Institute for
Interlaboratory Studies

Results of Proficiency Test
Biogasoline E85
June 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. C.M. Nijssen-Wester
Correctors: ing. A.S. Noordman-de Neef & ing. G.A. Oosterlaken-Buijs
Approved by: ing. A.S. Noordman-de Neef

Report: iis22B05

September 2022

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1 INTRODUCTION

Since 2010 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Biogasoline E85 based on the latest version of EN15293 and ASTM D5798 every year. During the annual proficiency testing program 2021/2022 it was decided to continue the round robin for the analysis of Biogasoline E85.

In this interlaboratory study 14 laboratories in 10 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 analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send one sample Biogasoline E85 in a 1 L amber glass bottle labelled #22093.
The 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

A batch of approximately 30 liters of Biogasoline E85 was obtained from a local supplier. After homogenization 28 amber glass bottles of 1 L were filled and labelled #22093.
The homogeneity of the subsamples was checked by determination of Density at 15 °C in accordance with ISO12185 on 4 stratified randomly selected subsamples.

	Density at 15 °C in kg/m^3
sample #22093-1	784.78
sample #22093-2	784.77
sample #22093-3	784.80
sample #22093-4	784.78

Table 1: homogeneity test results of subsamples #22093

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)	0.04
reference test method	ISO12185:96
0.3 x R (reference test method)	0.45

Table 2: evaluation of the repeatability of subsamples #22093

The calculated repeatability is 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 sample of Biogasoline E85 labelled #22093 was sent on May 4, 2022. 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: Total Acidity as Acetic Acid, Appearance, Inorganic Chloride as Cl, Copper Corrosion 3 hrs 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 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 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 (derived from e.g. ISO or ASTM test methods), 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

In this proficiency test no problems were encountered with the dispatch of the samples. Two participants did not report any test results and all other participants reported test results before the final reporting date. Not all participants were able to report all tests requested. In total 12 participants reported 84 numerical test results. Observed was 1 outlying test result, which is 1.2%. 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 in appendix 1. 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 Total Oxygen content. For this test, the calculated reproducibility was compared against the estimated reproducibility calculated with 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 EN15491:21.

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. The reporting participants agreed on a value near or below the application range of the reference test method. 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 at 25 °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 EN15938:10.

Gum (solvent washed): This determination was not problematic. The reporting participants agreed on a test result of less than or equal to 1 mg/100mL. Therefore, no z-scores are calculated.

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

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

Ethanol: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ISO22854-B:21.

Ethers: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ISO22854-B:21.

Higher saturated (C3-C5) mono-alcohols: This determination was not problematic. The reporting participants agreed on a value below the application range of the reference test method. Therefore, no z-scores are calculated.

Total Oxygen content: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation.

pHe, LiCl electrode: Only three test results were reported, however these three test results were within in the reproducibility of test method EN15490:07. Therefore, the z-scores are calculated for indication only.

pHe, KCl electrode: Only three test results were reported, however these three test results were within in the reproducibility of test method ASTM D6423:20a. Therefore, the z-scores are calculated for indication only.

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

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

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in 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 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 reference methods are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Acidity as Acetic Acid	%M/M	6	0.0018	0.0007	0.0014
Appearance		8	C&B	n.a.	n.a.
Inorganic Chloride as Cl	mg/kg	7	1.0	0.5	0.6
Copper Corrosion 3 hrs 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	784.87	0.30	1.5

Parameter	unit	n	average	2.8 * sd	R(lit)
Electrical Conductivity at 25 °C	µS/cm	7	1.65	0.20	0.27
Gum (solvent washed)	mg/100mL	5	≤1	n.e.	n.e.
Oxidation Stability	minutes	5	>360	n.e.	n.e.
Methanol	%V/V	8	≤0.5	n.e.	n.e.
Ethanol	%V/V	9	84.01	2.76	4.85
Ethers	%V/V	4	1.61	0.35	0.33
Higher saturated mono-alcohols	%V/V	4	<0.1	n.e.	n.e.
Total Oxygen content	%M/M	7	29.7	1.2	2.0
pHe LiCl electrode		3	5.84	0.43	0.56
pHe KCl electrode		3	6.77	0.56	1.10
Phosphorus as P	mg/L	5	<0.15	n.e.	n.e.
Sulfate as SO4	mg/kg	6	<1	n.e.	n.e.
Sulfur	mg/kg	8	<3	n.e.	n.e.
Water	%M/M	11	0.189	0.019	0.022

Table 3: reproducibilities of tests on sample #22093

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 tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF JUNE 2022 WITH PREVIOUS PTS

	June 2022	June 2021	June 2020	May 2019	May 2018
Number of reporting laboratories	12	11	12	14	13
Number of test results	84	78	98	115	77
Number of statistical outliers	1	3	4	9	7
Percentage of statistical outliers	1.2%	3.8%	4.1%	7.8%	9.1%

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.

Parameter	June 2022	June 2021	June 2020	May 2019	May 2018
Total Acidity as Acetic Acid	++	++	+	++	+
Inorganic Chloride as Cl	+/-	-	+	--	-
Density at 15 °C	++	++	++	++	++
Electrical Conductivity at 25 °C	+	++	-	-	+
Gum (solvent washed)	n.e.	n.e.	++	++	++
Ethanol	+	++	++	++	+
Ethers	+/-	+	+	+	--

Parameter	June 2022	June 2021	June 2020	May 2019	May 2018
Total Oxygen content	+	++	++	-	+
pHe LiCl electrode	+	-	-	+	++
pHe KCl electrode	+	+	-	+/-	++
Sulfur	n.e.	++	n.e.	n.e.	n.e.
Water	+	-	+/-	+	++

Table 5: comparison of determinations to the reference test methods

The following performance categories were used:

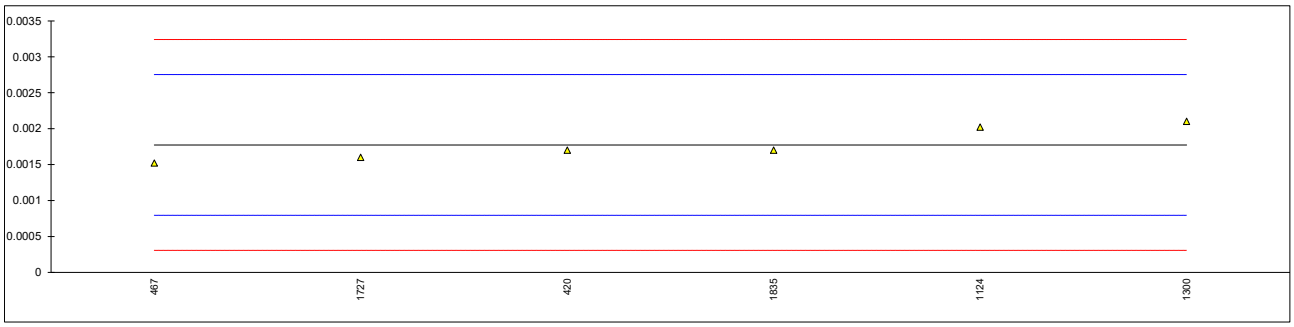
++ : group performed much better than the reference test method
+ : group performed better than the reference test method
+/- : group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.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

APPENDIX 1

Determination of Total Acidity as Acetic Acid on sample #22093; results in %M/M

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	D1613	<0.003		-----	
420	EN15491	0.0017		-0.15	
467	EN15491	0.00152		-0.52	
496		-----		-----	
862		-----		-----	
1033		-----		-----	
1124	EN15491	0.00202		0.50	
1299		-----		-----	
1300	EN15491	0.0021		0.67	
1459		-----		-----	
1727	EN15491	0.0016		-0.35	
1835	EN15491	0.0017		-0.15	
1984		-----		-----	

normality	unknown
n	6
outliers	0
mean (n)	0.00177
st.dev. (n)	0.000233
R(calc.)	0.00065
st.dev.(EN15491:21)	0.000489
R(EN15491:21)	0.00137



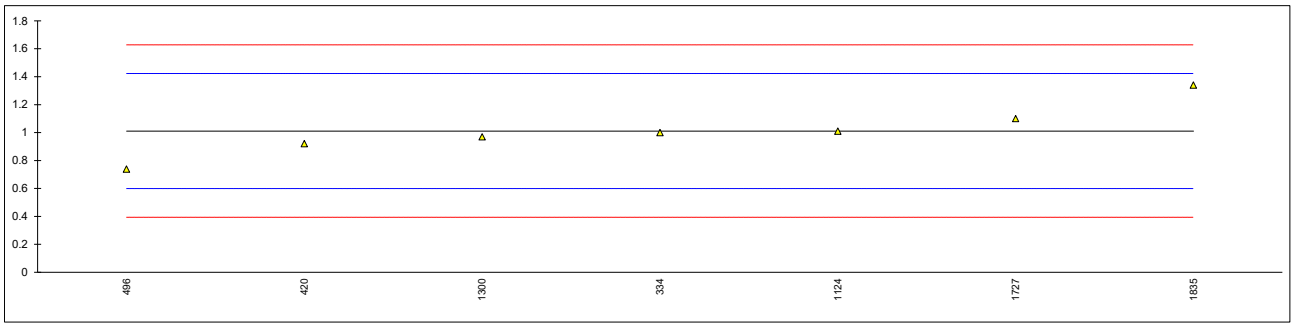
Determination of Appearance on sample #22093;

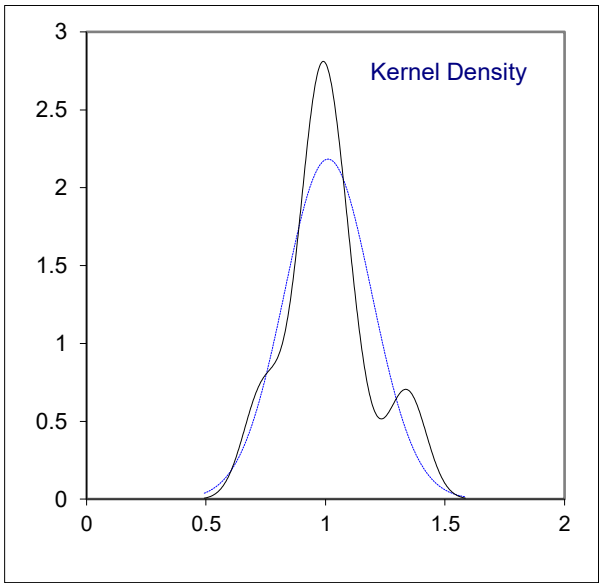
lab	method	value	mark	z(targ)	remarks
323	Visual	CBL		-----	
334	Visual	clear & bright		-----	
420	EN15769	clear, colourless		-----	
467		-----		-----	
496	Visual	clear & bright		-----	
862		-----		-----	
1033		-----		-----	
1124	EN15769	clear&bright		-----	
1299	Visual	Cl&Br		-----	
1300	EN15769	C&B		-----	
1459		-----		-----	
1727		-----		-----	
1835	EN15769	C&C		-----	
1984		-----		-----	
	n	8			
	mean (n)	Clear & Bright (C&B)			

Determination of Inorganic Chloride as Cl on sample #22093; results in mg/kg

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	EN15492	1.0		-0.05	
420	EN15484	0.92		-0.44	
467		-----		-----	
496	EN15492	0.739		-1.32	
862		-----		-----	
1033		-----		-----	
1124	EN15492	1.01		-0.01	
1299		-----		-----	
1300	EN15492	0.9690		-0.20	
1459		-----		-----	
1727	EN15492	1.1		0.43	
1835	EN15492	1.34		1.60	
1984		-----		-----	

normality	unknown
n	7
outliers	0
mean (n)	1.011
st.dev. (n)	0.1827
R(calc.)	0.512
st.dev.(EN15492:12)	0.2057
R(EN15492:12)	0.576





Determination of Copper Corrosion 3 hrs at 50 °C on sample #22093; rating

lab	method	value	mark	z(targ)	remarks
323	ISO2160	1A		-----	
334	D130	1a		-----	
420	ISO2160	1a		-----	
467	ISO2160	1a		-----	
496		-----		-----	
862		-----		-----	
1033		-----		-----	
1124	ISO2160	1a		-----	
1299	D130	1A		-----	
1300	ISO2160	1A		-----	
1459		-----		-----	
1727		-----		-----	
1835		-----		-----	
1984		-----		-----	
	n	7			
	mean (n)	1 (1a)			

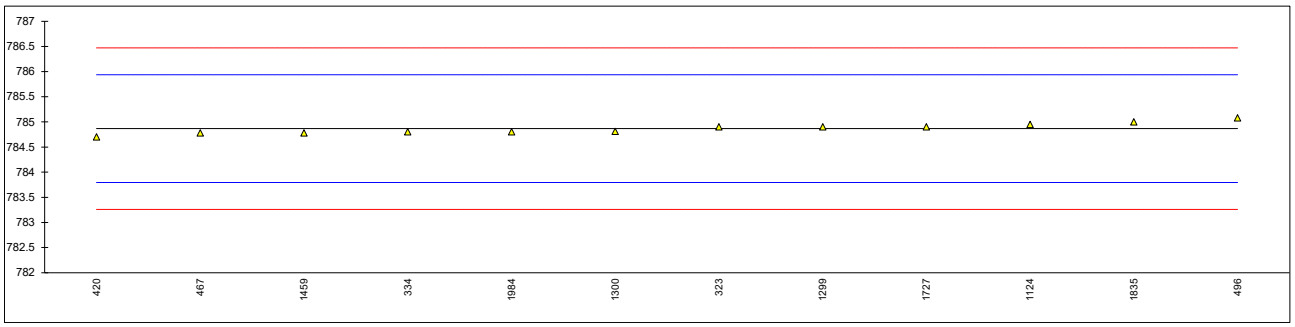
Determination of Copper as Cu on sample #22093; results in mg/kg

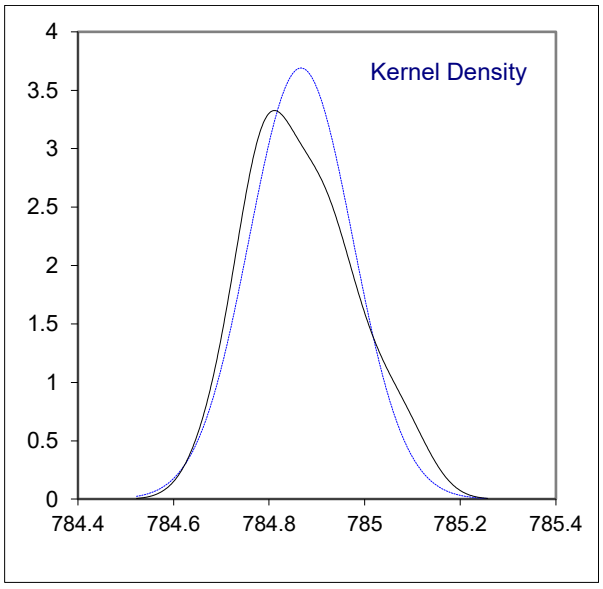
lab	method	value	mark	z(targ)	remarks
323	EN15488	< 0.070		-----	
334		-----		-----	
420	EN15837	<0,05		-----	
467		-----		-----	
496		-----		-----	
862		-----		-----	
1033		-----		-----	
1124	EN15488	<0.07		-----	
1299		-----		-----	
1300	EN15837	<0.05		-----	
1459		-----		-----	
1727		-----		-----	
1835	EN15837	0.06		-----	
1984		-----		-----	
	n	5			
	mean (n)	<0.07			Application range EN15488:07: 0.07 – 0.20 mg/kg

Determination of Density at 15 °C on sample #22093; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
323	ISO12185	784.9		0.06	
334	ISO12185	784.8		-0.12	
420	ISO12185	784.7		-0.31	
467	ISO12185	784.78		-0.16	
496	ISO12185	785.08		0.40	
862		-----		-----	
1033		-----		-----	
1124	ISO12185	784.95		0.16	
1299	D4052	784.9		0.06	
1300	ISO12185	784.81		-0.11	
1459	ISO12185	784.78		-0.16	
1727	ISO12185	784.9		0.06	
1835	ISO12185	785.0		0.25	
1984	ISO12185	784.8		-0.12	

normality	OK
n	12
outliers	0
mean (n)	784.867
st.dev. (n)	0.1081
R(calc.)	0.303
st.dev.(ISO12185:96)	0.5357
R(ISO12185:96)	1.5

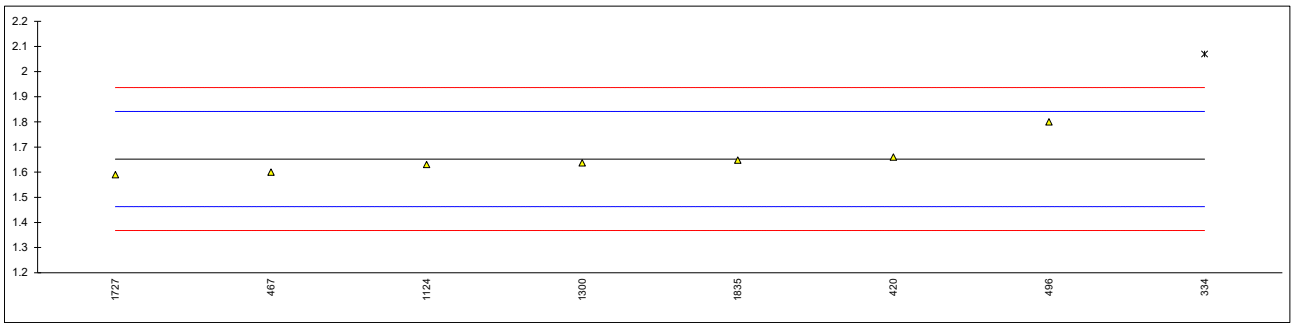


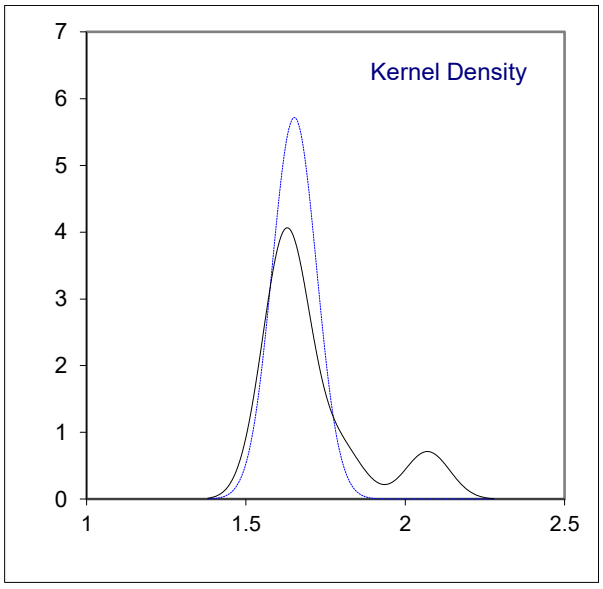


Determination of Electrical Conductivity at 25 °C on sample #22093; results in µS/cm

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	EN15938	2.07	C,G(0.05)	4.41	first reported: 1.06
420	EN15938	1.66		0.08	
467	EN15938	1.60		-0.55	
496	EN15938	1.8000		1.56	
862		-----		-----	
1033		-----		-----	
1124	EN15938	1.63		-0.23	
1299		-----		-----	
1300	EN15938	1.637		-0.16	
1459		-----		-----	
1727	EN15938	1.59		-0.66	
1835	EN15938	1.648		-0.04	
1984		-----		-----	

normality	unknown
n	7
outliers	1
mean (n)	1.652
st.dev. (n)	0.0698
R(calc.)	0.195
st.dev.(EN15938:10)	0.0947
R(EN15938:10)	0.265





Determination of Gum (solvent washed) on sample #22093; results in mg/100mL

lab	method	value	mark	z(targ)	remarks
323	ISO6246	< 0.5		-----	
334	ISO6246	<0.5		-----	
420	ISO6246	0.4		-----	
467		-----		-----	
496		-----		-----	
862		-----		-----	
1033		-----		-----	
1124		-----		-----	
1299	D381	1.0		-----	
1300	ISO6246	0.6		-----	
1459		-----		-----	
1727		-----		-----	
1835		-----		-----	
1984		-----		-----	
	n	5			
	mean (n)	≤1			

Determination of Oxidation Stability on sample #22093; results in minutes

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	ISO7536	>900		-----	
420	ISO7536	>600		-----	
467		-----		-----	
496		-----		-----	
862		-----		-----	
1033		-----		-----	
1124	ISO7536	>900		-----	
1299	D525	>900		-----	
1300	ISO7536	>900		-----	
1459		-----		-----	
1727		-----		-----	
1835		-----		-----	
1984		-----		-----	
	n	5			
	mean (n)	>360			

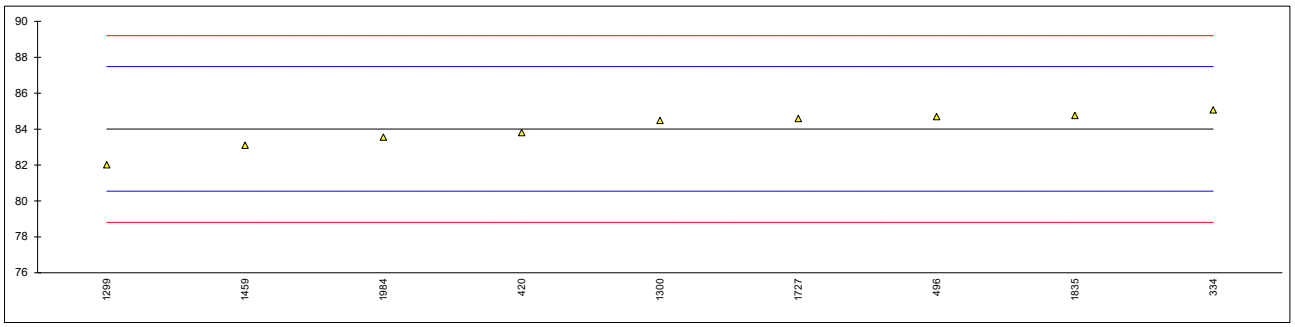
Determination of Methanol on sample #22093; results in %V/V

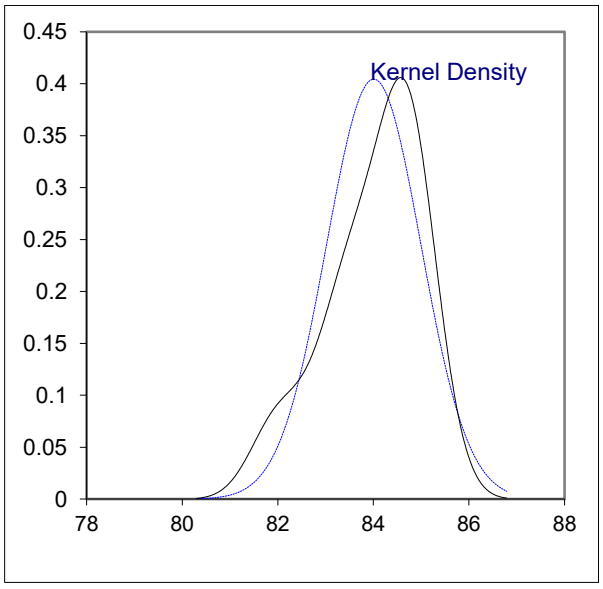
lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	ISO22854-B	<0.50		-----	
420	EN13132	<0,1		-----	
467		-----		-----	
496	ISO22854-B	<0.01		-----	
862		-----		-----	
1033		-----		-----	
1124	EN16761-1	< 0.1		-----	
1299	ISO22854-B	0.05		-----	
1300	EN16761-1	0.017		-----	
1459		-----		-----	
1727		-----		-----	
1835	In house	<0.01		-----	
1984	EN1601	<0.17		-----	
	n	8			
	mean (n)	≤0.5			Application range EN16761-1:15: 0.5 – 1.5% V/V

Determination of Ethanol on sample #22093; results in %V/V

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	ISO22854-B	85.07		0.61	
420	EN13132	83.8		-0.12	
467		-----		-----	
496	ISO22854-B	84.69		0.40	
862		-----		-----	
1033		-----		-----	
1124		-----		-----	
1299	ISO22854-B	82.01		-1.15	
1300	ISO22854-B	84.482		0.27	
1459	In house	83.1		-0.52	
1727	In house	84.59		0.34	
1835	In house	84.76		0.44	
1984	EN1601	83.55		-0.26	

normality	OK
n	9
outliers	0
mean (n)	84.006
st.dev. (n)	0.9869
R(calc.)	2.763
st.dev.(ISO22854-B:21)	1.7321
R(ISO22854-B:21)	4.85

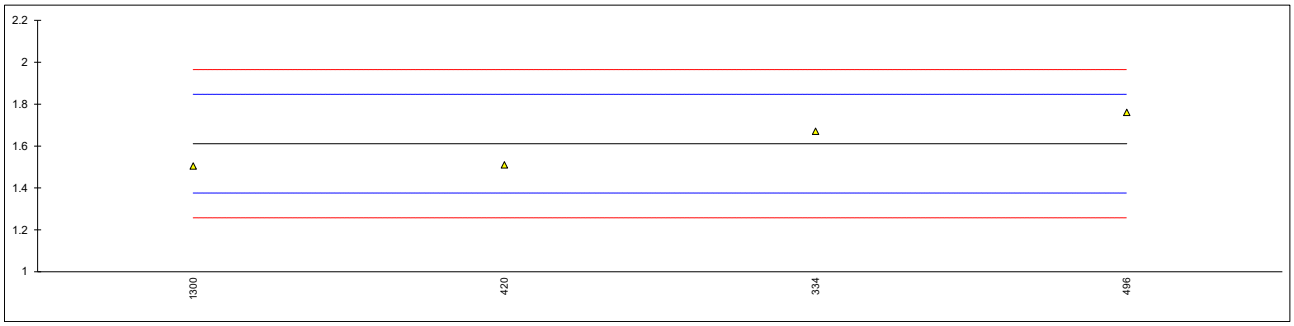




Determination of Ethers on sample #22093; results in %V/V

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	ISO22854-B	1.67		0.50	
420	EN13132	1.51		-0.86	
467		-----		-----	
496	ISO22854-B	1.76		1.26	
862		-----		-----	
1033		-----		-----	
1124		-----		-----	
1299		-----		-----	
1300	ISO22854-B	1.505		-0.90	
1459		-----		-----	
1727		-----		-----	
1835		-----		-----	
1984	EN1601	<0.17		<-12.23	Possibly a false negative test result?

normality	unknown
n	4
outliers	0
mean (n)	1.611
st.dev. (n)	0.1253
R(calc.)	0.351
st.dev.(ISO22854-B:21)	0.1179
R(ISO22854-B:21)	0.33



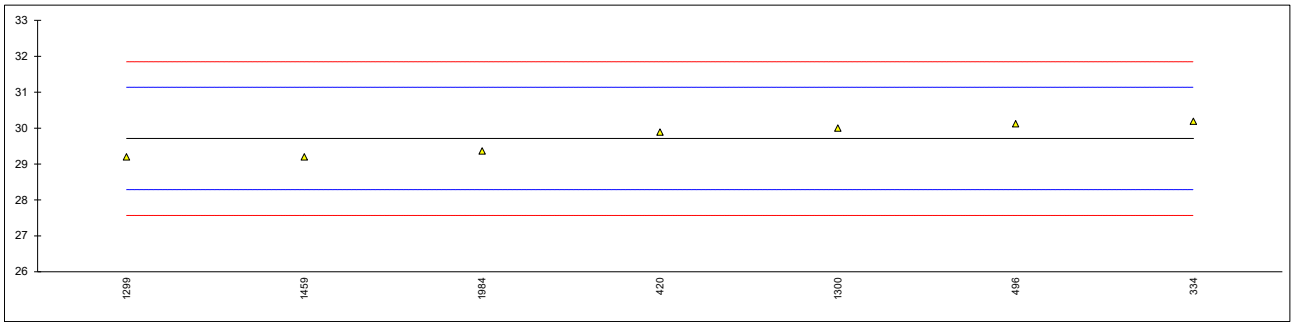
Determination of Higher saturated (C3-C5) mono-alcohols on sample #22093; results in %V/V

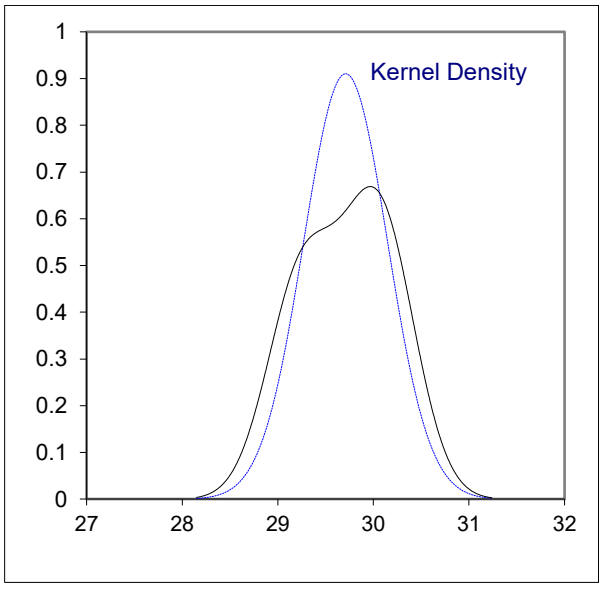
lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	ISO22854-B	0.04		-----	
420	EN13132	<0,1		-----	
467		-----		-----	
496	ISO22854-B	<0.01		-----	
862		-----		-----	
1033		-----		-----	
1124		-----		-----	
1299		-----		-----	
1300	ISO22854-B	0.075		-----	
1459		-----		-----	
1727		-----		-----	
1835		-----		-----	
1984		-----		-----	
	n	4			
	mean (n)	<0.1			Application range ISO22854-B:21: 1.4 - 2.5 % V/V

Determination of Total Oxygen content on sample #22093; results in %M/M

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	ISO22854-B	30.19		0.67	
420	EN13132	29.89		0.25	
467		-----		-----	
496	ISO22854-B	30.12		0.58	
862		-----		-----	
1033		-----		-----	
1124		-----		-----	
1299	ISO22854-B	29.20		-0.71	
1300	ISO22854-B	29.996		0.40	
1459	In house	29.2		-0.71	
1727		-----		-----	
1835		-----		-----	
1984	EN1601	29.365		-0.48	

normality	unknown
n	7
outliers	0
mean (n)	29.709
st.dev. (n)	0.4382
R(calc.)	1.227
st.dev.(Horwitz)	0.7133
R(Horwitz)	1.997

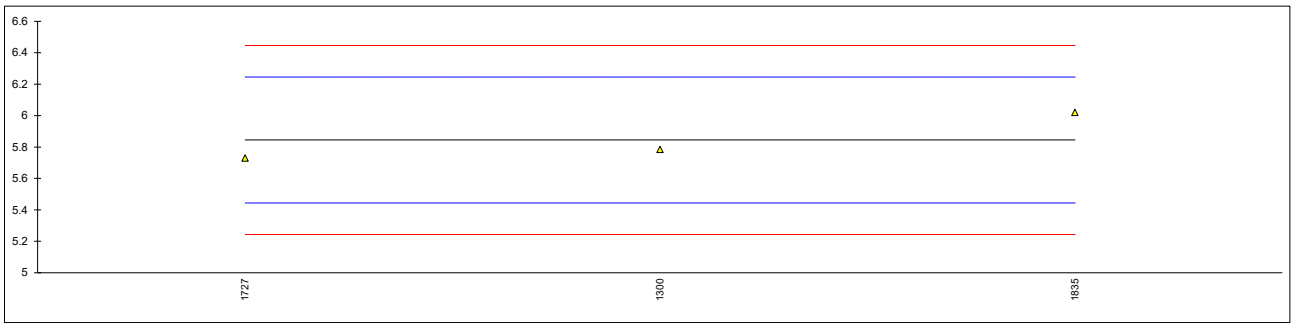




Determination of pHe with LiCl electrode on sample #22093;

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334		-----		-----	
420		-----		-----	
467		-----		-----	
496		-----		-----	
862		-----		-----	
1033		-----		-----	
1124		-----		-----	
1299		-----		-----	
1300	EN15490	5.784		-0.30	
1459		-----		-----	
1727	EN15490	5.73		-0.57	
1835	EN15490	6.02		0.87	
1984		-----		-----	

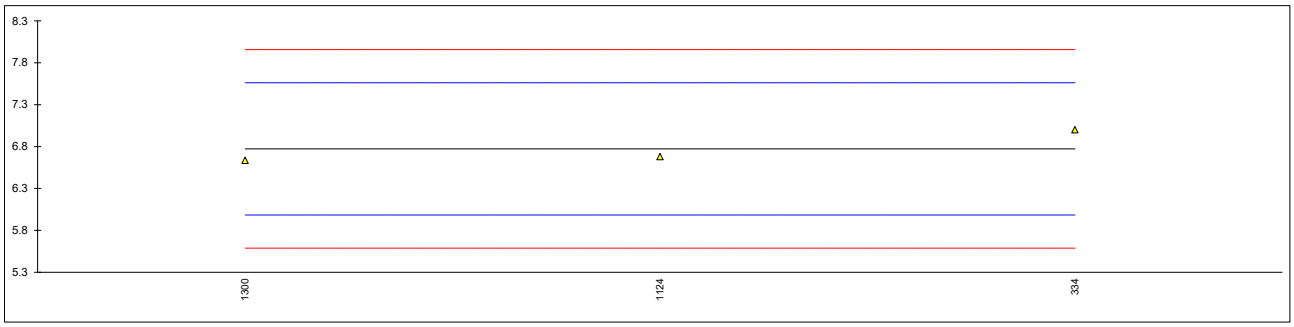
normality	unknown
n	3
outliers	0
mean (n)	5.845
st.dev. (n)	0.1542
R(calc.)	0.432
st.dev.(EN15490:07)	0.204
R(EN15490:07)	0.561



Determination of pHe with KCl electrode on sample #22093;

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	D6423	7.0		0.58	
420		-----		-----	
467		-----		-----	
496		-----		-----	
862		-----		-----	
1033		-----		-----	
1124	D6423	6.68		-0.23	
1299		-----		-----	
1300	D6423	6.637		-0.34	
1459		-----		-----	
1727		-----		-----	
1835		-----		-----	
1984		-----		-----	

normality	unknown
n	3
outliers	0
mean (n)	6.772
st.dev. (n)	0.1983
R(calc.)	0.555
st.dev.(D6423:20a)	0.3946
R(D6423:20a)	1.105



Determination of Phosphorus as P on sample #22093; results in mg/L

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334		-----		-----	
420	EN15837	<0,05		-----	
467		-----		-----	
496		-----		-----	
862		-----		-----	
1033		-----		-----	
1124	EN15487	<0.15		-----	
1299		-----		-----	
1300	EN15487	0.029		-----	
1459		-----		-----	
1727	EN15487	<0,13		-----	
1835	EN15837	<0.13		-----	
1984		-----		-----	
	n	5			
	mean (n)	<0.15			Application range EN15487:07: 0.15 – 1.5 mg/L

Determination of Sulfate as SO_4 on sample #22093; results in mg/kg

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	EN15492	<1.0		-----	
420		-----		-----	
467		-----		-----	
496	EN15492	0.152		-----	
862		-----		-----	
1033		-----		-----	
1124	EN15492	<1		-----	
1299		-----		-----	
1300	EN15492	0.104		-----	
1459		-----		-----	
1727	EN15492	<1		-----	
1835	EN15492	<1.0		-----	
1984		-----		-----	
	n	6			
	mean (n)	<1			application range EN15492:12: 1 – 20 mg/kg

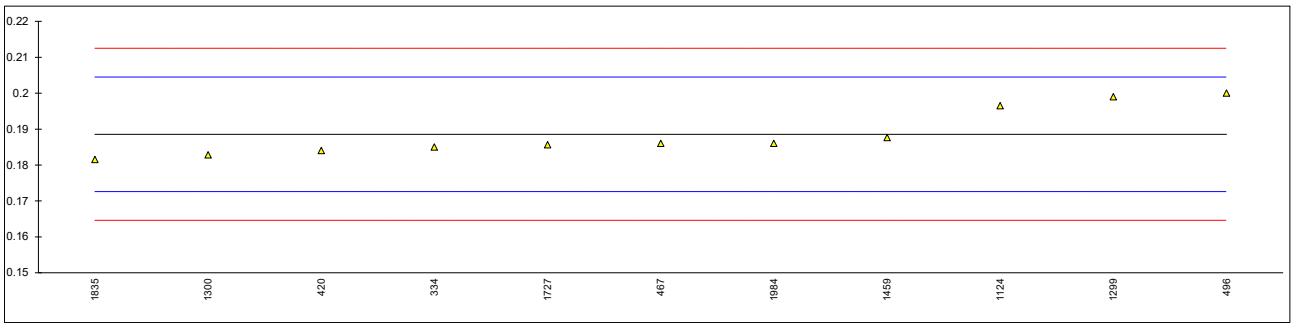
Determination of Sulfur on sample #22093; results in mg/kg

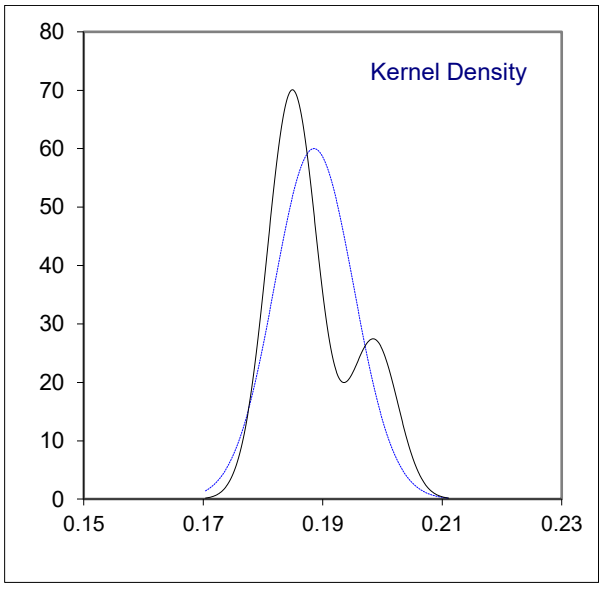
lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	ISO20846	<3		-----	
420	EN15486	1.24		-----	
467	ISO20846	<1		-----	
496		-----		-----	
862		-----		-----	
1033		-----		-----	
1124	EN15486	<2.0		-----	
1299		-----		-----	
1300	EN16997	0.8		-----	
1459	ISO20884	<0,5		-----	
1727		-----		-----	
1835	EN15486	<2.0		-----	
1984	ISO20846	<3		-----	
	n	8			
	mean (n)	<3			application range EN16997:17: 5 – 20 mg/kg

Determination of Water on sample #22093; results in %M/M

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	EN15489	0.185		-0.44	
420	EN15489	0.184		-0.57	
467	ISO12937	0.1860		-0.32	
496	EN15489	0.200		1.43	
862		-----		-----	
1033		-----		-----	
1124	EN15489	0.1965		1.00	
1299	ISO12937	0.199		1.31	
1300	EN15489	0.18284		-0.72	
1459	ISO12937	0.1876		-0.12	
1727	EN15489	0.1856		-0.37	
1835	EN15489	0.1815		-0.88	
1984	ISO12937	0.186		-0.32	

normality	suspect
n	11
outliers	0
mean (n)	0.1885
st.dev. (n)	0.00665
R(calc.)	0.0186
st.dev.(EN15489:07)	0.00798
R(EN15489:07)	0.0223





APPENDIX 2

Number of participants per country

1 lab in BELGIUM

1 lab in CHINA, People's Republic

1 lab in CZECH REPUBLIC

1 lab in ESTONIA

3 labs in FRANCE

1 lab in GERMANY

1 lab in LATVIA

3 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	= 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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6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
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12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)