Results of Proficiency Test
Biogasoline E85
June 2020

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. G.A. Oosterlaken-Buijs
Correctors: ing. A.S. Noordman-de Neef & ing. M. Meijer

Report: iis20B05

October 2020

CONTENTS

1 INTRODUCTION

Since 2010 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Biogasoline E85 every year. During the annual proficiency testing program 2019/2020 it was decided to continue the round robin for the analysis of Biogasoline E85 based on the scope of the latest version of EN15293 and ASTM D5798.

In this interlaboratory study 14 laboratories in 8 different countries registered for participation. See appendix 2 for the number of participants per country. In this report the results of the Biogasoline E85 proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send one sample of 1 liter Biogasoline E85 labelled #20083.
Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

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 organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

Approximately 60 liters of Biogasoline E85 was obtained from a local supplier. After homogenization 40 amber glass bottles of 1 liter were filled and labelled #20083. The homogeneity of the subsamples was checked by determination of Density at 15°C in accordance with ISO12185 on 8 stratified randomly selected subsamples.

	Density at 15°C in kg/m^3
Sample #20083-1	784.05
Sample #20083-2	784.05
Sample #20083-3	784.05
Sample #20083-4	784.04
Sample #20083-5	784.11
Sample #20083-6	784.05
Sample #20083-7	784.13
Sample #20083-8	784.04

Table 1: homogeneity test results of subsamples #20083

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Density at 15°C in kg/m^3
r (observed)	0.10
reference test method	ISO12185:96
0.3 * R (reference test method)	0.45

Table 2: evaluation of repeatability of the subsamples #20083

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

To each of the participating laboratories one bottle of 1L Biogasoline E85 labelled #20083 was sent on May 20, 2020. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Biogasoline E85 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 on sample #20083: Total Acidity as Acetic Acid, Appearance, Inorganic Chloride as Cl, Copper Corrosion 3hrs at 50°C, Copper as Cu, Density at 15°C, Electrical Conductivity at 25°C, Gum (solvent washed), Oxidation Stability, Methanol, Ethanol, Ethers, Higher saturated (C3-C5) mono-alcohols, Total Oxygen content, pHe (with LiCl and with KCL electrode), Phosphorus as P, Sulfate as SO4, Sulfur 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.

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 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 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. 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 may be 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 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 1 week. Finally, two participants did not report any test results. Not all laboratories were able to perform all requested analyzes. In total 12 participants reported 98 numerical test results. Observed were 4 outlying test results, which is 4.1%. In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as “not OK”, “suspect” or “unknown”. 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 where 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.

In the iis PT reports ASTM test methods are referred to with a number (e.g. D6423) and an added designation for the year that the test method was adopted or revised (e.g. D6423:18).

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 reproducibility estimated from the Horwitz equation.

Total Acidity as Acetic Acid: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN15491:07.

Appearance: This determination was not problematic. All reporting laboratories agreed about the appearance of the sample, clear and bright.

Inorganic Chloride as Cl: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN15492:12.

Copper Corrosion: This determination was not problematic. All reporting participants agreed on a test result of 1 (1a).

Copper as Cu: This determination was not problematic. Almost all reporting participants agreed on a test result below 0.07 mg/kg. Therefore, no z-scores are calculated.

Density at 15°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ISO12185:96.

Electrical Conductivity: 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 EN15938:10.

Gum (solvent washed): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ISO6246:17.

Oxidation Stability: This determination was not problematic. All reporting participants agreed on a test result above 360 minutes according to the specification EN15293:2018.

Methanol: This determination was not problematic. The reporting participants agreed on a value close or below the application range. Therefore, no z-scores are calculated.

Ethanol: 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 ISO22854-B:16.

Ethers: 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 ISO22854-B:16.

Higher saturated mono-alcohols: This determination was not problematic. The reporting participants agreed on a value below the application range. Therefore, no z-scores are calculated.

Total Oxygen: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility using the Horwitz equation.
Unfortunately, procedure B of ISO22854:16 doesn't provide precision data for Total Oxygen content. Therefore, it was decided to use the Horwitz equation.

pHe, LiCl electrode: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of EN15490:07.

pHe, KCl electrode: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D6423:18.

Phosphorus as P: This determination was not problematic. The reporting participants agreed on a value near or below the application range. Therefore, no z-scores are calculated.

Sulfate as SO_4: This determination was not problematic. The reporting participants agreed on a value near or below the application range. Therefore, no z-scores are calculated.

Sulfur: This determination was not problematic. The reporting participants agreed on a value near or below the application range. Therefore, no z-scores are calculated.

Water: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of EN15489:07.

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 using 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 EN and ISO test methods) or previous proficiency tests are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Acidity as Acetic Acid	%M/M	10	0.0018	0.0012	0.0014
Appearance		7	C&B	n.a.	n.a.
Inorganic Chloride as Cl	mg/kg	4	0.8	0.4	0.6
Copper Corrosion 3hrs at 50°C		7	1 (1a)	n.a.	n.a.
Copper as Cu	mg/kg	5	<0.07	n.e.	n.e.
Density at 15°C	kg/m^3	12	784.20	0.61	1.5
Electrical Conductivity at 25°C	µS/cm	7	1.13	0.23	0.20
Gum (solvent washed)	mg/100mL	4	0.35	0.49	1.82
Oxidation Stability	minutes	7	>360	n.a.	n.a.
Methanol	%V/V	8	<0.5	n.e.	n.e.
Ethanol	%V/V	10	84.54	2.32	4.85
Ethers	%V/V	6	1.54	0.17	0.33
Higher saturated mono-alcohols	%V/V	6	<0.2	n.e.	n.e.
Total Oxygen content	%M/M	8	30.1	1.0	2.0
pHe LiCl		4	5.67	0.76	0.55
pHe KCl		3	6.95	1.26	1.10
Phosphorus as P	mg/L	6	<0.2	n.e.	n.e.
Sulfate as SO4	mg/kg	5	<1	n.e.	n.e.
Sulfur	mg/kg	11	<5	n.e.	n.e.
Water	%M/M	11	0.181	0.024	0.022

Table 3: reproducibilities of tests on sample #20083

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF JUNE 2020 WITH PREVIOUS PTS

	June 2020	May 2019	May 2018	May 2017	May 2016
Number of reporting laboratories	12	14	13	16	16
Number of test results	98	115	77	91	117
Number of statistical outliers	4	9	7	5	7
Percentage of statistical outliers	4.1%	7.8%	9.1%	5.5%	6.0%

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.

	June 2020	May 2019	May 2018	May 2017	May 2016
Total Acidity as Acetic Acid	+	++	+	+	+
Inorganic Chloride as Cl	+	--	-	--	++
Density at 15°C	++	++	++	+/-	++
Electrical Conductivity at 25°C	-	-	+	+	-
Gum (solvent washed)	++	++	++	++	++
Methanol	n.e.	n.e.	n.e.	n.e.	-
Ethanol	++	++	+	++	+
Ethers	+	+	--	--	+/-
Total Oxygen content	++	-	+	++	++
pHe LiCl	-	+	++	--	--
pHe KCl	-	+/-	++		
Sulfate as SO4	n.e.	n.e.	n.e.	n.e.	(--)
Sulfur	n.e.	n.e.	n.e.	++	++
Water	+/-	+	++	-	+

Table 5: comparison determinations against the reference test method

NB. For results between brackets no z-scores are calculated

The following performance categories were used:

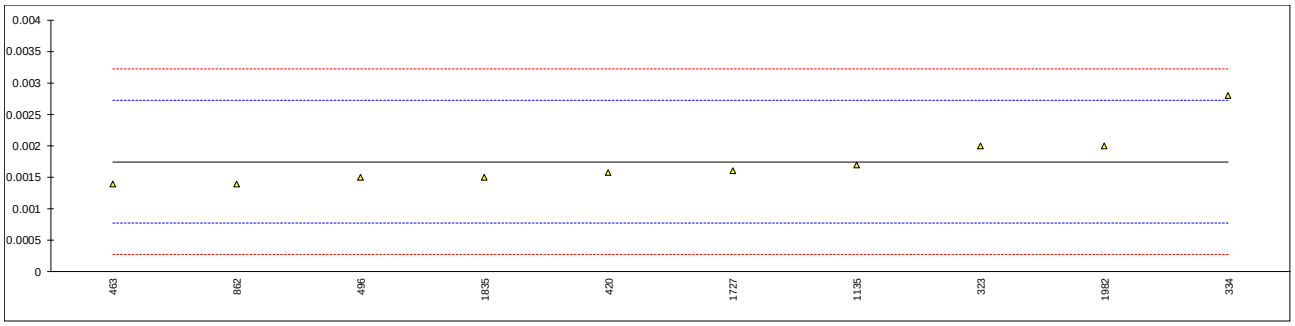
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test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

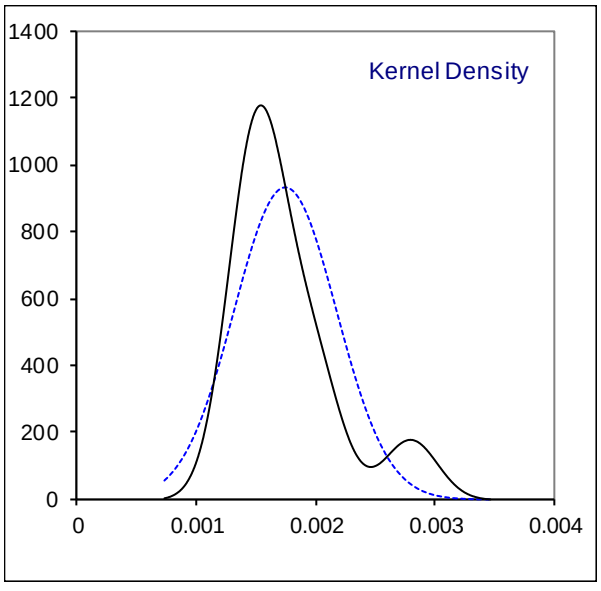
APPENDIX 1

Determination of Total Acidity as Acetic Acid on sample #20083; results in %M/M

lab	method	value	mark	z(targ)	remarks
323	EN15491	0.002		0.52	
334	EN15491	0.0028		2.15	
420	EN15491	0.00157	C	-0.36	first reported 0.0016 mg/kg
463	D1613	0.0014		-0.71	
496	EN15491	0.0015		-0.50	
862	D1613	0.0014		-0.71	
1033		-----		-----	
1135	D1613	0.0017		-0.10	
1459		-----		-----	
1727	EN15491	0.0016		-0.30	
1835	EN15491	0.0015		-0.50	
1982	D1613	0.0020		0.52	
1984		-----		-----	
6262		-----		-----	

normality	not OK
n	10
outliers	0
mean (n)	0.00175
st.dev. (n)	0.000429
R(calc.)	0.00120
st.dev.(EN15491:07)	0.000489
R(EN15491:07)	0.00137





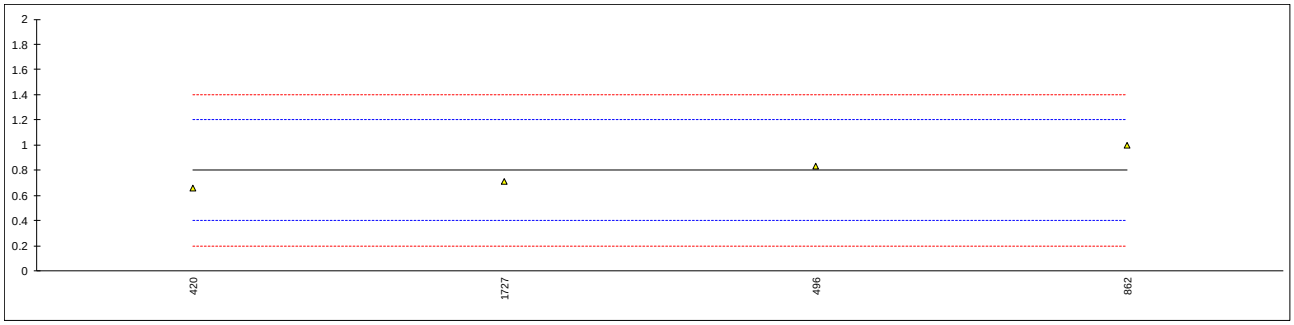
Determination of Appearance on sample #20083;

lab	method	value	mark	z(targ)	remarks
323	D4176	clear & bright		-----	
334	Visual	clear and bright		-----	
420	EN15769	clear, colorless		-----	
463		-----		-----	
496	Visual	c + b		-----	
862	Visual	Clear&bright		-----	
1033		-----		-----	
1135	Visual	Clear & Bright		-----	
1459		-----		-----	
1727		-----		-----	
1835	EN15769	C&C		-----	
1982		-----		-----	
1984		-----		-----	
6262		-----		-----	
	n	7			
	mean (n)	Clear & Bright (C&B)			

Determination of Inorganic Chloride as Cl on sample #20083; results in mg/kg

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	EN15492	<1		-----	
420	EN15484	0.66		-0.70	
463		-----		-----	
496	EN15492	0.83		0.15	
862	EN15492	1.0		1.00	
1033		-----		-----	
1135		-----		-----	
1459		-----		-----	
1727	EN15492	0.71		-0.45	
1835	EN15492	<1.0		-----	
1982		-----		-----	
1984		-----		-----	
6262		-----		-----	

normality	unknown
n	4
outliers	0
mean (n)	0.800
st.dev. (n)	0.1512
R(calc.)	0.423
st.dev.(EN15492:12)	0.2000
R(EN15492:12)	0.560



Determination of Copper Corrosion 3hrs at 50°C on sample #20083; rating

lab	method	value	mark	z(targ)	remarks
323	D130	1A		-----	
334	D130	1		-----	
420	ISO2160	class 1a		-----	
463	ISO2160	1A		-----	
496	ISO2160	1a		-----	
862	D130	1a		-----	
1033		-----		-----	
1135	D130	1A		-----	
1459		-----		-----	
1727		-----		-----	
1835		-----		-----	
1982		-----		-----	
1984		-----		-----	
6262		-----		-----	
	n	7			
	mean (n)	1 (1a)			

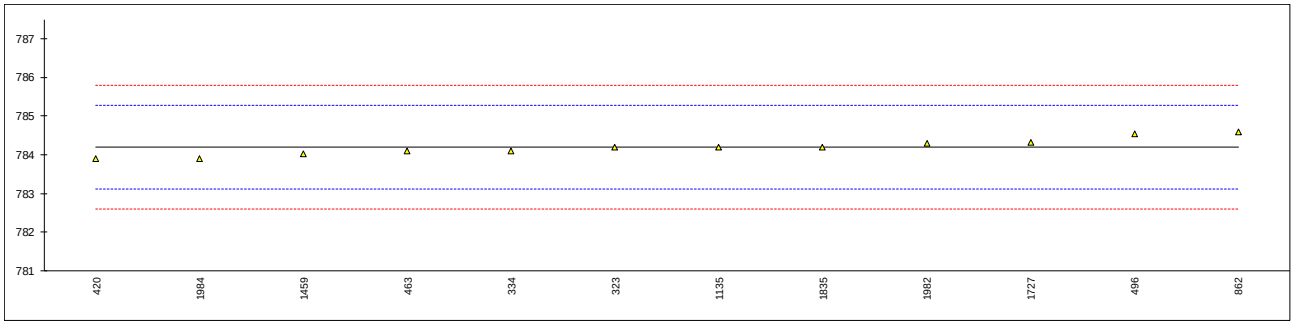
Determination of Copper as Cu on sample #20083; results in mg/kg

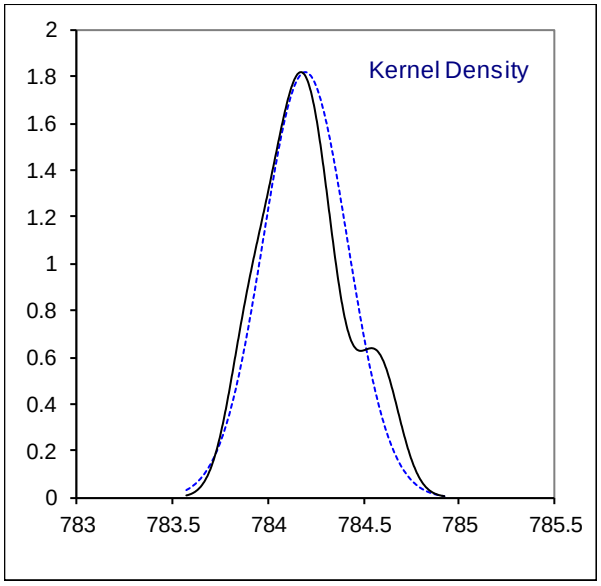
lab	method	value	mark	z(targ)	remarks
323	EN15488	<0.070		-----	
334	EN15488	<0.07		-----	
420	EN15837	<0,05		-----	
463		-----		-----	
496		-----		-----	
862	EN15488	<0.07		-----	
1033		-----		-----	
1135	UOP389	<0.1		-----	
1459		-----		-----	
1727		-----		-----	
1835	EN15837	<0.050		-----	
1982		-----		-----	
1984		-----		-----	
6262		-----		-----	
	n	5			
	mean (n)	<0.07			

Determination of Density at 15°C on sample #20083; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
323	ISO12185	784.2		0.00	
334	ISO12185	784.1		-0.18	
420	ISO12185	783.9		-0.56	
463	ISO12185	784.09		-0.20	
496	ISO12185	784.54		0.64	
862	ISO12185	784.6		0.75	
1033		-----		-----	
1135	ISO12185	784.2		0.00	
1459	ISO12185	784.03		-0.31	
1727	D4052	784.31		0.21	
1835	ISO12185	784.2		0.00	
1982	ISO12185	784.3		0.19	
1984	ISO12185	783.9		-0.56	
6262		-----		-----	

normality	OK
n	12
outliers	0
mean (n)	784.198
st.dev. (n)	0.2192
R(calc.)	0.614
st.dev.(ISO12185:96)	0.5357
R(ISO12185:96)	1.5

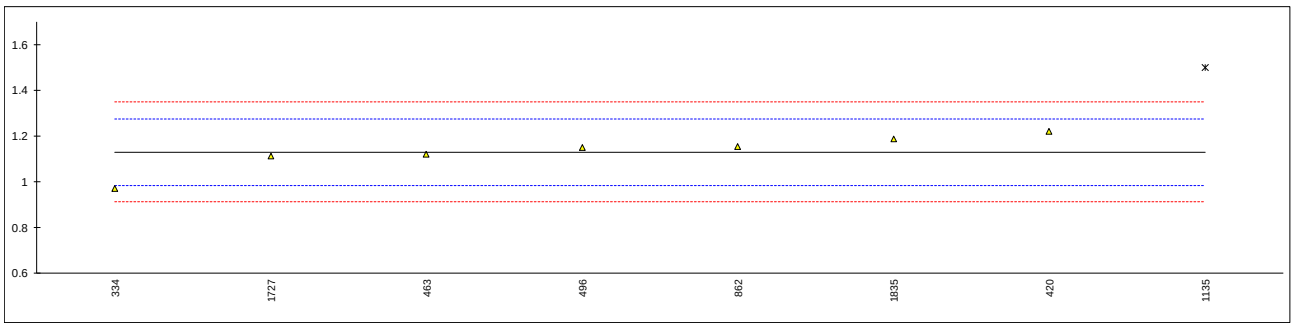


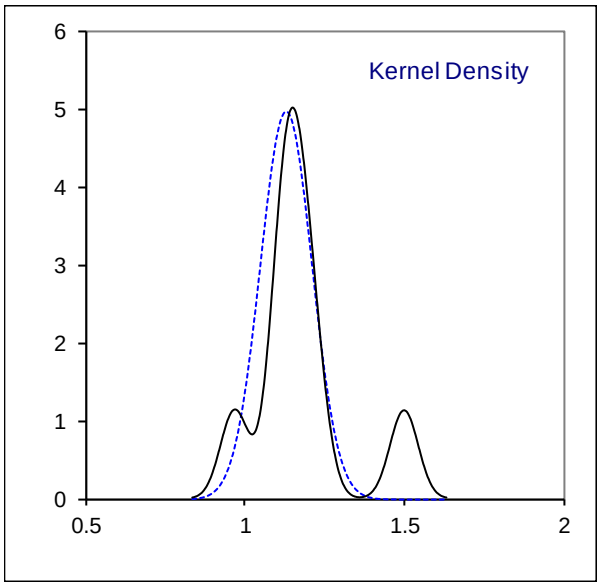


Determination of Electrical Conductivity at 25°C on sample #20083; results in µS/cm

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	EN15938	0.969		-2.22	
420	EN15938	1.22		1.24	
463	EN15938	1.12		-0.14	
496	EN15938	1.15		0.27	
862	EN15938	1.155		0.34	
1033		-----		-----	
1135	EN15938	1.498	C,G(0.05)	5.07	first reported 1.505
1459		-----		-----	
1727	EN15938	1.11		-0.28	
1835	EN15938	1.187		0.78	
1982		-----		-----	
1984		-----		-----	
6262		-----		-----	

normality	unknown
n	7
outliers	1
mean (n)	1.130
st.dev. (n)	0.0804
R(calc.)	0.225
st.dev.(EN15938:10)	0.0725
R(EN15938:10)	0.203

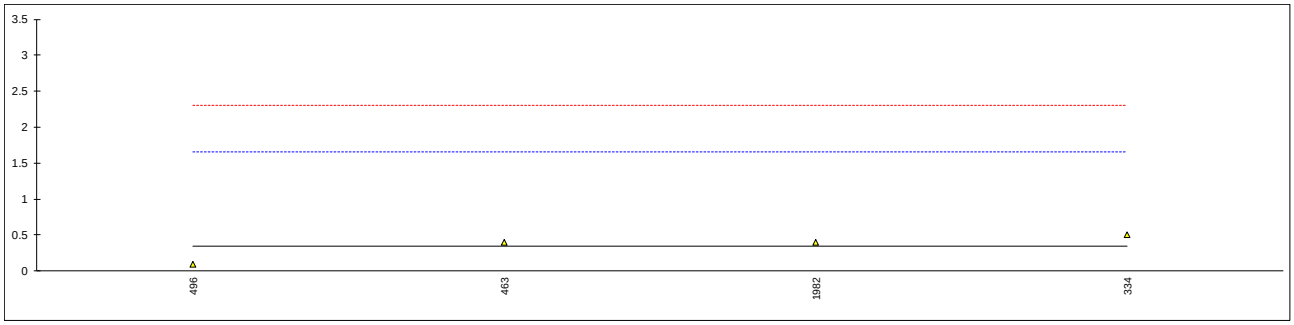




Determination of Gum (solvent washed) on sample #20083; results in mg/100mL

lab	method	value	mark	z(targ)	remarks
323	ISO6246	<0.5		-----	
334	ISO6246	0.5		0.23	
420	ISO6246	<1		-----	
463	ISO6246	0.4		0.08	
496	ISO6246	0.1		-0.38	
862	D381	<0.5		-----	
1033		-----		-----	
1135	ISO6246	<0.5		-----	
1459		-----		-----	
1727		-----		-----	
1835		-----		-----	
1982	ISO6246	0.4		0.08	
1984		-----		-----	
6262		-----		-----	

normality	unknown
n	4
outliers	0
mean (n)	0.350
st.dev. (n)	0.1732
R(calc.)	0.485
st.dev.(ISO6246:17)	0.6501
R(ISO6246:17)	1.820



Determination of Oxidation Stability on sample #20083; results in minutes

lab	method	value	mark	z(targ)	remarks
323	ISO7536	600		-----	
334	ISO7536	>900		-----	
420	ISO7536	>600		-----	
463	D525	>900		-----	
496	D525	>900		-----	
862	D525	>900		-----	
1033		-----		-----	
1135	ISO7536	>900		-----	
1459		-----		-----	
1727		-----		-----	
1835		-----		-----	
1982		-----		-----	
1984		-----		-----	
6262		-----		-----	
	n	7			
	mean (n)	>360			

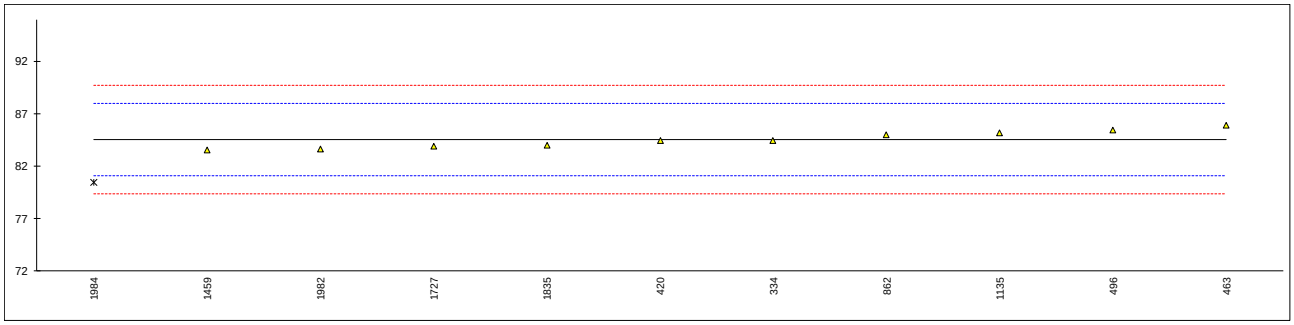
Determination of Methanol on sample #20083; results in %V/V

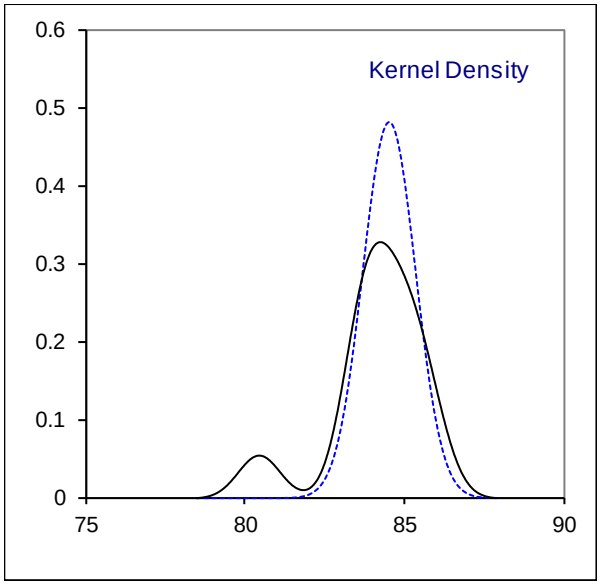
lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	ISO22854-B	0.03		-----	
420	EN13132	<0,1		-----	
463	EN13132	<0,2		-----	
496	EN13132	<0.10		-----	
862	D4815	<0.2		-----	
1033		-----		-----	
1135	ISO22854-B	0.39		-----	
1459		-----		-----	
1727		-----		-----	
1835	In house	<0.01		-----	
1982	D5501	<0,01		-----	
1984		-----		-----	
6262		-----		-----	
	n	8			
	mean (n)	<0.5			application range EN16761-1:16: 0.5 – 1.5 %V/V

Determination of Ethanol on sample #20083; results in %V/V

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	ISO22854-B	84.47		-0.04	
420	EN13132	84.42		-0.07	
463	EN13132	85.93		0.80	
496	ISO22854-B	85.48		0.54	
862	D4815	84.95		0.24	
1033		-----		-----	
1135	ISO22854-B	85.20	C	0.38	first reported 94.45
1459	In house	83.49		-0.61	
1727	In house	83.87		-0.39	
1835	In house	83.97		-0.33	
1982	D5501	83.61		-0.54	
1984	EN1601	80.45	D(0.05)	-2.36	
6262		-----		-----	

normality	OK
n	10
outliers	1
mean (n)	84.539
st.dev. (n)	0.8286
R(calc.)	2.320
st.dev.(ISO22854-B:16)	1.7321
R(ISO22854-B:16)	4.85

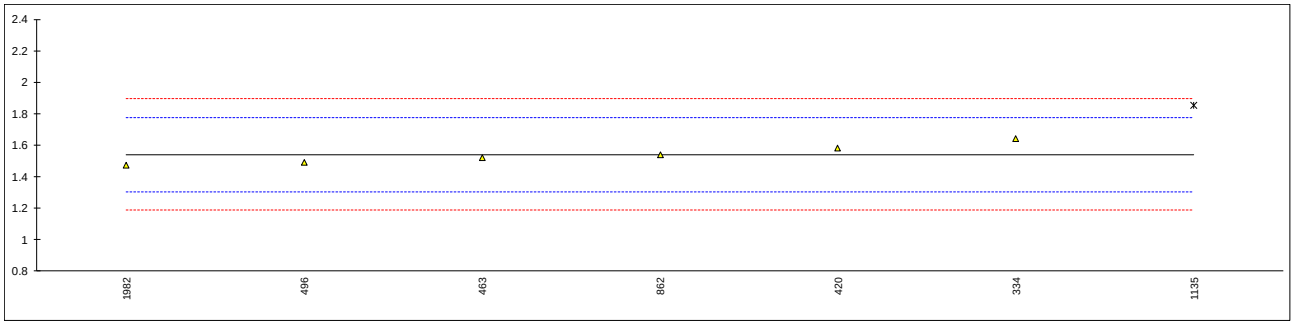


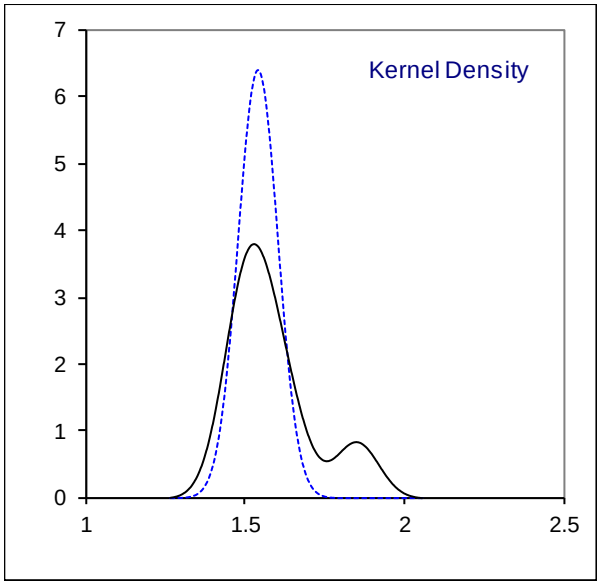


Determination of Ethers on sample #20083; results in %V/V

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	ISO22854-B	1.64		0.85	
420	EN13132	1.58		0.34	
463	EN13132	1.52		-0.17	
496	EN13132	1.49		-0.42	
862	D4815	1.54		0.00	
1033		-----		-----	
1135	ISO22854-B	1.85	G(0.05)	2.63	
1459		-----		-----	
1727		-----		-----	
1835		-----		-----	
1982	D4815	1.47		-0.59	
1984		-----		-----	
6262		-----		-----	

normality	unknown
n	6
outliers	1
mean (n)	1.540
st.dev. (n)	0.0623
R(calc.)	0.174
st.dev.(ISO22854-B:16)	0.1179
R(ISO22854-B:16)	0.33





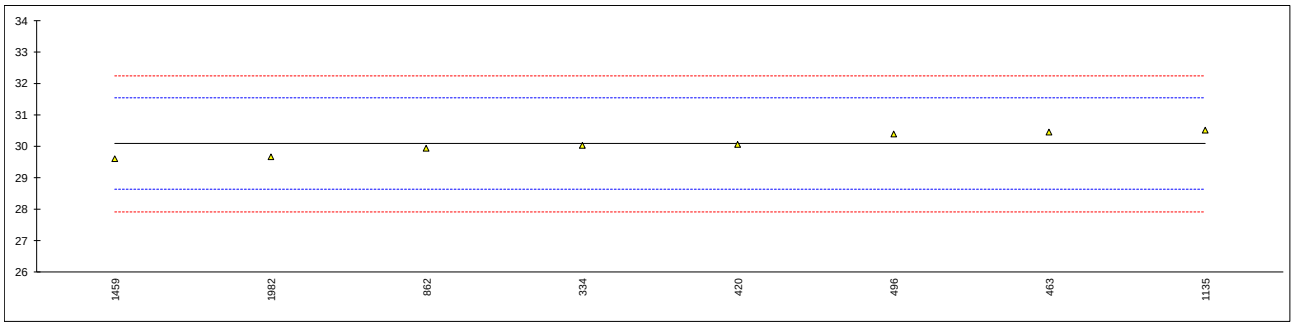
Determination of Higher saturated (C3-C5) mono-alcohols on sample #20083; results in %V/V

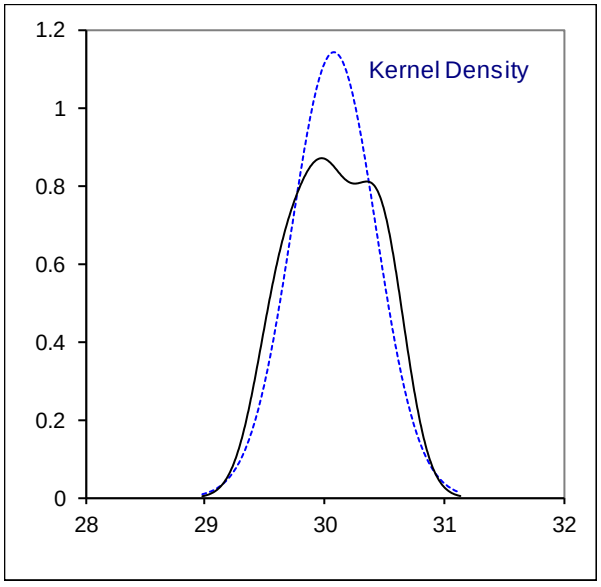
lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	ISO22854-B	0.06		-----	
420	EN13132	<0,1		-----	
463		-----		-----	
496	EN13132	<0.10		-----	
862	D4815	<0.2		-----	
1033		-----		-----	
1135	ISO22854-B	<0.1		-----	
1459		-----		-----	
1727		-----		-----	
1835		-----		-----	
1982	D4815	<0,20		-----	
1984		-----		-----	
6262		-----		-----	
	n	6			
	mean (n)	<0.2			application range ISO22854-B:16: 1.4 - < 2.5 %V/V

Determination of Total Oxygen content on sample #20083; results in %M/M

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	ISO22854-A	30.02		-0.08	
420	EN13132	30.06		-0.03	
463	EN13132	30.46		0.53	
496	ISO22854-B	30.400		0.44	
862	D4815	29.93		-0.21	
1033		-----		-----	
1135	ISO22854-B	30.51	C	0.59	first reported 33.75
1459	In house	29.61		-0.65	
1727		-----		-----	
1835		-----		-----	
1982	D4815	29.66		-0.58	
1984		-----		-----	
6262		-----		-----	

normality	unknown
n	8
outliers	0
mean (n)	30.081
st.dev. (n)	0.3496
R(calc.)	0.979
st.dev.(Horwitz)	0.7209
R(Horwitz)	2.018
compare	
R(ISO22854-A:16)	0.31

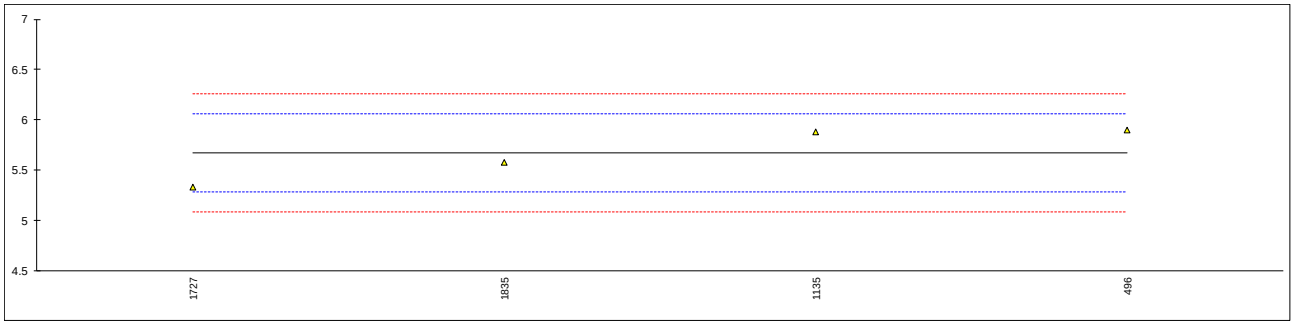




Determination of pHe on sample #20083; LiCl electrode

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334		-----		-----	
420		-----		-----	
463		-----		-----	
496	EN15490	5.90		1.17	
862		-----		-----	
1033		-----		-----	
1135	EN15490	5.88		1.07	
1459		-----		-----	
1727	EN15490	5.33		-1.76	
1835	EN15490	5.58		-0.48	
1982		-----		-----	
1984		-----		-----	
6262		-----		-----	

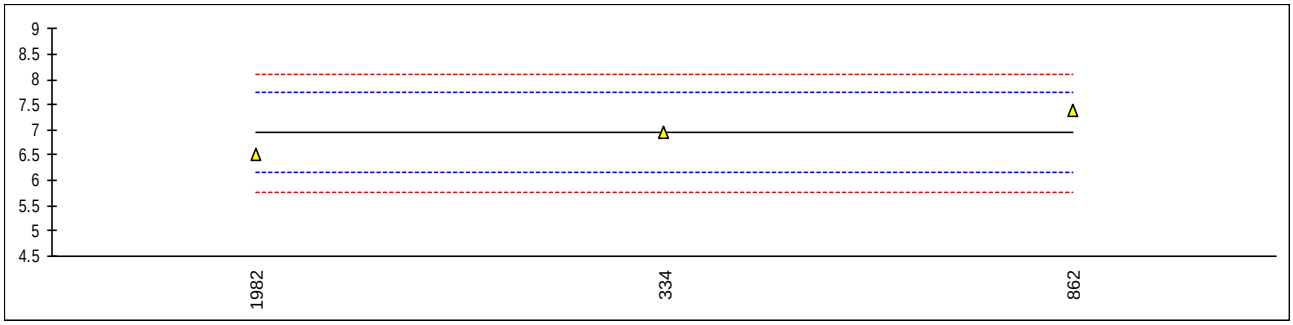
normality	unknown
n	4
outliers	0
mean (n)	5.673
st.dev. (n)	0.2712
R(calc.)	0.759
st.dev.(EN15490:07)	0.1945
R(EN15490:07)	0.545



Determination of pHe on sample #20083; KCl electrode

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	D6423	6.937	C	-0.02	reported as LiCl electrode
420		-----		-----	
463		-----		-----	
496		-----		-----	
862	D6423	7.40		1.16	
1033		-----		-----	
1135		-----		-----	
1459		-----		-----	
1727		-----		-----	
1835		-----		-----	
1982	D6423	6.5	C	-1.14	reported as LiCl electrode
1984		-----		-----	
6262		-----		-----	

normality	unknown
n	3
outliers	0
mean (n)	6.946
st.dev. (n)	0.4501
R(calc.)	1.260
st.dev.(D6423:18)	0.3914
R(D6423:18)	1.096



Determination of Phosphorus as P on sample #20083; results in mg/L

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	EN15487	<0.15		-----	
420	EN15837	<0,05		-----	
463		-----		-----	
496	EN15487	0.005		-----	
862	D3231	<0.2		-----	
1033		-----		-----	
1135	UOP389	<0.5		-----	
1459		-----		-----	
1727	EN15487	<0,15		-----	
1835	EN15837	<0.15		-----	
1982		-----		-----	
1984		-----		-----	
6262		-----		-----	
	n	6			
	mean (n)	<0.2			application range EN15487:07: 0.15 – 1.5 mg/L

Determination of Sulfate as SO_4 on sample #20083; results in mg/kg

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	EN15492	0.9		-----	
420		-----		-----	
463		-----		-----	
496	EN15492	0.23		-----	
862	EN15492	<1		-----	
1033		-----		-----	
1135		-----		-----	
1459		-----		-----	
1727	EN15492	0.26		-----	
1835	EN15492	<1.0		-----	
1982		-----		-----	
1984		-----		-----	
6262		-----		-----	
	n	5			
	mean (n)	<1			application range EN15492:12: 1 – 20 mg/kg

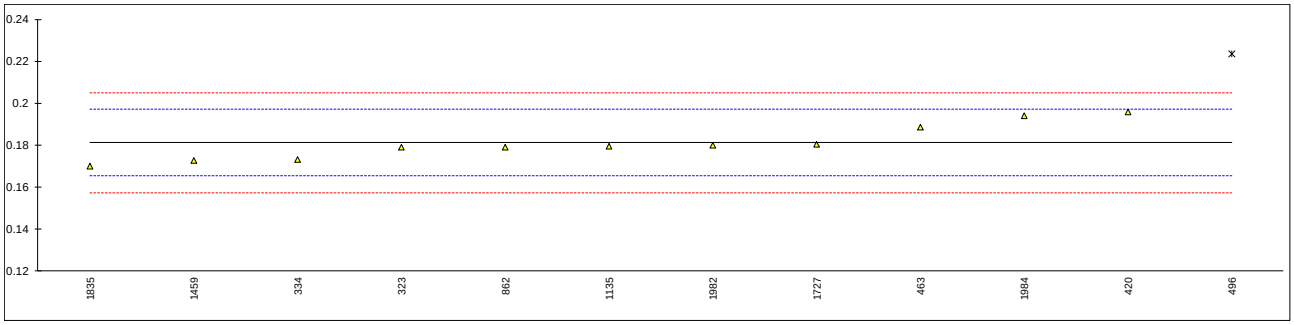
Determination of Sulfur on sample #20083; results in mg/kg

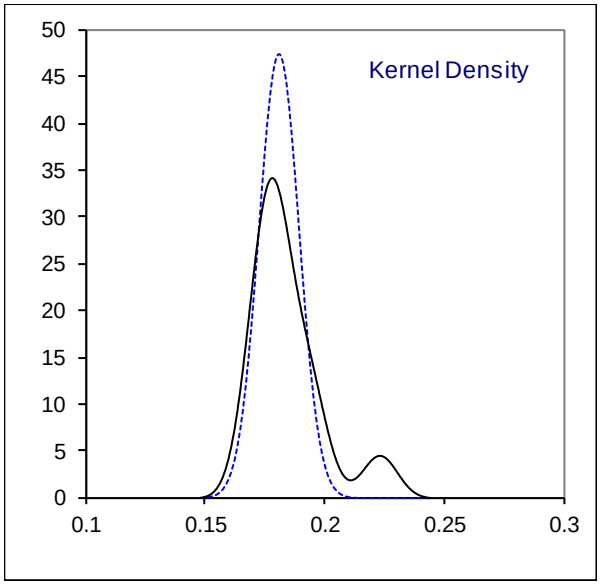
lab	method	value	mark	z(targ)	remarks
323	EN16997	<3.0		-----	
334	ISO20846	0.6		-----	
420	EN15486	0.65		-----	
463	ISO20846	<3		-----	
496	ISO20846	0.73		-----	
862	D5453	1.0		-----	
1033		-----		-----	
1135	ISO20846	0.9		-----	
1459	In house	< 1		-----	
1727		-----		-----	
1835	EN15486	<1.0		-----	
1982	D5453	0.7		-----	
1984	NFM 07 059	1		-----	
6262		-----		-----	
	normality	11			
	mean (n)	<5			application range EN16997:17: 5 – 20 mg/kg

Determination of Water on sample #20083; results in %M/M

lab	method	value	mark	z(targ)	remarks
323	EN15489	0.1790		-0.27	
334	EN15489	0.1733		-0.99	
420	EN15489	0.196		1.87	
463	ISO12937	0.18846		0.92	
496	EN15489	0.2235	D(0.05)	5.34	
862	D6304	0.179		-0.27	
1033		-----		-----	
1135	ISO12937	0.1797		-0.18	
1459	ISO12937	0.17265		-1.07	
1727	EN15489	0.1804		-0.09	
1835	EN15489	0.1701		-1.39	
1982	E1064	0.1800		-0.14	
1984	E1064	0.194		1.62	
6262		-----		-----	

normality	OK
n	11
outliers	1
mean (n)	0.1811
st.dev. (n)	0.00842
R(calc.)	0.0236
st.dev.(EN15489:07)	0.00793
R(EN15489:07)	0.0222





APPENDIX 2

Number of participants per country

3 labs in	BELGIUM
1 lab in	CHINA, People's Republic
1 lab in	CZECH REPUBLIC
3 labs in	FRANCE
2 labs in	GERMANY
2 labs in	SPAIN
1 lab in	SWEDEN
1 lab in	UNITED KINGDOM

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
fr.	= first reported
SDS	= Safety Data Sheet

Literature

1. iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2. Horwitz, R. Albert, J. AOAC Int, 79, 3, 589, (1996)
3. ASTM E178:08
4. ASTM E1301:03
5. ISO5725:86
6. ISO5725, parts 1-6:94
7. ISO13528:05
8. M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
9. W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
10. IP367:84
11. DIN38402 T41/42
12. P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
13. J.N. Miller, Analyst, 118, 455, (1993)
14. Analytical Methods Committee, Technical Brief, No. 4, January 2001
15. P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst 2002, 127, 1359-1364 (2002)
16. EN15293:11
17. Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)
18. M.A. Gonçalves et al., Evaluation study of different glass electrodes by an interlaboratory comparison for determining the pH of fuel ethanol, Sensors and Actuators, B 158, 327-332 (2011)