Results of Proficiency Test
Ethyl Acetate
March 2021

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2015 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Ethyl Acetate every two year. During the annual proficiency testing program 2020/2021 it was decided to continue the round robin for the analysis of Ethyl Acetate.

In this interlaboratory study 18 laboratories in 15 different countries registered for participation. See appendix 2 for the number of participants per country. In this report the results of the Ethyl Acetate 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 Ethyl Acetate, labelled #21026.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for 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 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 13L of Ethyl Acetate was obtained from a local supplier. After homogenisation 26 amber glass bottles of 0.5L were filled and labelled #21026.
The homogeneity of the subsamples was checked by the determination of Density at 20°C in accordance with ASTM D4052 and the determination of Water in accordance with ASTM E1064 on 4 stratified randomly selected subsamples.

	Density at 20°C in kg/L	Water in mg/kg
sample #21026-1	0.89880	1050
sample #21026-2	0.89883	1040
sample #21026-3	0.89883	1050
sample #21026-4	0.89883	1060

Table 1: homogeneity test results of subsamples #21026

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility of the reference test methods in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Density at 20°C in kg/L	Water in mg/kg
r (observed)	0.00004	22.9
reference test method	ISO12185:96	E1064:16
0.3 x R (reference test method)	0.00015	50.1

Table 2: evaluation of the repeatabilities of subsamples #21026

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

To each of the participating laboratories one sample of 0.5L Ethyl Acetate labelled #21026 was sent on February 24, 2021. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Ethyl Acetate 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: Acidity as Acetic acid, Appearance, Color Pt/Co, Density at 20°C, Specific Gravity 20/20°C, Distillation (IBP, 50% recovered, Dry Point and Distillation Range), Nonvolatile matter, Purity, Ethanol 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 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.

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 be used. For larger data sets (above 20 test results) Rosner's outlier test 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 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. 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 result tables of 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

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 reporting date and four other participants did not report any test results. Not all participants were able to report all tests requested.

In total 14 participants reported 99 numerical test results. Observed were 6 outlying test results, which is 6.1%. In proficiency tests 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 “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER TEST

In this section the reported 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 (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 results tables of appendix 1 only the method number and year of adoption or revision e.g. D1209:05 will be used.

Acidity as Acetic Acid: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D1613:17.

Appearance: This determination was not problematic. All reporting participants agreed about the appearance of the sample, which was bright, clear and free of suspended matter (Pass).

Color Pt/Co: The 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. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ISO12185:96.

Specific Gravity 20/20°C: 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 ISO12185:96.

Distillation: The determination was not problematic for Initial Boiling Point (IBP) and 50% recovery, and problematic for Dry Point (DP) and Distillation Range. No statistical outliers were observed. The calculated reproducibilities are in agreement with the requirements of ASTM D1078:19 automated and manual method for IBP and 50% recovery but not for DP and Distillation Range.

Nonvolatile matter: This determination may not be problematic. Eight participants reported a test result. Seven of them agreed on a value for Nonvolatile matter less than 1 mg/100mL. No z-scores were calculated because of the low amount of Nonvolatile matter.

Purity: This determination is very problematic. The deviation between the reported test results is too large compared to the requirements of ASTM D3545:06(2012). Therefore, no z-scores could be calculated.

Ethanol: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility calculated with the Horwitz equation.

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM E1064:16.

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 and ISO test methods) or estimated using the Horwitz equation are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Acidity as Acetic Acid	mg/kg	11	61.81	15.6	14
Appearance		11	Pass	n.a.	n.a.
Color Pt/Co		8	3.0	1.5	7
Density at 20°C	kg/L	11	0.8989	0.0002	0.0005
Specific Gravity 20/20°C		10	0.9005	0.0002	0.0005
Initial Boiling Point	°C	7	76.3	0.8	1.2
50% recovery	°C	6	77.1	0.4	0.5
Dry Point	°C	7	78.5	1.6	0.8
Distillation Range	°C	7	2.1	2.0	0.9
Nonvolatile matter	mg/100mL	7	<1	n.e.	n.e.
Purity	%M/M	5	98.2	1.4	(0.09)
Ethanol	mg/kg	5	43.8	8.2	11.1
Water	mg/kg	12	1082	71	172

Table 3: reproducibilities of tests on sample #21026

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2021 WITH PREVIOUS PTS.

	March 2021	March 2019	April 2017	April 2015
Number of reporting laboratories	14	15	15	12
Number of test results	99	157	246	180
Number of statistical outliers	6	2	11	3
Percentage of statistical outliers	6.1%	1.3%	4.5%	1.7%

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 against the requirements of the respective reference test methods. The conclusions are given in the following table.

	March 2021	March 2019	April 2017	April 2015
Acidity as Acetic Acid	-	--	-	++
Color Pt/Co	++	++	+/-	+
Density at 20°C	++	++	++	+
Specific Gravity 20/20°C	++	++	++	+
Initial Boiling Point	+	++	+	++
50% recovery	+	++	++	++
Dry Point	-	++	++	++
Distillation Range	--	+	+/-	+
Nonvolatile matter	n.e.	n.e.	-	--
Purity	(--)	++	+/-	+
Ethanol	+	+/-	-	-
Water	++	--	+	++

Table 5: comparison determinations against the reference test methods

The following performance categories were used:

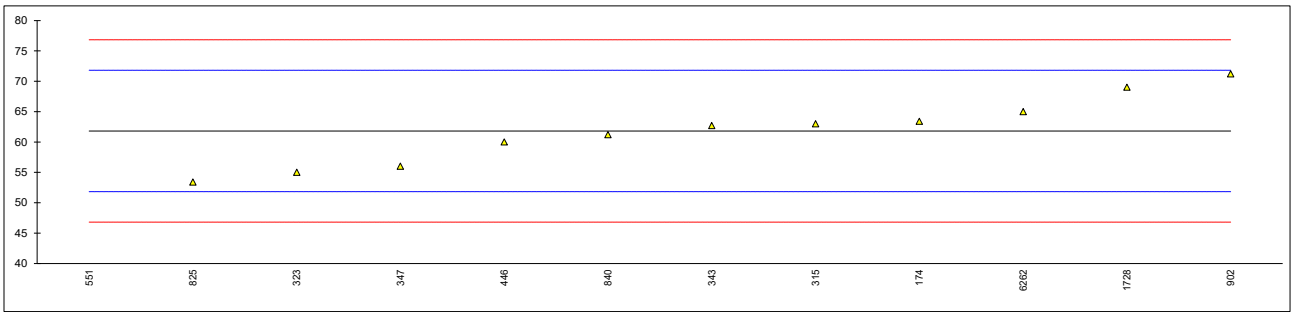
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals to the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test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

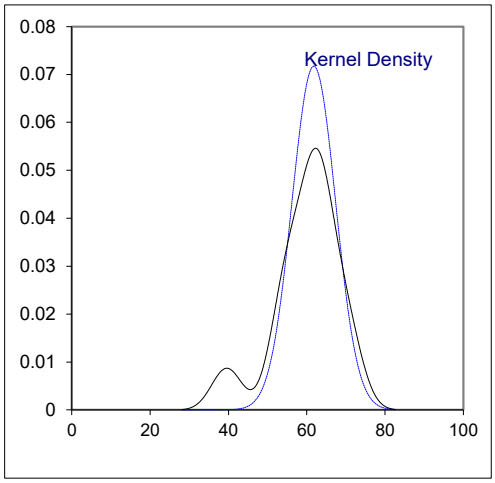
APPENDIX 1

Determination of Acidity as Acetic Acid on sample #21026; results in mg/kg

lab	method	value	mark	z(targ)	remarks
174	D1613	63.4		0.32	
315	D1613	63		0.24	
323	D1613	55		-1.36	
343	D1613	62.7		0.18	
347	D1613	56		-1.16	
446	D1613	60		-0.36	
551	D1613	39.6	G(0.05)	-4.44	
825	D1613	53.4		-1.68	
840	D1613	61.2		-0.12	
902	D1613	71.2		1.88	
912		-----		-----	
913		-----		-----	
963		-----		-----	
1010		-----		-----	
1728	D1613	69		1.44	
2458		-----		-----	
6262	D1613	65		0.64	
6281		-----		-----	

normality	OK
n	11
outliers	1
mean (n)	61.81
st.dev. (n)	5.561
R(calc.)	15.57
st.dev.(D1613:17)	5
R(D1613:17)	14





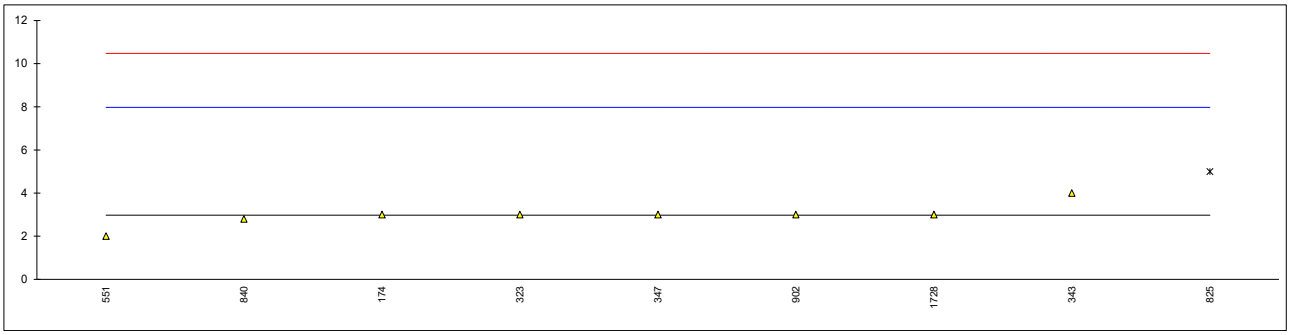
Determination of Appearance on sample #21026

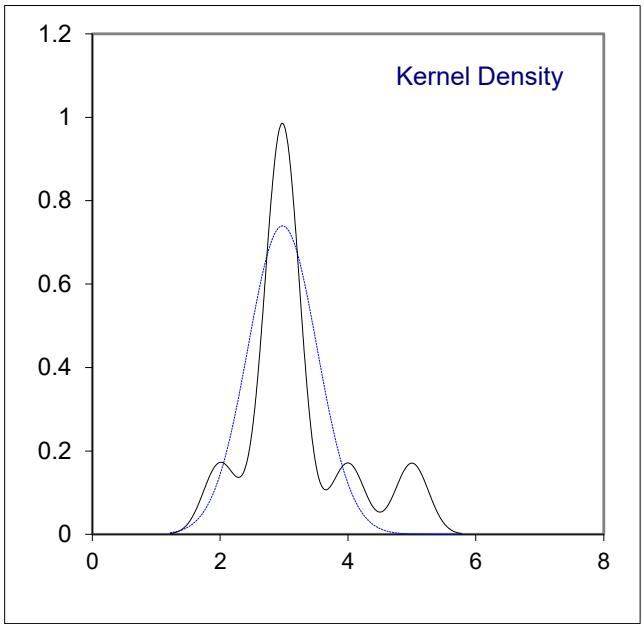
lab	method	value	mark	z(targ)	remarks
174	Visual	Clear & Free		-----	
315	E2680	Pass		-----	
323	INH-001	C&B		-----	
343	E2680	Pass		-----	
347	E2680	Pass		-----	
446	E2680	Pass		-----	
551	E2680	Pass		-----	
825		-----		-----	
840	E2680	Pass		-----	
902	E2680	PASS		-----	
912		-----		-----	
913		-----		-----	
963		-----		-----	
1010		-----		-----	
1728	Visual	CLEAR		-----	
2458		-----		-----	
6262	Visual	clear and bright		-----	
6281		-----		-----	
	n	11			
	mean (n)	Pass			

Determination of Color Pt/Co on sample #21026

lab	method	value	mark	z(targ)	remarks
174	D5386	3.0		0.01	
315	D1209	<5		-----	
323	D1209	3		0.01	
343	D5386	4		0.41	
347	D5386	3		0.01	
446	D1209	<5		-----	
551	D1209	2		-0.39	
825	D1209	5	G(0.01)	0.81	
840	D5386	2.8		-0.07	
902	D5386	3		0.01	
912		-----		-----	
913		-----		-----	
963		-----		-----	
1010		-----		-----	
1728	D1209	3		0.01	
2458		-----		-----	
6262	D1209	<5		-----	
6281		-----		-----	

normality	not OK
n	8
outliers	1
mean (n)	3.0
st.dev. (n)	0.54
R(calc.)	1.5
st.dev.(D1209:05)	2.50
R(D1209:05)	7

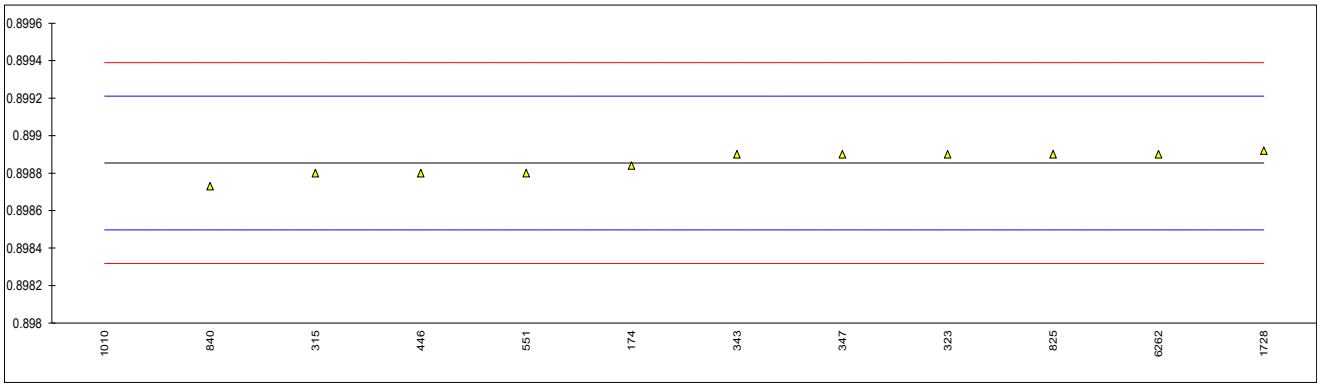


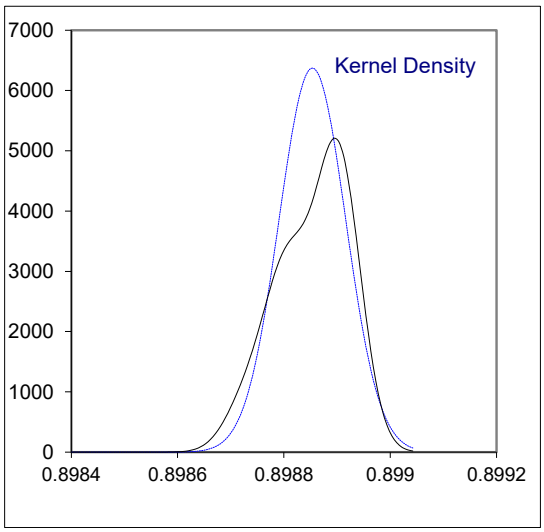


Determination of Density at 20°C on sample #21026; results in kg/L

lab	method	value	mark	z(targ)	remarks
174	D4052	0.89884		-0.08	
315	D4052	0.8988		-0.30	
323	D4052	0.8989		0.26	
343	D4052	0.8989		0.26	
347	D4052	0.8989		0.26	
446	D4052	0.8988		-0.30	
551	D4052	0.8988		-0.30	
825	ISO12185	0.8989		0.26	
840	D4052	0.89873		-0.69	
902		-----		-----	
912		-----		-----	
913		-----		-----	
963		-----		-----	
1010	D4052	0.8968	G(0.01)	-11.50	
1728	D4052	0.89892		0.37	
2458		-----		-----	
6262	D4052	0.8989		0.26	
6281		-----		-----	

normality	OK
n	11
outliers	1
mean (n)	0.89885
st.dev. (n)	0.000063
R(calc.)	0.00018
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005
Compare	
R(D4052:18a)	0.0005

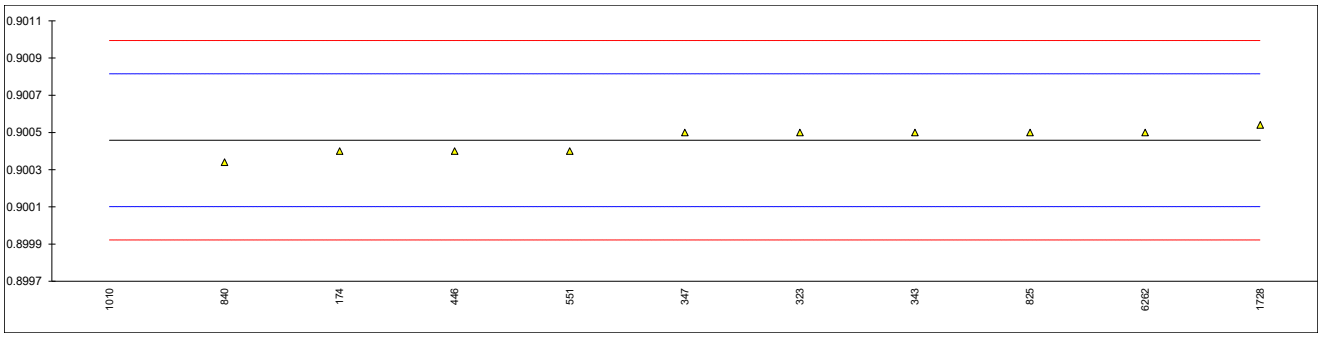


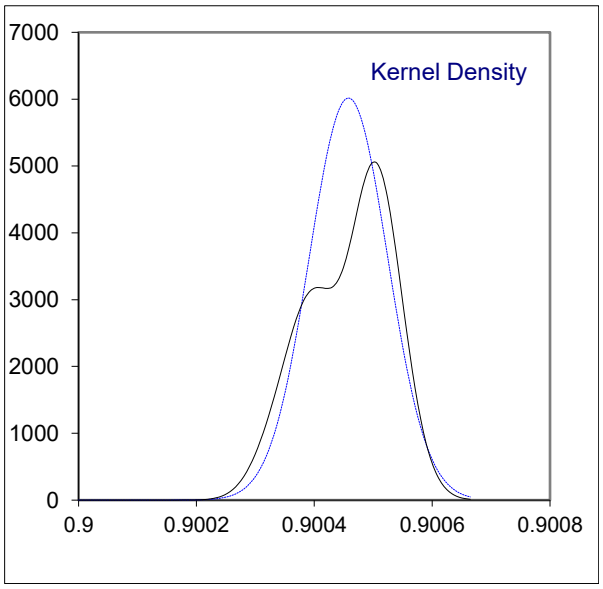


Determination of Specific Gravity 20/20°C on sample #21026

lab	method	value	mark	z(targ)	remarks
174	D4052	0.9004		-0.32	
315		-----		-----	
323	D4052	0.9005		0.24	
343	D4052	0.9005		0.24	
347	D4052	0.9005		0.24	
446	D4052	0.9004		-0.32	
551	D4052	0.9004		-0.32	
825	ISO12185	0.9005		0.24	
840	D4052	0.90034		-0.66	
902		-----		-----	
912		-----		-----	
913		-----		-----	
963		-----		-----	
1010	D4052	0.8984	G(0.01)	-11.52	
1728	D4052	0.90054		0.46	
2458		-----		-----	
6262	D4052	0.9005		0.24	
6281		-----		-----	

normality	OK
n	10
outliers	1
mean (n)	0.90046
st.dev. (n)	0.000066
R(calc.)	0.00019
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005
Compare	
R(D4052:18a)	0.0005





Determination of Distillation on sample #21026; results in °C

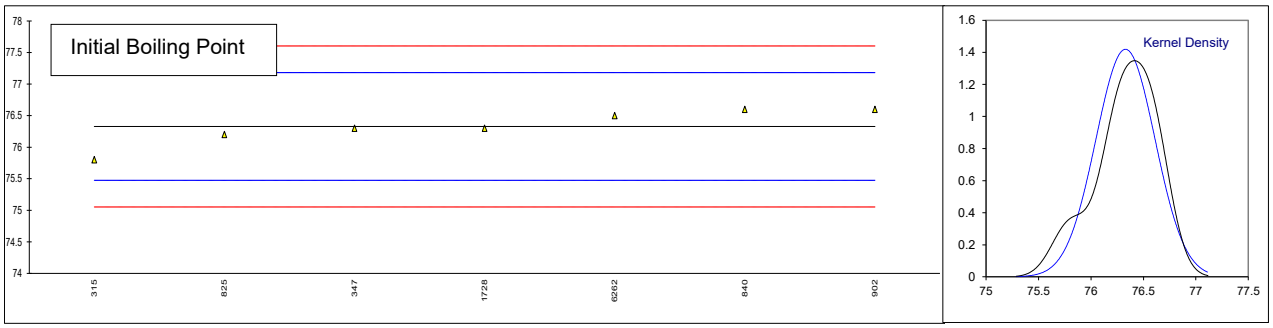
lab	method	IBP	mark	50% rec	mark	DP	mark	Distil. Range	mark
174		-----		-----		-----		-----	
315	D1078-automated	75.8		76.9		78.4		2.6	
323	D1078-manual	-----		-----		-----		-----	
343		-----		-----		-----		-----	
347	D1078-automated	76.3		-----		78.7		2.4	
446		-----		-----		-----		-----	
551		-----		-----		-----		-----	
825		76.2		77.1		79.0		2.8	
840	D1078-automated	76.60		77.27		78.75		2.15	
902	D1078-automated	76.6		77.2		77.6		1	
912		-----		-----		-----		-----	
913		-----		-----		-----		-----	
963		-----		-----		-----		-----	
1010		-----		-----		-----		-----	
1728	D1078-manual	76.3		77.2		79	C	2.7	C
2458		-----		-----		-----		-----	
6262	D1078-automated	76.5		77.0		77.8		1.3	
6281		-----		-----		-----		-----	
	normality	unknown		unknown		unknown		unknown	
	n	7		6		7		7	
	outliers	0		0		0		0	
	mean (n)	76.33		77.11		78.46		2.14	
	st.dev. (n)	0.281		0.140		0.563		0.711	
	R(calc.)	0.79		0.39		1.58		1.99	
	st.dev.(D1078-A:11)	0.425		0.188		0.300		0.330	
	R(D1078-A:11)	1.19		0.53		0.84		0.92	
	Compare								
	R(D1078-M:11)	0.82		0.50		1.02		0.83	

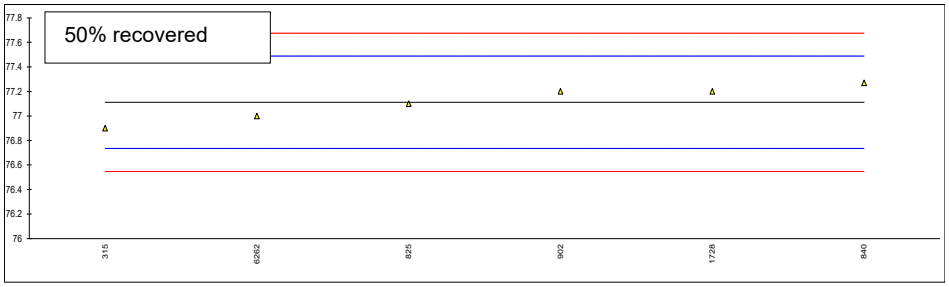
Lab 1728 first reported 80.9 for DP and 4.6 for Distillation range

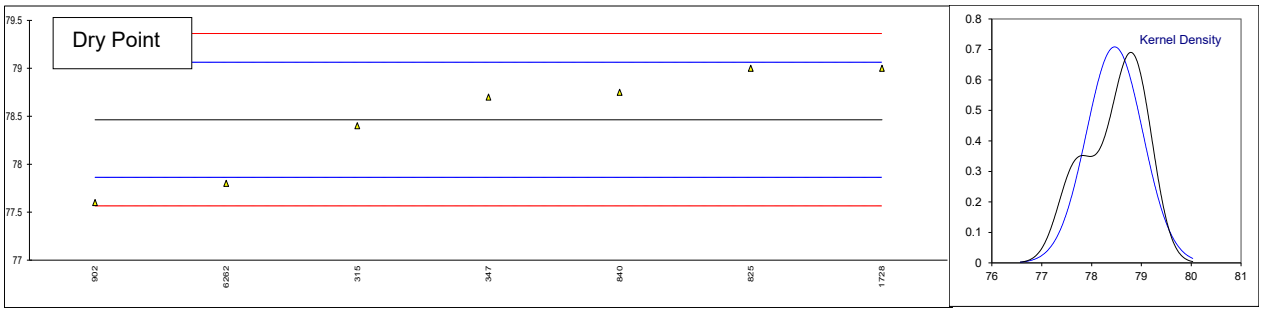
Theoretical mid boiling point = 77.2 °C

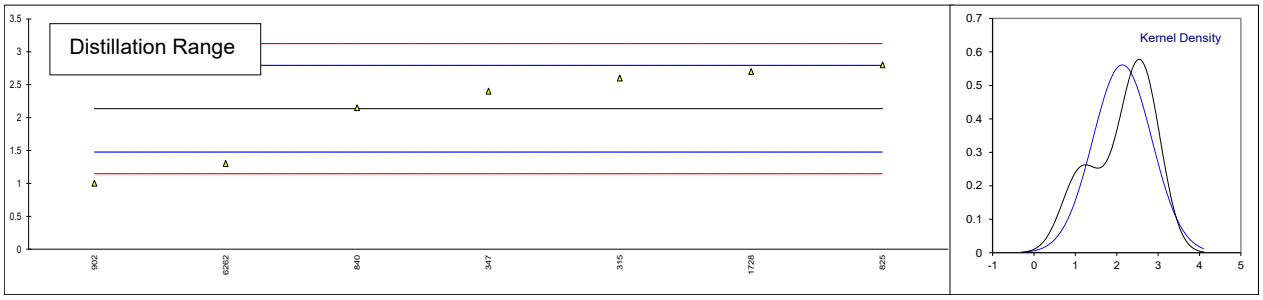
z-scores of Distillation on sample #21026

lab	method	IBP	50% rec	DP	Distil. Range
174		-----	-----	-----	-----
315	D1078-automated	-1.24	-1.13	-0.21	1.41
323	D1078-manual	-----	-----	-----	-----
343		-----	-----	-----	-----
347	D1078-automated	-0.07	-----	0.79	0.80
446		-----	-----	-----	-----
551		-----	-----	-----	-----
825		-0.30	-0.06	1.79	2.01
840	D1078-automated	0.64	0.84	0.95	0.04
902	D1078-automated	0.64	0.47	-2.88	-3.44
912		-----	-----	-----	-----
913		-----	-----	-----	-----
963		-----	-----	-----	-----
1010		-----	-----	-----	-----
1728	D1078-manual	-0.07	0.47	1.79	1.71
2458		-----	-----	-----	-----
6262	D1078-automated	0.40	-0.59	-2.22	-2.53
6281		-----	-----	-----	-----


Initial Boiling Point
78
77.5
77
76.5
76
75.5
75
74.5
74
315
825
347
1728
6262
840
902
Kernel Density
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
75
75.5
76
76.5
77
77.5


50% recovered
77.8
77.6
77.4
77.2
77
76.8
76.6
76.4
76.2
76
315
6262
825
902
1728
840


Dry Point
79.5
79
78.5
78
77.5
77
902
6262
315
347
840
825
1728
Kernel Density
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
76
77
78
79
80
81


Distillation Range
3.5
3
2.5
2
1.5
1
0.5
0
902
6262
840
347
315
1728
825
Kernel Density
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
-1
0
1
2
3
4
5

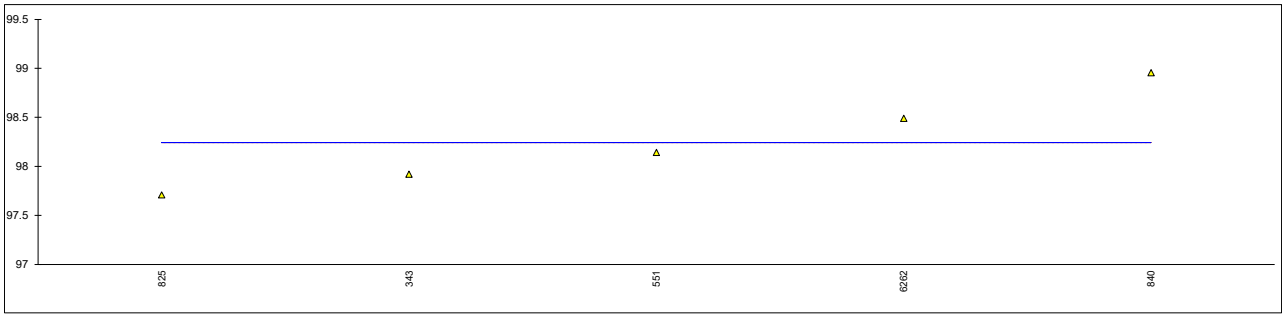
Determination of Nonvolatile matter on sample #21026; results in mg/100mL

lab	method	value	mark	z(targ)	remarks
174		-----		-----	
315	D1353	<1.0		-----	
323	D1353	-----		-----	
343	D1353	<1		-----	
347		-----		-----	
446	D1353	0		-----	
551	D1353	0.6		-----	
825	D1353	0.3		-----	
840	D1353	1.85		-----	Possibly a false positive test result?
902	D1353	<1		-----	
912		-----		-----	
913		-----		-----	
963		-----		-----	
1010		-----		-----	
1728		-----		-----	
2458		-----		-----	
6262	D1353	<1		-----	
6281		-----		-----	
	n	7			
	mean (n)	<1			

Determination of Purity on sample #21026; results in %M/M

lab	method	value	mark	z(targ)	remarks
174		-----		-----	
315		-----		-----	
323	INH-110	< 99.0		-----	
343	INH-243	97.92		-----	
347		-----		-----	
446		-----		-----	
551	D3545	98.1418		-----	
825	D3545	97.7080		-----	
840	INH-002	98.955		-----	
902		-----		-----	
912		-----		-----	
913		-----		-----	
963		-----		-----	
1010		-----		-----	
1728		-----		-----	
2458		-----		-----	
6262	D3545	98.49		-----	
6281		-----		-----	

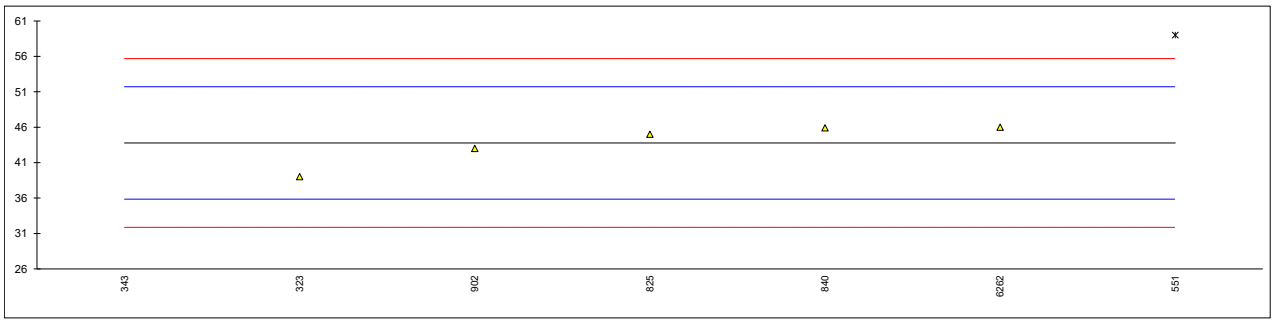
normality	unknown
n	5
outliers	0
mean (n)	98.24
st.dev. (n)	0.492
R(calc.)	1.38
st.dev.(D3545:06)	(0.032)
R(D3545:06)	(0.09)

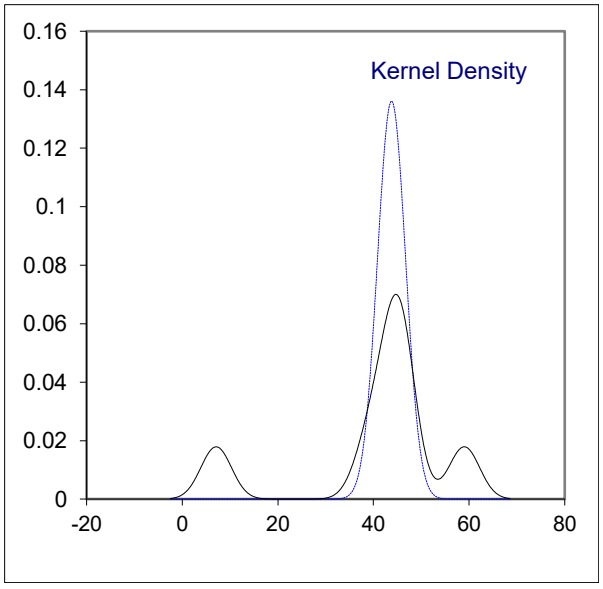


Determination of Ethanol on sample #21026; results in mg/kg

lab	method	value	mark	z(targ)	remarks
174		-----		-----	
315		-----		-----	
323	INH-110	39		-1.21	
343	INH-243	7.1	G(0.05)	-9.25	
347		-----		-----	
446		-----		-----	
551	D3545	59	D(0.05)	3.84	
825	D3545	45		0.31	
840	INH-002	45.9		0.53	
902	INH-128	43		-0.20	
912		-----		-----	
913		-----		-----	
963		-----		-----	
1010		-----		-----	
1728		-----		-----	
2458		-----		-----	
6262	D3545	46		0.56	
6281		-----		-----	

normality	unknown
n	5
outliers	2
mean (n)	43.78
st.dev. (n)	2.931
R(calc.)	8.21
st.dev.(Horwitz)	3.966
R(Horwitz)	11.10

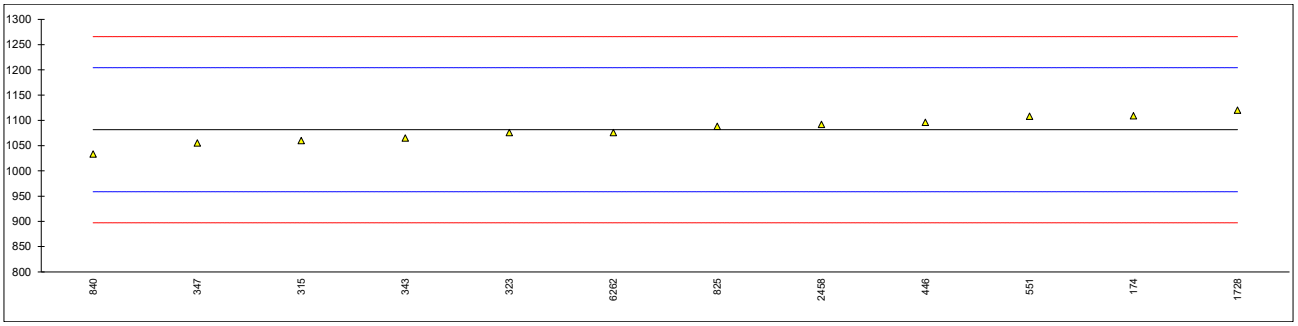


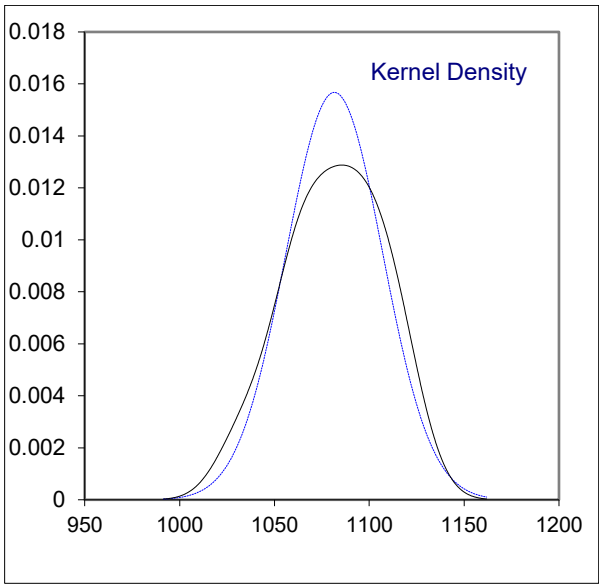


Determination of Water on sample #21026; results in mg/kg

lab	method	value	mark	z(targ)	remarks
174	E203	1109		0.45	
315	E1064	1060		-0.35	
323	E1064	1076		-0.09	
343	E1064	1065		-0.27	
347	E1064	1055	C	-0.43	First reported 1550
446	D1364	1096		0.24	
551	D1064	1108		0.43	
825	E1064	1088		0.11	
840	E1064	1033.2		-0.79	
902		-----		-----	
912		-----		-----	
913		-----		-----	
963		-----		-----	
1010		-----		-----	
1728	E1064	1120		0.63	
2458	EN13267	1092		0.17	
6262	E1064	1076		-0.09	
6281		-----		-----	

normality	OK
n	12
outliers	0
mean (n)	1081.52
st.dev. (n)	25.466
R(calc.)	71.30
st.dev.(E1064:16)	61.415
R(E1064:16)	171.96





APPENDIX 2

Number of participants per country

2 labs in BELGIUM
1 lab in BRAZIL
1 lab in GERMANY
2 labs in INDIA
1 lab in NETHERLANDS
1 lab in NORWAY
1 lab in ROMANIA
1 lab in SAUDI ARABIA
1 lab in SOUTH KOREA
2 labs in SPAIN
1 lab in THAILAND
1 lab in TURKEY
1 lab in UNITED KINGDOM
1 lab in UNITED STATES OF AMERICA
1 lab in VIETNAM

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

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
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7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
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10 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364 (2002)
11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)