	mean (n)	3.398			3.402
	st.dev. (n)	0.0947			0.0971
	R(calc.)	0.265			0.272
	st.dev.(D2810:13)	0.0455			0.0455
	R(D2810:13)	0.127			unknown
4.3 T					
					ж





Determination of pH of ten times diluted extract on sample #17641; unitless results

lah	method	value	mark	z(targ)	remarks
212	1504045	4.07	mark	_0 52	remaine
213	1304043	4.07		-0.52 0 = 0	
340	1304043	4.1Z		0.00	
302	1504045	4.29		4.32	
551	1504045	4.05		-0.96	
2115	1504045	4.1		0.14	
2120	1504045	4.272		3.93	
2129	100 1015				
2131	ISO4045	4.24		3.22	
2132	ISO4045	4.32		4.98	
2137	ISO4045	4.20		2.34	
2139	ISO4045	4.190		2.12	
2159					
2165	ISO4045	3.96		-2.93	
2172	ISO4045	4.05		-0.96	
2184	ISO4045	3.83		-5.79	
2213	ISO4045	4.00		-2.05	
2235	ISO4045	4.11		0.36	
2241	ISO4045	4.02		-1.61	
2247					
2255	ISO4045	4.06		-0.74	
2256	ISO4045	4.012		-1.79	
2273	ISO4045	4.30		4.54	
2290	ISO4045	4.067		-0.58	
2293					
2295	ISO4045	4.0		-2.05	
2300	ISO4045	7.21	R(0.01)	68.56	
2301					
2330	ISO4045	4.05		-0.96	
2350	ISO4045	4.17		1.68	
2351	ISO4045	4.01		-1.83	
2358	ISO4045	4.34		5.42	
2360	ISO4045	4.00		-2.05	
2364	ISO4045	4.10		0.14	
2370					
2374	ISO4045	4.10		0.14	
2378	ISO4045	4.06		-0.74	
2379	ISO4045	4.07		-0.52	
2380	ISO4045	4.12		0.58	
2381	ISO4045	4.20		2.34	
2385	ISO4045	4.020		-1.61	
2389	ISO4045	4.16		1.46	
2390	ISO4045	4.1		0.14	
2449					
2453					
2455	ISO4045	3.53	R(0.01)	-12.39	
2459	ISO4045	4.12	С	0.58	First reported 5.565
2460					
2477	ISO4045	4.22		2.78	
2488	ISO4045	4.3		4.54	
2489	ISO4045	3.98		-2.49	
2492		4.10		0.14	
2493					
2495	ISO4045	4.14		1.02	
2497	ISO4045	3.99		-2.27	
2501	ISO4045	3.92	_	-3.81	
2504	ISO4045	4.94	C,ex	18.62	First reported 4.88, test result excluded as "pH of extract" was outlier"
2511	ISO4045	4.128		0.76	
2519					
2532	ISO4045	4.08		-0.30	
2560	ISO4045	4.03		-1.39	
2561	ISO4045	4.27		3.88	
2563	ISO4045	3.96		-2.93	
2569	ISO4045	4.2		2.34	
2572					
2587					
2590	ISO4045	4.01		-1.83	
2592	ISO4045	4.01	_	-1.83	
2639	QB/T2724	4.07	С	-0.52	First reported 4.32
2643					
2650					
2656					
2666	ISO4045	3.90		-4.25	
2671	100 (0)-				
2674	ISO4045	4.15		1.24	
2695	ISO4045	4.020		-1.61	
2701	100 10				
2711	ISO4045	4.1		0.14	
2713					

lab	method	value	mark	z(targ)	remarks
2730	ISO4045	3.56	R(0.01)	-11.73	
2737	ISO4045	4.03		-1.39	
2741					
2743	ISO4045	4.320	ex	4.98	Test result excluded as "pH of extract" was outlier"
2756	INH-13	3.88		-4.69	
2769	ISO4045	4.360		5.86	
2783					
2789	ISO4045	4.43		7.40	
2792	ISO4045	4.08		-0.30	
2806	ISO4045	4.03		-1.39	
3146	ISO4045	4.07		-0.52	
3150	ISO4045	4.40		6.74	
3154					
3160	ISO4045	4.03		-1.39	
3172					
3179	ISO4045	3.91		-4.03	
3197	ISO4045	4.03		-1.39	
3209	ISO4045	4.036		-1.26	
3210	ISO4045	3.874		-4.83	
3216	ISO4045	4.05		-0.96	
3220	ISO4045	4.10		0.14	
3228	ISO4045	4.00		-2.05	
3237	100.00				
3243	ISO4045	4.05		-0.96	
3248					
	normality	OK			
	n	73			
	outliers	3 (+2excl)			
	mean (n)	4.093			
	st.dev. (n)	0.1246			
	R(calc.)	0.349			
	st.dev.(D2810:13)	0.0455			
	R(D2810:13)	0.127			
	. ,				





Determination of difference between pH of extract and pH of ten times diluted extract on sample #17641; unitless results

lah	mothod	value	mark	z(tora)	Pomarke
			IIIdi K	Z(lary)	Reliidiks
213	1504045	0.7			
340	1504045	0.69			
302	100 40 45				
551	1504045	0.61			
2115	1504045	0.6			
2120	1504045	0.897			
2129					
2131	ISO4045	0.79			
2132	ISO4045	0.71			
2137	ISO4045	0.7			
2139	ISO4045	0.845			
2159					
2165	ISO4045	0.65			
2172	ISO4045	0.70			
2184	ISO4045	0.54			
2213	ISO4045	0.60			
2235	ISO4045	0.54			
2241	ISO4045	0.64			
2247					
2255	ISO4045	0.68			
2256	ISO4045	0.569			
2273	ISO4045	0.92			
2290	ISO4045	0.66			
2293					
2295	ISO4045	0.62			
2300	ISO4045	3.4	R(0.01)		
2301					
2330	ISO4045	0.69			
2350	ISO4045	0.78			
2351	ISO4045	0.70			
2358	ISO4045	0.90			
2360	ISO4045	0.70			
2364	ISO4045	0.7			
2370	ISO4045	0.81			
2374	ISO4045	0.7			
2378	ISO4045	0.70			
2379	ISO4045	0.7			
2380	ISO4045	0.73			
2381	ISO4045	0.80			
2385	ISO4045	0.695			
2389	ISO4045	0.85			
2390	ISO4045	0.7			
2449					
2453					
2455	ISO4045	0.48	ex		Test result excluded as "pH of extract" was outlier"
2459	ISO4045	0.74	С		First reported 2.18
2460					
2477	ISO4045	0.84			
2488	ISO4045	0.9			
2489	ISO4045	0.65			
2492		0.70			
2493					
2495	ISO4045	0.71			
2497	ISO4045	0.4			
2501	ISO4045	0.67			
2504	ISO4045	0.99	ex		Test result excluded as "pH of extract" was outlier"
2511	ISO4045	0.734			
2519					
2532	ISO4045	0.61	-		
2560	ISO4045	0.64	С		Reported -0.64, iis changed it to absolute figure
2561	ISO4045	0.56			
2563	ISO4045	0.7			
2569		0.7			
2572					
2587					
2590	ISO4045	0.69			
2592	ISO4045	0.66			
2639	QB/T2724	0.69			
2643					
2650					
2656	100 in :-				
2666	1504045	0.50			
2671	100 40 45	 0 7 E			
2674	1504045	0.75			
2695	1504045	0.605			
2701	1004045		<u> </u>		First reported 4.1
2/11	1504045	0.7	C		гизтеропеа 4.1
2/13					

lab	method	value	mark	z(targ)	Remarks
2730	ISO4045	0.48	ex		Test result excluded as "pH of extract" was outlier"
2737	ISO4045	0.71			
2741					
2743	ISO4045	0.565	ex		Test result excluded as "pH of extract" was outlier"
2756	INH-13	0.58			
2769	ISO4045	0.783			
2783					
2789	ISO4045	0.78			
2792	ISO4045	0.81			
2806	ISO4045	0.72			
3146	ISO4045	0.73			
3150	ISO4045	0.86			
3154					
3160	ISO4045	0.66			
3172					
3179	ISO4045	0.63			
3197	ISO4045	0.64			
3209	ISO4045	0.683			
3210	ISO4045	0.481			
3216	ISO4045	0.7			
3220	ISO4045	0.65			
3228	ISO4045	0.65	С		First reported -0.65
3237					
3243	ISO4045	0.65			
3248					
	normality	ОК			
	n	73			
	outliers	1 (+4 excl)			
	mean (n)	0.694			
	st.dev. (n)	0.0979			
	R(calc.)	0.274			
	st.dev.(lit)	n.a.			
	R(lit)	n.a.			





APPENDIX 2 Analytical Details ISO17226-2

labnrs	1. Is your laboratory accredited in accordance with ISO/IEC17025?	Were the reagents checked for absence of formaldehyde?	If yes, please give absorbance of reagents	Was for "released Formaldehyde" the sample tested for other compounds which may cause a coloring with acetylacetone?	If yes, please give absorbance	Was the absorbance of the solution for the determination "released Formaldehyde" corrected for above absorbances?	If yes, please give absorbance of the sample solution before and after correction	Remarks on Additional Questions:
213	Yes	Yes	0,0051					
348	Yes	Yes	0.0271	Yes	0.723	No		
362	Yes	No						
551	Yes							
2115	Yes	Yes						
2120	No	Yes	Is depending on several circumstances					
2129	Yes	No	0,0029	No		No		
2131	Yes	No		No		No		
2132	Yes	Yes	0.0008	Yes	0.0907	Yes	Before 0.5927, After 0.5075	
2137	Yes	Yes	0.0004	Yes	0.0837	Yes	Before 0.84645 After 0.76365	
2139	No	No		No		No		
2159	Yes	No		No		No		-
2165	Yes							
2172	Yes	No		No		No		
2184								
2213	Yes	Yes						
2235	Yes							
2241	Yes	Yes						
2247	Yes	Yes	# 17635 :0.003; # 17636 :0.012;	No		No		
2255	Yes	No		No		No		
2256	Yes	Yes	0.001	Yes	0.3982	Yes	Before: 0.8810, After: 0.4828	
2273	Yes	Yes	0.034	No		No		
2290	Yes	Yes						
2293	Yes	Yes	-0.006	Yes	0.184	No		
2295	Yes	Yes	1nm	Yes	2.67nm	No		
2300	Yes	Yes	0.0	No		No		

				Was for "released		Was the absorbance		
	1. Is your			Formaldehyde" the		of the solution for the		
	laboratory	Were the		sample tested for		determination		
	accordance	checked for		which may cause a		Formaldehvde"	If ves, please give absorbance of	
	with	absence of	If yes, please give	coloring with	If yes, please give	corrected for above	the sample solution before and	Remarks on Additional
labnrs	ISO/IEC17025?	formaldehyde?	absorbance of reagents	acetylacetone?	absorbance	absorbances?	after correction	Questions:
2301								
2330	Yes	Yes	0.0257	No		No		
2350	No	No		No		No		
2351								
2358	Yes	Yes	0.052	No		No		
2360								
2364	Yes	Yes	0.0251	No		No		
2370	Yes	Yes	0.008Abs	No		No		
2374	Yes	No		No		No		
2378								
2379	No	Yes	0.0231	Yes	0.0006	No		
2380	Yes	No		No		No		
2381								
2385	Yes							
2389								
2390	Yes	Yes	0.000	No		No		
2449								
2453	No	Yes	0.033					
2455	Yes							
2459	Yes	No		No		No		
2460	Yes	Yes	0.017	Yes	0.339	No		
2477								
2488	Yes	Yes						
2489	Yes							
2492	Yes	No		No		No		
2493	Yes							
2495	Yes	No		No		No		
2497	Yes	No		No		No		
2501	Yes	Yes	Dimedone: 0.001	Yes	sodium dodecyl sulfate: 0.007	Yes	Before: 0.303, After: 0.295	The sample was diluted by 1000 times.

1. Is your baceredided in respinseNearther sample tested or sample solution bein sample tested or with accredided in test consolution bein test con					Was for "released		Was the absorbance		
laboratory accordance accordanceVere the sagents accordance of accordance of besched for types, please give absorbance of types, please give absorbance/determination formicabelyde' formicabelyde'determination formicabelyde' formicabelyde' formicabelyde'determination formicabelyde' formicabelyde'determination formica		1. Is your			Formaldehyde" the		of the solution for the		
accreated in accreated in absence of absence of absence of absence of absence of absence of absence of absence of absence of absence of eagens accreated in absence of absence of reagens accreated in absence of absence of reagens accreated in absence of absence of reagens accreated in absence of absence of absence of absence of reagens accreated in absence of absence of reagens accreated in accreated in absence of reagens accreated in absence of reagens absence of reagens accreated in absence of reagens accreated in accreated in accrea		laboratory	Were the		sample tested for		determination		
with laborsbisemo of isolic(710257bisemo of isolic(7102577Bisemo of isolic(7102577Bisemo of isolic(71025777Bisemo of isolic(7		accredited in	reagents		other compounds		"released Formaldebyde"	If yes, please give absorbance of	
IsburgISO/IEC17025?formaldehyde?alsorbanee of reagentsacetylacetone?alsorbanee?alter correctionQuestions:2504YesYes0.0038290NoNoNoNo25112519NoNo2519NoNoNoNo2520YesNoNANoNo2560YesNoN/ANoNo2561YesNoN/ANoNo2563YesNoNAYesYes2564YesNoYes <t< td=""><td></td><td>with</td><td>absence of</td><td>If yes, please give</td><td>coloring with</td><td>If yes, please give</td><td>corrected for above</td><td>the sample solution before and</td><td>Remarks on Additional</td></t<>		with	absence of	If yes, please give	coloring with	If yes, please give	corrected for above	the sample solution before and	Remarks on Additional
2504YesYes0.0038290NoNoNoIndependent2511In-In-In-IndependentIndependentIndependent2513NoNoIndependentNoIndependentIndependent2532YesNoNANoIndependentIndependent2560YesNoNANoNoIndependent2561YesYesYesIndependentYesIndependent2563YesNoIndependentYesIndependentIndependent2572In-IndependentIndependentIndependentIndependent2584YesNoIndependentIndependentIndependent2587YesNoIndependentIndependentIndependent2587YesNoIndependentIndependentIndependent2587YesNoIndependentIndependentIndependent2587YesNoIndependentIndependentIndependent2587YesNoIndependentIndependentIndependent2587YesNoIndependentIndependentIndependent2589YesNoIndependentIndependentIndependent2590YesNoIndependentIndependentIndependent2631YesNoIndependentIndependentIndependent2633YesNoIndependentIndependentIndepend	labnrs	ISO/IEC17025?	formaldehyde?	absorbance of reagents	acetylacetone?	absorbance	absorbances?	after correction	Questions:
26112519NoNoNoNoNoNo2532YesNoNoNoNo2560YesNoNANoNoN/A<	2504	Yes	Yes	0.0038290	No		No		
2519NoNoNoNoNoNo2529YesNoN/ANoNoNoN/A2560YesNoN/ANoNoN/A2561YesYesIYesNoN/A2563YesNoNaYesIN/A2564YesNoYesIN/AI2569YesNoIYesIII2569YesNoIYesIII2572IIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIII	2511								
2532VesNoNoNoNoNoNoNoNoNo2560YesYesYesYesYesYesYesYesYes2561YesNoYesYesYesYesYesYesYes2562YesNoYesYesYesYesYesYesYesYes2572YesNoYesYesYesYesYesYesYesYesYes2583YesNoYes	2519	No	No		No		No		
2560YesNoNoNoNoNoNoNoNA2561YesYesYesIYesII<	2532	Yes	No		No		No	-	-
2561YesYesYesYesYesYes 2563 YesNoIYesYesII 2569 YesIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIII	2560	Yes	No	N/A	No		No		N/A
263 Yes No Yes Yes Yes Image: Marcine Mar	2561	Yes	Yes		Yes		Yes		
2669YesImage: Additional system of the s	2563	Yes	No		Yes		Yes		
2572	2569	Yes							
2887YesNoNoNoNo2590YesNoIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIII	2572								
2590YesNo2592YesNo2639YesYes0.036Yes0.030No<	2587	Yes	No		No		No		
2592YesNoIndext (1)2639YesYes0.036Yes0.030NoIndext (1)Indext (1)2643YesYes0.0200NoIndext (1)Yesbefore : 0.8113, after: 0.7835Indext (1)2650Indext (1)Indext (1)Indext (1)Indext (1)Indext (1)Indext (1)2650NoNoIndext (1)Indext (1)Indext (1)Indext (1)Indext (1)Indext (1)2666YesNoIndext (1)NoIndext (1)Indext (1)Indext (1)Indext (1)Indext (1)2674Indext (1)Indext (1)Indext (1)Indext (1)Indext (1) <td>2590</td> <td>Yes</td> <td>No</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>	2590	Yes	No						
2639YesYes0.036Yes0.030NoNo2643YesYes0.0200NoYesbefore : 0.8113, after: 0.783526502656NoNoNoNo2666YesNoNoNoNo267126742675YesNo26742675YesNo26742675YesNo27012713NoYes0.0031YesBefore 0.1621x10 dilute, after 0.0434x10 dilute	2592	Yes	No						
2643 Yes Yes before : 0.8113, after: 0.7835 2650 2656 No No No No 2666 Yes No No No 2666 Yes No No No 2671 No No 2674 2674 2674 <td< td=""><td>2639</td><td>Yes</td><td>Yes</td><td>0.036</td><td>Yes</td><td>0.030</td><td>No</td><td></td><td></td></td<>	2639	Yes	Yes	0.036	Yes	0.030	No		
26502656NoNoNoNoNoNoInternational State2666YesNoInternational StateNoNoNoInternational State2671International StateInternational StateInternational StateInternational State2674International StateInternational StateInternational StateInternational StateInternational State2695YesNoInternational StateInternational StateInternational StateInternational StateInternational State2701International StateInternational StateInternational StateInternational StateInternational StateInternational State2713NoYes0.0031YesYesYesNoInternational StateInternational State	2643	Yes	Yes	0.0200	No		Yes	before : 0.8113, after: 0.7835	
2656NoNoNoNoNo2666YesNoNoNoNoImage: Second S	2650								
2666 Yes No No No No 2671 2674 2674 2674 2695 Yes No No 2701 2701 2711 2713 No Yes 0.0031 Yes Yes No4x10 dilute, after 0.0434x10 dilute	2656	No	No		No		No		
2671 2674 <td>2666</td> <td>Yes</td> <td>No</td> <td></td> <td>No</td> <td></td> <td>No</td> <td></td> <td></td>	2666	Yes	No		No		No		
2674 2695 Yes No No No No Image: Second Seco	2671								
2695 Yes No No No No 2701 2711 2713 No Yes 0.0031 Yes Yes Before 0.1621x10 dilute, after 0.0434x10 dilute	2674								
2701 2711 2713 No Yes 0.0031 Yes Yes Yes Dot 34x10 dilute, after 0.0434x10 dilute	2695	Yes	No		No		No		
2711 2713 No Yes 0.0031 Yes Yes Before 0.1621x10 dilute, after 0.0434x10 dilute 0.0434x10 dilute	2701								
2713 No Yes Yes Yes Before 0.1621x10 dilute, after 0.0434x10 dilute 0.0434x10 dilute 0.0434x10 dilute 0.0434x10 dilute	2711								
	2713	No	Yes	0.0031	Yes		Yes	Before 0.1621x10 dilute, after	
2730 No No	2730	No	No						
2737 Yes No No No	2737	Yes	No		No		No		
2741 Yes Yes 0.003 Yes 0.24 Yes Before: 5.04: after: 4.8 /	2741	Yes	Yes	0.003	Yes	0.24	Yes	Before: 5.04: after: 4.8	/
all measured against a				all measured against a					
blank constituted by all	0740			blank constituted by all					
Z/43 Yes The reagents NO	2/43	Yes	Yes	the reagents	NO		N0		
2756 Yes Yes observed for reagents	2756	Yes	Yes	observed for reagents					

	1. Is your laboratory accredited in	Were the		Was for "released Formaldehyde" the sample tested for other compounds		Was the absorbance of the solution for the determination		
	accordance	checked for		which may cause a		Formaldehyde"	If yes, please give absorbance of	
lahara	with	absence of	If yes, please give	coloring with	If yes, please give	corrected for above	the sample solution before and	Remarks on Additional
	150/IEC17025?	Tormaldenyde?	absorbance of reagents	acetylacetone?	absorbance	absorbances?		
2769	INO							
2783	NO	NO		NO		NO		Absorbance of reagents
2789	No	Yes	0 1114	Yes	0.0193	Yes	Before correction: 0,7402 After	(0,1114) is corrected by the spectophotometer and considered as base zero
2792	No							
2806	No							
3146	Yes	No		No		No		blank check
3150	Yes	No		No		No		
3154	Yes							
3160	No	No		No				
3172	Yes							
3179	Yes	Yes	-0,003	Yes	-0,004	No		
3197	Yes	Yes	0.0002	Yes	0.0004	No		
3209	Yes	No		No		No		
3210	Yes	Yes						
3216								
3220	Yes	Yes	Dimedone - Absorbance is 0.107	No		Yes	Before correction - 0.792; After correction - 0.685	
3228	Yes							
3237	Yes	No						
3243	Yes	No		No		No		
3248	Yes	Yes	0	Yes	0	Yes	0.0638	

APPENDIX 3

Number of participants per country

4 labs in BANGLADESH 1 lab in BRAZIL 1 lab in BULGARIA 1 lab in CAMBODIA, Kingdom of 1 lab in ETHIOPIA 3 labs in FRANCE 8 labs in GERMANY 1 lab in GUATEMALA 1 lab in HUNGARY 6 labs in HONG KONG 8 labs in INDIA 2 labs in INDONESIA 12 labs in ITALY 5 labs in SOUTH KOREA 2 labs in MEXICO 1 lab in MOROCCO 17 labs in P.R. of CHINA 4 labs in PAKISTAN 3 labs in PORTUGAL 5 labs in SPAIN 1 lab in SWITZERLAND 1 lab in TAIWAN R.O.C. 2 labs in THAILAND 1 lab in TUNISIA 6 labs in TURKEY 2 labs in U.S.A. 1 lab in UNITED KINGDOM 3 labs in VIETNAM

APPENDIX 4

Abbreviations:

C = final result after checking of first reported suspect tes	t result
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- 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
- n.a. = not applicable
- n.d. = not detected
- n.e. = not evaluated
- W = test result withdrawn on request of participant
- ex = test result excluded from the statistical evaluation

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