Results of Proficiency Test Formaldehyde and pH in Leather November 2018

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis18A12

January 2019

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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 the 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 requirements for leather, there are some Ecolabelling schemes imposing environmental requirements for textile & leather products on a voluntary basis. Well-known organizations 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 every year. During the annual proficiency testing program 2018/2019, it was decided to continue the round robin for the analysis of Formaldehyde content and pH in Leather.
In this interlaboratory study 106 laboratories in 30 different countries registered for participation in the PT Formaldehyde in Leather and 89 laboratories in 27 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 2018 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 analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send depending on the registration, one leather sample (labelled #18640) positive on Formaldehyde and a leather sample (labelled #18641) 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/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 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 first batch of dark brown leather squares was obtained from a third-party laboratory. After homogenization, 110 subsamples of approximately 6 grams were prepared. Each sample was packed in a polypropylene bag and wrapped in Aluminum foil and labelled #18640. The homogeneity of the subsamples was checked by the determination of the Formaldehyde content in accordance with an inhouse test method on 8 stratified randomly selected samples. See the following table for the test results.

	Formaldehyde in mg/kg
Sample #18640-1	87.9
Sample #18640-2	88.8
Sample #18640-3	91.2
Sample #18640-4	90.9
Sample #18640-5	87.3
Sample #18640-6	89.2
Sample #18640-7	90.0
Sample #18640-8	88.7

Table 1: homogeneity test results of subsamples #18640

From the above test results, the repeatability was calculated and compared with 0.3 times the corresponding reproducibility of the reference test method and in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Formaldehyde in mg/kg
r (observed)	3.8
reference test method	ISO17226-1:08
0.3*R (ref. test method)	16.5

Table 2: evaluation of the repeatability of subsamples #18640

The calculated repeatability was in agreement with 0.3 times the estimated reproducibility of the reference test method. Therefore, homogeneity of the subsamples #18640 was assumed.

The second batch was a black leather sample, which was shreddered into small pieces. After homogenization, 120 subsamples of approx. 10 grams were prepared. Each sample was packed in a polypropylene bag and wrapped in Aluminum foil and labelled sample #18641. The homogeneity of the subsamples was checked by the determination of pH in accordance an inhouse test method on 8 stratified randomly selected samples. See the following table for the test results.

	pH
Sample #18641-1	3.11
Sample #18641-2	3.13
Sample #18641-3	3.12
Sample #18641-4	3.13
Sample #18641-5	3.14
Sample #18641-6	3.14
Sample #18641-7	3.14
Sample #18641-8	3.12

Table 3: homogeneity test results of subsamples #18641

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

	pH
r (observed)	0.03
reference test method	ASTM D2810:13
0.3*R (ref. test method)	0.04

Table 4: repeatability of subsamples #18641

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 #18641 was assumed.

To each of the participants was sent depending on the registration: 1 sample labelled #18640 and/or 1 sample labelled #18641 on October 10, 2018.

2.5 ANALYSES

The participants were requested to determine on sample #18640, the content of Formaldehyde (HPLC) and/or the content of Formaldehyde (colorimetric). On sample #18641 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 requested to report if the laboratory was accredited for the requested components that were determined. It was also asked 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 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 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 appropriate 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 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 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 (no reanalyzes). 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 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.

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 Grubbs' test and by R(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. 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 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. 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 result tables in appendix 1.

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:

|z| < 1 good
1 < |z| < 2 satisfactory
2 < |z| < 3 questionable
3 < |z| unsatisfactory

4 EVALUATION

In this proficiency test, no problems were encountered with the delivery of the samples.
Six laboratories did not report any test results for the Formaldehyde determination and three laboratories did not report any test results for the pH determination.
Finally, in total for the two PTs the 114 reporting laboratories sent in total 396 numerical test results. Observed were 12 outlying test results, which is 3.0% 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 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.
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 on 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. 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 tests. 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 #18640:

Formaldehyde content (HPLC): This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated requirements of ISO17226-1:08.

Formaldehyde content (colorimetric): This determination was very problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the strict estimated requirements of ISO17226-2:08.

Sample #18641:

pH of extract: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D2810:13.

pH of ten times diluted extract: This determination was very problematic. Four 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.

Difference between pH of extract and pH ten times diluted 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 D2810:13.

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 (2.8*sd) and the target reproducibilities (ISO17226 and ASTM D2810), are compared in the next two tables.

parameter	unit	n	average	2.8 * sd	R (target)
Formaldehyde (HPLC)	mg/kg	84	71.9	46.8	43.5
Formaldehyde (colorimetric)	mg/kg	65	69.8	33.6	17.5

Table 5: reproducibilities of tests on sample #18640

parameter	unit	n	average	2.8 * sd	R (target)
pH of extract	-	84	3.57	0.17	0.13
pH of extract ten times diluted	-	76	4.04	0.27	0.13
Difference between pH	-	75	0.46	0.21	0.18

Table 6: reproducibilities of test on sample #18641

It can be concluded that, without statistical calculations, the group of participating laboratories has some difficulties with the determination of Formaldehyde (colorimetric) and pH, but have no problems with the HPLC analysis, when compared to the target test methods. See also the discussions in paragraphs 4.1 and 5.

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2018 WITH PREVIOUS PTS

Parameter	November 2018	November 2017	November 2016	October 2015	October 2014
Number of reporting labs	114	102	106	116	108
Number of results reported	396	378	240	239	224
Number of statistical outliers	12	16	16	7	7
Percentage outliers	3.0%	4.2%	6.7%	2.9%	3.1%

Table 7: comparison with previous proficiency tests

In Table 8 the relative uncertainties over the years are given. The uncertainty of the 2018 PT on the HPLC determination of Formaldehyde in leather is in line compared to the years before 2017, and is comparable to the uncertainty of the target test method. Apparently, the observed uncertainty in 2017 PT was remarkedly good.

Parameter	Nov 2018	Nov 2017	Nov 2016	Oct 2015	2013-2014	Est. from target test method
Formaldehyde (HPLC)	23%	9%	20%	23%	22-30%	22% (17226-1)
Formaldehyde (colorimetric)	17%	39%	26%	22%	25-33%	9% (17226-2)
pH (undiluted)	1.7%	2.8%	2.1%	2.6%	3.2%	0.9% (D2810)
pH (10x diluted)	2.3%	3.0%	2.3%	n.e.	n.e.	0.9% (D2810)

Table 8: development of relative uncertainties over the years

n.e. = not evaluated

Improvement is visible in 2018 PT for the colorimetric determination of Formaldehyde in Leather and for the pH determination (undiluted) in comparison with the results in previous PTs. However, they are 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.

The reproducibility of the colorimetric method (ISO17226 part 2) is (much) smaller than the reproducibility of the HPLC method (ISO17226 part 1). Maybe the precision data for the colorimetric method were obtained with samples and/or conditions that did not influence the variation (as the test method describes that the variation could be influenced by absorbances from the leather coloring).

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 77% of the reporting laboratories reported to be accredited for the determination of Formaldehyde in Leather and about 73% of the participating laboratories reported to be accredited for the determination of pH on Leather.

For this PT, a few analytical details of the determination of Formaldehyde (colorimetric) in Leather were asked. Most of the reporting participants (67%) reported to have further cut/grinded the sample before testing. Approximately 60% of the reporting laboratories used 2 grams as intake as prescribed in ISO17226 and most participants completed the test within one day.
For the determination pH on Leather also some analytical details were asked. Most of the reporting participants (70%) reported to have used 5 grams for intake. Eight other participants (11%) used only 2-2.5 grams and seven (10%) participants reported to have used 7.5 -12.5 grams.

5 DISCUSSION

The standard test method for Formaldehyde content is 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 selective for Formaldehyde alone and part 2 is not selective. Therefore, in theory, the test results from part 2 should be higher on average than the test results from part 1. Remarkedly, this is not observed in PT sample #18640 while it was seen in sample #17640 of 2017 PT (iis17A10).

Analytical Details of test method: ISO17226
In this PT some analytical details were asked about the sample pre-preparation, intake and the time to do the tests (see appendix 2). After evaluation of these details, it was noticed that the participants that further cut/ grinded the samples found on average a slightly higher content of Formaldehyde (see pag.14). The impact of intake or the time to perform the tests was not observed for this sample.

Sample #18640 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 Öko-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 Öko-Tex 100

When using ISO17226 part 1, all reporting laboratories would reject this sample for category A. For category B, thirty-four laboratories would reject this sample, while fifty-one other reporting laboratories would accept this sample. All of the reporting laboratories would accept this sample for category C.
When using ISO17226 part 2, all reporting laboratories would reject this sample for category A. For category B, twenty-six laboratories would reject this sample, while forty-one laboratories would accept this sample for category B. All of the reporting laboratories would accept this sample for category C.
Compared to other labelling standards different decisions may be made concerning the acceptance or rejection of the sample.

Analytical Details of test method: ISO4045/ASTM D2810
In this PT some analytical details were asked about sample intake and if additional steps were taken to wet the leather (see appendix 2) for the determination of the pH. No effect of intake or using an additional step to wet the leather was observed for this sample.

Sample #18641 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 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. Five 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

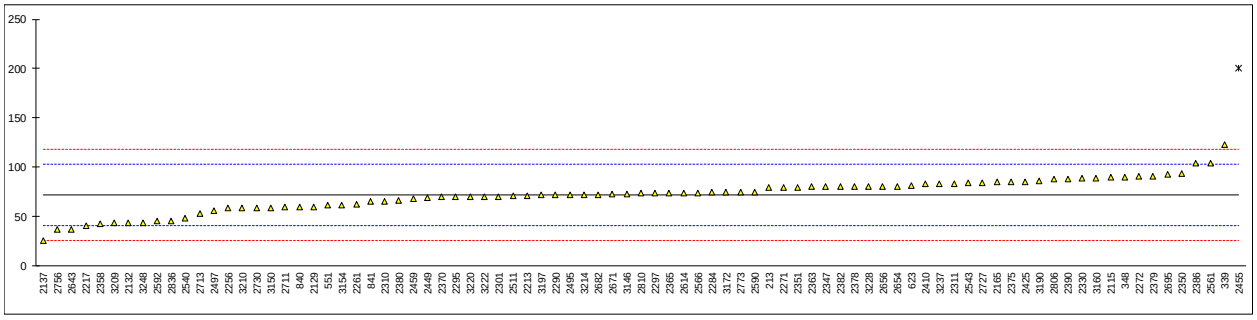
In this proficiency test the Formaldehyde content and pH were determined. The observed variation for the Formaldehyde content (HPLC method) in this interlaboratory study was in line with previous PTs, except for the 2017 PT. The observed variation for the Formaldehyde content (colorimetric) and pH in this interlaboratory study show improvement compared to the previous proficiency tests.
The variation 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.

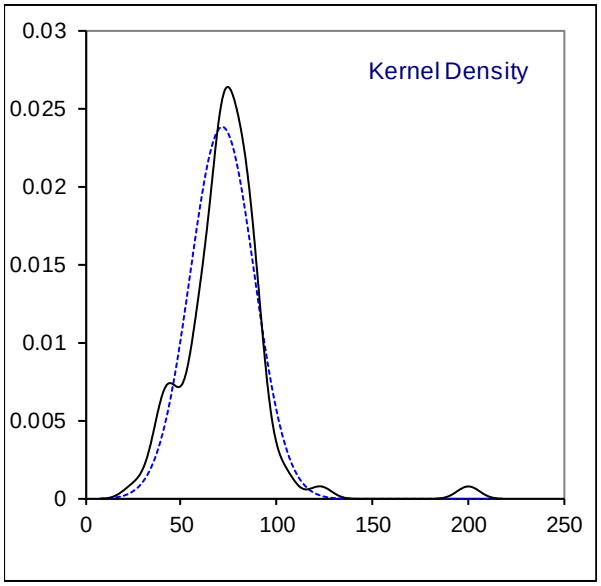
APPENDIX 1

Determination of Formaldehyde content (HPLC) on sample #18640; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
213	ISO17226-1	79.38		0.48	
339	In house	123		3.29	
348	In house	89.74		1.15	
362		-----		-----	
523		-----		-----	
551	In house	61.75		-0.65	
623	ISO17226-1	81.60		0.63	
840	ISO17226-1	59.7		-0.78	
841	ISO17226-1	64.92		-0.45	
2115	ISO17226-1	89.72		1.15	
2118		-----		-----	
2129	ISO17226-1	60		-0.76	
2132	ISO17226-1	43.4		-1.83	
2137	ISO17226-1	25.76		-2.97	
2138		-----		-----	
2165	ISO17226-1	84.8		0.83	
2213	ISO17226-1	71.2		-0.04	
2217	ISO17226-1	40.86		-2.00	
2256	ISO17226-1	58.5		-0.86	
2261	GB/T19941	62.41		-0.61	
2271	ISO17226-1	79.7		0.50	
2272	ISO17226-1	90.7		1.21	
2284	ISO17226-1	74.40		0.16	
2290	ISO17226-1	71.52		-0.02	
2293		-----		-----	
2295	ISO17226-1	70		-0.12	
2297	ISO17226-1	73.69		0.12	
2301	ISO17226-1	70.3		-0.10	
2310	ISO17226-1	65.03		-0.44	
2311	ISO17226-1	83.2		0.73	
2330	ISO17226-1	88.55		1.07	
2347	ISO17226-1	80		0.52	
2350	ISO17226-1	93.99		1.42	
2351	ISO17226-1	79.76		0.51	
2358	ISO17226-1	42.57		-1.89	
2363	ISO17226-1	80		0.52	
2365	ISO17226-1	73.73		0.12	
2370	ISO17226-1	69.80		-0.13	
2375	ISO17226-1	85.1		0.85	
2378	ISO17226-1	80.20		0.54	
2379	ISO17226-1	90.76		1.22	
2380	ISO17226-1	66.6		-0.34	
2381		-----		-----	
2382	ISO17226-1	80.1		0.53	
2386	In house	104		2.07	
2389		-----		-----	
2390	ISO17226-1	88.11		1.05	
2410	ISO17226-1	83		0.72	
2425	ISO17226-1	85.22		0.86	
2449	ISO17226-1	68.95		-0.19	
2452		-----		-----	
2455	ISO17226-1	200.5	C,R(0.01)	8.28	First reported 146.613
2459	ISO17226-1	68.29		-0.23	
2460		-----		-----	
2477		-----		-----	
2495	ISO17226-1	71.62		-0.02	
2497	ISO17226-1	56.22		-1.01	
2501		-----		-----	
2511	ISO17226-1	70.99		-0.06	
2540	ISO17226-1	48.35		-1.51	
2543	ISO17226-1	84.2		0.79	
2561	ISO17226-1	104.3		2.09	
2566	ISO17226-1	74.2		0.15	
2590	ISO17226-1	74.55		0.17	
2592	ISO17226-1	45.3		-1.71	
2614	ISO17226-1	73.89		0.13	
2639		-----		-----	
2643	ISO17226-1	37.30		-2.22	
2654	ISO17226-1	80.91		0.58	
2656	ISO17226-1	80.8		0.58	
2671	ISO17226-1	72.7		0.05	
2674		-----		-----	
2682	ISO17226-1	72.11		0.02	
2695	ISO17226-1	93.17		1.37	
2711	ISO17226-1	59.38		-0.80	

lab	method	value	mark	z(targ)	remarks	
2713	ISO17226-1	52.98		-1.22		
2727	ISO17226-1	84.50		0.81		
2730	ISO17226-1	59.16		-0.82		
2743		-----		-----		
2756	ISO17226-1	37.205		-2.23		
2773	ISO17226-1	74.5		0.17		
2783		-----		-----		
2806	ISO17226-1	87.8		1.03		
2810	ISO17226-1	73.52	C	0.11	First reported 17.25	
2829		-----		-----		
2836	ISO17226-1	45.56		-1.69		
2863		-----		-----		
3116		-----		-----		
3146	ISO17226-1	73.3		0.09		
3150	ISO17226-1	59.2		-0.81		
3154	ISO17226-1	61.81		-0.65		
3160	ISO17226-1	89.43		1.13		
3163		-----		-----		
3172	ISO17226-1	74.4		0.16		
3190	ISO17226-1	85.89		0.90		
3197	ISO17226-1	71.5		-0.02		
3209	ISO17226-1	43.352		-1.83		
3210	In house	58.81		-0.84		
3214	ISO17226-1	72.09		0.01		
3216		-----		-----		
3220	ISO17226-1	70.07		-0.12		
3222	ISO17226-1	70.12	C	-0.11	First reported 145.12	
3228	ISO17226-1	80.3		0.54		
3237	ISO17226-1	83.08		0.72		
3248	ISO17226-1	43.67		-1.81		
					Only further cut/grinded	Only intake 2 grams
	normality	OK			not OK	suspect
	n	84			58	52
	outliers	1			1	1
	mean (n)	71.860			76.558	71.495
	st.dev. (n)	16.7236	RSD = 23%		15.0889	16.1244
	R(calc.)	46.826			42.249	45.148
	st.dev.(ISO17226-1:08)	15.5364			16.6505	15.4498
	R(ISO17226-1:08)	43.502			46.621	43.259

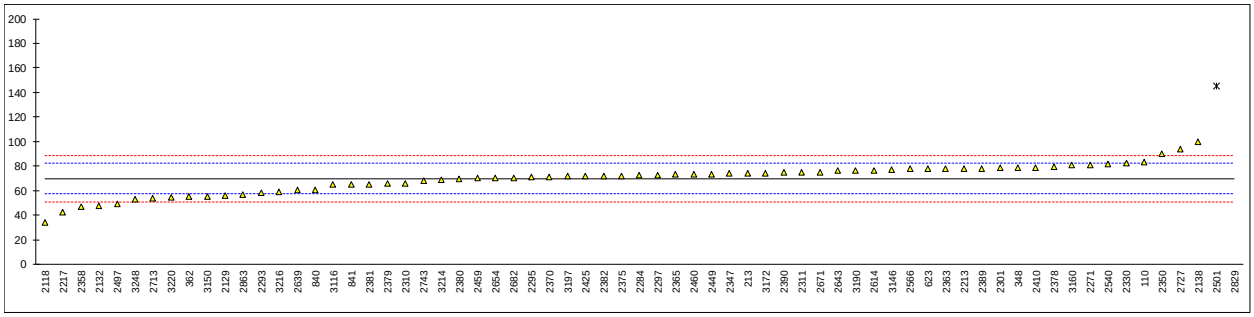


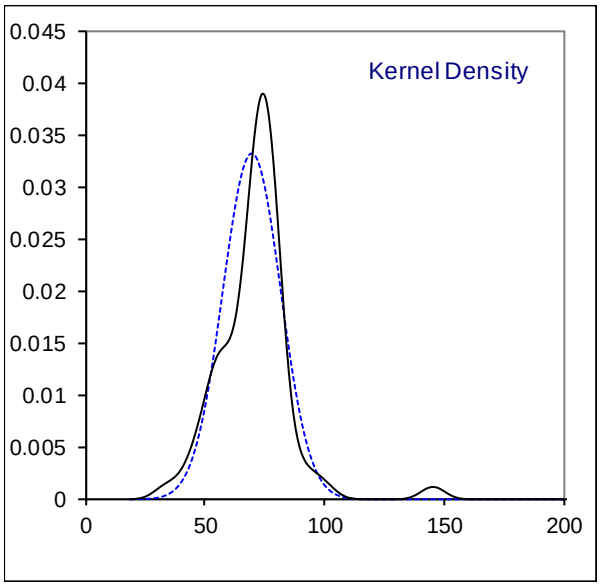


Determination of Formaldehyde content (colorimetric) on sample #18640; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	83.384		2.17	
213	ISO17226-2	74.4		0.73	
339		-----		-----	
348	In house	78.75		1.43	
362	ISO17226-2	55.23		-2.33	
523		-----		-----	
551		-----		-----	
623	ISO17226-2	77.61		1.25	
840	ISO17226-2	60.6		-1.47	
841	ISO17226-2	65.2		-0.73	
2115		-----		-----	
2118	ISO17226-2	34.065		-5.71	
2129	ISO17226-2	56.0		-2.20	
2132	ISO17226-2	47.5	C	-3.56	First reported 41.5
2137		-----		-----	
2138	ISO17226-2	100.1		4.84	
2165		-----		-----	
2213	ISO17226-2	78.1		1.33	
2217	ISO17226-2	42.35		-4.39	
2256		-----		-----	
2261		-----		-----	
2271	ISO17226-2	80.8		1.76	
2272		-----		-----	
2284	ISO17226-2	72.49		0.43	
2290		-----		-----	
2293	ISO17226-2	58.009		-1.88	
2295	ISO17226-2	71		0.19	
2297	ISO17226-2	72.76		0.47	
2301	ISO17226-2	78.50		1.39	
2310	ISO17226-2	66.0		-0.61	
2311	ISO17226-2	75.2		0.86	
2330	ISO17226-2	82.70		2.06	
2347	ISO17226-2	74		0.67	
2350	ISO17226-2	90.20		3.26	
2351		-----		-----	
2358	ISO17226-2	47.25		-3.60	
2363	ISO17226-2	77.9		1.29	
2365	ISO17226-2	73.13		0.53	
2370	ISO17226-2	71.20		0.22	
2375	ISO17226-2	72.1		0.37	
2378	ISO17226-2	79.30		1.52	
2379	ISO17226-2	65.764		-0.64	
2380	ISO17226-2	69.5		-0.05	
2381	ISO17226-2	65.20		-0.73	
2382	ISO17226-2	72.0		0.35	
2386		-----		-----	
2389	ISO17226-2	78.1		1.33	
2390	ISO17226-2	75.17		0.86	
2410	ISO17226-2	79		1.47	
2425	ISO17226-2	71.98		0.35	
2449	ISO17226-2	73.78		0.64	
2452		-----		-----	
2455		-----		-----	
2459	ISO17226-2	70.22		0.07	
2460	ISO17226-2	73.417		0.58	
2477		-----		-----	
2495		-----		-----	
2497	ISO17226-2	49.51		-3.24	
2501	ISO17226-2	145.40	R(0.01)	12.08	
2511		-----		-----	
2540	ISO17226-2	81.39		1.85	
2543		-----		-----	
2561		-----		-----	
2566	ISO17226-2	77.6		1.25	
2590		-----		-----	
2592		-----		-----	
2614	ISO17226-2	76.8		1.12	
2639	GB/T19941	60.23		-1.53	
2643	ISO17226-2	76.70		1.10	
2654	ISO17226-2	70.30		0.08	
2656		-----		-----	
2671	ISO17226-2	75.2		0.86	
2674		-----		-----	
2682	ISO17226-2	70.62		0.13	
2695		-----		-----	
2711		-----		-----	
2713	ISO17226-2	53.56		-2.59	

lab	method	value	mark	z(targ)	remarks
2727	ISO17226-2	93.70		3.82	
2730		-----		-----	
2743	ISO17226-2	68.03		-0.28	
2756		-----		-----	
2773		-----		-----	
2783		-----		-----	
2806		-----		-----	
2810		-----		-----	
2829	ISO17226-2	241.94	C,R(0.01)	27.50	First reported 190.34
2836		-----		-----	
2863	ISO17226-2	56.49		-2.13	
3116	ISO17226-2	64.95		-0.77	
3146	ISO17226-2	76.9		1.13	
3150	ISO17226-2	55.5		-2.28	
3154		-----		-----	
3160	ISO17226-2	80.65		1.73	
3163		-----		-----	
3172	ISO17226-2	74.4		0.73	
3190	ISO17226-2	76.76		1.11	
3197	ISO17226-2	71.7		0.30	
3209		-----		-----	
3210		-----		-----	
3214	ISO17226-2	69.09		-0.11	
3216	ISO17226-2	59.12		-1.71	
3220	ISO17226-2	54.85		-2.39	
3222		-----		-----	
3228	ISO17226-2	NA		-----	
3237		-----		-----	
3248	ISO17226-2	53	C	-2.68	First reported 38.15
	normality	OK			
	n	65			
	outliers	2			
	mean (n)	69.800			
	st.dev. (n)	12.0146	RSD = 17%		
	R(calc.)	33.641			
	st.dev.(ISO17226-2:08)	6.2591			
	R(ISO17226-2:08)	17.525			

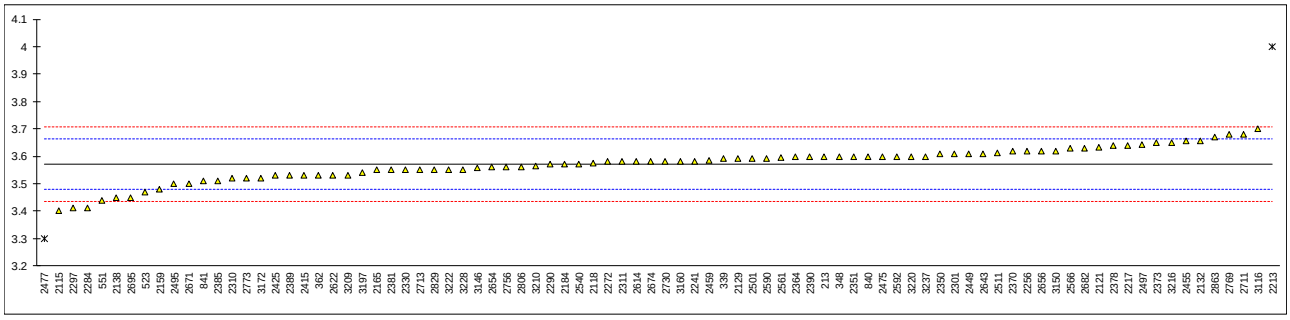


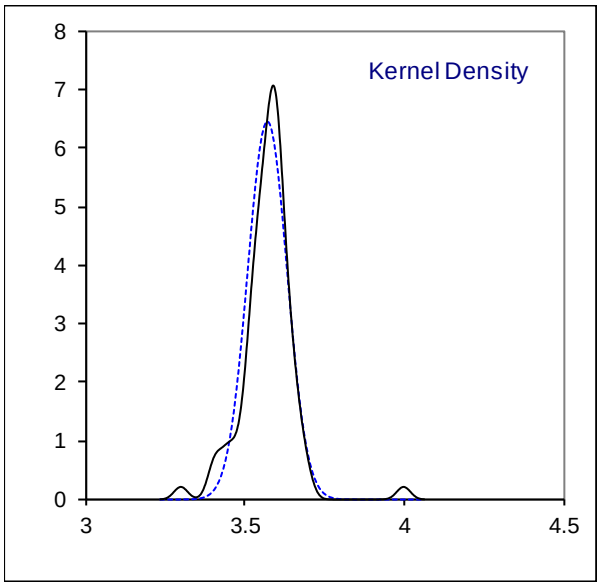


Determination of pH of extract on sample #18641; unitless results

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
213	ISO4045	3.6		0.62	
339	ISO4045	3.59		0.40	
348	ISO4045	3.60		0.62	
362	ISO4045	3.53		-0.92	
523	ISO4045	3.47		-2.24	
551	ISO4045	3.440		-2.90	
840	ISO4045	3.60		0.62	
841	ISO4045	3.51		-1.36	
2115	ISO4045	3.4		-3.78	
2118	ISO4045	3.575		0.07	
2121	ISO4045	3.634		1.37	
2129	ISO4045	3.59		0.40	
2132	ISO4045	3.657		1.87	
2138	ISO4045	3.45		-2.68	
2159	ISO4045	3.48		-2.02	
2165	ISO4045	3.55		-0.48	
2184	ISO4045	3.57		-0.04	
2213	ISO4045	4.00	R(0.01)	9.42	
2217	ISO4045	3.64		1.50	
2241	ISO4045	3.583		0.24	
2256	ISO4045	3.62		1.06	
2272	ISO4045	3.58		0.18	
2284	ISO4045	3.41		-3.56	
2290	ISO4045	3.57		-0.04	
2297	ISO4045	3.41		-3.56	
2301	ASTM D2810	3.61		0.84	
2310	ISO4045	3.52		-1.14	
2311	ISO4045	3.58		0.18	
2330	ISO4045	3.55		-0.48	
2350	ISO4045	3.61		0.84	
2351	ISO4045	3.60		0.62	
2364	ISO4045	3.60		0.62	
2370	ISO4045	3.62		1.06	
2373	ISO4045	3.65		1.72	
2374		-----		-----	
2378	ISO4045	3.64		1.50	
2381	ISO4045	3.55		-0.48	
2385	ISO4045	3.51		-1.36	
2389	ISO4045	3.53		-0.92	
2390	ISO4045	3.6		0.62	
2415	ISO4045	3.53		-0.92	
2425	ISO4045	3.53		-0.92	
2449	ISO4045	3.61		0.84	
2455	ISO4045	3.655		1.83	
2459	ISO4045	3.585		0.29	
2475	ISO4045	3.60		0.62	
2477	ISO4045	3.30	R(0.01)	-5.98	
2495	ISO4045	3.50		-1.58	
2497	ISO4045	3.641		1.52	
2501	ISO4045	3.59		0.40	
2511	ISO4045	3.612		0.88	
2540	ISO4045	3.57		-0.04	
2561	ISO4045	3.595		0.51	
2566	ISO4045	3.63		1.28	
2590	ISO4045	3.590		0.40	
2592	ISO4045	3.60		0.62	
2614	ISO4045	3.58		0.18	
2622	ISO4045	3.53		-0.92	
2643	ISO4045	3.61		0.84	
2654	ISO4045	3.56		-0.26	
2656	ISO4045	3.62		1.06	
2671	ISO4045	3.5		-1.58	
2674	ISO4045	3.58		0.18	
2682	ISO4045	3.63		1.28	
2695	ISO4045	3.450		-2.68	
2711	ISO4045	3.681		2.40	
2713	ISO4045	3.55		-0.48	
2730	ISO4045	3.58		0.18	
2756	In house	3.56		-0.26	
2769	ISO4045	3.680		2.38	
2773	ISO4045	3.52		-1.14	
2783		-----		-----	
2806	ISO4045	3.56		-0.26	
2829	ISO4045	3.55		-0.48	
2863	ISO4045	3.67		2.16	
3116	ISO4045	3.70		2.82	

lab	method	value	mark	z(targ)	remarks
3146	ISO4045	3.557		-0.33	
3150	ISO4045	3.62		1.06	
3160	ISO4045	3.58		0.18	
3172	ISO4045	3.52		-1.14	
3197	ISO4045	3.54		-0.70	
3209	ISO4045	3.532		-0.88	
3210	ISO4045	3.566		-0.13	
3216	ISO4045	3.65		1.72	
3220	ISO4045	3.60		0.62	
3222	ISO4045	3.55		-0.48	
3228	ISO4045	3.55		-0.48	
3237	ISO4045	3.6		0.62	
	normality	OK			
	n	84			
	outliers	2			
	mean (n)	3.572			
	st.dev. (n)	0.0618	RSD = 1.7%		
	R(calc.)	0.173			
	st.dev.(D2810:13)	0.0455			
	R(D2810:13)	0.127			

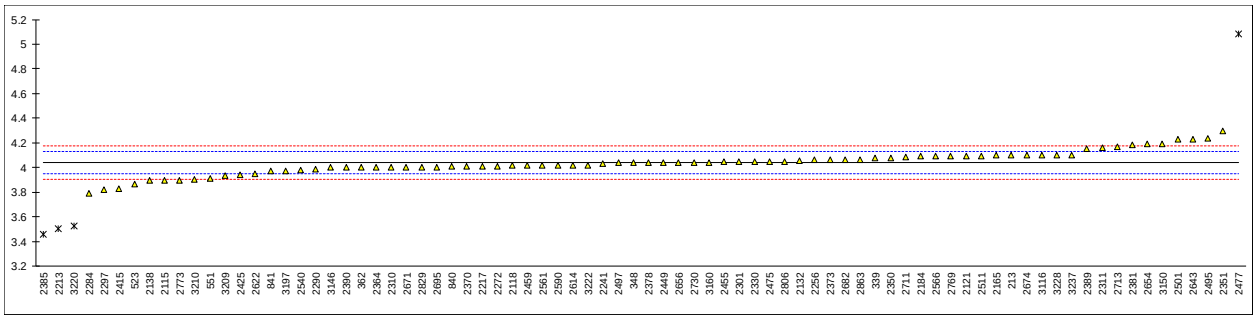


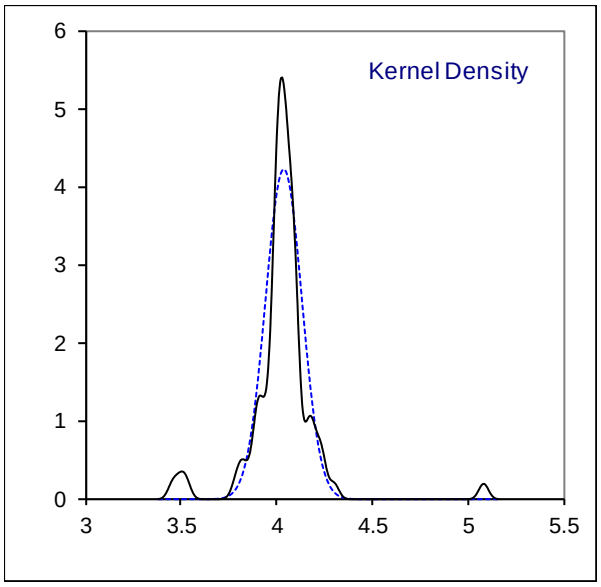


Determination of pH of ten times diluted extract on sample #18641; unitless results

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
213	ISO4045	4.1		1.36	
339	ISO4045	4.08		0.92	
348	ISO4045	4.04		0.04	
362	ISO4045	4.00		-0.84	
523	ISO4045	3.87		-3.70	
551	ISO4045	3.915		-2.71	
840	ISO4045	4.01		-0.62	
841	ISO4045	3.97		-1.50	
2115	ISO4045	3.9		-3.04	
2118	ISO4045	4.015		-0.51	
2121	ISO4045	4.092		1.18	
2129		-----		-----	
2132	ISO4045	4.057		0.41	
2138	ISO4045	3.90		-3.04	
2159		-----		-----	
2165	ISO4045	4.10		1.36	
2184	ISO4045	4.09		1.14	
2213	ISO4045	3.50	R(0.01)	-11.84	
2217	ISO4045	4.01		-0.62	
2241	ISO4045	4.030		-0.18	
2256	ISO4045	4.06		0.48	
2272	ISO4045	4.01		-0.62	
2284	ISO4045	3.79		-5.46	
2290	ISO4045	3.99		-1.06	
2297	ISO4045	3.82		-4.80	
2301	ASTM D2810	4.05		0.26	
2310	ISO4045	4.00		-0.84	
2311	ISO4045	4.16		2.68	
2330	ISO4045	4.05		0.26	
2350	ISO4045	4.08		0.92	
2351	ISO4045	4.30		5.76	
2364	ISO4045	4.00		-0.84	
2370	ISO4045	4.01		-0.62	
2373	ISO4045	4.06		0.48	
2374		-----		-----	
2378	ISO4045	4.04		0.04	
2381	ISO4045	4.18		3.12	
2385	ISO4045	3.46	R(0.01)	-12.72	
2389	ISO4045	4.15		2.46	
2390	ISO4045	4.0		-0.84	
2415	ISO4045	3.83		-4.58	
2425	ISO4045	3.94		-2.16	
2449	ISO4045	4.04		0.04	
2455	ISO4045	4.045		0.15	
2459	ISO4045	4.015		-0.51	
2475	ISO4045	4.05		0.26	
2477	ISO4045	5.08	R(0.01)	22.92	
2495	ISO4045	4.24		4.44	
2497	ISO4045	4.039		0.02	
2501	ISO4045	4.23		4.22	
2511	ISO4045	4.093		1.21	
2540	ISO4045	3.98		-1.28	
2561	ISO4045	4.015		-0.51	
2566	ISO4045	4.09		1.14	
2590	ISO4045	4.020		-0.40	
2592		-----		-----	
2614	ISO4045	4.02		-0.40	
2622	ISO4045	3.95		-1.94	
2643	ISO4045	4.23		4.22	
2654	ISO4045	4.19		3.34	
2656	ISO4045	4.04		0.04	
2671	ISO4045	4.0		-0.84	
2674	ISO4045	4.10		1.36	
2682	ISO4045	4.06		0.48	
2695	ISO4045	4.005		-0.73	
2711	ISO4045	4.087		1.07	
2713	ISO4045	4.17		2.90	
2730	ISO4045	4.04		0.04	
2756		-----		-----	
2769	ISO4045	4.090		1.14	
2773	ISO4045	3.90		-3.04	
2783		-----		-----	
2806	ISO4045	4.05		0.26	
2829	ISO4045	4.00		-0.84	
2863	ISO4045	4.06		0.48	
3116	ISO4045	4.10		1.36	

lab	method	value	mark	z(targ)	remarks
3146	ISO4045	3.999		-0.86	
3150	ISO4045	4.19		3.34	
3160	ISO4045	4.04		0.04	
3172		-----		-----	
3197	ISO4045	3.97		-1.50	
3209	ISO4045	3.931		-2.36	
3210	ISO4045	3.905		-2.93	
3216		-----		-----	
3220	ISO4045	3.53	R(0.01)	-11.18	
3222	ISO4045	4.02		-0.40	
3228	ISO4045	4.10		1.36	
3237	ISO4045	4.1		1.36	
	normality	OK			
	n	76			
	outliers	4			
	mean (n)	4.038			
	st.dev. (n)	0.0946	RSD = 2.3%		
	R(calc.)	0.265			
	st.dev.(D2810:13)	0.0455			
	R(D2810:13)	0.127			

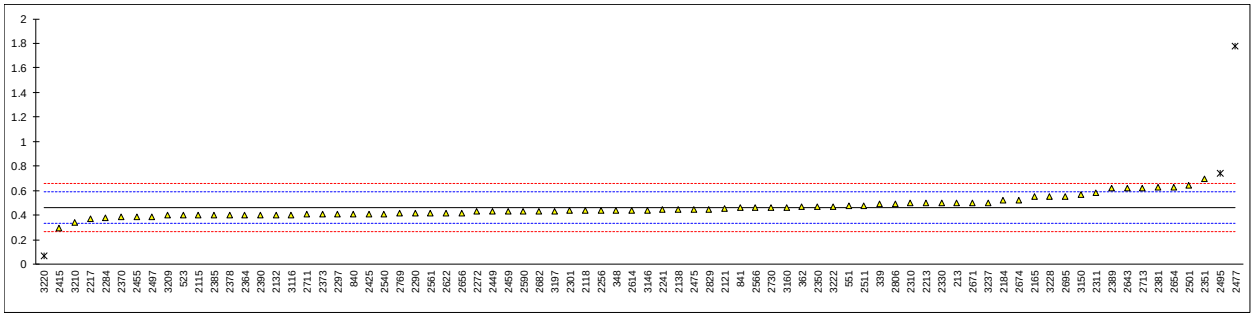


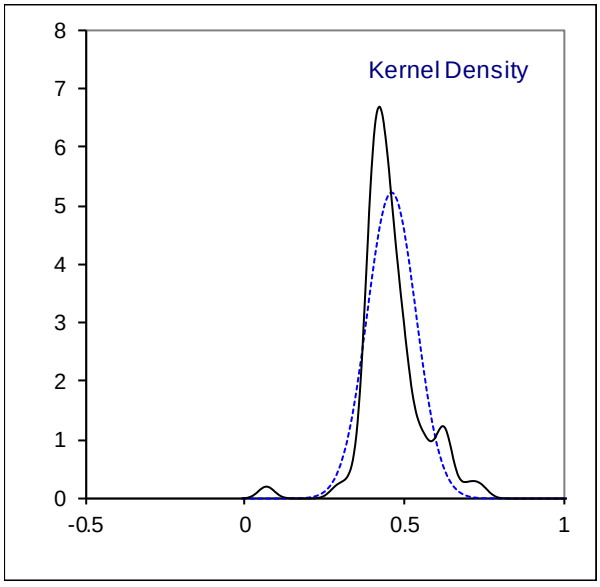


Determination of difference between pH of extract and pH of ten times diluted extract on sample #18641; unitless results

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
213	ISO4045	0.5		0.59	
339	ISO4045	0.49		0.43	
348	ISO4045	0.44		-0.35	
362	ISO4045	0.47		0.12	
523	ISO4045	0.4		-0.97	
551	ISO4045	0.475		0.20	
840	ISO4045	0.41		-0.81	
841	ISO4045	0.46		-0.04	
2115	ISO4045	0.4		-0.97	
2118	ISO4045	0.44		-0.35	
2121	ISO4045	0.458		-0.07	
2129		-----		-----	
2132	ISO4045	0.40		-0.97	
2138	ISO4045	0.45		-0.19	
2159		-----		-----	
2165	ISO4045	0.55		1.36	
2184	ISO4045	0.52		0.90	
2213	ISO4045	0.50		0.59	
2217	ISO4045	0.37		-1.44	
2241	ISO4045	0.447		-0.24	
2256	ISO4045	0.44		-0.35	
2272	ISO4045	0.43		-0.50	
2284	ISO4045	0.38		-1.28	
2290	ISO4045	0.42		-0.66	
2297	ISO4045	0.41		-0.81	
2301	ASTM D2810	0.44		-0.35	
2310	ISO4045	0.50		0.59	
2311	ISO4045	0.58		1.83	
2330	ISO4045	0.5		0.59	
2350	ISO4045	0.47		0.12	
2351	ISO4045	0.70		3.70	
2364	ISO4045	0.40		-0.97	
2370	ISO4045	0.39		-1.12	
2373	ISO4045	0.41		-0.81	
2374		-----		-----	
2378	ISO4045	0.40		-0.97	
2381	ISO4045	0.63		2.61	
2385	ISO4045	0.40		-0.97	
2389	ISO4045	0.62		2.45	
2390	ISO4045	0.4		-0.97	
2415	ISO4045	0.295		-2.60	
2425	ISO4045	0.41		-0.81	
2449	ISO4045	0.43		-0.50	
2455	ISO4045	0.39		-1.12	
2459	ISO4045	0.43		-0.50	
2475	ISO4045	0.45		-0.19	
2477	ISO4045	1.78	R(0.01)	20.50	
2495	ISO4045	0.74	R(0.05)	4.32	
2497	ISO4045	0.39		-1.12	
2501	ISO4045	0.64		2.76	
2511	ISO4045	0.481		0.29	
2540	ISO4045	0.41		-0.81	
2561	ISO4045	0.42		-0.66	
2566	ISO4045	0.46		-0.04	
2590	ISO4045	0.430		-0.50	
2592		-----		-----	
2614	ISO4045	0.44		-0.35	
2622	ISO4045	0.42		-0.66	
2643	ISO4045	0.62		2.45	
2654	ISO4045	0.63		2.61	
2656	ISO4045	0.42		-0.66	
2671	ISO4045	0.5		0.59	
2674	ISO4045	0.52		0.90	
2682	ISO4045	0.43		-0.50	
2695	ISO4045	0.555		1.44	
2711	ISO4045	0.406		-0.88	
2713	ISO4045	0.62		2.45	
2730	ISO4045	0.46		-0.04	
2756		-----		-----	
2769	ISO4045	0.415		-0.74	
2773		-----		-----	
2783		-----		-----	
2806	ISO4045	0.49		0.43	
2829	ISO4045	0.45		-0.19	
2863		-----		-----	
3116	ISO4045	0.40		-0.97	

lab	method	value	mark	z(targ)	remarks
3146	ISO4045	0.442		-0.32	
3150	ISO4045	0.57		1.68	
3160	ISO4045	0.46		-0.04	
3172		-----		-----	
3197	ISO4045	0.43		-0.50	
3209	ISO4045	0.399		-0.98	
3210	ISO4045	0.339		-1.92	
3216		-----		-----	
3220	ISO4045	0.07	R(0.01)	-6.10	
3222	ISO4045	0.47		0.12	
3228	ISO4045	0.55		1.36	
3237	ISO4045	0.5		0.59	
	normality	not OK			
	n	75			
	outliers	3			
	mean (n)	0.462			
	st.dev. (n)	0.0763	RSD = 17%		
	R(calc.)	0.214			
	st.dev.(D2810:13)	0.0643			
	R(D2810:13)	0.180			





APPENDIX 2

Analytical details for sample #18640 (Formaldehyde Determination)

lab	ISO/IEC17025 accredited	Sample further grinded/cut	Sample intake (in grams)	Number of days to complete the test	Remarks
110	Yes	Further Cut	1 gram	one	
213	Yes	---			
339	No	Further Cut	2g	2 days	
348	Yes	Further Cut	2	1	
362	Yes	Used as received	2.0054	1	
523	---	---			
551	Yes	Used as received	2.0	One	
623	---	---			
840	Yes	Further Cut	2g	1 day	
841	Yes	Used as received	2	1	
2115	Yes	Further Cut	2,0 g	1 day	
2118	No	Used as received	2	1 day	
2129	Yes	Used as received			
2132	Yes	Used as received	2g	1 day	
2137	Yes	Further Cut	0.4 g	1 day	
2138	Yes	Used as received	2.005 g	same day	
2165	Yes	Further Cut	2.000	<2	
2213	Yes	Used as received	2 grams	Same day of analysis	
2217	Yes	Used as received	2.0	1	ISO 17226-1 method not accredited
2256	Yes	Further Grinded	2 gram	1 days	
2261	Yes	Used as received	2.0 grams	In one day.	
2271	Yes	Further Cut	2.0g	1 day	
2272	Yes	Further Cut	2g		
2284	Yes	Further Cut	2.0001 grams	1 day	
2290	---	---			
2293	Yes	Used as received	2.00	1 day after received it	
2295	Yes	Further Cut	2 g	2 days	
2297	Yes	Further Cut	2	1	
2301	Yes	Further Cut	1	1	
2310	Yes	Further Cut	4 grams	2 days	
2311	Yes	Further Cut	2	1	
2330	Yes	Further Cut	1.00 grams	1 day	
2347	Yes	Further Cut			
2350	Yes	Further Cut	1 g	1 day	
2351	Yes	Further Cut	1g	1day	
2358	Yes	Used as received	5	7	
2363	Yes	Further Cut	2g	1 day	
2365	Yes	Used as received	1g	In 6 hours.	
2370	Yes	Further Cut	2 g	2 days	
2375	Yes	Further Cut	2 gr		
2378	Yes	Used as received	2g	15days	
2379	Yes	Further Cut	1 g	5 days for HPLC and 1 day for colorimetric	
2380	Yes	Used as received	2.00 g	2 days	
2381	Yes	Further Cut	2	2	
2382	No	Further Cut	1g	one day	
2386	Yes	Further Cut	1	1	
2389	---	---			
2390	Yes	Further Cut	2 grams	One day	
2410	Yes	Further Cut	2 g	5days	
2425	Yes	Further Cut	1	Same day	
2449	Yes	Further Grinded	2	1	
2452	---	---			
2455	Yes	Further Cut	2.03 grams	1	
2459	Yes	Further Cut	2.0 grams	1 day	
2460	Yes	Further Cut	2.0 g	one day	
2477	---	---			
2495	Yes	Further Cut	1.00	8	
2497	Yes	---	2	2	
2501	Yes	Further Cut	1.9932g	1 day	
2511	No	Further Cut	4 GRAMS	ONE DAY	
2540	Yes	Used as received	2 g	1 day	
2543	Yes	Further Cut	1,0 g	2 days	
2561	---	---			
2566	Yes	Further Cut	1.0010 gm	within one week	
2590	Yes	Further Cut	2.007	1 day	
2592	---	---			
2614	Yes	Further Cut	2.0 grams	one day	
2639	Yes	Further Cut	approx. 4 grams.	one day	
2643	Yes	Further Cut	6 g (2 g, 2g, 2g)	3 weeks	
2654	Yes	Further Grinded	2.0018	1 DAY	
2656	No	Further Cut	2	1	
2671	Yes	Used as received	2.0	10	
2674	---	---			
2682	Yes	Further Cut	2.0001	one day	
2695	Yes	Further Cut	2 grams per test	ONE DAY	

lab	ISO/IEC17025 accredited	Sample further grinded/cut	Sample intake (in grams)	Number of days to complete the test	Remarks
2711	---	---			
2713	Yes	Further Cut	2 g.	1 day	
2727	Yes	Further Cut	0,5	one day	
2730	No	Used as received	2	1	
2743	Yes	Further Cut	1.5	1	
2756	---	---			
2773	Yes	Further Cut	2.0g	1 day	
2783	---	---			
2806	Yes	Further Cut			
2810	Yes	Further Cut	4	2	
2829	No	Further Grinded	2	1	
2836	Yes	Further Cut	2 grams per replicate	1 day	No sufficient sample mass, results were reported in wet basis.
2863	No	Further Cut	about 2 g		
3116	Yes	Further Cut	2g	1 day	
3146	Yes	Further Cut	6 gram	3 different days	
3150	Yes	Further Cut	1		
3154	---	---			
3160	No	Further Cut	2 g	One day	
3163	---	---			
3172	Yes	---			
3190	Yes	Further Cut	1.0000	1	None
3197	Yes	Used as received	2 grams	10 days	
3209	Yes	Used as received	2g	1	
3210	Yes	Further Cut	1	1	
3214	Yes	Further Cut	2g	2 days	
3216	---	---			
3220	Yes	Further Cut	2.0gms	1week	
3222	No	Further Cut	2	8	
3228	Yes	Further Cut	1g	3days	
3237	Yes	Further Cut	2 g	24 days	
3248	Yes	Used as received	2.00 grams	1 day	

Analytical details for sample #18641 (pH Determination)

lab	ISO/IEC17025 accredited	Sample intake (in grams)	Additional steps to wet the sample
110	---		---
213	Yes		---
339	No	10 g	No
348	No	5	No
362	Yes	2g	No
523	No	5	No
551	No	5.0	No
840	Yes	2.5g	No
841	Yes	5	No
2115	Yes	5 g	No
2118	Yes	10 grams	No
2121	Yes	10	No
2129	Yes		---
2132	Yes	5g	No
2138	Yes	5.000 g	No
2159	Yes	5	No
2165	Yes	2	No
2184	Yes	2.5g	No
2213	Yes	5 grams	No
2217	Yes	5 g	Yes: Shaker
2241	Yes	5	No
2256	Yes	5	No
2272	---		---
2284	Yes	5.0007grams	No
2290	---		---
2297	Yes	5	No
2301	Yes	5	No
2310	Yes	7.5 grams	No
2311	Yes	5	No
2330	Yes	5 g	No
2350	No	5 g	No
2351	Yes	10g	No
2364	Yes	5.00g	No
2370	Yes	5 g	No
2373	Yes	2.5	No
2374	---		---
2378	Yes	5g	No
2381	Yes	5.0	No
2385	Yes	5	Yes: a vacuum step
2389	---		---
2390	Yes	5.0054, 5.0046	No
2415	Yes	2.5	No
2425	Yes	5	No
2449	Yes	5	No
2455	---		---
2459	Yes	5	No
2475	No	5	No
2477	Yes	5	No
2495	Yes	5	No
2497	Yes	5	No
2501	Yes	5.0023g and 5.0032g	No
2511	No	10 GRAMS	No
2540	Yes	5 g	No
2561	---		---
2566	Yes	5.0020gm	Yes
2590	Yes	5,004	No
2592	---		---
2614	Yes	5 grams	No
2622	No	5	No
2643	Yes	5 g	No
2654	Yes	5.0041	No
2656	No	5	No
2671	Yes	5	No
2674	Yes	5g	No
2682	Yes	5.0001	No
2695	Yes	5 grams for each analysis	No
2711	No	5.021	No
2713	Yes	about 5 g.	No
2730	No	5	No
2756	---		---
2769	Yes	5	No
2773	Yes	2.5	No
2783	---		---
2806	Yes		---
2829	No	5	No
2863	No	5	No

lab	ISO/IEC17025 accredited	Sample intake (in grams)	Additional steps to wet the sample
3116	Yes	5 grams	No
3146	Yes	12 gram	No
3150	Yes	2,5	No
3160	Yes	5 g	No
3172	Yes		---
3197	Yes	5 grams	No
3209	Yes	5g	No
3210	Yes	5.0	No
3216	Yes	5.0	No
3220	Yes	5gm	No
3222	Yes	5	No
3228	Yes	5g	No
3237	Yes	5 g	No

APPENDIX 3

Number of participants per country for iis18A12F

3 labs in	BANGLADESH
1 lab in	BELGIUM
3 labs in	BRAZIL
1 lab in	BULGARIA
1 lab in	CAMBODIA
1 lab in	ETHIOPIA
4 labs in	FRANCE
5 labs in	GERMANY
1 lab in	GUATEMALA
4 labs in	HONG KONG
1 lab in	HUNGARY
8 labs in	INDIA
2 labs in	INDONESIA
13 labs in	ITALY
6 labs in	KOREA
3 labs in	MEXICO
2 labs in	MOROCCO
19 labs in	P.R. of CHINA
5 labs in	PAKISTAN
1 lab in	POLAND
1 lab in	PORTUGAL
3 labs in	SPAIN
2 labs in	TAIWAN R.O.C.
1 lab in	THAILAND
1 lab in	THE NETHERLANDS
2 labs in	TUNISIA
5 labs in	TURKEY
3 labs in	U.S.A.
1 lab in	UNITED KINGDOM
3 labs in	VIETNAM

Number of participants per country for iis18A12P

2 labs in	BANGLADESH
1 lab in	BELGIUM
1 lab in	BRAZIL
1 lab in	BULGARIA
1 lab in	CAMBODIA
1 lab in	ETHIOPIA
6 labs in	FRANCE
4 labs in	GERMANY
3 labs in	HONG KONG
1 lab in	HUNGARY
8 labs in	INDIA
1 lab in	INDONESIA
12 labs in	ITALY
4 labs in	KOREA
1 lab in	MEXICO
2 labs in	MOROCCO
15 labs in	P.R. of CHINA
5 labs in	PAKISTAN
1 lab in	POLAND
1 lab in	PORTUGAL
3 labs in	SPAIN
1 lab in	TAIWAN R.O.C.
1 lab in	TUNISIA
4 labs in	TURKEY
3 labs 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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