Results of Proficiency Test
Biogasoline E85
May 2015

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

1 INTRODUCTION

Since 2010, the Institute for Interlaboratory Studies organizes a proficiency scheme for Biogasoline E85. During the annual proficiency testing program 2014/2015, it was decided to continue the round robin for the analysis of Biogasoline E85. In this interlaboratory study 14 laboratories in 10 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the Biogasoline E85 2015 proficiency test are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. It was decided to evaluate the E85 gasoline according to the two different test scopes of ASTM D5798 and of CEN/TS15293. The analyses for fit-for-use and homogeneity testing were subcontracted. In this proficiency test, the participants received one sample of Biogasoline E85.
Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially of participant's data. Also customer's satisfaction is measured on a 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 Organization, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol can be downloaded via the FAQ page of the iis website.

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 about 60 litres of Biogasoline E85 was purchased at a local pump station. After homogenisation, 38 brown glass bottles of 1 litre (labelled #15063) were filled. The homogeneity of the subsamples #15063 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 #15063-1	778.12
Sample #15063-2	778.17
Sample #15063-3	778.23
Sample #15063-4	778.19
Sample #15063-5	778.16
Sample #15063-6	778.16
Sample #15063-7	778.20
Sample #15063-8	778.22

Table 1: homogeneity test results of subsamples #15063

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 (sample #15063)	0.10
reference test	ISO12185:96
0.3*R (reference test)	0.45

Table 2: evaluation of repeatability of the subsamples #15063

The calculated repeatability is less than 0.3 times the reproducibility of the corresponding reference method. Therefore, homogeneity of the subsamples #15063 was assumed.

One 1L bottle with sample #15063 was sent to each of the participants on April 22, 2015.

2.5 ANALYSIS

The participants were requested to determine on sample #15063: Acidity (Total), Chloride (Inorganic), Copper Corrosion, Copper, Density, Electrical Conductivity, Existent Gum (washed), Oxidation Stability, pHe, Phosphorus, Sulphate, Sulphur, Water, Ethanol and higher saturated alcohols, Methanol, Ethers (5 or more C atoms), Higher saturated monoalcohols (C3-C5), Total Organically bound oxygen.
To get comparable results a detailed report form, on which the units were prescribed as well as the required standards and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The detailed report form was also made available for download on the iis website www.iisnl.com. A SDS and a form to confirm receipt of the samples were added to the sample package.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were received. The original reported results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.
Directly after deadline, a reminder fax was sent to those laboratories that did not report results at that moment. Shortly after the deadline, the available results were screened for suspect data. A 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 raw data of these tests (no reanalysis). Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

Statistical calculations were performed as described in the report ‘iis Interlaboratory Studies- Protocol for the Organisation, Statistics and Evaluation’ of April 2014 (iis-protocol, version 3.3).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. 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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.
In accordance with ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test and by R(0.05) for the Rosner General ESD test (see appendix 3, no 16). 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. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to visualise 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 analysis 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 standard. 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 (see appendix 3; nos.13 and 14). Also a normal Gauss curve was projected over the Kernel Density Graph.

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 reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8.

The z-scores were calculated in accordance with:

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

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 the fit-for-useness of the reported test result.

The $z_{(target)}$ scores are listed in the 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:

	$\lvert z \rvert < 1$	good
$1 <$	$\lvert z \rvert < 2$	satisfactory
$2 <$	$\lvert z \rvert < 3$	questionable
$3 <$	$\lvert z \rvert$	unsatisfactory

4 EVALUATION

No problems were encountered during the execution of this proficiency test. One laboratory did not report any test result. Not all laboratories were able to perform all requested analyses. Finally, 13 laboratories did report 110 numerical results. Observed was 1 outlying result, which is 0.9%. In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

In this section the results are discussed per test.

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. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN15491:07.

Chloride, Inorganic: Only five participants reported a test result for inorganic chloride, of which three a test result below the application range of EN15492. Therefore no significant conclusions were drawn.

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

Copper as Cu: No significant conclusions were drawn. Only three test results were reported.

Density at15ºC: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility 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 despite the low concentration level. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of ISO6246:95.

Oxidation stab.: In this determination no problems have been observed. All reporting participants agreed on a result above 900 minutes.

pHe: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of EN15490:07. A possible cause may be the use of different glass electrodes, see reference 17. Electrodes with LiCl give significantly lower pHe values than other types of electrodes.

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

Sulphate: One possibly false positive test result was reported. Four other participants agreed on a sulphate concentration below the application range. Therefore no significant conclusions were drawn.

Sulphur: This determination may not be problematic. No statistical outliers were observed. Although the consensus value is below the application range of EN15486:07 (5 – 20 mg/kg), the calculated reproducibility is in good agreement with the requirements estimated from EN15486:07.

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of EN15489:07.

Ethanol and higher saturated alcohols: This determination (%M/M) may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of EN1601:14.

Methanol: The reporting participants agreed on a value below the detection limit (<0.2%V/V). Therefore no significant conclusions were drawn.

Ethers (5 or more C atoms): This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of EN1601:14. The low number of reported test results (only 4 laboratories reported a numerical result) may (partly) explain for the large spread.

Higher sat. alcohols: Only three participants reported a test result for higher saturated alcohols, of which two a test result below the application range of EN1601. Therefore no significant conclusions were drawn.

Tot. org. bound oxygen: Only four laboratories reported a test result. The reported test results are all far above the application range of EN1601 and of EN13132 (0.17 – 3.7%M/M). However, the calculated reproducibility is in agreement with the estimated (extrapolated) reproducibility requirement of EN1601:14.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories. The assigned values, calculated reproducibilities and reproducibilities, derived from literature standards (in casu ASTM, ISO, EN standards) are compared in the next table.

Parameter	unit	n	mean	2.8 * sd	R (lit)
Acidity as acetic acid, Total	%M/M	9	0.0015	0.0005	0.0014
Chloride, Inorganic	mg/kg	5	0.8	1.1	(0.6)
Copper corr. 3 hrs at 50°C	-----	8	1(1A)	n.a.	n.a.
Copper as Cu	mg/kg	3	n.a.	n.a.	n.a.
Density at 15°C	kg/m^3	13	778.3	0.5	1.5
Electrical Conductivity	µS/cm	5	1.19	0.18	0.21
Existent Gum (washed)	mg/100mL	5	0.6	1.0	1.4
Oxidation Stability	min.	7	>900	n.a.	n.a.
pHe		7	6.8	1.2	0.7
Phosphorus as P	mg/L	4	<0.15	n.a.	n.a.
Sulphate	mg/kg	4	<1.0	n.a.	n.a.
Sulphur as S	mg/kg	11	2.60	1.08	2.09
Water	%M/M	12	0.169	0.016	0.022
Ethanol and higher sat. alc.	%M/M	6	76.4	6.0	5.0
Methanol	%V/V	4	<0.2	n.a.	n.a.
Ethers (5 or more C-atoms)	%V/V	4	0.43	0.21	0.10
Higher saturated monoalc.	%V/V	3	<0.5	n.a.	n.a.
Tot. Organically Bound Oxygen	%M/M	4	27.2	2.6	(2.6)

table 3: performance evaluation sample #15063

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

4.3 COMPARISON OF PROFICIENCY TEST OF MAY 2015 WITH PREVIOUS PTS

Determination	May 2015	May 2014	May 2013	May 2012
Number of reporting labs	13	16	16	14
Number of results reported	110	126	110	103
Statistical outliers	1	2	5	3
Percentage outliers	0.9 %	1.6 %	4.5 %	2.9 %

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 standards. The conclusions are given the following table:

Determination	May 2015	May 2014	May 2013	May 2012
Acidity as acetic acid, Total	++	+	+/-	--
Chloride, Inorganic	(--)	n.e.	+	n.e.
Copper corr. 3 hrs at 50°C	n.e.	++	++	++
Copper as Cu	n.e.	n.e.	n.e.	n.e.
Density at 15°C	++	+	+/-	-
Electrical Conductivity	+	+/-	-	n.e.
Existent Gum (washed)	+	+	(+/-)*	(--)*
Oxidation Stability	n.e.	n.e.	n.e.	n.e.
pHe	--	--	--	--
Phosphorus as P	n.e.	n.e.	n.e.	n.e.
Sulphate	n.e.	n.e.	n.e.	n.e.
Sulphur as S	++	++	++	++
Water	+	-	+/-	+/-
Ethanol and higher sat. alc.	-	--	--	--
Methanol	n.e.	n.e.	n.e.	++
Ethers (5 or more C-atoms)	--	n.e.	--	n.e.
Higher saturated monoalcohols	n.e.	n.e.	n.e.	n.e.
Total organically bound oxygen	+/-	n.e.	n.e.	n.e.

table 5: comparison of the quality of the determinations against the respective standard requirements

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

The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

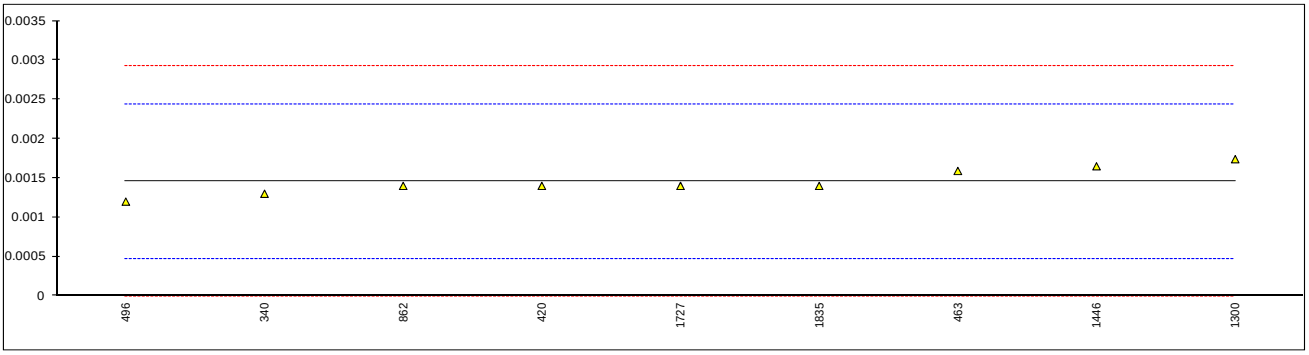
++: group performed much better than the standard
+ : group performed better than the standard
+/-: group performance equals the standard
- : group performed worse than the standard
-- : group performed much worse than the standard
n.e.: not evaluated

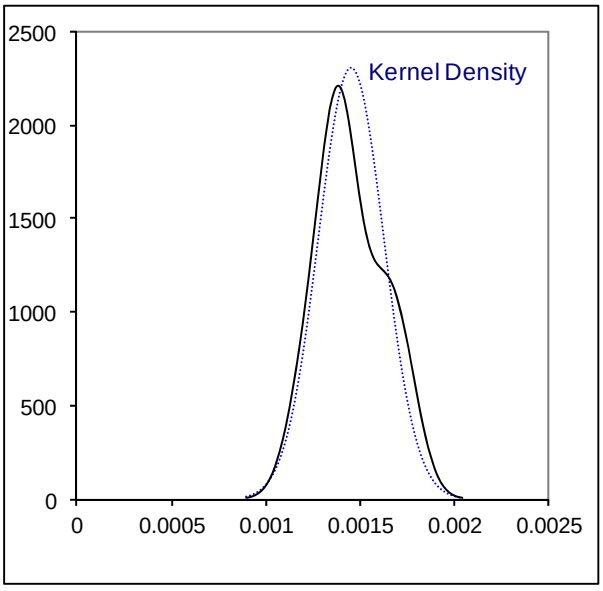
APPENDIX 1

Determination of Total Acidity as Acetic Acid on sample #15063; results in %M/M

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340	EN15491	0.0013		-0.31	
420	EN15491	0.0014		-0.11	
463	EN15491	0.00159		0.28	
496	EN15491	0.0012		-0.52	
862	EN15491	0.0014		-0.11	
1161		-----		-----	
1300	EN15491	0.00174		0.59	
1446	EN15491	0.00165		0.40	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727	EN15491	0.0014		-0.11	
1835	EN15491	0.0014		-0.11	

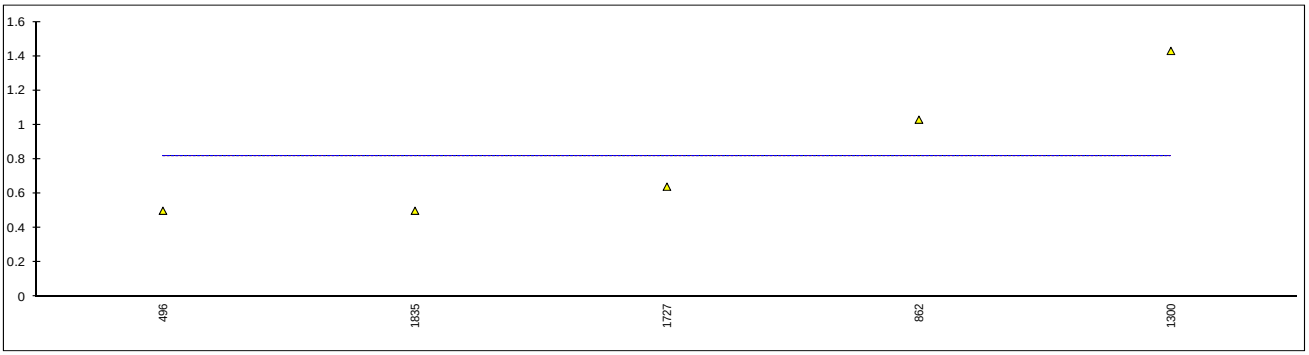
normality	OK
n	9
outliers	0
mean (n)	0.00145
st.dev. (n)	0.000173
R(calc.)	0.00048
R(EN15491:07)	0.00137





Determination of Inorganic Chloride on sample #15063; results in mg/kg

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340		-----		-----	
420		-----		-----	
463		-----		-----	
496	EN15492	0.5		-----	
862	EN15492	1.03		-----	
1161		-----		-----	
1300	EN15492	1.43	C	-----	First reported 3.27
1446		-----		-----	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727	EN15492	0.64		-----	
1835	EN15492	0.5		-----	
	normality	unknown			
	n	5			
	outliers	0			
	mean (n)	0.820			
	st.dev. (n)	0.4042			
	R(calc.)	1.132			
	R(EN15492:12)	(0.562)			Application range : 1.0 – 2.5 mg/kg



Determination of Copper Corrosion 3hrs/50°C on sample #15063; rating

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340	ISO2160	1A		-----	
420	ISO2160	1A		-----	
463	D130	1A		-----	
496	D130	1A		-----	
862	D130	1A		-----	
1161	D130	1A		-----	
1300	ISO2160	1A		-----	
1446		-----		-----	
1459		-----		-----	
1634	D130	1A		-----	
1706		-----		-----	
1727		-----		-----	
1835		-----		-----	
	normality	n.a.			
	n	8			
	outliers	n.a.			
	mean (n)	1A			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(ISO2160:98)	n.a.			

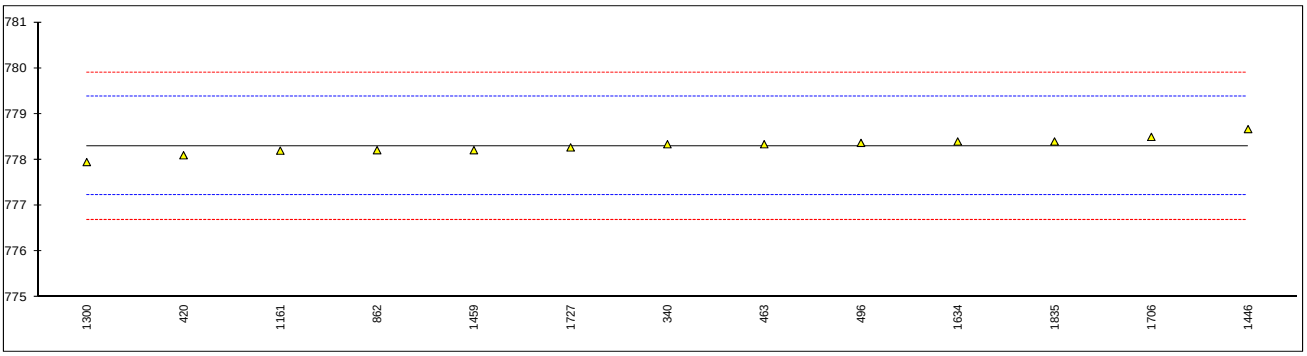
Determination of Copper as Cu on sample #15063; results in mg/kg

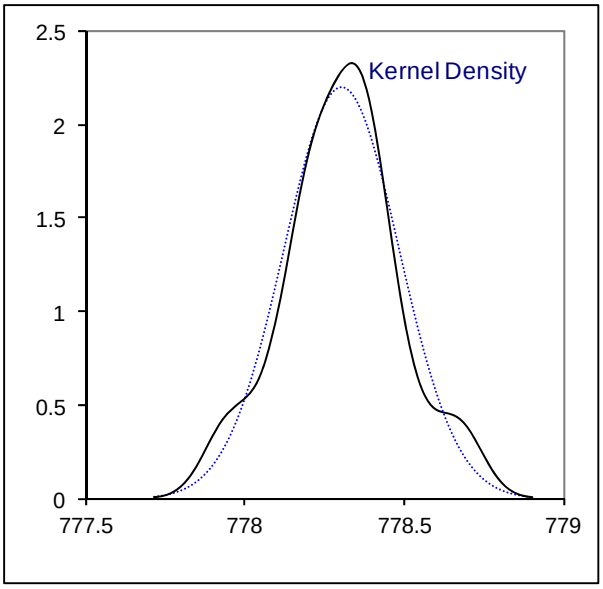
lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340		-----		-----	
420	EN15488	0.126		-----	
463		-----		-----	
496		-----		-----	
862	EN15488	<0.01		-----	
1161		-----		-----	
1300	EN15837	0.0272		-----	
1446		-----		-----	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727		-----		-----	
1835		-----		-----	
	normality	n.a.			
	n	3			
	outliers	n.a.			
	mean (n)	n.a.			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(EN15488:07)	n.a.			

Determination of Density at 15°C on sample #15063; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340	ISO12185	778.34		0.07	
420	ISO12185	778.1		-0.38	
463	ISO12185	778.34		0.07	
496	ISO12185	778.37		0.12	
862	D4052	778.21		-0.18	
1161	ISO12185	778.2		-0.20	
1300	ISO12185	777.95		-0.66	
1446	ISO12185	778.67		0.68	
1459	ISO12185	778.21		-0.18	
1634	D4052	778.4		0.18	
1706	ISO12185	778.5		0.36	
1727	D4052	778.27		-0.06	
1835	D4052	778.4		0.18	

normality	OK
n	13
outliers	0
mean (n)	778.30
st.dev. (n)	0.181
R(calc.)	0.51
R(ISO12185:96)	1.50

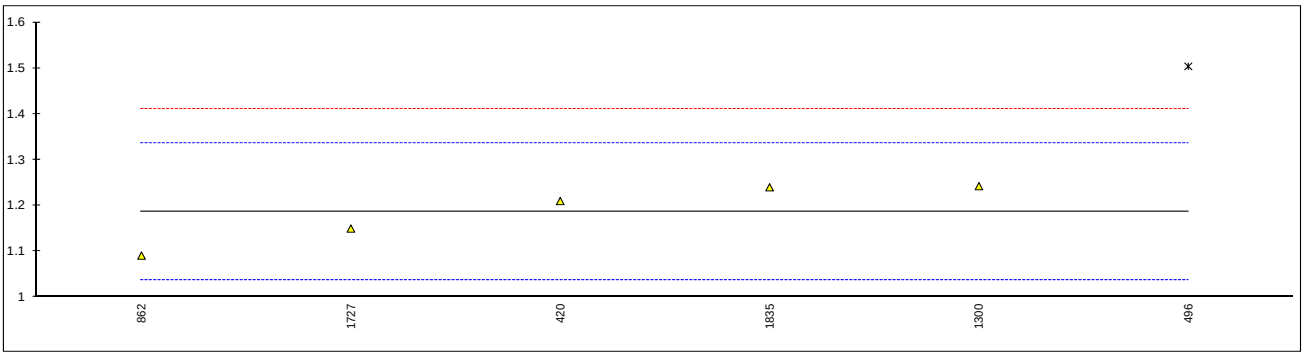




Determination of Electrical Conductivity at 25°C on sample #15063; results in µS/cm

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340		-----		-----	
420	EN15938	1.21		0.31	
463		-----		-----	
496	EN15938	1.504	D(0.05)	4.23	
862	EN15938	1.091		-1.28	
1161		-----		-----	
1300	EN15938	1.2425		0.74	
1446		-----		-----	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727	EN15938	1.15		-0.49	
1835	EN15938	1.24		0.71	

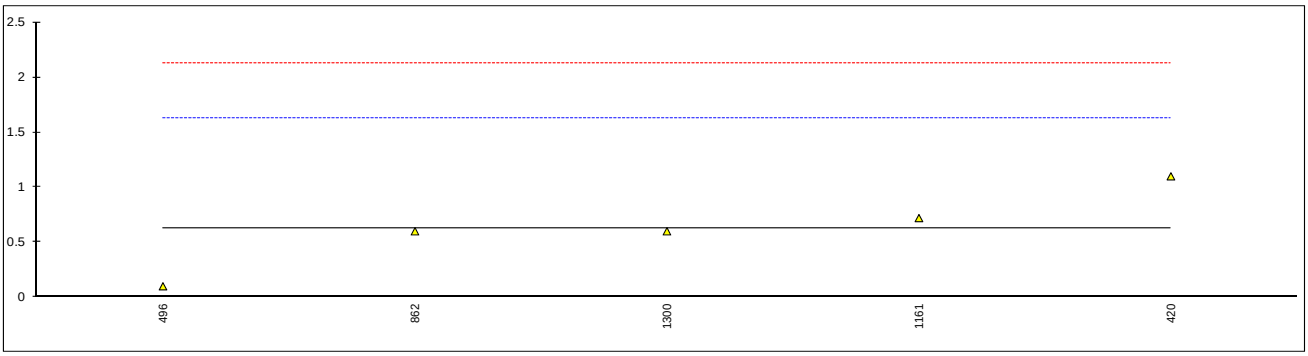
normality	unknown
n	5
outliers	1
mean (n)	1.187
st.dev. (n)	0.0652
R(calc.)	0.183
R(EN15938:10)	0.210



Determination of Existent Gum (solvent washed) on sample #15063; results in mg/100ml

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340	ISO6246	<1		-----	
420	ISO6246	1.1		0.95	
463	ISO6246	<0.5		-----	
496	ISO6246	0.1		-1.04	
862	ISO6246	0.6		-0.05	
1161	ISO6246	0.72		0.19	
1300	ISO6246	0.6		-0.05	
1446		-----		-----	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727		-----		-----	
1835		-----		-----	

normality	unknown
n	5
outliers	0
mean (n)	0.62
st.dev. (n)	0.358
R(calc.)	1.00
R(ISO6246:95)	1.41 (solvent washed)



Determination of Oxidation Stability on sample #15063; results in minutes

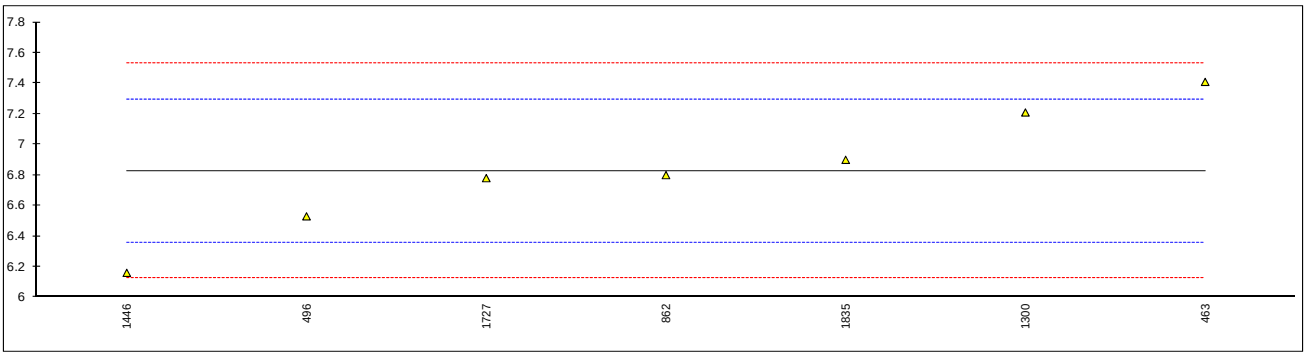
lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340	ISO7536	>900		-----	
420	ISO7536	>900		-----	
463	ISO7536	>900		-----	
496	ISO7536	>900		-----	
862	D525	>900		-----	
1161	ISO7536	>900		-----	
1300	ISO7536	>900		-----	
1446		-----		-----	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727		-----		-----	
1835		-----		-----	

normality	n.a.
n	7
outliers	n.a.
mean (n)	>900
st.dev. (n)	n.a.
R(calc.)	n.a.
R(ISO7536:94)	n.a.

Determination of pHe on sample #15063;

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340		-----		-----	
420		-----		-----	
463	EN15490	7.41		2.49	
496	EN15490	6.53		-1.27	
862	EN15490	6.80		-0.12	
1161		-----		-----	
1300	EN15490	7.21		1.64	
1446	EN15490	6.16		-2.85	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727	EN15490	6.78		-0.20	
1835	EN15490	6.9		0.31	

normality	unknown
n	7
outliers	0
mean (n)	6.827
st.dev. (n)	0.4139
R(calc.)	1.159
R(EN15490:07)	0.655



Determination of Phosphorus as P on sample #15063; results in mg/L

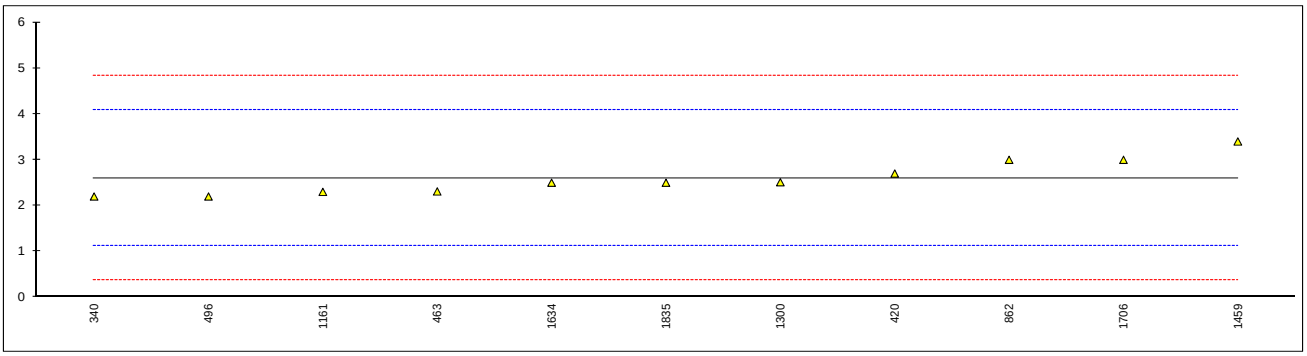
lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340		-----		-----	
420		-----		-----	
463		-----		-----	
496	EN15487	0.0016		-----	
862	EN15487	0.08		-----	
1161		-----		-----	
1300	EN15487	0.029		-----	
1446		-----		-----	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727		-----		-----	
1835	EN15487	<0.15		-----	
	normality	unknown			
	n	4			
	outliers	0			
	mean (n)	<0.15			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(EN15487:07)	n.a.			Application range: 0.15 – 1.50 mg/L

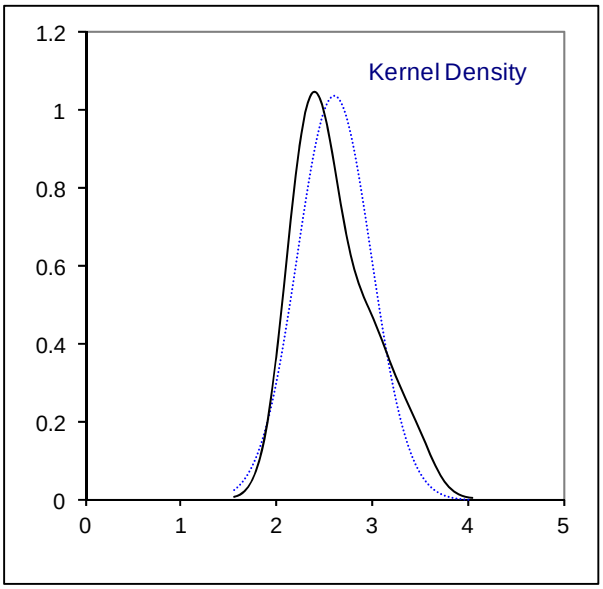
Determination of Sulphate on sample #15063; results in mg/kg

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340		-----		-----	
420		-----		-----	
463		-----		-----	
496	EN15492	<0.1		-----	
862	EN15492	1.37		-----	Possibly a false positive test result?
1161		-----		-----	
1300	EN15492	0.607		-----	
1446		-----		-----	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727	EN15492	<1		-----	
1835	EN15492	<1.0		-----	
	normality	n.a.			
	n	4			
	outliers	n.a.			
	mean (n)	<1.0			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(EN15492:12)	n.a.			Application range: 1 – 20 mg/kg

Determination of Sulphur as S on sample #15063; results in mg/kg

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340	EN15486	2.2		-0.54	
420	ISO20846	2.7		0.13	
463	D5453	2.31		-0.39	
496	EN15486	2.20		-0.54	
862	D5453	3.0		0.53	
1161	ISO20846	2.3		-0.41	
1300	EN15486	2.510		-0.12	
1446		-----		-----	
1459		3.4		1.07	
1634	ISO20846	2.5		-0.14	
1706	EN15486	3.0		0.53	
1727		-----		-----	
1835	EN15486	2.5		-0.14	
	normality	OK			
	n	11			
	outliers	0			
	mean (n)	2.602			
	st.dev. (n)	0.3863			
	R(calc.)	1.082			
	R(EN15486:07)	2.086			Application range : 5 – 20 mg/kg

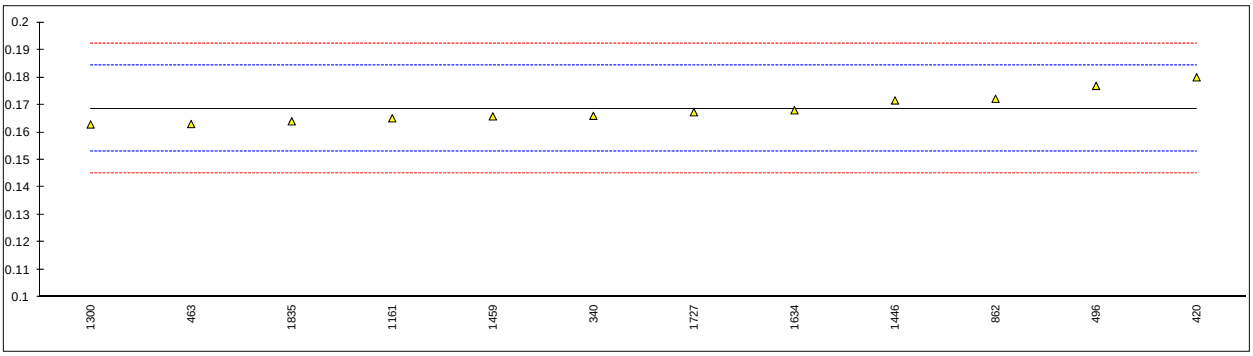


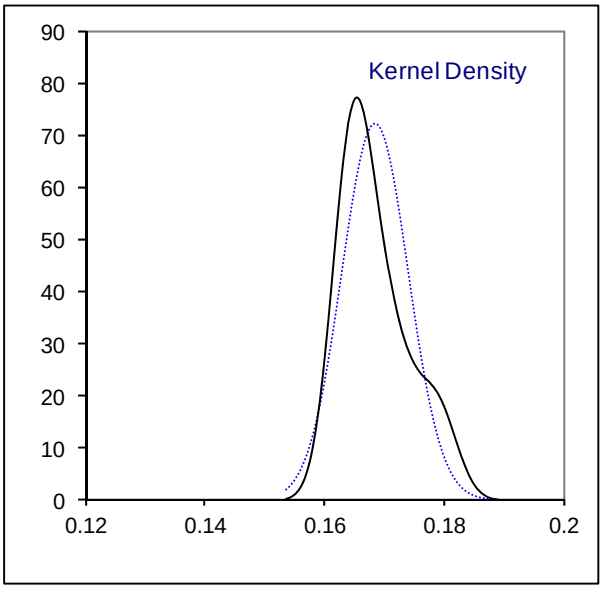


Determination of Water on sample #15063; results in %M/M

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340	EN15489	0.166		-0.33	
420	EN15489	0.180		1.46	
463	EN15489	0.1630		-0.71	
496	EN15489	0.1769		1.06	
862	EN15489	0.1722		0.46	
1161	EN15489	0.1651		-0.44	
1300	EN15489	0.16286		-0.73	
1446	ISO760	0.1716		0.39	
1459	EN15489	0.1658		-0.35	
1634	ISO12937	0.168		-0.07	
1706		-----		-----	
1727	EN15489	0.1673		-0.16	
1835	EN15489	0.164		-0.58	

normality	suspect
n	12
outliers	0
mean (n)	0.1686
st.dev. (n)	0.00552
R(calc.)	0.0155
R(EN15489:07)	0.0220



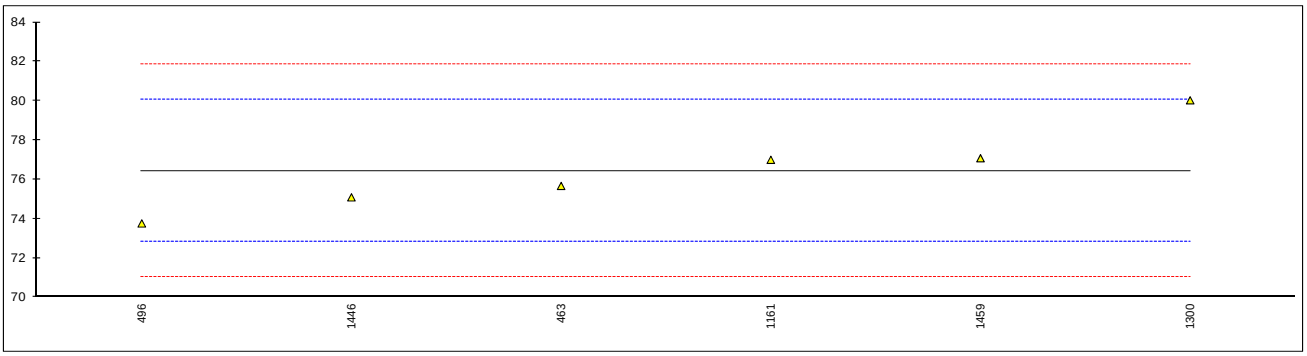


Determination of Ethanol and higher saturated alcohols on sample #15063; results in %M/M

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340		-----		-----	
420		-----		-----	
463	EN13132	75.67		-0.43	
496	EN1601	73.76		-1.49	
862		-----		-----	
1161	EN13132	77.0		0.31	
1300	EN1601	80.03		1.99	
1446	EN1601	75.09		-0.75	
1459		77.08		0.36	
1634		-----		-----	
1706		-----		-----	
1727		-----		-----	
1835		-----		-----	

	normality	unknown
	n	6
	outliers	0
	mean (n)	76.438
	st.dev. (n)	2.1545
	R(calc.)	6.033
	R(EN1601:14)	5.042

Application range: 20 - 100 %M/M



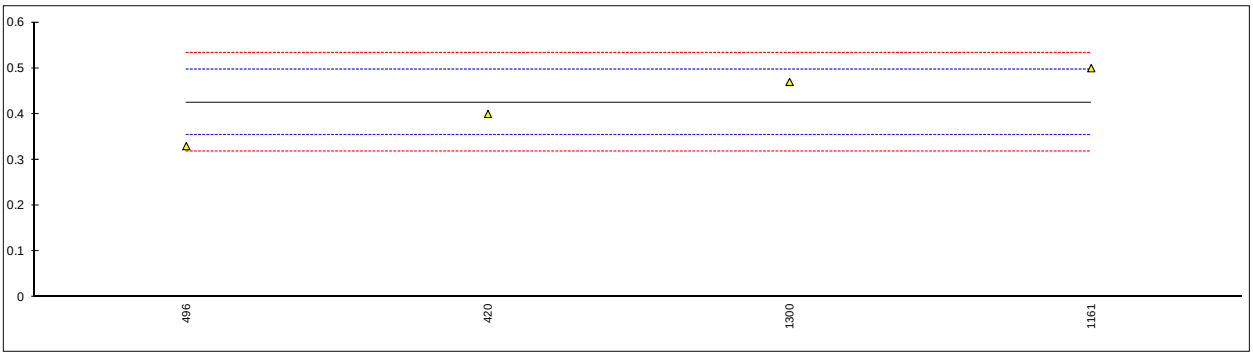
Determination of Methanol on sample #15063; results in %V/V

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340		-----		-----	
420	EN13132	<0.1		-----	
463	EN13132	<0.2		-----	
496	EN1601	<0.10		-----	
862		-----		-----	
1161		-----		-----	
1300	EN1601	0.00050		-----	
1446		-----		-----	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727		-----		-----	
1835		-----		-----	
	normality	n.a.			
	n	4			
	outliers	n.a.			
	mean (n)	<0.2			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(EN1601:14)	n.a.			

Determination of Ethers (5 or more C atoms) on sample #15063; results in %V/V

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340		-----		-----	
420	EN13132	0.40		-0.70	
463	EN13132	<0.2		<-6.30	false negative test result?
496	EN1601	0.33		-2.66	
862		-----		-----	
1161	EN13132	0.5		2.10	
1300	EN1601	0.47		1.26	
1446		-----		-----	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727		-----		-----	
1835		-----		-----	

normality	n.a.
n	4
outliers	0
mean (n)	0.425
st.dev. (n)	0.0759
R(calc.)	0.213
R(EN1601:14)	0.100



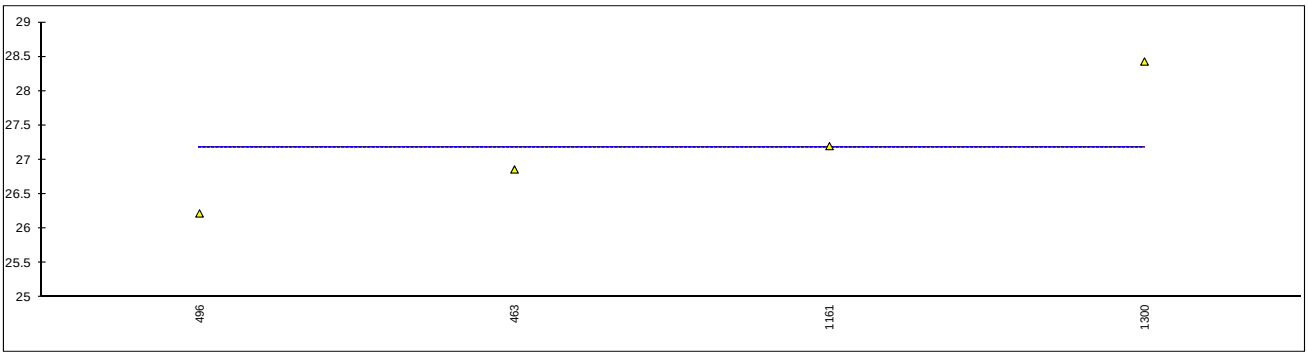
Determination of Higher saturated monoalcohols (C3-C5) on sample #15063; results in %V/V

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340		-----		-----	
420		-----		-----	
463	EN13132	0.41		-----	
496	EN1601	0.08		-----	
862		-----		-----	
1161		-----		-----	
1300	EN1601	0.021		-----	
1446		-----		-----	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727		-----		-----	
1835		-----		-----	

normality	n.a.
n	3
outliers	n.a.
mean (n)	<0.50
st.dev. (n)	n.a.
R(calc.)	n.a.
R(EN1601:14)	n.a.

Determination of Total Organically Bound Oxygen on sample #15063; results in %M/M

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340		-----		-----	
420		-----		-----	
463	EN13132	26.86		-0.34	
496	EN1601	26.220		-1.02	
862		-----		-----	
1161	EN13132	27.2		0.02	
1300	EN1601	28.43		1.34	
1446		-----		-----	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727		-----		-----	
1835		-----		-----	
	normality	unknown			
	n	4			
	outliers	0			
	mean (n)	27.177			
	st.dev. (n)	0.9286			
	R(calc.)	2.600			
	R(EN1601:14)	2.617			Application range: 0.17 – 3.7 %M/M



APPENDIX 2

Number of participants per country

1 lab in AUSTRIA
2 labs in CZECH REPUBLIC
1 lab in ESTONIA
3 labs in FRANCE
1 lab in GERMANY
1 lab in P.R. of CHINA
1 lab in PORTUGAL
2 labs in SPAIN
1 lab in SWEDEN
1 lab in TURKEY

APPENDIX 3

Abbreviations:

C	= final result after checking of first reported suspect 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
ex	= excluded from calculations
E	= error in calculations
n.a.	= not applicable
n.e.	= not evaluated
W	= withdrawn
fr.	= first reported
U	= reported in different unit
SDS	= Safety Data Sheet

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, April 2014

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3 ASTM E1301-03

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12 J.N. Miller, Analyst, 118, 455, (1993)

13 Analytical Methods Committee Technical Brief, No4 January 2001

14 The Royal Society of Chemistry 2002, Analyst 2002, 127 page1359-1364, P.J. Lowthian and M. Thompson. (see www.rsc.org/suppdata/an/b2/b205600n/)

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17 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 pp 327-332 (2011)