

Results of Proficiency Test
Biogasoline E85
May 2012

Organised by: Institute for Interlaboratory Studies
Spijkensisse, 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 2011/2012. In this interlaboratory study 15 laboratories in 9 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the 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), which can be downloaded from www.iisnl.com.

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 #12072). The homogeneity of the subsamples #12072 was checked by determination of Density @ 15°C in accordance with ASTM D4052:11 on 8 stratified randomly selected samples.

	Density @ 15°C in kg/m ³
Sample #12072-1	779.06
Sample #12072-2	779.05
Sample #12072-3	779.04
Sample #12072-4	779.05
Sample #12072-5	779.05
Sample #12072-6	779.06
Sample #12072-7	779.08
Sample #12072-8	779.10

table 1: homogeneity test results of subsamples #12072

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 #12072)	0.05
reference test	ISO12185:96
0.3*R (reference test)	0.15

table 2: evaluation of repeatability of the subsamples #12072

The calculated repeatability is each less than 0.3 times the reproducibility of the corresponding reference method. Therefore, homogeneity of the subsamples #12072 was assumed.

To the participants: one 1L bottle with sample #12072 was sent on May 2, 2012.

2.5 ANALYSIS

The participants were requested to determine on sample #12072: Acidity, Copper, Copper Corrosion, Density, Existent Gum, Inorganic Chloride (mg/l and mg/kg), 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 and a letter of instructions were prepared and made available for download on the iis website.

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; 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.

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. One laboratory did not report any test results. Not all laboratories were able to perform all analyses requested. Finally, 14 laboratories did report 103 numerical results. Observed were 3 outlying results, which is 2.9%. In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

In this section the results are discussed per test. All data sets proved to have a normal distribution, except for the Density results. For this determination a not normal data distribution was found.

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

Copper as Cu: No significant conclusions were drawn as only two numerical results were reported. All 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.

Density @15°C: This determination was problematic. Only one statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ISO12185:96.

Existent Gum: The consensus value was near or below the detection limit. Therefore no significant conclusions were drawn. No statistical outliers were observed.

Inorganic Chloride: The results of this determination were reported in mg/l and in mg/kg. 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 determination may not be problematic.

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

pHe: This determination was problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of EN15490:07. The EN15490 method uses a combined pH electrode, filled with a 1 M solution of Lithium Chloride in 99 % (V/V) Ethanol. The D6423 test method uses an Orion combination electrode. Use of different electrodes will likely give different test results.

Phosphorus: Due the low concentration (0.083 mg/L) in this sample no conclusions were drawn. The application range of EN15487:08 is 0.15 – 1.50 mg/L. All participants agreed on a value near or below the detection limit.

Sulphur: This determination may be 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 EN15486:07. No statistical outliers were observed.

Water: This determination was not problematic. Only one statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of EN15489:07.

Ethanol: This determination (%V/V and %M/M) may be very problematic. No statistical outliers were observed. However the calculated reproducibilities are not at all in agreement with the requirements of ASTM D5501:09. However, the precision of D5501 was determined using 93-97% ethanol and it may not be valid for 74% ethanol.

Ether content: No significant conclusions were drawn. Only three laboratories reported a numerical result.

Methanol: This determination may not be problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of EN1601:97.

Higher saturated alcohols: No significant conclusions were drawn. Only three laboratories reported a numerical result.

Total Organic Bound Oxygen: Only three laboratories reported a numerical result of which one apparently contains a calculation error. When the theoretical values were estimated from the reported test results, the determination appears to be very problematic. The problems observed with the ethanol determination of course are transferred 1:1 to this determination.

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	7	0.0014	0.0024	0.0014
Copper as Cu	mg/kg	2	n.e	n.e	n.e
Copper corr. 3 hrs @ 50°C	-----	10	1 (1a)	n.e	n.e
Density @ 15°C	kg/m ³	13	779.1	0.7	0.5
Existent Gum (washed)	mg/100mL	6	0.8	1.6	(1.1)
Inorganic Chloride	mg/L	2	0.44	n.e.	n.e.
Inorganic Chloride	mg/kg	3	0.62	n.e.	n.e.
Oxidation Stability	min.	8	>900	n.e.	n.e.
pHe		8	7.64	1.10	0.73
Phosphorous	mg/L	2	0.08	n.e.	n.e.
Sulphur	mg/kg	5	1.26	0.80	1.87
Water	%M/M	10	0.19	0.02	0.02
- Ethanol	%M/M	8	74.1	1.5	0.5
- Ethanol	%V/V	9	72.6	1.6	0.5
- Ether content	%V/V	3	0.26	0.10	0.10
- Methanol	%V/V	4	0.19	0.05	0.10
- Higher saturated monoalc.	%V/V	3	0.09	n.e.	n.e.
- Total organic bound oxygen	%M/M	2	26.1	n.e.	0.3

table 3: performance evaluation sample #12072

*() = near or below the detection limit

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 2012 WITH PREVIOUS PTS

Determination	May 2012	May 2011	May 2010
Number of reporting labs	14	20	17
Number of results reported	103	155	150
Statistical outliers	3	8	9
Percentage outliers	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 2012	May 2011	May 2010
Acidity as acetic acid	--	+/-	+/-
Copper as Cu	n.e.	n.e.	n.e.
Copper corr. 3 hrs @ 50°C	++	++	++
Density @ 15°C	-	+/-	--
Existent Gum (washed)	(--)*	(+)*	-
Inorganic Chloride	n.e.	++	++
Oxidation Stability	n.e.	n.e.	n.e.
pHe	--	--	--
Phosphorous	n.e.	(-)*	(--)*
Sulphur	++	++	++
Water	+/-	++	++
- Ethanol	--	--	--
- Ether content	n.e.	--	n.e.
- Higher saturated monoalc.	n.e.	n.e.	n.e.
- Methanol	++	n.e.	n.e.
- Total organic bound oxygen	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

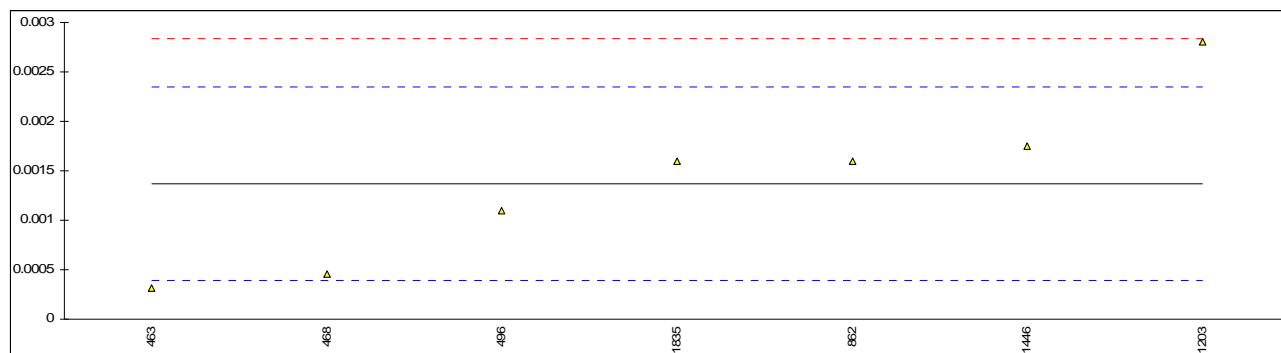
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

- ++: 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 #12072; results in %M/M

lab	method	value	mark	z(targ)	remarks
343	EN15491	<0.003		----	
420		----		----	
441		----		----	
447		----		----	
463	D1613	0.00031		-2.18	
468	EN15491	0.00046		-1.87	
496	EN15491	0.0011		-0.56	
862	EN15491	0.0016		0.46	
1033		----		----	
1203	D1613	0.0028		2.91	
1446	EN15491	0.00175		0.77	
1634		----		----	
1706		----		----	
1835	EN15491	0.0016		0.46	
2493		----		----	
normality	OK				
n	7				
outliers	0				
mean (n)	0.00137				
st.dev. (n)	0.000848				
R(calc.)	0.00238				
R(EN15491:07)	0.00137				



Determination of Copper on sample #12072; results in mg/kg

lab	method	value	mark	z(targ)	remarks
343	EN15488	<0.07		----	
420	EN15488	0.11		----	
441		----		----	
447		----		----	
463		----		----	
468	D1688	<0.05		----	
496		----		----	
862	EN15488	0.0005		----	
1033		----		----	
1203		----		----	
1446		----		----	
1634		----		----	
1706		----		----	
1835		----		----	
2493		----		----	
	normality	n.a.			
	n	2			
	outliers	0			
	mean (n)	n.a.			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(EN15488:07)	n.a.			

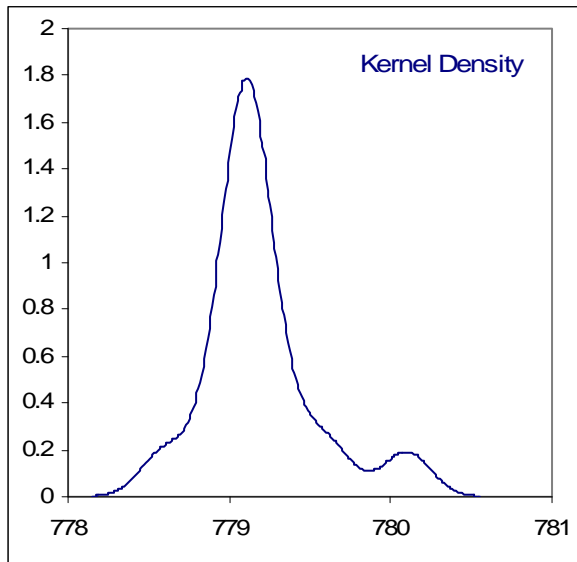
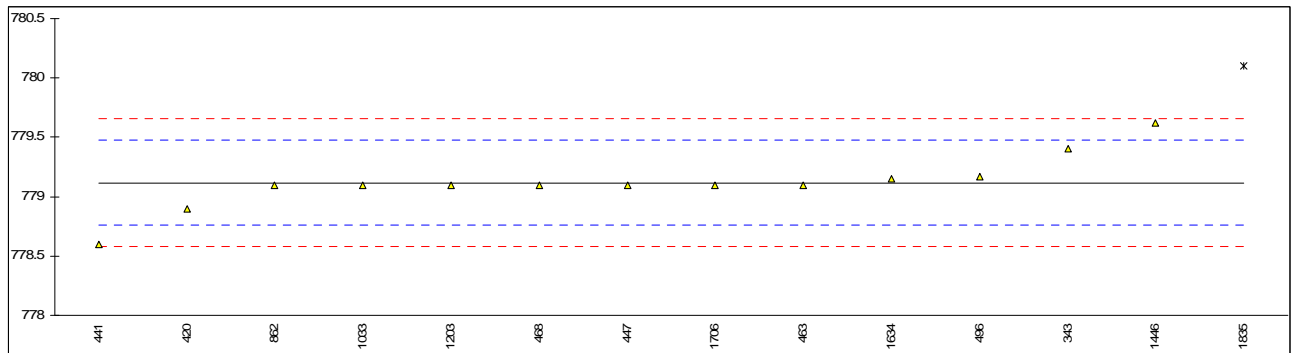
Determination of Copper Corrosion 3hrs/50°C on sample #12072; rating

lab	method	value	mark	z(targ)	remarks
343	D130	1A		----	
420	ISO2160	1		----	
441		----		----	
447	IP154	1A		----	
463	ISO2160	1A		----	
468	ISO2160	1A		----	
496	ISO2160	1A		----	
862	D130	1A		----	
1033	IP154	1B		----	
1203	ISO2160	1		----	
1446		----		----	
1634	ISO2160	1A		----	
1706		----		----	
1835		----		----	
2493		----		----	
	normality	n.a.			
	n	10			
	outliers	0			
	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 #12072; results in kg/m³

lab	method	value	mark	z(targ)	remarks
343	ISO12185	779.40		1.58	
420	ISO12185	778.9		-1.22	
441	D4052	778.6		-2.90	
447	IP365	779.1		-0.10	
463	ISO12185	779.10		-0.10	
468	ISO12185	779.10		-0.10	
496	ISO12185	779.17		0.29	
862	ISO12185	779.1		-0.10	
1033	IP365	779.1		-0.10	
1203	ISO12185	779.1		-0.10	
1446	ISO12185	779.624		2.83	
1634	ISO12185	779.146		0.15	
1706	ISO12185	779.1		-0.10	
1835	D4052	780.1	G(0.05)	5.50	
2493		-----		-----	

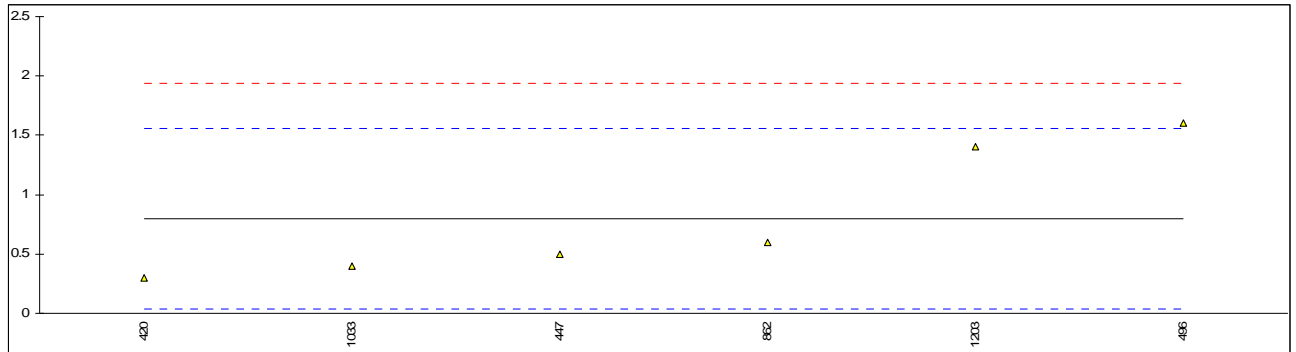
normality not OK
n 13
outliers 1
mean (n) 779.12
st.dev. (n) 0.234
R(calc.) 0.66
R(ISO12185:96) 0.50



Determination of Existent Gum (washed) on sample #12072; results in mg/100ml

lab	method	value	mark	z(targ)	remarks
343	ISO6246	<1		0.52	
420	ISO6246	0.3		-1.31	
441		----		----	
447	IP131	0.5		-0.79	
463	ISO6246	<0.5		<-0.79	
468	ISO6246	<0.5		<-0.79	
496	ISO6246	1.6		2.10	
862	ISO6246	0.6		-0.53	
1033	IP131	0.4		-1.05	
1203	ISO6246	1.4		1.58	
1446		----		----	
1634		----		----	
1706		----		----	
1835		----		----	
2493		----		----	

normality n.a.
 n 6
 outliers 0
 mean (n) 0.80
 st.dev. (n) 0.555
 R(calc.) 1.55
 R(ISO6246:97) 1.07



Determination of Inorganic Chloride on sample #12072; results in mg/L

lab	method	value	mark	z(targ)	remarks
343	EN15492	<0.8		----	
420		----		----	
441		----		----	
447		----		----	
463		----		----	
468	EN15492	<0.5		----	
496	EN15492	0.48		----	
862		----		----	
1033		----		----	
1203	EN15492	0.39		----	
1446		----		----	
1634		----		----	
1706		----		----	
1835		----		----	
2493		----		----	
	normality	n.a.			
	n	2			
	outliers	0			
	mean (n)	0.435			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(EN15492:08)	n.a.			Application range: 0.8 – 2.0 mg/L

Determination of Inorganic Chloride on sample #12072; results in mg/kg

lab	method	value	mark	z(targ)	remarks
343		----		----	
420		----		----	
441		----		----	
447		----		----	
463		----		----	
468		<0.5		----	
496	EN15492	0.62		----	
862	IMPCA002	0.74		----	
1033		----		----	
1203		0.51		----	
1446		----		----	
1634		----		----	
1706		----		----	
1835		----		----	
2493		----		----	
	normality	n.a.			
	n	3			
	outliers	0			
	mean (n)	0.623			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(EN15492:08)	n.a.			Application range; 1.0 – 2.5 mg/kg

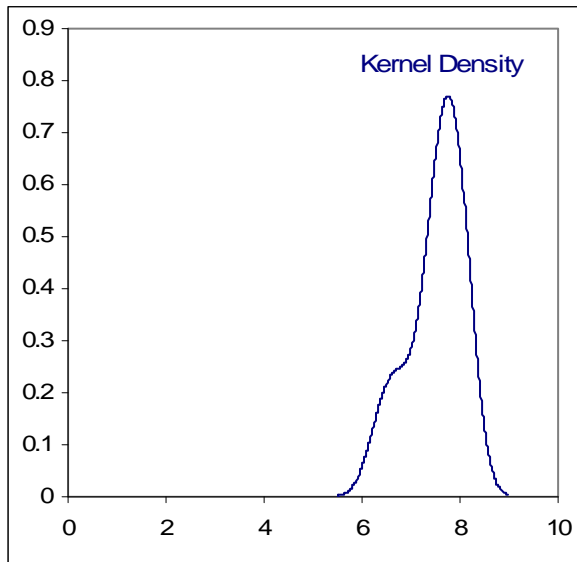
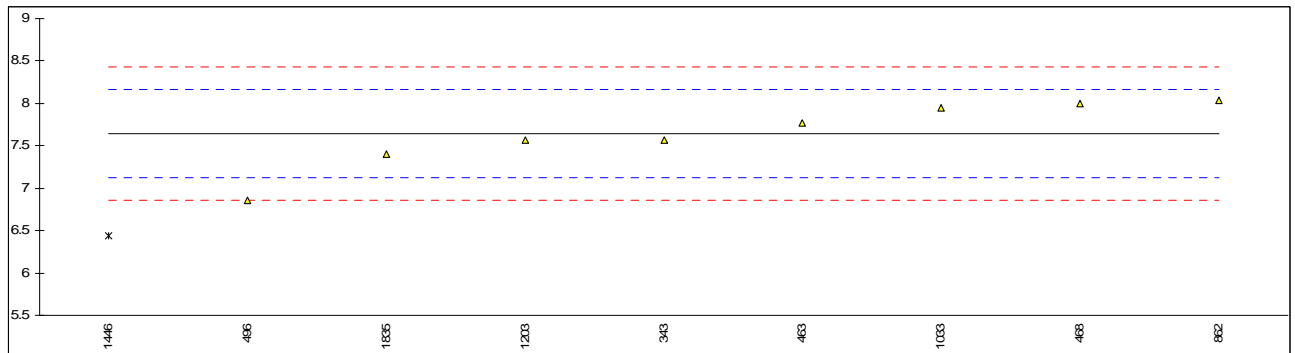
Determination of Oxidation Stability on sample #12072; results in minutes

lab	method	value	mark	z(targ)	remarks
343	D525	397	ex	----	False positive?
420	ISO7536	>900		----	
441		----		----	
447	IP40	>900		----	
463	D525	>900		----	
468		----		----	
496	ISO7536	>900		----	
862	D525	>900		----	
1033	IP40	>960		----	
1203	ISO7536	>900		----	
1446		----		----	
1634		----		----	
1706		----		----	
1835		----		----	
2493		----		----	
	normality	n.a.			
	n	7			
	outliers	0			
	mean (n)	>900			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(ISO7536:94)	n.a.			

Determination of pH_e on sample #12072;

lab	method	value	mark	z(targ)	remarks
343	EN15490	7.57		-0.28	
420		-----		-----	
441		-----		-----	
447		-----		-----	
463	D6423	7.77		0.48	
468	D6423	8.0		1.36	
496	EN15490	6.855		-3.01	
862	EN15490	8.03		1.48	
1033	EN15490	7.95		1.17	
1203	EN15490	7.57		-0.28	
1446	EN15490	6.44	G(0.05)	-4.59	
1634		-----		-----	
1706		-----		-----	
1835	EN15490	7.40		-0.93	
2493		-----		-----	

normality OK
n 8
outliers 1
mean (n) 7.643
st.dev. (n) 0.3925
R(calc.) 1.099
R(EN15490:07) 0.734



Determination of Phosphorus on sample #12072; results in mg/l

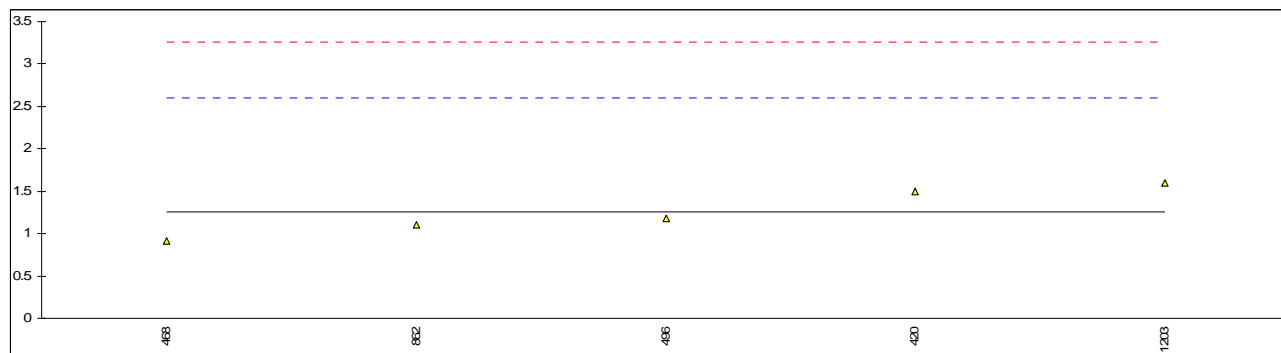
lab	method	value	mark	z(targ)	remarks
343	EN15487	<0.15		----	
420		----		----	
441		----		----	
447		----		----	
463		----		----	
468	EN15487	<0.10		----	
496		----		----	
862	EN15487	0.096		----	
1033		----		----	
1203		----		----	
1446		----		----	
1634		----		----	
1706		----		----	
1835	EN15487	0.07		----	
2493		----		----	
	normality	n.a.			
	n	2			
	outliers	0			
	mean (n)	0.083			
	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 #12072; results in mg/kg

lab	method	value	mark	z(targ)	remarks
343	EN15486	<5		----	
420	EN20846	1.5		0.36	
441				----	
447	D5453	<1.0		<-0.39	
463				----	
468	EN15486	0.91		-0.52	
496	EN15486	1.18		-0.12	
862	EN15486	1.1		-0.24	
1033				----	
1203	EN15486	1.6		0.51	
1446				----	
1634				----	
1706				----	
1835				----	
2493				----	

normality n.a.
n 5
outliers 0
mean (n) 1.258
st.dev. (n) 0.2862
R(calc.) 0.801
R(EN15486:07) 1.871

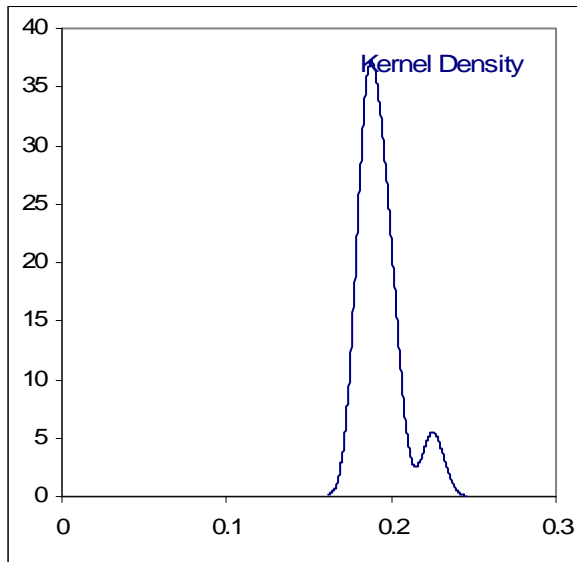
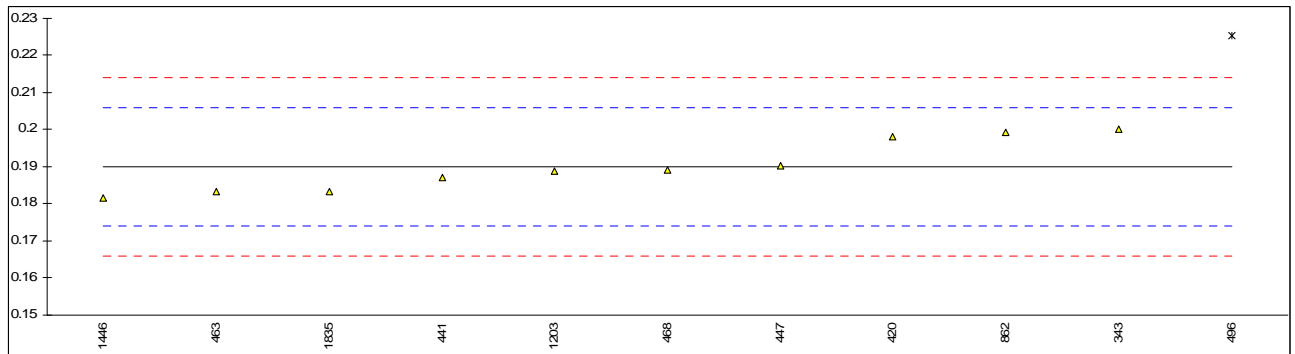
Application range: 5 – 20 mg/kg



Determination of Water on sample #12072; results in %M/M

lab	method	value	mark	z(targ)	remarks
343	EN15489	0.20	C	1.25	First reported 0.23
420	EN12937	0.198	C	1.00	First reported 0.153
441	EN15489	0.187		-0.38	
447	IP539	0.1904		0.05	
463	EN12937	0.1832		-0.85	
468	EN15489	0.189		-0.13	
496	EN15489	0.2254	G(0.01)	4.43	
862	EN15489	0.1992		1.15	
1033		----		----	
1203	EN15489	0.1887		-0.17	
1446	ISO760	0.1815		-1.07	
1634		----		----	
1706		----		----	
1835	EN15489	0.1832		-0.85	
2493		----		----	

normality OK
 n 10
 outliers 1
 mean (n) 0.1900
 st.dev. (n) 0.00687
 R(calc.) 0.0192
 R(EN15489:07) 0.0224

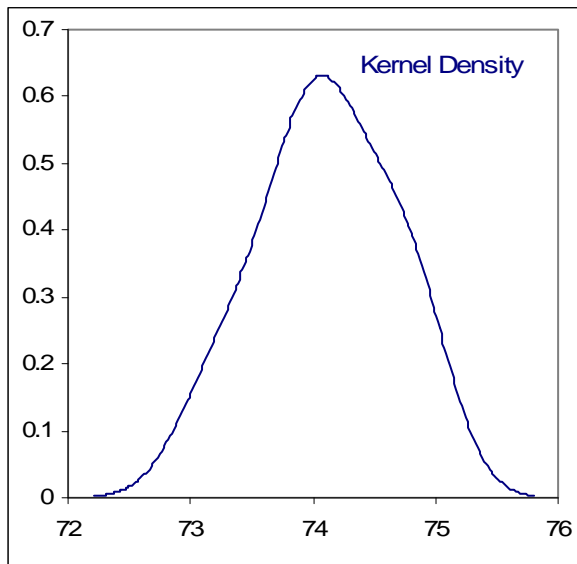
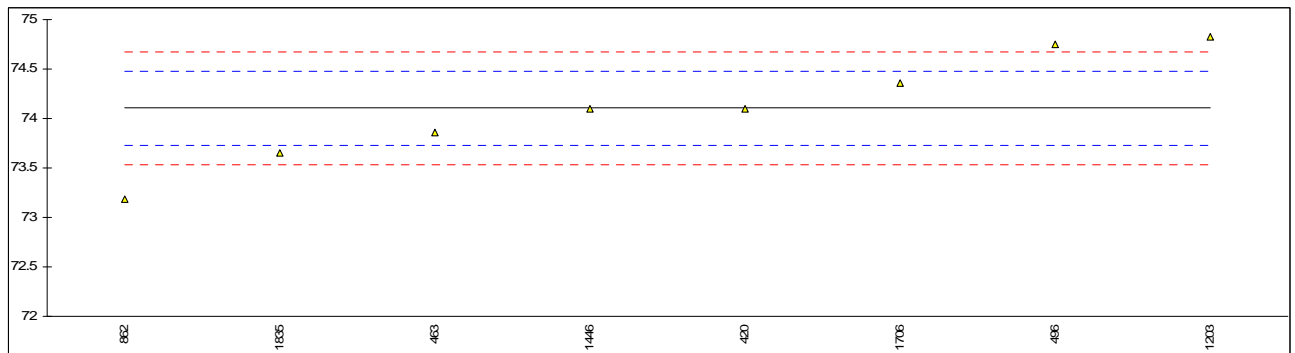


Determination of Ethanol content on sample #12072; results in %M/M

lab	method	value	mark	z(targ)	remarks
343		----		----	
420	EN13132	74.1		-0.02	
441		----		----	
447		----		----	
463	EN13132	73.86		-1.29	
468		----		----	
496	D5501	74.747		3.39	
862	D5501	73.19	C	-4.83	First reported 68.09
1033		----		----	
1203	EN1601	74.83		3.83	
1446	INH-GC	74.10		-0.02	
1634		----		----	
1706	EN13132	74.36		1.35	
1835	in house	73.65		-2.40	
2493		----		----	

normality OK
n 8
outliers 0
mean (n) 74.105
st.dev. (n) 0.5487
R(calc.) 1.536
R(D5501:09) 0.530

Application range: 93 – 97 %M/M

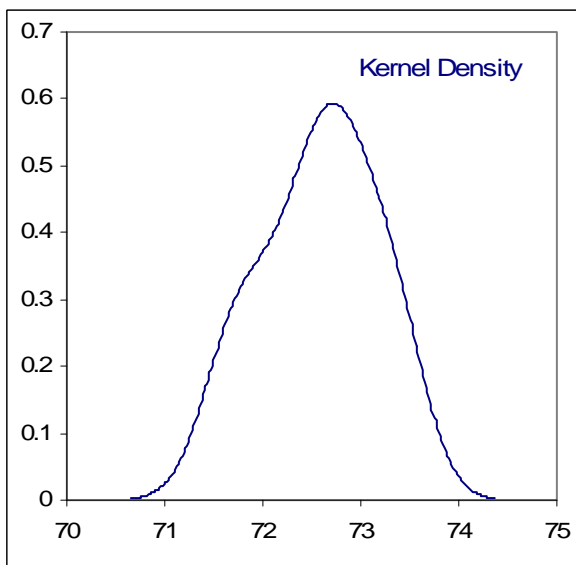
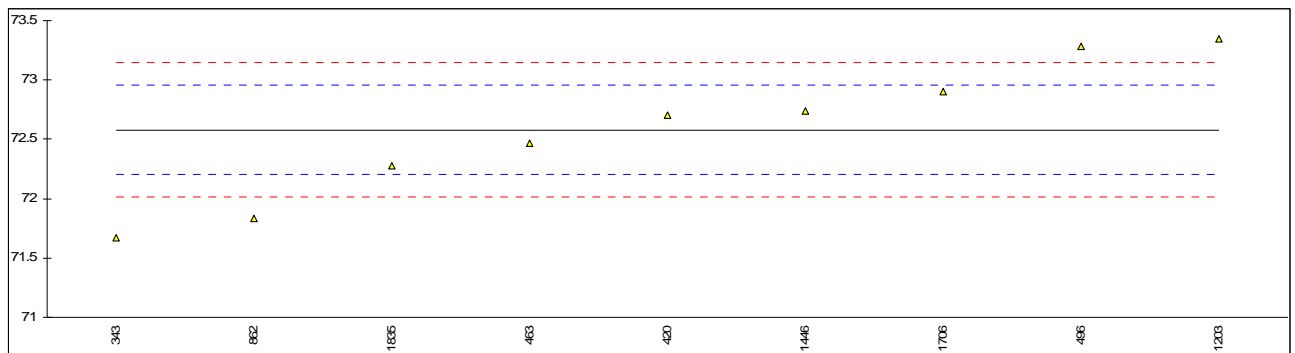


Determination of Ethanol content on sample #12072; results in %V/V

lab	method	value	mark	z(targ)	remarks
343	INH-1887	71.67		-4.81	
420	EN13132	72.7		0.63	
441		----		----	
447		----		----	
463	EN13132	72.47		-0.58	
468		----		----	
496	D5501	73.280		3.70	
862	D5501	71.83	C	-3.96	First reported 66.82
1033		----		----	
1203	EN1601	73.35		4.07	
1446	INH-GC	72.74		0.85	
1634		----		----	
1706		72.9		1.69	
1835	in house	72.28		-1.58	
2493		----		----	

normality OK
n 9
outliers 0
mean (n) 72.580
st.dev. (n) 0.5829
R(calc.) 1.632
R(D5501:09) 0.530

Application range: 93 – 97 %V/V



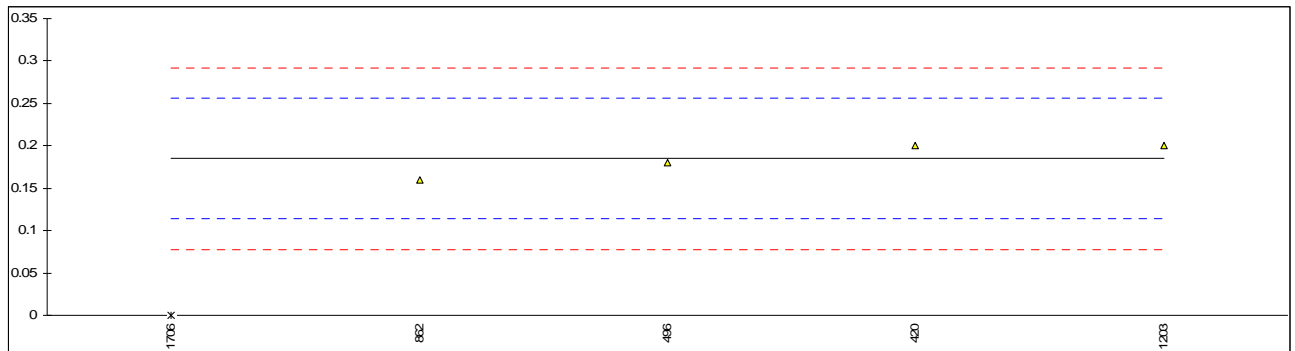
Determination of Ether content on sample #12072; results in %V/V

lab	method	value	mark	z(targ)	remarks
343		----		----	
420		----		----	
441		----		----	
447		----		----	
463		----		----	
468		----		----	
496	EN1601	0.22		-0.99	
862	D4815	0.256		0.02	
1033		----		----	
1203	EN1601	0.29		0.97	
1446		----		----	
1634		----		----	
1706		----		----	
1835		----		----	
2493		----		----	
	normality	n.a.			
	n	3			
	outliers	0			
	mean (n)	0.255			
	st.dev. (n)	0.0350			
	R(calc.)	0.098			
	R(EN1601:97)	0.100			

Determination of Methanol on sample #12072; results in %V/V

lab	method	value	mark	z(targ)	remarks
343		----		----	
420	EN13132	0.20		0.42	
441		----		----	
447		----		----	
463	EN13132	<0.2	C	<0.42	First reported 0.33
468		----		----	
496	EN1601	0.18		-0.14	
862	D4815	0.16		-0.70	
1033		----		----	
1203	EN1601	0.20		0.42	
1446		----		----	
1634		----		----	
1706	EN13132	0.0	ex	-5.18	Result excluded as zero is not a real value
1835		----		----	
2493		----		----	

normality n.a.
 n 4
 outliers 0
 mean (n) 0.185
 st.dev. (n) 0.0191
 R(calc.) 0.054
 R(EN1601:97) 0.100



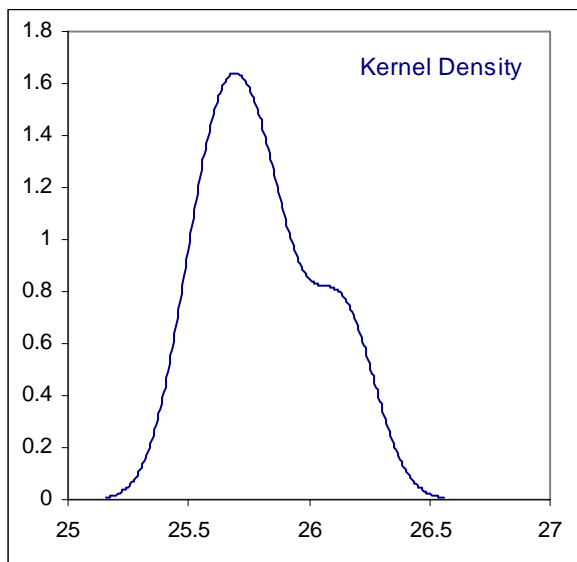
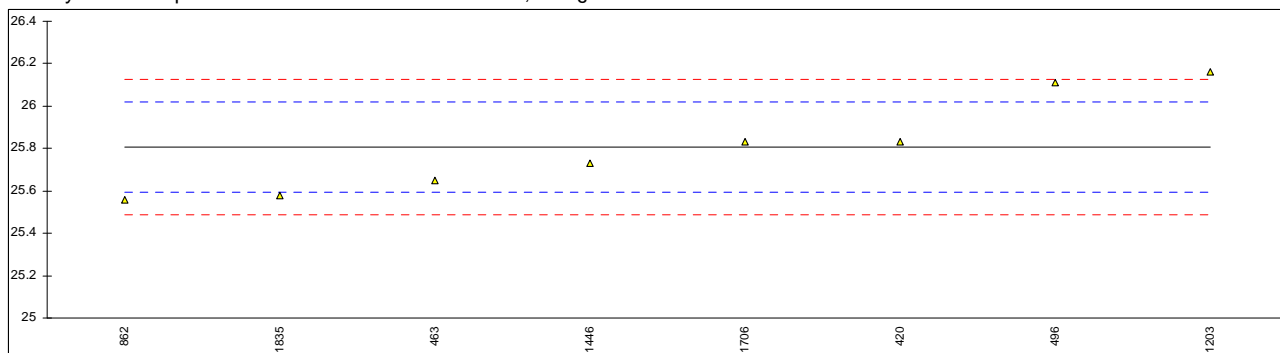
Determination of Higher saturated monoalcohols (C3-C5) on sample #12072; results in %V/V

lab	method	value	mark	z(targ)	remarks
343		----		----	
420		----		----	
441		----		----	
447		----		----	
463		----		----	
468		----		----	
496	EN1601	0.09		----	
862	D4815	0.091		----	
1033		----		----	
1203	EN1601	0.08		----	
1446		----		----	
1634		----		----	
1706		----		----	
1835		----		----	
2493		----		----	
	normality	n.a.			
	n	3			
	outliers	0			
	mean (n)	0.087			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(EN1601:97)	n.a.			

Determination of Total Org Bound Oxygen on sample #12072; results in %M/M

lab	method	value	mark	z(targ)	remarks	calc. by iis, see §4.1
343		----		----		----
420		----		----		25.83
441		----		----		----
447		----		----		----
463		----		----		25.65
468		----		----		----
496	EN1601	26.109		----		26.11
862	D4815	23.78	E, ex	----	calculation error?	25.56
1033		----		----		----
1203	EN1601	26.16		----		26.16
1446		----		----		25.73
1634		----		----		----
1706		----		----		25.83
1835		----		----		25.58
2493		----		----		----
normality		n.a.				OK
n		2				8
outliers		0				0
mean (n)		26.13				25.81
st.dev. (n)		n.a.				0.225
R(calc.)		n.a.				0.63
R(EN1601:97)		0.3				0.30

calc. 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 GERMANY

2 labs in HUNGARY

1 lab in P.R. of CHINA

1 lab in PORTUGAL

2 labs in SPAIN

2 labs in SWEDEN

3 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.s.	= 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 <http://www.rsc.org/suppdata/an/b2/b205600n/>)