

Results of Proficiency Test
Ethanol (Fuel grade)
December 2016

Organised by: Institute for Interlaboratory Studies (iis)
Spijkenisse, the Netherlands

Authors: ing. R.J. Starink
Correctors: dr. R.G. Visser & ing. A.S. Noordman-de Neef
Report: iis16C10

February 2017

CONTENTS

1	INTRODUCTION	3
2	SET UP	3
2.1	ACCREDITATION	3
2.2	PROTOCOL	3
2.3	CONFIDENTIALITY STATEMENT	3
2.4	SAMPLES	4
2.5	STABILITY OF THE SAMPLES.....	4
2.6	ANALYSES.....	4
3	RESULTS	5
3.1	STATISTICS	5
3.2	GRAPHICS	6
3.3	Z-SCORES	6
4	EVALUATION.....	7
4.1	EVALUATION PER TEST	7
4.2	PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES.....	10
4.3	COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2016 WITH PREVIOUS PTS.....	11

Appendices:

1.	Data and statistical results.....	12
2.	Number of participants in alphabetical country order	52
3.	Abbreviations and literature	53

1 INTRODUCTION

Since 1995, the Institute for Interlaboratory Studies (iis) organizes a proficiency test (PT) for Ethanol (Fuel grade) in accordance with the latest applicable version of the specifications EN15376 and ASTM D4806.

In this interlaboratory study 61 laboratories from 32 different countries for the PT on Ethanol (Fuel grade) did register for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2016 interlaboratory study on Ethanol (Fuel grade) are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET-UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. In this proficiency test the participants received a 1 litre bottle with Ethanol (Fuel grade), labelled #16260. Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for the statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

The necessary bulk material for sample #16260 was obtained from an European supplier. The approximately 125 litres bulk material was homogenised in a pre-cleaned drum. After homogenisation 98 amber glass bottles of 1 litre were filled with Ethanol (Fuel grade) and labelled #16260. The homogeneity of the sub samples #16260 was checked by determination of Density in accordance with ASTM D4052 and Water in accordance with EN15489 on 8 stratified randomly selected samples.

	Density at 20°C in kg/L	Water in %M/M
Sample #16260-1	0.79424	0.177
Sample #16260-2	0.79423	0.178
Sample #16260-3	0.79422	0.179
Sample #16260-4	0.79422	0.179
Sample #16260-5	0.79422	0.180
Sample #16260-6	0.79422	0.178
Sample #16260-7	0.79422	0.179
Sample #16260-8	0.79422	0.179

Table 1: Homogeneity test results of sub samples #16260

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility of the respective reference test method in agreement with the procedure of ISO 13528, Annex B2 in the next table.

	Density at 20°C in kg/L	Water in %M/M
r (observed)	0.00002	0.003
reference test method	ISO12185:96	EN15489:07
0.3 x R (ref. test method)	0.00015	0.007

Table 2: Evaluation of the repeatabilities of sub samples #16260

Each of the calculated repeatabilities was less than 0.3 times the corresponding reproducibility of the respective reference test method. Therefore, homogeneity of the subsamples of #16260 was assumed.

To each of the participating laboratories, 1 x 1 litre bottle (labelled #16260) was sent on November 9, 2016. A SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Ethanol (Fuel grade), packed in the amber glass bottles, was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were asked to determine on sample #16260: Acidity, Appearance, Chloride as Cl (Inorganic), Copper, Density at 20°C, Electrical conductivity at 25°C, Involatile material content, Nitrogen, pH_e, Phosphorus as P, Sulphate, Sulphur and Water (coulometric and titrimetric), Ethanol (acc. EN15721 and ASTM D5501), Higher Alcohols (acc. EN15721), Impurities (acc. EN15721) and Methanol (acc. EN15721 and ASTM D5501).

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results more, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical calculations.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalysis). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<... ' or '>... ' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the results of the statistical evaluation should be used with due care.

According to ISO 5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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

3.2 GRAPHICS

In order to 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 test results are plotted. The corresponding laboratory numbers are on the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM, EN or ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

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

The $z_{(\text{target})}$ scores are listed in the test result tables in appendix 1.

Absolute values for $z < 2$ are very common and absolute values for $z > 3$ are very rare.

The usual interpretation of z-scores is as follows:

	$ z < 1$	good
1 <	$ z < 2$	satisfactory
2 <	$ z < 3$	questionable
3 <	$ z $	unsatisfactory

4 EVALUATION

In this proficiency test some problems were encountered during the dispatch of the samples. Participants in Brazil, India and Indonesia received the samples late or not at all due to problems with clearance of the samples at customs. One participant reported the test results after the final reporting date and four participants did not report any test results at all. Not all laboratories were able to report all analyses requested.

In total 57 reported seventy-six participants reported 476 numerical test results on Ethanol (Fuel grade). Observed were 31 outlying test results, which is 6.5% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

In this section, the results are discussed per test. The reported test methods and requirements were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the reported test results. The abbreviations, used in these tables, are listed in appendix 3.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D5501) and an added designation for the year that the method was adopted or revised (e.g. D5501:12). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D5501:12 (2016)). In the results tables of Appendix 1 only the method number and year of adoption or revision will be used.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care.

Acidity: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of EN15491:07 (and ASTM D1613:06(2012) and ASTM D7795-B:15).

- Appearance: This determination was not problematic. All reporting participants agreed about the appearance of sample #16260 as Pass (clear and free of suspended matter).
- Chloride, Inorganic: All test results were below the application range of the method EN15492:12 (1 – 30 mg/kg) and ASTM D7319:13 (1 – 50 mg/kg). Therefore no significant conclusions were drawn.
- Copper: Almost all laboratories reported a 'less than' test result. Therefore no statistical conclusions were drawn.
- Density at 20°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ISO12185:96.
- Electrical Conductivity: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of EN15938:10.
- Involatile matter: All test results were below the application range of the method EN15691:09 (10 – 25 mg/100ml). Therefore no significant conclusions were drawn.
- Nitrogen: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D4629:12.
- pHe: It is known that the pHe determined with a LiCl electrode will be lower than the pHe determined with a KCl electrode. Two test methods are available for the determination of the pHe of Ethanol: ASTM D6423, that describes the use of a KCl electrode and EN15490, that describes the use of a LiCl electrode. Both test methods are used in this PT and therefore the reported pHe test results for were split up into pHe (KCl) and pHe (LiCl) and evaluated separately.
- pHe (KCl): This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of D6423:14.
- pHe (LiCl): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of EN15490:07.
- Phosphorous: Almost all test results were near or below the application range of method EN15487:07 (0.15 – 1.50 mg/kg). Therefore no statistical conclusions were drawn.

- Sulphate:** This determination may be problematic depending on the test method used for evaluation. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of EN15492:12 (and ASTM D7328:16). However, the calculated reproducibility is in full agreement with the less strict reproducibility requirements of ASTM D7319:13.
- Total Sulphur:** This determination may be problematic depending on the test method used for evaluation. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of EN15485:07. However, the calculated reproducibility is not in agreement with the reproducibility requirements of EN15486:07 or ASTM D5453:09.
- Water:** This determination is not problematic for coulometric method and the titrimetric Karl Fisher method. In total six statistical outliers were observed. The calculated reproducibility for the coulometric method after rejection of the statistical outliers is in good agreement with the requirements of EN15489:07, ASTM E1064:12 and ASTM D6304:16e1. For the titrimetric Karl Fisher method the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM E203:08 and EN15692:09.
- GC general:** In previous round robins it became clear that the test results reported for the Ethanol content is depending on the test method used by the laboratory. The test method EN15721 uses a different definition for Ethanol than ASTM D5501 does. Therefore, it was decided to request the ethanol content for both definitions in this proficiency test.
- Ethanol (EN15721):** In EN15721 the purity (the ethanol content) is defined as:
Ethanol (incl. higher alcohols) = 100% - impurity% - methanol %, where the higher alcohols consequently are not included in "impurity%".
This determination was very problematic for a number of laboratories. Seven statistical outliers were observed. Two other laboratories probably did not include the "higher alcohols" in the Ethanol content. The test results of both laboratories were excluded from the statistical calculations. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of EN15721:13.
- Higher Alcohols (EN15721):** In EN15721 the higher alcohol content is defined as:
the sum of n-propanol%, n-butanol%, sec-butanol%, isopropanol%, 2-methyl-1-butanol% and 3-methyl-1-butanol%.
This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of EN15721:13.
- Impurities (EN15721):** This determination was very problematic. In EN15721 the impurity content is defined as: content of all components except for Ethanol%, Methanol% and the "higher alcohols"%. Two statistical outliers were observed; both test results probably included incorrectly the higher alcohols. The

calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the estimated reproducibility limits using the Horwitz equation (nine components).

Methanol: This determination was problematic. Six statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated requirements calculated using the Horwitz equation, but in agreement with the estimated requirements of ASTM D5501:12. When using standard EN15721:13 a negative value for the reproducibility is found at this concentration level.

Ethanol (D5501): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5501:12.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant reference test method and the reproducibility as found for the group of participating laboratories. The average results per sample, calculated reproducibilities and reproducibilities, derived from reference test methods (in casu ASTM, ISO, EN and IEC test methods) are compared in the next tables.

Parameter	unit	n	average	2.8 *sd	R (lit)
Acidity as Acetic acid	mg/kg	42	23.6	10.1	13.7
Appearance		47	pass	n.a.	n.a.
Chloride as Cl, Inorganic	mg/kg	15	0.10	(0.23)	(0.51)
Copper	mg/kg	20	<0.07	n.a.	n.a.
Density at 20°C	kg/L	57	0.7900	0.0002	0.0005
Electrical conductivity	µS/cm	27	2.4	0.6	0.3
Involatile material content	mg/100 mL	22	1.1	(2.1)	(0.3)
Nitrogen	mg/kg	17	3.3	2.4	1.5
pHe (KCl)		19	7.04	1.15	1.09
pHe (LiCl)		8	6.48	0.68	0.62
Phosphorus as P	mg/L	23	<0.15	n.a.	n.a.
Sulphate	mg/kg	25	5.66	4.77	1.32
Total Sulphur	mg/kg	30	1.41	2.25	3.36
Water (coulometric)	%M/M	42	0.185	0.019	0.022
Water (titrimetric)	%M/M	25	0.186	0.022	0.078
Ethanol (EN15721)	%M/M	15	99.924	0.066	0.045
Higher Alcohols (EN15721)	%M/M	27	0.189	0.061	0.053
Impurities (EN15721)	%M/M	20	0.071	0.075	0.018
Methanol	%M/M	21	0.005	0.002	0.001
Ethanol (D5501)	%M/M	20	99.679	0.215	0.993

Table 3: Reproducibilities of sample #16260

Results between brackets should be used with care, because the average was near or below the application range.

Without further statistical calculations, it can be concluded that for several tests there is a good compliance of the group of participating laboratories with the relevant test methods or the rather strict calculated estimates using the Horwitz equation. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2016 WITH PREVIOUS PTS

	<i>December 2016</i>	<i>December 2015</i>	<i>November 2014</i>	<i>November 2013</i>
Number of reporting labs	57	68	68	71
Number of results reported	476	899	817	880
Statistical outliers	31	39	42	41
Percentage outliers	6.5%	4.3%	5.1%	4.7%

Table 4: Comparison with previous proficiency tests.

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

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

Determination	<i>December 2016</i>	<i>December 2015</i>	<i>November 2014</i>	<i>November 2013</i>
Acidity as Acetic Acid	+	+/-	+/-	+
Chloride as Cl, Inorganic	(++)	-	+	-
Density at 20°C	++	++	++	+
Electrical conductivity	--	-	--	--
Involatile Matter content	(--)	(--)	(--)	(--)
Nitrogen	--	--	--	-
pHe	+/-	n.e.	n.e.	n.e.
Phosphorus as P	n.e.	n.e.	(+/-)	(--)
Sulphate	--	(--)	(--)	(--)
Total Sulphur	+	++	++	++
Water (coulometric)	+	+/-	+/-	+
Water (titrimetric)	++	++	++	++
Ethanol (EN15721)	-	--	n.e.	n.e.
Higher Alcohols (EN15721)	-	-	n.e.	n.e.
Impurities (EN15721)	--	--	n.e.	n.e.
Methanol	-	--	++	++
Ethanol (D5501)	++	++	--	++

Table 5: Comparison determinations against the reference test method

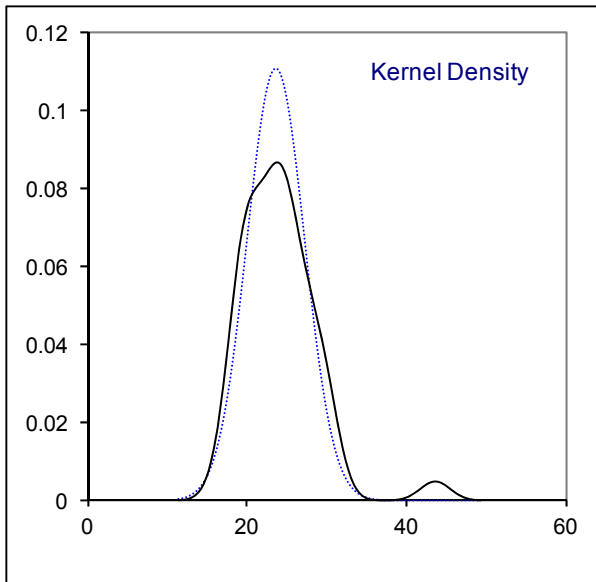
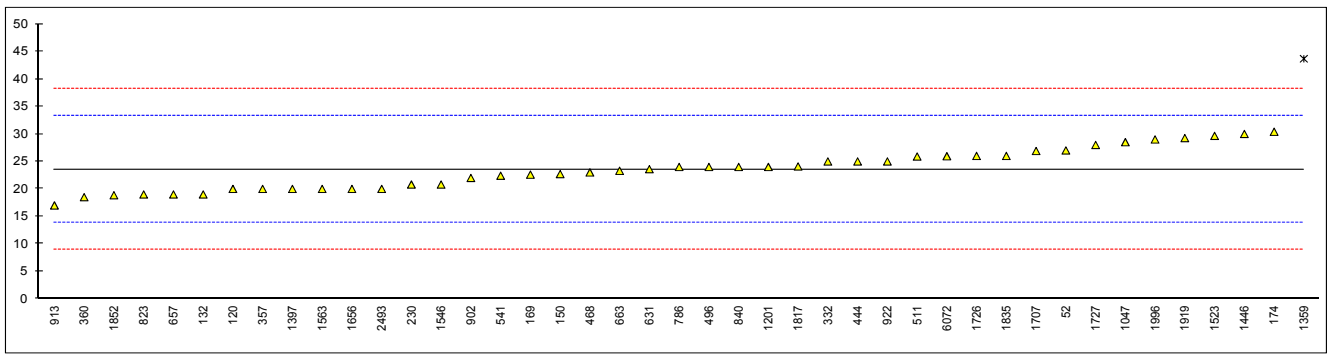
Results between brackets should be used with care, because the average was near or below the application range.

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

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

APPENDIX 1**Determination of Acidity, Total as Acetic Acid on sample #16260; results in mg/kg**

lab	method	value	mark	z(targ)	remarks
52	D1613	27		0.70	
120	D7795	20.0		-0.73	
132	D7795	19		-0.94	
150	D1613	22.7		-0.18	
169	D1613	22.6		-0.20	
171	EN15491	<30		----	
174	D1613	30.4		1.39	
230	D1613	20.8		-0.57	
311	EN15491	<30		----	
323	EN15491	<30		----	
329	EN15491	<30		----	
332	EN15491	25		0.29	
333	EN15491	<30		----	
334		----		----	
337		----		----	
338		----		----	
340	EN15491	<30		----	
357	EN15491	20		-0.73	
360	D1613	18.5		-1.04	
391		----		----	
444	EN15491	25		0.29	
447		----		----	
468	EN15491	23		-0.12	
496	EN15491	24		0.09	
511	D1613	25.9		0.47	
541	D1613	22.4		-0.24	
551		----		----	
554		----		----	
556		----		----	
621		----		----	
631	D1613	23.6		0.00	
657	D1613	19		-0.94	
663	D1613	23.3		-0.06	
786	EN15491	24		0.09	
823	D1613	19		-0.94	
840	D1613	24	C	0.09	First reported 12.14 acc. ASTM D7795
902	D1613	22		-0.32	
913	D1613	17		-1.34	
922	D1613	25		0.29	
1047	EN15491	28.5		1.01	
1126		----		----	
1201	EN15491	24		0.09	
1359	EN15491	43.67	R(0.01)	4.11	
1397	EN15491	20		-0.73	
1446	EN15491	30		1.31	
1523	ISO1388/2	29.65		1.24	
1546	EN15491	20.8	C	-0.57	First reported 0.00398 %M/M
1563	EN15491	20		-0.73	
1605		----		----	
1656	EN15491	20	C	-0.73	First reported 50
1707	D1613	26.9		0.68	
1726	EN15491	26		0.50	
1727	EN15491	28		0.90	
1788		----		----	
1817		24.09		0.11	
1835	EN15491	26		0.50	
1852	EN15491	18.86		-0.96	
1919	EN15491	29.25		1.16	
1996	EN15491	29.0		1.11	
2493	EN15491	20		-0.73	
6072	NBR9866-12	25.95		0.49	
	normality	OK			
	n	42			
	outliers	1			
	mean (n)	23.576			
	st.dev. (n)	3.6123			
	R(calc.)	10.114			
	R(EN15491:07)	13.700			Application range: 30 -150 mg/kg
	For comparison:				
	R(D1613:06)	14.000			Application range: <500 mg/kg
	R(D7795-B:15)	12.812			Application range: <200 mg/kg



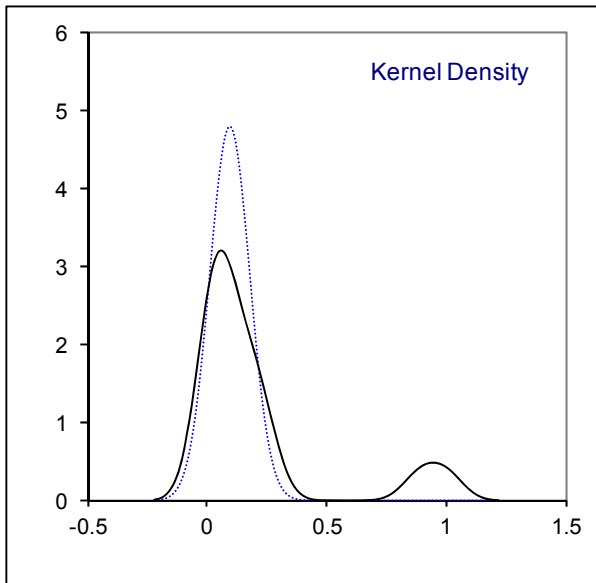
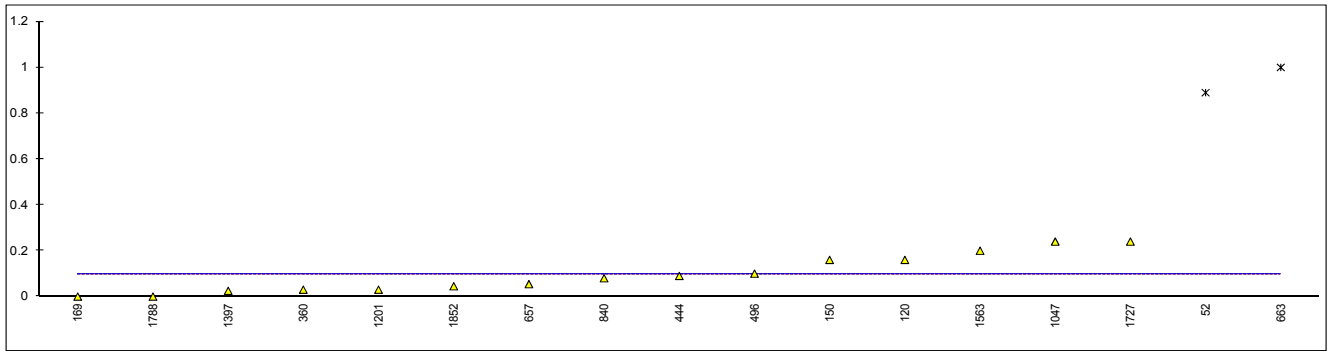
--- Empty page ---

Determination of Appearance on sample #16260;

lab	method	value	mark	z(targ)	remarks
52	D4176	Pass		----	
120	Visual	C&B		----	
132	D4176	Clear & Bright		----	
150	E2680	Pass		----	
169	D4176	Pass		----	
171	EN15769	Clear and colourless		----	
174	Visual	CFSM		----	
230	Visual	Clear & Bright		----	
311	EN15769	clear & colourless		----	
323	E2680	Clear & Bright		----	
329	E2680	clear & bright		----	
332	EN15769	Clear and colourless		----	
333	EN15769	Clear and colorless		----	
334	Visual	Clear & Bright		----	
337	Visual	Clear and bright		----	
338	Visual	Clear and bright		----	
340		----		----	
357	E2680	Pass		----	
360	EN15769	Clear and Colourless Liq.		----	
391	E2680	Pass		----	
444	EN15769	Pass		----	
447	Visual	Clear & Bright		----	
468	EN15769	C&C		----	
496	EN15769	clear and colourless		----	
511	EN15769	Clear & Bright		----	
541	Visual	C&B		----	
551		----		----	
554		----		----	
556		----		----	
621		----		----	
631	Visual	Clear & bright		----	
657	E2680	PASS		----	
663	Visual	Clear and bright		----	
786	E2680	Pass		----	
823	E2680	Pass		----	
840	E2680	Pass		----	
902	EN15769	PASS		----	
913	D2680	CLEAR		----	
922	Visual	Clear & Bright		----	
1047		----		----	
1126		----		----	
1201	D4176	Clear and bright		----	
1359	Visual	clear and colourless		----	
1397		----		----	
1446		----		----	
1523		----		----	
1546	EN15769	Clear and colorless		----	
1563	EN15769	Clear and Colourless		----	
1605		----		----	
1656	EN15769	Pass		----	
1707	Visual	C&B		----	
1726	EN15769	Clear&colorless		----	
1727	Visual	Clear&colorless		----	
1788	Visual	Clear		----	
1817		----		----	
1835	EN15769	CCL		----	
1852	Visual	clear and bright		----	
1919		----		----	
1996	EN15769	clear&colorless		----	
2493		----		----	
6072	Visual	Clear&bright		----	
n		47			
mean (n)		Pass (B&C)			

Determination of Chlorides, Inorganic as Cl on sample #16260; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	In house	0.89	G(0.01)	----	
120	D7319	0.16		----	
132	D7319	<1		----	
150	D7328	0.16		----	
169	D7319	0.000		----	
171	EN15492	<1.0		----	
174	D7319	<1.0		----	
230	D512	<2		----	
311	EN15492	<1.0		----	
323	EN15492	<1.0		----	
329	EN15492	<1,0		----	
332		----		----	
333	EN15492	<1.0		----	
334	EN15492	<1		----	
337		----		----	
338		----		----	
340		----		----	
357	EN15492	< 1		----	
360	EN15492	0.03		----	
391		----		----	
444	EN15492	0.09		----	
447		----		----	
468	EN15492	<1,0		----	
496	EN15492	0.1		----	
511		----		----	
541		----		----	
551		----		----	
554		----		----	
556		----		----	
621		----		----	
631		----		----	
657	D7328	0.054		----	
663	ISO6227	1.00	G(0.05)	----	
786		----		----	
823	D7319	<1.0		----	
840	INMPCA02	0.08		----	
902		----		----	
913		----		----	
922	D7328	<0.75		----	
1047	EN15492	0.24		----	
1126		----		----	
1201	EN15492	0.03		----	
1359		----		----	
1397	EN15492	0.025		----	
1446		----		----	
1523		----		----	
1546		----		----	
1563	EN15492	0.2		----	
1605		----		----	
1656	EN15492	<0.1		----	
1707		----		----	
1726		----		----	
1727	EN15492	0.24		----	
1788		0		----	
1817		----		----	
1835	EN15492	<1,0		----	
1852	EN15492	0.045		----	
1919		----		----	
1996	EN15492	<2		----	
2493	EN15492	<0,04		----	
6072		----		----	
	normality	OK			
	n	15			
	outliers	2			
	mean (n)	0.097			
	st.dev. (n)	0.0834			
	R(calc.)	0.233			
	R(EN15492:12)	(0.507)			Application range: 1 – 30 mg/kg
	For comparison				
	R(D7319:13)	(0.081)			Application range: 1 – 50 mg/kg



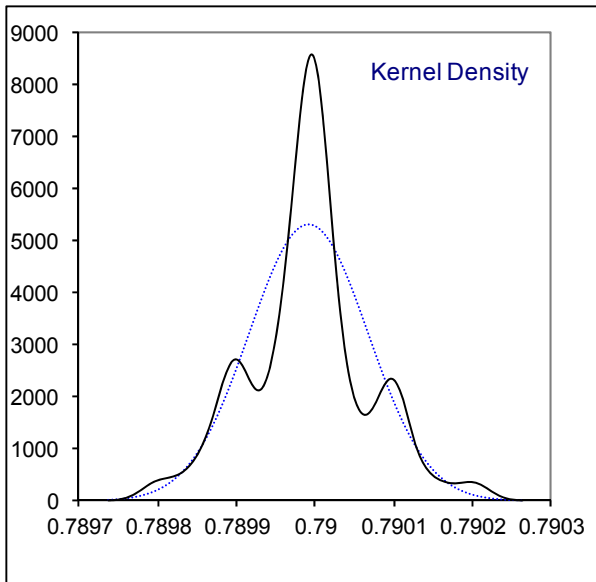
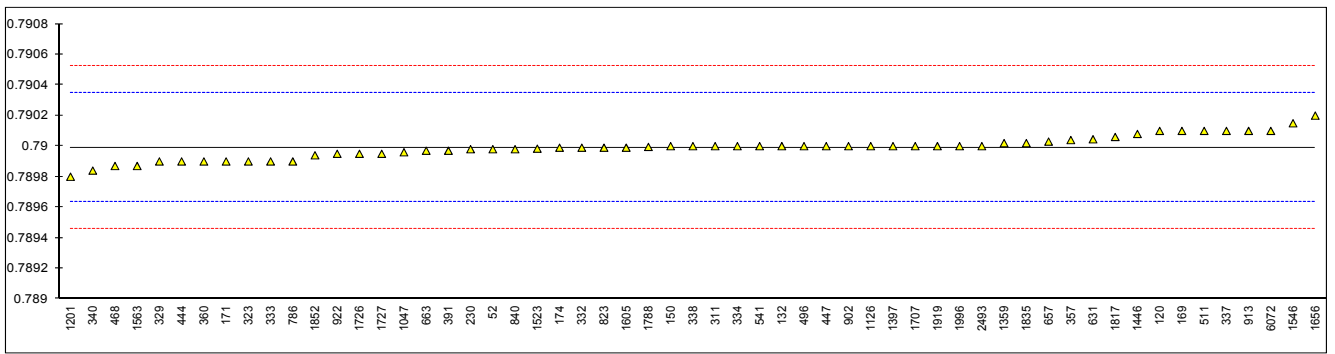
--- Empty page ---

Determination of Copper as Cu on sample #16260; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	D1688	<0.05		----	
120	D1688	<0.05		----	
132	D1688	<0.05		----	
150	D1688	<0.1		----	
169	D1688	0.000		----	
171		----		----	
174		----		----	
230		----		----	
311	EN15837	<0.050		----	
323	EN15488	<0.070		----	
329	EN15488	<0,07		----	
332		----		----	
333	EN15488	<0.07		----	
334		----		----	
337		----		----	
338		----		----	
340		----		----	
357		----		----	
360	EN15837	< 0.05		----	
391		----		----	
444	EN15488	<0.002		----	
447		----		----	
468	EN15488	<0,1		----	
496		----		----	
511	D1688	0.0037		----	
541		----		----	
551		----		----	
554		----		----	
556		----		----	
621		----		----	
631	D1688	0.01		----	
657		----		----	
663	INH-12441	0.01		----	
786		----		----	
823	UOP389	<0.01		----	
840	D1688	<0.05		----	
902		----		----	
913		----		----	
922	D1688	<0.05		----	
1047	EN15837	<0,035		----	
1126		----		----	
1201	EN15488	<0.1		----	
1359	EN15488	<1		----	
1397		----		----	
1446		----		----	
1523		----		----	
1546		----		----	
1563	EN15488	0.000		----	
1605		----		----	
1656	D1688-A	<0.01		----	
1707		----		----	
1726		----		----	
1727		----		----	
1788		----		----	
1817		----		----	
1835		----		----	
1852		----		----	
1919		----		----	
1996	EN15488	<0.05		----	
2493		----		----	
6072		----		----	
	normality	unknown			
	n	20			
	outliers	n.a.			
	mean (n)	<0.07			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(EN15488:07)	n.a.			
	For comparison				
	R(D1688:12)	n.a.			

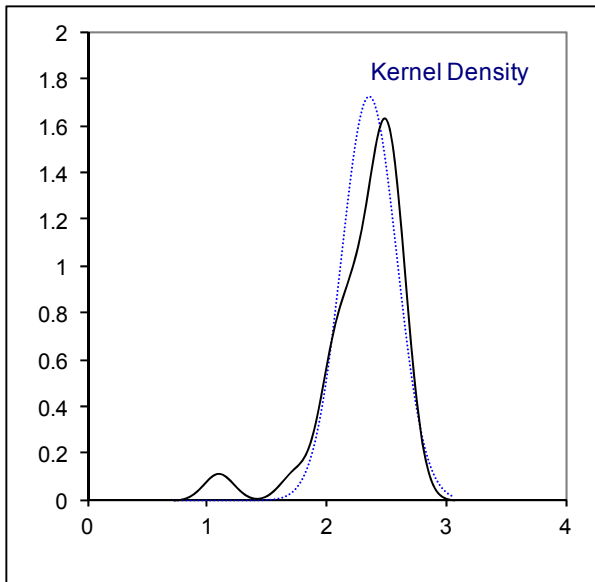
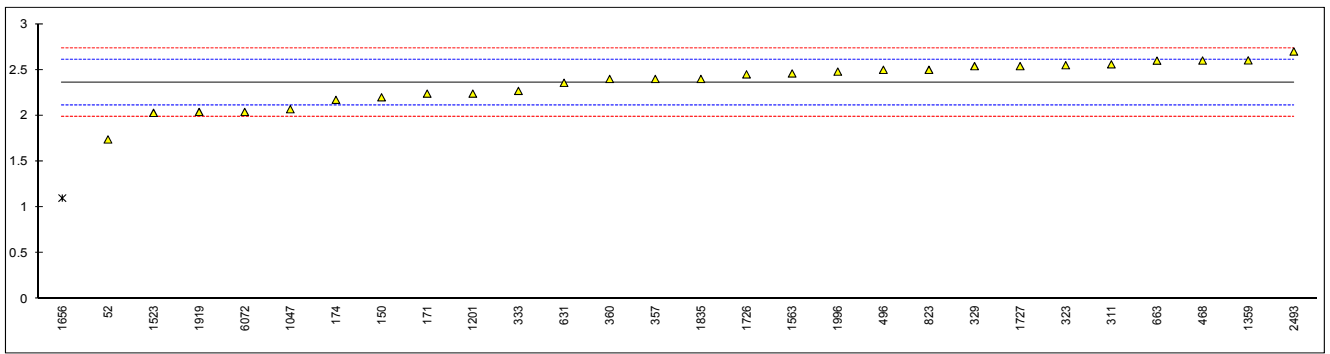
Determination of Density at 20°C on sample #16260; results in kg/L

lab	method	Value	mark	z(targ)	remarks
52	D4052	0.78998		-0.06	
120	D4052	0.7901		0.61	
132	D4052	0.7900	C	0.05	First reported 0.7905
150	D4052	0.7900		0.05	
169	D4052	0.7901		0.61	
171	D4052	0.7899		-0.51	
174	D4052	0.78999		-0.01	
230	D4052	0.78998		-0.06	
311	ISO12185	0.7900		0.05	
323	D4052	0.7899		-0.51	
329	D4052	0.7899		-0.51	
332	ISO12185	0.78999		-0.01	
333	ISO12185	0.7899		-0.51	
334	ISO12185	0.7900		0.05	
337	ISO12185	0.7901		0.61	
338	ISO12185	0.7900		0.05	
340	ISO12185	0.78984		-0.85	
357	D4052	0.79004		0.27	
360	ISO12185	0.7899		-0.51	
391	ISO12185	0.78997		-0.12	
444	D4052	0.7899		-0.51	
447	D4052	0.7900		0.05	
468	ISO12185	0.78987		-0.68	
496	ISO12185	0.79000		0.05	
511	D4052	0.79010		0.61	
541	ISO12185	0.7900		0.05	
551		----		----	
554		----		----	
556		----		----	
621		----		----	
631	D4052	0.790045		0.30	
657	D4052	0.79003		0.22	
663	D4052	0.78997		-0.12	
786	D4052	0.7899		-0.51	
823	ISO12185	0.78999		-0.01	
840	D4052	0.78998		-0.06	
902	D4052	0.7900		0.05	
913	D4052	0.7901		0.61	
922	D4052	0.78995		-0.23	
1047	ISO12185	0.78996		-0.18	
1126	ISO12185	0.790		0.05	
1201	ISO12185	0.7898		-1.07	
1359	ISO12185	0.79002		0.16	
1397	ISO12185	0.790		0.05	
1446	ISO12185	0.79008		0.50	
1523	D4052	0.7899835		-0.04	
1546	ISO12185	0.79015		0.89	
1563	INH-035	0.78987		-0.68	
1605	D4052	0.789990		-0.01	
1656	D4052	0.7902		1.17	
1707	ISO12185	0.79000		0.05	
1726	D4052	0.78995		-0.23	
1727	D4052	0.78995		-0.23	
1788	D4052	0.789995		0.02	
1817		0.79006		0.38	
1835	ISO12185	0.79002		0.16	
1852	ISO12185	0.78994		-0.29	
1919	ISO12185	0.7900		0.05	
1996	ISO12185	0.7900		0.05	
2493	ISO12185	0.7900		0.05	
6072	D4052	0.7901		0.61	
	normality	OK			
	n	57			
	outliers	0			
	mean (n)	0.78999			
	st.dev. (n)	0.000075			
	R(calc.)	0.00021			
	R(ISO12185:96)	0.00050			



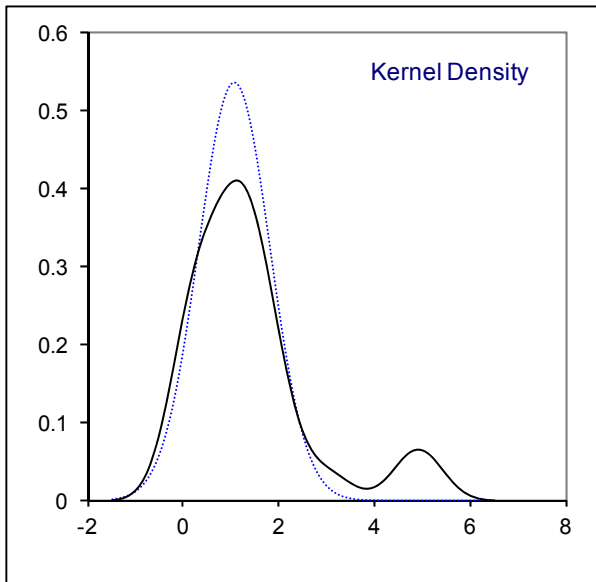
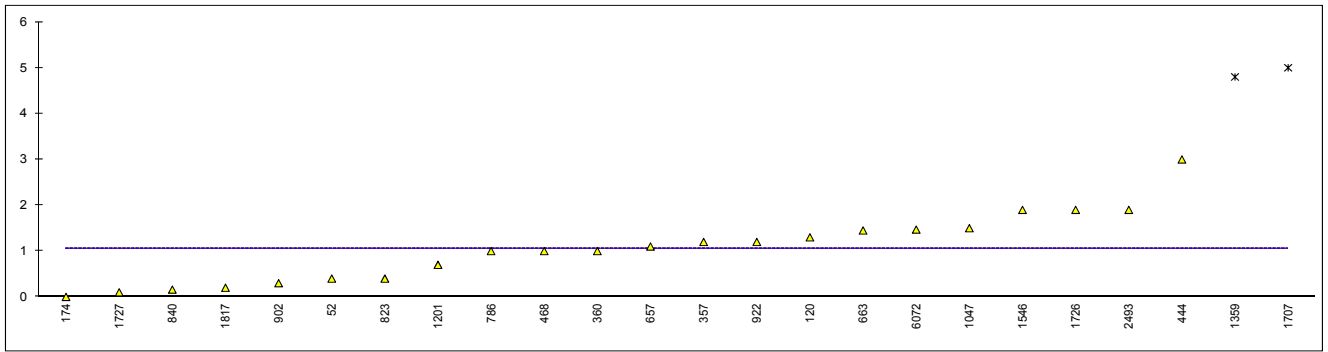
Determination of Electrical conductivity at 25°C on sample #16260; results in µS/cm

lab	method	value	mark	z(targ)	remarks
52	D1125	1.74		-4.96	
120		----		----	
132		----		----	
150	EN15938	2.2		-1.27	
169		----		----	
171	EN15938	2.24		-0.95	
174	EN15938	2.172		-1.50	
230		----		----	
311	EN15938	2.56		1.61	
323	EN15938	2.55		1.53	
329	EN15938	2.54		1.45	
332		----		----	
333	EN15938	2.27		-0.71	
334		----		----	
337		----		----	
338		----		----	
340		----		----	
357	EN15938	2.40		0.33	
360	EN15938	2.40		0.33	
391		----		----	
444		----		----	
447		----		----	
468	EN15938	2.601		1.94	
496	EN15938	2.5		1.13	
511		----		----	
541		----		----	
551		----		----	
554		----		----	
556		----		----	
621		----		----	
631	D1125	2.358		-0.01	
657		----		----	
663	D1125-A	2.6		1.93	
786		----		----	
823	EN15938	2.5		1.13	
840		----		----	
902		----		----	
913		----		----	
922		----		----	
1047	EN15938	2.07		-2.31	
1126		----		----	
1201	EN15938	2.240		-0.95	
1359	EN15938	2.603		1.96	
1397		----		----	
1446		----		----	
1523	D2624	2.03		-2.63	
1546		----		----	
1563	EN15938	2.46		0.81	
1605		----		----	
1656	EN15938	1.1	C,R(0.01)	-10.09	First reported 1.25
1707		----		----	
1726	EN15938	2.45		0.73	
1727	EN15938	2.54		1.45	
1788		----		----	
1817		----		----	
1835	EN15938	2.40		0.33	
1852		----		----	
1919	EN15938	2.04		-2.55	
1996	EN15938	2.48		0.97	
2493	EN15938	2.7		2.74	
6072	NBR10547	2.04		-2.55	
	normality	OK			
	n	27			
	outliers	1			
	mean (n)	2.359			
	st.dev. (n)	0.2310			
	R(calc.)	0.647			
	R(EN15938:10)	0.349			



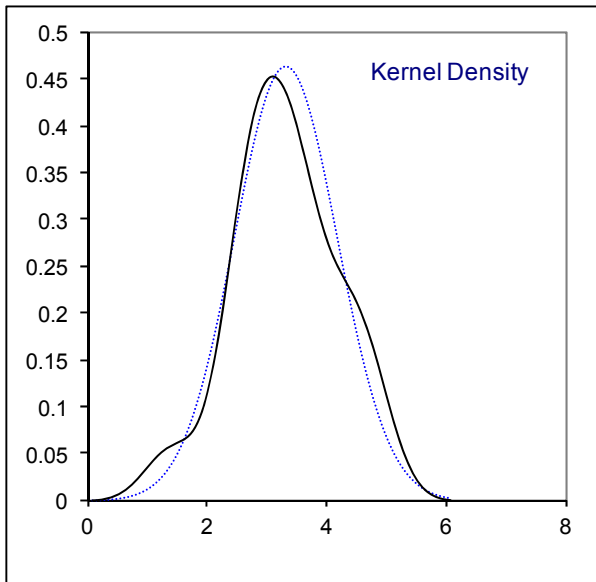
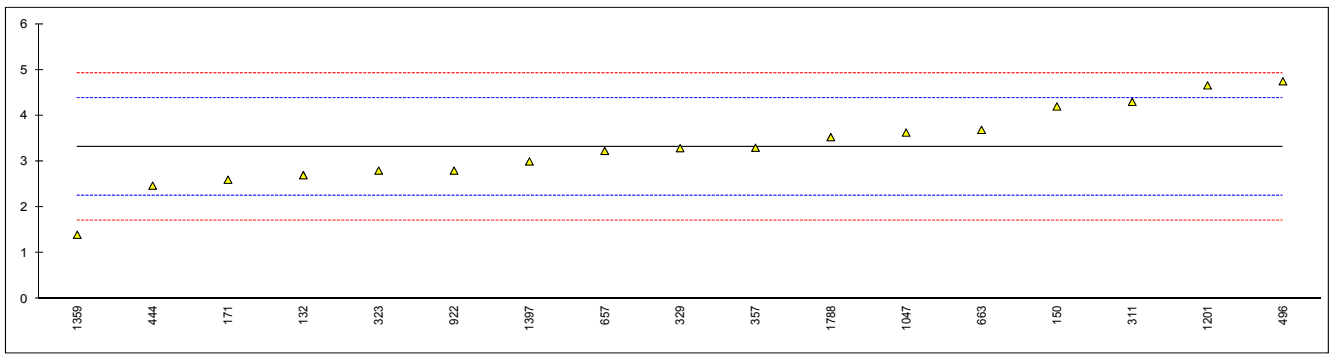
Determination of Involatile material content on sample #16260; results in mg/100mL

lab	method	value	mark	z(targ)	remarks
52	D1353	0.4		----	
120	EN15691	1.3		----	
132		----		----	
150	D1353	<1		----	
169		----		----	
171	EN15691	<10		----	
174	D1353	0		----	
230	D1353	<1		----	
311	EN15691	<10		----	
323	EN15691	<10		----	
329	EN15691	<10		----	
332		----		----	
333		----		----	
334		----		----	
337		----		----	
338		----		----	
340		----		----	
357	EN15691	1.2		----	
360	EN15691	1.0		----	
391		----		----	
444	EN15691	3		----	
447		----		----	
468	EN15691	1.0		----	
496		----		----	
511		----		----	
541	EN15691	<10		----	
551		----		----	
554		----		----	
556		----		----	
621		----		----	
631		----		----	
657	D1353	1.1		----	
663	D1353	1.45		----	
786	D1353	1.0		----	
823	D1353	0.4		----	
840	D1353	0.16		----	
902	D1353	0.3		----	
913		----		----	
922	D1353	1.20		----	
1047	EN15691	1.5		----	
1126		----		----	
1201	EN15691	0.7		----	
1359	EN15691	4.8	DG(0.01)	----	
1397		----		----	
1446		----		----	
1523		----		----	
1546	EN15691	1.9		----	
1563	EN15691	<10		----	
1605		----		----	
1656	EN15691	<1		----	
1707	EN15691	5.0	C,DG(0.01)	----	First reported 9.1
1726	EN15691	1.9		----	
1727	EN15691	0.1		----	
1788		----		----	
1817		0.2		----	
1835	EN15691	<10		----	
1852		----		----	
1919		----		----	
1996	EN15691	<10		----	
2493	D1353	1.9		----	
6072	NBR8911	1.47		----	
	normality	OK			
	n	22			
	outliers	2			
	mean (n)	1.054			
	st.dev. (n)	0.7456			
	R(calc.)	2.088			
	R(EN15691:09)	(0.255)			Application range: 10 – 25 mg/100ml
	For comparison				
	R(D1353:13)	(2.110)			



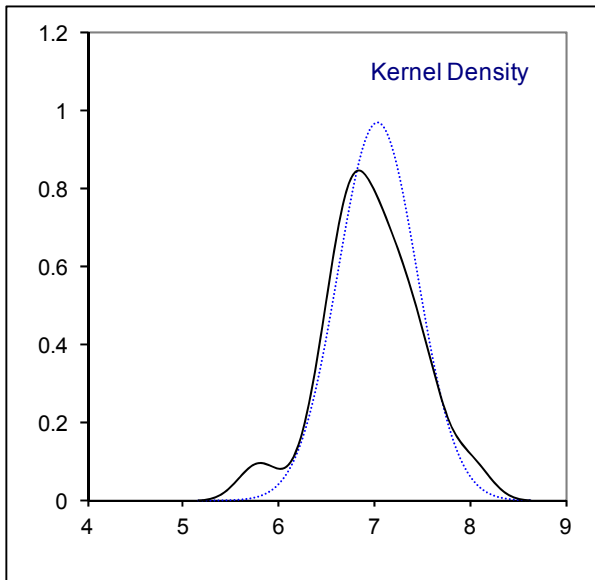
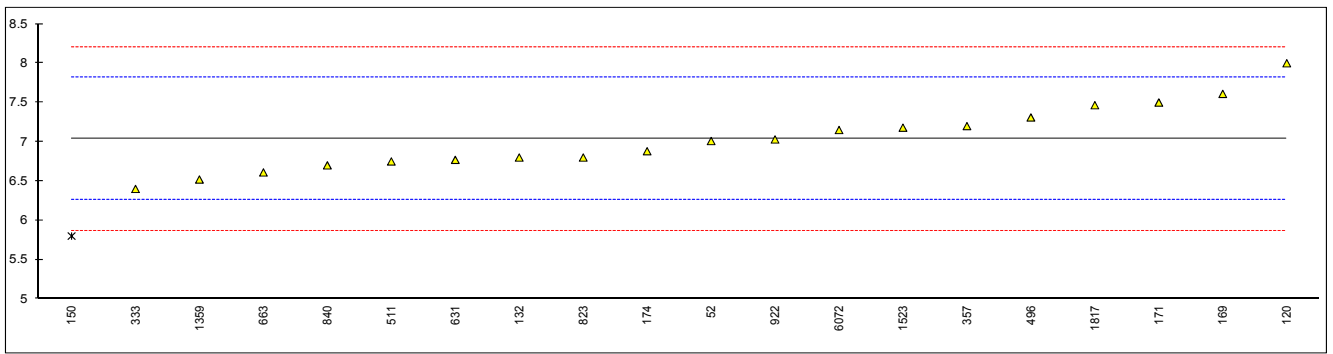
Determination of Nitrogen on sample #16260; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52		----		----	
120		----		----	
132	D4629	2.7		-1.15	
150	D4629	4.2		1.65	
169		----		----	
171	D4629	2.6		-1.33	
174		----		----	
230		----		----	
311	D4629	4.3		1.84	
323	D4629	2.8		-0.96	
329	D5762	3.29		-0.05	
332		----		----	
333		----		----	
334		----		----	
337		----		----	
338		----		----	
340		----		----	
357	D4629	3.3		-0.03	
360		----		----	
391		----		----	
444	D4629	2.472		-1.57	
447		----		----	
468		----		----	
496	D4629	4.75		2.68	
511		----		----	
541		----		----	
551		----		----	
554		----		----	
556		----		----	
621		----		----	
631		----		----	
657	D4629	3.23		-0.16	
663	D4629	3.69		0.70	
786		----		----	
823		----		----	
840		----		----	
902		----		----	
913		----		----	
922	D4629	2.80		-0.96	
1047	D4629	3.63		0.59	
1126		----		----	
1201	D4629	4.66		2.51	
1359	In house	1.40		-3.57	
1397	In house	3		-0.59	
1446		----		----	
1523		----		----	
1546		----		----	
1563		----		----	
1605		----		----	
1656		----	W	----	Result with drawn, test result reported was 5.5
1707		----		----	
1726		----		----	
1727		----		----	
1788	D4629	3.53		0.40	
1817		----		----	
1835		----		----	
1852		----		----	
1919		----		----	
1996		----		----	
2493		----		----	
6072		----		----	
	normality	OK			
	n	17			
	outliers	0			
	mean (n)	3.315			
	st.dev. (n)	0.8592			
	R(calc.)	2.406			
	R(D4629:12)	1.500			Application range : 0.3 – 100 mg/kg



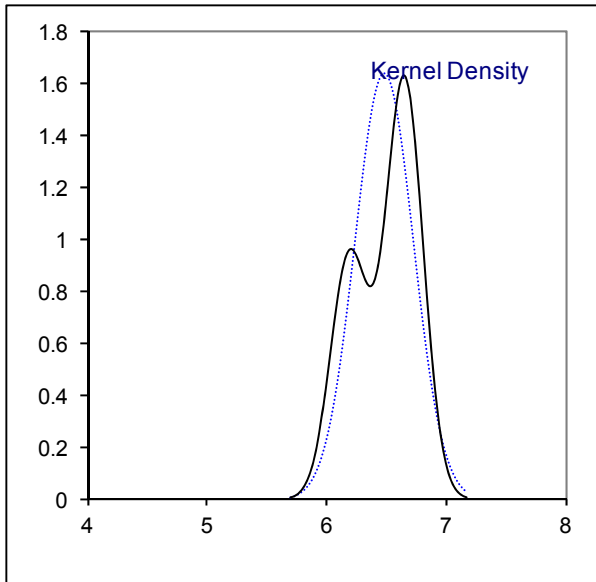
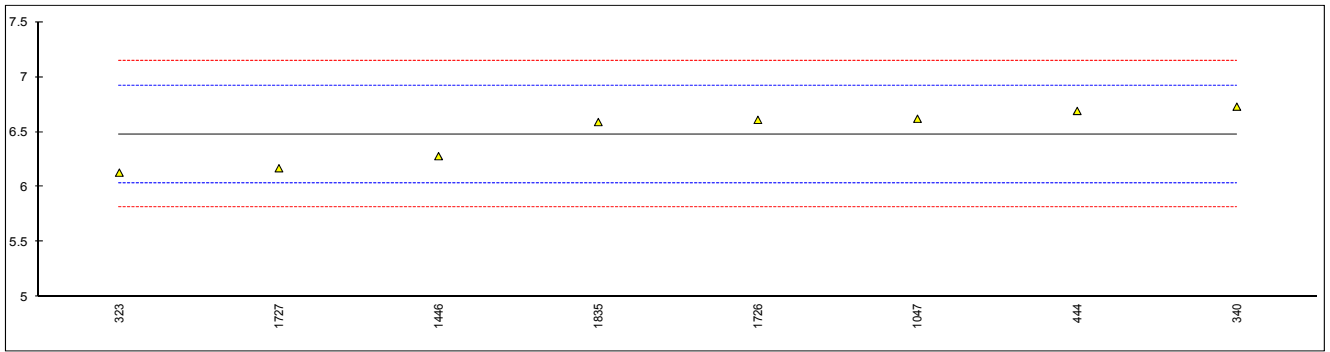
Determination of pHe (KCl) on sample #16260;

lab	method	electrode	value	mark	z(targ)	remarks
52	D6423	----	7.01		-0.07	
120	D6423	----	8.0		2.47	
132	D6423	KCl	6.8		-0.61	
150	D6423	KCl	5.8	C,G(0.05)	-3.17	First reported 8.811
169	D6423	KCl	7.61		1.47	
171	D6423	----	7.5		1.19	
174	D6423	KCl	6.88		-0.40	
230		----	----		----	
311		----	----		----	
323		----	----		----	
329		----	----		----	
332		----	----		----	
333	EN15492-mod.	KCl	6.4		-1.63	
334		----	----		----	
337		----	----		----	
338		----	----		----	
340		----	----		----	
357	D6423	----	7.2		0.42	
360		----	----		----	
391		----	----		----	
444		----	----		----	
447		----	----		----	
468		----	----		----	
496	INH-2976	KCl	7.31		0.70	
511	D6423	----	6.75		-0.73	
541		----	----		----	
551		----	----		----	
554		----	----		----	
556		----	----		----	
621		----	----		----	
631	D6423	KCl	6.77		-0.68	
657		----	----		----	
663	D6423	KCl	6.61		-1.09	
786		----	----		----	
823	D6423	KCl	6.8		-0.61	
840	D6423	KCl	6.70		-0.86	
902		----	----		----	
913		----	----		----	
922	D6423	KCl	7.03		-0.02	
1047		----	----		----	
1126		----	----		----	
1201		----	----		----	
1359	In house	KCl	6.52		-1.32	
1397		----	----		----	
1446		----	----		----	
1523	D6423	KCl	7.18		0.37	
1546		----	----		----	
1563		----	----		----	
1605		----	----		----	
1656		----	----		----	
1707		----	----		----	
1726		----	----		----	
1727		----	----		----	
1788		----	----		----	
1817	D6423	KCl	7.466		1.10	
1835		----	----		----	
1852		----	----		----	
1919		----	----		----	
1996		----	----		----	
2493		----	----		----	
6072	D6423	KCl	7.15		0.29	
	normality		OK			
	n		19			
	outliers		1			
	mean (n)		7.036			
	st.dev. (n)		0.4116			
	R(calc.)		1.152			
	R(D6423:14)		1.091			



Determination of pHe (LiCl) on sample #16260;

lab	method	Electrode	value	mark	z(targ)	remarks
52		----	----		----	
120		----	----		----	
132		----	----		----	
150		----	----		----	
169		----	----		----	
171		----	----		----	
174		----	----		----	
230		----	----		----	
311		----	----		----	
323	EN15490	LiCl	6.13		-1.57	
329		----	----		----	
332		----	----		----	
333		----	----		----	
334		----	----		----	
337		----	----		----	
338		----	----		----	
340	EN15490	LiCl	6.73		1.14	
357		----	----		----	
360		----	----		----	
391		----	----		----	
444	EN15490	Polymer elec	6.691		0.96	
447		----	----		----	
468		----	----		----	
496		----	----		----	
511		----	----		----	
541		----	----		----	
551		----	----		----	
554		----	----		----	
556		----	----		----	
621		----	----		----	
631		----	----		----	
657		----	----		----	
663		----	----		----	
786		----	----		----	
823		----	----		----	
840		----	----		----	
902		----	----		----	
913		----	----		----	
922		----	----		----	
1047	EN15490	LiCl	6.62		0.64	
1126		----	----		----	
1201		----	----		----	
1359		----	----		----	
1397		----	----		----	
1446	EN15490	LiCl	6.28		-0.89	
1523		----	----		----	
1546		----	----		----	
1563		----	----		----	
1605		----	----		----	
1656		----	----		----	
1707		----	----		----	
1726	EN15490	LiCl	6.61		0.60	
1727	EN15490	LiCl	6.17		-1.39	
1788		----	----		----	
1817		----	----		----	
1835	EN15490	LiCl	6.59		0.51	
1852		----	----		----	
1919		----	----		----	
1996		----	----		----	
2493		----	----		----	
6072		----	----		----	
	normality		unknown			
	n		8			
	outliers		0			
	mean (n)		6.478			
	st.dev. (n)		0.2432			
	R(calc.)		0.681			
	R(EN15490:07)		0.622			



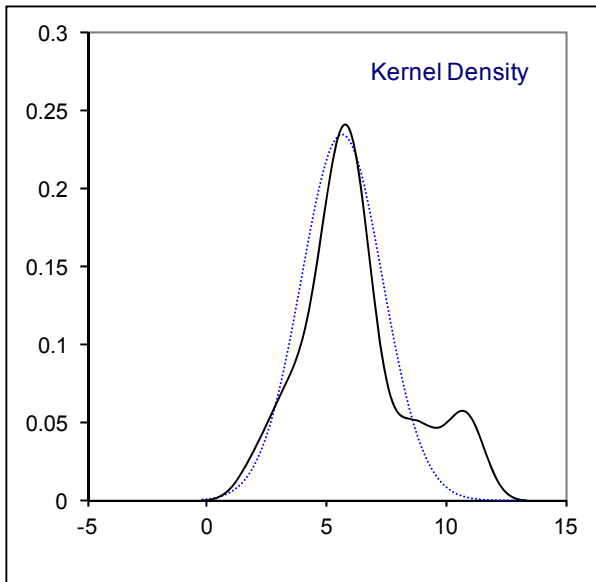
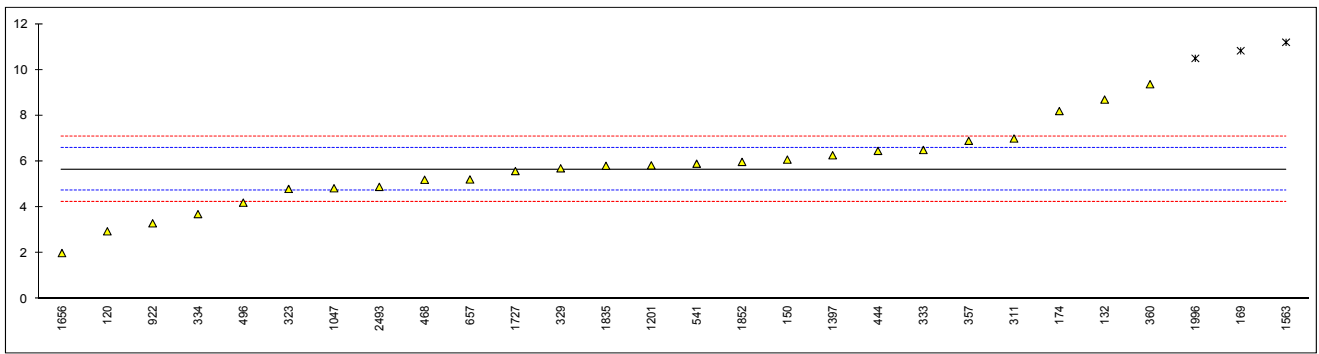
--- empty page ---

Determination of Phosphorus on sample #16260; results in mg/L

lab	method	value	mark	z(targ)	remarks
52	D3231	0.016		----	
120		----		----	
132		----		----	
150	D3231	<0.20		----	
169		----		----	
171	EN15487	<0.15		----	
174		----		----	
230		----		----	
311	EN15837	<0.13		----	
323	EN15487	<0.15		----	
329	EN15487	<0,15		----	
332		----		----	
333		----		----	
334		----		----	
337		----		----	
338		----		----	
340		----		----	
357		----		----	
360	EN15837	< 0.15		----	
391		----		----	
444	EN15487	0.0047		----	
447		----		----	
468	EN15487	<0,15		----	
496	EN15487	0.0017		----	
511		----		----	
541	EN15487	<0.15		----	
551		----		----	
554		----		----	
556		----		----	
621		----		----	
631		----		----	
657		----		----	
663		----		----	
786		----		----	
823	UOP389	<0.11		----	
840	UOP389	0.04		----	
902		----		----	
913		----		----	
922		----		----	
1047		<0,10		----	
1126		----		----	
1201	EN15487	<0.1		----	
1359	EN15487	0.004		----	
1397	EN15487	<0,1		----	
1446		----		----	
1523		----		----	
1546		----		----	
1563	EN15487	<0.15		----	
1605		----		----	
1656	EN15487	<0.01		----	
1707		----		----	
1726	EN15487	0.02		----	
1727	EN15487	<0,15		----	
1788		----		----	
1817		----		----	
1835	EN15487	<0.15		----	
1852		----		----	
1919		----		----	
1996	EN15487	<0.15		----	
2493	EN15487	<0,01		----	
6072		----		----	
	normality	unknown			
	n	23			
	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

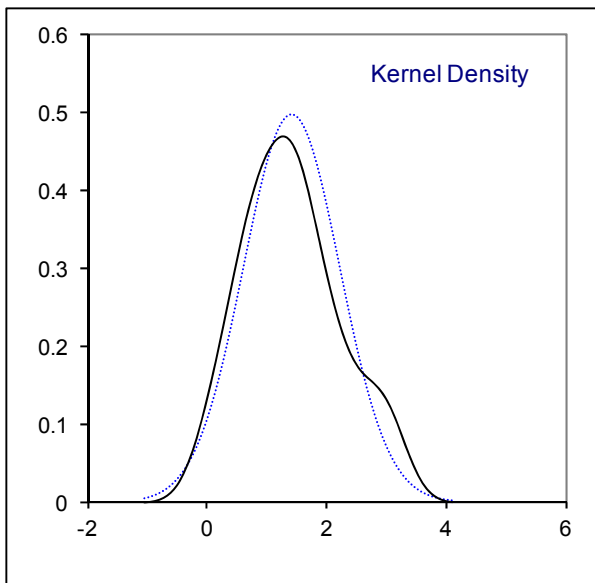
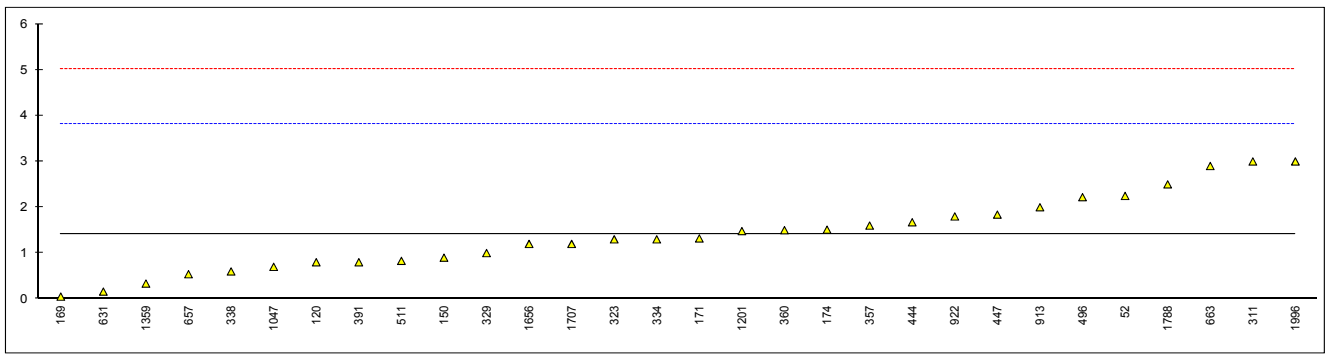
Determination of Sulphate on sample #16260; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52		----		----	
120	D7319	2.95	C	-5.72	First reported 1.06
132	D7319	8.7		6.44	
150	D7328	6.08		0.90	
169	D7319	10.83	R(0.05)	10.95	
171	EN15492	<1.0		<-9.85	False negative test result?
174	D7319	8.2	C	5.39	First reported 21.9
230		----		----	
311	EN15492	7.0		2.85	
323	EN15492	4.8		-1.81	
329	EN15492	5.7		0.10	
332		----		----	
333	EN15492	6.5		1.79	
334	EN15492	3.7		-4.14	
337		----		----	
338		----		----	
340		----		----	
357	EN15492	6.9		2.63	
360	EN15492	9.37		7.86	
391		----		----	
444	EN15492	6.46		1.70	
447		----		----	
468	EN15492	5.2		-0.96	
496	EN15492	4.20		-3.08	
511		----		----	
541	D7328	5.9	C	0.52	First reported <0.55
551		----		----	
554		----		----	
556		----		----	
621		----		----	
631		----		----	
657	D7328	5.214		-0.93	
663		----		----	
786		----		----	
823		----		----	
840		----		----	
902		----		----	
913		----		----	
922	D7328	3.30		-4.98	
1047	EN15492	4.83		-1.74	
1126		----		----	
1201	EN15492	5.83		0.37	
1359		----		----	
1397	EN15492	6.27		1.30	
1446		----		----	
1523		----		----	
1546		----		----	
1563	EN15492	11.2	R(0.05)	11.73	
1605		----		----	
1656	EN15492	2	C	-7.73	First reported 1.1
1707		----		----	
1726		----		----	
1727	EN15492	5.58		-0.16	
1788		----		----	
1817		----		----	
1835	EN15492	5.81		0.33	
1852	EN15492	5.98		0.69	
1919		----		----	
1996	EN15492	10.5	R(0.05)	10.25	
2493	EN15492	4.89		-1.62	
6072		----		----	
	normality	OK			
	n	25			
	outliers	3			
	mean (n)	5.655			
	st.dev. (n)	1.7032			
	R(calc.)	4.769			
	R(EN15492:12)	1.323			Application range : 1 – 20 mg/kg
	For comparison				
	R(D7319:13)	4.584			Application range : 1 – 50 mg/kg
	R(D7328:16)	2.101			Application range : 0.55 – 20 mg/kg



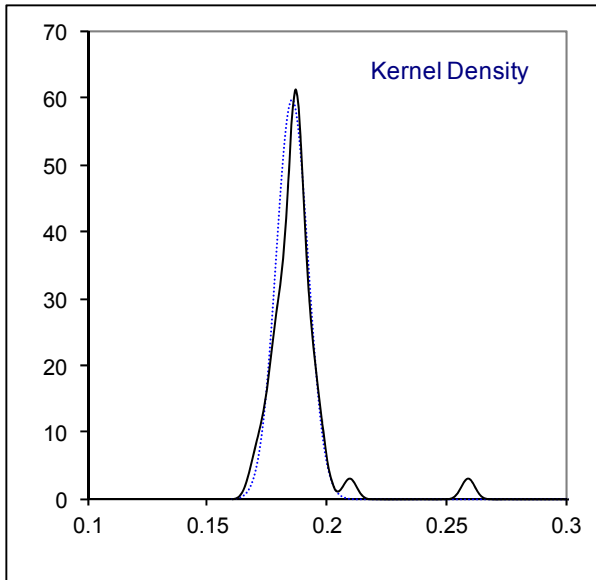
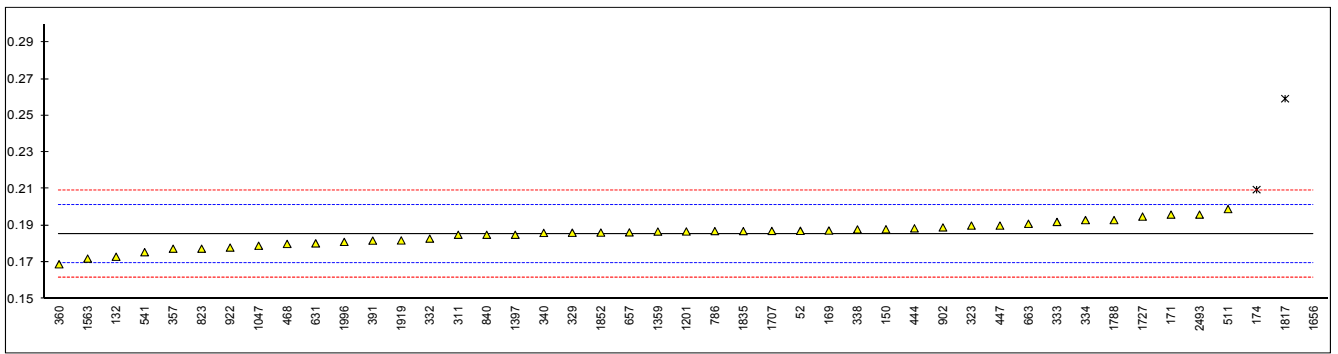
Determination of total Sulphur on sample #16260; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	D5453	2.247		0.70	
120	D5453	0.8		-0.51	
132	D5453	<1		----	
150	D5453	0.9		-0.43	
169	D5453	0.05		-1.13	
171	EN15485	1.32		-0.08	
174	D5453	1.512		0.08	
230		----		----	
311	EN15486	3.0		1.33	
323	D5453	1.3		-0.09	
329	EN15485	1		-0.34	
332		----		----	
333	ISO20846	< 3		----	
334	ISO20846	1.3		-0.09	
337		----		----	
338	D5453	0.6		-0.68	
340	ISO20846	<3		----	
357	D5453	1.6		0.16	
360	EN15486	1.50		0.07	
391	EN15485	0.80		-0.51	
444	D5453	1.673		0.22	
447	D5453	1.84		0.36	
468	EN15485	<2		----	
496	D5453	2.22		0.68	
511	D5453	0.83		-0.48	
541	D5453	<1		----	
551		----		----	
554		----		----	
556		----		----	
621		----		----	
631	D5453	0.16		-1.04	
657	D5453	0.539		-0.73	
663	D5453	2.90		1.24	
786		----		----	
823		----		----	
840		----		----	
902		----		----	
913	D5453	2.0		0.49	
922	D5453	1.80		0.32	
1047	EN15485	0.7		-0.59	
1126		----		----	
1201	EN15485	1.48		0.06	
1359	In house	0.335		-0.90	
1397	D5453	<3,0		----	
1446		----		----	
1523		----		----	
1546	ISO20846	Under 3,0		----	
1563		----		----	
1605		----		----	
1656	EN15486	1.2		-0.18	
1707	EN15485	1.20		-0.18	
1726		----		----	
1727		----		----	
1788	D5453	2.50		0.91	
1817		----		----	
1835	ISO20846	<5.0		----	
1852		----		----	
1919		----		----	
1996	EN15485	3.0		1.33	
2493		----		----	
6072		----		----	
	normality	OK			
	n	30			
	outliers	0			
	mean (n)	1.410			
	st.dev. (n)	0.8041			
	R(calc.)	2.251			
	R(EN15485:07)	3.359			Application range : 7 – 20 mg/kg
	For comparison				
	R(EN15486:07)	1.896			Application range : 5 – 20 mg/kg
	R(D5453:16e1)	0.750			Application range : 1 – 8000 mg/kg



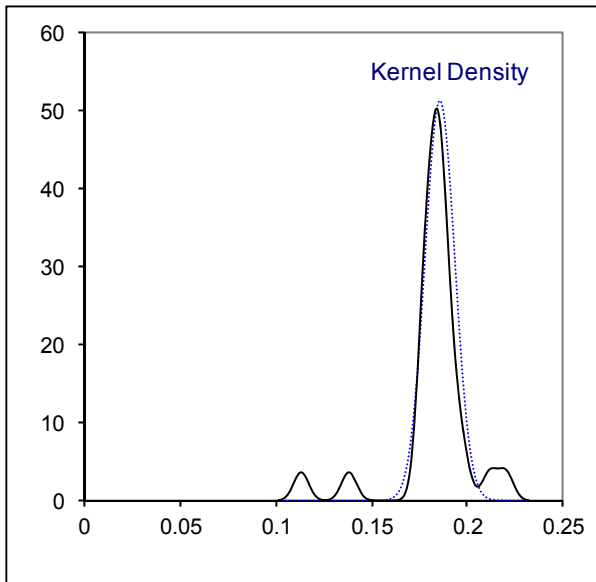
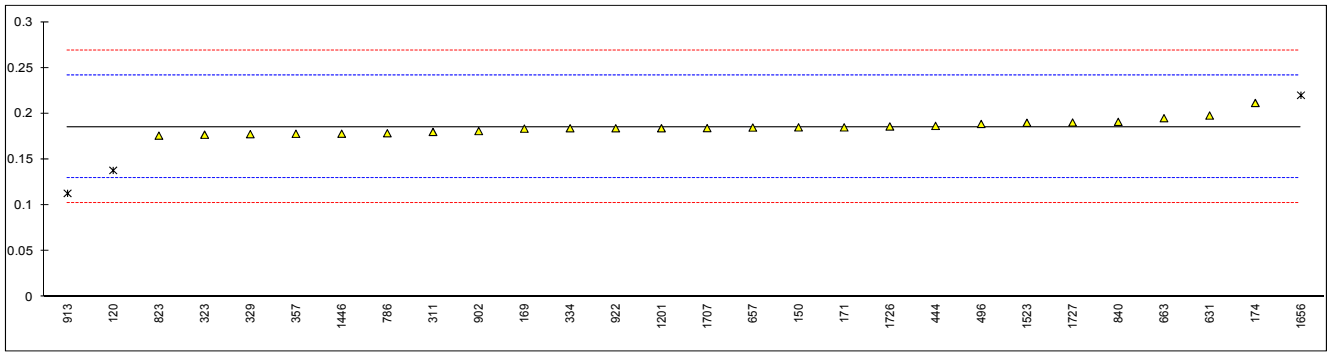
Determination of Water (coulometric) on sample #16260; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	E1064	0.18716		0.22	
120		----		----	
132	E1064	0.173		-1.56	
150	E1064	0.188		0.33	
169	E1064	0.1873		0.24	
171	EN15489	0.196		1.33	
174	E1064	0.2096	C,R(0.05)	3.04	First reported 0.218655
230		----		----	
311	EN15489	0.185		-0.05	
323	EN15489	0.190		0.58	
329	EN15489	0.1861		0.09	
332	EN15489	0.1829		-0.31	
333	EN15489	0.192		0.83	
334	EN15489	0.193		0.96	
337		----		----	
338	ISO12937	0.1879		0.32	
340	EN15489	0.186		0.08	
357	E1064	0.1774		-1.00	
360	EN15489	0.1689		-2.07	
391	EN15489	0.1818		-0.45	
444	EN15489	0.1885		0.39	
447	IP438	0.190		0.58	
468	EN15489	0.18		-0.68	
496		----		----	
511	E1064	0.199		1.71	
541	E1064	0.1755		-1.24	
551		----		----	
554		----		----	
556		----		----	
621		----		----	
631	D6304	0.1803		-0.64	
657	E1064	0.1863		0.11	
663	E1064	0.191		0.71	
786	E1064	0.1870		0.20	
823	E1064	0.1774		-1.00	
840	E1064	0.1850		-0.05	
902	EN15489	0.1890		0.45	
913		----		----	
922	D6304	0.1780		-0.93	
1047	EN15489	0.179		-0.80	
1126		----		----	
1201	EN15489	0.1868		0.18	
1359	EN15489	0.18676		0.17	
1397	EN15489	0.185		-0.05	
1446		----		----	
1523		----		----	
1546		----		----	
1563	EN15489	0.172		-1.68	
1605		----		----	
1656	EN15489	0.37	C,R(0.01)	23.20	First reported 0.25
1707	EN15489	0.1871		0.22	
1726		----		----	
1727	EN15489	0.1949		1.20	
1788	D6304	0.193		0.96	
1817		0.259238	C,R(0.01)	9.28	First reported 0.227752
1835	EN15489	0.1870		0.20	
1852	EN15489	0.1862		0.10	
1919	EN15489	0.18190		-0.44	
1996	EN15489	0.18110		-0.54	
2493	EN15489	0.19600		1.33	
6072		----	W	----	Result withdrawn, test result reported was 0.201
	normality	OK			
	n	42			
	outliers	3			
	mean (n)	0.18539			
	st.dev. (n)	0.006666			
	R(calc.)	0.01867			
	R(EN15489:07)	0.02229			
	For comparison				
	R(E1064:16)	0.03152			
	R(D6304:16e1)	0.15435			



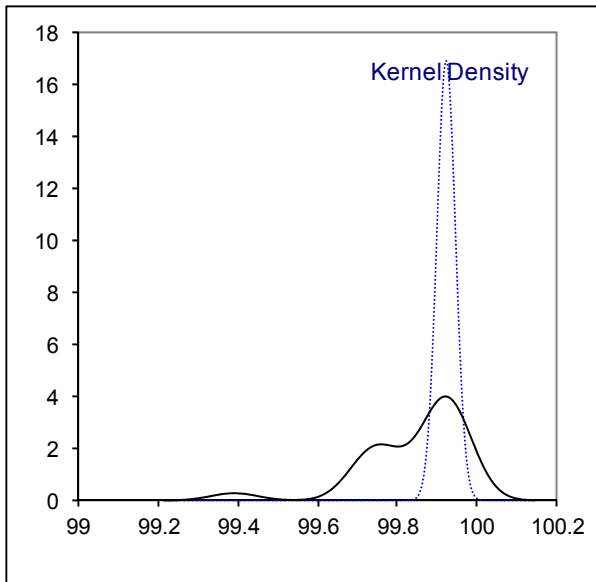
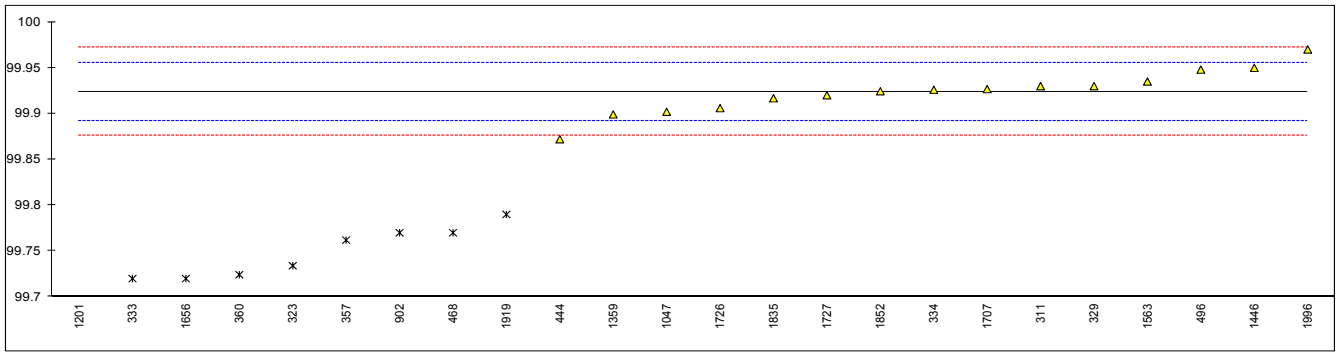
Determination of Water (titrimetric) on sample #16260; results in %M/M

lab	method	value	mark	z(targ)	remarks
52		----		----	
120	E203	0.138	C,R(0.01)	-1.71	First reported 0.128
132		----		----	
150	E203	0.185		-0.02	
169	E203	0.1836		-0.07	
171	E203	0.185		-0.02	
174	E203	0.2116		0.94	
230		----		----	
311	E203	0.180		-0.20	
323	E203	0.177		-0.31	
329	E203	0.1776		-0.28	
332		----		----	
333		----		----	
334	E203	0.184		-0.05	
337		----		----	
338		----		----	
340		----		----	
357	E203	0.1780		-0.27	
360		----		----	
391		----		----	
444	E203	0.1866		0.04	
447		----		----	
468		----		----	
496	E203	0.18875		0.12	
511		----		----	
541		----		----	
551		----		----	
554		----		----	
556		----		----	
621		----		----	
631	E203	0.1979		0.44	
657	E203	0.1848		-0.03	
663	E203	0.195		0.34	
786	E203	0.1786		-0.25	
823	E203	0.176		-0.34	
840	E203	0.1910		0.20	
902	E203	0.181		-0.16	
913	E203	0.113	R(0.01)	-2.60	
922	E203	0.1840		-0.05	
1047		----		----	
1126		----		----	
1201	E203	0.184		-0.05	
1359		----		----	
1397		----		----	
1446	ISO760	0.1781		-0.27	
1523	E203	0.19		0.16	
1546		----		----	
1563		----		----	
1605		----		----	
1656	E203	0.2200	R(0.01)	1.24	
1707	E203	0.1842		-0.05	
1726	EN15692	0.1860		0.02	
1727	EN15692	0.1903		0.17	
1788		----		----	
1817		----		----	
1835		----		----	
1852		----		----	
1919		----		----	
1996		----		----	
2493		----		----	
6072		----		----	
	normality	not OK			
	n	25			
	outliers	3			
	mean (n)	0.18552			
	st.dev. (n)	0.007794			
	R(calc.)	0.02182			
	R(E203:16)	0.07800			
	For comparison				
	R(EN15692:09)	0.09671			



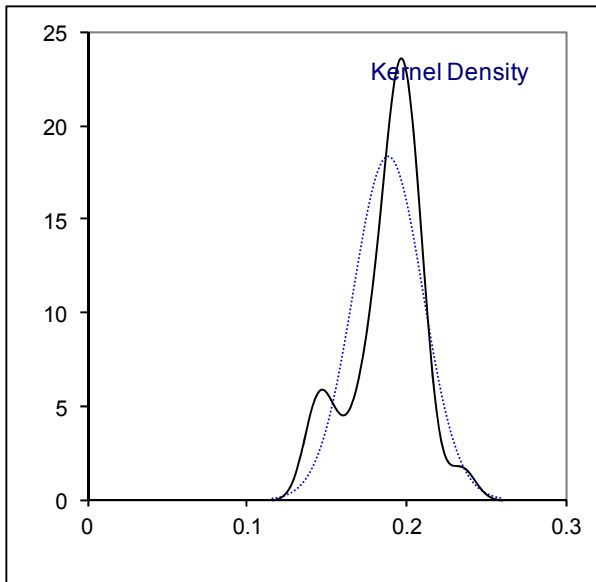
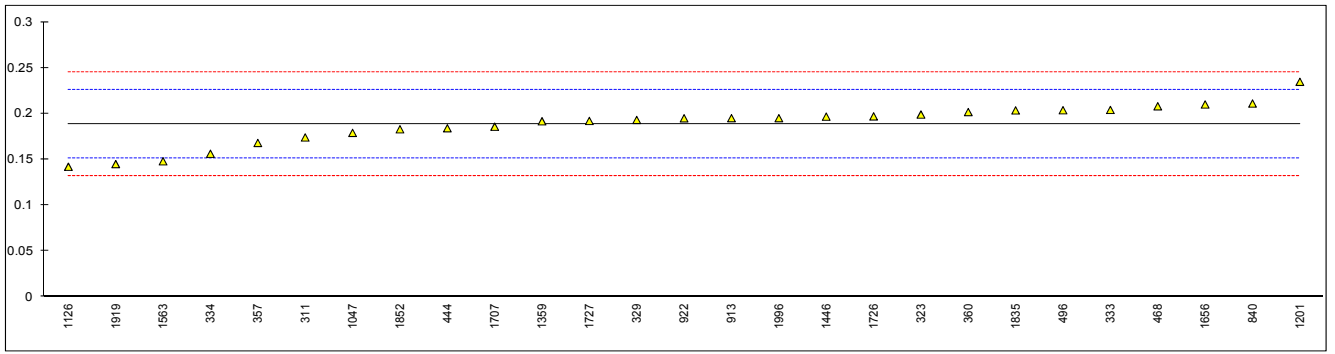
Determination of Ethanol acc. to EN15721 on sample #16260 in %M/M

lab	method	value	mark	z(targ)	remarks
52		----		----	
120		----		----	
132		----		----	
150		----		----	
169		----		----	
171		----		----	
174		----		----	
230		----		----	
311	EN15721	99.93	C	0.39	First reported 99.93
323	EN15721	99.734	R(0.01)	-11.84	
329	EN15721	99.93		0.39	
332		----		----	
333	EN15721	99.72	R(0.01)	-12.71	
334	EN15721	99.926		0.14	
337		----		----	
338		----		----	
340		----		----	
357	EN15721	99.762	ex	-10.09	Result excluded; probably included higher alcohols in impurities
360	EN15721	99.7242	R(0.01)	-12.45	
391		----		----	
444	EN15721	99.872		-3.23	
447	IP466	>98.0		----	
468	EN15721	99.77	R(0.01)	-9.59	
496	EN15721	99.9480		1.51	
511		----		----	
541		----		----	
551		----		----	
554		----		----	
556		----		----	
621		----		----	
631		----		----	
657		----		----	
663		----		----	
786		----		----	
823		----		----	
840		----		----	
902	INH-0001	99.77	R(0.01)	-9.59	
913		----		----	
922		----		----	
1047	EN15721	99.902		-1.36	
1126		----		----	
1201	EN15721	99.39	C,R(0.01)	-33.31	First reported 99.528
1359	EN15721	99.8991		-1.54	
1397		----		----	
1446	EN15721	99.95		1.64	
1523		----		----	
1546		----		----	
1563	EN15721	99.935		0.70	
1605		----		----	
1656	EN15721	99.72	R(0.01)	-12.71	
1707	EEC2870/2000R	99.92675		0.19	
1726	EN15721	99.906		-1.11	
1727	EN15721	99.920		-0.23	
1788		----		----	
1817		----		----	
1835	EN15721	99.9168		-0.43	
1852	EN15721	99.9244		0.04	
1919	EN15721	99.79	ex	-8.35	Result excluded; probably included higher alcohols in impurities
1996	EN15721	99.97		2.89	
2493		----		----	
6072		----		----	
	normality	OK			
	n	15			
	outliers	7 (+2 excl)			
	mean (n)	99.92373			
	st.dev. (n)	0.023560			
	R(calc.)	0.06597			
	R(EN15721:13)	0.04486			



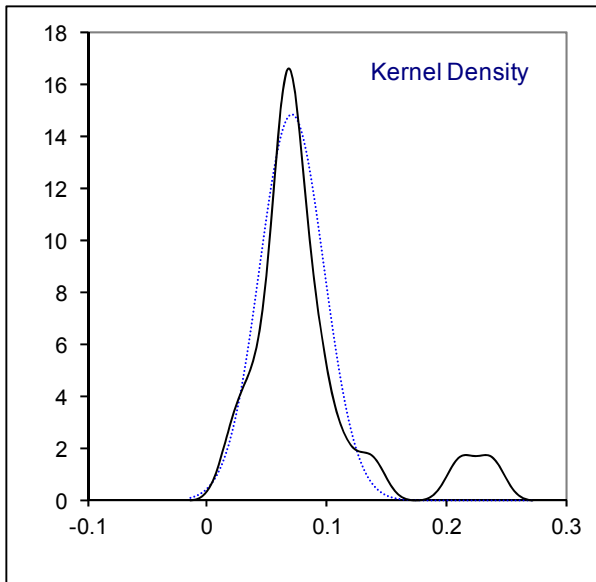
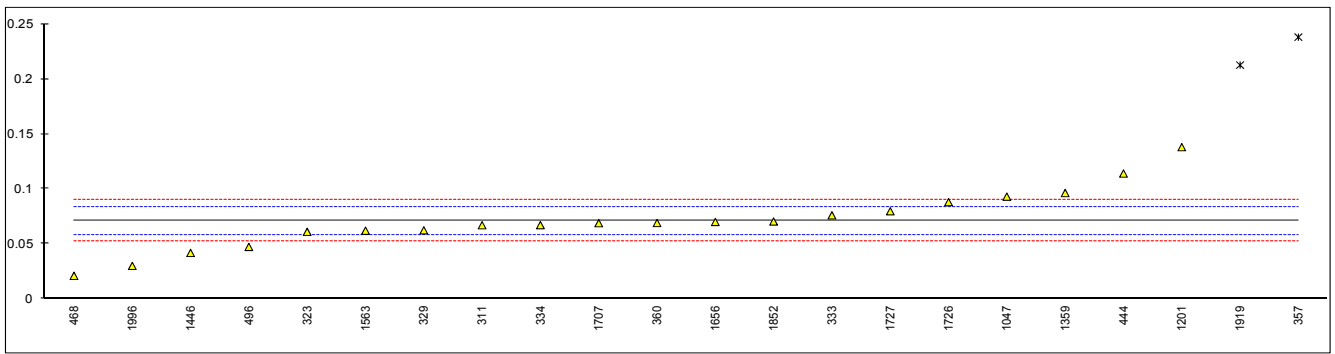
Determination of Higher Alcohols acc. to EN15721 on sample #16260; results in %M/M

lab	method	value	mark	z(targ)	remarks
52		----		----	
120		----		----	
132		----		----	
150		----		----	
169		----		----	
171		----		----	
174		----		----	
230		----		----	
311	EN15721	0.174		-0.78	
323	EN15721	0.199		0.55	
329	EN15721	0.1931		0.23	
332		----		----	
333	EN15721	0.204		0.81	
334	EN15721	0.1562		-1.73	
337		----		----	
338		----		----	
340		----		----	
357	EN15721	0.168		-1.10	
360	EN15721	0.2017		0.69	
391		----		----	
444	EN15721	0.184		-0.25	
447	IP466	<0.2		----	
468	EN15721	0.208		1.02	
496	EN15721	0.2037		0.80	
511		----		----	
541		----		----	
551		----		----	
554		----		----	
556		----		----	
621		----		----	
631		----		----	
657		----		----	
663		----		----	
786		----		----	
823		----		----	
840	EN15721	0.2110		1.18	
902		----		----	
913	INH-0001	0.1950		0.33	
922	INH-0001	0.1949		0.33	
1047	EN15721	0.179		-0.52	
1126	EN15721	0.142		-2.48	
1201	EN15721	0.2347		2.44	
1359	EN15721	0.19162		0.15	
1397		----		----	
1446	EN15721	0.1967		0.42	
1523		----		----	
1546		----		----	
1563	EN15721	0.148		-2.16	
1605		----		----	
1656	EN15721	0.21		1.13	
1707	EEC2870/2000R	0.1857		-0.16	
1726	EN15721	0.197		0.44	
1727	EN15721	0.1920		0.17	
1788		----		----	
1817		----		----	
1835	EN15721	0.2035		0.79	
1852	EN15721	0.1829		-0.31	
1919	EN15721	0.1449		-2.33	
1996	EN15721	0.195		0.33	
2493		----		----	
6072		----		----	
	normality	OK			
	n	27			
	outliers	0			
	mean (n)	0.18873			
	st.dev. (n)	0.021748			
	R(calc.)	0.06090			
	R(EN15721:13)	0.05269			



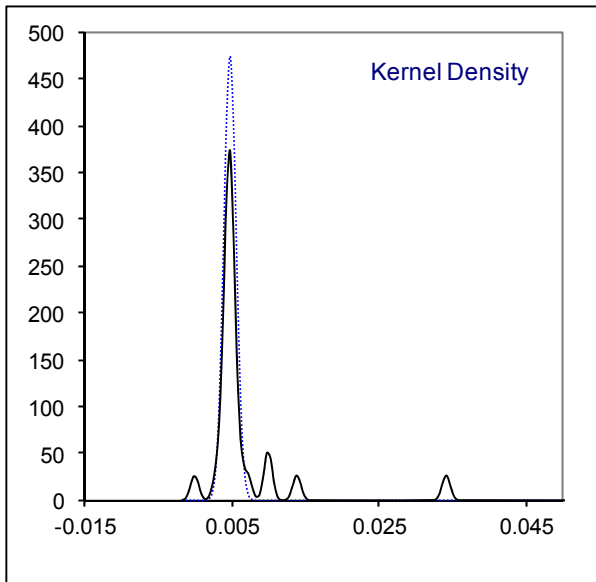
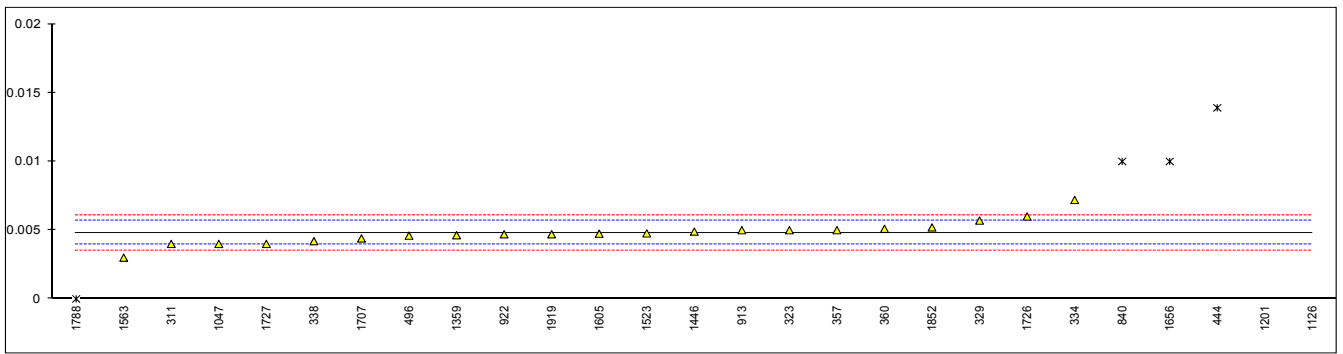
Determination of Impurities acc. to EN15721 on sample #16260; results in %M/M

lab	method	value	mark	z(targ)	remarks
52		----		----	
120		----		----	
132		----		----	
150		----		----	
169		----		----	
171		----		----	
174		----		----	
230		----		----	
311	EN15721	0.067	C	-0.65	First reported 0.241
323	EN15721	0.061		-1.60	
329	EN15721	0.0624		-1.38	
332		----		----	
333	EN15721	0.076		0.76	
334	EN15721	0.0671		-0.64	
337		----		----	
338		----		----	
340		----		----	
357	EN15721	0.238	R(0.01)	26.26	Probably included higher alcohols in impurities
360	EN15721	0.0690		-0.34	
391		----		----	
444	EN15721	0.114		6.75	
447		----		----	
468	EN15721	0.021		-7.89	
496	EN15721	0.0473		-3.75	
511		----		----	
541		----		----	
551		----		----	
554		----		----	
556		----		----	
621		----		----	
631		----		----	
657		----		----	
663		----		----	
786		----		----	
823		----		----	
840		----		----	
902		----		----	
913		----		----	
922		----		----	
1047	EN15721	0.093		3.44	
1126		----		----	
1201	EN15721	0.138	C	10.52	First reported 0.438
1359	EN15721	0.09624		3.95	
1397		----		----	
1446	EN15721	0.0418		-4.62	
1523		----		----	
1546		----		----	
1563	EN15721	0.062		-1.44	
1605		----		----	
1656	EN15721	0.07		-0.18	
1707	EEC2870/2000R	0.06885		-0.36	
1726	EN15721	0.088		2.65	
1727	EN15721	0.0797		1.35	
1788		----		----	
1817		----		----	
1835	EN15721	<0.1		----	
1852	EN15721	0.0704		-0.12	
1919	EN15721	0.2126	R(0.01)	22.26	Probably included higher alcohols in impurities
1996	EN15721	0.03		-6.47	
2493		----		----	
6072		----		----	
	normality	suspect			
	n	20			
	outliers	2			
	mean (n)	0.07114			
	st.dev. (n)	0.026891			
	R(calc.)	0.07530			
	R(Horwitz (n=9))	0.01779			



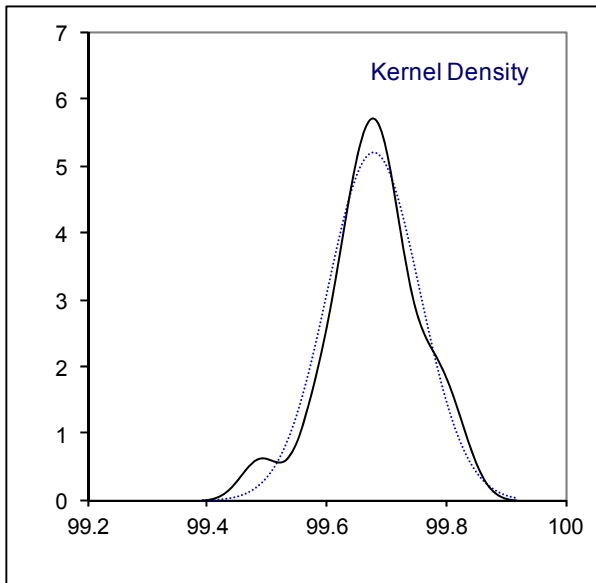
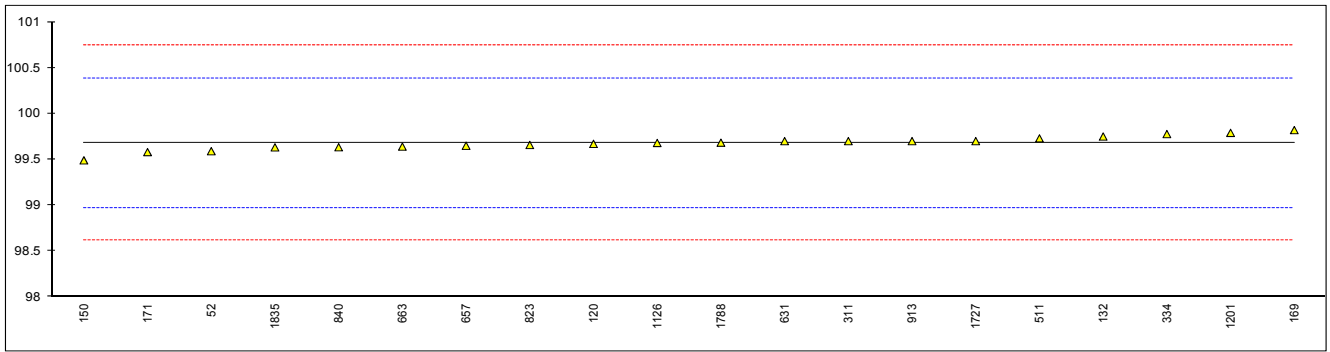
Determination of Methanol on sample #16260; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	D5501	<0.01		----	
120		----		----	
132	D5501	<0.01		----	
150		----		----	
169	D5501	<0.01		----	
171		----		----	
174		----		----	
230		----		----	
311	EN15721	0.004		-1.87	
323	EN15721	0.005		0.46	
329	EN15721	0.0057		2.09	
332		----		----	
333	EN15721	<0.100		----	
334	EN15721	0.0072		5.59	
337		----		----	
338	EN15721	0.0042		-1.40	
340		----		----	
357	EN15721	0.005		0.46	
360	EN15721	0.0051		0.70	
391		----		----	
444	EN15721	0.0139	R(0.01)	21.21	
447	IP466	<0.2		----	
468	EN15721	<0,01		----	
496	EN15721	0.0046		-0.47	
511		----		----	
541		----		----	
551		----		----	
554		----		----	
556		----		----	
621		----		----	
631	D5501	<0.01	C	----	First reported 0.02
657	D5501	<0.01	C	----	First reported 0.0204
663	D5501	<0.01		----	
786		----		----	
823	D5501	<0.01		----	
840	D5501	0.010	R(0.01)	12.12	
902		----		----	
913	INH-0001	0.0050		0.46	
922	INH-0001	0.0047		-0.24	
1047	EN15721	0.004		-1.87	
1126	EN15721	0.124	C,R(0.01)	277.86	First reported 0.023
1201	EN15721	0.0342	R(0.01)	68.53	
1359	EN15721	0.00463		-0.40	
1397		----		----	
1446	EN15721	0.0049		0.23	
1523	D5501	0.004768		-0.08	
1546		----		----	
1563	EN15721	0.003		-4.20	
1605		0.00474		-0.14	
1656	EN15721	0.01	R(0.01)	12.12	
1707	EEC2870/2000R	0.0044		-0.94	
1726	EN15721	0.006		2.79	
1727	EN15721	0.004		-1.87	
1788	D5501	0.00	R(0.05)	-11.19	
1817		----		----	
1835	EN15721	<0.1		----	
1852	EN15721	0.0052		0.93	
1919	EN15721	0.0047		-0.24	
1996	EN15721	<0.001		<-8.87	Possibly a false negative test result?
2493		----		----	
6072		----		----	
	normality	not OK			
	n	21			
	outliers	6			
	mean (n)	0.00480			
	st.dev. (n)	0.000842			
	R(calc.)	0.00236			
	R(Horwitz)	0.00120			
	For comparison				
	R(D5501:12)	0.01312			Application range : 0.01 – 0.6%M/M
	R(EN15721:12)	-0.00469			Application range: 0.1 – 3 %M/M



Determination of Ethanol acc. to ASTM D5501 on sample #16260; results in %V/V

lab	method	value	mark	z(targ)	remarks
52	D5501	99.59		-0.25	
120	D5501	99.67		-0.02	
132	D5501	99.75		0.20	
150	D5501	99.49		-0.53	
169	D5501	99.820		0.40	
171	D5501	99.58		-0.28	
174		----		----	
230		----		----	
311	D5501	99.70		0.06	
323		----		----	
329		----		----	
332		----		----	
333		----		----	
334	INH-5001	99.777		0.28	
337		----		----	
338		----		----	
340		----		----	
357		----		----	
360		----		----	
391		----		----	
444		----		----	
447		----		----	
468		----		----	
496		----		----	
511	D5501	99.73		0.14	
541		----		----	
551		----		----	
554		----		----	
556		----		----	
621		----		----	
631	D5501	99.70		0.06	
657	D5501	99.6494		-0.08	
663	D5501	99.640		-0.11	
786		----		----	
823	D5501	99.6577		-0.06	
840	D5501	99.633		-0.13	
902		----		----	
913	D5501	99.70		0.06	
922		----		----	
1047		----		----	
1126	D5501	99.68		0.00	
1201	D5501	99.79	C	0.31	First reported 0
1359		----		----	
1397		----		----	
1446		----		----	
1523		----		----	
1546		----		----	
1563		----		----	
1605		----		----	
1656		----		----	
1707		----		----	
1726		----		----	
1727	D5501	99.70		0.06	
1788	D5501	99.6829		0.01	
1817		----		----	
1835	D5501	99.632		-0.13	
1852		----		----	
1919		----		----	
1996		----		----	
2493		----		----	
6072		----		----	
	normality	OK			
	n	20			
	outliers	0			
	mean (n)	99.67859			
	st.dev. (n)	0.076752			
	R(calc.)	0.21490			
	R(D5501:12)	0.99302			



APPENDIX 2**Number of participating laboratories per country:**

1 lab in ARGENTINA
3 labs in BELGIUM
3 labs in BRAZIL
2 labs in BULGARIA
1 lab in CANADA
1 lab in COLOMBIA
1 lab in CROATIA
1 lab in CZECH REPUBLIC
1 lab in FINLAND
6 labs in FRANCE
2 labs in GERMANY
1 lab in HUNGARY
1 lab in INDIA
1 lab in INDONESIA
2 labs in ITALY
1 lab in MAURITIUS
5 labs in NETHERLANDS
1 lab in PAKISTAN
1 lab in PERU
1 lab in PHILIPPINES
1 lab in POLAND
1 lab in PORTUGAL
1 lab in RUSSIAN FEDERATION
1 lab in SINGAPORE
1 lab in SOUTH KOREA
3 labs in SPAIN
2 labs in SWEDEN
2 labs in THAILAND
2 labs in TURKEY
4 labs in UNITED KINGDOM
6 labs in UNITED STATES OF AMERICA
1 lab in VIETNAM

APPENDIX 3

Abbreviations:

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= probably an error in calculations
U	= test result probably reported in a different unit
W	= test result withdrawn on request participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= Safety Data Sheet

Literature:

- 1 iis, Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, April 2014
- 2 W. Horwitz and R. Albert, J. AOAC Int., Vol. 79, 3, p. 589, (1996)
- 3 ASTM E178:02
- 4 ASTM E1301:03
- 5 ISO 5725-86
- 6 ISO 5725, parts 1-6, 1994
- 7 ISO 13528
- 8 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 9 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 10 IP 367/84
- 11 DIN 38402 T41/42
- 12 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 13 J.N. Miller, Analyst, 118, 455, (1993)
- 14 Analytical Methods Committee Technical brief, No 4. January 2001
- 15 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry 2002, Analyst, 2002, 127, page 1359-1364.
- 16 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)
- 17 M.A. Gonçalves et.al., Sensors and Actuators B158, (2011) pages 327-332