Results of Proficiency Test Formaldehyde and pH in Leather November 2020

Organized by: Institute for Interlaboratory Studies
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CONTENTS

1 INTRODUCTION 3

2 SET UP 3

2.1 QUALITY SYSTEM 3

2.2 PROTOCOL 3

2.3 CONFIDENTIALITY STATEMENT 3

2.4 SAMPLES 4

2.5 ANALYZES 5

3 RESULTS 6

3.1 STATISTICS 6

3.2 GRAPHICS 7

3.3 Z-SCORES 7

4 EVALUATION 8

4.1 EVALUATION PER SAMPLE AND PER TEST 8

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES 9

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2020 WITH PREVIOUS PTS 10

4.4 EVALUATION OF THE ANALYTICAL DETAILS 10

5 DISCUSSION 11

6 CONCLUSION 12

Appendices:

1. Data, statistical and graphic results 13
2. Analytical details 23
3. Number of participants per country 27
4. Abbreviations and literature 28

1 INTRODUCTION

Since 2013 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Formaldehyde and pH in Leather every year. During the annual proficiency testing program 2020/2021 it was decided to continue the proficiency tests for the analyzes of Formaldehyde content and pH in Leather.
In this interlaboratory study 100 laboratories in 28 different countries registered for participation in the PT Formaldehyde in Leather and 81 laboratories in 26 different countries registered for participation in the PT pH on Leather. See appendix 3 for the number of participants per country.
In this report the results of the Formaldehyde and pH in Leather proficiency tests 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. Depending on the registration it was decided to send one leather sample labelled #20715 positive on Formaldehyde and/or one leather sample labelled #20720 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 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

The batch selected for Formaldehyde determination was a grinded dark red Leather. After homogenization 130 bags were filled with approximately 6 grams and labelled #20715. Each subsample was wrapped in Aluminum foil and again packed in a bag.
The homogeneity of the subsamples was checked by the determination of the Formaldehyde content in accordance with an ISO17226-1 test method on 8 stratified randomly selected subsamples.

	Formaldehyde in mg/kg
Sample #20715-1	66.740
Sample #20715-2	63.010
Sample #20715-3	64.013
Sample #20715-4	65.401
Sample #20715-5	66.158
Sample #20715-6	66.651
Sample #20715-7	66.140
Sample #20715-8	67.977

Table 1: homogeneity test results of subsamples #20715

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)	4.454
reference test method	ISO17226-1:08
0.3*R (reference test method)	11.836

Table 2: evaluation of the repeatability of subsamples #20715

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

The batch selected for the pH determination was a brown Leather which was grinded. After homogenization 130 bags were filled with approximately 10 grams and labelled #20720.
The homogeneity of the subsamples was checked by the determination of the pH in accordance with ISO4045 on 7 stratified randomly selected subsamples.

	pH of extract
Sample #20720-1	2.79
Sample #20720-2	2.77
Sample #20720-3	2.81
Sample #20720-4	2.81

	pH of extract
Sample #20720-5	2.80
Sample #20720-6	2.78
Sample #20720-7	2.79

Table 3: homogeneity test results of subsamples #20720

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.

	pH of extract
r (observed)	0.04
reference test method	ASTM D2810:18
0.3*R (reference test method)	0.04

Table 4: evaluation of the repeatability of subsamples #20720

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

Depending on the registration to each of the participants one sample labelled #20715 and/or one sample labelled #20720 was sent on October 21, 2020.

2.5 ANALYZES

The participants were requested to determine Formaldehyde on samples #20715 and to determine pH of extract, pH of ten times diluted extract and the difference between the two pH measurements on sample #20720.
It was also requested to report if the laboratory was accredited for the requested components that were determined and to report some analytical details.

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 test 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 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.

According to ISO5725 the original test results per determination were submitted to Dixon’s, Grubbs’ 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 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. 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 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. In some cases, a reproducibility based on former iis proficiency tests could be 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-use.
The z-scores were calculated according to:

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

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:

	$\lvert z \rvert$	< 1	good
1 <	$\lvert z \rvert$	< 2	satisfactory
2 <	$\lvert z \rvert$	< 3	questionable
3 <	$\lvert z \rvert$		unsatisfactory

4 EVALUATION

In this interlaboratory study some problems were encountered with the dispatch of the samples due to the COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with one week.
For the Formaldehyde proficiency test three laboratories reported the test results after the extended closing date and eight laboratories did not report any test results.
For the pH proficiency test three laboratories reported the test results after the extended reporting date and six laboratories did not report any test results.

In total over the two proficiency tests 106 reporting laboratories reported 356 numerical test results. Observed were 14 outlying test results, which is 3.9% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal Gaussian distribution. These are referred as “not OK” or “suspect”. The statistical evaluation of these data 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 test methods are also in the tables together with the reported test results 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. Therefore, the target reproducibilities were estimated from the reproducibility data as mentioned in the annexes of ISO17226-1 and ISO17226-2.

Test methods ASTM D2810 and ISO4045 are considered to be the official test methods for the determination pH on Leather. 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. As a rule of thumb, 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 (thus factor of 2 instead of 3). Also, the repeatability and reproducibility are based on the values of duplicate measurements. Therefore, in this report the reproducibility for this test is calculated by three times the repeatability times the square root of two (0.127 pH units), assuming that the sample material was not sufficient for most participants to perform the determination at least in duplicate.

Sample #20715

Formaldehyde content (HPLC): This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in good agreement with the estimated requirements of ISO17226-1:08.

Formaldehyde content (colorimetric): This determination was problematic for a number of laboratories. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated requirements of ISO17226-2:08.

Sample #20720

pH of extract: This determination may be problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated requirements of ASTM D2810:18.

pH of ten times diluted extract: This determination was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the estimated requirements of ASTM D2810:18.

Difference between pH of extract and pH ten times diluted extract: This determination may be problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated requirements of ASTM D2810:18.

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, the calculated reproducibility (2.8*standard deviation) and the target reproducibility are compared in the next two tables.

Component	unit	n	average	2.8 * sd	R(target)
Formaldehyde (HPLC)	mg/kg	80	61.6	20.4	36.7
Formaldehyde (colorimetric)	mg/kg	52	45.6	9.7	12.2

Table 5: reproducibilities of tests on sample #20715

Parameter	unit	n	average	2.8 * sd	R(target)
pH of extract	-	72	3.51	0.22	0.13
pH of extract ten times diluted	-	71	4.14	0.38	0.13
Difference between pH	-	67	0.62	0.24	0.18

Table 6: reproducibilities of test on sample #20720

Without further statistical calculations, the group of participating laboratories have some difficulties with the determination of the pH but have no problems with the Formaldehyde analyzes. See also the discussions in paragraphs 4.1 and 5.

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2020 WITH PREVIOUS PTS

	November 2020	November 2019	November 2018	November 2017	November 2016
Number of reporting laboratories	106	136	114	102	106
Number of test results	356	441	396	378	240
Number of statistical outliers	14	17	12	16	16
Percentage of statistical outliers	3.9%	3.9%	3.0%	4.2%	6.7%

Table 7: comparison with previous proficiency tests

The performance of the determinations of the proficiency test was compared expressed as relative standard deviation (RSD) of the PTs, see below table.

Parameter	November 2020	November 2019	November 2018	November 2017	2013-2016	Target
Formaldehyde (HPLC)	12%	12%	23%	9%	20-30%	22%
Formaldehyde (colorimetric)	8%	8%	17%	39%	22-33%	9%
pH of extract	2.3%	2.5%	1.7%	2.8%	2.1-3.2%	0.9%
pH of extract ten times diluted	3.3%	2.3%	2.3%	3.0%	2.3%	0.9%

Table 8: development of uncertainties over the years

The uncertainties for 2020 are equal to the uncertainties of 2019 for the HPLC and Colorimetric determinations of Formaldehyde in Leather. Both meet again the estimated targets from the reference test methods.

For the pH determination the group did improve for pH of extract but did not improve for pH of extract ten times diluted with the uncertainties of the previous proficiency tests. The pH determination is not at all in agreement with the uncertainties as mentioned in the respective reference test methods. These targets are most likely too strict to be met.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

The reported details of the analytical test methods are listed in appendix 2.
About 85% of all laboratories reported to be accredited for the determination of Formaldehyde in Leather and about 70% of all the laboratories reported to be accredited for the determination of pH on Leather.

For this PT a few analytical details of the determination of Formaldehyde in Leather was asked. Approximately 60% of all laboratories used 2 grams as intake as prescribed in ISO17226 and 50% of all participants completed the test within one or two days.
No impact by sample intake or the duration of the tests was observed for this sample.

For the determination of pH on Leather also some analytical details were asked.
About 65% of the participants reported to have used 5 grams for intake. No effect of intake or using an additional step to wet the Leather was observed for this sample.

5 DISCUSSION

The Formaldehyde test method ISO17226 part 1 and part 2 describe both the determination of the Formaldehyde content by extraction of the Formaldehyde from the 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 by colorimetric analysis in part 2. 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 should be higher on average than the test results from part 1. Remarkably, this is not observed in PT sample #20715.

Sample #20715 compared 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 Oeko-Tex Standard 100 (see table 9), 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 9: summary of limits from Standard GB20400:2006 and Oeko-Tex 100

When looking at the HPLC test results all reporting laboratories would reject this sample for category A. For category B again all reporting laboratories would accept this sample except five laboratories. All of the reporting laboratories would accept this sample for category C. When looking at the Colorimetric test results all reporting laboratories would reject this sample for category A. For category B all laboratories would accept this sample except two laboratories. All of the reporting laboratories would accept this sample for category C.

Two different test methods are available to determine the pH on Leather, ASTM D2810 and ISO4045. The difference between both test methods is the dilution of the extract (10 times) in ISO4045 when the pH of the undiluted extract is not between 4.00 and 10.00. Three 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 observed variation for the Formaldehyde content (both methods) in this interlaboratory study did improve compared with previous PTs.
The observed variation for the pH in this interlaboratory study was in line with the previous proficiency tests.

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.

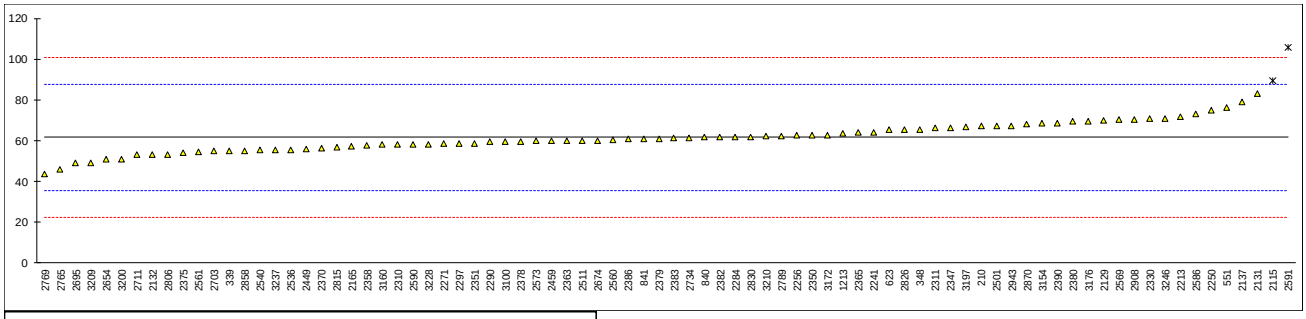
APPENDIX 1

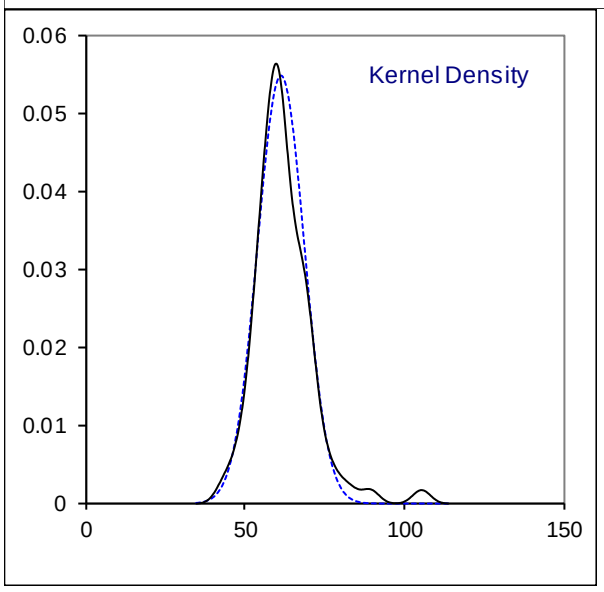
Determination of Formaldehyde content (HPLC) on sample #20715; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210	GB/T19941-1	67.16		0.42	
230		-----		-----	
339	ISO1726Mod.	55		-0.50	
348	In house	65.4		0.29	
362		-----		-----	
551	ISO17226-1	76.4210		1.13	
623	ISO17226-1	65.19		0.27	
840	ISO17226-1	61.65		0.00	
841	ISO17226-1	60.77		-0.06	
1213	ISO17226-1	63.65		0.16	
2115	ISO17226-1	89.5	R(0.05)	2.13	
2129	ISO17226-1	70		0.64	
2131	In house	83		1.63	
2132	ISO17226-1	53.1		-0.65	
2137	ISO17266-1	78.92		1.32	
2165	ISO17226-1	57.3		-0.33	
2213	ISO17226-1	71.8		0.78	
2241	ISO17226-1	63.83		0.17	
2250	ISO17226-1	74.71		1.00	
2256	ISO17226-1	62.5975		0.08	
2265		-----		-----	
2271	ISO17226-1	58.46		-0.24	
2284	ISO17226-1	61.94		0.03	
2290	ISO17226-1	59.5		-0.16	
2297	ISO17226-1	58.6		-0.23	
2310	ISO17226-1	58		-0.27	
2311	ISO17226-1	66.287		0.36	
2330	ISO17226-1	70.92		0.71	
2347		66.3		0.36	
2350	ISO17226-1	62.72		0.09	
2351	ISO17226-1	58.71		-0.22	
2357		-----		-----	
2358	ISO17226-1	57.66		-0.30	
2363	ISO17226-1	60		-0.12	
2365	ISO17226-1	63.8		0.17	
2366	ISO17226-1	-----		-----	
2370	ISO17226-1	56.12		-0.42	
2375	ISO17226-1	54.0		-0.58	
2378	ISO17226-1	59.68		-0.15	
2379	ISO17226-1	61.03		-0.04	
2380	ISO17226-1	69.6		0.61	
2381		-----		-----	
2382	ISO17226-1	61.7		0.01	
2383	ISO17226-1	61.4		-0.02	
2386	In house	60.66		-0.07	
2390	ISO17226-1	68.66		0.54	
2449	ISO17226-1	55.7		-0.45	
2459	ISO17226-1	59.87		-0.13	
2460		-----		-----	
2501	ISO17226-1	67.40		0.44	
2511		60.019		-0.12	
2536	ISO17226-1	55.42		-0.47	
2540	ISO17226-1	55.21		-0.49	
2560	ISO17226-1	60.20		-0.11	
2561	ISO17226-1	54.67		-0.53	
2563		-----		-----	
2569	ISO17226-1	70.4		0.67	
2573	ISO17226-1	59.8		-0.14	
2586	ISO17226-1	73.30		0.89	
2590	ISO17226-1	58.191		-0.26	
2591	ISO17226-1	105.57	R(0.01)	3.36	
2639		-----		-----	
2643		-----		-----	
2644		-----		-----	
2654	ISO17226-1	50.80		-0.82	
2674	ISO17226-1	60.1		-0.11	
2695	ISO17226-1	49.000		-0.96	
2703	ISO17226-1	54.95		-0.51	
2711	ISO17226-1	53.0		-0.66	
2734	ISO17226-1	61.490		-0.01	
2756		-----		-----	
2765	ISO17226-1	45.9		-1.20	
2769	ISO17226-1	43.5		-1.38	

lab	method	value	mark	z(targ)	remarks
2789	ISO17226-1	62.3		0.05	
2806	ISO17226-1	53.3		-0.63	
2815	ISO17226-1	56.90		-0.36	
2826	ISO17226-1	65.3		0.28	
2830	ISO17226-1	61.965		0.03	
2858	ISO17226-1	55.00		-0.50	
2870	ISO17226-1	68.1		0.50	
2908	ISO17226-1	70.41		0.67	
2941		-----		-----	
2943	ISO17226-1	67.405		0.44	
3100	ISO17226-1	59.67		-0.15	
3116		-----		-----	
3134		-----		-----	
3154	ISO17226-1	68.406		0.52	
3160	ISO17226-1	57.97		-0.28	
3172	ISO17226-1	62.75		0.09	
3176	ISO17226-1	69.62		0.61	
3192		-----		-----	
3197	ISO17226-1	66.7		0.39	
3200	ISO17226-1	51.03		-0.81	
3209	ISO17226-1	49.22		-0.94	
3210	ISO17226-1	61.98		0.03	
3228	ISO17226-1	58.2		-0.26	
3237	ISO17226-1	55.4		-0.47	
3246	ISO17226-1	71.01		0.72	
3248		-----		-----	

normality	OK	
n	80	
outliers	2	
mean (n)	61.598	
st.dev. (n)	7.2887	RSD = 12%
R(calc.)	20.408	
st.dev.(ISO17226-1:08)	13.1027	
R(ISO17226-1:08)	36.688	



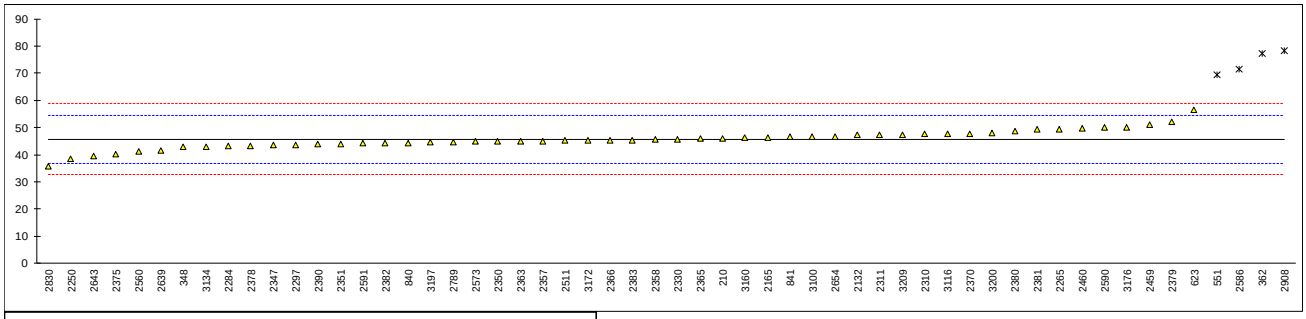


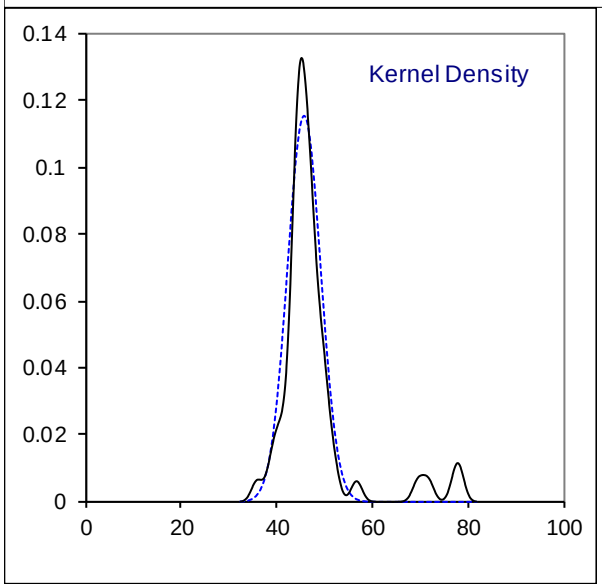
Determination of Formaldehyde content (colorimetric) on sample #20715; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210	GB/T19441-2	46.11		0.11	
230		-----		-----	
339		-----		-----	
348	In house	43.0		-0.61	
362	ISO17226-2	77.3	C,R(0.01)	7.26	First reported 82
551	ISO17226-2	69.43	C,R(0.01)	5.46	First reported 61.3586
623	ISO17226-2	56.60		2.51	
840	ISO17226-2	44.42		-0.28	
841	ISO17226-2	46.60		0.22	
1213		-----		-----	
2115		-----		-----	
2129		-----		-----	
2131		-----		-----	
2132	ISO17226-2	47.2		0.36	
2137		-----		-----	
2165	ISO17226-2	46.4		0.17	
2213		-----	W	-----	Test result withdrawn reported 59
2241		-----		-----	
2250	ISO17226-2	38.60		-1.62	
2256		-----		-----	
2265	ISO17226-2	49.36		0.85	
2271	ISO17226-2	-----		-----	
2284	ISO17226-2	43.19		-0.56	
2290		-----		-----	
2297	ISO17226-2	43.7		-0.45	
2310	ISO17226-2	47.5		0.43	
2311	ISO17226-2	47.363		0.39	
2330	ISO17226-2	45.73		0.02	
2347		43.6		-0.47	
2350	ISO17226-2	44.89		-0.17	
2351	ISO17226-2	44.06		-0.36	
2357	ISO17226-2	45.1		-0.12	
2358	ISO17226-2	45.61		-0.01	
2363	ISO17226-2	45		-0.15	
2365	ISO17226-2	46.1		0.11	
2366	ISO17226-2	45.3		-0.08	
2370	ISO17226-2	47.73		0.48	
2375	ISO17226-2	40.2		-1.25	
2378	ISO17226-2	43.21		-0.56	
2379	ISO17226-2	52.05		1.47	
2380	ISO17226-2	48.8		0.72	
2381	ISO17226-2	49.20		0.82	
2382	ISO17226-2	44.3		-0.31	
2383	ISO17226-2	45.4		-0.06	
2386		-----		-----	
2390	ISO17226-2	44.03		-0.37	
2449		-----		-----	
2459	ISO17226-2	51.23		1.28	
2460	ISO17226-2	49.85		0.97	
2501		-----		-----	
2511		45.13		-0.12	
2536		-----		-----	
2540		-----		-----	
2560	ISO17226-2	41.10		-1.04	
2561		-----		-----	
2563		-----		-----	
2569		-----		-----	
2573	ISO17226-2	44.8		-0.19	
2586	ISO17226-2	71.58	C,R(0.01)	5.95	First reported 62.71
2590	ISO17226-2	49.900		0.98	
2591	ISO17226-2	44.11		-0.35	
2639	GB/T19941-2	41.495		-0.95	
2643	ISO17226-2	39.60		-1.39	
2644		-----		-----	
2654	ISO17226-2	46.75		0.25	
2674		-----		-----	
2695		-----		-----	
2703		-----		-----	
2711		-----		-----	
2734		-----		-----	
2756		-----		-----	
2765		-----		-----	
2769		-----		-----	

lab	method	value	mark	z(targ)	remarks
2789	ISO17226-2	44.7		-0.22	
2806		-----		-----	
2815		-----		-----	
2826		-----		-----	
2830	ISO17226-2	35.82		-2.25	
2858		-----		-----	
2870		-----		-----	
2908	ISO17226-2	78.11	C,R(0.01)	7.45	First reported 69.17
2941		-----		-----	
2943		-----		-----	
3100	ISO17226-2	46.63		0.23	
3116	ISO17226-2	47.70		0.47	
3134	ISO17226-2	43.04		-0.60	
3154		-----		-----	
3160	ISO17226-2	46.18		0.12	
3172	ISO17226-2	45.14		-0.12	
3176	ISO17226-2	49.91		0.98	
3192		-----		-----	
3197	ISO17226-2	44.5		-0.26	
3200	ISO17226-2	48.01		0.54	
3209	ISO17226-2	47.41		0.41	
3210		-----		-----	
3228	ISO17226-2	-----		-----	
3237		-----		-----	
3246	ISO17226-2	-----		-----	
3248		-----		-----	

normality	suspect	
n	52	
outliers	4	
mean (n)	45.642	
st.dev. (n)	3.4661	RSD = 8%
R(calc.)	9.705	
st.dev.(ISO17226-2:08)	4.3600	
R(ISO17226-2:08)	12.208	



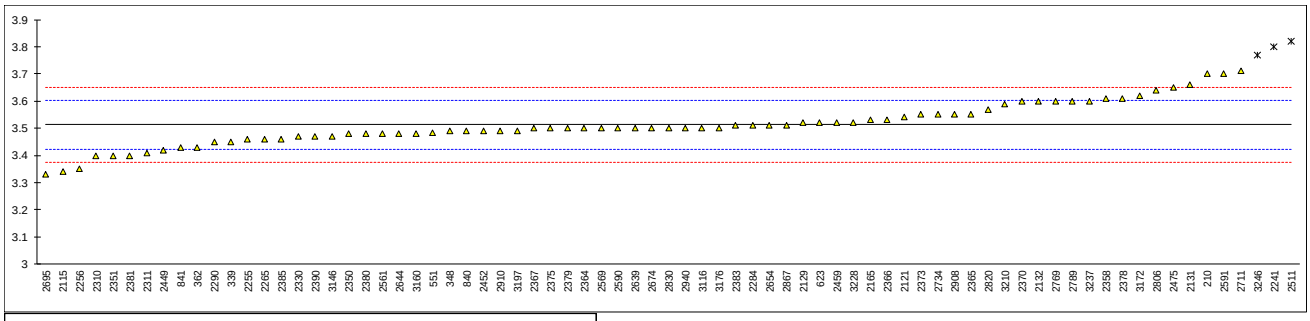


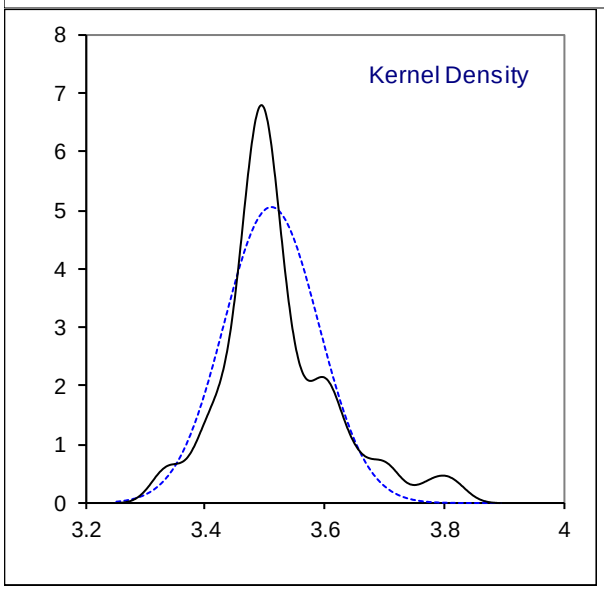
Determination of pH of extract on sample #20720; unitless results

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210	ISO4045	3.70	C	4.12	First reported 3.72
230		-----		-----	
339	ISO4045	3.45		-1.38	
348	ISO4045	3.49		-0.50	
362	ISO4045	3.43		-1.82	
551	ISO4045	3.485		-0.61	
623	ISO4045	3.520		0.16	
840	ISO4045	3.49		-0.50	
841	ISO4045	3.43		-1.82	
2115	ISO4045	3.34		-3.80	
2121	ISO4045	3.54		0.60	
2129	ISO4045	3.52		0.16	
2131	In house	3.66	C	3.24	First reported 6.66
2132	ISO4045	3.60		1.92	
2165	ISO4045	3.53		0.38	
2241	ISO4045	3.80	C,R(0.05)	6.32	First reported 4.058
2255	ISO4045	3.46		-1.16	
2256	ISO4045	3.35		-3.58	
2265	ISO4045	3.46		-1.16	
2284	ISO4045	3.510		-0.06	
2290	ISO4045	3.45		-1.38	
2310	ISO4045	3.40		-2.48	
2311	ISO4045	3.41		-2.26	
2330	ISO4045	3.47		-0.94	
2350	ISO4045	3.48		-0.72	
2351	ISO4045	3.40		-2.48	
2358	ISO4045	3.61		2.14	
2364	ISO4045	3.50		-0.28	
2365	ISO4045	3.552		0.86	
2366	ISO4045	3.53		0.38	
2367	ISO4045	3.50		-0.28	
2370	ISO4045	3.60		1.92	
2373	ISO4045	3.55		0.82	
2375	ISO4045	3.5		-0.28	
2378	ISO4045	3.61		2.14	
2379	ISO4045	3.50		-0.28	
2380	ISO4045	3.48		-0.72	
2381	ISO4045	3.40		-2.48	
2383	ISO4045	3.509	C	-0.08	First reported 7.201
2385	ISO4045	3.46		-1.16	
2390	ISO4045	3.47		-0.94	
2449	ISO4045	3.42	C	-2.04	First reported 3.31
2452	ISO4045	3.49		-0.50	
2459	ISO4045	3.52		0.16	
2475	ISO4045	3.65		3.02	
2511		3.819	R(0.05)	6.74	
2561	ISO4045	3.48		-0.72	
2563		-----		-----	
2569	ISO4045	3.5		-0.28	
2590	ISO4045	3.50		-0.28	
2591	ISO4045	3.7		4.12	
2639	QB/T2724	3.50		-0.28	
2644	ISO4045	3.48		-0.72	
2654	ISO4045	3.510		-0.06	
2674	ISO4045	3.50		-0.28	
2695	ISO4045	3.33	C	-4.02	First reported 3.32
2711	ISO4045	3.71	C	4.34	First reported 3.752
2734	ISO4045	3.55	C	0.82	First reported 3.30
2756		-----		-----	
2769	ISO4045	3.60		1.92	
2789	ISO4045	3.60		1.92	
2806	ISO4045	3.64		2.80	
2820	ISO4045	3.57		1.26	
2830	ISO4045	3.50		-0.28	
2867	ISO4045	3.51		-0.06	
2908	ISO4045	3.55		0.82	
2910	ISO4045	3.49		-0.50	
2940		-----		-0.28	
2941		-----		-----	
3116	ISO4045	3.50	C	-0.28	First reported 3.70
3146	ISO4045	3.470		-0.94	
3160	ISO4045	3.48		-0.72	
3172	ISO4045	3.618		2.32	

lab	method	value	mark	z(targ)	remarks
3176	ISO4045	3.50		-0.28	
3197	ISO4045	3.49		-0.50	
3210	ISO4045	3.59		1.70	
3228	ISO4045	3.52		0.16	
3237	ISO4045	3.6		1.92	
3246	ISO4045	4.02	R(0.01)	5.66	
3248		-----		-----	

	normality	OK
	n	72
	outliers	3
	mean (n)	3.513
	st.dev. (n)	0.0790
RSD = 2.2%		
	R(calc.)	0.221
	st.dev.(D2810:18)	0.0455
	R(D2810:18)	0.127



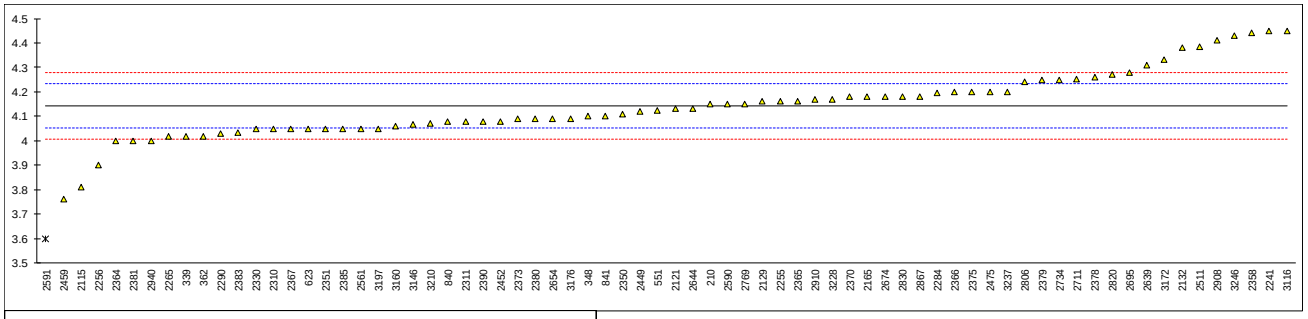


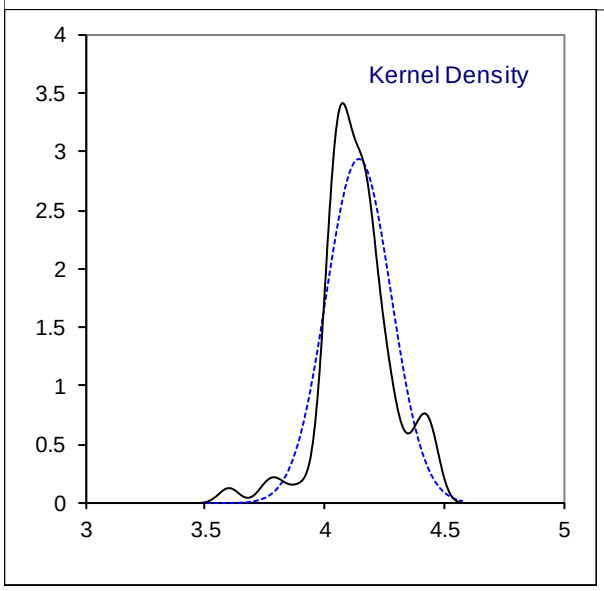
Determination of pH of ten times diluted extract on sample #20720; unitless results

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210	ISO4045	4.15	C	0.13	First reported 4.3
230		-----		-----	
339	ISO4045	4.02		-2.73	
348	ISO4045	4.10		-0.97	
362	ISO4045	4.02		-2.73	
551	ISO4045	4.125		-0.42	
623	ISO4045	4.050		-2.07	
840	ISO4045	4.08		-1.41	
841	ISO4045	4.10		-0.97	
2115	ISO4045	3.81		-7.35	
2121	ISO4045	4.13		-0.31	
2129	ISO4045	4.16		0.35	
2131		-----		-----	
2132	ISO4045	4.38		5.19	
2165	ISO4045	4.18		0.79	
2241	ISO4045	4.45		6.73	
2255	ISO4045	4.16		0.35	
2256	ISO4045	3.90		-5.37	
2265	ISO4045	4.02		-2.73	
2284	ISO4045	4.197		1.16	
2290	ISO4045	4.03		-2.51	
2310	ISO4045	4.05		-2.07	
2311	ISO4045	4.08		-1.41	
2330	ISO4045	4.05		-2.07	
2350	ISO4045	4.11		-0.75	
2351	ISO4045	4.05		-2.07	
2358	ISO4045	4.44		6.51	
2364	ISO4045	4.00		-3.17	
2365	ISO4045	4.161		0.37	
2366	ISO4045	4.20		1.23	
2367	ISO4045	4.05		-2.07	
2370	ISO4045	4.18		0.79	
2373	ISO4045	4.09		-1.19	
2375	ISO4045	4.2		1.23	
2378	ISO4045	4.26		2.55	
2379	ISO4045	4.25		2.33	
2380	ISO4045	4.09		-1.19	
2381	ISO4045	4.00		-3.17	
2383	ISO4045	4.032	C	-2.47	First reported 3.509
2385	ISO4045	4.05		-2.07	
2390	ISO4045	4.08		-1.41	
2449	ISO4045	4.12		-0.53	
2452	ISO4045	4.08		-1.41	
2459	ISO4045	3.76		-8.45	
2475	ISO4045	4.2		1.23	
2511		4.386		5.32	
2561	ISO4045	4.05		-2.07	
2563		-----		-----	
2569		-----		-----	
2590	ISO4045	4.15		0.13	
2591	ISO4045	3.6	R(0.05)	-11.97	
2639	QB/T2724	4.31		3.65	
2644	ISO4045	4.13		-0.31	
2654	ISO4045	4.090		-1.19	
2674	ISO4045	4.18		0.79	
2695	ISO4045	4.28	C	2.99	First reported 4.22
2711	ISO4045	4.251		2.35	
2734	ISO4045	4.25	C	2.33	First reported 3.98
2756		-----		-----	
2769	ISO4045	4.15		0.13	
2789		-----		-----	
2806	ISO4045	4.24		2.11	
2820	ISO4045	4.27		2.77	
2830	ISO4045	4.18		0.79	
2867	ISO4045	4.18		0.79	
2908	ISO4045	4.41	C	5.85	First reported 44.1
2910	ISO4045	4.17		0.57	
2940	QB/T2724	4.00		-3.17	
2941		-----		-----	
3116	ISO4045	4.45		6.73	
3146	ISO4045	4.068		-1.68	
3160	ISO4045	4.06		-1.85	
3172	ISO4045	4.330		4.09	

lab	method	value	mark	z(targ)	remarks
3176	ISO4045	4.09		-1.19	
3197	ISO4045	4.05		-2.07	
3210	ISO4045	4.07		-1.63	
3228	ISO4045	4.17		0.57	
3237	ISO4045	4.2		1.23	
3246	ISO4045	4.43		6.29	
3248		-----		-----	

	normality	OK
	n	71
	outliers	1
	mean (n)	4.144
	st.dev. (n)	0.1360 RSD = 3.3%
	R(calc.)	0.381
	st.dev.(D2810:18)	0.0455
	R(D2810:18)	0.127



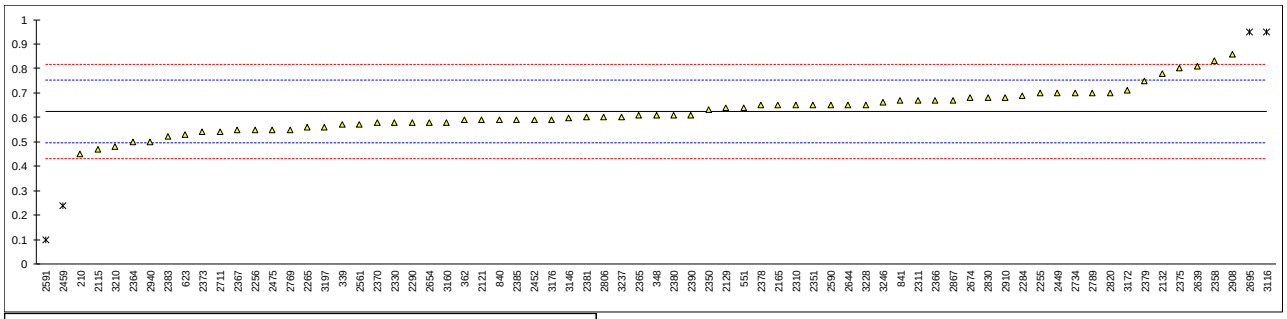


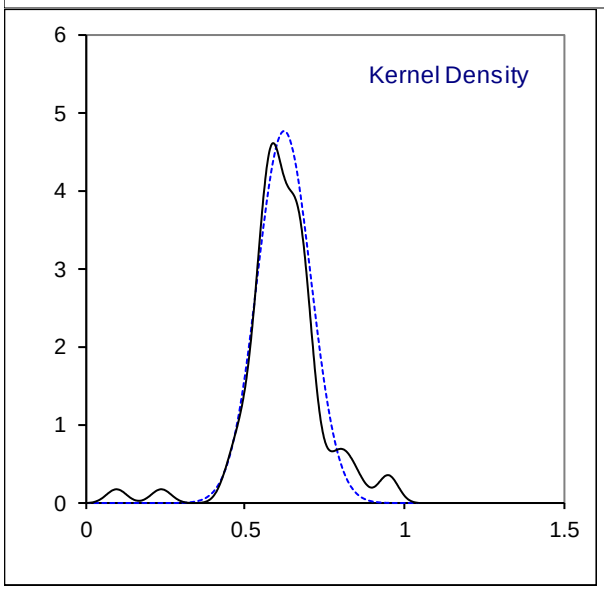
Determination of difference between pH of extract and pH of ten times diluted extract on sample #20720; unitless results

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210	ISO4045	0.45	C	-2.71	First reported 0.55
230		-----		-----	
339	ISO4045	0.57		-0.85	
348	ISO4045	0.61		-0.23	
362	ISO4045	0.59		-0.54	
551	ISO4045	0.64		0.24	
623	ISO4045	0.530		-1.47	
840	ISO4045	0.59		-0.54	
841	ISO4045	0.67		0.71	
2115	ISO4045	0.47		-2.40	
2121	ISO4045	0.59		-0.54	
2129	ISO4045	0.640		0.24	
2131		-----		-----	
2132	ISO4045	0.78		2.42	
2165	ISO4045	0.65		0.40	
2241		-----		-----	
2255	ISO4045	0.7		1.17	
2256	ISO4045	0.55		-1.16	
2265	ISO4045	0.56		-1.00	
2284	ISO4045	0.687		0.97	
2290	ISO4045	0.58		-0.69	
2310	ISO4045	0.65		0.40	
2311	ISO4045	0.67		0.71	
2330	ISO4045	0.58		-0.69	
2350	ISO4045	0.63		0.09	
2351	ISO4045	0.65		0.40	
2358	ISO4045	0.83		3.20	
2364	ISO4045	0.50		-1.94	
2365	ISO4045	0.609		-0.24	
2366	ISO4045	0.67		0.71	
2367	ISO4045	0.55		-1.16	
2370	ISO4045	0.58		-0.69	
2373	ISO4045	0.54		-1.31	
2375	ISO4045	0.8		2.73	
2378	ISO4045	0.65		0.40	
2379	ISO4045	0.75		1.95	
2380	ISO4045	0.61		-0.23	
2381	ISO4045	0.60		-0.38	
2383	ISO4045	0.523		-1.58	
2385	ISO4045	0.59		-0.54	
2390	ISO4045	0.61		-0.23	
2449	ISO4045	0.70	C	1.17	First reported 0.81
2452	ISO4045	0.59		-0.54	
2459	ISO4045	0.24	R(0.05)	-5.98	
2475	ISO4045	0.55		-1.16	
2511		-----		-----	
2561	ISO4045	0.57		-0.85	
2563		-----		-----	
2569		-----		-----	
2590	ISO4045	0.65		0.40	
2591	ISO4045	0.1	R(0.01)	-8.16	
2639	QB/T2724	0.81		2.89	
2644	ISO4045	0.65		0.40	
2654	ISO4045	0.58		-0.69	
2674	ISO4045	0.68		0.86	
2695	ISO4045	0.950	C,R(0.05)	5.06	First reported 0.930
2711	ISO4045	0.541	C	-1.30	First reported 0.499
2734	ISO4045	0.70	C	1.17	First reported 0.68
2756		-----		-----	
2769	ISO4045	0.55		-1.16	
2789	ISO4045	0.70		1.17	
2806	ISO4045	0.60		-0.38	
2820	ISO4045	0.70		1.17	
2830	ISO4045	0.68		0.86	
2867	ISO4045	0.67		0.71	
2908	ISO4045	0.86		3.66	
2910	ISO4045	0.68		0.86	
2940	QB/T2724	0.50		-1.94	
2941		-----		-----	
3116	ISO4045	0.95	C,R(0.05)	5.06	First reported 0.75
3146	ISO4045	0.598		-0.41	
3160	ISO4045	0.58		-0.69	
3172	ISO4045	0.712		1.36	

lab	method	value	mark	z(targ)	remarks
3176	ISO4045	0.59		-0.54	
3197	ISO4045	0.56		-1.00	
3210	ISO4045	0.48		-2.25	
3228	ISO4045	0.65		0.40	
3237	ISO4045	0.6		-0.38	
3246	ISO4045	0.66		0.55	
3248		-----		-----	

normality	OK
n	67
outliers	4
mean (n)	0.624
st.dev. (n)	0.0838
R(calc.)	0.235
st.dev.(D2810:18)	0.0643
R(D2810:18)	0.180





APPENDIX 2

Analytical details for sample #20715 (Formaldehyde Determination)

lab	ISO/IEC17025 accredited	Sample intake (in grams)	Number of days to complete the test
110	---		
210	Yes		
230	---		
339	No	2	16
348	Yes	2	2
362	Yes	2.00g	1 day
551	Yes	2 g	
623	Yes	1 Gram	1 day
840	Yes	2g	1 day
841	Yes	2 grams	1 days
1213	Yes		
2115	Yes	2 g	1 day
2129	Yes	1g	29.10./02.11./03.11.
2131	No	2	1
2132	Yes	2g	2 days
2137	Yes	1	1
2165	Yes	2.0g	1 day
2213	Yes	2 gram	2 gram
2241	Yes	0.5g	one day
2250	Yes	1 g/2 g	2 days
2256	Yes	2.0083g	1 day
2265	No	2 g	in 7 days
2271	Yes	1.0006g	1 day
2284	Yes	ISO17226-1:2.03g ISO17226-2:2.02g	3 days
2290	Yes		
2297	Yes	1g	24hrs
2310	No	1	10
2311	Yes	1	10
2330	Yes	2 grams	1 day
2347	---		
2350	Yes	2 g	1
2351	Yes	ISO17226-1: 1g ISO17226-2: 1g	16
2357	Yes	2	
2358	Yes	1.0 g	with 1 day
2363	Yes	2g	24h
2365	Yes	1g	ISO17226-1:11.18-11.19 (2 DAYs) ISO17226-2:11.18 (1 DAYs)
2366	Yes	0.5g	1d
2370	Yes	2 g	2 days
2375	---		
2378	Yes	2g	7 days
2379	Yes	(HPLC) 1 gr. (Colorimetric) 1 gr / 25 mL	3 days
2380	Yes	2.00 g	Two
2381	Yes	2 gm	2
2382	Yes	2g	14
2383	Yes		1
2386	Yes	1	1
2390	Yes	2.0056g	Within 1day
2449	No	2grams	
2459	---		
2460	No	2	19 days
2501	Yes	2.0133 grams	4 days
2511	---		
2536	Yes	2.0003	Not applicable
2540	Yes	2 g	1day
2560	Yes	2.0 gm	One day
2561	Yes	2	1 sample on day 1, 2 samples on day 2
2563	---		
2569	Yes	2 gm	one day
2573	Yes	2g	1day
2586	Yes	2 g	1 day
2590	Yes	1g	1 day
2591	Yes	2.0 grams	18/11/2020 by Colorimetric method. 23/11/2020 by HPLC.
2639	No	2.0072g	one day
2643	Yes	2.017	3
2644	---		
2654	Yes	2	
2674	Yes	1.0g	performed during 2020/11/09 to 2020/11/13
2695	Yes	4 g	1 day
2703	Yes	2.00g	7 days from receipt of sample
2711	No	2.000g	1
2734	Yes	2.0	19 g
2756	---		
2765	Yes	1,44 g	1 day

lab	ISO/IEC17025 accredited	Sample intake (in grams)	Number of days to complete the test
2769	Yes	2	
2789	Yes	2	2
2806	Yes		
2815	Yes	2 g	in 1 day
2826	Yes	2g	Within 1 day
2830	Yes	2.00 gram	1
2858	Yes	0.701 gm	Two days and four trials
2870	Yes	2 gm	Performed extraction and analysis in same day.
2908	Yes	2 grams	the determination of formaldehyde was in 23-11-2020
2941	---		
2943	No	2.0 g	20 days
3100	Yes	1g	
3116	Yes	2g	Within 1 day
3134	No	1g/25 ml, twice,	sample received 27-10-2020 , analysed 19 and 20 November 2020.
3154	Yes		
3160	Yes	2 g	1
3172	Yes		
3176	Yes	2	7
3192	---		
3197	Yes	2 g	
3200	Yes	2.0026/2.0015	one day
3209	Yes	2g	10
3210	Yes	2g	1 day
3228	Yes	1	5
3237	Yes	2 gr	Same day
3246	Yes	1.00g	7 days
3248	---		

Analytical details for sample #20720 (pH Determination)

lab	ISO/IEC17025 accredited	Sample intake (in grams)	Additional steps to wet the sample
110	---		---
210	Yes		---
230	---		---
339	No	5	No
348	No	5	No
362	Yes	5.00g	No
551	No	5g	No
623	Yes	2.5 grams	No
840	Yes	5g	No
841	Yes	5.0008 grams	Yes: used deion water
2115	Yes	5 g	No
2121	Yes	2 x 4,95 g	No
2129	Yes	2	No
2131	Yes	2	No
2132	Yes	5g	No
2165	Yes	2.0G	No
2241	Yes	5g	No
2255	Yes	5	No
2256	Yes	5gram	No
2265	No	5 g	No
2284	Yes	5 grams for each sample.	No
2290	Yes		---
2310	Yes	10	No
2311	Yes	5	No
2330	Yes	5.00 g	No
2350	No	5.0 g	No
2351	Yes	10g	No
2358	Yes	5.0 g	No
2364	Yes	5.00g per specimen	No
2365	Yes	2g	No
2366	Yes	1g	No
2367	Yes	5g	No
2370	Yes	5g	No
2373	Yes	5g	No
2375	---		---
2378	Yes	5.001g	No
2379	Yes	5.0 grams per each	No
2380	Yes	5.0 g	No
2381	Yes	5	No
2383	Yes	5g	No
2385	No	4	No
2390	Yes	5.0088g	No
2449	Yes	5grams	No
2452	Yes		---
2459	---		---
2475	No	5g	No
2511	---		---
2561	Yes	2 x 5 g extractions	Yes: shaken vigorously by hand
2563	---		---
2569	---		---
2590	Yes	5g	No
2591	Yes	5 grams	No
2639	No	5.0002g	No
2644	No	5 g	No
2654	Yes	5	No
2674	Yes	5g	No
2695	Yes	10g	No
2711	No	5.011g	No
2734	Yes	5.0 g	No
2756	---		---
2769	Yes	5	No
2789	---		---
2806	Yes	5 GRAMS	No
2820	Yes	5	No
2830	Yes	5.00 grames	No
2867	Yes	0.3g	No
2908	Yes	5.00 grams	No
2910	Yes	5g	No
2940	No	5g	No
2941	---		---
3116	Yes	5g	No
3146	Yes	2 * 2.00 gram	No
3160	Yes	5 g	No

lab	ISO/IEC17025 accredited	Sample intake (in grams)	Additional steps to wet the sample
3172	Yes		---
3176	Yes	5	No
3197	Yes	5 g	No
3210	Yes	5	No
3228	No	2.5g	No
3237	Yes	5 gr	No
3246	Yes	5.00	No
3248	---		---

APPENDIX 3

Number of participants per country

iis20A15F	
5 labs in	BANGLADESH
1 lab in	BRAZIL
3 labs in	BULGARIA
1 lab in	CAMBODIA
1 lab in	ETHIOPIA
2 labs in	FRANCE
7 labs in	GERMANY
1 lab in	GREECE
5 labs in	HONG KONG
5 labs in	INDIA
1 lab in	INDONESIA
10 labs in	ITALY
1 lab in	MAURITIUS
1 lab in	MEXICO
4 labs in	MOROCCO
23 labs in	P.R. of CHINA
3 labs in	PAKISTAN
1 lab in	PORTUGAL
4 labs in	SOUTH KOREA
4 labs in	SPAIN
2 labs in	SWITZERLAND
1 lab in	TAIWAN
1 lab in	THAILAND
1 lab in	TUNISIA
4 labs in	TURKEY
1 lab in	U.S.A.
2 labs in	UNITED KINGDOM
5 labs in	VIETNAM

iis20A15P	
3 labs in	BANGLADESH
1 lab in	BRAZIL
2 labs in	BULGARIA
1 lab in	CAMBODIA
1 lab in	ETHIOPIA
4 labs in	FRANCE
5 labs in	GERMANY
4 labs in	HONG KONG
3 labs in	INDIA
1 lab in	INDONESIA
9 labs in	ITALY
1 lab in	MAURITIUS
4 labs in	MOROCCO
18 labs in	P.R. of CHINA
3 labs in	PAKISTAN
1 lab in	PORTUGAL
1 lab in	SOUTH KOREA
4 labs in	SPAIN
1 lab in	SWITZERLAND
1 lab in	TAIWAN
1 lab in	THAILAND
2 labs in	TUNISIA
4 labs in	TURKEY
1 lab in	U.S.A.
1 lab in	UNITED KINGDOM
4 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
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 evaluations

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