

Results of Proficiency Test
Biogasoline E85
May 2013

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

On request of several participants, the Institute for Interlaboratory Studies decided to organise a proficiency test for the analysis of Biogasoline E85 during the annual proficiency testing program 2012/2013. In this interlaboratory study 16 laboratories in 11 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the 2013 Biogasoline E85 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 the two different test scopes of ASTM D5798 and of EN15293. 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 and ILAC-G13:2007. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality 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 January 2010 (iis-protocol, version 3.2). 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 65 litres of Biogasoline E85 was purchased at a local pump station. After homogenisation, the material was transferred into 48 brown glass bottles of 1 litre (labelled #13072). The homogeneity of the subsamples #13072 was checked by determination of Density @15°C in accordance with ASTM D4052:11 on 7 stratified randomly selected samples.

	Density @ 15°C in kg/m ³
Sample #13072-1	757.94
Sample #13072-2	757.96
Sample #13072-3	757.92
Sample #13072-4	757.94
Sample #13072-5	757.96
Sample #13072-6	757.97
Sample #13072-7	758.02

table 1: homogeneity test results of subsamples #13072

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 @ 15 °C in kg/m ³
r (sample #13072)	0.09
reference test	ISO12185:96
0.3*R (reference test)	0.15

table 2: evaluation of repeatability of the subsamples #13072

The calculated repeatability is less than 0.3 times the reproducibility of the corresponding reference method. Therefore, homogeneity of the subsamples #13072 was assumed.

To the participants: one 1L bottle with sample #13072 was sent on May 8, 2013.

2.5 ANALYSIS

The participants were requested to determine on sample #13072: Acidity, Copper, Copper Corrosion, Density, Electric Conductivity, Existent Gum, Inorganic Chloride (mg/l and mg/kg), Oxidation Stability, pHe, Phosphorous, Sulphur, Water, Ethanol (%M/M and %V/V), Ether (C5 – Higher), Methanol, Higher saturated monoalcohols (C3-C8), Total organic bound oxygen.

To get comparable results a detailed report form, on which the units were prescribed as well as some of the required standards and a letter of instructions were prepared and 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 gathered. The original data are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers. Directly after the deadline, a reminder fax was sent to the laboratories that had not reported 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 results. 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 January 2010 (iis-protocol, version 3.2).

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. 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 to 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. 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 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 analysis results are plotted. The corresponding laboratory numbers are under 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 "x". 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).

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

The z-scores were calculated in accordance with:

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

The $Z_{(\text{target})}$ 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:

$ 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. Not all the laboratories were able to perform all requested analyses. Finally, 16 laboratories did report 110 numerical results. Observed were 5 outlying results, which is 4.5 %. 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. For the determination of Copper, Methanol, Higher saturated monoalcohols (C3 – C5) and Total Organic Bound Oxygen too few test results were reported for a meaningful statistical evaluation.

The other data sets proved to have a normal distribution.

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

Copper as Cu: No significant conclusions were drawn. One numerical result was reported and three other participants agreed on a value near or below the detection limit.

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

Density @15°C: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is nearly in agreement with the requirements of ISO12185:96.

Electric Conductivity: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outlier is not 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 ISO6246:97.

Inorganic Chloride: This determination was not problematic. The results of this determination were reported in mg/l (EN15492:08) and in mg/kg (EN15492:12). The application ranges are 0.8 – 2.0 mg/l and 1.0 - 2.5 mg/kg. Both consensus values were below the application ranges, but the calculated reproducibilities are both in agreement with the requirements.

Oxidation stab.: In this determination no problems have been observed. All reporting participants agreed on a result above 360 minutes.

pHe: This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of EN15490:07.

Phosphorus: Only one numerical test result was reported. All the other participants agreed on a value below the application range. Therefore no significant conclusions were drawn.

Sulphur: This determination may be not problematic. One statistical outlier was observed. Although the consensus value is below the application range of EN15486:07 (5 – 20 mg/kg), the calculated reproducibility, after rejection of the statistical outlier, 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: This determination (%M/M and %V/V) was problematic. Both results of one laboratory appeared to be a statistical outlier. The calculated reproducibilities, after rejection of the statistical outliers are both not in agreement with the requirements of ASTM D5501:12.

Organic oxygenate compounds according to EN1601:97

The gas chromatographic determination of Organic compounds according to EN1601:97 may be not optimized for Biogasoline E85. Oxygenated compounds with low content in diluted E85 fuel could not be identified with the regular test method. The determinations are still being investigated in terms of correct application and precision (see EN15293:11, p3,10).

Ether (C5 – Higher): This determination may be problematic. Only four laboratories reported a test result. The calculated reproducibility is not in agreement with the requirements of EN1601:97.

Methanol: This determination may be very problematic. Three laboratories reported the presence and three laboratories reported the absence of methanol.

Higher sat. alcohols: Only one numerical test result was reported. Therefore no significant conclusions were drawn.

Tot. org. bound oxygen: Only three laboratories reported a test result. When the theoretical values were estimated from the other reported test results, the determination appears to be very problematic. The estimated reproducibility after rejection of one statistical outlier is not at all in agreement with the requirements of EN1601:97.

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	%M/M	10	0.0014	0.0014	0.0015
Copper as Cu	mg/kg	4	n.e.	n.e.	n.e.
Copper corr. 3 hrs @ 50°C	-----	11	1 (1a)	n.e.	n.e.
Density @ 15°C	kg/m ³	13	779.29	0.56	0.50
Electric Conductivity	µS/cm	6	1.19	0.27	0.21
Existent Gum (washed)	mg/100mL	8	0.6	0.8	0.8
Inorganic Chloride	mg/L	5	0.54	0.15	0.37
Inorganic Chloride	mg/kg	5	0.63	0.54	0.55
Oxidation Stability	min.	8	>360	n.e.	n.e.
pHe		8	7.35	2.04	0.71
Phosphorous	mg/L	7	<0.15	n.e.	n.e.
Sulphur	mg/kg	7	1.37	0.46	1.89
Water	%M/M	13	0.19	0.023	0.022
Ethanol	%M/M	7	74.26	1.70	1.19
	%V/V	8	72.89	1.54	1.20
Organic oxygenate comp.					
- Ether content	%V/V	4	0.20	0.23	0.10
- Methanol	%V/V	3	n.e.	n.e.	n.e.
- Higher saturated monoalc.	%V/V	3	<0.2	n.e.	n.e.
-Total Organic Bound Oxygen	%M/M	3	26.6	n.e.	(0.3)

table 3: performance evaluation sample #13072

Without further statistical calculations, it can be concluded that for several tests there is not 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 2013 WITH PREVIOUS PTS

Determination	May 2013	May 2012	May 2011	May 2010
Number of reporting labs	16	14	20	17
Number of results reported	110	103	155	150
Statistical outliers	5	3	8	9
Percentage outliers	4.5 %	2.9 %	5.2 %	6.0 %

table 4: comparison with previous proficiency tests

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency tests was compared against the requirements of the respective standards. The conclusions are given the following table:

Determination	May 2013	May 2012	May 2011	May 2010
Acidity as acetic acid	+/-	--	+/-	+/-
Copper as Cu	n.e.	n.e.	n.e.	n.e.
Copper corr. 3 hrs @ 50°C	++	++	++	++
Density @ 15°C	+/-	-	+/-	--
Electric Conductivity	-	n.e.	n.e.	n.e.
Existent Gum (washed)	(+/-)*	(--)*	(+)*	-
Inorganic Chloride	+	n.e.	++	++
Oxidation Stability	n.e.	n.e.	n.e.	n.e.
pHe	--	--	--	--
Phosphorous	n.e.	n.e.	(-)*	(--)*
Sulphur	++	++	++	++
Water	+/-	+/-	++	++
Ethanol	--	--	--	--
Organic oxygenate comp.				
-Ether content	--	n.e.	--	n.e.
-Methanol	n.e.	++	n.e.	n.e.
-Higher saturated monoalc.	n.e.	n.e.	n.e.	n.e.
-Total organic bound oxygen	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

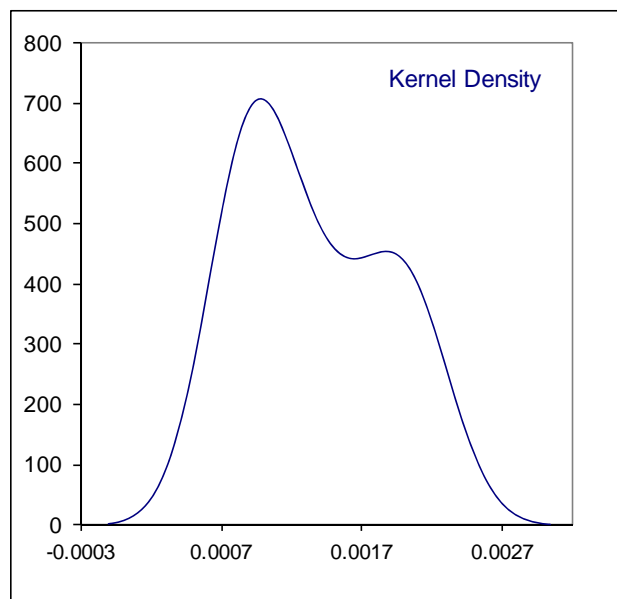
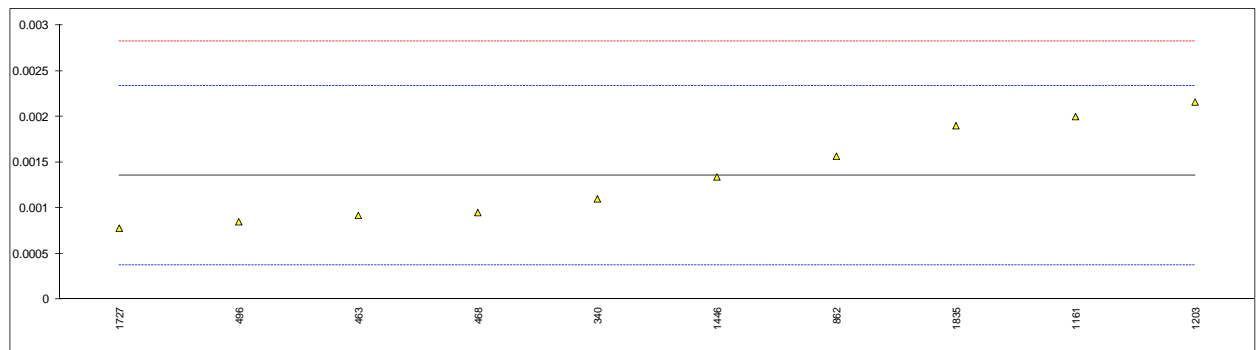
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

- ++: 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 #13072; results in %M/M

lab	method	value	mark	z(targ)	remarks
340	EN15491	0.0011		-0.52	
343	EN15491	<0.003		----	
420		----		----	
441		----		----	
447		----		----	
463	D1613	0.00092		-0.89	
468	EN15491	0.00095		-0.83	
496	EN15491	0.00085		-1.04	
862	D7795	0.001567		0.43	
1161	EN15491	0.002		1.31	
1203	EN15491	0.00216		1.64	
1446	EN15491	0.00134		-0.03	
1634		----		----	
1706		----		----	
1727	EN15491	0.00078		-1.18	
1835	EN15491	0.0019		1.11	
normality		OK			
n		10			
outliers		0			
mean (n)		0.00136			
st.dev. (n)		0.000517			
R(calc.)		0.00145			
R(EN15491:07)		0.00137			



Determination of Copper on sample #13072; results in mg/kg

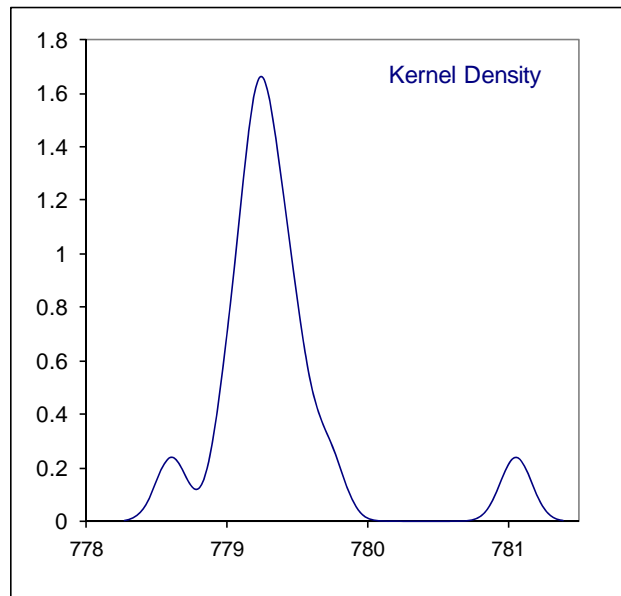
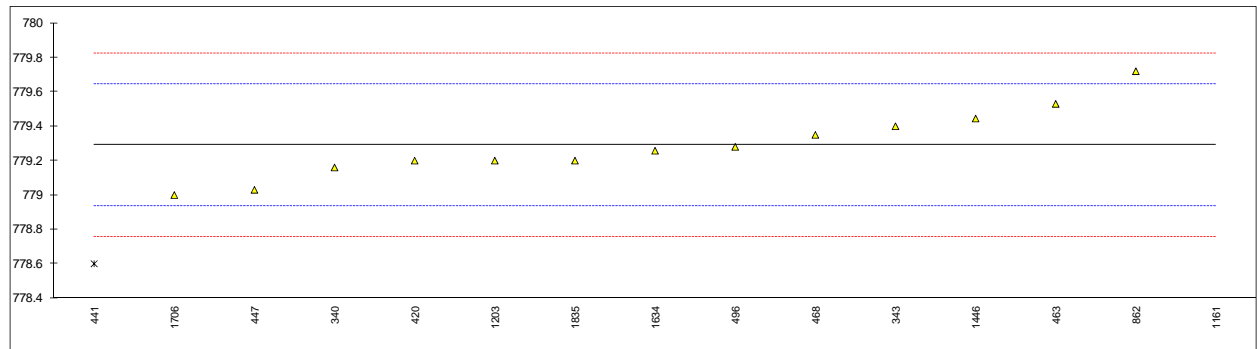
lab	method	value	mark	z(targ)	remarks
340		----		----	
343	EN15488	<0.070		----	
420	EN15488	0.16		----	
441		----		----	
447		----		----	
463		----		----	
468	EN15488	<0.07		----	
496		----		----	
862	EN15488	<0.005		----	
1161		----		----	
1203		----		----	
1446		----		----	
1634		----		----	
1706		----		----	
1727		----		----	
1835		----		----	
	normality	n.a.			
	n	4			
	outliers	n.a.			
	mean (n)	n.a.			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(EN15488:07)	n.a.			

Determination of Copper Corrosion 3hrs/50°C on sample #13072; rating

lab	method	value	mark	z(targ)	remarks
340	ISO2160	1A		----	
343	ISO2160	1A		----	
420	ISO2160	1A		----	
441		----		----	
447	D130	1A		----	
463	ISO2160	1A		----	
468	ISO2160	1A		----	
496	D130	1A		----	
862	D130	1A		----	
1161	ISO2160	1A		----	
1203	ISO2160	1		----	
1446		----		----	
1634	ISO2160	1A		----	
1706		----		----	
1727		----		----	
1835		----		----	
	normality	n.a.			
	n	11			
	outliers	n.a.			
	mean (n)	1 (1a)			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(ISO2160:98)	n.a.			

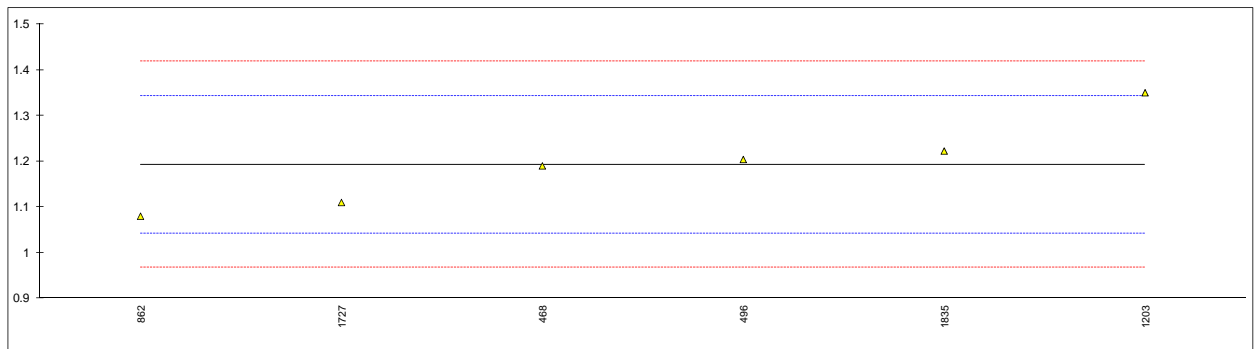
Determination of Density @ 15°C on sample #13072; results in kg/m³

lab	method	value	mark	z(targ)	remarks
340	ISO12185	779.16		-0.73	
343	ISO12185	779.4		0.62	
420	ISO12185	779.2		-0.50	
441	D4052	778.6	G(0.05)	-3.86	
447	IP365	779.03		-1.46	
463	ISO12185	779.53		1.34	
468	ISO12185	779.35		0.34	
496	ISO12185	779.28		-0.06	
862	ISO12185	779.72		2.41	
1161	ISO12185	781.05	G(0.01)	9.86	
1203	ISO12185	779.20		-0.50	
1446	ISO12185	779.445		0.87	
1634	ISO12185	779.2575		-0.18	
1706	ISO12185	779.0		-1.62	
1727		----		----	
1835	D4052	779.2		-0.50	
normality		OK			
n		13			
outliers		2			
mean (n)		779.290			
st.dev. (n)		0.1992			
R(calc.)		0.558			
R(ISO12185:96)		0.500			



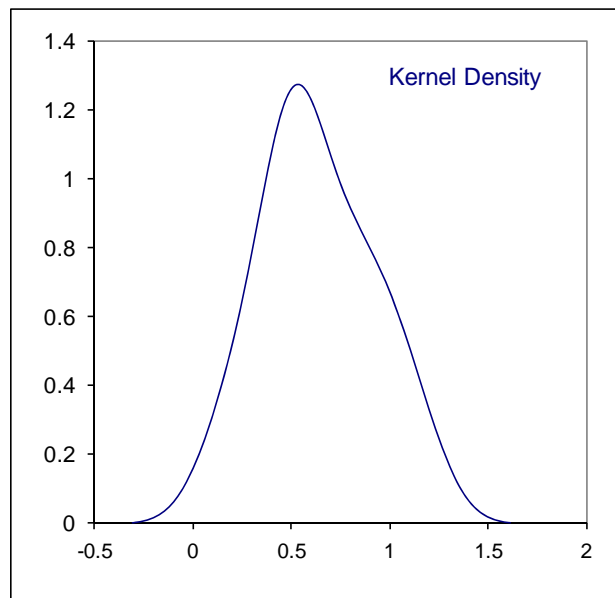
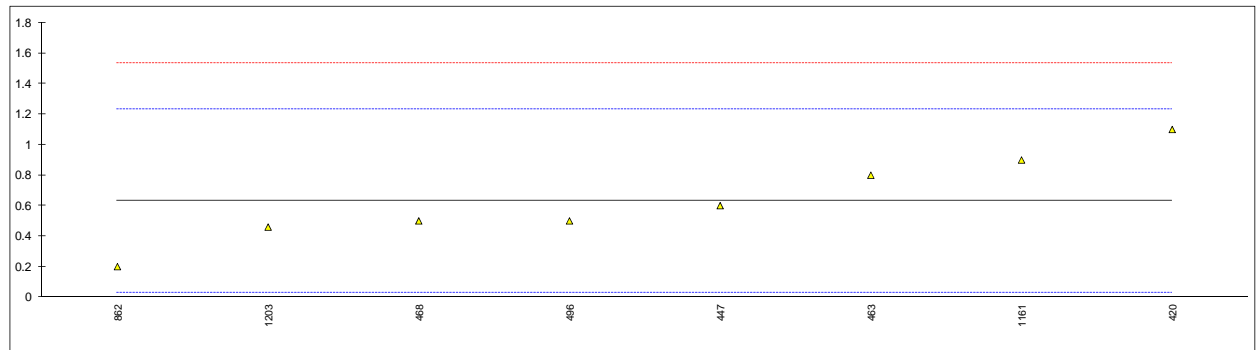
Determination of Electric Conductivity on sample #13072; results in $\mu\text{S}/\text{cm}$

lab	method	value	mark	z(targ)	remarks
340		----		----	
343		----		----	
420		----		----	
441		----		----	
447		----		----	
463		----		----	
468	EN15938	1.19		-0.04	
496	EN15938	1.204		0.15	
862	EN15938	1.080		-1.50	
1161		----		----	
1203	EN15938	1.35		2.09	
1446		----		----	
1634		----		----	
1706		----		----	
1727	EN15938	1.11		-1.10	
1835	EN15938	1.2225		0.40	
normality		OK			
n		6			
outliers		0			
mean (n)		1.193			
st.dev. (n)		0.0952			
R(calc.)		0.267			
R(EN15938:10)		0.211			



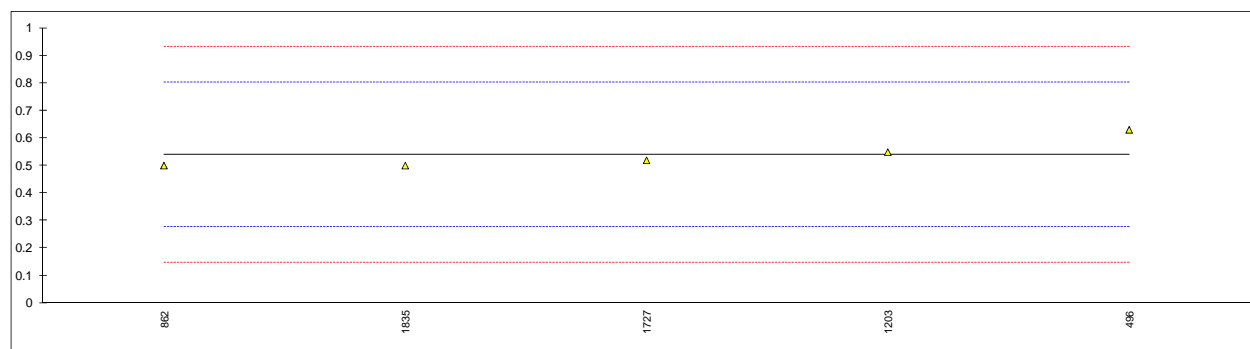
Determination of Existent Gum (washed) on sample #13072; results in mg/100ml

lab	method	value	mark	z(targ)	remarks
340		----		----	
343	ISO6246	<1		----	
420	ISO6246	1.1		1.55	
441		----		----	
447	IP131	0.6		-0.11	
463	ISO6246	0.8		0.56	
468	ISO6246	0.5		-0.44	
496	ISO6246	0.5		-0.44	
862	ISO6246	0.2		-1.44	
1161	ISO6246	0.9		0.89	
1203	ISO6246	0.46		-0.57	
1446		----		----	
1634		----		----	
1706		----		----	
1727		----		----	
1835		----		----	
normality		OK			
n		8			
outliers		0			
mean (n)		0.63			
st.dev. (n)		0.286			
R(calc.)		0.80			
R(ISO6246:97)		0.84			



Determination of Inorganic Chloride on sample #13072; results in mg/L

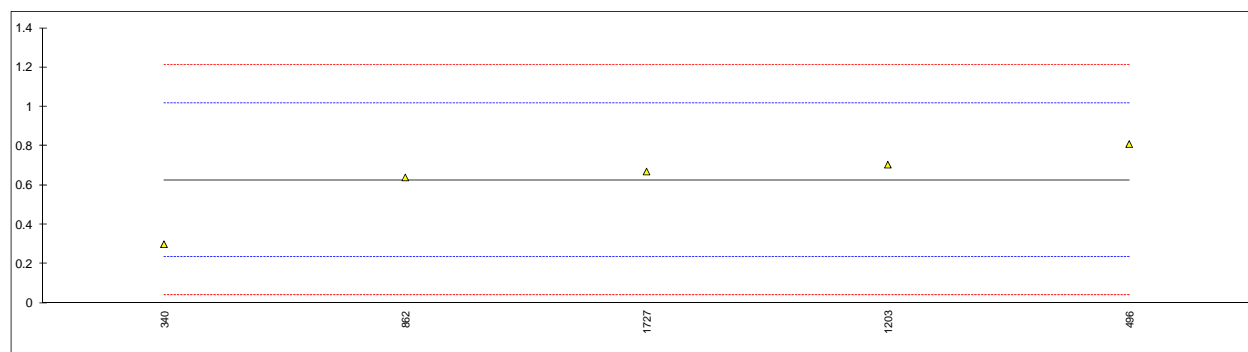
lab	method	value	mark	z(targ)	remarks
340		----		----	
343		----		----	
420		----		----	
441		----		----	
447		----		----	
463		----		----	
468	EN15492	<1.0		----	
496	EN15492	0.63		0.69	
862	EN15492	0.50		-0.30	
1161		----		----	
1203	EN15484	0.549		0.07	
1446		----		----	
1634		----		----	
1706		----		----	
1727	EN15492	0.519		-0.16	
1835	EN15492	0.50		-0.30	
	normality	OK			
	n	5			
	outliers	0			
	mean (n)	0.540			
	st.dev. (n)	0.0544			
	R(calc.)	0.152			
	R(EN15492:08)	0.367			application range: 0.8 – 2.0 mg/L



Determination of Inorganic Chloride on sample #13072; results in mg/kg

lab	method	value	mark	z(targ)	remarks
340	EN15484	0.3		-1.66	
343		----		----	
420		----		----	
441		----		----	
447		----		----	
463		----		----	
468		<1.0		----	
496		0.81		0.95	
862	EN15492	0.64		0.08	
1161		----		----	
1203		0.705		0.41	
1446		----		----	
1634		----		----	
1706		----		----	
1727		0.670		0.23	
1835		----		----	
normality		OK			
n		5			
outliers		0			
mean (n)		0.625			
st.dev. (n)		0.1927			
R(calc.)		0.539			
R(EN15492:12)		0.547			

application range: 1.0 – 2.5 mg/kg

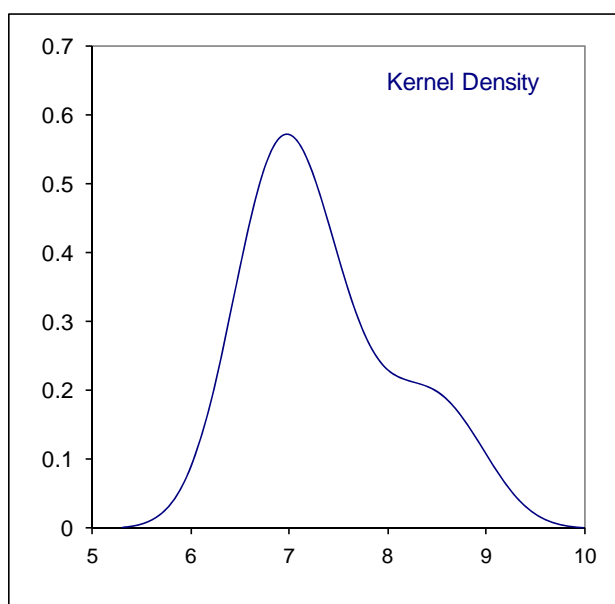
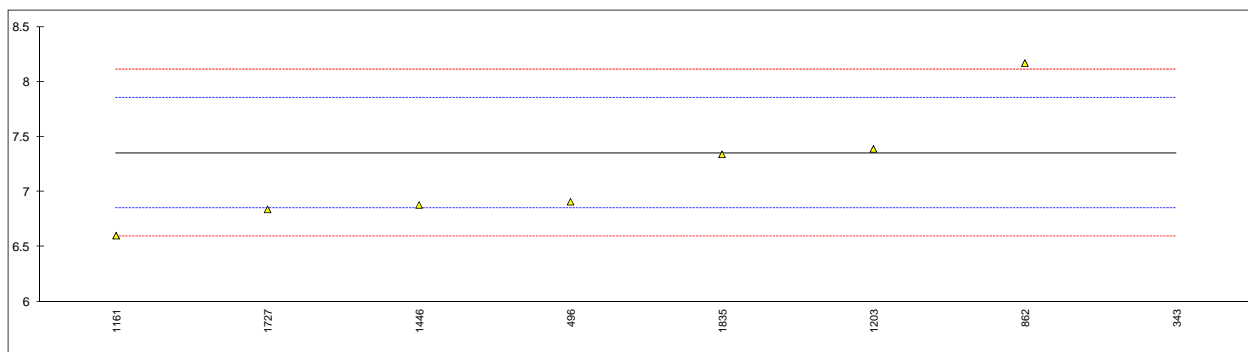


Determination of Oxidation Stability on sample #13072; results in minutes

lab	method	value	mark	z(targ)	remarks
340		----		----	
343	ISO7536	>360		----	
420	ISO7536	>900		----	
441		----		----	
447	ISO7536	>900		----	
463	D525	>360		----	
468		----		----	
496	ISO7536	>1000		----	
862	D525	1738		----	
1161	ISO7536	>900		----	
1203	ISO7536	>900		----	
1446		----		----	
1634		----		----	
1706		----		----	
1727		----		----	
1835		----		----	
	normality	n.a.			
	n	8			
	outliers	n.a.			
	mean (n)	>360			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(ISO7536:94)	n.a.			

Determination of pH_e on sample #13072;

lab	method	value	mark	z(targ)	remarks
340		----		----	
343	EN15490	8.69		5.31	
420		----		----	
441		----		----	
447		----		----	
463		----		----	
468		----		----	
496	EN15490	6.910		-1.76	
862	EN15490	8.17		3.24	
1161	EN15490	6.601		-2.98	
1203	EN15490	7.39		0.15	
1446	EN15490	6.88		-1.87	
1634		----		----	
1706		----		----	
1727	EN15490	6.84		-2.03	
1835	EN15490	7.34		-0.05	
normality		OK			
n		8			
outliers		0			
mean (n)		7.353			
st.dev. (n)		0.7275			
R(calc.)		2.037			
R(EN15490:07)		0.706			

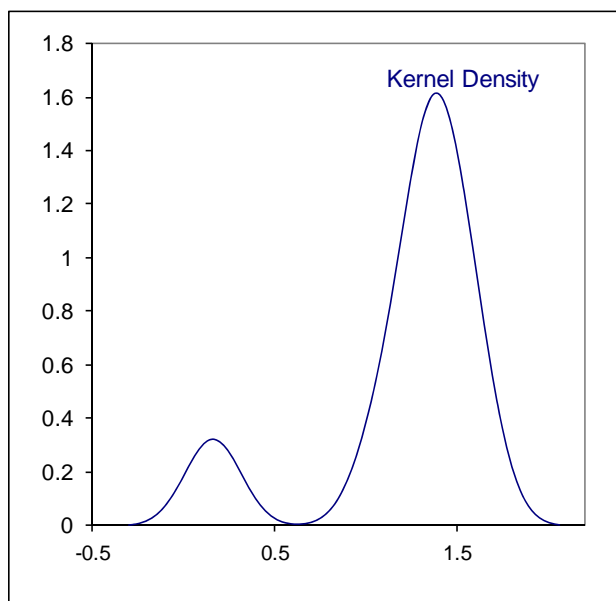
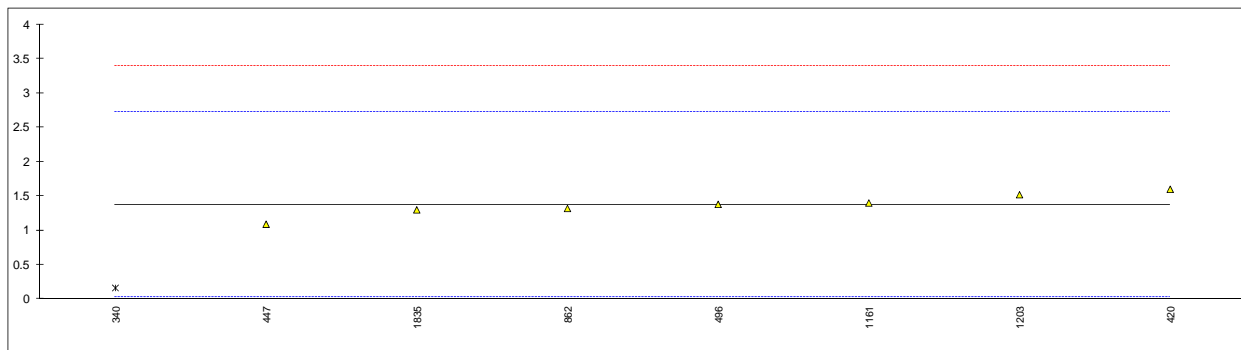


Determination of Phosphorus on #sample #13072; results in mg/L

lab	method	value	mark	z(targ)	remarks
340		----		----	
343	EN15487	<0.15		----	
420		----		----	
441		----		----	
447		----		----	
463		----		----	
468	EN15487	<0.15		----	
496	EN15487	<0.15		----	
862	EN15487	0.04		----	
1161		----		----	
1203	EN15487	<0.15		----	
1446		----		----	
1634		----		----	
1706		----		----	
1727	EN15487	<0.01		----	
1835	EN15487	<0.15		----	
	normality	n.a.			
	n	7			
	outliers	n.a.			
	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

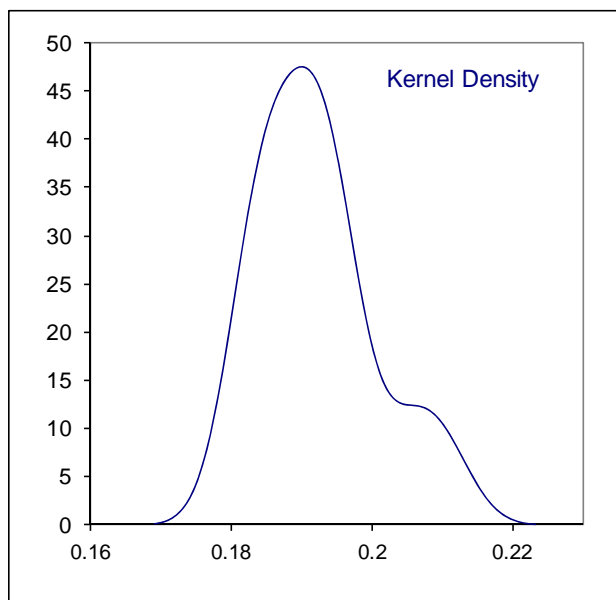
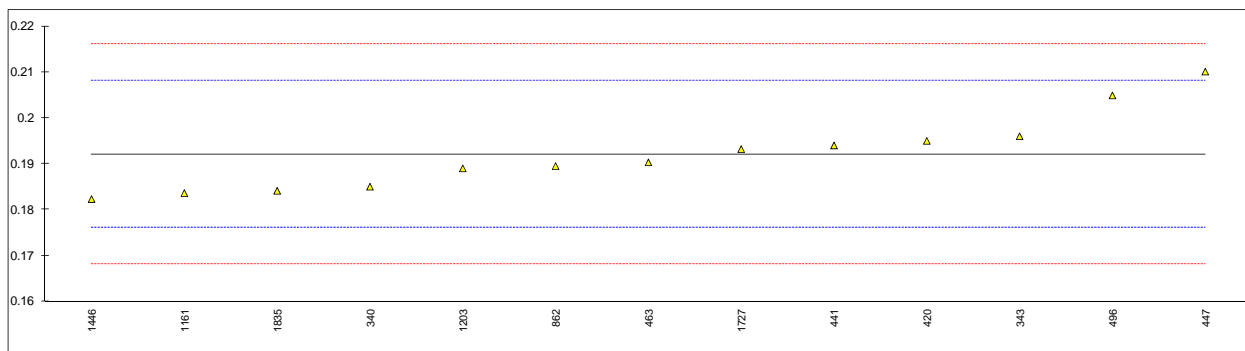
Determination of Sulphur on sample #13072; results in mg/kg

lab	method	value	mark	z(targ)	remarks
340	EN15486	0.16	G(0.01)	-1.80	
343	EN15486	<5		-----	
420	ISO20846	1.6		0.34	
441				-----	
447	IP490	1.09		-0.42	
463	D5453	<1		-----	
468	EN15486	<1.0		-----	
496	EN15486	1.38		0.01	
862	EN15486	1.32		-0.08	
1161	EN20846	1.4		0.04	
1203	EN15486	1.52		0.22	
1446				-----	
1634				-----	
1706				-----	
1727				-----	
1835	EN15486	1.3		-0.11	
normality		OK			
n		7			
outliers		1			
mean (n)		1.373			
st.dev. (n)		0.1644			
R(calc.)		0.460			
R(EN15486:07)		1.890	application range: 5 – 20 mg/kg		



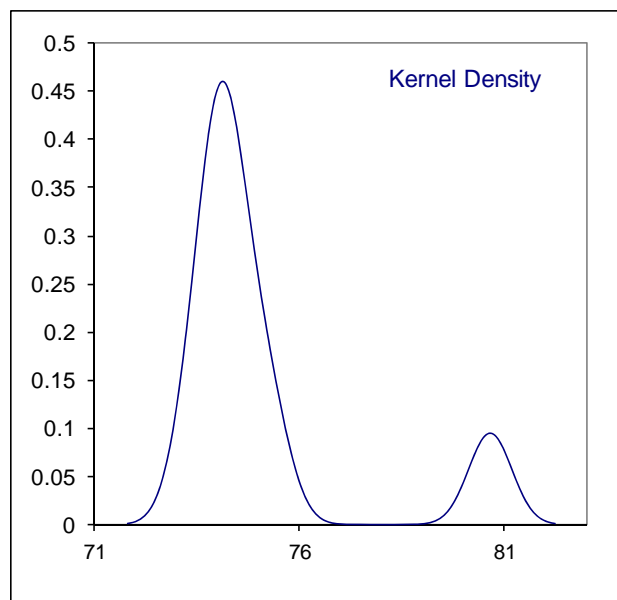
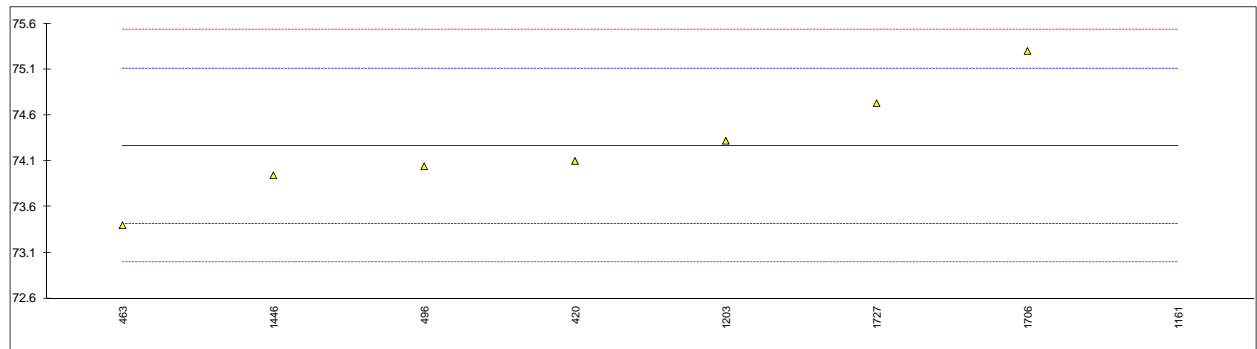
Determination of Water on sample #13072; results in %M/M

lab	method	value	mark	z(targ)	remarks
340	EN15489	0.185		-0.88	
343	EN15489	0.196		0.49	
420	ISO12937	0.195		0.37	
441	EN15489	0.194		0.24	
447	IP438	0.2101		2.25	
463	ISO12937	0.1903		-0.22	
468		----		----	
496	EN15489	0.2049		1.60	
862	EN15489	0.1895		-0.32	
1161	EN15489	0.1836	EN15489	-1.06	
1203	EN15489	0.189		-0.38	
1446	ISO760	0.1823		-1.22	
1634		----		----	
1706		----		----	
1727	EN15489	0.1932		0.14	
1835	EN15489	0.1841		-1.00	
normality		OK			
n		13			
outliers		0			
mean (n)		0.1921			
st.dev. (n)		0.00826			
R(calc.)		0.0231			
R(EN15489:07)		0.0224			



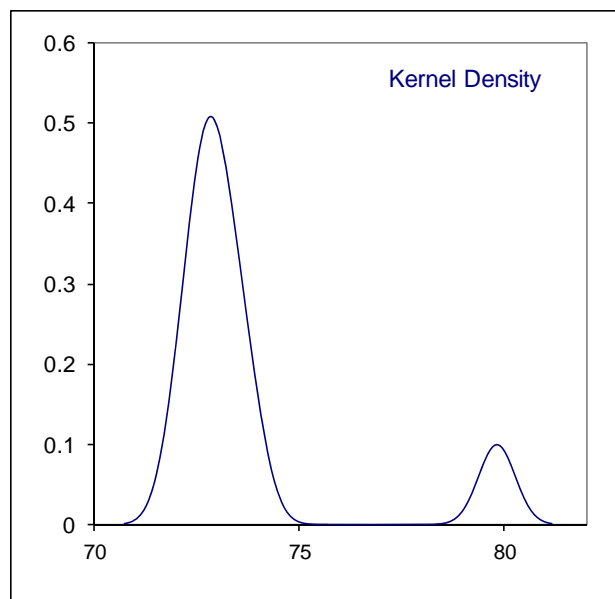
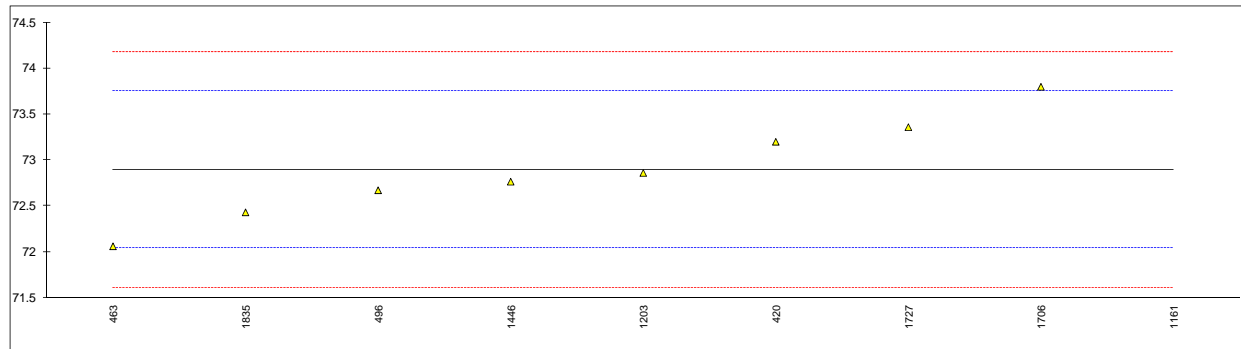
Determination of Ethanol content on sample #13072; results in %M/M

lab	method	value	mark	z(targ)	remarks
340		-----		-----	
343		-----		-----	
420	ISO13132	74.1		-0.38	
441		-----		-----	
447		-----		-----	
463	EN13132	73.40		-2.04	
468		-----		-----	
496	D5501	74.042		-0.52	
862		-----		-----	
1161	EN13132	80.66	G(0.01)	15.12	
1203	EN22854	74.32	C	0.14	first reported 69.61
1446	EN13132	73.945		-0.75	
1634		-----		-----	
1706	EN13132	75.3		2.45	
1727	D5501	74.73	C	1.10	first reported 72.18
1835		-----		-----	
normality		OK			
n		7			
outliers		1			
mean (n)		74.262			
st.dev. (n)		0.6080			
R(calc.)		1.702			
R(D5501:12)		1.185			
				application range: 20 – 100 %M/M	



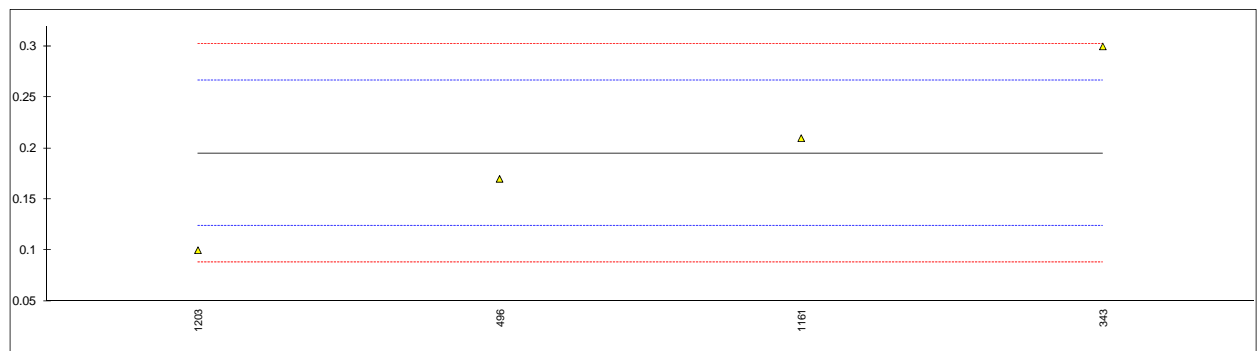
Determination of Ethanol content on sample #13072; results in %V/V

lab	method	value	mark	z(targ)	remarks
340		----		----	
343		----		----	
420	ISO13132	73.2		0.72	
441		----		----	
447		----		----	
463	EN13132	72.06		-1.95	
468		----		----	
496	D5501	72.671		-0.52	
862		----		----	
1161	EN13132	79.8	G(0.01)	16.14	
1203		72.86		-0.08	
1446	EN13132	72.765		-0.30	
1634		----		----	
1706		73.8		2.12	
1727		73.36	C	1.09	first reported 70.86
1835	in house	72.43		-1.08	
normality		OK			
n		8			
outliers		1			
mean (n)		72.893			
st.dev. (n)		0.5490			
R(calc.)		1.537			
R(D5501:12)		1.198			application range: 20 – 100 %V/V



Determination of Ethers (C5 – Higher) on sample #13072; results in %V/V

lab	method	value	mark	z(targ)	remarks
340		----		----	
343	EN13132	0.3		2.94	
420		----		----	
441		----		----	
447		----		----	
463		----		----	
468		----		----	
496	EN1601	0.17		-0.70	
862		----		----	
1161	EN13132	0.21		0.42	
1203	EN22854	0.10		-2.66	
1446		----		----	
1634		----		----	
1706		----		----	
1727		----		----	
1835		----		----	
	normality	OK			
	n	4			
	outliers	0			
	mean (n)	0.20			
	st.dev. (n)	0.083			
	R(calc.)	0.23			
	R(EN1601:97)	0.10			



Determination of Methanol on sample #13072; results in %V/V

lab	method	value	mark	z(targ)	remarks
340		----		----	
343	EN13132	<0.2		----	
420		----		----	
441		----		----	
447		----		----	
463	EN13132	0.34		----	
468		----		----	
496	EN1601	0.17		----	
862		----		----	
1161	EN13132	<0.2		----	
1203	EN22854	<0.02		----	
1446		----		----	
1634		----		----	
1706		----		----	
1727	EN1601	0.654		----	
1835		----		----	
	normality	n.a.			
	n	3			
	outliers	n.a.			
	mean (n)	n.a.			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(EN1601:97)	n.a.			

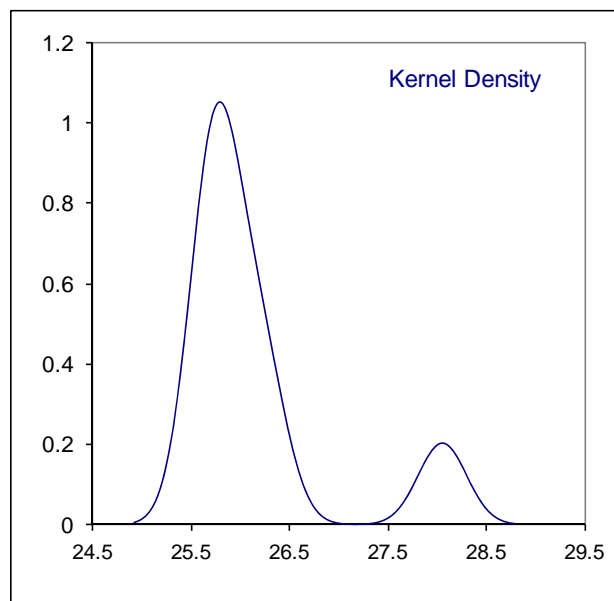
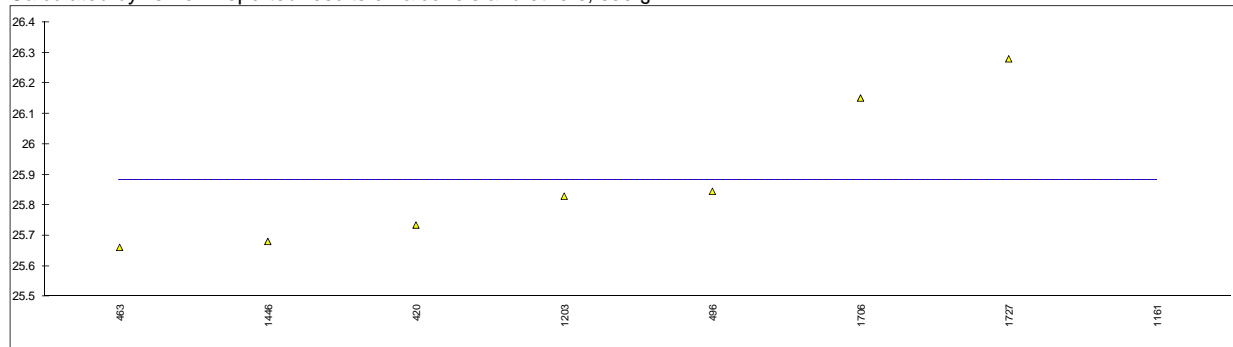
Determination of Higher saturated monoalcohols (C3-C5) on sample #13072; results in %V/V

lab	method	value	mark	z(targ)	remarks
340		----		----	
343		----		----	
420		----		----	
441		----		----	
447		----		----	
463		----		----	
468		----		----	
496	EN1601	0.08		----	
862		----		----	
1161	EN13132	<0.2		----	
1203	EN22854	<0.02		----	
1446		----		----	
1634		----		----	
1706		----		----	
1727		----		----	
1835		----		----	
	normality	unknown			
	n	3			
	outliers	n.a.			
	mean (n)	<0.2			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(EN1601:97)	n.a.			

Determination of Total Org Bound Oxygen on sample #13072; results in %M/M

lab	method	value	mark	z(targ)	remarks	calc. by iis, see §4.1	mark
340		----		----		----	
343		----		----		----	
420		----		----		25.74	
441		----		----		----	
447		----		----		----	
463		----		----		25.66	
468		----		----		----	
496	EN1601	25.845		----		25.85	
862		----		----		----	
1161	EN13132	28.09		----		28.05	G(0.01)
1203	EN22854	25.85	C	----	first reported 24.24	25.83	
1446		----		----		25.68	
1634		----		----		----	
1706		----		----		26.15	
1727		----		----		26.28	
1835		----		----		----	
normality		n.a.				OK	
n		3				7	
outliers		0				1	
mean (n)		26.60				25.88	
st.dev. (n)		n.a.				0.240	
R(calc.)		n.a.				0.67	
R(EN1601:97)		(0.30)			application range 0.17 – 3.7 M/M%	(0.30)	

Calculated by iis from reported results on alcohols and ethers, see §4.1:



APPENDIX 2

Number of participants per country

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

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
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, January 2010
- 2 ASTM E178-02
- 3 ASTM E1301-03
- 4 ISO 5725-86
- 5 ISO 5725, parts 1-6, 1994
- 6 ISO13528-05
- 7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 9 IP 367/84
- 10 DIN 38402 T41/42
- 11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 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/)
- 15 EN15293:11