Results of Proficiency Test
Acetic Acid
February 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

1 INTRODUCTION

Since 2004, the Institute for Interlaboratory Studies organizes a proficiency scheme for Acetic Acid. The proficiency scheme for Acetic Acid was organized every year till 2011 and every two years after 2011. During the annual proficiency test program of 2018/2019, it was decided to continue the proficiency test for the analysis of Acetic Acid in accordance with the latest applicable version of the product specification ASTM D3620.
In this interlaboratory study, 21 laboratories in 13 different countries did register for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2019 proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one bottle of 0.5L Acetic Acid, labelled #19002.
Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluations.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. 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 organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of 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 necessary bulk material of Acetic Acid was obtained from a chemical producer. The approximately 25 liters of Acetic Acid was spiked with 208 mg Iron(III)Chloride.$6H_2O$. After homogenization, this material was divided over 42 amber glass bottles of 0.5 L and labelled #19002.
The homogeneity of subsamples #19002 was checked by determination of Chloride in accordance with an in-house test method and Density in accordance with ASTM D4052 on 8 stratified randomly selected samples.

	Chloride in mg/kg	Density at 20°C in kg/l
sample #19002-1	3.6	1.04948
sample #19002-2	3.6	1.04946
sample #19002-3	3.6	1.04950
sample #19002-4	3.6	1.04949
sample #19002-5	3.6	1.04949
sample #19002-6	3.6	1.04949
sample #19002-7	3.6	1.04950
sample #19002-8	3.6	1.04948

Table 1: homogeneity test results of subsamples #19002

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

	Chloride in mg/kg	Density at 20°C in kg/l
r (observed)	0.0	0.00004
reference method	Horwitz	ISO12185:96
0.3*R (reference method)	0.40	0.00015

Table 2: evaluation of the repeatabilities of subsamples #19002

The calculated repeatabilities were both in agreement with 0.3 times the corresponding reproducibility of the reference methods. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories 1*0.5 L amber glass bottle, labelled #19002 was sent on January 23, 2019. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Acetic Acid packed in amber glass bottles was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were asked to determine: Acetaldehyde, Appearance, Anorganic Chloride as Cl, Color Pt/Co, Density at 20°C, Formic Acid, Freezing Point, Iron as Fe, Nonvolatile Matter, Purity via Freezing Point, Purity via Titration, Sulfate as SO_4 and Water.

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

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test 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.co.uk/sgs-iis/. 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/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under ‘Remarks’ in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). For the statistical evaluation, the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by

the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either ‘unknown’, ‘OK’, ‘suspect’ or ‘not OK’. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO5725 the original test results per determination were submitted to Dixon’s and/or 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, 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 these with a factor of 2.8.

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis, the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve 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, EN or ISO reproducibilities, 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) = (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

In this interlaboratory study, some problems were encountered with dispatch of the samples. Participants in Brazil received the sample late and reported therefore the test results after the final reporting date. All participants reported test results. Not all laboratories were able to perform all analyses requested.
In total 21 participants reported 124 numerical test results. Observed were 5 outlying results, which is 4.0% of the total of 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 “unknown” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER TEST

In this section, the test results are discussed per test. The test methods, which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the reported data. The abbreviations, used in these tables, are listed in appendix 3.

For comparison of the results of this interlaboratory study, the requirements from the specification ASTM D3620:04 (2017) “Standard Specification for Glacial Acetic Acid” were used. Regretfully, for many determinations this specification is referring to ASTM E302:95 “Standard Test Methods for Monobasic Organic Acids”, which was withdrawn already in 2001. As there was no replacement, this specification was used as reference method.

The target reproducibility used for the determination of the Purity by Freezing Point is calculated from the values in table 1 from ASTM E302 and the target reproducibility from ASTM E302.
For the determination of the Purity by Titration the used method for comparison is ASTM E301:94 which was also withdrawn in 2001 with no replacement. However, no other useful standardized method is published yet.
Unfortunately, a suitable reference test method, providing the precision data, is not available for all determinations. For the tests, that have no available precision data, the calculated reproducibility was compared against the reproducibility estimated from the Horwitz equation.
In the iis PT reports, ASTM methods are referred to with a number (e.g. D1209) and an added designation for the year that the method was adopted or revised (e.g. D1209:05). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D1209:05 (2011)). In the results tables of appendix 1 only the method number and year of adoption or revision will be used.

Acetaldehyde: Five participants reported a test result and therefore no z-scores were calculated. Please note that ASTM D2191 is meant for vinyl acetate.

Appearance: This determination was not problematic. All reporting participants agreed about the appearance; bright, clear and free of suspended matter or pass in accordance with ASTM E2680:16.

Chloride, Anorganic as Cl: 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 reproducibility using the Horwitz equation.

Color Pt/Co: 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 requirements of ASTM D1209:05(2011).

Density at 20°C: 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 requirements of ISO12185:96.

Formic Acid: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of ASTM D3546:05(2011).

Freezing Point: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of ASTM E302:95 (withdrawn).

Iron as Fe: 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 requirements of ASTM E394:15.

Nonvolatile Matter: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D1353:13.

Purity (from Freezing Point): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility limits based on the requirements of ASTM E302:94 (withdrawn).

Purity (Titration): This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of ASTM E301:94 (withdrawn).

Sulfate as SO4: Three participants reported a test result and all three test results were “less than”. Therefore, no z-scores were calculated.

Water: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of the withdrawn method ASTM E302:95 (withdrawn).

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant reference test methods and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average test results, the calculated reproducibilities (2.8 * standard deviation) and the target reproducibilities derived from literature reference test methods (in casu ASTM and EN standards) are presented in the next tables.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acetaldehyde	mg/kg	5	<20	n.a.	n.a.
Appearance		17	Pass	n.a.	n.a.
Chloride, Anorganic as Cl	mg/kg	7	3.6	0.8	1.3
Color Pt/Co		16	10.8	2.7	7
Density at 20°C	kg/L	18	1.0495	0.0004	0.0005
Formic Acid	mg/kg	9	98	231	360
Freezing Point	°C	15	16.31	0.16	0.25
Iron as Fe	mg/kg	13	1.9	0.4	0.9
Nonvolatile Matter	mg/100 mL	5	2.0	2.5	0.9
Purity (by Freezing Point)	%M/M	14	99.84	0.08	0.13
Purity (via Titration)	%M/M	5	99.75	0.35	0.54
Sulfate as SO_4	mg/kg	3	<1	n.a.	n.a.
Water	%M/M	17	0.131	0.027	0.050

Table 3: reproducibilities of tests on sample #19002

Without further statistical calculations it can be concluded that for a number tests there is a good compliance of the group of participating laboratories with the relevant reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF FEBRUARY 2019 WITH PREVIOUS PTS

	February 2019	February 2017	February 2015	February 2013	February 2011
Number of reporting participants	21	22	22	23	28
Number of results reported	124	152	159	177	236
Statistical outliers	5	5	6	10	10
Percentage outliers	4.0%	3.3%	3.8%	5.7%	4.2%

Table 4: comparison with previous proficiency tests.

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency test was compared against the requirements of the respective reference test methods. The conclusions are given in the following table.

Determination	February 2019	February 2017	February 2015	February 2013	February 2011
Acetaldehyde	n.e.	n.e.	n.e.	++	++
Chloride, Anorganic as Cl	+	++	++	++	++
Color	++	++	+	++	++
Density at 20°C	+	++	++	++	++
Formic Acid	+	++	++	++	++
Freezing Point	+	++	++	++	++
Iron as Fe	++	++	-	++	++
Nonvolatile matter	--	-	+/-	--	++
Purity (by Freezing point)	+	++	++	++	++
Purity (via Titration)	+	++	++	+	++
Sulfate as SO_4	n.e.	n.e.	n.e.	--	--
Water	+	++	++	++	++

Table 5: comparison determinations against the reference test method

The performance of the determinations against the requirements of the respective reference test methods is listed in the above table. The following performance categories were used:

++: group performed much better than the reference test method
+ : group performed better than the reference test method
+/-: group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.: not evaluated

APPENDIX 1

Determination of Acetaldehyde on sample #19002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173		-----		-----	
174		-----		-----	
311		-----		-----	
319		-----		-----	
323	D2191	<10		-----	
343		-----		-----	
347		-----		-----	
357		-----		-----	
395		-----		-----	
551		-----		-----	
558		-----		-----	
609		-----		-----	
663		-----		-----	
859	D2191	10		-----	
861		-----		-----	
912		-----		-----	
913	IS695	<20	C	-----	First reported <0.002 mg/kg
963	D2191	10		-----	
1091		-----		-----	
1294		0		-----	
6221		-----		-----	
	n	5			
	mean (n)	<20			

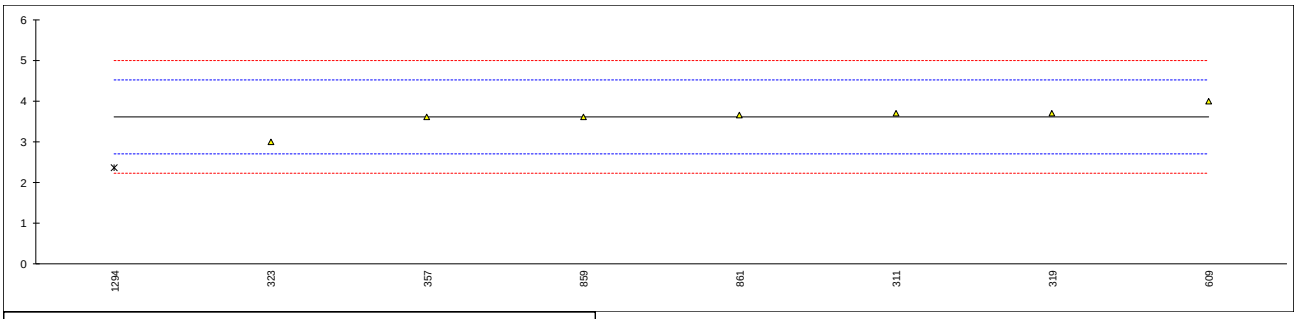
Determination of Appearance on sample #19002;

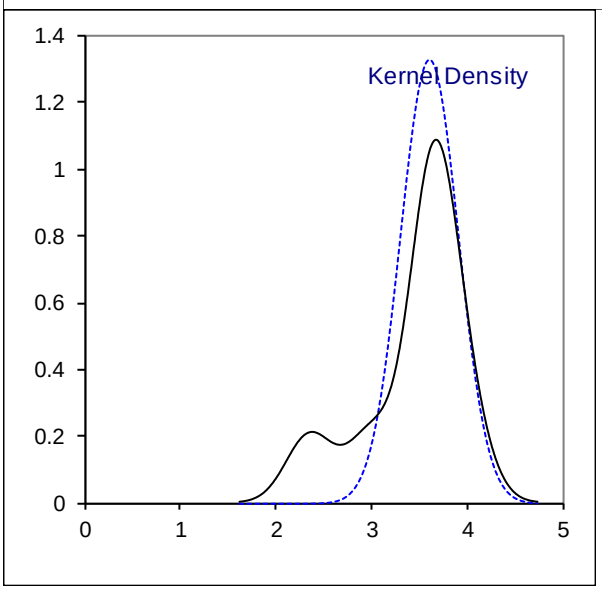
lab	method	value	mark	z(targ)	remarks
173	E2680	Pass		-----	
174	Visual	Clear & Free		-----	
311	E2680	pass		-----	
319	Visual	clear colorless liquid		-----	
323	E2680	clear & bright		-----	
343	E2680	Pass		-----	
347	E2680	pass		-----	
357	E2680	Pass		-----	
395	E2680	PASS		-----	
551	E2680	Pass		-----	
558		-----		-----	
609	E2680	PASS		-----	
663		-----		-----	
859	E2680	C&B		-----	
861	Visual	Bright&Clear		-----	
912	E2680	Pass		-----	
913	E2680	Clear & Bright		-----	
963	Visual	pass		-----	
1091		-----		-----	
1294	Visual	Clear		-----	
6221		-----		-----	
	n	17			
	mean (n)	Pass			

Determination of Chloride, Anorganic as Cl on sample #19002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173	INH-221	>0.5		-----	
174		-----		-----	
311	INH-158	3.7		0.20	
319	ISO753-8	3.7		0.20	
323	E2469	3		-1.28	
343		-----		-----	
347		-----		-----	
357	INH-709TI	3.6		-0.02	
395		-----		-----	
551		-----		-----	
558		-----		-----	
609	INH-70020	4		0.83	
663		-----		-----	
859	IMPCA002	3.6		-0.02	
861	IMPCA002	3.65		0.09	
912		-----		-----	
913		-----		-----	
963	INH-1351	<2		<-3.38	Possibly a false negative test result?
1091		-----		-----	
1294		2.36	G(0.05)	-2.62	
6221		-----		-----	

normality	not OK
n	7
outliers	1
mean (n)	3.607
st.dev. (n)	0.3006
R(calc.)	0.842
st.dev.(Horwitz)	0.4758
R(Horwitz)	1.332

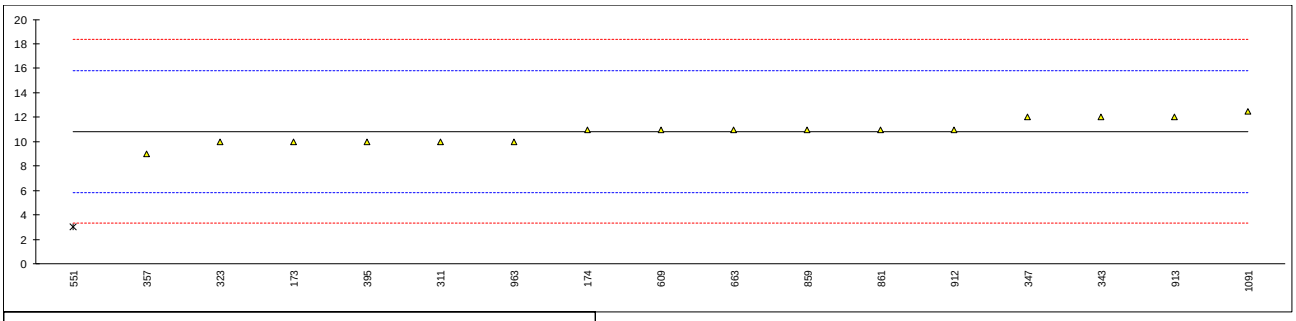


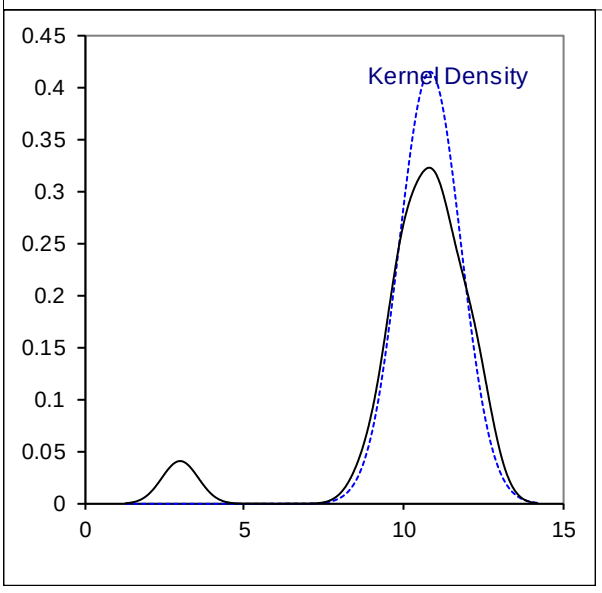


Determination of Color Pt/Co on sample #19002

lab	method	value	mark	z(targ)	remarks
173	D1209	10		-0.34	
174	D5386	11		0.06	
311	E302	10		-0.34	
319		-----		-----	
323	D1209	10		-0.34	
343	D5386	12		0.46	
347	D5386	12		0.46	
357	D5386	9		-0.74	
395	D1209	10		-0.34	
551	D1209	3	G(0.01)	-3.14	
558	D1209	<5		-----	
609	D1209	11		0.06	
663	D1209	11		0.06	
859	D1209	11		0.06	
861	D1209	11		0.06	
912	D5386	11		0.06	
913	D5386	12		0.46	
963	D1209	10		-0.34	
1091	D5386	12.5		0.66	
1294		<10		-----	
6221		-----		-----	

normality	OK
n	16
outliers	1
mean (n)	10.84
st.dev. (n)	0.961
R(calc.)	2.69
st.dev.(D1209:05)	2.5
R(D1209:05)	7

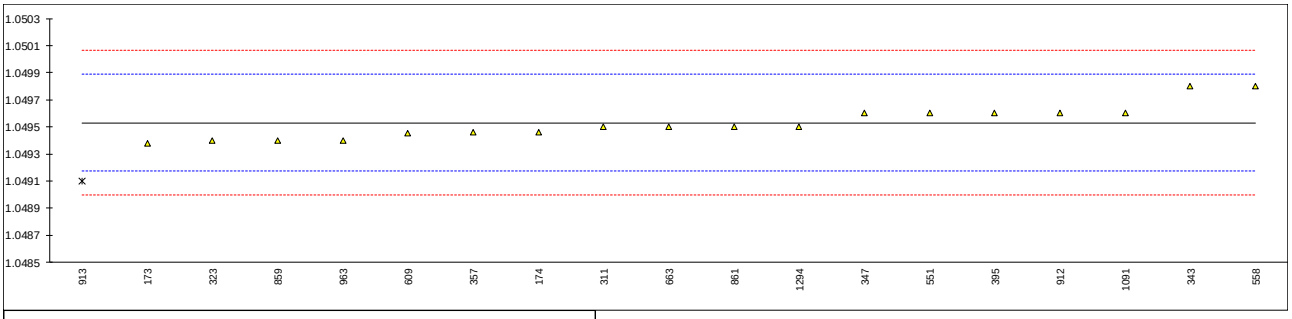


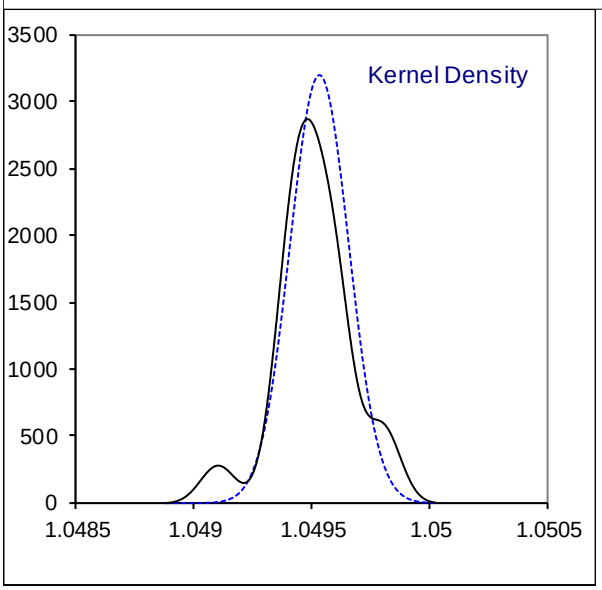


Determination of Density at 20°C on sample #19002; results in kg/L

lab	method	value	mark	z(targ)	remarks
173	D4052	1.04938		-0.84	
174	D4052	1.04946		-0.39	
311	D4052	1.0495		-0.17	
319		-----		-----	
323	ISO12185	1.0494		-0.73	
343	D4052	1.0498		1.51	
347	D4052	1.0496		0.39	
357	D4052	1.04946		-0.39	
395	D4052	1.0496		0.39	
551	D4052	1.0496		0.39	
558	D4052	1.0498		1.51	
609	D4052	1.04945		-0.45	
663	D4052	1.0495		-0.17	
859	D4052	1.0494		-0.73	
861	D4052	1.0495		-0.17	
912	D4052	1.0496		0.39	
913	D4052	1.0491	G(0.05)	-2.41	
963	ISO12185	1.0494		-0.73	
1091	D4052	1.0496		0.39	
1294	D4052	1.0495		-0.17	
6221		-----		-----	

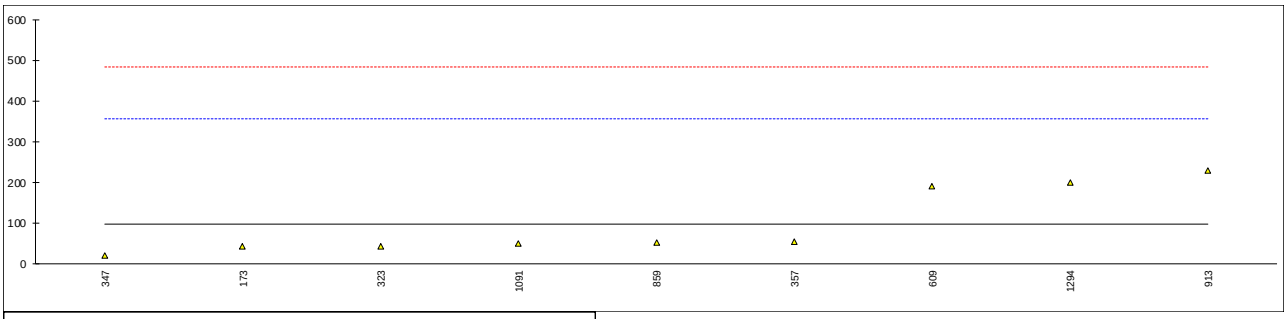
normality	suspect
n	18
outliers	1
mean (n)	1.04953
st.dev. (n)	0.000124
R(calc.)	0.00035
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

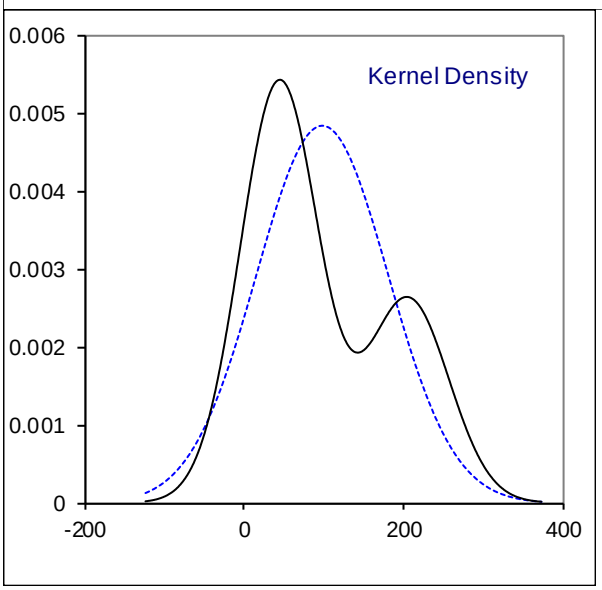




Determination of Formic Acid on sample #19002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173	D3546	43.8		-0.42	
174		-----		-----	
311		-----		-----	
319		-----		-----	
323	D3546	44		-0.42	
343		-----		-----	
347	D3546	20		-0.61	
357	D3546	55		-0.34	
395		-----		-----	
551		-----		-----	
558		-----		-----	
609	D3546	190		0.71	
663		-----		-----	
859	D3546	52		-0.36	
861		-----		-----	
912		-----		-----	
913	IS695	230		1.02	
963		-----		-----	
1091	D3546	51		-0.37	
1294	D3546	200		0.79	
6221		-----		-----	
	normality	OK			
	n	9			
	outliers	0			
	mean (n)	98.42			
	st.dev. (n)	82.464			
	R(calc.)	230.9			
	st.dev.(D3546:05)	128.57			
	R(D3546:05)	360			Compare R(iis17C02) = 117.6 at 59.1 mg/kg

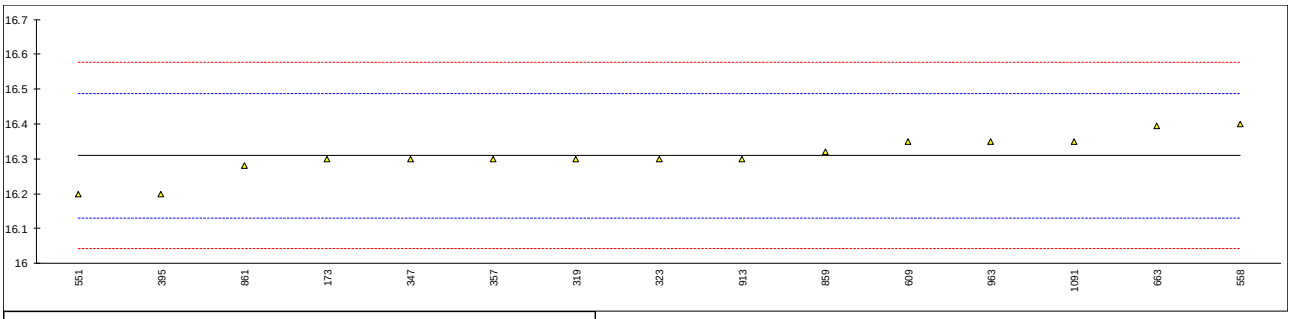


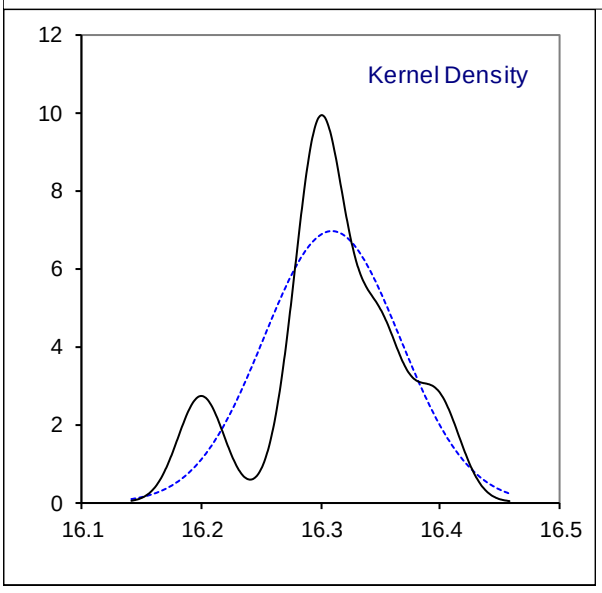


Determination of Freezing Point on sample #19002; results in °C

lab	method	value	mark	z(targ)	remarks
173	INH-124	16.3		-0.11	
174		-----		-----	
311		-----		-----	
319	E302	16.30		-0.11	
323	E302	16.30		-0.11	
343		-----		-----	
347	E302	16.30		-0.11	
357	E302	16.30		-0.11	
395	INH-124	16.2		-1.23	
551	E302	16.2		-1.23	
558	E302	16.4		1.01	
609	INH-70013	16.35		0.45	
663	D6875	16.394		0.95	
859	E302	16.32		0.12	
861	E302	16.28		-0.33	
912		-----		-----	
913	E302	16.3		-0.11	
963	E302	16.35		0.45	
1091	E302	16.35		0.45	
1294		-----		-----	
6221		-----		-----	

normality	OK
n	15
outliers	0
mean (n)	16.310
st.dev. (n)	0.0572
R(calc.)	0.160
st.dev.(E302:95)	0.0893
R(E302:95)	0.25

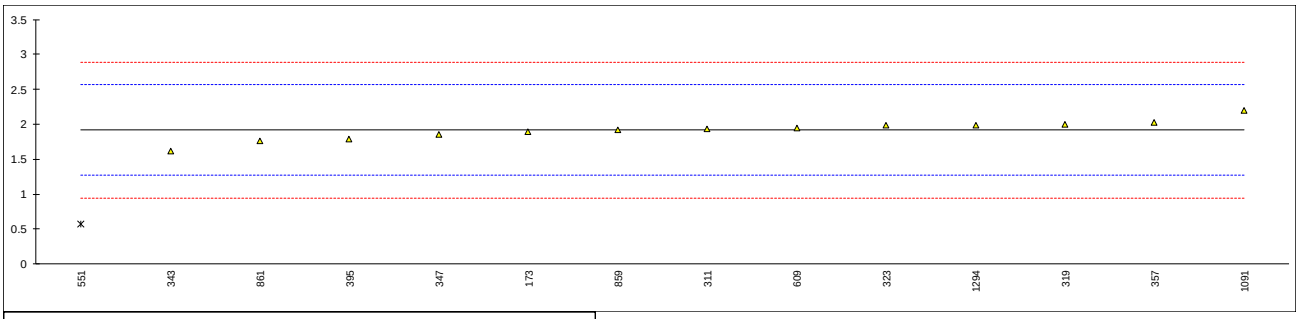


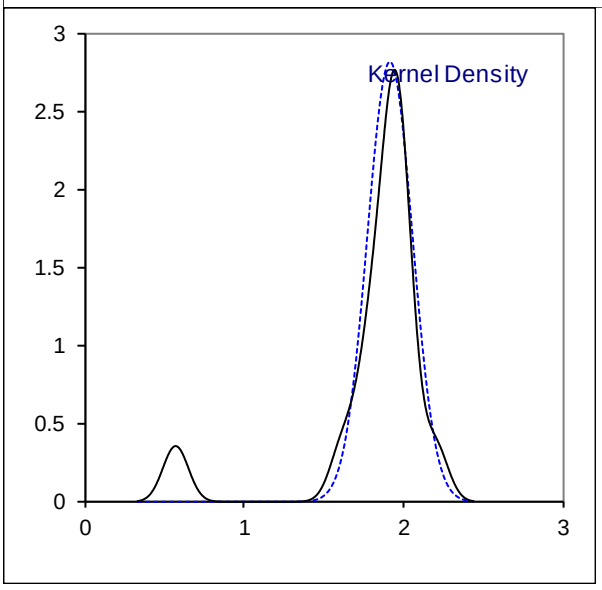


Determination of Iron as Fe on sample #19002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173	E394	1.90		-0.05	
174		-----		-----	
311	E394	1.93		0.05	
319	E394	2.0		0.26	
323	E394	1.98		0.20	
343	E394	1.62		-0.91	
347	E394	1.85		-0.20	
357	E394	2.02		0.32	
395	E394	1.791		-0.38	
551	E394	0.57	G(0.01)	-4.15	
558		-----		-----	
609	E394	1.946		0.10	
663		-----		-----	
859	E394	1.92		0.01	
861	E394	1.76		-0.48	
912		-----		-----	
913		-----		-----	
963		-----		-----	
1091	E394	2.20		0.88	
1294		1.98		0.20	
6221		-----		-----	

normality	suspect
n	13
outliers	1
mean (n)	1.915
st.dev. (n)	0.1414
R(calc.)	0.396
st.dev.(E394:15)	0.3241
R(E394:15)	0.908

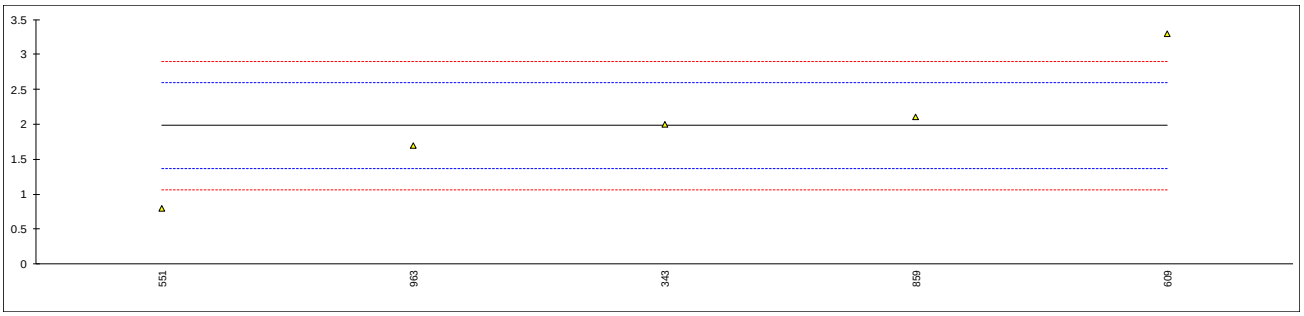




Determination of Nonvolatile Matter on sample #19002; results in mg/100 mL

lab	method	value	mark	z(targ)	remarks
173		-----		-----	
174		-----		-----	
311		-----		-----	
319		-----		-----	
323	D1353	<1		<-3.21	Possibly a false negative test result?
343	D1353	2.0		0.07	
347		-----		-----	
357	D1353	<1		<-3.21	Possibly a false negative test result?
395		-----		-----	
551	D1353	0.8		-3.87	
558		-----		-----	
609	D1353	3.3		4.33	
663		-----		-----	
859	D1353	2.1		0.39	
861		-----		-----	
912		-----		-----	
913		-----		-----	
963	D1353	1.7		-0.92	
1091		-----		-----	
1294		-----		-----	
6221		-----		-----	

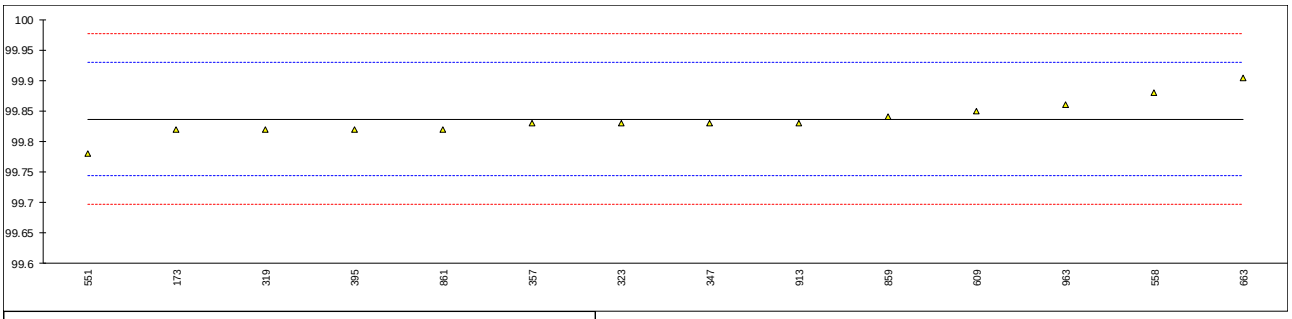
normality	unknown
n	5
outliers	0
mean (n)	1.98
st.dev. (n)	0.898
R(calc.)	2.52
st.dev.(D1353:13)	0.305
R(D1353:13)	0.85

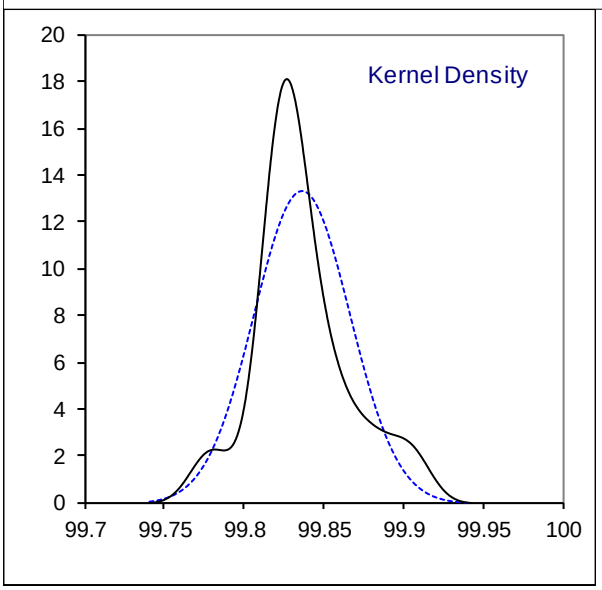


Determination of Purity via Freezing Point on sample #19002; results in %M/M

lab	method	value	mark	z(targ)	remarks
173	INH-124	99.82		-0.36	
174		-----		-----	
311		-----		-----	
319	E302	99.82		-0.36	
323	E302	99.83		-0.14	
343		-----		-----	
347	E302	99.83		-0.14	
357	E302	99.83		-0.14	
395	INH-124	99.82		-0.36	
551	E302	99.78		-1.22	
558	E302	99.88		0.93	
609	INH-70014	99.85		0.29	
663	BS579part2	99.904		1.45	
859	E302	99.84		0.07	
861	E302	99.82		-0.36	
912		-----		-----	
913	E302	99.83		-0.14	
963	E302	99.86		0.50	
1091		-----		-----	
1294		-----		-----	
6221		-----		-----	

normality	suspect
n	14
outliers	0
mean (n)	99.837
st.dev. (n)	0.0299
R(calc.)	0.084
st.dev.(E302:95)	0.0464
R(E302:95)	0.13

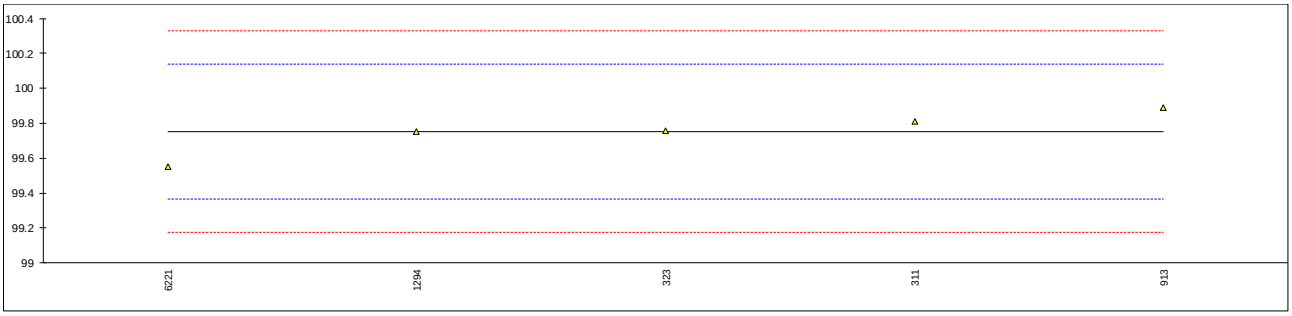




Determination of Purity via titration on sample #19002; results in %M/M

lab	method	value	mark	z(targ)	remarks
173		-----		-----	
174		-----		-----	
311	E301	99.81		0.30	
319		-----		-----	
323	E301	99.76		0.04	
343		-----		-----	
347		-----		-----	
357		-----		-----	
395		-----		-----	
551		-----		-----	
558		-----		-----	
609		-----		-----	
663		-----		-----	
859		-----		-----	
861		-----		-----	
912		-----		-----	
913	E301	99.89		0.72	
963		-----		-----	
1091		-----		-----	
1294		99.75		-0.01	
6221	EN13194	99.55		-1.05	

normality	unknown
n	5
outliers	0
mean (n)	99.752
st.dev. (n)	0.1258
R(calc.)	0.352
st.dev.(E301:94)	0.1929
R(E301:94)	0.54



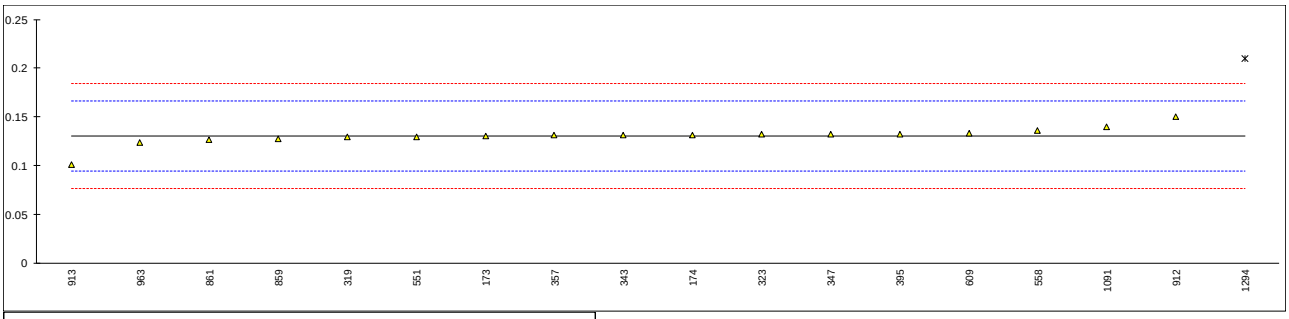
Determination of Sulfate as SO_4 on sample #19002, results in mg/kg

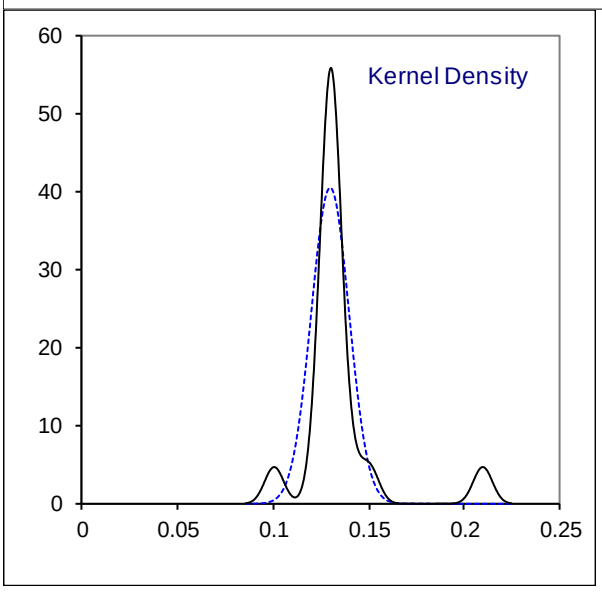
lab	method	value	mark	z(targ)	remarks
173		-----		-----	
174		-----		-----	
311		-----		-----	
319		-----		-----	
323	EN15492	<1		-----	
343	INH-1283	<1		-----	
347		-----		-----	
357	EN15492	< 0,1		-----	
395		-----		-----	
551		-----		-----	
558		-----		-----	
609		-----		-----	
663		-----		-----	
859		-----		-----	
861		-----		-----	
912		-----		-----	
913		-----		-----	
963		-----		-----	
1091		-----		-----	
1294		-----		-----	
6221		-----		-----	
	n	3			
	mean (n)	<1			

Determination of Water on sample #19002, results in %M/M

lab	method	value	mark	z(targ)	remarks
173	E203	0.13035		-0.01	
174	E203	0.1313		0.04	
311		-----		-----	
319	E1064	0.130		-0.03	
323	E302	0.132		0.08	
343	E1064	0.1312		0.03	
347	E302	0.1320		0.08	
357	E203	0.131		0.02	
395	E1064	0.1324		0.10	
551	E203	0.130		-0.03	
558	E1064	0.1363		0.32	
609	D1364	0.133		0.13	
663		-----		-----	
859	E1064	0.128		-0.15	
861	D1364	0.1266		-0.22	
912	E203	0.1507		1.13	
913	E203	0.101		-1.66	
963	E203	0.124		-0.37	
1091	E1064	0.1404		0.55	
1294		0.21	G(0.01)	4.45	
6221		-----		-----	

normality	not OK
n	17
outliers	1
mean (n)	0.13060
st.dev. (n)	0.009649
R(calc.)	0.02702
st.dev.(E302:95)	0.017857
R(E302:95)	0.050





APPENDIX 2

Number of participants per country

2 labs in BELGIUM
2 labs in BRAZIL
2 labs in CHINA, People's Republic
1 lab in FINLAND
2 labs in INDIA
1 lab in ITALY
1 lab in MALAYSIA
2 labs in NETHERLANDS
2 labs in SAUDI ARABIA
2 labs in SPAIN
1 lab in THAILAND
1 lab in TURKEY
2 labs in UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations:

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= possibly an error in calculations
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
SDS	= Safety Data Sheet

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 ASTM E178:16
3 ASTM E1301:95(2003)
4 ISO 5725:86 (1994)
5 ISO 5725, parts 1-6, 1994
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8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
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10 DIN 38402 T41/42
11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
12 J.N. Miller, Analyst, 118, 455, (1993)
13 Analytical Methods Committee Technical Brief, No 4, January 2001
14 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst 2002, 127, 1359-1364, (2002)
15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)
16 Horwitz, W and Albert, R, J. AOAC Int, 79, 3, 589, (1996)