Results of Proficiency Test
Biogasoline E85
May 2018

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis18B03

August 2018

CONTENTS

1 INTRODUCTION

Since 2010, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Biogasoline E85. During the annual proficiency testing program 2017/2018, it was decided to continue the round robin for the analysis of Biogasoline E85 in accordance with the latest applicable version of the specification for ASTM D5798 and/of CEN/TS15293. In this interlaboratory study 13 laboratories in 9 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2018 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 organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one sample (1 litre) of Biogasoline E85.
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/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

The necessary sample material of 60 litres of Biogasoline E85 was purchased from a local supplier. After homogenisation, 38 brown glass bottles of 1 litre (labelled #18083) were filled. The homogeneity of the subsamples #18083 was checked by determination of Density at 15°C in accordance with ASTM D4052 on 8 stratified randomly selected samples.

	Density at 15°C in kg/m^3
Sample #18083-1	783.92
Sample #18083-2	783.97
Sample #18083-3	784.00
Sample #18083-4	783.94
Sample #18083-5	783.98
Sample #18083-6	783.99
Sample #18083-7	783.99
Sample #18083-8	783.99

Table 1: homogeneity test results of subsamples #18083

From the above test results, the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method which is in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density at 15°C in kg/m^3
r (observed)	0.08
reference test method	ISO12185:96
0.3 * R (reference test method)	0.45

Table 2: evaluation of repeatability of the subsamples #18083

The calculated repeatability is less than 0.3 times the reproducibility of the corresponding reference test method. Therefore, homogeneity of the subsamples #18083 was assumed.

One 1L bottle labelled #18083 was sent to each of the participating laboratories on May 2, 2018. A SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Biodiesel E85, packed in an amber glass bottle, was checked. The material was found to be sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were requested to determine on sample #18083: Total Acidity (as Acetic Acid), Chloride (Inorganic), Copper Corrosion, Copper, Density, Electrical Conductivity, Existent Gum (solvent washed), Oxidation Stability, Methanol, Ethanol and higher saturated alcohols, Ethers (5 or more C atoms), Higher saturated monoalcohols (C3-C5), Total Organically bound oxygen, pHe, Phosphorus, Sulphate, Sulphur 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 to round the test results, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment.
Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalysis). Additional or corrected test results are used for data analysis and 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 March 2017 (iis-protocol, version 3.4).

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 ISO 5725 the original test results per determination were submitted to Dixon's, 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.

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. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM, ISO or IP reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of 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 targets values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.
The z-scores were calculated according to:

$z_{(target)}$ = (test result - average of PT) / target standard deviation

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare.
The usual interpretation of z-scores is as follows:

	|z| < 1	good
1 <	|z| < 2	satisfactory
2 <	|z| < 3	questionable
3 <	|z|	unsatisfactory

4 EVALUATION

No problems were encountered during the execution of this proficiency test. All laboratories reported test results. Not all laboratories were able to perform all requested analyses. Finally, 13 laboratories did report 77 numerical test results. Observed were 7 outlying test results, which is 9.1%. In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

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

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.

Total Acidity: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of EN15491:07.

Chloride, Inorganic: This determination may be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of EN15492:12. The low number of reported test results may (partly) explain the large variation.

Copper corrosion: No problems have been observed. All reporting participants agreed on a test result of 1 (1a).

Copper as Cu: No significant conclusions were drawn. Only three “less than” test results were reported.

Density at 15°C: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ISO12185:96.

Electrical Conductivity: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN15938:10.

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

Oxidation Stab.: In this determination, no problems have been observed. All reporting participants agreed on a test result above 360 minutes according to specification EN15293:2011.

Methanol: Only two participants reported a numerical test result, all other laboratories reported a less than test result. Therefore, no significant conclusions were drawn.

Ethanol and higher saturated alcohols: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of EN1601:14.

Ethers (5 or more C atoms): This determination may be problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of EN1601:14. The low number of reported test results may (partly) explain this larger variation.

Higher sat. alcohols: Only five participants reported a test result for higher saturated alcohols. Four of them reported a less than test result. Therefore, no significant conclusions were drawn.

Tot. org. bound oxygen: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN1601:14.

pHe: This determination may be problematic as two different electrodes were used (KCl and LiCl electrodes). Therefore, it was decided to split the test results based on the electrode used. It is known that electrodes with LiCl give significantly lower pHe values than other types of electrodes (e.g. KCl electrodes) (see lit.18)
The determination was not problematic for the laboratories that used a KCl electrode. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of D6423:14.
The determination was not problematic for the laboratories that used a LiCl electrode. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of EN15490:07.

Phosphorus: The reporting participants agreed on a value close or below the application range. Therefore, no significant conclusions were drawn.

Sulphate: The reporting participants agreed on a value close or below the application range. Therefore, no significant conclusions were drawn.

Sulphur: The reporting participants agreed on a value near or below the application range. Therefore, no significant conclusions were drawn

Water: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good 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 relevant reference test method and the reproducibility as found for the group of participating laboratories. The target reproducibilities derived from reference test methods (in casu ASTM, ISO, EN reference test methods) are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acidity as Acetic Acid, Total	%M/M	8	0.0014	0.0007	0.0014
Chloride, Inorganic	mg/kg	3	1.0	0.8	0.6
Copper Corrosion 3 hrs at 50°C		7	1a	n.a.	n.a.
Copper as Cu	mg/kg	3	<0.07	n.a.	n.a.
Density at 15°C	kg/m^3	11	784.1	0.3	1.5
Electrical Conductivity at 25°C	µS/cm	5	1.17	0.18	0.21
Existent Gum (washed)	mg/100mL	6	0.48	0.88	2.01
Oxidation Stability	minutes	7	>360	n.a.	n.a.
Methanol	%V/V	8	<0.2	n.a.	n.a.
Ethanol and higher saturated alcohols	%V/V	9	83.69	4.01	5.51
Ethers (5 or more C-atoms)	%V/V	5	1.58	0.49	0.22
Higher saturated mono alcohols	%V/V	5	<0.2	n.a.	n.a.
Total organically bound Oxygen	%M/M	7	29.7	1.8	2.8
pHe KCl		3	7.04	0.55	1.09
pHe LiCl		4	6.11	0.08	0.59
Phosphorus as P	mg/L	5	<0.2	n.a.	n.a.
Sulphate	mg/kg	5	<1	n.a.	n.a.
Sulphur as S	mg/kg	7	<1	n.a.	n.a.
Water	%M/M	9	0.171	0.010	0.022

Table 3: performance evaluation sample #18083

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

4.3 COMPARISON OF PROFICIENCY TEST OF MAY 2018 WITH PREVIOUS PTS

Determination	May 2018	May 2017	May 2016	May 2015	May 2014
Number of reporting labs	13	16	16	13	16
Number of test results reported	77	91	117	110	126
Statistical outliers	7	5	7	1	2
Percentage outliers	9.1%	5.5%	6.0%	0.9%	1.6%

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 the following table:

	May 2018	May 2017	May 2016	May 2015	May 2014
Acidity as Acetic Acid, Total	+	+	+	++	+
Chloride, Inorganic	-	--	++	(--)*	n.e.
Copper Corrosion 3 hrs at 50°C	n.e.	n.e.	n.e.	n.e.	++
Copper as Cu	n.e.	n.e.	n.e.	n.e.	n.e.
Density at 15°C	++	+/-	++	++	+
Electrical Conductivity at 25°C	+	+	-	+	+/-
Existent Gum (washed)	++	++	++	+	+
Oxidation Stability	n.e.	n.e.	n.e.	n.e.	n.e.
Methanol	n.e.	n.e.	-	n.e.	n.e.
Ethanol and higher saturated alcohols	+	++	+	-	--
Ethers (5 or more C-atoms)	--	--	+/-	--	n.e.
Higher saturated mono alcohols	n.e.	n.e.	n.e.	n.e.	n.e.
Total organically bound Oxygen	+	++	++	+/-	n.e.
pHe KCl	++	-	--	--	--
pHe LiCl	++				
Phosphorus as P	n.e.	n.e.	n.e.	n.e.	n.e.
Sulphate	n.e.	n.e.	(--)*	n.e.	n.e.
Sulphur as S	n.e.	++	++	++	++
Water	++	-	+	+	-

Table 5: comparison of the quality of the determinations against the reference test methods

*() = assigned value was near or below the detection limit

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

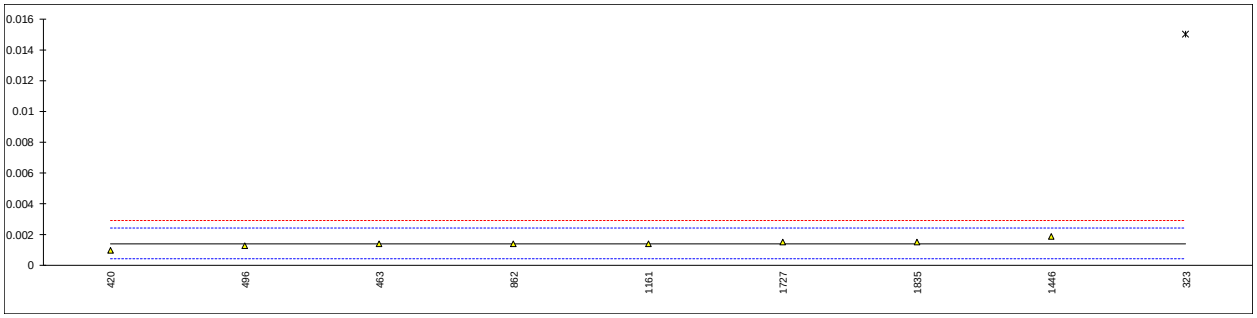
++: group performed much better than the reference test method
+ : group performed better than the reference test method
+/-: group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.: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

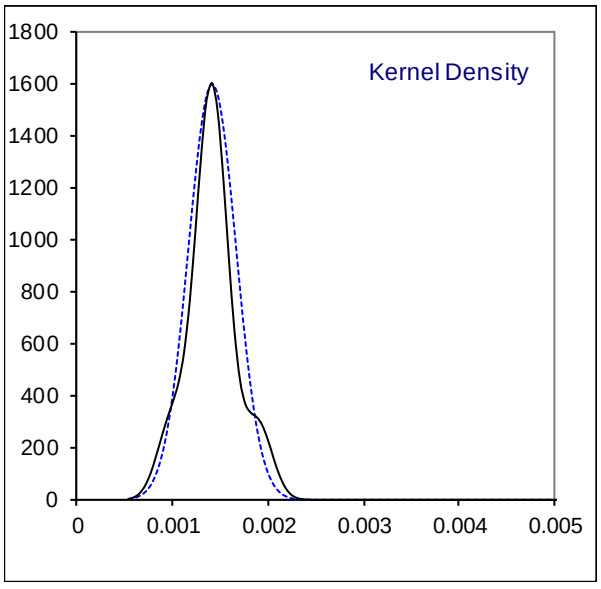
APPENDIX 1

Determination of Total Acidity as Acetic Acid on sample #18083; results in %M/M

lab	method	value	mark	z(targ)	remarks
323	EN15491	0.015	G(0.01)	27.74	
334		-----		-----	
420	EN15491	0.0010		-0.87	
447		-----		-----	
463	EN15491	0.0014		-0.05	
496	EN15491	0.0013		-0.26	
862	EN15491	0.0014		-0.05	
1161	EN15491	0.0014		-0.05	
1446	EN15491	0.0019		0.97	
1459		-----		-----	
1727	EN15491	0.0015		0.15	
1835	EN15491	0.0015		0.15	
1984		-----		-----	

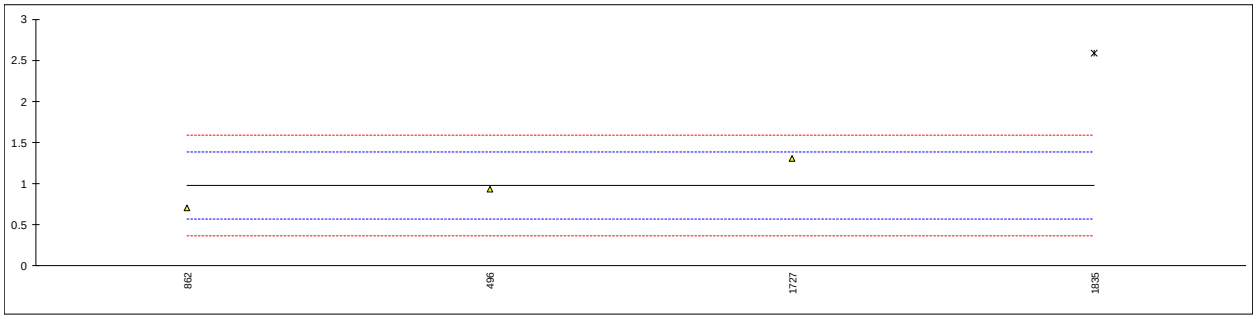
normality	not OK
n	8
outliers	1
mean (n)	0.00142
st.dev. (n)	0.000249
R(calc.)	0.00070
st.dev.(EN15491:07)	0.000489
R(EN15491:07)	0.00137





Determination of Inorganic Chloride on sample #18083; results in mg/kg

lab	method	value	mark	z(targ)	remarks
323	EN15492	<1		-----	
334		-----		-----	
420		-----		-----	
447		-----		-----	
463		-----		-----	
496	EN15492	0.93		-0.23	
862	EN15492	0.7		-1.35	
1161		-----		-----	
1446		-----		-----	
1459		-----		-----	
1727	EN15492	1.3		1.58	
1835	EN15492	2.59	G(0.05)	7.88	Analytical modifications written in EN15293 were used
1984		-----		-----	
	normality	unknown			
	n	3			
	outliers	1			
	mean (n)	0.977			
	st.dev. (n)	0.3027			
	R(calc.)	0.848			
	st.dev.(EN15492:12)	0.2047			
	R(EN15492:12)	0.573			Application range: 1 – 30 mg/kg



Determination of Copper Corrosion 3hrs at 50°C on sample #18083; rating

lab	method	value	mark	z(targ)	remarks
323	ISO2160	1A		-----	
334		-----		-----	
420	ISO2160	class 1a		-----	
447	D130	1a		-----	
463	ISO2160	1A		-----	
496	ISO2160	1a		-----	
862	D130	1a		-----	
1161	ISO2160	1a		-----	
1446		-----		-----	
1459		-----		-----	
1727		-----		-----	
1835		-----		-----	
1984		-----		-----	
	n	7			
	mean (n)	1a			

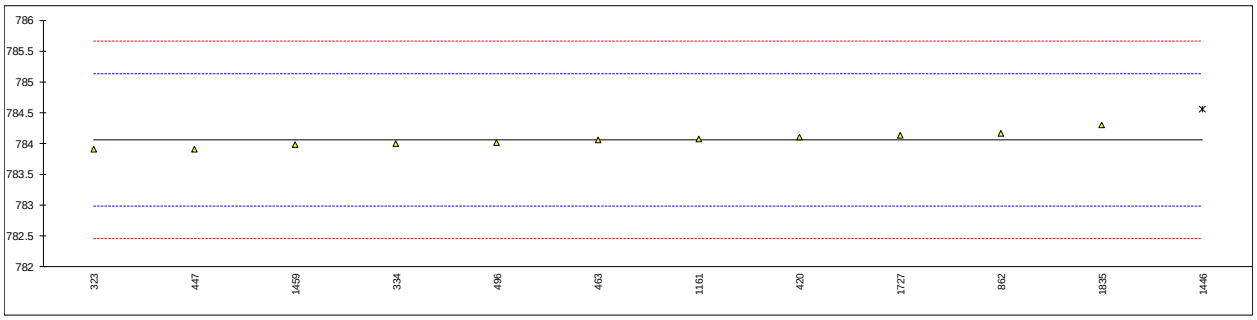
Determination of Copper as Cu on sample #18083; results in mg/kg

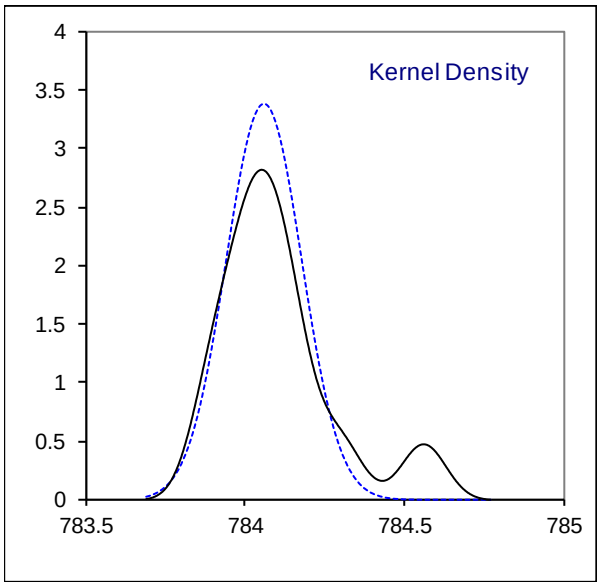
lab	method	value	mark	z(targ)	remarks
323	EN15488	<0.070		-----	
334		-----		-----	
420	EN15837	< 0,05		-----	
447		-----		-----	
463		-----		-----	
496		-----		-----	
862	EN15488	<0.07		-----	
1161		-----		-----	
1446		-----		-----	
1459		-----		-----	
1727		-----		-----	
1835		-----		-----	
1984		-----		-----	
	n	3			
	mean (n)	<0.07			

Determination of Density at 15°C on sample #18083; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
323	ISO12185	783.9		-0.30	
334	ISO12185	784		-0.11	
420	ISO12185	784.1		0.07	
447	D4052	783.9		-0.30	
463	ISO12185	784.06		0.00	
496	ISO12185	784.02		-0.07	
862	ISO12185	784.17		0.21	
1161	ISO12185	784.08		0.04	
1446	ISO12185	784.56	G(0.05)	0.93	
1459	ISO12185	783.99		-0.13	
1727	D4052	784.14		0.15	
1835	ISO12185	784.3		0.45	
1984		-----		-----	

normality	OK
n	11
outliers	1
mean (n)	784.060
st.dev. (n)	0.1181
R(calc.)	0.331
st.dev.(ISO12185:96)	0.5357
R(ISO12185:96)	1.5

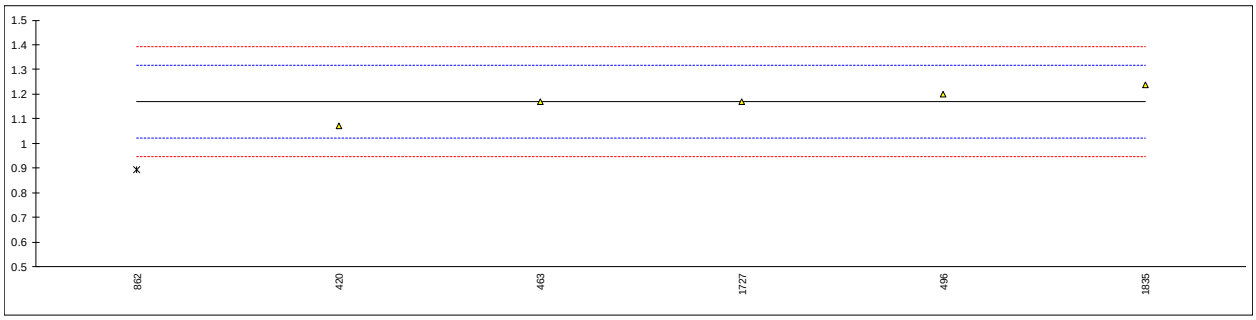




Determination of Electrical Conductivity at 25°C on sample #18083; results in µS/cm

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334		-----		-----	
420	EN15938	1.07		-1.34	
447		-----		-----	
463	EN15938	1.17		0.00	
496	EN15938	1.200		0.41	
862	EN15938	0.893	G(0.05)	-3.73	
1161		-----		-----	
1446		-----		-----	
1459		-----		-----	
1727	EN15938	1.17		0.00	
1835	EN15938	1.239		0.93	
1984		-----		-----	

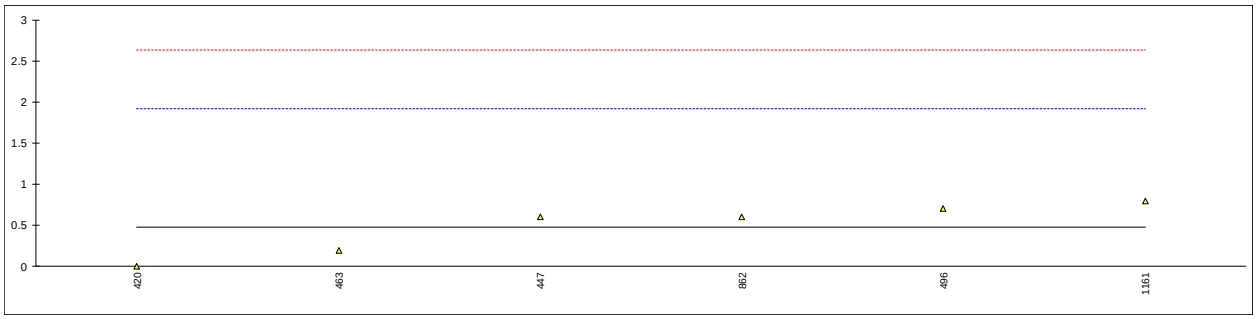
normality	unknown
n	5
outliers	1
mean (n)	1.170
st.dev. (n)	0.0626
R(calc.)	0.175
st.dev.(EN15938:10)	0.0742
R(EN15938:10)	0.208



Determination of Existent Gum (solvent washed) on sample #18083; results in mg/100ml

lab	method	value	mark	z(targ)	Remarks
323	ISO6246	<0.5		-----	
334		-----		-----	
420	ISO6246	0.0004		-0.67	Perhaps reported in a deviating unit (g/100ml)?
447	D381	0.6		0.16	
463	ISO6246	0.2		-0.40	
496	ISO6246	0.7		0.30	
862	D381	0.6		0.16	
1161	ISO6246	0.8		0.44	
1446		-----		-----	
1459		-----		-----	
1727		-----		-----	
1835		-----		-----	
1984		-----		-----	

normality	unknown
n	6
outliers	0
mean (n)	0.483
st.dev. (n)	0.3124
R(calc.)	0.875
st.dev.(ISO6246:17)	0.7162
R(ISO6246:17)	2.005



Determination of Oxidation Stability on sample #18083; results in minutes

lab	method	value	mark	z(targ)	remarks
323	ISO7536	>900		-----	
334		-----		-----	
420	ISO7536	> 600		-----	
447	D525	>900		-----	
463	ISO7536	>360		-----	
496	ISO7536	>360		-----	
862	D525	>480		-----	
1161	ISO7536	>900		-----	
1446		-----		-----	
1459		-----		-----	
1727		-----		-----	
1835		-----		-----	
1984		-----		-----	
	n	7			
	mean (n)	>360			

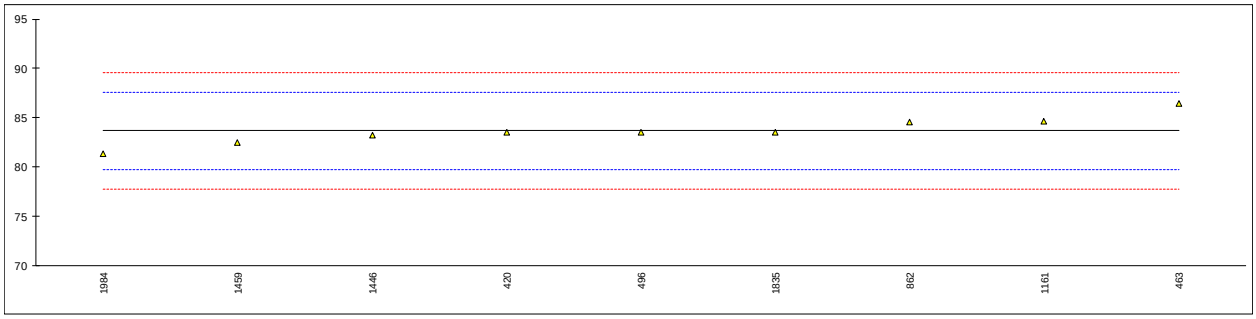
Determination of Methanol on sample #18083; results in %V/V

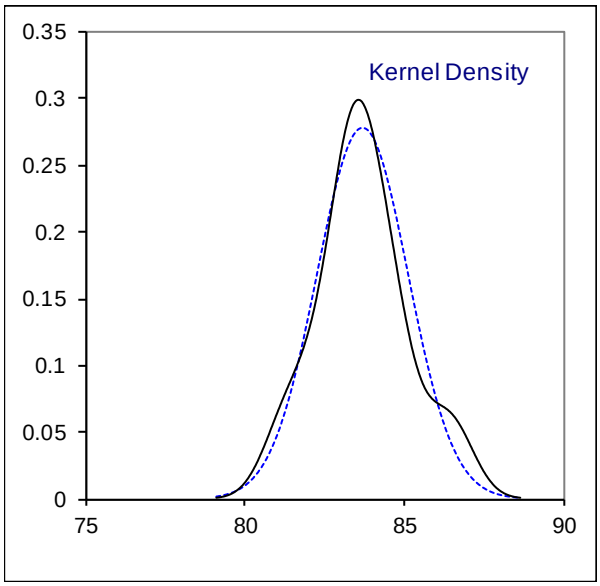
lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334		-----		-----	
420	EN13132	< 0,1		-----	
447		-----		-----	
463	EN13132	<0,2		-----	
496	EN1601	<0.01		-----	
862	D4815	<0.2		-----	
1161	ISO22854	<0,17		-----	
1446		-----		-----	
1459		-----		-----	
1727		0.005		-----	
1835	In house	0.005		-----	
1984		<0.17		-----	
	n	8			
	mean (n)	<0.2			

Determination of Ethanol and higher saturated alcohols on sample #18083; results in %V/V

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334		-----		-----	
420	EN13132	83.5		-0.10	
447		-----		-----	
463	EN13132	86.41		1.38	
496	EN1601	83.51		-0.09	
862	D4815	84.53		0.43	
1161	ISO22854	84.66		0.49	
1446		83.28		-0.21	
1459		82.45		-0.63	
1727		-----		-----	
1835	In house	83.54		-0.08	
1984		81.3333		-1.20	

normality	suspect
n	9
outliers	0
mean (n)	83.690
st.dev. (n)	1.4316
R(calc.)	4.009
st.dev.(EN1601:14)	1.9677
R(EN1601:14)	5.510

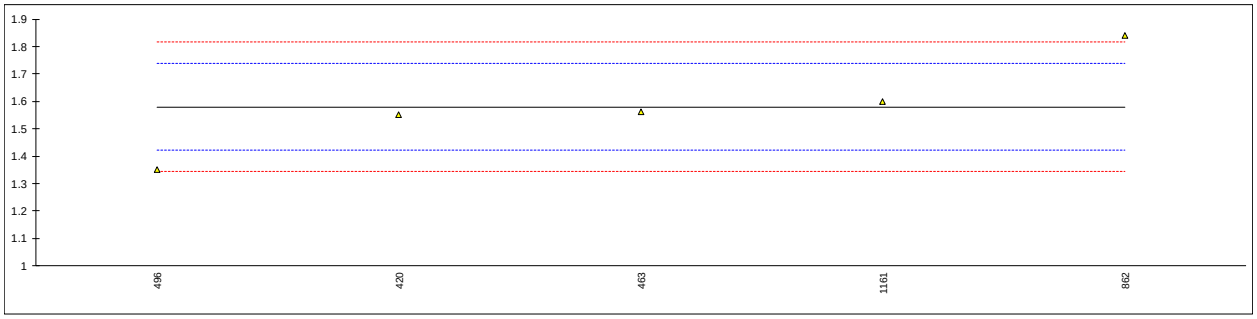




Determination of Ethers (C5 or more C atoms) on sample #18083; results in %V/V

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334		-----		-----	
420	EN13132	1.55		-0.38	
447		-----		-----	
463	EN13132	1.56		-0.25	
496	EN1601	1.35		-2.91	
862	D4815	1.84		3.29	
1161	ISO22854	1.6		0.25	
1446		-----		-----	
1459		-----		-----	
1727		-----		-----	
1835		-----		-----	
1984		<0.17		<-17.83	Possibly a false negative test result?

normality	unknown
n	5
outliers	0
mean (n)	1.580
st.dev. (n)	0.1748
R(calc.)	0.489
st.dev.(EN1601:14)	0.0791
R(EN1601:14)	0.222



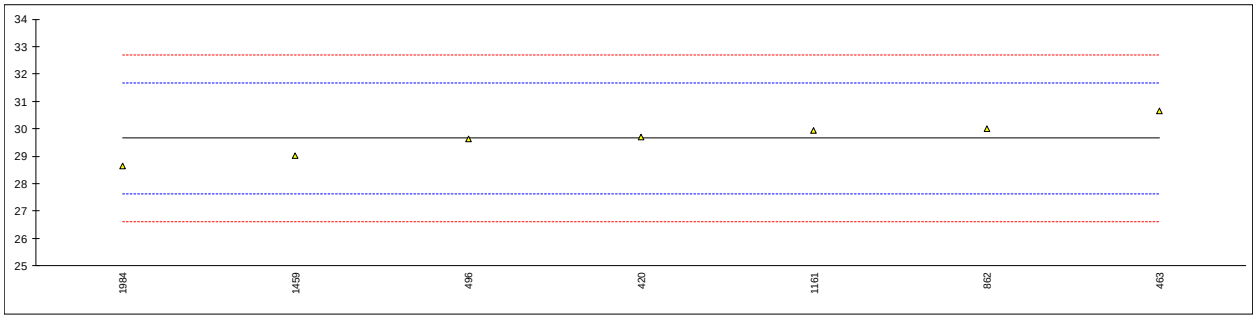
Determination of Higher saturated monoalcohols (C3-C5) on sample #18083; results in %V/V

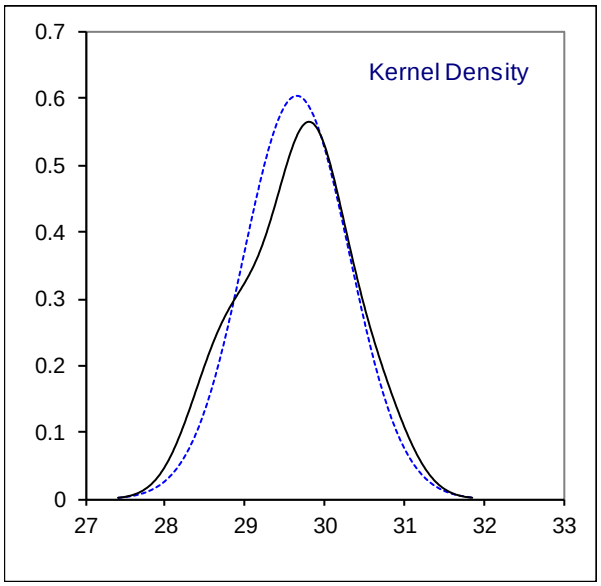
lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334		-----		-----	
420	EN13132	< 0,1		-----	
447		-----		-----	
463		-----		-----	
496	EN1601	0.18		-----	
862	D4815	<0.2		-----	
1161	ISO22854	<0,17		-----	
1446		-----		-----	
1459		-----		-----	
1727		-----		-----	
1835		-----		-----	
1984		<0.17		-----	
	n	5			
	mean (n)	<0.2			

Determination of Total Organically Bound Oxygen on sample #18083; results in %M/M

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334		-----		-----	
420	EN13132	29.7		0.04	
447		-----		-----	
463	EN13132	30.64		0.97	
496	EN1601	29.629		-0.03	
862	D4815	30.0		0.34	
1161	ISO22854	29.95		0.29	
1446		-----		-----	
1459		29.03		-0.62	
1727		-----		-----	
1835		-----		-----	
1984		28.6325		-1.01	

normality	unknown
n	7
outliers	0
mean (n)	29.654
st.dev. (n)	0.6602
R(calc.)	1.849
st.dev.(EN1601:14)	1.0154
R(EN1601:14)	2.843



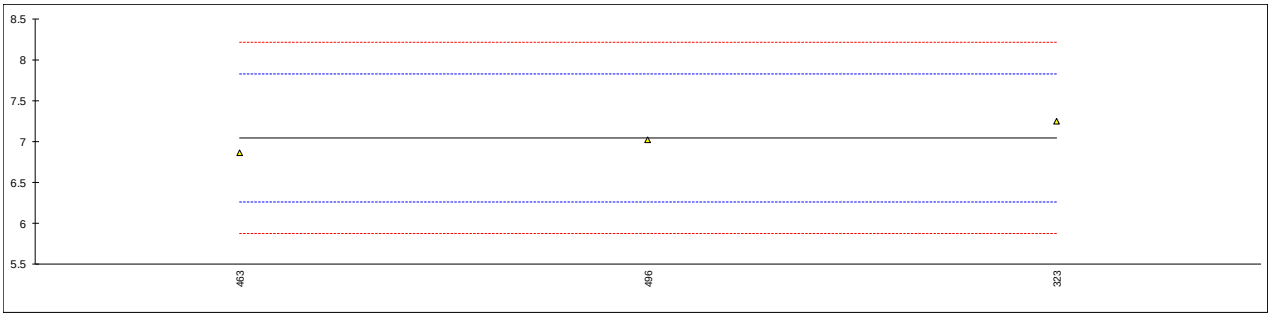


Determination of pHe on sample #18083;

KCl electrode

lab	method	value	mark	z(targ)	Electrode	remarks
323	EN15490	7.25		0.53	KCl	
334		-----		-----	-----	
420		-----		-----	-----	
447		-----		-----	-----	
463	In house	6.857		-0.48	KCl	
496	EN15490	7.025		-0.05	KCl	
862		-----		-----	-----	
1161		-----		-----	-----	
1446		-----		-----	-----	
1459		-----		-----	-----	
1727		-----		-----	-----	
1835		-----		-----	-----	
1984		-----		-----	-----	

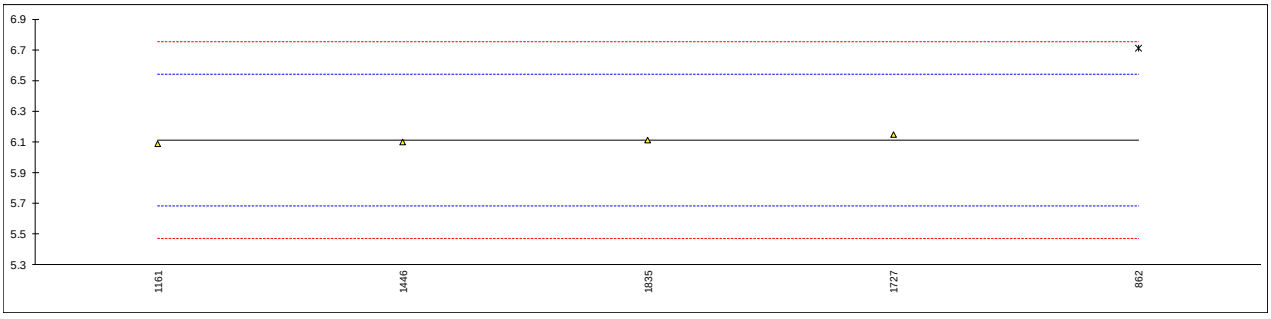
normality	unknown
n	3
outliers	0
mean (n)	7.044
st.dev. (n)	0.1972
R(calc.)	0.552
st.dev.(D6423:14)	0.3895
R(D6423:14)	1.091



LiCl electrode

lab	method	value	mark	z(targ)	Electrode	remarks
323		-----		-----	-----	
334		-----		-----	-----	
420		-----		-----	-----	
447		-----		-----	-----	
463		-----		-----	-----	
496		-----		-----	-----	
862	D6423	6.71	G(0.01)	2.86	LiCl	
1161	EN15490	6.085		-0.13	LiCl	
1446	EN15490	6.1		-0.05	LiCl	
1459		-----		-----	-----	
1727	EN15490	6.15		0.18	LiCl	
1835	EN15490	6.11		-0.01	LiCl	
1984		-----		-----	-----	

normality	unknown
n	4
outliers	1
mean (n)	6.111
st.dev. (n)	0.0278
R(calc.)	0.078
st.dev.(EN15490:07)	0.2095
R(EN15490:07)	0.587



Determination of Phosphorus as P on sample #18083; results in mg/L

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334		-----		-----	
420	EN15837	< 0,05		-----	
447		-----		-----	
463		-----		-----	
496	EN15487	0.008		-----	
862	D3231	<0.2		-----	
1161		-----		-----	
1446		-----		-----	
1459		-----		-----	
1727	EN15487	<0,15		-----	
1835	EN15487	<0.15		-----	
1984		-----		-----	
	n	5			
	mean (n)	<0.2			Application range: 0.15 – 1.5 mg/L

Determination of Sulphate on sample #18083; results in mg/kg

lab	method	value	mark	z(targ)	remarks
323	EN15492	<1		-----	
334		-----		-----	
420		-----		-----	
447		-----		-----	
463		-----		-----	
496	EN15492	0.19		-----	
862	EN15492	<1		-----	
1161		-----		-----	
1446		-----		-----	
1459		-----		-----	
1727	EN15492	<1		-----	
1835	EN15492	<1.0		-----	
1984		-----		-----	
	n	5			
	mean (n)	<1			Application range: 1 - 20 mg/kg

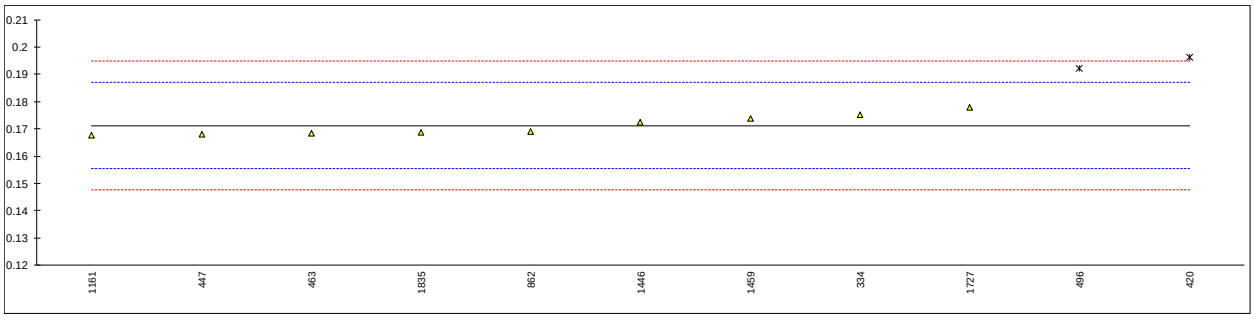
Determination of Sulphur as S on sample #18083; results in mg/kg

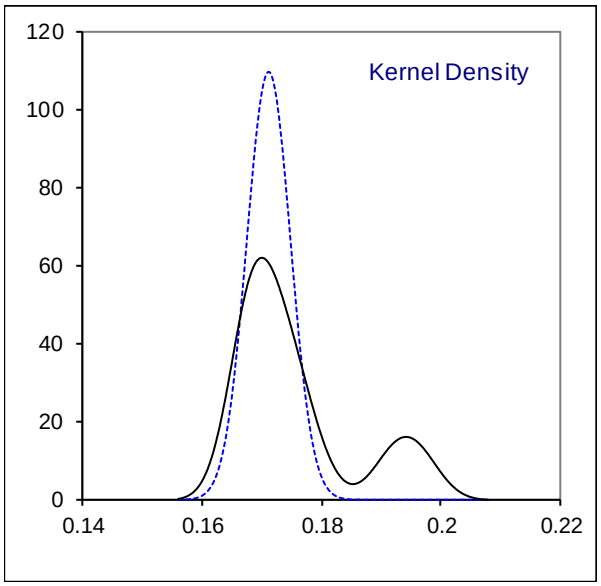
lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	EN15486	0.4		-----	
420	EN15486	0.75		-----	
447	IP490	<3.0		-----	
463	D5453	0.47		-----	
496	EN15486	0.69		-----	
862	D5453	<1		-----	
1161	ISO20846	0.356		-----	
1446		-----		-----	
1459	In house	<5	C	-----	First reported 8.2
1727		-----		-----	
1835	EN15486	<1.0		-----	
1984		-----		-----	
	n	7			
	mean (n)	<1			
					Apllication range EN15486 : 5 – 20 mg/kg

Determination of Water, coulometric on sample #18083; results in %M/M

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
334	EN15489	0.175		0.48	
420	EN15489	0.19625	DG(0.01)	3.19	
447	IP438	0.168		-0.41	
463	D6304	0.1685		-0.34	
496	EN15489	0.192	DG(0.01)	2.65	
862	EN15489	0.169		-0.28	
1161	EN15489	0.1677		-0.44	
1446	ISO760	0.1723		0.14	
1459	ISO12937	0.1737		0.32	
1727	EN15489	0.1778		0.84	
1835	EN15489	0.1687		-0.32	
1984		-----		-----	

normality	OK
n	9
outliers	2
mean (n)	0.1712
st.dev. (n)	0.00365
R(calc.)	0.0102
st.dev.(EN15489:07)	0.00786
R(EN15489:07)	0.0220





APPENDIX 2

Number of participants per country

1 lab in	BELGIUM
1 lab in	CHINA, People's Republic
2 labs in	CZECH REPUBLIC
3 labs in	FRANCE
1 lab in	GERMANY
2 labs in	SPAIN
1 lab in	SWEDEN
1 lab in	TURKEY
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	= probably an error in calculations
U	= test result probably reported in a different unit
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 test result
SDS	= Safety Data Sheet

Literature

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