Results of Proficiency Test Formaldehyde and pH in Leather November 2019

Organised by: Institute for Interlaboratory Studies
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1 INTRODUCTION

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 2019/2020, it was decided to continue the proficiency test for the analyzes of Formaldehyde content and pH in Leather.
In this interlaboratory study 125 laboratories in 31 different countries registered for participation in the PT Formaldehyde in Leather and 101 laboratories in 30 different countries registered for participation in the PT pH on Leather. In total 142 participants were registered. See appendix 3 for the number of participants per country. In this report, the results of the 2019 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. It was decided to send depending on the registration, one leather sample (labelled #19640) positive on Formaldehyde and one leather sample (labelled #19641) 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 brown leather which was grinded. After homogenization 140 subsamples of approximately 6 grams each were packed in a polypropylene bag, wrapped in Aluminum foil and labelled #19640.
The homogeneity of the subsamples was checked by the determination of the Formaldehyde content in accordance with an in-house test method on 8 stratified randomly selected samples.

	Formaldehyde in mg/kg
Sample #19640-1	130.87
Sample #19640-2	133.56
Sample #19640-3	133.98
Sample #19640-4	134.18
Sample #19640-5	133.25
Sample #19640-6	136.43
Sample #19640-7	139.46
Sample #19640-8	134.54

Table 1: homogeneity test results of subsamples #19640

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 ISO13528, Annex B2 in the next table.

	Formaldehyde in mg/kg
r (observed)	7.05
reference test method	ISO17226-1:08
0.3*R (ref. test method)	25.54

Table 2: evaluation of the repeatability of subsamples #19640

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 black leather which was also grinded. After homogenization 140 subsamples of approximately 10 grams each were packed in a polypropylene bag and labelled #19641.
The homogeneity of the subsamples was checked by the determination of the pH in accordance with an in-house test method on 8 stratified randomly selected samples.

	pH of extract
Sample #19641-1	3.53
Sample #19641-2	3.53
Sample #19641-3	3.53
Sample #19641-4	3.55
Sample #19641-5	3.50
Sample #19641-6	3.48
Sample #19641-7	3.50
Sample #19641-8	3.47

Table 3: homogeneity test results of subsamples #19641

From the above test results the relative standard deviation RSD_r were calculated and compared with 0.3 times the corresponding relative standard deviation RSD_R of the reference method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	pH of extract
RSD_r (observed)	0.8%
Reference test method	ASTM D2810:18
0.3 x RSD_R (ref. test method)	0.3%
0.3 x RSD_R (previous PTs)	0.8%

Table 4: evaluation of the relative standard deviation of subsamples #19641

The calculated relative standard deviation RSD_r of the subsamples was not in agreement with 0.3 times the corresponding RSD_R from ASTM D2810:18. However it is in agreement with 0.3 times the RSD_R from previous proficiency tests (see chapter 4.3, table 8). Therefore, the homogeneity of the subsamples was assumed.

Depending on the registration to each of the participants one sample labelled #19640 and/or one sample labelled #19641 was sent on October 9, 2019.

2.5 ANALYZES

The participants were requested to determine on sample #19640 the content of Formaldehyde (HPLC and Colorimetric) and on sample #19641 the pH of extract, pH of ten times diluted extract and the difference between the two pH measurements.
It was also requested to report if the laboratory was accredited for the components that were determined and 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 results cannot be used for meaningful statistical evaluations.

To get comparable 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 method 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 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 dataset 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’ 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 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 as met for all evaluated tests, therefore, the uncertainty of all assigned values maybe 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, e.g. ASTM, ISO reproducibilities, 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:

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

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare.
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

During the execution of this proficiency test no problems were encountered with the dispatch of the samples.
For the Formaldehyde proficiency test eight laboratories did not report any test results and one laboratory reported the test results after the final reporting date.
For the pH proficiency test four laboratories did not report any test results. No laboratory reported the test results after the final reporting date.
Finally, in total 136 reporting laboratories reported 441 numerical test results. Observed were 17 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 methods are also in the table together with the original data. 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 D2810:18 and ISO4045:08 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 #19640

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

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

Sample #19641

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

pH of 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 requirements of ASTM D2810:18.

Difference between pH of extract and pH ten times diluted extract: This determination may be problematic. Two 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 results, the calculated reproducibilities (2.8*standard deviation) and the target reproducibilities (ISO17226 and ASTM D2810), are compared in the next two tables.

Component	unit	n	average	2.8 * sd	R (target)
Formaldehyde (HPLC)	mg/kg	90	114.7	39.4	71.9
Formaldehyde (colorimetric)	mg/kg	60	87.4	20.6	21.4

Table 5: reproducibilities of tests on sample #19640

Parameter	unit	n	average	2.8 * sd	R (target)
pH of extract	-	95	3.56	0.25	0.13
pH of extract ten times diluted	-	89	4.08	0.26	0.13
Difference between pH	-	90	0.52	0.21	0.18

Table 6: reproducibilities of test on sample #19641

It can be concluded that, without statistical calculations, the group of participating laboratories has some difficulties with the determination of the pH, but have no problems with the Formaldehyde analyzes, 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 2019 WITH PREVIOUS PTS

	November 2019	November 2018	November 2017	November 2016	October 2015
Number of reporting labs	136	114	102	106	116
Number of results reported	441	396	378	240	239
Number of statistical outliers	17	12	16	16	7
Percentage outliers	3.9%	3.0%	4.2%	6.7%	2.9%

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

Table 8: development of uncertainties over the years

Improvement is visible in 2019 PT for the HPLC and Colorimetric determinations of Formaldehyde in Leather in comparison with the results in previous PTs. Both meet the estimated targets from the reference test methods.

For the pH determination the group did not improve and are in line 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. This same phenomenon was also noticed for the homogeneity testing. It was therefore decided to assume homogeneity of the subsamples based on the performance of the group in previous PT's.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

The reported details of the analytical test methods are listed in appendix 2. About 78% of all laboratories reported to be accredited for the determination of Formaldehyde in Leather and about 76% 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. The majority of the participants (75%) reported to have used the sample as received before testing, which was expected as the PT sample was grinded. However, no deviating test results were observed for participants that reported to further cut or to further grind the sample. Approximately 54% 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. The majority of the participants (63%) reported to have used 5 grams for intake. Fourteen other participants (14%) used only 1-2.5 grams and seven (7%) participants reported to have used 7.5 - 10 grams. 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. Remarkedly, this is not observed in PT sample #19640.

Sample #19640 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 reject this sample except three 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 again all laboratories would reject this sample except five laboratories. 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.

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

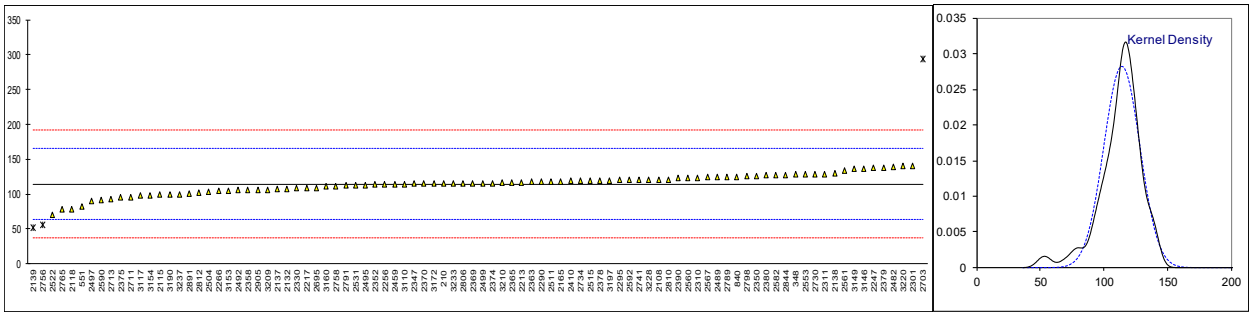
Determination of Formaldehyde content (HPLC) on sample #19640; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210	ISO17226-1	115.1		0.02	
230		-----		-----	
339		-----		-----	
348	In house	127.91		0.52	
362		-----		-----	
523		-----		-----	
551	In house	82.050	C	-1.27	First reported 301.7226
622		-----		-----	
623		-----		-----	
840	ISO17226-1	125.2		0.41	
2108	ISO17226-1	120.8		0.24	
2115	ISO17226-1	98.88		-0.61	
2118	ISO17226-1	78.675		-1.40	
2129		-----		-----	
2132	ISO17226-1	107.6		-0.27	
2137	ISO17226-1	107		-0.30	
2138	ISO17226-1	129.21		0.57	
2139	ISO17226-1	51.6	R(0.01)	-2.45	
2165	ISO17226-1	118.5		0.15	
2213	ISO17226-1	117		0.09	
2217	ISO17226-1	108.89		-0.22	
2247	ISO17226-1	138.11		0.91	
2256	ISO17226-1	114.3		-0.01	
2266	ISO17226-1	104.75		-0.39	
2273		-----		-----	
2290	ISO17226-1	118.12		0.13	
2293		-----		-----	
2295	ISO17226-1	120		0.21	
2301	ISO17226-1	140.73		1.02	
2310	ISO17226-1	123.7		0.35	
2311	ISO17226-1	129.06		0.56	
2330	ISO17226-1	108.17		-0.25	
2347	ISO17226-1	115		0.01	
2350	ISO17226-1	126.35		0.46	
2352	ISO17226-1	114.2		-0.02	
2357		-----		-----	
2358	ISO17226-1	106		-0.34	
2363	ISO17226-1	118		0.13	
2365	ISO17226-1	116.67		0.08	
2366	ISO17226-1	out of capability		-----	
2369	ISO17226-1	115.61		0.04	
2370	ISO17226-1	115		0.01	
2374	ISO17226-1	115.91		0.05	
2375	ISO17226-1	94.97		-0.77	
2378	ISO17226-1	119.60		0.19	
2379	ISO17226-1	138.38		0.92	
2380	ISO17226-1	127.0		0.48	
2381		-----		-----	
2382		-----		-----	
2390	ISO17226-1	122.87		0.32	
2410	ISO17226-1	119		0.17	
2449		-----		-----	
2452		-----		-----	
2453		-----		-----	
2455		-----		-----	
2459	ISO17226-1	114.40		-0.01	
2460		-----		-----	
2482	ISO17226-1	138.5		0.93	
2488		-----		-----	
2489	ISO17226-1	125		0.40	
2492	ISO17226-1	105.9		-0.34	
2495	ISO17226-1	112.94		-0.07	
2497	ISO17226-1	90.37		-0.95	
2499	GB/T19941	115.80		0.04	
2501		-----		-----	
2504	ISO17226-1	103.23		-0.44	
2511	ISO17226-1	118.40		0.15	
2515	ISO17226-1	119.55		0.19	
2522	GB/T19941	69.73		-1.75	
2531	ISO17226-1	112.37		-0.09	
2532	ISO17226-1	123.05		0.33	
2553	ISO17226-1	128.14		0.52	
2560	ISO17226-1	123.28		0.34	

lab	method	value	mark	z(targ)	remarks
2561	ISO17226-1	133.3		0.73	
2563		-----		-----	
2567	ISO17226-1	124.3		0.38	
2582	ISO17226-1	127.17	C	0.49	First reported 42.39
2590	ISO17226-1	92.13		-0.88	
2592	ISO17226-1	120		0.21	
2602		-----		-----	
2639		-----		-----	
2644		-----		-----	
2656		-----		-----	
2695	ISO17226-1	108.994		-0.22	
2703	ISO17226-1	293.7	R(0.01)	6.97	
2711	ISO17226-1	95.2		-0.76	
2713	ISO17226-1	92.6		-0.86	
2730	ISO17226-1	128.29		0.53	
2734	ISO17226-1	119.00		0.17	
2741	ISO17226-1	120		0.21	
2756	ISO17226-1	56.0	R(0.01)	-2.28	
2758	ISO17226-1	111.92		-0.11	
2765	ISO17226-1	78.534		-1.41	
2789	ISO17226-1	125.1		0.41	
2791	ISO17226-1	112.01		-0.10	
2798	ISO17226-1	126.0		0.44	
2806	ISO17226-1	115.5		0.03	
2810	ISO17226-1	121.05		0.25	
2812	ISO17226-1	102.59		-0.47	
2830		-----		-----	
2844	ISO17226-1	127.81		0.51	
2877		-----		-----	
2891	ISO17226-1	100.9		-0.54	
2904		-----		-----	
2905	ISO17226-1	106.04		-0.34	
3110	ISO17226-1	114.4		-0.01	
3116		-----		-----	
3117	ISO17226-1	98.18		-0.64	
3146	ISO17226-1	136.5		0.85	
3149	ISO17226-1	136.0		0.83	
3153	ISO17226-1	105.185		-0.37	
3154	ISO17226-1	98.21		-0.64	
3160	ISO17226-1	111.22		-0.13	
3172	ISO17226-1	115.0		0.01	
3176		-----		-----	
3190	ISO17226-1	98.93		-0.61	
3197	ISO17226-1	119.9		0.20	
3209	ISO17226-1	106.25		-0.33	
3210	ISO17226-1	116.45		0.07	
3220	ISO17226-1	140.51		1.01	
3228	ISO17226-1	120		0.21	
3233	ISO17226-1	115.16		0.02	
3237	ISO17226-1	99.8		-0.58	
3248		-----		-----	

	normality	OK
	n	90
	outliers	3
	mean (n)	114.657
	st.dev. (n)	14.0629
	R(calc.)	39.376
	st.dev.(ISO17226-1:08)	25.6853
	R(ISO17226-1:08)	71.919

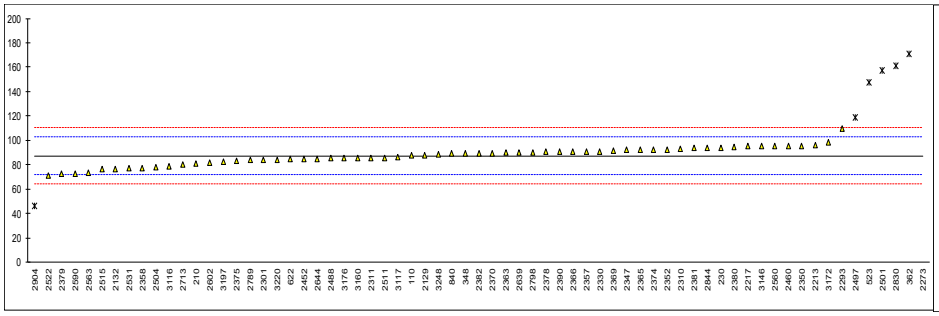
RSD = 12%

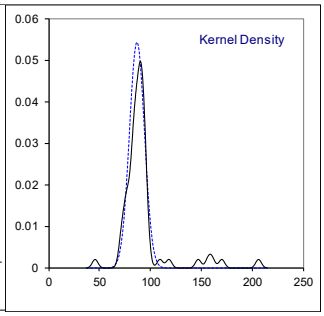


Determination of Formaldehyde content (colorimetric) on sample #19640; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	87.8207		0.06	
210	ISO17226-2	80.7		-0.87	
230	ISO17226-2	94.21		0.90	
339		-----		-----	
348	In house	89.23		0.24	
362	ISO17226-2	170.72	R(0.01)	10.91	
523	ISO17226-2	147.5258	R(0.01)	7.87	
551		-----		-----	
622	ISO17226-2	84.41	C	-0.39	First reported 37.08
623		-----		-----	
840	ISO17226-2	89.2		0.24	
2108		-----		-----	
2115		-----		-----	
2118		-----		-----	
2129	ISO17226-2	87.93		0.07	
2132	ISO17226-2	76.7		-1.40	
2137		-----		-----	
2138		-----		-----	
2139		-----		-----	
2165		-----		-----	
2213	ISO17226-2	96		1.13	
2217	ISO17226-2	95.1		1.01	
2247		-----		-----	
2256		-----		-----	
2266		-----		-----	
2273	ISO17226-2	206.78	C,R(0.01)	15.63	First reported 179.65
2290		-----		-----	
2293	ISO17226-2	109.93	C	2.95	First reported 129.93
2295		-----		-----	
2301	ISO17226-2	84.14	C	-0.42	First reported 140.03
2310	ISO17226-2	93.2		0.76	
2311	ISO17226-2	85.83		-0.20	
2330	ISO17226-2	91.12		0.49	
2347	ISO17226-2	92		0.61	
2350	ISO17226-2	95.72		1.09	
2352	ISO17226-2	92.4		0.66	
2357	ISO17226-2	91.1		0.49	
2358	ISO17226-2	77.50		-1.29	
2363	ISO17226-2	90		0.34	
2365	ISO17226-2	92.07		0.62	
2366	ISO17226-2	91.08		0.49	
2369	ISO17226-2	91.64		0.56	
2370	ISO17226-2	89.42		0.27	
2374	ISO17226-2	92.33		0.65	
2375	ISO17226-2	83.1		-0.56	
2378	ISO17226-2	90.60		0.42	
2379	ISO17226-2	72.69		-1.92	
2380	ISO17226-2	94.6		0.95	
2381	ISO17226-2	93.82		0.84	
2382	ISO17226-2	89.4		0.27	
2390	ISO17226-2	90.7		0.44	
2410		-----		-----	
2449		-----		-----	
2452	ISO17226-2	85.0		-0.31	
2453		-----		-----	
2455		-----		-----	
2459		-----		-----	
2460	ISO17226-2	95.648		1.08	
2482		-----		-----	
2488	ISO17226-2	85.2		-0.28	
2489		-----		-----	
2492		-----		-----	
2495		-----		-----	
2497	ISO17226-2	118.77	R(0.01)	4.11	
2499		-----		-----	
2501	ISO17226-2	157.39	C,R(0.01)	9.16	First reported 138.83
2504	ISO17226-2	77.69		-1.27	
2511	ISO17226-2	85.87		-0.20	
2515	ISO17226-2	76.67		-1.40	
2522	GB/T19941	71.34		-2.10	
2531	ISO17226-2	76.93		-1.37	
2532		-----		-----	
2553		-----		-----	
2560	ISO17226-2	95.62		1.08	

lab	method	value	mark	z(targ)	remarks
2561		-----		-----	
2563	ISO17226-2	73.5		-1.82	
2567		--		-----	
2582		-----		-----	
2590	ISO17226-2	72.88		-1.90	
2592		-----		-----	
2602	ISO17226-2	81.5		-0.77	
2639	GB/T19941	90.10		0.36	
2644	ISO17226-2	85.0		-0.31	
2656		-----		-----	
2695		-----		-----	
2703		-----		-----	
2711		-----		-----	
2713	ISO17226-2	80.5		-0.90	
2730		-----		-----	
2734		-----		-----	
2741		-----		-----	
2756		-----		-----	
2758		-----		-----	
2765		-----		-----	
2789	ISO17226-2	83.9		-0.45	
2791		-----		-----	
2798	ISO17226-2	90.3		0.38	
2806		-----		-----	
2810		-----		-----	
2812		-----		-----	
2830	ISO17226-2	161.2	R(0.01)	9.66	
2844	ISO17226-2	93.84		0.85	
2877		-----		-----	
2891		-----		-----	
2904	ISO17226-2	46.26	R(0.01)	-5.38	
2905		-----		-----	
3110		-----		-----	
3116	ISO17226-2	78.38		-1.18	
3117	ISO17226-2	86.51		-0.11	
3146	ISO17226-2	95.28		1.04	
3149		-----		-----	
3153		-----		-----	
3154		-----		-----	
3160	ISO17226-2	85.77		-0.21	
3172	ISO17226-2	98.6		1.47	
3176	ISO17226-2	85.490		-0.25	
3190		-----		-----	
3197	ISO17226-2	82.3		-0.66	
3209		-----		-----	
3210		-----		-----	
3220	ISO17226-2	84.34		-0.40	
3228		-----		-----	
3233		-----		-----	
3237		-----		-----	
3248	GB/T19941	88.4		0.13	
	normality	OK			
	n	60			
	outliers	7			
	mean (n)	87.371			
	st.dev. (n)	7.3545	RSD = 8%		
	R(calc.)	20.593			
	st.dev.(ISO17226-2:08)	7.6403			
	R(ISO17226-2:08)	21.393			



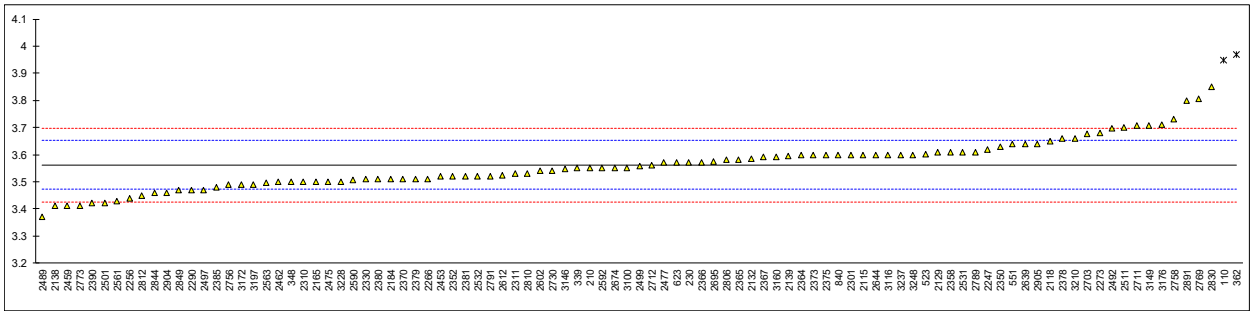


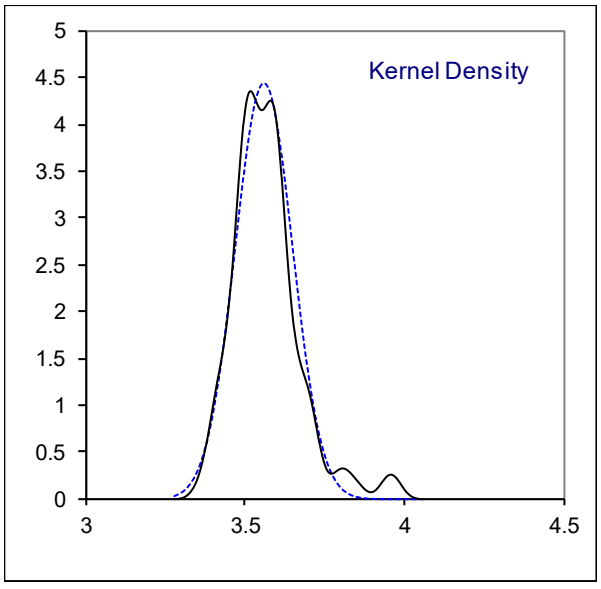
Determination of pH of extract on sample #19641; unitless results

lab	method	value	mark	z(targ)	remarks
110	In house	3.95	R(0.01)	8.53	
210	ISO4045	3.55		-0.27	
230	ISO4045	3.57		0.17	
339	ISO4045	3.55		-0.27	
348	ISO4045	3.50		-1.37	
362	ISO4045	3.97	C,R(0.01)	8.97	First reported 4.97
523	ISO4045	3.602		0.88	
551	ISO4045	3.640		1.71	
623	ISO4045	3.57		0.17	
840	ISO4045	3.60		0.83	
2115	ISO4045	3.60		0.83	
2118	ISO4045	3.65		1.93	
2129	ISO4045	3.61		1.05	
2132	ISO4045	3.585		0.50	
2138	ISO4045	3.41		-3.35	
2139	ISO4045	3.595		0.72	
2165	ISO4045	3.50		-1.37	
2184	ISO4045	3.51		-1.15	
2241		-----		-----	
2247	ISO4045	3.62	C	1.27	First reported 3.31
2256	ISO4045	3.44		-2.69	
2266	ISO4045	3.51		-1.15	
2273	ISO4045	3.68		2.59	
2290	ISO4045	3.47		-2.03	
2301	ASTM D2810	3.60		0.83	
2310	ISO4045	3.5		-1.37	
2311	ISO4045	3.53		-0.71	
2330	ISO4045	3.51		-1.15	
2350	ISO4045	3.63		1.49	
2352	ISO4045	3.52		-0.93	
2358	ISO4045	3.61		1.05	
2364	ISO4045	3.60		0.83	
2365	ISO4045	3.582		0.44	
2366	ISO4045	3.57		0.17	
2367	ISO4045	3.59		0.61	
2370	ISO4045	3.51		-1.15	
2373	ISO4045	3.60		0.83	
2375	ISO4045	3.6		0.83	
2378	ISO4045	3.66		2.15	
2379	ISO4045	3.51	C	-1.15	First reported 5.91
2380	ISO4045	3.51		-1.15	
2381	ISO4045	3.52		-0.93	
2385	ISO4045	3.48		-1.81	
2390	ISO4045	3.42		-3.13	
2453	ISO4045	3.52		-0.93	
2459	ISO4045	3.41		-3.35	
2462	ISO4045	3.50		-1.37	
2475	ISO4045	3.50		-1.37	
2477	ISO4045	3.57		0.17	
2489	ISO4045	3.37		-4.23	
2492	ISO4045	3.696		2.95	
2497	ISO4045	3.47		-2.03	
2499	ISO4045	3.556		-0.13	
2501	ISO4045	3.42		-3.13	
2511	ISO4045	3.7		3.03	
2531	ISO4045	3.61		1.05	
2532	ISO4045	3.52		-0.93	
2561	ISO4045	3.43		-2.91	
2563	ISO4045	3.498		-1.41	
2590	ISO4045	3.505		-1.26	
2592	ISO4045	3.55		-0.27	
2602	ISO4045	3.54		-0.49	
2612	ISO4045	3.52475		-0.82	
2639		3.64		1.71	
2644	ISO4045	3.60		0.83	
2656		-----		-----	
2674	ISO4045	3.55		-0.27	
2695	ISO4045	3.575		0.28	
2703	ISO4045	3.675		2.48	
2705		-----		-----	
2711	ISO4045	3.707		3.19	
2712	ISO4045	3.56		-0.05	
2730	ISO4045	3.54		-0.49	
2756	In house	3.49		-1.59	
2758	ISO4045	3.73		3.69	

lab	method	value	mark	z(targ)	remarks
2769	ISO4045	3.805		5.34	
2773	ISO4045	3.41	C	-3.35	First reported 3.28
2789	ISO4045	3.61		1.05	
2791	ISO4045	3.52		-0.93	
2806	ISO4045	3.58		0.39	
2810	ISO4045	3.53		-0.71	
2812	ISO4045	3.45		-2.47	
2830	ISO4045	3.85		6.33	
2844	ISO4045	3.46		-2.25	
2849	ISO4045	3.468	C	-2.07	First reported 4.006
2877		-----		-----	
2891	ISO4045	3.80	C	5.23	First reported 3.31
2904	ISO4045	3.46		-2.25	
2905	ISO4045	3.64		1.71	
3100	ISO4045	3.55		-0.27	
3116	ISO4045	3.60		0.83	
3146	ISO4045	3.547		-0.33	
3149	ISO4045	3.707		3.19	
3160	ISO4045	3.59		0.61	
3172	ISO4045	3.49		-1.59	
3176	ISO4045	3.71		3.25	
3197	ISO4045	3.49		-1.59	
3210	ISO4045	3.66		2.15	
3228	ISO4045	3.50		-1.37	
3237	ISO4045	3.6		0.83	
3248	ISO4045	3.60	C	0.83	First reported 3.13

				Use only 5 gram for intake:	
	normality	OK		OK	
	n	95		63	
	outliers	2		1	
	mean (n)	3.562		3.552	
	st.dev. (n)	0.0898	RSD = 2.5%	0.0923	RSD = 2.6%
	R(calc.)	0.251		0.258	
	st.dev.(D2810:18)	0.0455		0.0455	
	R(D2810:18)	0.127		0.127	



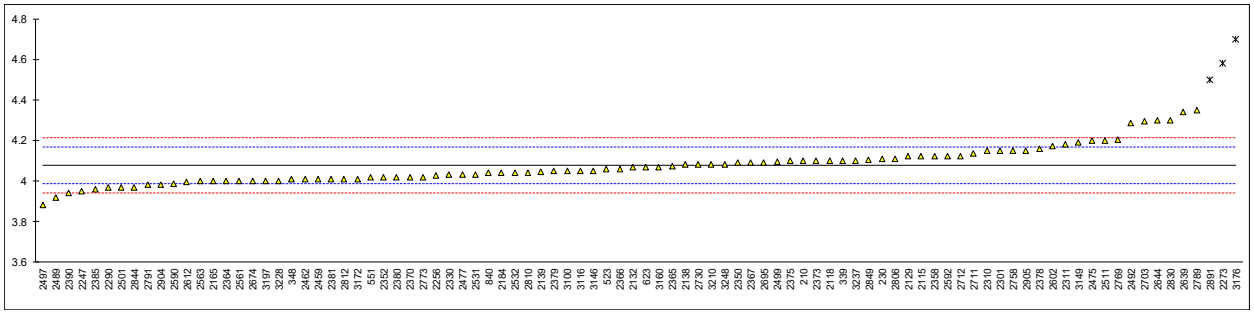


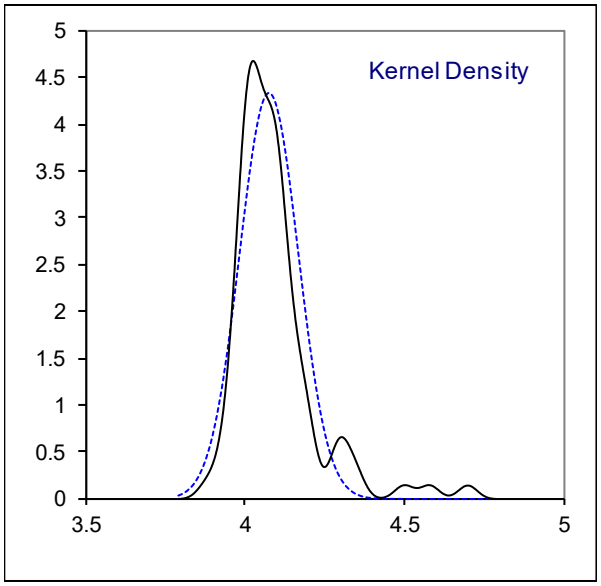
Determination of pH of ten times diluted extract on sample #19641; unitless results

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210	ISO4045	4.1		0.55	
230	ISO4045	4.11	C	0.77	First reported 4.63
339	ISO4045	4.10		0.55	
348	ISO4045	4.01		-1.43	
362		-----		-----	
523	ISO4045	4.057		-0.40	
551	ISO4045	4.016		-1.30	
623	ISO4045	4.07		-0.11	
840	ISO4045	4.04		-0.77	
2115	ISO4045	4.12		0.99	
2118	ISO4045	4.10		0.55	
2129	ISO4045	4.12		0.99	
2132	ISO4045	4.07		-0.11	
2138	ISO4045	4.08		0.11	
2139	ISO4045	4.045		-0.66	
2165	ISO4045	4.00		-1.65	
2184	ISO4045	4.04		-0.77	
2241		-----		-----	
2247	ISO4045	3.95		-2.75	
2256	ISO4045	4.025		-1.10	
2266		-----		-----	
2273	ISO4045	4.58	C,R(0.01)	11.11	First reported 4.38
2290	ISO4045	3.97		-2.31	
2301	ASTM D2810	4.15		1.65	
2310	ISO4045	4.15		1.65	
2311	ISO4045	4.18		2.31	
2330	ISO4045	4.03		-0.99	
2350	ISO4045	4.09		0.33	
2352	ISO4045	4.02		-1.21	
2358	ISO4045	4.12		0.99	
2364	ISO4045	4.00	C	-1.65	First reported 3.2
2365	ISO4045	4.071		-0.09	
2366	ISO4045	4.06		-0.33	
2367	ISO4045	4.09		0.33	
2370	ISO4045	4.02		-1.21	
2373	ISO4045	4.10		0.55	
2375	ISO4045	4.1		0.55	
2378	ISO4045	4.16		1.87	
2379	ISO4045	4.05	C	-0.55	First reported 6.48
2380	ISO4045	4.02		-1.21	
2381	ISO4045	4.01		-1.43	
2385	ISO4045	3.96		-2.53	
2390	ISO4045	3.94		-2.97	
2453		-----		-----	
2459	ISO4045	4.01		-1.43	
2462	ISO4045	4.01		-1.43	
2475	ISO4045	4.20		2.75	
2477	ISO4045	4.03		-0.99	
2489	ISO4045	3.92		-3.41	
2492	ISO4045	4.286		4.64	
2497	ISO4045	3.88		-4.29	
2499	ISO4045	4.096		0.46	
2501	ISO4045	3.97		-2.31	
2511	ISO4045	4.2	C	2.75	First reported 4.3
2531	ISO4045	4.03		-0.99	
2532	ISO4045	4.04		-0.77	
2561	ISO4045	4.00		-1.65	
2563	ISO4045	3.998		-1.70	
2590	ISO4045	3.985		-1.98	
2592	ISO4045	4.12		0.99	
2602	ISO4045	4.17		2.09	
2612	ISO4045	3.9945		-1.77	
2639		4.34		5.83	
2644	ISO4045	4.30		4.95	
2656		-----		-----	
2674	ISO4045	4.00		-1.65	
2695	ISO4045	4.09		0.33	
2703	ISO4045	4.295		4.84	
2705		-----		-----	
2711	ISO4045	4.136		1.34	
2712	ISO4045	4.12		0.99	
2730	ISO4045	4.08		0.11	
2756		-----		-----	
2758	ISO4045	4.15		1.65	

lab	method	value	mark	z(targ)	remarks
2769	ISO4045	4.203		2.81	
2773	ISO4045	4.02	C	-1.21	First reported 3.66
2789	ISO4045	4.35		6.05	
2791	ISO4045	3.98		-2.09	
2806	ISO4045	4.11		0.77	
2810	ISO4045	4.04		-0.77	
2812	ISO4045	4.01		-1.43	
2830	ISO4045	4.30		4.95	
2844	ISO4045	3.97		-2.31	
2849	ISO4045	4.102	C	0.59	First reported 4.742
2877		-----		-----	
2891	ISO4045	4.50	C,R(0.01)	9.35	First reported 3.7
2904	ISO4045	3.98		-2.09	
2905	ISO4045	4.15		1.65	
3100	ISO4045	4.05		-0.55	
3116	ISO4045	4.05		-0.55	
3146	ISO4045	4.051		-0.53	
3149	ISO4045	4.191		2.55	
3160	ISO4045	4.07		-0.11	
3172	ISO4045	4.01		-1.43	
3176	ISO4045	4.7	C,R(0.01)	13.75	First reported 4.3
3197	ISO4045	4.00		-1.65	
3210	ISO4045	4.08		0.11	
3228	ISO4045	4.00		-1.65	
3237	ISO4045	4.1		0.55	
3248	ISO4045	4.08	C	0.11	First reported 3.68

	normality	suspect	
	n	89	
	outliers	3	
	mean (n)	4.075	
	st.dev. (n)	0.0921	RSD = 2.3%
	R(calc.)	0.258	
	st.dev.(D2810:18)	0.0455	
	R(D2810:18)	0.127	



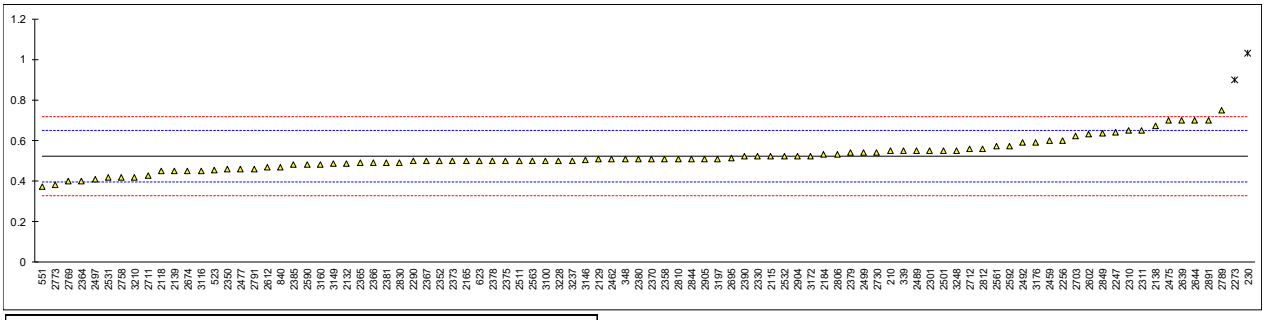


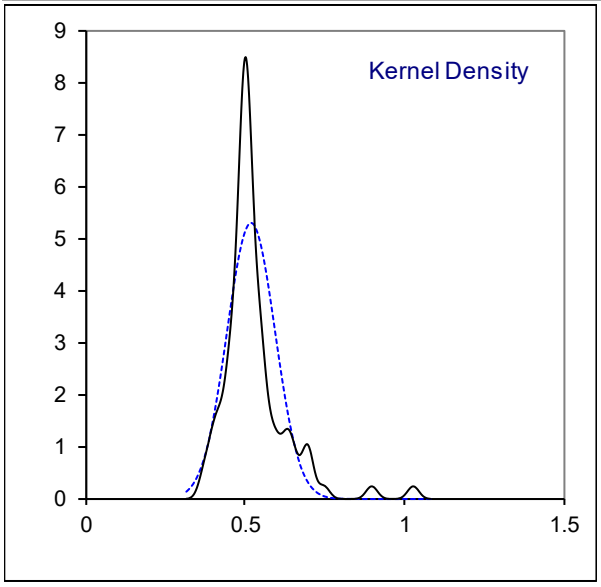
Determination of difference between pH of extract and pH of ten times diluted extract on sample #19641; unitless results

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210	ISO4045	0.55		0.43	
230	ISO4045	1.03	C,R(0.01)	7.90	First reported 1.06
339	ISO4045	0.55		0.43	
348	ISO4045	0.51		-0.19	
362		-----		-----	
523	ISO4045	0.455		-1.04	
551	ISO4045	0.373		-2.32	
623	ISO4045	0.5		-0.34	
840	ISO4045	0.47		-0.81	
2115	ISO4045	0.52		-0.03	
2118	ISO4045	0.45		-1.12	
2129	ISO4045	0.51		-0.19	
2132	ISO4045	0.485		-0.58	
2138	ISO4045	0.67		2.30	
2139	ISO4045	0.450		-1.12	
2165	ISO4045	0.50		-0.34	
2184	ISO4045	0.53		0.12	
2241		-----		-----	
2247	ISO4045	0.64		1.83	
2256	ISO4045	0.60		1.21	
2266		-----		-----	
2273	ISO4045	0.9	C,R(0.01)	5.88	First reported 0.70
2290	ISO4045	0.5		-0.34	
2301	ASTM D2810	0.55		0.43	
2310	ISO4045	0.65		1.99	
2311	ISO4045	0.65		1.99	
2330	ISO4045	0.52		-0.03	
2350	ISO4045	0.46		-0.97	
2352	ISO4045	0.50		-0.34	
2358	ISO4045	0.51		-0.19	
2364	ISO4045	0.40		-1.90	
2365	ISO4045	0.489		-0.52	
2366	ISO4045	0.49		-0.50	
2367	ISO4045	0.50		-0.34	
2370	ISO4045	0.51		-0.19	
2373	ISO4045	0.50		-0.34	
2375	ISO4045	0.5		-0.34	
2378	ISO4045	0.50		-0.34	
2379	ISO4045	0.54	C	0.28	First reported 0.57
2380	ISO4045	0.51		-0.19	
2381	ISO4045	0.49		-0.50	
2385	ISO4045	0.48		-0.66	
2390	ISO4045	0.52		-0.03	
2453		-----		-----	
2459	ISO4045	0.60		1.21	
2462	ISO4045	0.51		-0.19	
2475	ISO4045	0.7		2.77	
2477	ISO4045	0.46		-0.97	
2489	ISO4045	0.55		0.43	
2492	ISO4045	0.590		1.06	
2497	ISO4045	0.41		-1.74	
2499	ISO4045	0.540		0.28	
2501	ISO4045	0.55		0.43	
2511	ISO4045	0.5	C	-0.34	First reported 0.6
2531	ISO4045	0.42		-1.59	
2532	ISO4045	0.52		-0.03	
2561	ISO4045	0.57		0.74	
2563	ISO4045	0.5		-0.34	
2590	ISO4045	0.4800		-0.66	
2592	ISO4045	0.57		0.74	
2602	ISO4045	0.63		1.68	
2612	ISO4045	0.46975		-0.82	
2639		0.70		2.77	
2644	ISO4045	0.70		2.77	
2656		-----		-----	
2674	ISO4045	0.45		-1.12	
2695	ISO4045	0.515		-0.11	
2703	ISO4045	0.62		1.52	
2705		-----		-----	
2711	ISO4045	0.429		-1.45	
2712	ISO4045	0.56		0.59	
2730	ISO4045	0.54		0.28	
2756		-----		-----	
2758	ISO4045	0.42		-1.59	

lab	method	value	mark	z(targ)	remarks
2769	ISO4045	0.398		-1.93	
2773	ISO4045	0.38		-2.21	
2789	ISO4045	0.75		3.54	
2791	ISO4045	0.46		-0.97	
2806	ISO4045	0.53		0.12	
2810	ISO4045	0.51		-0.19	
2812	ISO4045	0.56		0.59	
2830	ISO4045	0.49		-0.50	
2844	ISO4045	0.51		-0.19	
2849	ISO4045	0.634	C	1.74	First reported 0.736
2877		-----		-----	
2891	ISO4045	0.7	C	2.77	First reported 0.4
2904	ISO4045	0.52		-0.03	
2905	ISO4045	0.51		-0.19	
3100	ISO4045	0.50		-0.34	
3116	ISO4045	0.45		-1.12	
3146	ISO4045	0.504		-0.28	
3149	ISO4045	0.484		-0.59	
3160	ISO4045	0.48		-0.66	
3172	ISO4045	0.52		-0.03	
3176	ISO4045	0.59		1.06	
3197	ISO4045	0.51		-0.19	
3210	ISO4045	0.42		-1.59	
3228	ISO4045	0.50		-0.34	
3237	ISO4045	0.5		-0.34	
3248	ISO4045	0.55		0.43	

normality	OK
n	90
outliers	2
mean (n)	0.522
st.dev. (n)	0.0752
R(calc.)	0.211
st.dev.(D2810:18)	0.0643
R(D2810:18)	0.180





APPENDIX 2

Analytical details for sample #19640 (Formaldehyde Determination)

lab	ISO/IEC17025 accredited	Sample further grinded/cut	Sample intake (in grams)	Number of days to complete the test
110	Yes	Used as received	2	1
210	Yes	---		
230	Yes	Used as received	2	1
339	---	---		
348	Yes	Used as received	2	2
362	Yes	Used as received	2	1
523	No	Used as received	2	1
551	Yes	Further Cut	2.0	1
622	Yes	Used as received	2	1
623	---	---		
840	Yes	Used as received	2	1
2108	Yes	Used as received	0,5	
2115	---	---		
2118	No	Used as received		1
2129	Yes	Used as received		
2132	Yes	Used as received	2	1
2137	Yes	Used as received	2	1
2138	Yes	Used as received	2	3
2139	Yes	Used as received	4.4	2
2165	Yes	Further Cut	2	7
2213	Yes	Used as received	1	5
2217	Yes	Used as received	2,0223	6
2247	---	---		
2256	Yes	Used as received	2	
2266	Yes	Further Grinded	2	
2273	Yes	Used as received	2	1
2290	---	---		
2293	Yes	Used as received	2	1
2295	Yes	Further Cut	2.5	
2301	Yes	Used as received	1	1
2310	Yes	Used as received	4	2
2311	Yes	Used as received	2	17
2330	Yes	Further Cut	1	2
2347	Yes	---		
2350	Yes	Used as received	2	1
2352	Yes	Used as received	0.5 / 1	15
2357	Yes	Used as received	2	
2358	Yes	Further Cut	1	2
2363	Yes	---		
2365	Yes	Used as received	5	3
2366	Yes	Used as received	1	1
2369	Yes	Used as received	2 / 0.5	
2370	Yes	Used as received	2	2
2374	Yes	Used as received	1.0 / 0.9	15
2375	Yes	Further Cut		
2378	Yes	Used as received	2	15
2379	No	Used as received	1	2
2380	Yes	Used as received	2.00	2
2381	Yes	Used as received	2	2
2382	Yes	Further Cut	2	7
2390	Yes	Used as received	2.0035	2
2410	Yes	Used as received	0.5	7
2449	---	---		
2452	Yes	Used as received	2.5083	9
2453	---	---		
2455	---	---		
2459	Yes	Used as received	2.0	1
2460	Yes	Used as received	2	1
2482	Yes	Used as received	2	1
2488	Yes	Used as received	2	
2489	Yes	Used as received	2	1
2492	Yes	Further Cut	1.0	
2495	Yes	Used as received	1,0	9
2497	---	---		
2499	Yes	Used as received	1.9974	1
2501	Yes	Used as received	2.0 / 2.0	1
2504	Yes	Used as received	2	1
2511	---	---		
2515	Yes	Used as received	1	1
2522	Yes	Used as received	2	4
2531	Yes	Used as received	2	1
2532	Yes	Used as received	1	1
2553	Yes	Used as received	2.0053	1

lab	ISO/IEC17025 accredited	Sample further grinded/cut	Sample intake (in grams)	Number of days to complete the test
2560	Yes	Used as received	2.0	7
2561	Yes	Used as received	2	1
2563	Yes	Used as received	1 - 2	1
2567	Yes	Further Cut	1.0	3
2582	Yes	Used as received	2.0	1
2590	Yes	Used as received	2.0	1
2592	Yes	Used as received	2	10
2602	Yes	Used as received	2	1
2639	Yes	Used as received	3.9927	4
2644	No	Used as received	2	1
2656	---	---		
2695	Yes	Used as received	4	1
2703	Yes	Used as received	2	1
2711	No	Used as received	2,008	
2713	Yes	Used as received	2	1
2730	No	Used as received	1,5	
2734	Yes	Used as received		1
2741	Yes	Further Cut	2	2
2756	Yes	Used as received	2.0	
2758	No	Used as received	1	1
2765	Yes	Used as received	2	1
2789	Yes	Used as received	1	7
2791	Yes	Used as received	2	1
2798	Yes	Used as received	0.5	
2806	Yes	Further Cut		
2810	Yes	Used as received	4.0	1
2812	No	Further Cut	2	1
2830	---	---		
2844	No	Used as received	2 +/- 0.1	1
2877	---	---		
2891	Yes	Used as received	4 / 2	2
2904	No	Used as received	2 / 2	1
2905	No	Used as received	2 per test	29
3110	Yes	Used as received	0.5	
3116	Yes	Used as received	2	
3117	Yes	Used as received	2.00	1
3146	Yes	Used as received	2.00	28
3149	Yes	Used as received	2	2
3153	Yes	Used as received	2	1
3154	Yes	Used as received	2	1
3160	Yes	Used as received	2	1
3172	Yes	Further Grinded		
3176	No	Used as received	1	8
3190	Yes	Used as received	0.9970	20
3197	Yes	Used as received	2	
3209	Yes	Used as received	0.5	8
3210	Yes	Used as received	1	1
3220	Yes	Used as received	2	2
3228	Yes	Used as received	1	15
3233	No	Used as received	2.0594	3
3237	---	---		
3248	Yes	Used as received	2	1

Analytical details for sample #19641 (pH Determination)

lab	ISO/IEC17025 accredited	Sample intake (in grams)	Additional steps to wet the sample
110	Yes		---
210	Yes		---
230	Yes	2.5	No
339	---		---
348	No	5	No
362	Yes	5	No
523	No	5	No
551	Yes	5,0021	No
623	Yes	2	No
840	Yes	8	Yes: other
2115	Yes	5	No
2118	Yes	5+-0.1	No
2129	Yes		---
2132	Yes	5.0001	No
2138	Yes	5	No
2139	Yes	10	No
2165	Yes	5	No
2184	No	5	No
2241	---		---
2247	---		---
2256	Yes	5.0001 / 4.9753	No
2266	Yes	2.5	No
2273	Yes	5.0	No
2290	---		---
2301	---		---
2310	Yes	7.5	No
2311	Yes	5	No
2330	Yes	5	No
2350	No	5.0	No
2352	Yes		No
2358	Yes	5	No
2364	Yes	5.00	No
2365	Yes	2	No
2366	Yes	1	No
2367	Yes	5	No
2370	Yes	5.0	No
2373	Yes	5	No
2375	Yes	2.5	No
2378	Yes	5	No
2379	Yes	5.00	No
2380	Yes	5.00	No
2381	Yes	5	No
2385	Yes	5.02	No
2390	Yes	5.0019	No
2453	No	5	No
2459	Yes	5	No
2462	Yes	10	No
2475	No	5	No
2477	Yes	4.9961	No
2489	Yes	5	No
2492	Yes	2.5	No
2497	---		---
2499	Yes	5	No
2501	Yes	4.9947 / 5.0003	No
2511	---		---
2531	Yes	5	No
2532	Yes	2.5	No
2561	No	5	No
2563	Yes	5	No
2590	Yes	5	No
2592	Yes	5	No
2602	Yes	1,000	No
2612	Yes	2,5	No
2639	Yes	5.0015	No
2644	Yes	10	No
2656	---		---
2674	Yes	5	No
2695	Yes	10	No
2703	Yes	5	No
2705	---		---
2711	No	5,000	No
2712	Yes	5	Yes: a vacuum step
2730	No	5	No

lab	ISO/IEC17025 accredited	Sample intake (in grams)	Additional steps to wet the sample
2756	Yes	5.0	No
2758	No	1.25	No
2769	Yes	5	No
2773	Yes	5	No
2789	Yes	5	No
2791	Yes	5	No
2806	Yes	5	---
2810	Yes	10.0	No
2812	Yes	5	No
2830	---		---
2844	No	5.03	No
2849	Yes	5,0	No
2877	---		---
2891	Yes	5	Yes: other
2904	No	5	No
2905	No	5	No
3100	Yes	5	No
3116	Yes	5	No
3146	Yes	2.00	No
3149	Yes	5	No
3160	Yes	5	No
3172	Yes	2.5	No
3176	Yes	2.5	No
3197	Yes	5	No
3210	Yes	5	No
3228	Yes	5	No
3237	Yes	5	No
3248	Yes	5	No

APPENDIX 3
Number of participants per country

iis19A14F	
3 labs in	BANGLADESH
1 lab in	BELGIUM
2 labs in	BRAZIL
1 lab in	BULGARIA
2 labs in	CAMBODIA
1 lab in	ETHIOPIA
6 labs in	FRANCE
8 labs in	GERMANY
1 lab in	GUATEMALA
8 labs in	HONG KONG
1 lab in	HUNGARY
9 labs in	INDIA
3 labs in	INDONESIA
16 labs in	ITALY
1 lab in	MAURITIUS
2 labs in	MEXICO
2 labs in	MOROCCO
20 labs in	P.R. of CHINA
3 labs in	PAKISTAN
2 labs in	PORTUGAL
5 labs in	SOUTH KOREA
4 labs in	SPAIN
2 labs in	SRI LANKA
1 lab in	SWITZERLAND
1 lab in	TAIWAN R.O.C.
2 labs in	THAILAND
2 labs in	TUNISIA
8 labs in	TURKEY
2 labs in	U.S.A.
2 labs in	UNITED KINGDOM
4 labs in	VIETNAM

iis19A14P	
2 labs in	BANGLADESH
1 lab in	BELGIUM
2 labs in	BRAZIL
1 lab in	BULGARIA
1 lab in	CAMBODIA
1 lab in	ETHIOPIA
6 labs in	FRANCE
7 labs in	GERMANY
7 labs in	HONG KONG
8 labs in	INDIA
2 labs in	INDONESIA
13 labs in	ITALY
1 lab in	LUXEMBOURG
1 lab in	MAURITIUS
2 labs in	MEXICO
2 labs in	MOROCCO
16 labs in	P.R. of CHINA
2 labs in	PAKISTAN
1 lab in	POLAND
3 labs in	PORTUGAL
3 labs in	SOUTH KOREA
4 labs in	SPAIN
1 lab in	SWITZERLAND
2 labs in	TAIWAN R.O.C.
1 lab in	THAILAND
1 lab in	TUNISIA
5 labs in	TURKEY
1 lab in	U.S.A.
2 labs in	UNITED KINGDOM
2 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

Literature:

1. iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2. Oeko-Tex Standard 100; January 2019
3. Blue Sign (BSSL) version 6.0 July 01, 2016
4. Impacts of Environmental Standards and requirements in EU Countries. Aug 99
5. Horwitz, R. Albert, Journal of AOAC International, 79-3, 589, (1996.)
6. P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
7. W.J. Conover, Practical Nonparametric Statistics, J. Wiley&Sons. NY, 302, (1971)
8. ISO 5725:86
9. ISO 5725. parts 1-6:94
10. ISO105 E4:94
11. ISO14184-1:94
12. ISO13528:05
13. M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
14. Analytical Methods Committee, Technical brief, No 4, January 2001
15. P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst 2002, 127, 1359-1364 (2002)
16. Official Journal of the European Communities, L133/29, May 2002.
17. Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)