

Results of Proficiency Test  
Biogasoline E85  
May 2010

Organised by: Institute for Interlaboratory Studies  
Spijkensisse, the Netherlands

Authors: ing R. J. Starink  
Correctors: dr. R.G. Visser & ing. M. Audier  
Report: iis10B02E85

July 2010

**CONTENTS**

1	INTRODUCTION .....	3
2	SET UP .....	3
2.1	QUALITY SYSTEM .....	3
2.2	PROTOCOL.....	3
2.3	CONFIDENTIALY STATEMENT .....	3
2.4	SAMPLES .....	4
2.5	ANALYSES .....	4
3	RESULTS.....	5
3.1	STATISTICS .....	5
3.2	GRAPHICS .....	5
3.3	Z-SCORES .....	6
4	EVALUATION .....	6
4.1	EVALUATION PER TEST .....	6
4.2	PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES.....	8

## Appendices:

1.	Data, statistical results and graphic results.....	9
2.	Number of participants per country .....	24
3.	Abbreviations and literature .....	25

## **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 2009/2010. In this international interlaboratory study 19 laboratories in 13 different countries have participated. See appendix 2 for a list of participants in alphabetical country order. In this report, the results of the Biogasoline E85 proficiency test are presented and discussed.

## **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 EN15293. The analyses for fit-for-use and homogeneity testing were subcontracted. In this proficiency test, the participants received one sample of Biogasoline E85: 1\*1 litre (labelled #1046).

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 guide 43, ISO17043:2010 and ILAC-G13:2007. This ensures 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), which can be downloaded from [www.iisnl.com](http://www.iisnl.com).

### **2.3 CONFIDENTIALITY STATEMENT**

All data present 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, the material was transferred into 58 brown glass bottles of 1 litre (labelled #1046). The homogeneity of the subsamples #1046 was checked by determination of Density @ 15°C in accordance with ASTM D4052:09 and Water in accordance with ASTM E1064:08 on 8 stratified randomly selected samples.

	Density @ 15°C in kg/L	Water in %M/M
Sample #1046-1	0.78474	0.219
Sample #1046-2	0.78472	0.218
Sample #1046-3	0.78473	0.219
Sample #1046-4	0.78475	0.219
Sample #1046-5	0.78474	0.218
Sample #1046-6	0.78479	0.220
Sample #1046-7	0.78483	0.218
Sample #1046-8	0.78483	0.219

table 1: homogeneity test of subsamples #1046

From the above test results, the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density @ 15 °C in kg/L	Water in %M/M
r (sample #1046)	0.00012	0.002
reference test	ISO12185:96	EN15489:08
0.3*R (reference test)	0.00015	0.007

table 2: repeatabilities of the subsamples #1046

The calculated repeatabilities are each less than 0.3 times the reproducibility of the corresponding reference method. Therefore, homogeneity of the subsamples #1046 was assumed.

To the participants: 1\*1 litre of sample #1046 was sent on April 28, 2010.

## 2.5 ANALYSIS

The participants were requested to determine on sample #1046: Acidity, Copper, Copper Strip Corrosion, Density, Existent Gum, Inorganic Chloride, Oxidation Stability, pHe, Phosphorous, Sulphur, Water, Ethanol (%V/V and %M/M), Methanol, Higher Saturated Monoalcohols (C3-C8), Ethers, Oxygen content.

To get comparable results a detailed report form, on which the units were prescribed as well as some of the required standards, was sent together with each set of samples. Also, a letter of instructions and a SDS were added to the 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. 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; nr.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. 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. In total five laboratories reported the results after the final reporting date and two participants decided not to report any results.

Most laboratories reported results, but not all laboratories were able to perform all analyses requested. Finally, 17 laboratories did send in 150 numerical results. Observed were 9 outlying 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. All data sets proved to have a normal distribution.

Acidity: This determination is not problematic. No statistical outliers were observed and the calculated reproducibility is in full agreement with the requirements of EN15491:08.

Copper as Cu: No significant conclusions were drawn as only one numerical result was reported.

- Copper strip: No problems have been observed, all reporting participants agreed on a result of 1.
- Density @15°C: This determination was problematic for several laboratories. Two statistical outliers were observed and the calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ISO12185:96.
- Existent Gum: This determination was problematic at this low level of 0.7 mg/100mL. Although no statistical outliers were observed, the calculated reproducibility is not in agreement with the requirements of ISO6246:97.
- Inorganic Chloride: Due the low concentration of inorganic chloride (0.45 mg/L) in this sample no conclusions were drawn. The application range of EN15492:08 is 4 -30 mg/L.
- Oxidation stab.: In this determination no problems have been observed. All reporting participants agreed on a result above 900 minutes.
- pHe: This determination was very problematic. Only one statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of EN15490:08, nor with the requirements of ASTM D6423:08.
- Phosphorus: Due the low concentration (0.12 mg/L) in this sample no conclusions were drawn. The application range of EN15487:08 is 0.15 – 1.50 mg/L.
- Sulphur: This determination seems not problematic. Although the consensus value is below the application range of EN15486 (5 – 20 mg/kg), the calculated reproducibility is in good agreement with the requirements estimated from EN15486:08. No statistical outliers were observed.
- Water: This determination was problematic for several laboratories. Two statistical outliers and one false negative result were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of EN15489:08.
- Ethanol: This determination (%V/V and %M/M) may be very problematic. In total three statistical outliers were observed. After rejection the statistical outliers, both calculated reproducibilities are not at all in agreement with the requirements of ASTM D5501:09. However, the precision of D5501 were determined using 93-97% ethanol and it may not be valid for 85% ethanol.

Methanol: No significant conclusions were drawn as only a very few numerical results were reported. The reported results vary over a wide range from 0.0079 – 0.3164%V/V.

Higher saturated alcohols: No significant conclusions were drawn as only a few numerical results were reported.

Ethers: No significant conclusions were drawn as only a few numerical results were reported.

Oxygen: This determination was problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the 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	11	0.00161	0.00130	0.00137
Copper as Cu	mg/kg	-	n.a.	n.a.	n.a.
Copper Strip 3 hrs @ 50°C	-----	14	1 (1a)	n.a.	n.a.
Density @ 15°C	kg/m <sup>3</sup>	16	784.82	0.83	0.50
Existent Gum (washed)	mg/100mL	8	0.71	1.17	0.95
Inorganic Chloride	mg/L	6	0.45	0.29	(1.19)
Oxidation Stability	min.	8	>900	n.a.	n.a.
pHe		11	7.10	1.43	0.68
Phosphorous	mg/L	5	0.12	0.15	(0.07)
Sulphur	mg/kg	12	2.39	1.05	2.05
Water	%M/M	13	0.221	0.015	0.023
- Ethanol	%V/V	11	84.175	2.562	0.530
- Ethanol	%M/M	10	85.269	2.073	0.530
- Methanol	%V/V	5	n.a.	n.a.	n.a.
- Higher saturated monoalc.	% V/V	3	n.a.	n.a.	n.a.
- Ethers (C5-highers)	%V/V	7	n.a.	n.a.	n.a.
- Oxygen content	%M/M	7	29.60	0.71	0.30

table 3: performance evaluation sample #1046

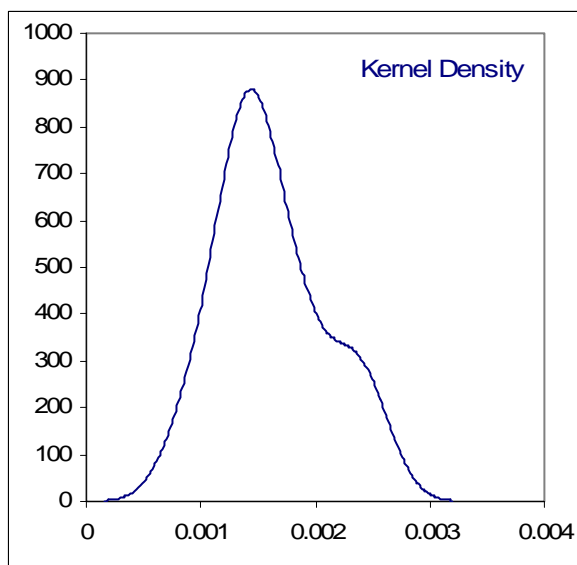
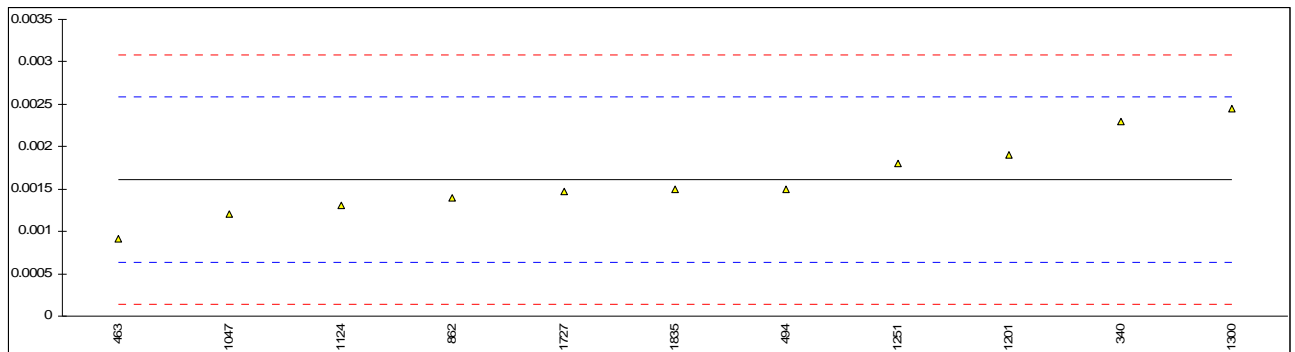
Without further statistical calculations, it can be concluded that for several tests there is not a good compliance of the group of participants with the relevant standards. The problematic tests have been discussed in paragraph 4.1.



**APPENDIX 1**

Determination of Total Acidity as Acetic Acid on sample #1046; results in %M/M

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
340	EN15491	0.0023		1.41	
463	D1613	0.00091		-1.43	
494	EN15491	0.0015		-0.23	
496	EN15491	<0.003		-----	
862	EN15491	0.0014		-0.43	
1017		-----		-----	
1033		-----		-----	
1047	EN15491	0.0012		-0.84	
1080	in house	<0.01		-----	
1121		-----		-----	
1124	EN15491	0.0013		-0.64	
1154		-----		-----	
1201	EN15491	0.0019		0.59	
1251	EN15491	0.0018		0.38	
1300	EN15491	0.00245		1.71	
1706		-----		-----	
1727	EN15491	0.00147		-0.29	
1835	EN15491	0.0015	C	-0.23	First reported 15
normality	OK				
n	11				
outliers	0				
mean (n)	0.00161				
st.dev. (n)	0.000463				
R(calc.)	0.00130				
R(EN15491:08)	0.00137				



## Determination of Copper on sample #1046; results in mg/kg

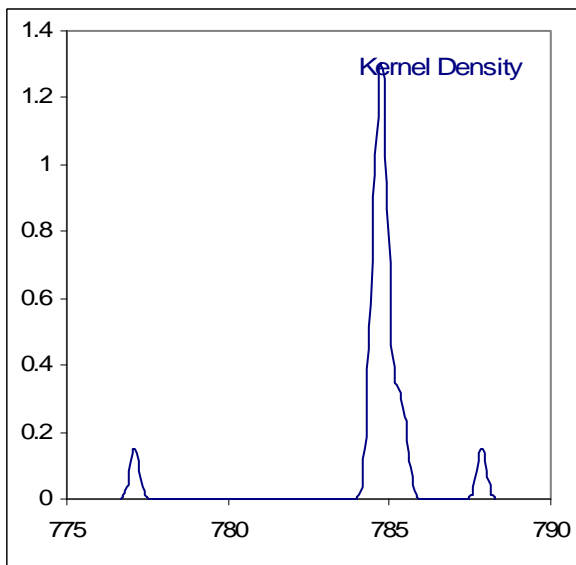
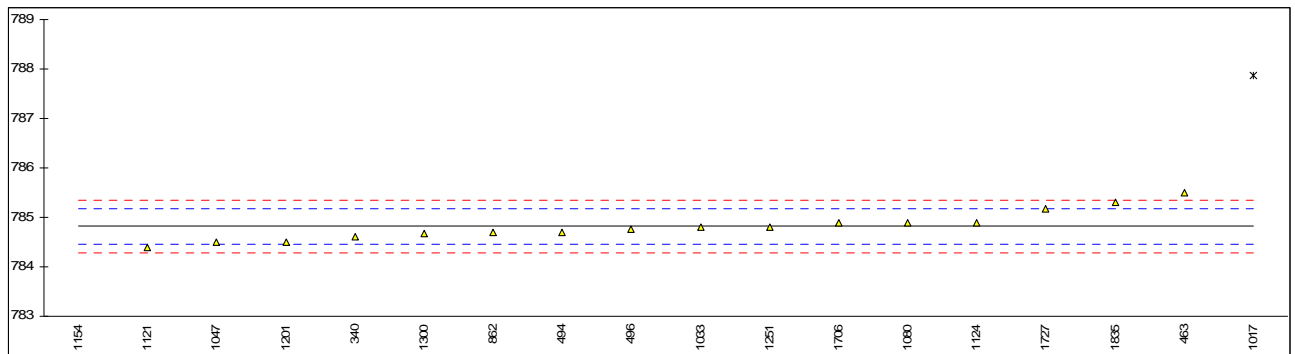
lab	method	value	mark	z(targ)	remarks
323		----		----	
340		----		----	
463		----		----	
494		----		----	
496		----		----	
862		----		----	
1017		----		----	
1033		----		----	
1047		----		----	
1080		----		----	
1121		----		----	
1124	EN15488	0.003		----	
1154		----		----	
1201	EN15488	<0.1		----	
1251		----		----	
1300		----		----	
1706		----		----	
1727		----		----	
1835		----		----	
	normality	n.a.			
	n	1			
	outliers	0			
	mean (n)	n.a.			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(EN15488:08)	n.a.			

## Determination of Copper strip Corrosion 3hrs/50°C on sample #1046; rating

lab	method	value	mark	z(targ)	remarks
323		----		----	
340	ISO2160/D130	1a		----	
463	D130	1a		----	
494	ISO2160	1		----	
496	D130	1a		----	
862	D130	1a		----	
1017	ISO2160/D130	1a		----	
1033	IP154	1a		----	
1047	ISO2160	1a		----	
1080	D130	1a		----	
1121	IP154	1a		----	
1124	ISO2160	1a		----	
1154		----		----	
1201	D130	1a		----	
1251	D130	1a		----	
1300	ISO2160	1a		----	
1706		----		----	
1727		----		----	
1835		----		----	
	normality	n.a.			
	n	14			
	outliers	0			
	mean (n)	1 (1a)			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(ISO2160)	n.a.			

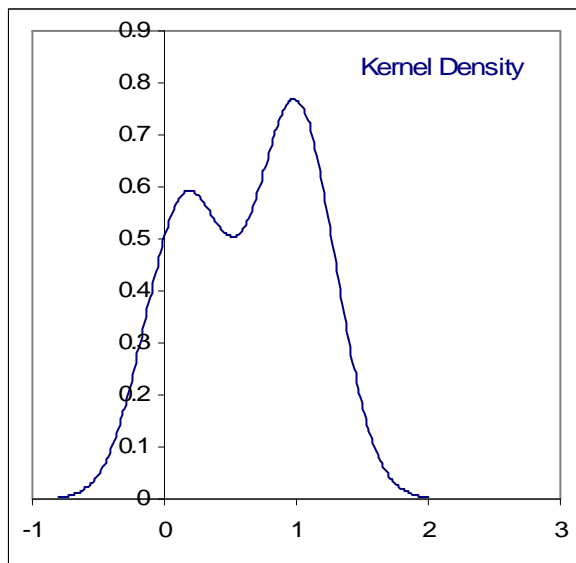
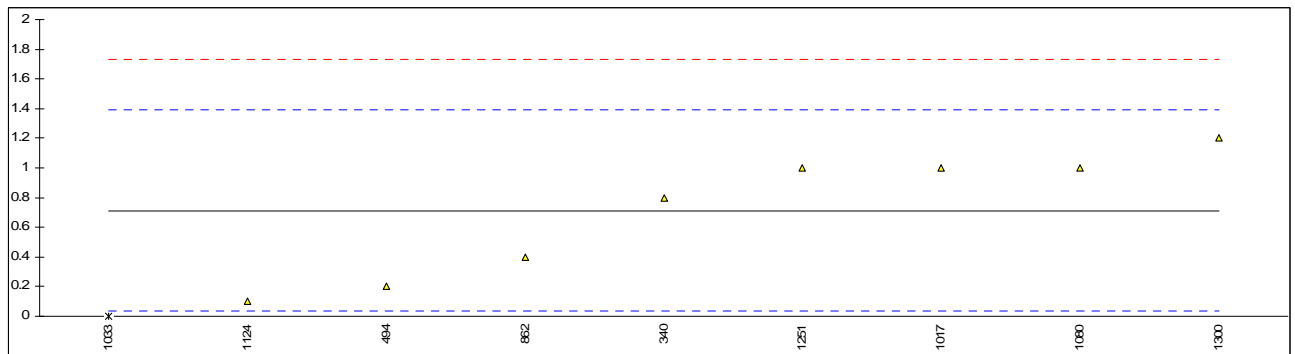
Determination of Density @ 15°C on sample #1046; results in kg/m<sup>3</sup>

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
340	ISO12185	784.61		-1.17	
463	ISO12185	785.5	C	3.81	First reported 787.5
494	ISO12185	784.7	C	-0.67	First reported 748.7
496	ISO12185	784.76		-0.33	
862	D4052	784.70		-0.67	
1017	ISO12185	787.86	G(0.01)	17.03	
1033	IP365	784.8		-0.11	
1047	ISO12185	784.5		-1.79	
1080	ISO12185	784.9		0.45	
1121	IP365	784.4		-2.35	
1124	ISO12185	784.90		0.45	
1154	ISO12185	777.1	C,G(0.01)	-43.23	Reported 0.7771
1201	ISO12185	784.5		-1.79	
1251	ISO12185	784.8		-0.11	
1300	ISO12185	784.67		-0.84	
1706	ISO12185	784.9		0.45	
1727	ISO12185	785.17		1.96	
1835	D4052	785.3	C	2.69	First reported 0.7853
normality	OK				
n	16				
outliers	2				
mean (n)	784.82				
st.dev. (n)	0.296				
R(calc.)	0.83				
R(ISO12185:96)	0.50				



Determination of Existent Gum (washed) on sample #1046; results in mg/100mL

lab	method	value	mark	z(targ)	remarks
323		----		----	
340	ISO6246	0.8		0.26	
463	ISO6246	<0.5		----	
494	ISO6246	0.2		-1.51	
496	ISO6246	<1		----	
862	D381	0.4		-0.92	
1017	ISO6246	1		0.85	
1033	IP131	0.0	ex	-2.10	Result excluded, not a real result
1047	ISO6246	<1.0		----	
1080	ISO6246	1.0		0.85	
1121		----		----	
1124	ISO6246	0.1		-1.81	
1154		----		----	
1201	ISO6246	<0.5		----	
1251	ISO6246	1		0.85	
1300	ISO6246	1.2		1.44	
1706		----		----	
1727		----		----	
1835		----		----	
normality		OK			
n		8			
outliers		0			
mean (n)		0.71			
st.dev. (n)		0.419			
R(calc.)		1.17			
R(ISO6246:97)		0.95			

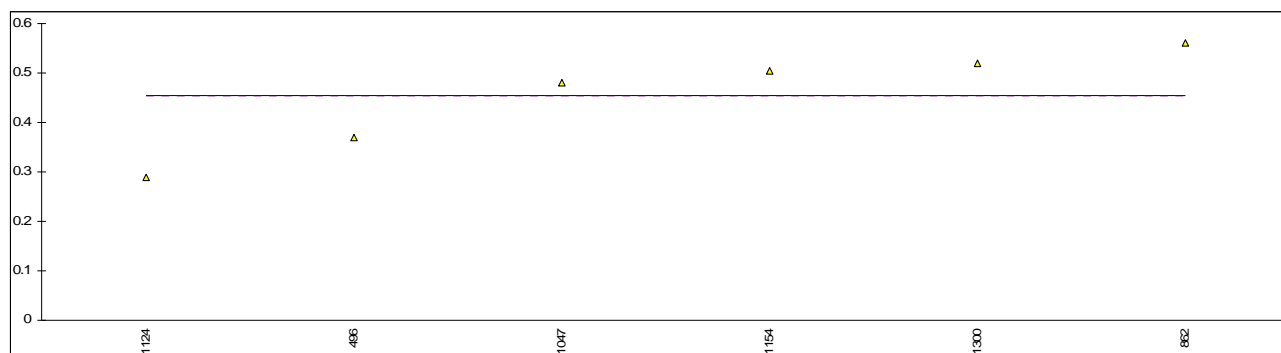


Determination of Inorganic Chloride on sample #1046; results in mg/L

lab	method	value	mark	z(targ)	remarks
323		----		----	
340		----		----	
463		----		----	
494		----		----	
496	inh-395	0.37		----	
862	EN15492	0.56		----	
1017		----		----	
1033		----		----	
1047	EN15484	0.48		----	
1080		----		----	
1121		----		----	
1124	EN15492	0.29		----	
1154	EN15492	0.504		----	
1201	EN15492	<1		----	
1251	EN15492	<5		----	
1300	EN15492	0.5185		----	
1706		----		----	
1727		----		----	
1835		----		----	

normality OK  
n 6  
outliers 0  
mean (n) 0.454  
st.dev. (n) 0.1025  
R(calc.) 0.287  
R(EN15492:08) (1.192)

Application range: 4 – 30 mg/L



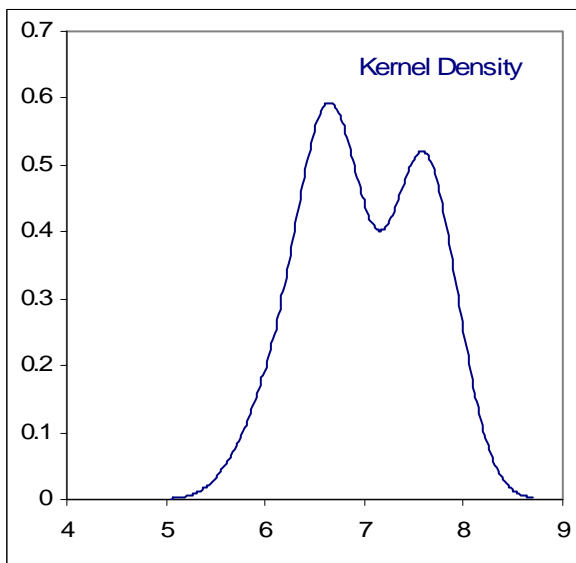
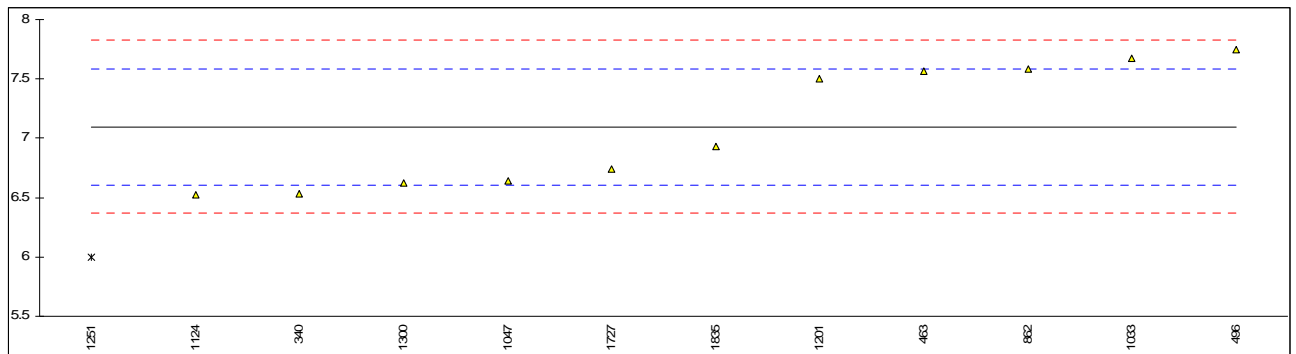
## Determination of Oxidation Stability on sample #1046; results in minutes

lab	method	value	mark	z(targ)	remarks
323		----		----	
340	ISO7536	>960		----	
463		----		----	
494	D525	>900		----	
496	D525	>1000		----	
862	D525	>900		----	
1017		----		----	
1033	IP460	>960		----	
1047		----		----	
1080		----		----	
1121		----		----	
1124	ISO7536	>900		----	
1154		----		----	
1201	D525	>900		----	
1251		----		----	
1300	ISO7536	>900		----	
1706		----		----	
1727		----		----	
1835		----		----	
	normality	n.a.			
	n	0			
	outliers	0			
	mean (n)	>900			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(ISO7536:96)	n.a.			

Determination of pH<sub>e</sub> on sample #1046;

lab	method	value	mark	z(targ)	remarks
323		----		----	
340	EN15490	6.53		-2.32	
463	D6423	7.564		1.93	
494		----		----	
496	EN15490	7.75		2.69	
862	D6423	7.58		1.99	
1017		----		----	
1033	D6423	7.67		2.36	
1047	EN15490	6.64		-1.87	
1080		----		----	
1121		----		----	
1124	D6423	6.523		-2.35	
1154		----		----	
1201	EN15490	7.5		1.66	
1251	EN15490	6.0	G(0.05)	-4.50	
1300	EN15490	6.619		-1.96	
1706		----		----	
1727	EN15490	6.74		-1.46	
1835	EN15490	6.93		-0.68	
normality		OK			
n		11			
outliers		1			
mean (n)		7.095			
st.dev. (n)		0.5111			
R(calc.)		1.431			
R(EN15490:08)		0.681			

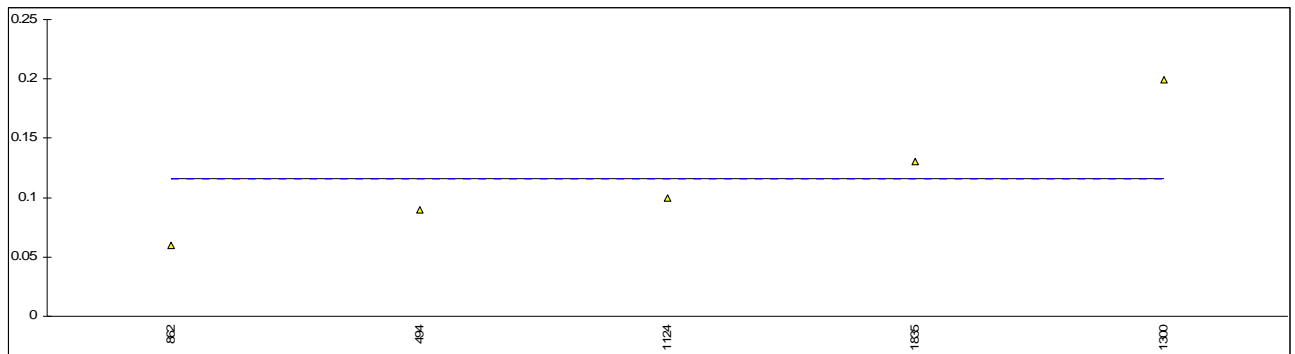
Compare R(D6423) = 0.520





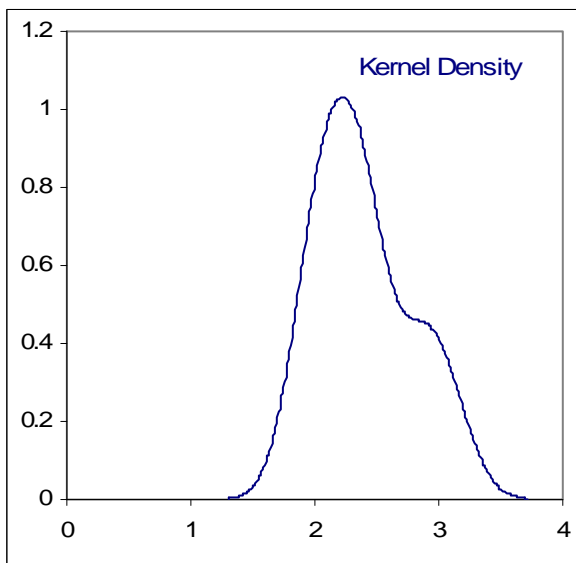
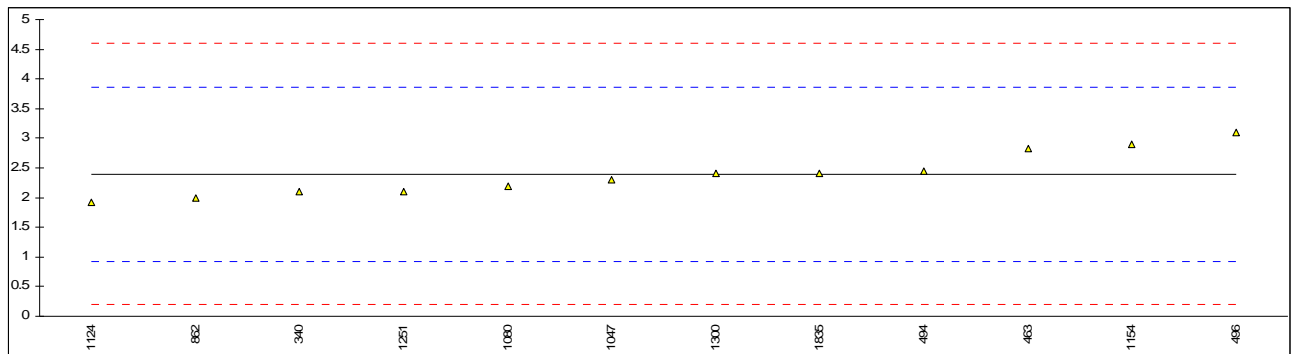
Determination of Phosphorus on sample #1046; results in mg/L

lab	method	value	mark	z(targ)	remarks
323		----		----	
340		----		----	
463		----		----	
494	EN15487	0.09		----	
496	EN15487	<0.15		----	
862	D3231	0.06		----	
1017		----		----	
1033		----		----	
1047	EN15487	<0.15		----	
1080		----		----	
1121		----		----	
1124	D3231	0.10		----	
1154		----		----	
1201	EN15487	<0.5		----	
1251		----		----	
1300	EN15487	0.1996		----	
1706		----		----	
1727	EN15487	<0.20		----	
1835	EN15487	0.13		----	
	normality	OK			
	n	5			
	outliers	0			
	mean (n)	0.116			
	st.dev. (n)	0.0530			
	R(calc.)	0.149			
	R(EN15487:08)	(0.070)			
					Compare R(D3231) = 0.130
					Application range 0.15 – 1.50 mg/L



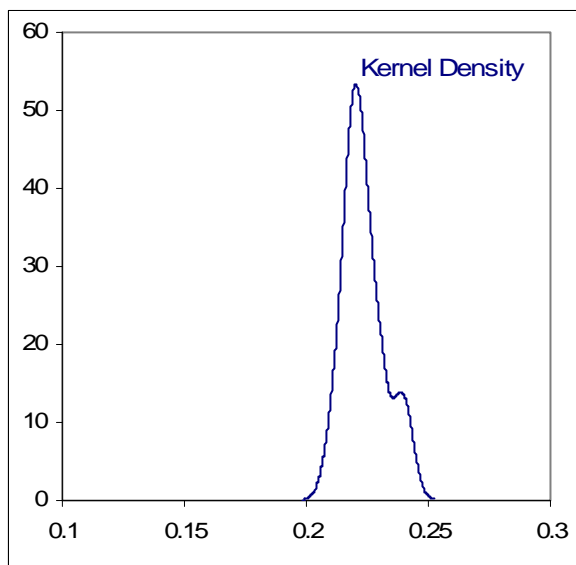
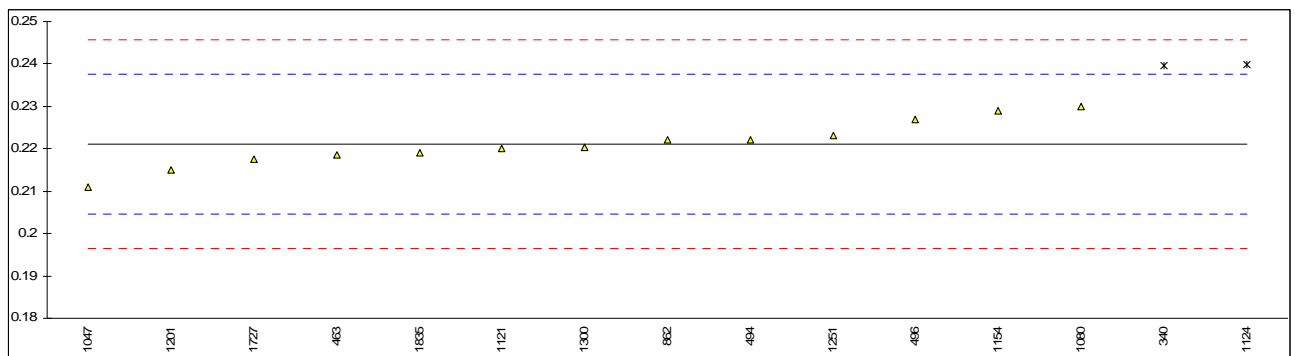
Determination of Sulphur on sample #1046; results in mg/kg

lab	method	value	mark	z(targ)	remarks
323		----		----	
340	EN15486	2.1		-0.40	
463	EN15486	2.83		0.60	
494	EN15486	2.45		0.08	
496	EN15486	3.1		0.96	
862	D5453	2.0		-0.54	
1017		----		----	
1033		----		----	
1047	EN15486	2.3		-0.13	
1080	ISO20846	2.2		-0.26	
1121		----		----	
1124	ISO20884	1.92		-0.65	
1154	ISO20846	2.9		0.69	
1201	EN15486	<1		<-1.90	False negative result?
1251	EN15486	2.1		-0.40	
1300	EN15486	2.409		0.02	
1706		----		----	
1727		----		----	
1835	EN15486	2.41		0.02	
normality		OK			
n		12			
outliers		0			
mean (n)		2.39			
st.dev. (n)		0.375			
R(calc.)		1.05			
R(EN15486:08)		2.05			
					Compare R(ISO20846) = 1.39
					Application range : 5 – 20 mg/kg



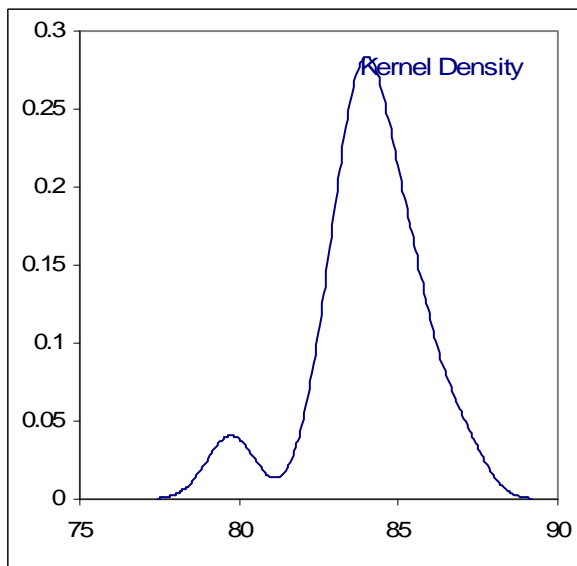
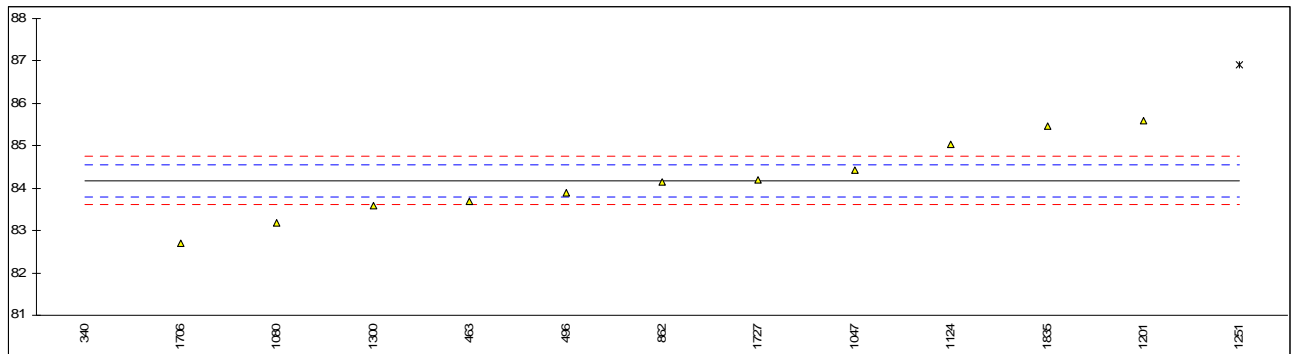
Determination of Water on sample #1046; results in %M/M

lab	method	value	mark	z(targ)	remarks
323		-----		-----	
340	EN15489	0.23950	DG(0.05)	2.24	
463	ISO12937	0.2185		-0.32	
494	EN15489	0.2220		0.11	
496	EN15489	0.227		0.72	
862	E1064	0.2220		0.11	
1017		-----		-----	
1033	IP438	<0.0030	C	-----	False negative result? First reported 0
1047	EN15489	0.211		-1.23	
1080	D6304	0.23		1.09	
1121	IP539	0.220		-0.13	
1124	EN15489	0.2398	DG(0.05)	2.28	
1154	EN12937	0.229		0.96	
1201	EN15489	0.215		-0.74	
1251	EN15489	0.223		0.23	
1300	EN15489	0.2202		-0.11	
1706		-----		-----	
1727	EN15489	0.2175		-0.44	
1835	EN15489	0.2191		-0.24	
normality	OK				
n	13				
outliers	2				
mean (n)	0.2211				
st.dev. (n)	0.00537				
R(calc.)	0.0150				
R(EN15489:08)	0.0230				Compare R(E1064) = 0.0071



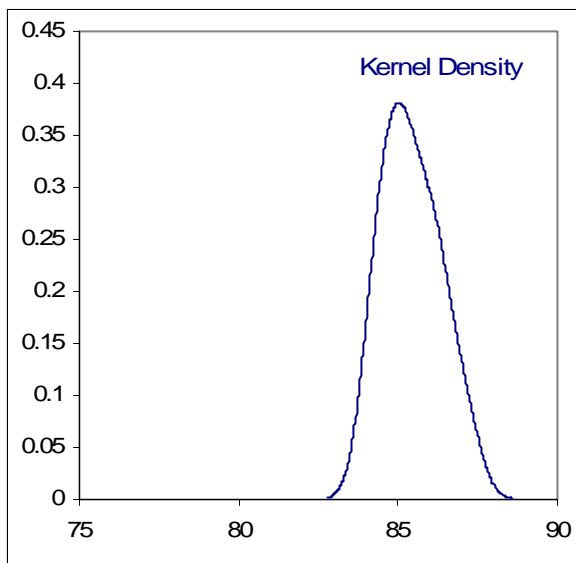
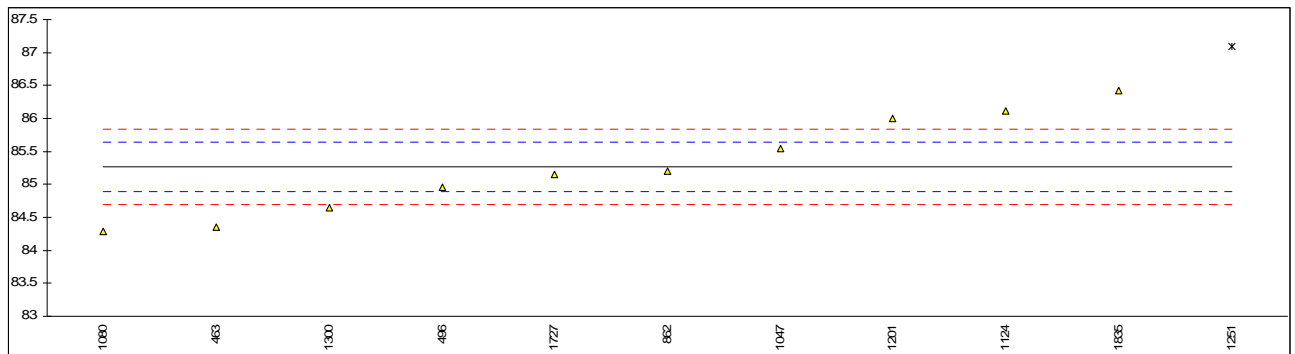
Determination of Ethanol content on sample #1046; results in %V/V

lab	method	value	mark	z(targ)	remarks
323		----		----	
340	EN1601	79.74	G(0.05)	-23.43	
463	inh-01	83.68		-2.61	
494		----		----	
496	D5501	83.893		-1.49	
862	D5501	84.153		-0.11	
1017		----		----	
1033		----		----	
1047	EN1601	84.43		1.35	
1080	in house	83.18		-5.25	
1121		----		----	
1124	D5501	85.04		4.57	
1154		----		----	
1201	in house	85.6		7.53	
1251	in house	86.9	G(0.05)	14.40	
1300	D5501	83.5752		-3.17	
1706	EN13132Mod	82.7		-7.79	
1727	D5501	84.20		0.13	
1835	in house	85.47		6.84	
normality		OK			
n		11			
outliers		2			
mean (n)		84.1747			
st.dev. (n)		0.914985			
R(calc.)		2.5620			
R(D5501:09)		0.5300			



Determination of Ethanol content on sample #1046; results in %M/M

lab	method	value	mark	z(targ)	remarks
323		----		----	
340		----		----	
463	inh-01	84.36		-4.80	
494		----		----	
496	D5501	84.962		-1.62	
862	D5501	85.204		-0.34	
1017		----		----	
1033		----		----	
1047	EN1601	85.54		1.43	
1080	in house	84.28		-5.23	
1121		----		----	
1124	D5501	86.11		4.44	
1154		----		----	
1201	in house	86.0		3.86	
1251	in house	87.1	G(0.05)	9.67	
1300	D5501	84.6542		-3.25	
1706		----		----	
1727	D5501	85.16		-0.58	
1835	in house	86.42		6.08	
normality		OK			
n		10			
outliers		1			
mean (n)		85.2690			
st.dev. (n)		0.74032			
R(calc.)		2.0729			
R(D5501:09)		0.5300			



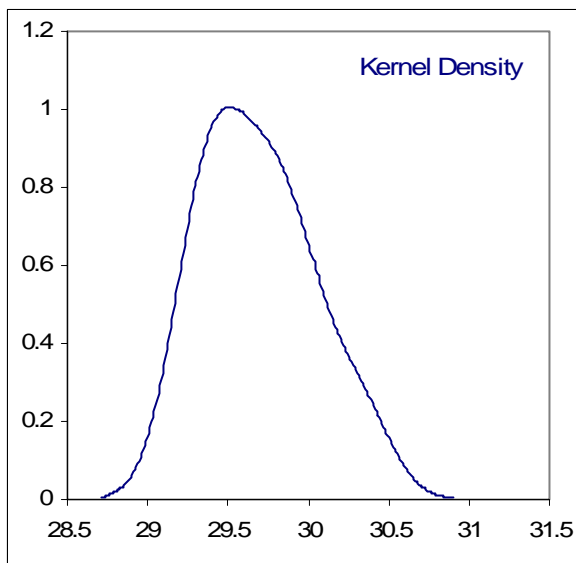
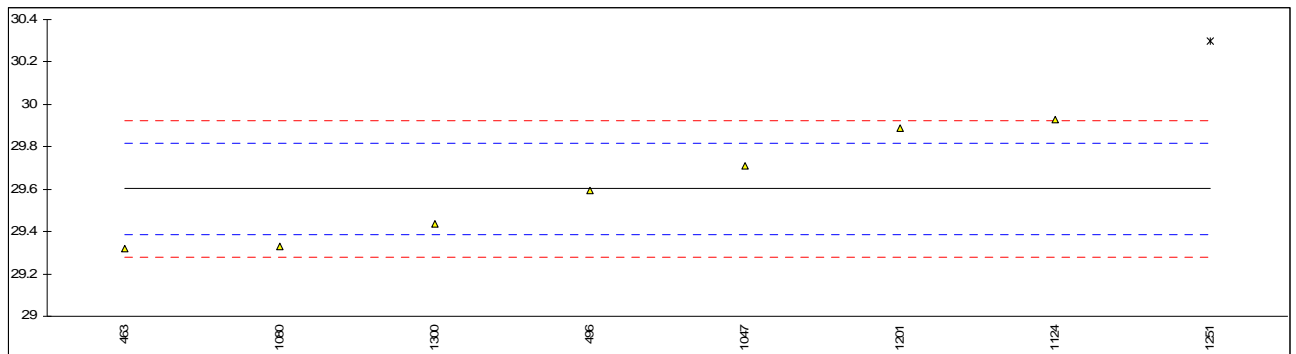
## Determination of Methanol on sample #1046; results in %V/V

lab	method	MeOH	mark	Higher sat.alc. *)	mark	Ethers	mark	Remarks
323		----		----		----		
340		----		----		----		
463	EN13132	<0.2		<0.2		<0.2		
494		----		----		----		
496	EN1601	0.05		0.14		0.15		
862	D5501	0.0079		----		----		
1017		----		----		----		
1033		----		----		----		
1047	EN1601	<0.17		<0.17		0.25	C	First reported <0.17
1080		----		0.03		0.34		
1121		----		----		----		
1124	EN13132	<0.2		<0.2		0.16		
1154		----		----		----		
1201	in house	0.90		<0.1		0.28		
1251		----		----		----		
1300	EN1601	0.3164		0.0137		0.3195		
1706		----		----		----		
1727		----		----		----		
1835	in house	0.0085		----		0.353		
	normality	n.a.		n.a.		n.a.		
	n	5		3		7		
	outliers	0		0		0		
	mean (n)	n.a.		n.a.		n.a.		
	st.dev. (n)	n.a.		n.a.		n.a.		
	R(calc.)	n.a.		n.a.		n.a.		
	R(EN1601:97)	n.a.		n.a.		n.a.		

\*) higher saturated monoalcohols (C3 – C8)

Determination of Oxygen content on sample #1046; results in %M/M

lab	method	value	mark	z(targ)	remarks
323		----		----	
340		----		----	
463	EN13132	29.32		-2.63	
494		----		----	
496	EN1601	29.591		-0.10	
862		----		----	
1017		----		----	
1033		----		----	
1047	EN1601	29.71		1.01	
1080	in house	29.33		-2.53	
1121		----		----	
1124	EN13132	29.93		3.07	
1154		----		----	
1201	in house	29.89		2.69	
1251	in house	30.3	G(0.05)	6.52	
1300	EN1601	29.438		-1.52	
1706		----		----	
1727		----		----	
1835		----		----	
normality		OK			
n		7			
outliers		1			
mean (n)		29.601			
st.dev. (n)		0.2526			
R(calc.)		0.707			
R(EN1601:97)		0.300			



## **APPENDIX 2**

### **Number of participants per country**

- 1 laboratory in AUSTRIA
- 2 laboratories in BELGIUM
- 1 laboratory in ESTONIA
- 2 laboratories in FRANCE
- 2 laboratories in GERMANY
- 1 laboratory in HUNGARY
- 1 laboratory in LATVIA
- 1 laboratory in P.R. of CHINA
- 1 laboratory in POLAND
- 2 laboratories in SPAIN
- 1 laboratory in SWEDEN
- 2 laboratories in THE NETHERLANDS
- 2 laboratories 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
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 <http://www.rsc.org/suppdata/an/b2/b205600n/>)
- 15 H. Verplaetse and M. Lacourt, Accred Qual Assur (2006) 11:521-522