Results of Proficiency Test
Biogasoline E85
May 2016

Organised by: **Institute for Interlaboratory Studies**
                       Spijkenisse, the Netherlands

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Appendices:

1   INTRODUCTION

Since 2010, the Institute for Interlaboratory Studies organizes a proficiency scheme for Biogasoline E85. During the annual proficiency testing program 2015/2016, it was decided to continue the round robin for the testing of Biogasoline E85. In this interlaboratory study 17 laboratories in 12 different countries have registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the Biogasoline E85 2016 proficiency test are presented and discussed. This report is also electronically available through the iis website 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 Biogasoline according to the test scopes of ASTM D5798 and of CEN/TS15293. The analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one sample of Biogasoline E85 to the participants. Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1   QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2   PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol is electronically available through the iis website site 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 about 39 litres of Biogasoline E85 was purchased at a local pump station. After homogenisation, 37 brown glass bottles of 1 litre (labelled #16083) were filled. The homogeneity of the subsamples #16083 was checked by determination of Density at 15°C in accordance with ASTM D4052 on 4 stratified randomly selected samples.

	Density at 15°C in kg/m^3
Sample #16083-1	781.31
Sample #16083-2	781.27
Sample #16083-3	781.38
Sample #16083-4	781.30

Table 1: homogeneity test results of subsamples #16083

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)	0.13
reference test method	ISO12185:96
0.3 * R (reference test method)	0.45

Table 2: evaluation of repeatability of the subsamples #16083

The calculated repeatability is less than 0.3 times the reproducibility of the corresponding reference test method. Therefore, homogeneity of the subsamples #16083 was assumed.

One 1L bottle labelled #16083 was sent to each of the participating laboratories on May 4, 2016.

2.5 STABILITY OF THE SAMPLES

The stability of Biodiesel E85, packed in the brown glass bottles, 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 #16083: Total Acidity (as Acetic Acid), Chloride (Inorganic), Copper Corrosion, Copper, Density, Electrical Conductivity, Existent Gum (solvent washed), Oxidation Stability, Ethanol and higher saturated alcohols, Ethers (5 or more C atoms), Higher saturated monoalcohols (C3-C5), Methanol, Total Organically bound oxygen, pHe, Phosphorus, Sulphate, Sulphur and Water.

To get comparable test results a detailed report form, on which the units were prescribed as well as the reference test methods and a letter of instructions were prepared and made

available on the data entry portal www.kpmd.co.uk/sgs-iis/. A SDS and a form to confirm receipt of the sample were added to the sample package.

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 April 2014 (iis-protocol, version 3.3).
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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the test results 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. 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 test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.
Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM 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.

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:

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

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:

$$| z | < 1 \quad \text{good}$$
$$1 < | z | < 2 \quad \text{satisfactory}$$
$$2 < | z | < 3 \quad \text{questionable}$$
$$3 < | z | \quad \text{unsatisfactory}$$

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, 16 laboratories did report 117 numerical test results. Observed were 7 outlying test results, which is 6.0%. 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: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN15492:12.

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

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

Density at15°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 problematic. One test result was excluded as zero is not a real test result. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of EN15938:10. The low number of reported test results (only 4 laboratories reported a numerical result) may (partly) explain this larger variation.

Existent Gum: This determination was not problematic.Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers 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 test result above 360 minutes according to specification EN15293:2011.

Ethanol and higher saturated alcohols: 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 EN1601:14.

Ethers (5 or more C atoms): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN1601:14.

Higher sat. alcohols: Only three participants reported a test result for higher saturated alcohols. Therefore no significant conclusions were drawn.

Methanol: 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 EN1601:14.

Tot. org. bound oxygen: 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 EN1601:14.

pHe: This determination was problematic. No statistical outliers were observed and one test result excluded. Test method EN15490 mentions to use LiCl electrodes. Electrodes with LiCl give significantly lower pHe values than other types of electrodes (e.g. KCl electrodes) (see lit. 18). The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of EN15490:07.

Phosphorus: The reporting participants agreed on a value 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: 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. One statistical outlier was observed. However, the calculated reproducibility 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	10	0.0019	0.0012	0.0014
Chloride, Inorganic	mg/kg	4	1.0	0.3	0.6
Copper Corrosion 3 hrs at 50°C		11	1 or 1a	n.a.	n.a.
Copper as Cu	mg/kg	3	<0.1	n.a.	n.a.
Density at 15°C	kg/m^3	14	781.5	0.5	1.5
Electrical Conductivity	µS/cm	4	2.02	0.52	0.31
Existent Gum (washed)	mg/100mL	6	0.9	0.6	1.5
Oxidation Stability	minutes	9	>360	n.a.	n.a.
Ethanol and higher saturated alcohols	%V/V	10	78.7	3.3	5.2
Ethers (5 or more C-atoms)	%V/V	6	0.50	0.15	0.15
Higher saturated mono alcohols	%V/V	3	<0.2	n.a.	n.a.
Methanol	%V/V	4	0.08	0.14	0.12
Total organically bound Oxygen	%M/M	6	27.8	0.9	2.7
pHe		6	6.5	1.1	0.6
Phosphorus as P	mg/L	3	<0.15	n.a.	n.a.
Sulphate	mg/kg	3	0.36	0.60	(0.23)*
Sulphur as S	mg/kg	11	2.22	0.79	2.03
Water	%M/M	13	0.192	0.013	0.022

Table 3: performance evaluation sample #16083

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

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 reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3   COMPARISON OF PROFICIENCY TEST OF MAY 2016 WITH PREVIOUS PTS

Determination	May 2016	May 2015	May 2014	May 2013	May 2012
Number of reporting labs	16	13	16	16	14
Number of test results reported	117	110	126	110	103
Statistical outliers	7	1	2	5	3
Percentage outliers	6.0%	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 reference test methods. The conclusions are given the following table:

Determination	May 2016	May 2015	May 2014	May 2013	May 2012
Acidity as Acetic Acid, Total	+	++	+	+/-	--
Chloride, Inorganic	++	(--)*	n.e.	+	n.e.
Copper Corrosion 3 hrs at 50°C	n.e.	n.e.	++	++	++
Copper as Cu	n.e.	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.	n.e.
Ethanol and higher saturated alcohols	+	-	--	--	--
Ethers (5 or more C-atoms)	+/-	--	n.e.	--	n.e.
Higher saturated mono alcohols	n.e.	n.e.	n.e.	n.e.	n.e.
Methanol	-	n.e.	n.e.	n.e.	++
Total organically bound Oxygen	++	+/-	n.e.	n.e.	n.e.
pHe	--	--	--	--	--
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	++	++	++	++	++
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 #16083; results in %M/M

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340	EN15491	<0.003		-----	
420	EN15491	0.0018		-0.23	
447		-----		-----	
463	EN15491	0.00163		-0.58	
494	EN15491	0.0015		-0.84	
496	EN15491	0.0019		-0.03	
862	EN15491	0.002		0.18	
970		-----		-----	
1047	EN15491	0.0021		0.38	
1161	EN15491	0.0024		1.00	
1446	EN15491	0.0021		0.38	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727	EN15491	0.0026		1.40	
1835	EN15491	0.0011		-1.66	

normality	OK
n	10
outliers	0
mean (n)	0.00191
st.dev. (n)	0.000436
R(calc.)	0.00122
R(EN15491:07)	0.00137
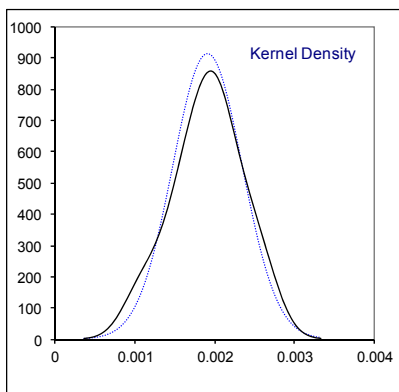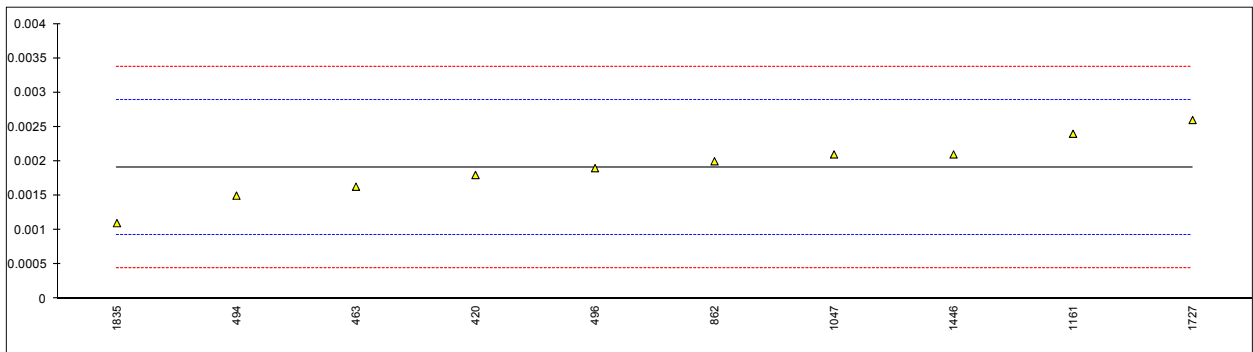
Determination of Inorganic Chloride on sample #16083; results in mg/kg

lab	method	value	mark	z(targ)	remarks
334	EN15492	1.1		0.67	
340	ISO6227	<1		-----	
420		-----		-----	
447		-----		-----	
463		-----		-----	
494		-----		-----	
496	EN15492	0.9		-0.31	
862		-----		-----	
970		-----		-----	
1047	EN15492	0.95		-0.06	
1161		-----		-----	
1446		-----		-----	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727		-----		-----	
1835	EN15492	0.9		-0.31	

normality	unknown	
n	4	
outliers	0	
mean (n)	0.9625	
st.dev. (n)	0.09465	
R(calc.)	0.2650	
R(EN15492:12)	0.5722	Application range: 1 – 30 mg/kg
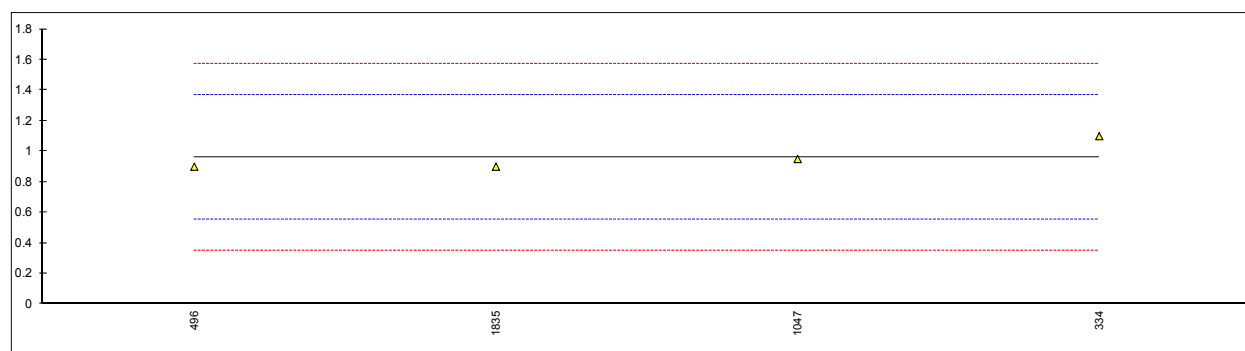
Determination of Copper Corrosion 3hrs at 50°C on sample #16083; rating

lab	method	value	mark	z(targ)	remarks
334	D130	1a		-----	
340	D130	1a		-----	
420	ISO2160	Class 1a		-----	
447	D130	1a		-----	
463	ISO2160	1A		-----	
494	ISO2160	1a		-----	
496	D130	1a		-----	
862	D130	1a		-----	
970		-----		-----	
1047	ISO2160	1		-----	
1161	ISO2160	1A		-----	
1446		-----		-----	
1459		-----		-----	
1634	D130	1a		-----	
1706		-----		-----	
1727		-----		-----	
1835		-----		-----	

normality	n.a.	
n	11	
outliers	0	
mean (n)	1 or 1a	
st.dev. (n)	n.a.	
R(calc.)	n.a.	
R(lit.)	n.a.	

Determination of Copper as Cu on sample #16083; results in mg/kg

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340		-----		-----	
420	EN15837	<0,1		-----	
447		-----		-----	
463		-----		-----	
494		-----		-----	
496		-----		-----	
862	EN15488	<0.07		-----	
970		-----		-----	
1047	EN15837	<0,020		-----	
1161		-----		-----	
1446		-----		-----	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727		-----		-----	
1835		-----		-----	

normality	n.a.	
n	3	
outliers	0	
mean (n)	<0.1	
st.dev. (n)	n.a.	
R(calc.)	n.a.	
R(EN15488:07)	n.a.	Application range: 0.07 – 0.20 mg/kg

Determination of Density at 15°C on sample #16083; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
334	ISO12185	781.3		-0.31	
340	ISO12185	781.43		-0.06	
420	ISO12185	781.5		0.07	
447	D4052	781.3		-0.31	
463	ISO12185	781.27		-0.36	
494	ISO12185	781.8		0.63	
496	ISO12185	781.55		0.16	
862		-----		-----	
970		-----		-----	
1047	ISO12185	781.4		-0.12	
1161	ISO12185	781.25		-0.40	
1446	ISO12185	781.74		0.52	
1459	ISO12185	781.37		-0.18	
1634	ISO12185	781.454		-0.02	
1706	ISO12185	782.1	G(0.05)	1.19	
1727	D4052	781.53		0.12	
1835	ISO12185	781.6		0.25	

normality	OK
n	14
outliers	1
mean (n)	781.464
st.dev. (n)	0.1692
R(calc.)	0.474
R(ISO12185:96)	1.500

Kernel Density
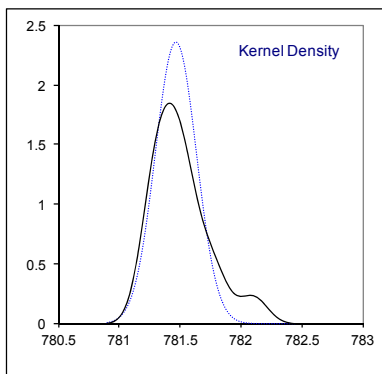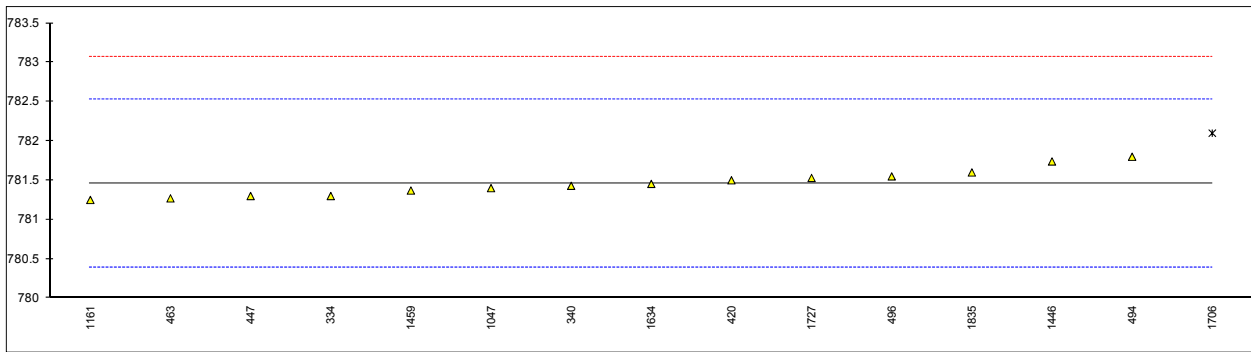
Determination of Electrical Conductivity at 25°C on sample #16083; results in µS/cm

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340		-----		-----	
420	EN15938	1.94		-0.75	
447		-----		-----	
463		-----		-----	
494	EN15938	0	ex	-18.31	see §4.1
496	EN15938	2.3		2.50	
862		-----		-----	
970		-----		-----	
1047		-----		-----	
1161		-----		-----	
1446		-----		-----	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727	EN15938	1.913		-1.00	
1835	EN15938	1.94		-0.75	

normality	unknown
n	4
outliers	1
mean (n)	2.023
st.dev. (n)	0.1849
R(calc.)	0.518
R(EN15938:10)	0.309
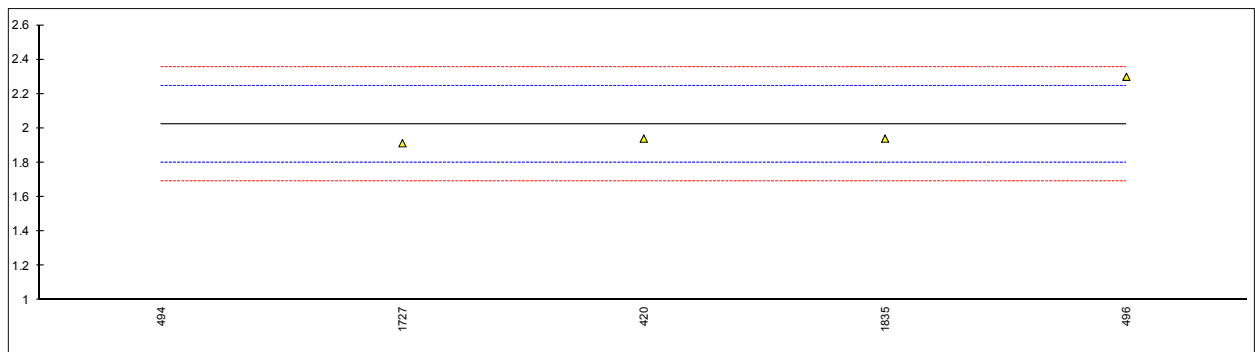
Determination of Existent Gum (solvent washed) on sample #16083; results in mg/100ml

lab	method	value	mark	z(targ)	remarks
334	ISO6246	1		0.26	
340	ISO6246	1		0.26	
420	ISO6246	2.7	DG(0.01)	3.35	
447	D381	<0.5		-----	
463		-----		-----	
494	ISO6246	0.90		0.08	
496	ISO6246	0.5		-0.65	
862	D381	3.0	DG(0.01)	3.90	
970		-----		-----	
1047	ISO6246	1.0		0.26	
1161	ISO6246	0.75		-0.20	
1446		-----		-----	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727		-----		-----	
1835		-----		-----	

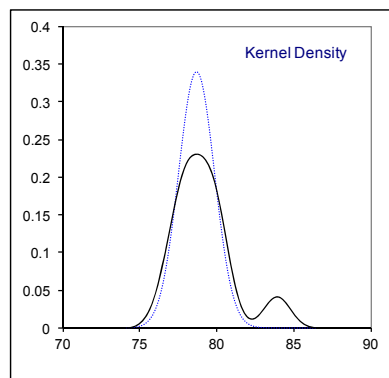|	
normality	unknown
n	6
outliers	2
mean (n)	0.86
st.dev. (n)	0.201
R(calc.)	0.56
R(ISO6246:95)	1.54
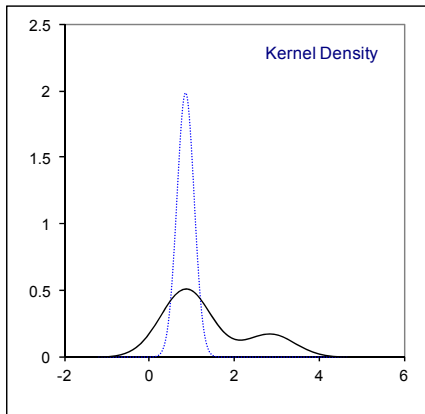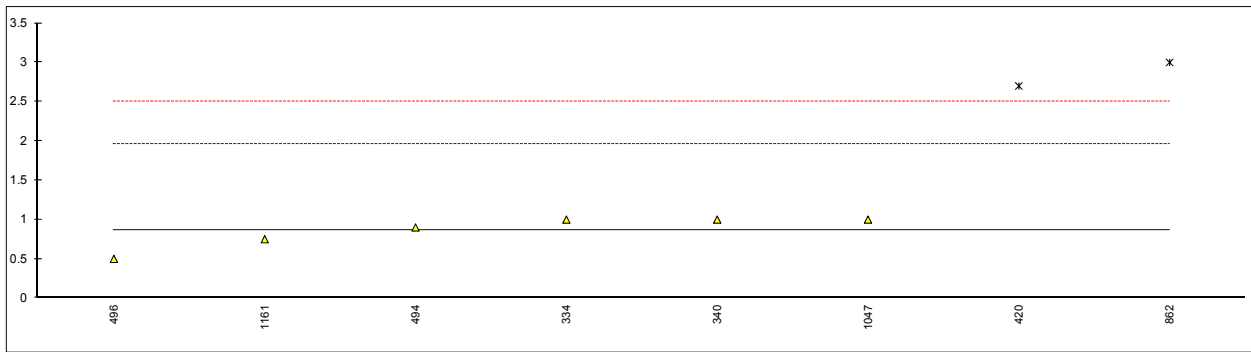
Determination of Oxidation Stability on sample #16083; results in minutes

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340	ISO7536	>900		-----	
420	ISO7536	>900		-----	
447	D525	>900		-----	
463	D525	>900		-----	
494	ISO7536	1317.33		-----	
496	ISO7536	>900		-----	
862	D525	>900		-----	
970		-----		-----	
1047	ISO7536	>360		-----	
1161	ISO7536	>900		-----	
1446		-----		-----	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727		-----		-----	
1835		-----		-----	

normality	n.a.
n	9
outliers	0
mean (n)	>360
st.dev. (n)	n.a.
R(calc.)	n.a.
R(ISO7536:94)	n.a.

Determination of Ethanol and higher saturated alcohols on sample #16083; results in %V/V

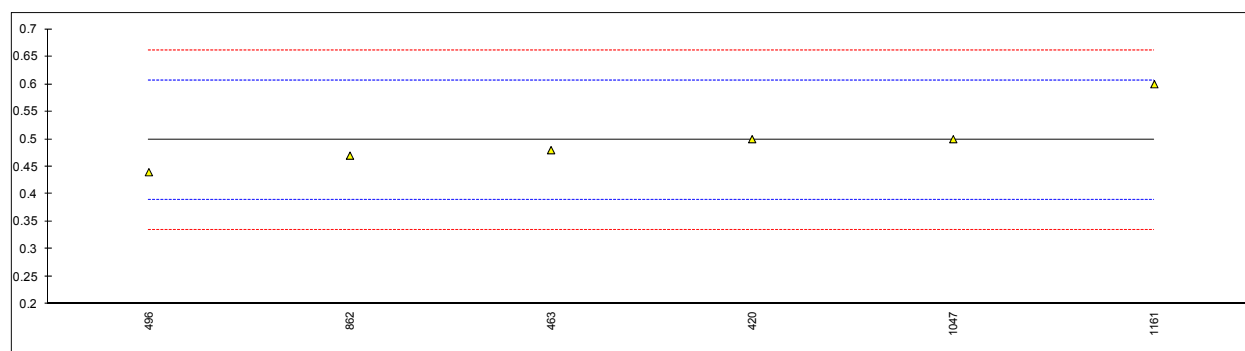| lab	method	value	mark	z(targ)	remarks
334	ISO22854	79.49		0.44	
340		-----		-----	
420	EN13132	77.9		-0.42	
447	EN13132	83.94	D(0.05)	2.84	
463	EN13132	79.99		0.71	
494		-----		-----	
496	EN1601	79.55		0.47	
862	D5501	77.90		-0.42	
970		-----		-----	
1047	EN1601	78.5		-0.09	
1161	EN13132	78.8		0.07	
1446		-----		-----	did not report result in %V/V but reported: 78.15%M/M (EN1601/EN13132)
1459		76.71		-1.06	
1634		-----		-----	
1706	In house	80.36		0.91	
1727		-----		-----	
1835	In house	77.55		-0.61	

normality	OK
n	10
outliers	1
mean (n)	78.675
st.dev. (n)	1.1725
R(calc.)	3.283
R(EN1601:14)	5.186
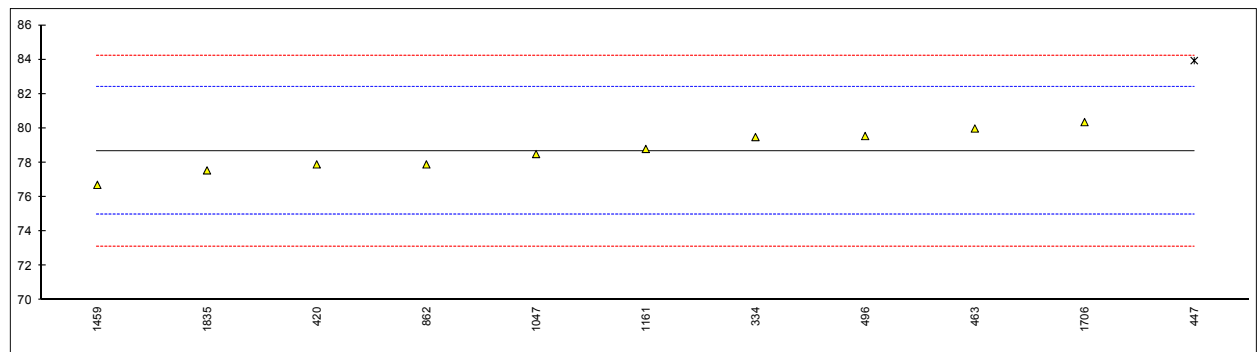
Determination of Ethers (5 or more C atoms) on sample #16083; results in %V/V

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340		-----		-----	
420	EN13132	0.5		0.03	
447		-----		-----	
463	EN13132	0.48		-0.34	
494		-----		-----	
496	EN1601	0.44		-1.08	
862	D4815	0.47		-0.52	
970		-----		-----	
1047	EN1601	0.5		0.03	
1161	EN13132	0.60		1.87	
1446		-----		-----	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727		-----		-----	
1835		-----		-----	

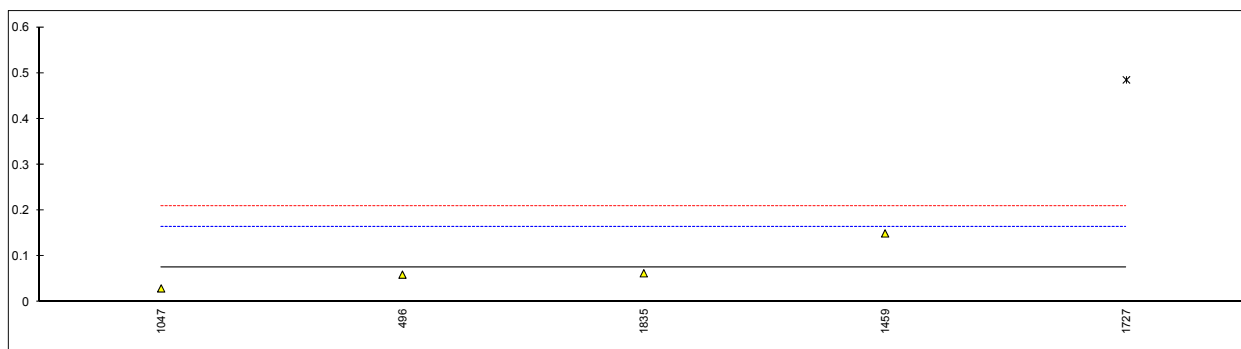|	
normality	unknown
n	6
outliers	0
mean (n)	0.4983
st.dev. (n)	0.05456
R(calc.)	0.1528
R(EN1601:14)	0.1519

Determination of Higher saturated monoalcohols (C3-C5) on sample #16083; results in %V/V

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340		-----		-----	
420		-----		-----	
447		-----		-----	
463	EN13132	<0,2		-----	
494		-----		-----	
496	EN1601	0.07		-----	
862		-----		-----	
970		-----		-----	
1047		-----		-----	
1161	EN13132	<0,17		-----	
1446		-----		-----	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727		-----		-----	
1835		-----		-----	

normality	n.a.
n	3
outliers	0
mean (n)	<0.2
st.dev. (n)	n.a.
R(calc.)	n.a.
R(EN1601:14)	n.a.

Determination of Methanol on sample #16083; results in %V/V

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340		-----		-----	
420	EN13132	<0,1		-----	
447		-----		-----	
463	EN13132	<0,2		-----	
494		-----		-----	
496	EN1601	0.06		-0.35	
862	D4815	<0.01		-----	
970		-----		-----	
1047	EN1601	0.03		-1.03	
1161	EN13132	<0,17		-----	
1446		-----		-----	
1459		0.15		1.67	
1634		-----		-----	
1706		-----		-----	
1727	D5501	0.485	C,D(0.05)	9.19	first reported: 0.395; a false positive test result?
1835	In house	0.063		-0.29	

|	
normality	unknown
n	4
outliers	1
mean (n)	0.07575
st.dev. (n)	0.051694
R(calc.)	0.14474
R(EN1601:14)	0.12468

Determination of Total Organically Bound Oxygen on sample #16083; results in %M/M

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340		-----		-----	
420	EN13132	27.6		-0.18	
447	EN13132	30.76	D(0.01)	3.13	
463	EN13132	28.30		0.55	
494		-----		-----	
496	EN1601	27.695		-0.08	
862		-----		-----	
970		-----		-----	
1047	EN1601	27.81		0.04	
1161	EN13132	27.9		0.13	
1446		-----		-----	
1459		27.33		-0.46	
1634		-----		-----	
1706		-----		-----	
1727		-----		-----	
1835		-----		-----	

normality	unknown
n	6
outliers	1
mean (n)	27.772
st.dev. (n)	0.3248
R(calc.)	0.909
R(EN1601:14)	2.671
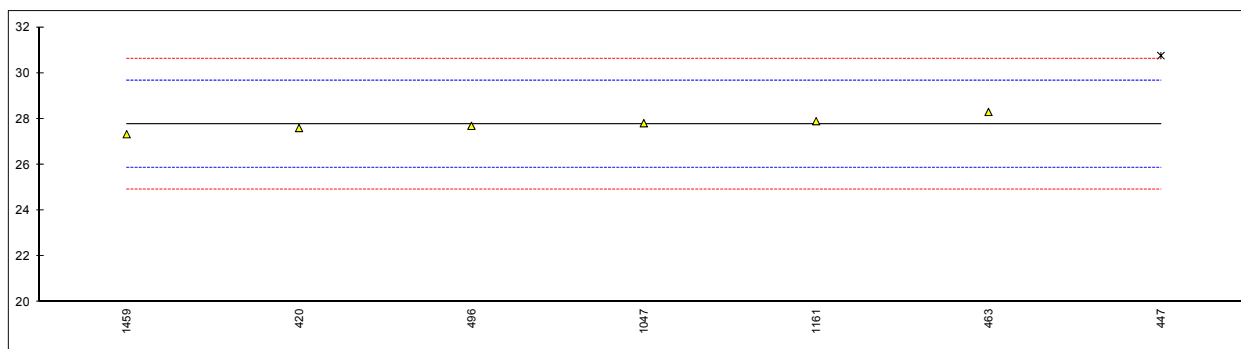
Determination of pHe on sample #16083;

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340	EN15490	7.12	ex	2.83	used KCl electrode
420		-----		-----	
447		-----		-----	
463		-----		-----	
494		-----		-----	
496	EN15490	7.21		3.23	used LiCl electrode
862		-----		-----	
970		-----		-----	
1047	EN15490	6.54		0.22	used LiCl electrode
1161	EN15490	6.075		-1.87	used LiCl electrode
1446	EN15490	6.28		-0.95	used LiCl electrode
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727	EN15490	6.24		-1.13	used LiCl electrode
1835	EN15490	6.6		0.49	used LiCl electrode

|	
normality	unknown
n	6
outliers	0+1ex
mean (n)	6.4908
st.dev. (n)	0.40294
R(calc.)	1.1282
R(EN15490:07)	0.6231
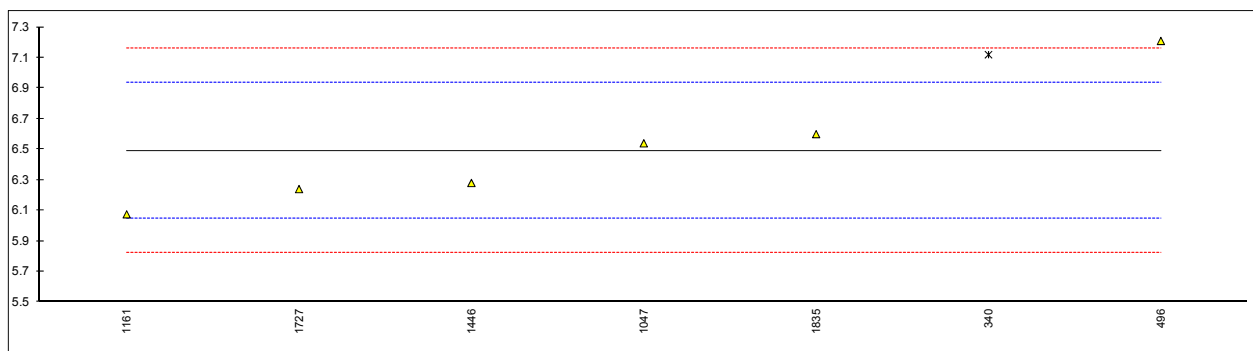
Determination of Phosphorus as P on sample #16083; results in mg/L

lab	method	value	mark	z(targ)	remarks
334		-----		-----	
340		-----		-----	
420	EN15487	<0,1		-----	
447		-----		-----	
463		-----		-----	
494		-----		-----	
496	EN15487	0.041		-----	
862		-----		-----	
970		-----		-----	
1047		-----		-----	
1161		-----		-----	
1446		-----		-----	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727		-----		-----	
1835	EN15487	<0.15		-----	
	normality	n.a.			
	n	3			
	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 #16083; results in mg/kg

lab	method	value	mark	z(targ)	remarks
334	EN15492	0.55		-----	
340		-----		-----	
420		-----		-----	
447		-----		-----	
463		-----		-----	
494		-----		-----	
496	EN15492	0.4		-----	
862		-----		-----	
970		-----		-----	
1047		-----		-----	
1161		-----		-----	
1446		-----		-----	
1459		-----		-----	
1634		-----		-----	
1706		-----		-----	
1727		-----		-----	
1835	EN15492	0.13		-----	

normality	unknown
n	3
outliers	0
mean (n)	0.36
st.dev. (n)	0.213
R(calc.)	0.60
R(EN15492:12)	(0.23)

Application range 0.5 – 20 mg/kg
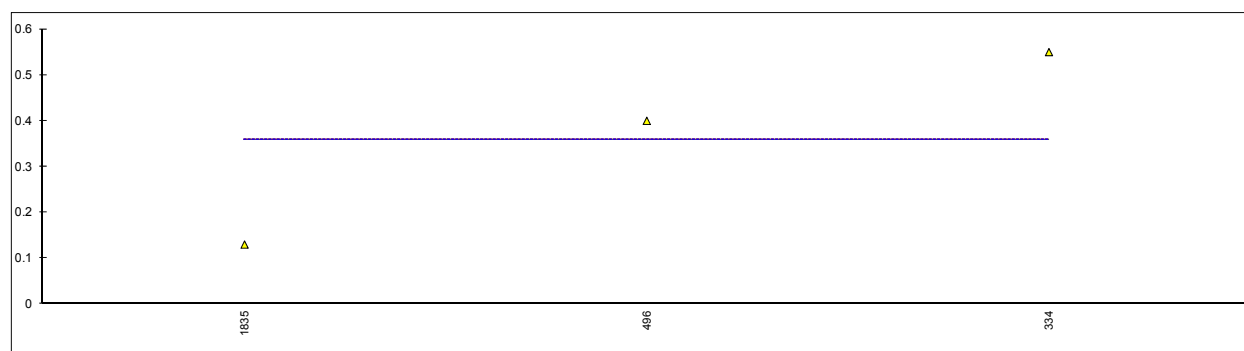
Determination of Sulphur as S on sample #16083; results in mg/kg

lab	method	value	mark	z(targ)	remarks
334	EN15486	2		-0.31	
340		-----		-----	
420	EN15486	2.43		0.29	
447	ISO20846	<3		-----	
463	D5453	1.75		-0.65	
494	EN15486	2.4		0.25	
496	EN15486	2.08		-0.20	
862	D5453	1.88		-0.47	
970		-----		-----	
1047	EN15486	2.4		0.25	
1161	ISO20846	2.3		0.11	
1446		-----		-----	
1459		-----		-----	
1634	ISO20846	2.1		-0.17	
1706	ISO20846	2.7		0.66	
1727		-----		-----	
1835	EN15486	2.4		0.25	

normality	OK	
n	11	
outliers	0	
mean (n)	2.222	
st.dev. (n)	0.2823	
R(calc.)	0.790	
R(EN15486:07)	2.025	Application range: 5 – 20 mg/kg

Kernel Density
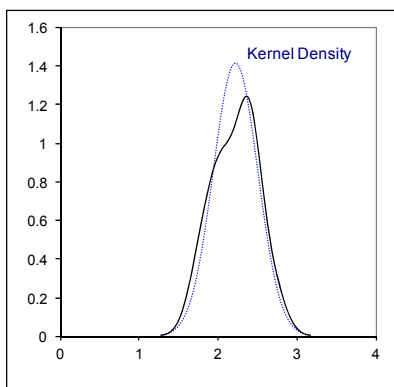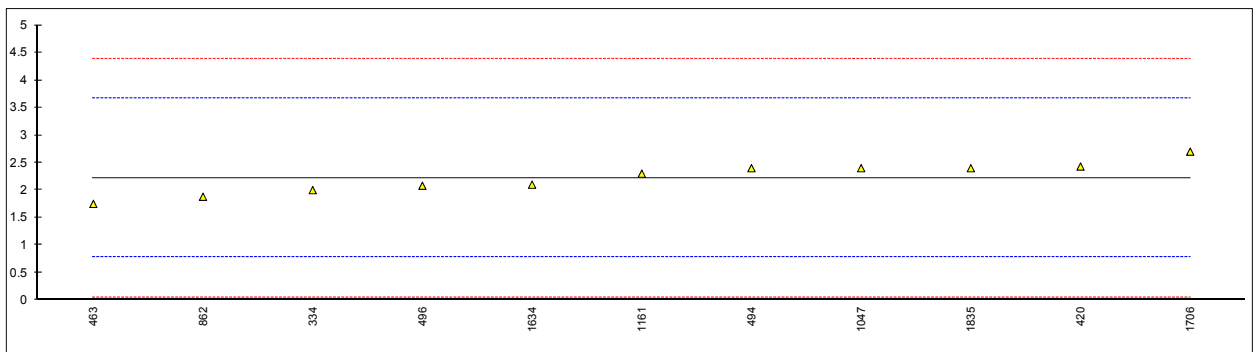
Determination of Water, coulometric on sample #16083; results in %M/M

lab	method	value	mark	z(targ)	remarks
334	EN15489	0.194		0.27	
340	EN15489	0.1924		0.07	
420	EN15489	0.201		1.14	
447	IP438	0.193		0.14	
463	D6304	0.19085		-0.13	
494	EN15489	0.1894		-0.31	
496	EN15489	0.2166	G(0.01)	3.09	
862	D6304	0.1898		-0.26	
970		-----		-----	
1047		-----		-----	
1161	EN15489	0.1899063		-0.24	
1446	ISO760	0.1838		-1.01	
1459	ISO12937	0.1968		0.62	
1634	ISO12937	0.1851		-0.84	
1706		-----		-----	
1727	EN15489	0.1911		-0.09	
1835	EN15489	0.1970		0.64	

normality	OK
n	13
outliers	1
mean (n)	0.1919
st.dev. (n)	0.00472
R(calc.)	0.0132
R(EN15489:07)	0.0224
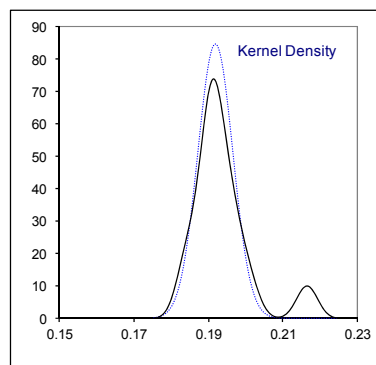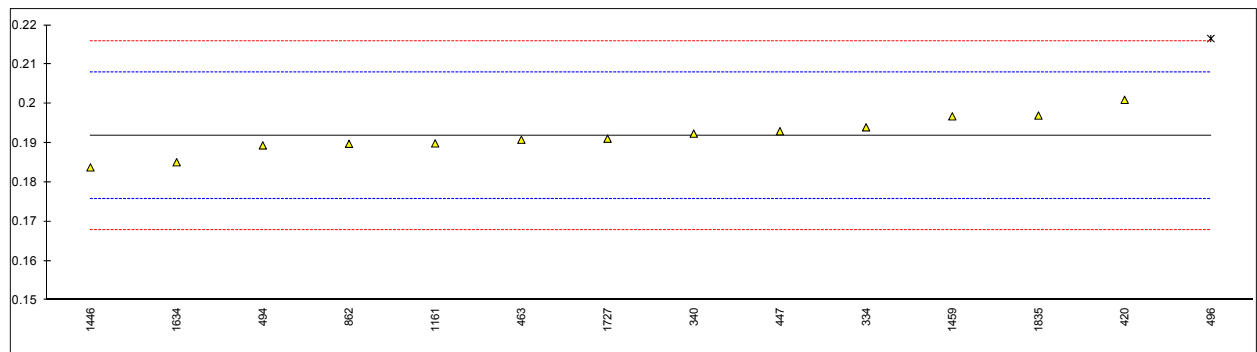
APPENDIX 2

Number of participants per country

    1 lab in AUSTRIA
    1 lab in CHINA, People's Republic
    2 labs in CZECH REPUBLIC
    3 labs in FRANCE
    2 labs in GERMANY
    1 lab in OMAN
    1 lab in POLAND
    1 lab in PORTUGAL
    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 calculations
n.a.   = not applicable
n.e.   = not evaluated
n.d.   = not detected
fr.   = first reported
SDS   = Safety Data Sheet

Literature

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18.   M.A. Gonçalves et al., Evaluation study of different glass electrodes by an interlaboratory comparison for determining the pH of fuel ethanol, Sensors and Actuators B 158 pp 327-332 (2011)