Results of Proficiency Test
Acetic Acid
February 2021

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2004 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Acetic Acid in accordance with the latest version of ASTM D3620. The proficiency scheme for Acetic Acid was organized since 2011 every two years. During the annual proficiency test program of 2020/2021 it was decided to continue the proficiency test for the analysis of Acetic Acid

In this interlaboratory study 18 laboratories in 12 different countries registered for participation. See appendix 2 for the number of participants per country. In this report the results of the Acetic acid 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/IEC17025 accredited laboratory.
It was decided to send one bottle of 0.5L Acetic Acid, labelled #21002.
The 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/IEC17043:2010 (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

A batch of approximately 30 liters of Acetic Acid was obtained from a local supplier. The batch of Acetic Acid was spiked with Iron Chloride and Sodium Sulfate. After homogenization 58 amber glass bottles of 0.5L were filled and labeled #21002.
The homogeneity of the subsamples was checked by determination of Chloride in accordance with an in-house test method and Density at 20°C in accordance with ASTM D4052 on 8 stratified randomly selected subsamples.

	Chloride in mg/kg	Density at 20°C in kg/l
sample #21002-1	2.4	1.04938
sample #21002-2	2.4	1.04938
sample #21002-3	2.4	1.04938
sample #21002-4	2.4	1.04938
sample #21002-5	2.5	1.04938
sample #21002-6	2.4	1.04938
sample #21002-7	2.4	1.04937
sample #21002-8	2.5	1.04938

Table 1: homogeneity test results of subsamples #21002

From the above test results the repeatabilities were calculated and compared with 0.3 times the reproducibility of the corresponding reference methods 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)	0.13	0.00001
reference method	Horwitz	ISO12185:96
0.3 x R (reference method)	0.29	0.00015

Table 2: evaluation of the repeatabilities of subsamples #21002

The calculated repeatabilities were 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 one sample of 0.5L Acetic Acid labelled #21002 was sent on January 13, 2021. 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 ANALYZES

The participants were requested to determine: Acetaldehyde, Appearance, Inorganic 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 sample as if it was a routine sample 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 (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. 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 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.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can e used. For larger data sets (above 20 test results) Rosner’s outlier tests can be used. 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

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. 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, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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. Therefore, 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

Some problems were encountered with the dispatch of the samples due to COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another week. One participant reported test results after the extended final reporting date and three other participants did not report any test results. Not all participants were able to report all tests requested.
In total 15 participants reported 97 numerical test results. Observed were 4 outlying test results, which is 4.1%. In proficiency test outlier percentages of 3% - 7.5% are quite normal.

Not all 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 original data. The abbreviations, used in these tables, are explained in appendix 3.

Unfortunately, a suitable reference test method, providing the precision data, is not available for all determinations. For these tests the calculated reproducibility was compared against the estimated reproducibility calculated with the Horwitz equation.

In the iis PT reports ASTM test methods are referred to with a number and if appropriate an indication of sub test method (e.g. D1209) and an added designation for the year that the test 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 (2019). In the test results tables of appendix 1 only the method number (sub) and year of adoption or revision (e.g. D1209:05) 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. Please note: This test method is withdrawn in 2021 with no replacement.

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.

Inorganic Chloride as Cl: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility calculated with the Horwitz equation.

Color Pt/Co: 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 ASTM D1209:05(2019).

Density at 20°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ISO12185:96.

Formic Acid: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D3546:05(2019).

Freezing Point: 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 ASTM E302:95. Please note: This test method is withdrawn in 2001 with no replacement.

Iron as Fe: 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 ASTM E394:15.

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

Purity via Freezing Point: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM E302:95.
Please note: This test method is withdrawn in 2001 with no replacement.

Purity via Titration: Two participants reported a test result and therefore no z-scores were calculated.

Sulfate as SO4: No participants reported a test result. In the last five iis proficiency tests we noticed a decline in number of reported test results. Therefore, we will withdraw this parameter from the test scope.

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM E302:95.
Please note: This test method is withdrawn in 2001 with no replacement.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method or as declared by the estimated target reproducibility calculated with the Horwitz equation and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods (in casu ASTM test methods) or estimated using the Horwitz equation are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Acetaldehyde	mg/kg	4	<15	n.e.	n.e.
Appearance		14	Pass	n.a.	n.a.
Inorganic Chloride as Cl	mg/kg	5	2.7	0.7	1.0
Color Pt/Co		13	9.4	3.9	7
Density at 20°C	kg/L	14	1.0494	0.0002	0.0005
Formic Acid	mg/kg	4	26	28	360
Freezing Point	°C	11	16.36	0.12	0.25
Iron as Fe	mg/kg	12	1.2	0.3	0.6
Nonvolatile matter	mg/100 mL	4	1.8	2.3	0.8
Purity via Freezing Point	%M/M	12	99.85	0.07	0.13

Parameter	unit	n	average	2.8 * sd	R(lit)
Purity via Titration	%M/M	2	99.90	n.a.	n.a.
Sulfate as SO_4	mg/kg	0	0	n.a.	n.a.
Water	mg/kg	14	1048	152	500

Table 3: reproducibilities of tests on sample #21002

Without further statistical calculations, it can be concluded that for many tests there is a good compliance of the group of participants with the reference test methods. The problematic test has been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF FEBRUARY 2021 WITH PREVIOUS PTS

	February 2021	February 2019	February 2017	February 2015	February 2013
Number of reporting laboratories	15	21	22	22	23
Number of test results	97	124	152	159	177
Number of statistical outliers	4	5	5	6	10
Percentage of statistical outliers	4.1%	4.0%	3.3%	3.8%	5.7%

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 tests was compared to the requirements of the reference test methods. The conclusions are given in the following table.

Determination	February 2021	February 2019	February 2017	February 2015	February 2013
Acetaldehyde	n.e.	n.e.	n.e.	n.e.	++
Inorganic 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	n.e.	+	++	++	+
Sulfate as SO_4	n.e.	n.e.	n.e.	n.e.	--
Water	++	+	++	++	++

Table 5: comparison determinations against the reference test method

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 #21002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173	INH-245	<1		-----	
174		-----		-----	
319		-----		-----	
323	D2191	<10		-----	
343		-----		-----	
347		-----		-----	
357		-----		-----	
395		-----		-----	
551	D2191	<10		-----	
558		-----		-----	
609		-----		-----	
663		-----		-----	
859	D2191	30		-----	Possibly a false positive test result?
861		-----		-----	
912		-----		-----	
913		-----		-----	
963	D2191	10.0		-----	
6262		-----		-----	
	n	4			
	mean (n)	<15			

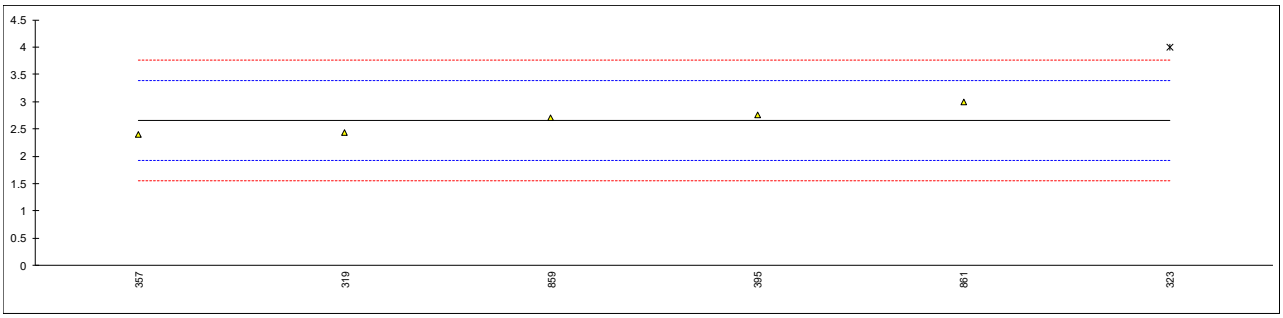
Determination of Appearance on sample #21002;

lab	method	value	mark	z(targ)	remarks
173	E2680	Pass		-----	
174	Visual	clear & free		-----	
319	Visual	clear colorless liquid		-----	
323	In house	clear and bright liquid		-----	
343	E2680	pass		-----	
347	E2680	Pass		-----	
357	E2680	Pass		-----	
395	E2680	PASS		-----	
551	E2680	Pass		-----	
558		-----		-----	
609	E2680	PASS		-----	
663		-----		-----	
859	E2680	Pass		-----	
861	Visual	bright clear		-----	
912		-----		-----	
913		-----		-----	
963	E2680	Clear and Bright		-----	
6262	Visual	Bright and clear		-----	
	n	14			
	mean (n)	Pass			

Determination of Inorganic Chloride as Cl on sample #21002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173	INH-221	<0.5		<-5.88	Possibly a false negative test result?
174		-----		-----	
319	ISO753/8	2.44		-0.59	
323	INH-008	4	G(0.05)	3.66	
343		-----		-----	
347		-----		-----	
357	INH-709	2.4		-0.70	
395	IMPCA002	2.75		0.25	
551		-----		-----	
558		-----		-----	
609	INH-70020	<3		-----	
663		-----		-----	
859	IMPCA002	2.7		0.11	
861	IMPCA002	3.0		0.93	
912		-----		-----	
913		-----		-----	
963	JIS K1351	<2		-----	
6262	E2469	<1		<-4.52	Possibly a false negative test result?

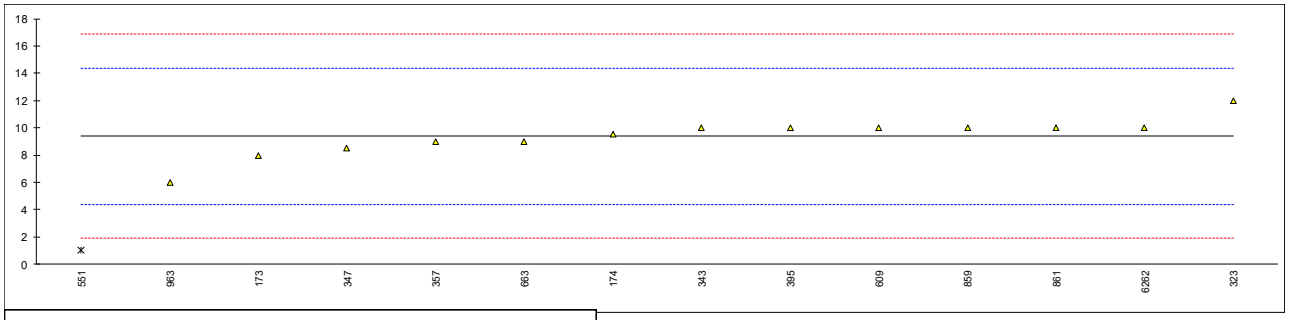
normality	unknown
n	5
outliers	1
mean (n)	2.658
st.dev. (n)	0.2456
R(calc.)	0.688
st.dev.(Horwitz)	0.3671
R(Horwitz)	1.028

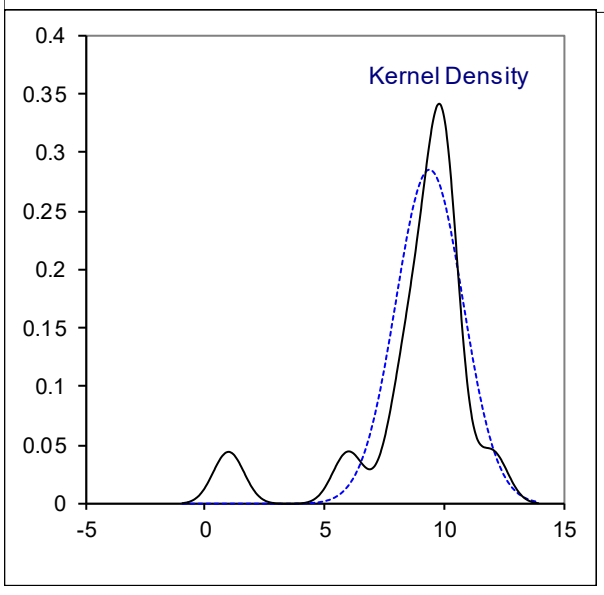


Determination of Color Pt/Co on sample #21002

lab	method	value	mark	z(targ)	remarks
173	D1209	8		-0.55	
174	D5386	9.52		0.05	
319		-----		-----	
323	D5386	12		1.05	
343	D5386	10		0.25	
347	D5386	8.5		-0.35	
357	D5386	9		-0.15	
395	D1209	10		0.25	
551	D1209	1	G(0.01)	-3.35	
558		-----		-----	
609	D1209	10		0.25	
663	D1209	9		-0.15	
859	D1209	10		0.25	
861	D5386	10		0.25	
912		-----		-----	
913		-----		-----	
963	D1209	6		-1.35	
6262	D1209	10		0.25	

normality	not OK
n	13
outliers	1
mean (n)	9.39
st.dev. (n)	1.402
R(calc.)	3.93
st.dev.(D1209:05)	2.5
R(D1209:05)	7

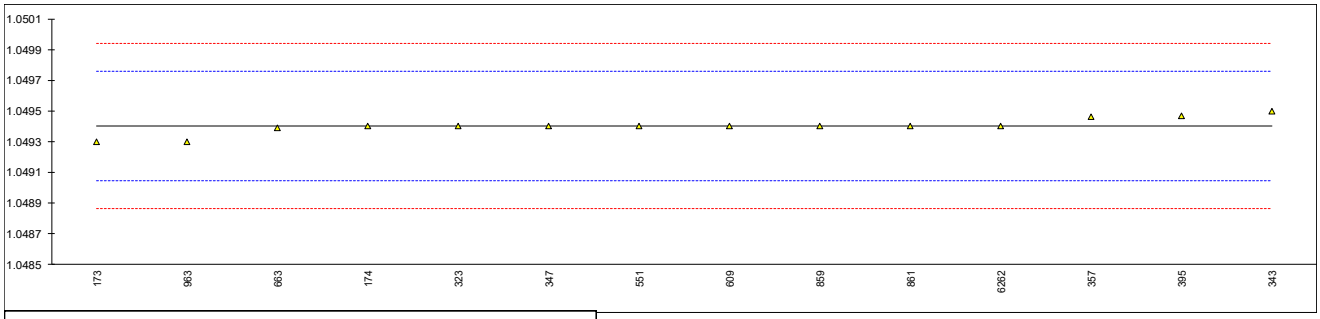


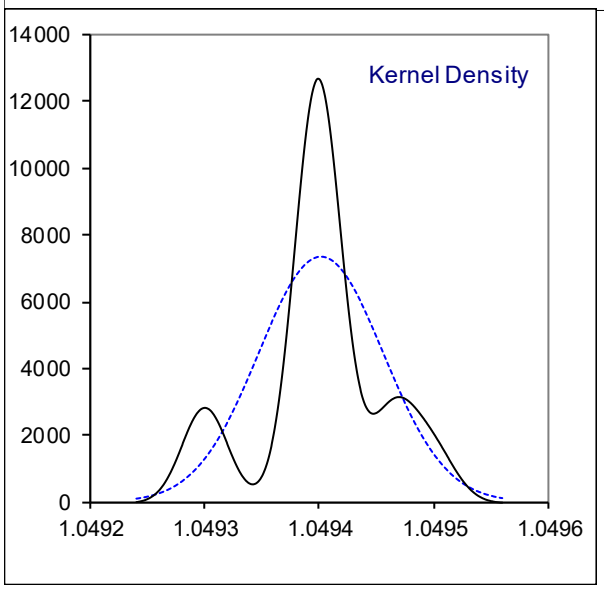


Determination of Density at 20°C on sample #21002; results in kg/L

lab	method	value	mark	z(targ)	remarks
173	D4052	1.0493		-0.57	
174	D4052	1.0494		-0.01	
319		-----		-----	
323	D4052	1.0494		-0.01	
343	D4052	1.0495		0.55	
347	D4052	1.0494		-0.01	
357	D4052	1.04946		0.33	
395	D4052	1.04947		0.38	
551	D4052	1.0494		-0.01	
558		-----		-----	
609	D4052	1.04940		-0.01	
663	D4052	1.04939		-0.06	
859	D4052	1.0494		-0.01	
861	D4052	1.0494		-0.01	
912		-----		-----	
913		-----		-----	
963	ISO12185	1.0493		-0.57	
6262	D4052	1.0494		-0.01	

normality	OK
n	14
outliers	0
mean (n)	1.04940
st.dev. (n)	0.000054
R(calc.)	0.00015
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

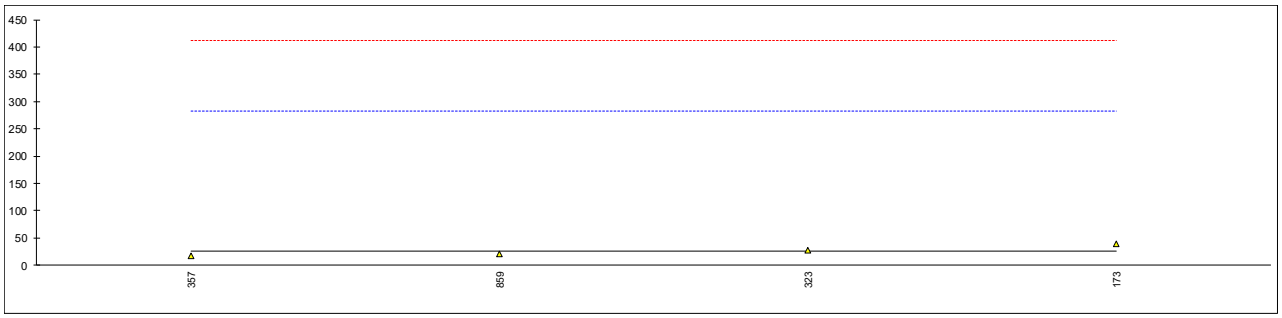




Determination of Formic Acid on sample #21002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173	D3546	40		0.11	
174		-----		-----	
319		-----		-----	
323	D3546	27		0.01	
343		-----		-----	
347		-----		-----	
357	D3546	18		-0.06	
395		-----		-----	
551		-----		-----	
558		-----		-----	
609		-----		-----	
663		-----		-----	
859	D3546	20		-0.05	
861		-----		-----	
912		-----		-----	
913		-----		-----	
963		-----		-----	
6262	D3546	<100		-----	

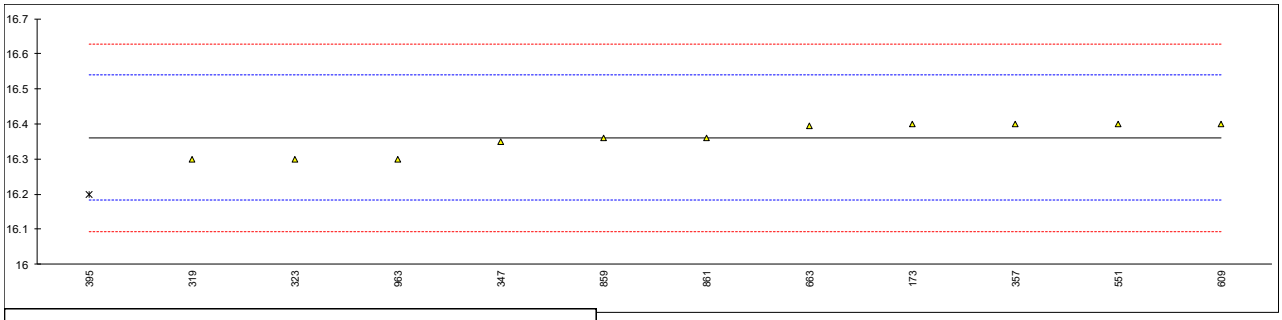
normality	unknown
n	4
outliers	0
mean (n)	26.25
st.dev. (n)	9.946
R(calc.)	27.8
st.dev.(D3546:05)	128.57
R(D3546:05)	360

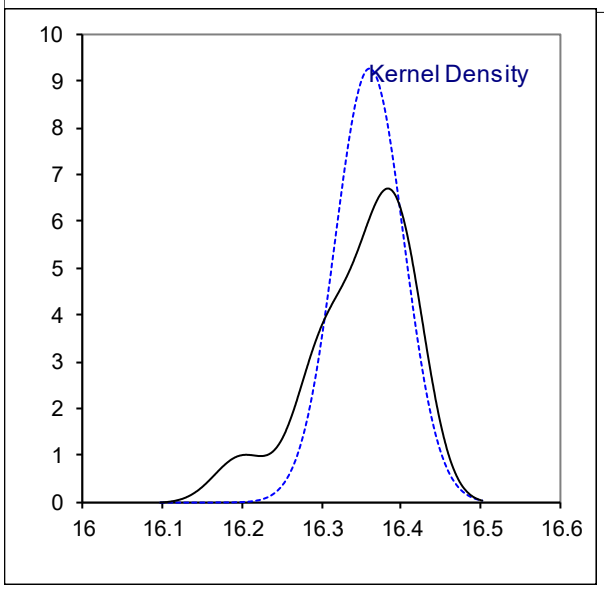


Determination of Freezing Point on sample #21002; results in °C

lab	method	value	mark	z(targ)	remarks
173	INH-124	16.40		0.44	
174		-----		-----	
319	E302	16.30		-0.68	
323	E302	16.30		-0.68	
343		-----		-----	
347	E302	16.35		-0.12	
357	E302	16.40		0.44	
395	INH-124	16.2	C,D(0.05)	-1.80	First reported 16
551	E302	16.4		0.44	
558		-----		-----	
609	INH-70013	16.40		0.44	
663	D6875	16.394		0.38	
859	E302	16.36		0.00	
861	E302	16.36		0.00	
912		-----		-----	
913		-----		-----	
963	E302	16.3		-0.68	
6262		-----		-----	

normality	OK
n	11
outliers	1
mean (n)	16.360
st.dev. (n)	0.0430
R(calc.)	0.120
st.dev.(E302:95)	0.0893
R(E302:95)	0.25

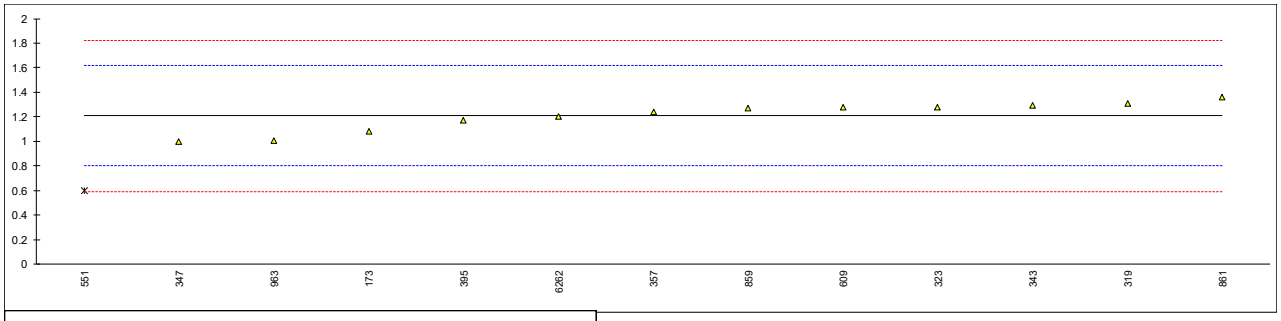


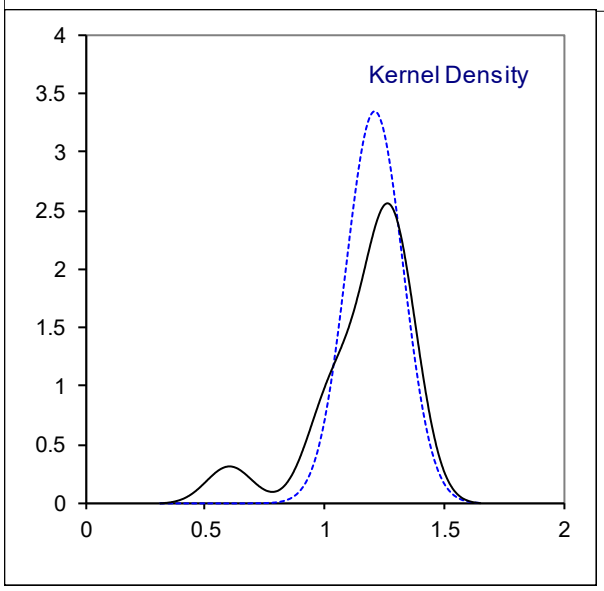


Determination of Iron as Fe on sample #21002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173	E394	1.08		-0.62	
174		-----		-----	
319	E394	1.31		0.50	
323	E394	1.28		0.36	
343	E394	1.29	C	0.40	First reported 1.89
347	E394	1.0		-1.01	
357	E394	1.24		0.16	
395	E394	1.169		-0.19	
551	E394	0.6	G(0.01)	-2.97	
558		-----		-----	
609	E394	1.279		0.35	
663		-----		-----	
859	E394	1.27		0.31	
861	E394	1.36		0.75	
912		-----		-----	
913		-----		-----	
963	E394	1.01		-0.97	
6262	E394	1.2		-0.04	

normality	OK
n	12
outliers	1
mean (n)	1.207
st.dev. (n)	0.1190
R(calc.)	0.333
st.dev.(E394:15)	0.2043
R(E394:15)	0.572

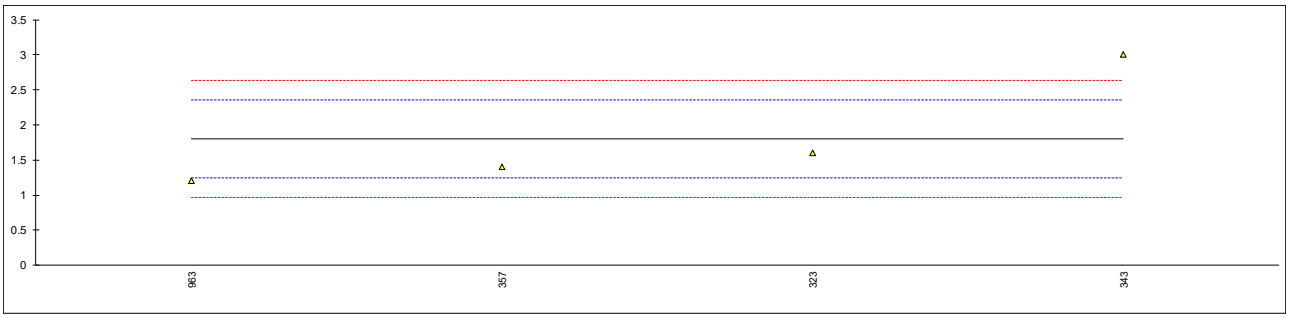




Determination of Nonvolatile matter on sample #21002; results in mg/100 mL

lab	method	value	mark	z(targ)	remarks
173		-----		-----	
174		-----		-----	
319		-----		-----	
323	D1353	1.6		-0.72	
343	D1353	3		4.33	
347		-----		-----	
357	D1353	1.4		-1.44	
395		-----		-----	
551		-----		-----	
558		-----		-----	
609		-----		-----	
663		-----		-----	
859		-----		-----	
861		-----		-----	
912		-----		-----	
913		-----		-----	
963	D1353	1.2		-2.16	
6262	D1353	<1		-----	

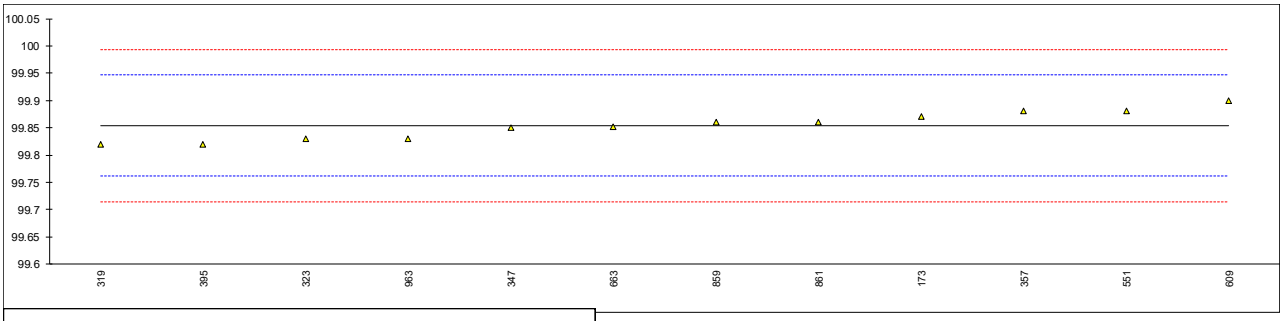
normality	unknown
n	4
outliers	0
mean (n)	1.80
st.dev. (n)	0.816
R(calc.)	2.29
st.dev.(D1353:13)	0.277
R(D1353:13)	0.78

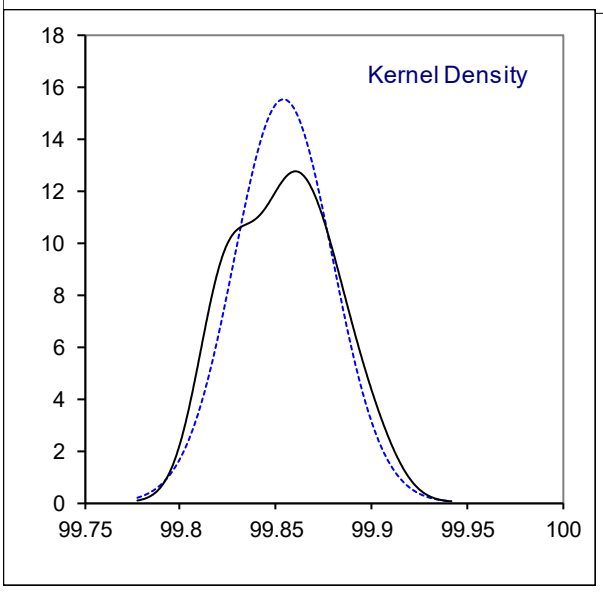


Determination of Purity via Freezing Point on sample #21002; results in %M/M

lab	method	value	mark	z(targ)	remarks
173	INH-124	99.87		0.34	
174		-----		-----	
319	E302	99.82		-0.74	
323	E302	99.83		-0.52	
343		-----		-----	
347	E302	99.85		-0.09	
357	E302	99.88		0.55	
395	INH-124	99.82	C	-0.74	First reported 99.67
551	E302	99.88		0.55	
558		-----		-----	
609	INH-70014	99.90		0.98	
663	BS576	99.852		-0.05	
859	E302	99.86		0.12	
861	E302	99.86		0.12	
912		-----		-----	
913		-----		-----	
963	E302	99.83		-0.52	
6262		-----		-----	

normality	OK
n	12
outliers	0
mean (n)	99.854
st.dev. (n)	0.0257
R(calc.)	0.072
st.dev.(E302:95)	0.0464
R(E302:95)	0.13





Determination of Purity via Titration on sample #21002; results in %M/M

lab	method	value	mark	z(targ)	remarks
173		-----		-----	
174		-----		-----	
319		-----		-----	
323	E301	99.82		-----	
343		-----		-----	
347		-----		-----	
357		-----		-----	
395		-----		-----	
551		-----		-----	
558		-----		-----	
609		-----		-----	
663		-----		-----	
859		-----		-----	
861		-----		-----	
912		-----		-----	
913		-----		-----	
963		-----		-----	
6262	E301	99.97		-----	
	n	2			
	mean (n)	99.895			

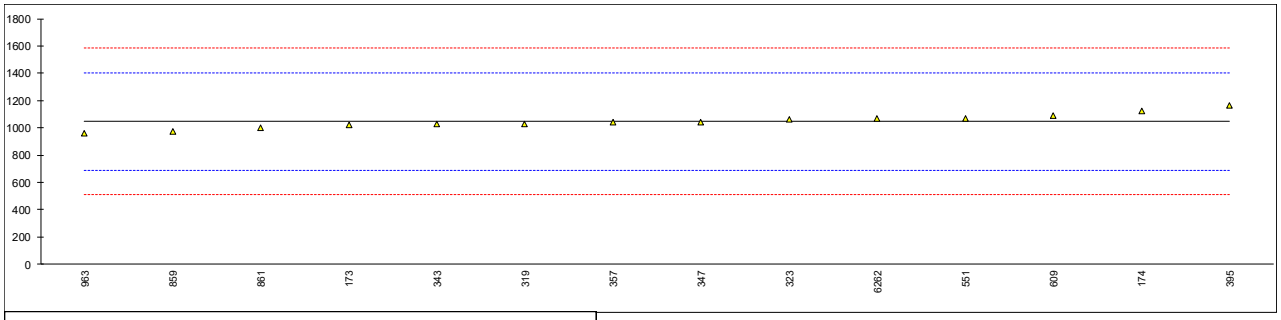
Determination of Sulfate as SO_4 on sample #21002, results in mg/kg

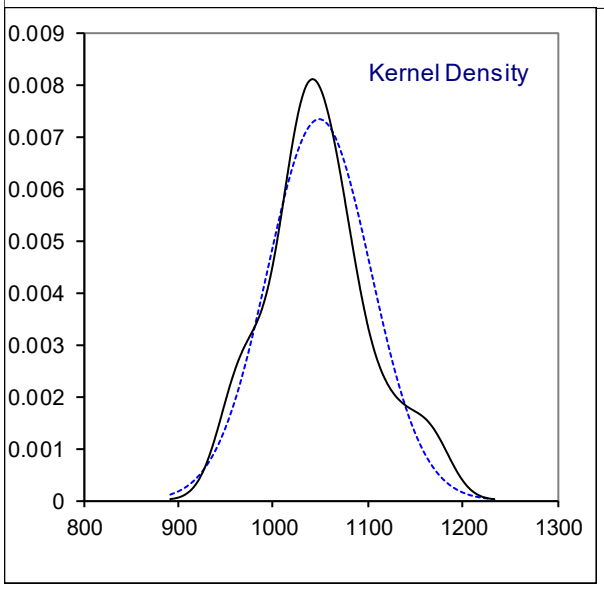
lab	method	value	mark	z(targ)	remarks
173		-----		-----	
174		-----		-----	
319		-----		-----	
323		-----		-----	
343		-----		-----	
347		-----		-----	
357		-----		-----	
395		-----		-----	
551		-----		-----	
558		-----		-----	
609		-----		-----	
663		-----		-----	
859		-----		-----	
861		-----		-----	
912		-----		-----	
913		-----		-----	
963		-----		-----	
6262		-----		-----	
	n	0			
	mean (n)	unknown			

Determination of Water on sample #21002, results in mg/kg

lab	method	value	mark	z(targ)	remarks
173	E203	1020		-0.16	
174	E203	1122		0.42	
319	E1064	1030	C	-0.10	First reported 0.103 mg/kg
323	E302	1060	C	0.07	First reported 0.1060 mg/kg
343	E1064	1030		-0.10	
347	E1064	1040		-0.04	
357	E203	1040	C	-0.04	First reported 0.104 mg/kg
395	E1064	1163	C	0.65	First reported 0.1163 mg/kg
551	E203	1069		0.12	
558		-----		-----	
609	D1364	1090	C	0.24	Reported 0.109 mg/kg
663		-----		-----	
859	E1064	973		-0.42	
861	D1364	1004		-0.24	
912		-----		-----	
913		-----		-----	
963	E203	960.0		-0.49	
6262	D1364	1067		0.11	

normality	OK
n	14
outliers	0
mean (n)	1047.7
st.dev. (n)	54.26
R(calc.)	151.9
st.dev.(E302:95)	178.57
R(E302:95)	500





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

1 lab in NETHERLANDS

1 lab in SAUDI ARABIA

2 labs in SPAIN

1 lab in THAILAND

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	= calculation difference between reported test result and result calculated by iis
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

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4 ASTM E1301:03
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6 ISO5725, parts 1-6:94
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13 Analytical Methods Committee, Technical Brief, No 4, January 2001
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