Results of Proficiency Test
Ethanol (Fuel/ Bio-grade)
December 2018

CONTENTS

Appendices:

1   INTRODUCTION

Since 1995, the Institute for Interlaboratory Studies (iis) organizes a proficiency test (PT) for
Ethanol (Fuel/ Bio-grade) in accordance with the latest applicable version of the specifications
EN15376 and ASTM D4806 every year.
In this interlaboratory study 54 laboratories from 30 different countries for the PT on Ethanol
(Fuel/ Bio-grade) did register for participation. See appendix 2 for the number of participants per
country. In this report, the results of the 2018 interlaboratory study on Ethanol (Fuel/ Bio-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 two different samples of Ethanol (Fuel/ Bio-grade), a 1 litre bottle labelled
#18240 and a 50mL bottle labelled #18241 for Inorganic Chloride, Sulfate and total Sulfur
determination only. Participants were requested to report rounded and unrounded test results.
The unrounded test results were preferably used for 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 June 2018 (iis-protocol, version 3.5). 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 #18240 was obtained from a European supplier. The approximately 85 litres bulk material was homogenised in a pre-cleaned drum. After homogenisation 79 amber glass bottles of 1 litre were filled and labelled #18240. The homogeneity of the subsamples #18240 was checked by determination of Density in accordance with ASTM D4052 and Water in accordance with ASTM E203 on 8 stratified randomly selected samples.

	Density at 15°C in kg/L	Water in %M/M
Sample #18240-1	0.79456	0.303
Sample #18240-2	0.79454	0.303
Sample #18240-3	0.79454	0.301
Sample #18240-4	0.79455	0.303
Sample #18240-5	0.79454	0.300
Sample #18240-6	0.79454	0.303
Sample #18240-7	0.79454	0.302
Sample #18240-8	0.79454	0.302

Table 1: homogeneity test results of subsamples #18240

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities of the reference test methods in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Density at 15°C in kg/L	Water in %M/M
r (observed)	0.00002	0.003
reference test method	ISO12185:96	E203:16
0.3 x R (ref. test method)	0.00015	0.023

Table 2: evaluation of the repeatabilities of subsamples #18240

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibilities of the reference test methods. Therefore, homogeneity of the subsamples of #18240 was assumed. Please note that the Density for homogeneity purposes was determined at 15°C. The Density determination as requested from the participants was at 20°C.

The necessary bulk material for sample #18241 was obtained from a European supplier. To approximately 3.9 kg of this material, the following components, dissolved in water, were added:

Component	Amount
Sodium Chloride (NaCl)	39 mg
Sodium Sulfate (Na_2SO_4)	23 mg

Table 3: preparation table for sample #18241

After homogenisation, 78 PE bottles of 50mL were filled and labelled #18241. The homogeneity of subsamples #18241 was checked by determination of Sulfate as SO_4 in accordance with EN15492 on 8 stratified randomly selected samples.

	Sulfate in mg SO_4/kg
Sample #18241-1	4.19
Sample #18241-2	4.33
Sample #18241-3	4.76
Sample #18241-4	4.45
Sample #18241-5	4.83
Sample #18241-6	4.76
Sample #18241-7	4.34
Sample #18241-8	4.61

Table 4: homogeneity test results of subsamples #18241

From the above test results the repeatability was calculated and compared with 0.3 times the corresponding reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Sulfate in mg SO_4/kg
r (observed)	0.67
reference test method	D7319:17
0.3 x R (ref. test method)	1.16

Table 5: evaluation of the repeatability of subsamples #18241

The calculated repeatability was in agreement with 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples of #18241 was assumed.

To each of the participating laboratories, 1 x 1 litre bottle labelled #18240 and 1 x 50mL bottle labelled #18241 was sent on November 7th, 2018. An SDS was added to the sample package.

2.5   STABILITY OF THE SAMPLES

The stability of Ethanol (Fuel/ Bio-grade) packed in an amber glass bottle 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 #18240: Acidity Total, Appearance, Copper as Cu, Density at 20°C, Electrical conductivity at 25°C, Nonvolatile matter, Nitrogen, pHe (KCl & LiCl), Phosphorus as P, Water (coulometric and titrimetric), Ethanol incl. higher alcohols (acc. EN15721), Higher Alcohols (acc. EN15721), Impurities (acc. EN15721), Methanol and Ethanol by mass and by volume (acc. ASTM D5501).

The participants were asked to determine on sample #18241: Inorganic Chloride as Cl, Sulfate as SO_4 and Sulfur.

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, 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 organisation 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 June 2018 (iis-protocol, version 3.5).
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 ISO5725 the original test results per determination were submitted to Dixon's and/or 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. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying these 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 reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.
The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

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

The z-scores were calculated according to:

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

The $z_{(target)}$ scores are listed in the result tables of 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:

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

4   EVALUATION

In this proficiency test, no major problems were encountered with the dispatch of the samples. Two participants reported the test results after the final reporting date and one participant did not report any test results at all. Not all laboratories were able to report all analyses requested.

In total 53 laboratories reported 473 numerical test results. Observed were 14 outlying test results, which is 3.0% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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, see also paragraph 3.1.

4.1   EVALUATION PER SAMPLE AND PER TEST

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

Unfortunately, a suitable standard test method, providing the precision data, is not available for all determinations. For the test, that have no available precision data, the calculated reproducibility was compared against the reproducibility estimated from the Horwitz equation.

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.

Sample #18240

Acidity, Total: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN15491:07, ASTM D1613:17 and ASTM D7795-B:15.

Appearance: This determination was not problematic. All reporting participants agreed about the appearance as Pass (Clear and Bright).

Copper as Cu: Almost all participants reported a 'less than' test result. Therefore no z-scores were calculated.

Density at 20°C: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ISO12185:96.

Electrical conductivity at 25°C: This determination was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of EN15938:10.

Nonvolatile matter: All test results were below the application range of the method EN15691:09 (10-25 mg/100mL). Therefore no z-scores were calculated.

Nitrogen: This determination was problematic at the low level of 0.8 mg/kg. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D4629:12. The low number of reported test results may (partly) explain the large variation.

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 very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of D6423:14.

pHe (LiCl):   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 EN15490:07. The low number of reported test results may (partly) explain the large variation.

Phosphorus as P: Almost all test results were near or below the application range of method EN15487:07 (0.15-1.50 mg/kg). Therefore no z-scores were calculated.

Water (coulometric): This determination may be problematic depending on the test method used. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of EN15489:07 but is in agreement with ASTM E1064:16 and ASTM D6304:16e1.

Water (titrimetric): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM E203:16 and EN15692:09.

GC general: The test results reported for the Ethanol content is depending on the test method used by the laboratory. Test method EN15721 uses a different definition for Ethanol than ASTM D5501. Therefore, the participants in this proficiency test were requested to report the ethanol content for each of the two definitions.

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 <u>not</u> included in "impurity%" but in Ethanol content.
This determination was very problematic. No statistical outliers were observed. The calculated reproducibility is not at all 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 not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of EN15721:13.

Impurities (EN15721): This determination may be problematic. In EN15721 the impurity content is defined as: content of all components except for Ethanol%, Methanol% and the higher alcohols%. One statistical outlier was observed and 11 other test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the estimated reproducibility limits using the Horwitz equation based on nine components.

Methanol: This determination may be problematic depending on the test method used. Four 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. A negative value for the reproducibility is found at this concentration level for test method EN15721:13.

Ethanol (D5501): This determination was not problematic for Ethanol by mass and Ethanol by volume. No statistical outliers were observed but two test results were excluded. For both Ethanol by mass and Ethanol by volume the calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ASTM D5501:12 (2016).

Sample #18241

Chloride, Inorganic: This determination was problematic. No statistical outliers were observed, but one test result was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the estimated requirements calculated using the Horwitz equation nor with ASTM D7319:17 and EN15492:12. Because of the strict reproducibility of ASTM D7319:17 and EN15492:12 it was decided to evaluate the test results with the estimated reproducibility using the Horwitz equation. The average recovery of Inorganic Chloride (theoretical increment of 6.2 mg Chloride/kg) may be sufficient (<90%), the actual Chloride content is unknown.

Sulfate as SO_4: This determination was problematic. No statistical outliers were observed, but one test result was excluded. The calculated reproducibility is not in agreement with the requirements of ASTM D7319:17 and not at all in agreement with EN15492:12 and ASTM D7328:16. The average recovery of Sulfate (theoretical increment of 4.3 mg Sulfate/kg) may be good (<97%), the actual Sulfate content is unknown.

Sulfur: This determination may be problematic depending on the test method used. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN15485:07, but not in agreement with the reproducibility requirements of EN15486:07 and ASTM D5453:16e1.

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 number of significant test results, the average result, the calculated reproducibility (2.8 * sd) and the target reproducibilities derived from reference test methods (in casu ASTM test methods) are compared in next table.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acidity, Total as Acetic acid	mg/kg	47	24.5	11.8	13.7
Appearance		43	Pass	n.a.	n.a.
Copper as Cu	mg/kg	19	<0.07	n.a.	n.a.
Density at 20°C	kg/L	48	0.7903	0.0002	0.0005
Electrical conductivity at 25°C	µS/cm	24	0.83	0.37	0.17
Nonvolatile matter	mg/100mL	20	0.70	0.96	(0.13)
Nitrogen	mg/kg	8	0.79	1.20	0.72
pHe (KCl)		18	6.31	2.75	1.13
pHe (LiCl)		8	4.84	0.77	0.46
Phosphorus as P	mg/L	17	<0.15	n.a.	n.a.
Water (coulometric)	%M/M	38	0.313	0.027	0.025
Water (titrimetric)	%M/M	29	0.308	0.041	0.078
Ethanol incl. Higher Alcohols (EN15721)	%M/M	27	99.521	0.565	0.167
Higher Alcohols (EN15721)	%M/M	27	0.265	0.079	0.075

Parameter	unit	n	average	2.8 * sd	R (lit)
Impurities (EN15721)	%M/M	13	0.511	0.216	0.190
Methanol	%M/M	28	0.015	0.006	0.003
Ethanol (D5501)	%M/M	15	99.247	0.884	0.996
Ethanol (D5501)	%V/V	14	99.376	0.935	0.995

Table 6: reproducibilities of tests on sample #18240
Results between brackets should be used with care

Parameter	unit	n	average	2.8 * sd	R (lit)
Chloride, Inorganic as Cl	mg/kg	24	5.6	2.5	1.9
Sulfate as SO_4	mg/kg	23	4.2	5.5	3.6
Sulfur	mg/kg	31	3.3	2.6	3.5

Table 7: reproducibilities of tests on sample #18241
Reproducibility between brackets is estimated and should be used with care

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 2018 WITH PREVIOUS PTS

	December 2018	December 2017	December 2016	December 2015	November 2014
Number of reporting labs	53	59	57	68	68
Number of results reported	473	537	476	899	817
Statistical outliers	14	22	31	39	42
Percentage outliers	3.0%	4.1%	6.5%	4.3%	5.1%

Table 8: comparison of statistical summary parameters 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	December 2018	December 2017	December 2016	December 2015	November 2014
Acidity, Total as Acetic Acid	+	-	+	+/-	+/-
Copper as Cu	n.e.	n.e.	n.e.	n.e.	n.e.
Density at 20°C	++	++	++	++	++
Electrical conductivity at 25°C	--	--	--	-	--
Nonvolatile matter	(--)	(--)	(--)	(--)	(--)
Nitrogen	-	-	--	--	--
pHe	-	-	+/-	n.e.	n.e.

Determination	December 2018	December 2017	December 2016	December 2015	November 2014
Phosphorus as P	n.e.	n.e.	n.e.	n.e.	(+/-)
Water (coulometric)	+/-	+/-	+	+/-	+/-
Water (titrimetric)	+	++	++	++	++
Ethanol (EN15721)	--	-	-	--	n.e.
Higher Alcohols (EN15721)	+/-	+/-	-	-	n.e.
Impurities (EN15721)	-	-	--	--	n.e.
Methanol	--	-	-	--	++
Ethanol (D5501)	+	+	++	++	--
Chloride, Inorganic as Cl	-	+	(++)	-	+
Sulfate as SO$_4$	-	--	--	(--)	(--)
Sulfur	+	+	+	++	++

Table 9: Comparison determinations against the reference test method

Results between brackets should 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 #18240; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	D1613	23		-0.31	
120	D1613	25.5	C	0.20	reported in 25.5 %M/M
150	D7795	22		-0.52	
169	D7795	28		0.71	
171	EN15491	24		-0.11	
174	D1613	19		-1.13	
175	D7795	22.68		-0.38	
194		-----		-----	
230	D1613	17.54		-1.43	
311	D1613	30		1.12	
323	EN15491	27		0.51	
329	EN15491	28		0.71	
333	EN15491	26		0.30	
334	EN15491	20		-0.92	
337		-----		-----	
343	EN15491	30		1.12	
357	EN15491	21		-0.72	
360	EN15491	20		-0.92	
391		-----		-----	
396	D1613	24		-0.11	
444	EN15491	26		0.30	
496	EN15491	28		0.71	
541		-----		-----	
551	D1613	21.1		-0.70	
554	D1613	21.7		-0.58	
631	D1613	26.6		0.42	
633	D1613	15.3		-1.88	
657	D1613	21.5978		-0.60	
663	D1613	25.8		0.26	
823	D1613	21		-0.72	
840	D1613	30.5		1.22	
913	D1613	23		-0.31	
922	D1613	30.51		1.22	
1040	EN15491	20		-0.92	
1201	EN15491	20.7		-0.78	
1300	EN15491	25.1		0.12	
1320	EN15491	21		-0.72	
1359	EN15491	35.3044		2.20	
1397	EN15491	40	R(0.05)	3.16	
1438		-----		-----	
1446	EN15491	27		0.51	
1523	ISO1388/2	24.88		0.07	
1563		-----		-----	
1656	EN15491	30		1.12	
1710	EN15491	23		-0.31	
1712	EN15491	20		-0.92	
1726	EN15491	29		0.92	
1727	EN15491	30		1.12	
1817	ISO1388/2	23.34		-0.24	
1835	EN15491	29		0.92	
1852	EN15491	20		-0.92	
1919	EN15491	30.4		1.20	
6072	D1613	23		-0.31	
6214		22		-0.52	

normality	OK		
n	47		
outliers	1		
mean (n)	24.522		
st.dev. (n)	4.2137		
R(calc.)	11.798		
st.dev.(EN15491:07)	4.8929		
R(EN15491:07)	13.7		application range: 30 - 150 mg/kg
Compare:			
R(D1613:17)	14		application range: <500 mg/kg
R(D7795-B:15)	13.024		application range: <200 mg/kg
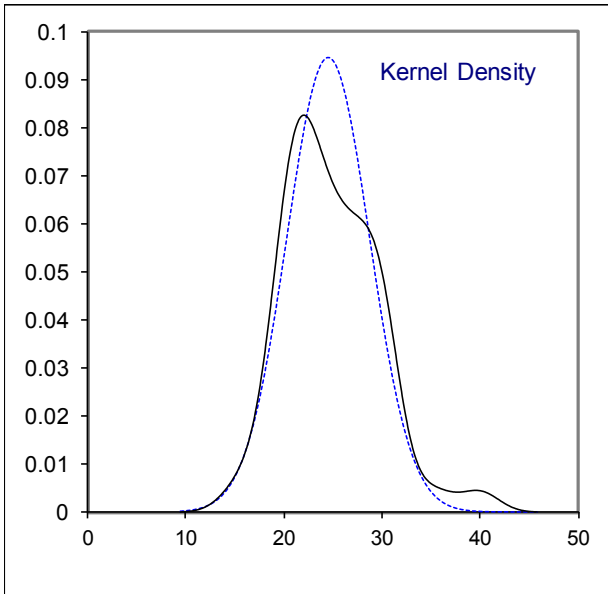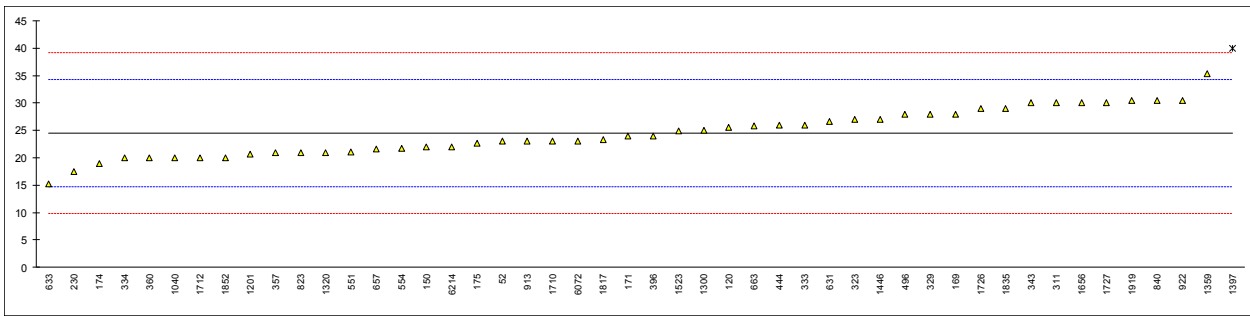
Determination of Appearance on sample #18240;

lab	method	value	mark	z(targ)	remarks
52	D4176	Pass		-----	
120	D4176	Clear and Bright		-----	
150		-----		-----	
169	D4176	Pass		-----	
171	Visual	Clear and Free		-----	
174	Visual	Clear & Free		-----	
175	D4176	clear and bright		-----	
194	Visual	Clear and bright		-----	
230	Visual	Clear & Bright		-----	
311	EN15769	colourless & clear		-----	
323	D4176	pass		-----	
329	Visual	clear		-----	
333	EN15769	Clear and Colourless		-----	
334		-----		-----	
337	Visual	colorless		-----	
343		C&B		-----	
357	E2680	Pass		-----	
360	EN15769	Clear and Colourless		-----	
391		-----		-----	
396	E2680	Pass		-----	
444	EN15769	Pass		-----	
496	EN15769	clear and colourless		-----	
541	E2680	Pass		-----	
551	E2680	Pass		-----	
554	Visual	Pass		-----	
631	Visual	clear and bright		-----	
633	Visual	Clear & Bright		-----	
657	E2680	Pass		-----	
663	Visual	Clear & Bright		-----	
823	E2680	Pass		-----	
840	E2680	Pass		-----	
913	E2680	Clear & Bright		-----	
922	Visual	Clear & Bright		-----	
1040	Visual	clear & bright		-----	
1201	Visual	Clear&Colourless		-----	
1300	EN15769	clear&colorless		-----	
1320		-----		-----	
1359	Visual	Clear / Bright		-----	
1397	EN15769	clear, colourless		-----	
1438		-----		-----	
1446		-----		-----	
1523		-----		-----	
1563		-----		-----	
1656	EN15769	pass		-----	
1710	D4176	Clear&Bright		-----	
1712	EN15769	C&B		-----	
1726	EN15769	Clear&colourless		-----	
1727	Visual	Clear&Colorless		-----	
1817		-----		-----	
1835	EN15769	C&C		-----	
1852		-----		-----	
1919		-----		-----	
6072	Visual	Libre de contaminantes suspendidos o precipitados		-----	
6214	EN15769	clear and colourless		-----	
	n	43			
	mean (n)	Pass (Clear & Bright)			

Determination of Copper as Cu on sample #18240; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	D1688	<0.05		-----	
120		-----		-----	
150		-----		-----	
169		-----		-----	
171	EN15488	<0.070		-----	
174		-----		-----	
175	D1688	0.001		-----	
194		-----		-----	
230	In house	0.02		-----	
311	EN15837	<0.0050		-----	
323	EN15488	<0.070		-----	
329		-----		-----	
333	EN15488	< 0.07		-----	
334		-----		-----	
337		-----		-----	
343	EN15488	<0,070		-----	
357		-----		-----	
360	EN15837	< 0.050		-----	
391		-----		-----	
396		-----		-----	
444		-----		-----	
496		-----		-----	
541	NBR11331	<0.1		-----	
551	OGC2047	<0.04		-----	
554		-----		-----	
631	D1688	<0.05		-----	
633		-----		-----	
657		-----		-----	
663	INH-12414	0.001		-----	
823	UOP389	<0.01		-----	
840	D1688	<0.05		-----	
913		-----		-----	
922	D1688	<0.05		-----	
1040		-----		-----	
1201	EN15488	<1		-----	
1300	EN15837	<0.05		-----	
1320		-----		-----	
1359	EN15488	1.0800		-----	possibly a false positive test result?
1397		-----		-----	
1438		-----		-----	
1446		-----		-----	
1523		-----		-----	
1563		-----		-----	
1656	D1688-A	<0.1		-----	
1710		-----		-----	
1712	EN15488	<0,07		-----	
1726		-----		-----	
1727		-----		-----	
1817		-----		-----	
1835	EN15837	<0.050		-----	
1852		-----		-----	
1919		-----		-----	
6072		-----		-----	
6214		0.00322		-----	

normality	unknown	
n	19	
outliers	n.a.	
mean (n)	<0.07	
st.dev. (n)	n.a.	
R(calc.)	n.a.	
st.dev.(EN15488:07)	n.a.	
R(EN15488:07)	n.a.	application range 0.07 – 0.20 mg/kg
Compare:		
R(D1688:12)	n.a.	

Determination of Density at 20°C on sample #18240; results in kg/L

lab	method	value	mark	z(targ)	remarks
52	D4052	0.7903		0.05	
120	D4052	0.7904		0.61	
150	D4052	0.7903		0.05	
169	D4052	0.7905		1.17	
171	ISO12185	0.7903		0.05	
174	D4052	0.7903		0.05	
175	D4052	0.79028		-0.06	
194	D4052	0.7910	R(0.01)	3.97	
230	D4052	0.79030		0.05	
311	D4052	0.7902		-0.51	
323	ISO12185	0.7902		-0.51	
329	D4052	0.7902		-0.51	
333	ISO12185	0.7902		-0.51	
334	ISO12185	0.7901		-1.07	
337	ISO12185	0.7905		1.17	
343	ISO12185	0.7903		0.05	
357	D4052	0.79028		-0.06	
360	ISO12185	0.7902		-0.51	
391	ISO12185	0.7902		-0.51	
396	D4052	0.7902		-0.51	
444	D4052	0.7903		0.05	
496	ISO12185	0.79027		-0.12	
541	D4052	0.79030		0.05	
551	D4052	0.79033		0.22	
554	D4052	0.7904		0.61	
631	D4052	0.79026		-0.17	
633	D4052	0.7903		0.05	
657	D4052	0.79030		0.05	
663	D4052	0.79032		0.16	
823	D4052	0.79031		0.11	
840	D4052	0.79034		0.27	
913	D4052	0.7905		1.17	
922	D4052	0.79013		-0.90	
1040	ISO12185	0.79033		0.22	
1201	D4052	0.7903		0.05	
1300	ISO12185	0.79027		-0.12	
1320		-----		-----	
1359	ISO12185	0.790273		-0.10	
1397		-----		-----	
1438		-----		-----	
1446	ISO12185	0.79036		0.39	
1523	D4052	0.790256		-0.20	
1563		-----		-----	
1656	D4052	0.7902		-0.51	
1710	ISO12185	0.7903		0.05	
1712	ISO12185	0.7905		1.17	
1726	D4052	0.79026		-0.17	
1727	D4052	0.79023		-0.34	
1817	Table OIML	0.79031		0.11	
1835	ISO12185	0.79027		-0.12	
1852		-----		-----	
1919	ISO12185	0.790310		0.11	
6072	D4052	0.79026		-0.17	
6214		0.79022		-0.40	

normality	suspect	
n	48	
outliers	1	
mean (n)	0.79029	
st.dev. (n)	0.000087	
R(calc.)	0.00024	
st.dev.(ISO12185:96)	0.000179	
R(ISO12185:96)	0.0005	
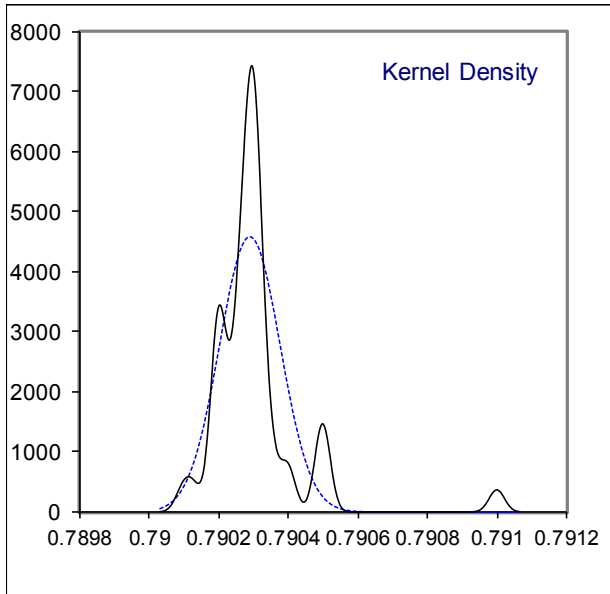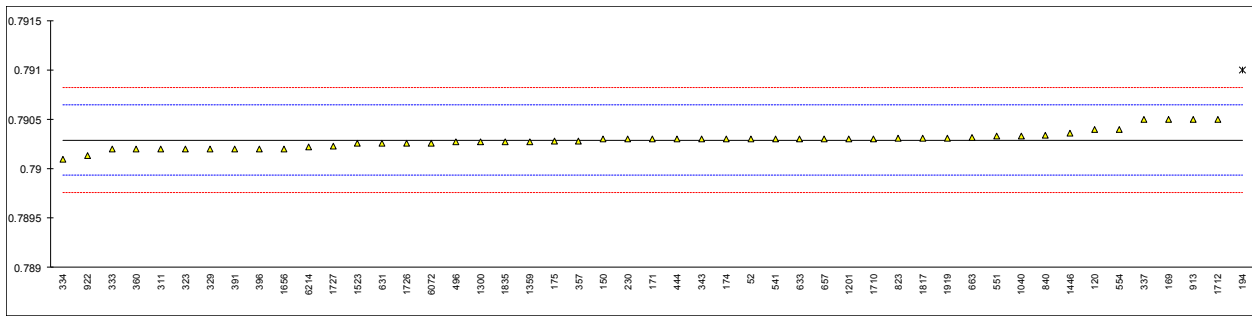
Determination of Electrical conductivity at 25°C on sample #18240; results in µS/cm

lab	method	value	mark	z(targ)	remarks
52	D1125Mod.	0.91		1.36	
120		-----		-----	
150		0.9		1.19	
169		-----		-----	
171	EN15938	0.78		-0.82	
174	D1125-A	<10		-----	
175		-----		-----	
194		-----		-----	
230		-----		-----	
311	EN15938	0.60		-3.83	
323	EN15938	<0.3		<-8.86	possibly a false negative test result?
329	EN15938	0.55		-4.67	
333	EN15938	1.01		3.03	
334	EN15938	0.83		0.02	
337		-----		-----	
343		-----		-----	
357		-----		-----	
360	EN15938	0.80		-0.49	
391		-----		-----	
396		-----		-----	
444		-----		-----	
496	EN15938	0.8		-0.49	
541		-----		-----	
551	NBR10547	0.88		0.85	
554	NBR10547	0.85		0.35	
631	D1125-A	0.92		1.52	
633		-----		-----	
657		-----		-----	
663		-----		-----	
823	D1125	0.9		1.19	
840		-----		-----	
913		-----		-----	
922		-----		-----	
1040		-----		-----	
1201	EN15938	1.142		5.24	
1300	EN15938	0.617		-3.55	
1320		-----		-----	
1359	EN15938	0.9655		2.28	
1397		-----		-----	
1438		-----		-----	
1446		-----		-----	
1523	D2624	0.74		-1.49	
1563		-----		-----	
1656	EN15938	<1		-----	
1710		-----		-----	
1712	EN15938	0.740		-1.49	
1726	EN15938	0.85		0.35	
1727	EN15938	0.8505		0.36	
1817	In house	0.920		1.52	
1835	EN15938	0.80		-0.49	
1852		-----		-----	
1919	EN15938	0.37	R(0.05)	-7.69	
6072	NBR10547	0.792		-0.62	
6214		0.75		-1.32	
	normality	OK			
	n	24			
	outliers	1			
	mean (n)	0.829			
	st.dev. (n)	0.1302			
	R(calc.)	0.365			
	st.dev.(EN15938:10)	0.0597			
	R(EN15938:10)	0.167			

Kernel Density
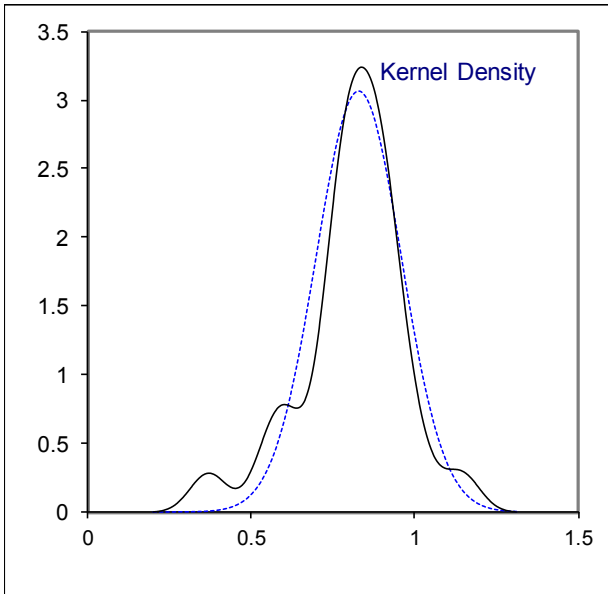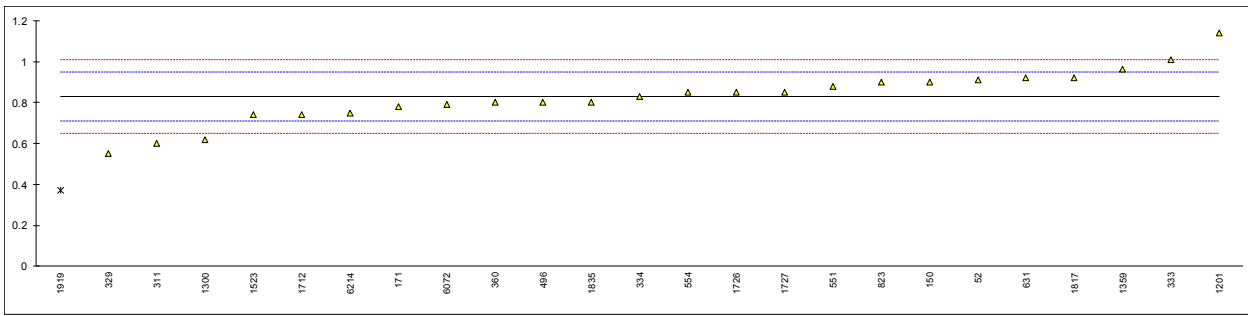
Determination of Nonvolatile matter on sample #18240; results in mg/100mL

lab	method	value	mark	z(targ)	remarks
52	D1353	<1		-----	
120		-----		-----	
150	D1353	0		-----	
169	D1353	0.8		-----	
171	EN15691	0.0007		-----	
174	D1353	0.2		-----	
175		-----		-----	
194		-----		-----	
230	D1353	0.5		-----	
311	EN15691	<10		-----	
323	EN15691	<1		-----	
329	EN15691	0.6		-----	
333	EN15691	< 10		-----	
334		-----		-----	
337		-----		-----	
343	EN15691	1		-----	
357	EN15691	1.0		-----	
360	EN15691	1.0		-----	
391		-----		-----	
396		-----		-----	
444	EN15691	<10		-----	
496		-----		-----	
541	D1353	1.30		-----	
551	D1353	0.8		-----	
554	D1353	0.8		-----	
631		-----		-----	
633		-----		-----	
657	D1353	0.8		-----	
663		-----		-----	
823		-----		-----	
840	D1353	0.54		-----	
913		-----		-----	
922	D1353	0.80		-----	
1040		-----		-----	
1201	EN15691	0.55		-----	
1300	EN15691	0.6		-----	
1320		-----		-----	
1359	EN15691	3.1	R(0.01)	-----	
1397		-----		-----	
1438		-----		-----	
1446		-----		-----	
1523		-----		-----	
1563		-----		-----	
1656	EN15691	<1		-----	
1710	EN15691	0.7		-----	
1712	EN15691	<5		-----	
1726	EN15691	1.1		-----	
1727	EN15691	0.9		-----	
1817		-----		-----	
1835	EN15691	<10		-----	
1852		-----		-----	
1919		-----		-----	
6072		-----		-----	
6214		2.6	R(0.01)	-----	

normality	OK		
n	20		
outliers	2		
mean (n)	0.70		
st.dev. (n)	0.342		
R(calc.)	0.96		
st.dev.(EN15691:09)	(0.046)		
R(EN15691:09)	(0.13)		application range: 10 – 25 mg/100mL
Compare:			
R(D1353:13)	(0.30)		

Kernel Density
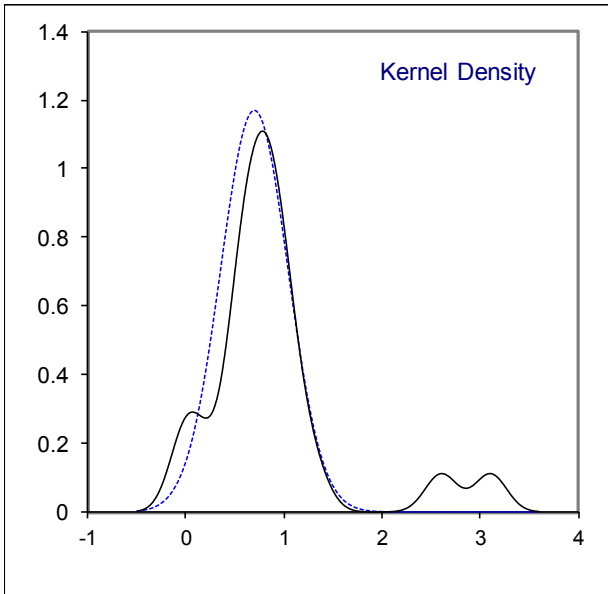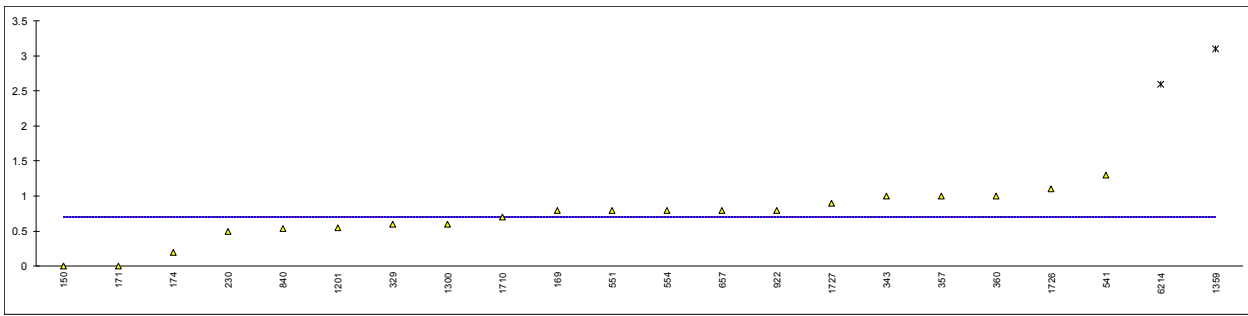
Determination of Nitrogen on sample #18240; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	D4629	0.6		-0.73	
120		-----		-----	
150	D4629	<0.3		-----	
169		-----		-----	
171	D4629	<0.3		-----	
174		-----		-----	
175		-----		-----	
194		-----		-----	
230		-----		-----	
311		-----		-----	
323	D4629	<1		-----	
329		-----		-----	
333		-----		-----	
334		-----		-----	
337		-----		-----	
343		-----		-----	
357	D4629	0.5		-1.12	
360		-----		-----	
391		-----		-----	
396		-----		-----	
444	D4629	0.687		-0.39	
496		-----		-----	
541		-----		-----	
551	D4629	1.02		0.91	
554		-----		-----	
631		-----		-----	
633		-----		-----	
657	D4629	1.2527		1.82	
663		-----		-----	
823		-----		-----	
840		-----		-----	
913		-----		-----	
922	D4629	<0.3		-----	
1040		-----		-----	
1201	D4629	<1		-----	
1300	D4629	1.469	C	2.67	first reported 3.469
1320		-----		-----	
1359	In house	0.17		-2.42	
1397		-----		-----	
1438		-----		-----	
1446		-----		-----	
1523		-----		-----	
1563		-----		-----	
1656		-----		-----	
1710	D4629	0.6		-0.73	
1712		-----		-----	
1726		-----		-----	
1727		-----		-----	
1817		-----		-----	
1835		-----		-----	
1852		-----		-----	
1919		-----		-----	
6072		-----		-----	
6214		-----		-----	

normality	unknown	
n	8	
outliers	0	
mean (n)	0.787	
st.dev. (n)	0.4275	
R(calc.)	1.197	
st.dev.(D4629:12)	0.2556	
R(D4629:12)	0.716	application range 0.3 - 100 mg/kg

Kernel Density
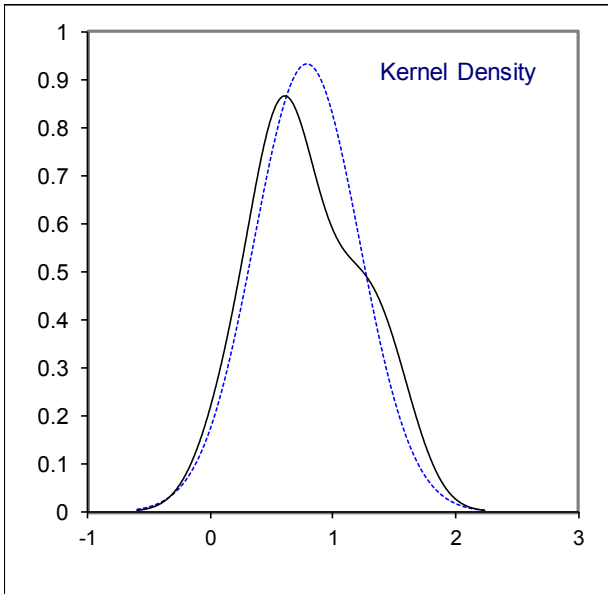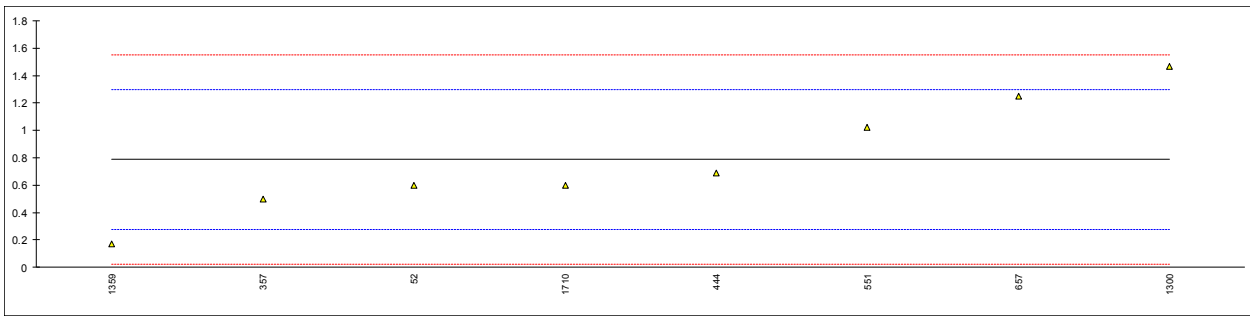
Determination of pHe (KCl) on sample #18240;

lab	method	value	mark	z(targ)	remarks
52	D6423	6.9		1.46	
120		-----		-----	
150	D6423	5.48		-2.06	
169	D6423	6.46		0.37	no details on which type of electrode was used
171	D6423	6.4		0.22	
174	D6423	6.7		0.97	
175		-----		-----	
194		-----		-----	
230		-----		-----	
311		-----		-----	
323		-----		-----	
329		-----		-----	
333	EN15490	5.27		-2.59	
334		-----		-----	
337		-----		-----	
343		-----		-----	
357	D6423	5.0		-3.26	
360		-----		-----	
391		-----		-----	
396		-----		-----	
444		-----		-----	
496	D6423	7.42		2.76	
541		-----		-----	
551	D6423	5.21		-2.74	
554	D6423	5.80		-1.27	
631	D6423	7.82		3.75	
633		-----		-----	
657		-----		-----	
663	D6423	7.37		2.63	
823	D6423	5.1		-3.01	
840		-----		-----	
913		-----		-----	
922	D6423	5.83		-1.19	
1040		-----		-----	
1201	EN15490	7.56		3.10	
1300		-----		-----	
1320		-----		-----	
1359	EN15490	7.747		3.57	
1397		-----		-----	
1438		-----		-----	
1446		-----		-----	
1523	D6423	6.19		-0.30	
1563		-----		-----	
1656		-----		-----	
1710		-----		-----	
1712		-----		-----	
1726		-----		-----	
1727		-----		-----	
1817		-----		-----	
1835		-----		-----	
1852		-----		-----	
1919		-----		-----	
6072	D6423	5.34		-2.41	
6214		-----		-----	
	normality	OK			
	n	18			
	outliers	0			
	mean (n)	6.311			
	st.dev. (n)	0.9822			
	R(calc.)	2.750			
	st.dev.(D6423:14)	0.4025			
	R(D6423:14)	1.127			
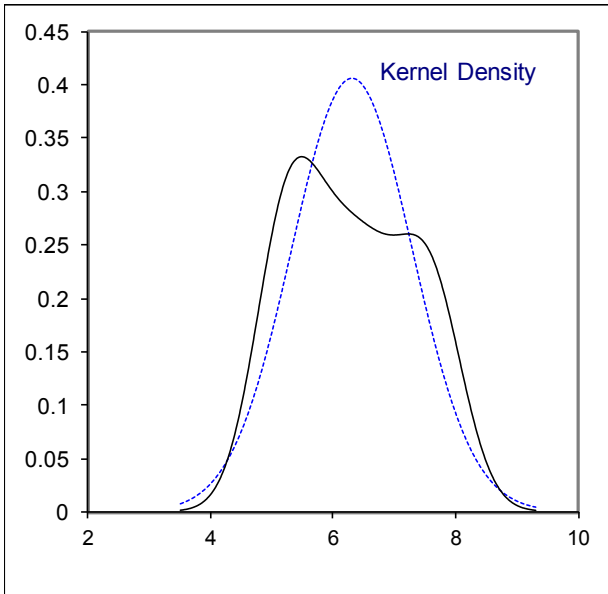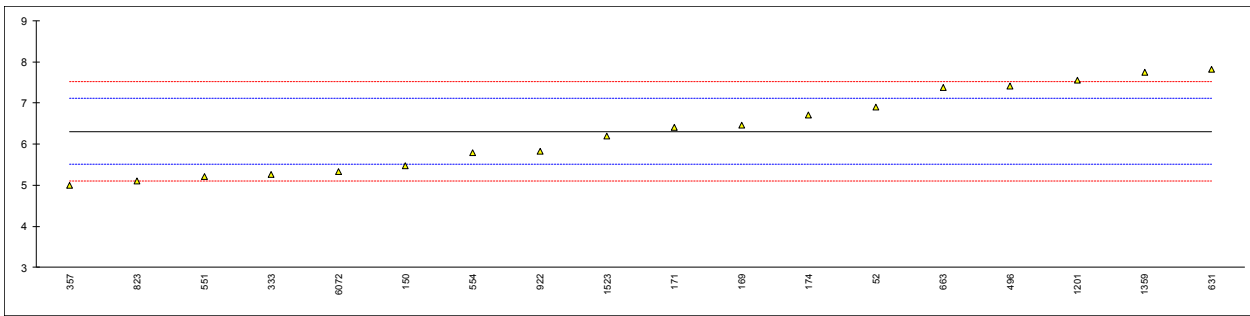
Determination of pHe (LiCl) on sample #18240;

lab	method	value	mark	z(targ)	remarks
52		-----		-----	
120	D6423	4.39		-2.70	
150		-----		-----	
169		-----		-----	
171		-----		-----	
174		-----		-----	
175	D6423	6.28	D(0.05)	8.70	possibly used a KCl electrode?
194		-----		-----	
230		-----		-----	
311		-----		-----	
323	EN15490	4.66		-1.07	
329		-----		-----	
333		-----		-----	
334	EN15490	4.92		0.50	
337		-----		-----	
343		-----		-----	
357		-----		-----	
360		-----		-----	
391		-----		-----	
396		-----		-----	
444		-----		-----	
496		-----		-----	
541		-----		-----	
551		-----		-----	
554		-----		-----	
631		-----		-----	
633		-----		-----	
657		-----		-----	
663		-----		-----	
823		-----		-----	
840		-----		-----	
913		-----		-----	
922		-----		-----	
1040		-----		-----	
1201		-----		-----	
1300	EN15490	5.352		3.10	
1320		-----		-----	
1359		-----		-----	
1397		-----		-----	
1438		-----		-----	
1446	EN15490	4.90		0.38	
1523		-----		-----	
1563		-----		-----	
1656		-----		-----	
1710		-----		-----	
1712		-----		-----	
1726	EN15490	4.76		-0.47	
1727	EN15490	4.96		0.74	
1817		-----		-----	
1835	EN15490	4.76		-0.47	
1852		-----		-----	
1919		-----		-----	
6072		-----		-----	
6214		-----		-----	

normality	suspect
n	8
outliers	1
mean (n)	4.838
st.dev. (n)	0.2763
R(calc.)	0.774
st.dev.(EN15490:07)	0.1659
R(EN15490:07)	0.464

Kernel Density
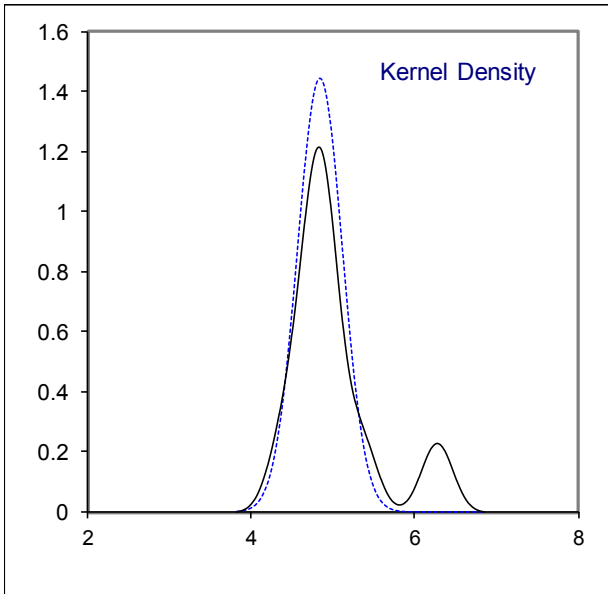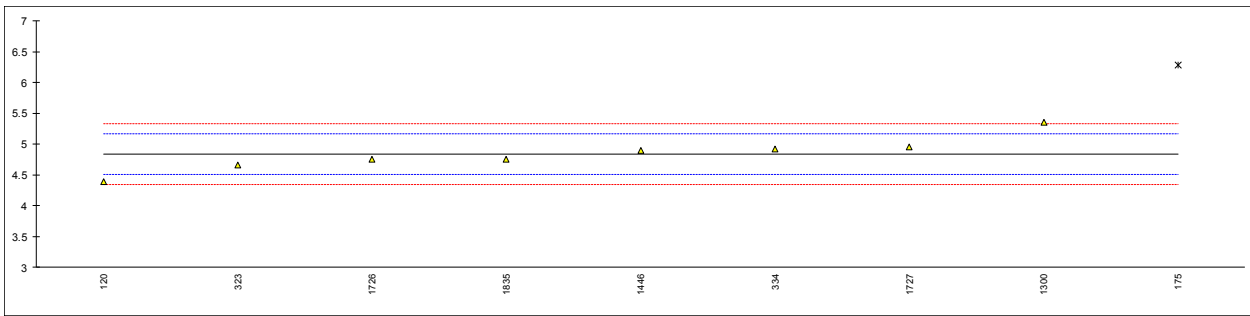
- Empty page -

Determination of Phosphorus as P on sample #18240; results in mg/L

lab	method	value	mark	z(targ)	remarks
52	D3231	<0.20		-----	
120		-----		-----	
150	D3231	<0.20		-----	
169		-----		-----	
171	EN15487	<0.15		-----	
174		-----		-----	
175		-----		-----	
194		-----		-----	
230		-----		-----	
311	EN15837	<0.13		-----	
323	EN15487	<0.15		-----	
329	EN15487	0.004		-----	
333	EN15487	<0.15		-----	
334		-----		-----	
337		-----		-----	
343		-----		-----	
357		-----		-----	
360	EN15837	< 0.10		-----	
391		-----		-----	
396		-----		-----	
444	EN15487	<0.15		-----	
496	EN15487	0.0007		-----	
541		-----		-----	
551	OGC2047	<0.13		-----	
554		-----		-----	
631		-----		-----	
633		-----		-----	
657		-----		-----	
663		-----		-----	
823		-----		-----	
840		-----		-----	
913		-----		-----	
922		-----		-----	
1040		-----		-----	
1201	EN15487	<0.01		-----	
1300	EN15487	0.01		-----	
1320		-----		-----	
1359		-----		-----	
1397		-----		-----	
1438		-----		-----	
1446		-----		-----	
1523		-----		-----	
1563		-----		-----	
1656	EN15487	<0.01		-----	
1710		-----		-----	
1712	EN15487	<0,15		-----	
1726	EN15487	0.068		-----	
1727	EN15487	<0,01		-----	
1817		-----		-----	
1835	EN15837	0.05		-----	
1852		-----		-----	
1919		-----		-----	
6072		-----		-----	
6214		0.011		-----	

normality	unknown	
n	17	
outliers	0	
mean (n)	<0.15	
st.dev. (n)	n.a.	
R(calc.)	n.a.	
st.dev.(EN15487:07)	n.a.	
R(EN15487:07)	n.a.	application range: 0.15 - 1.50 mg/L

## Determination of Water (coulometric) on sample #18240; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	E1064	0.306		-0.76	
120	E1064	0.316	C	0.38	reported 0.316 mg/kg
150	E1064	0.3190		0.72	
169	E1064	0.3143		0.18	
171	EN15489	0.302		-1.21	
174		-----		-----	
175		-----		-----	
194		-----		-----	
230		-----		-----	
311	EN15489	0.314		0.15	
323	EN15489	0.3136		0.11	
329	D6304	0.3145		0.21	
333	EN15489	0.327		1.63	
334	EN15489	0.314		0.15	
337		-----		-----	
343	EN15489	0.304		-0.98	
357	E1064	0.2889		-2.70	
360	EN15489	0.3196		0.79	
391		-----		-----	
396	E1064	0.299		-1.55	
444	EN15489	0.3136		0.11	
496	EN15489	0.348	R(0.05)	4.01	
541	E1064	0.3071		-0.63	
551	E1064	0.319		0.72	
554	E1064	0.320		0.83	
631		-----		-----	
633	D6304	0.361	R(0.01)	5.48	
657	E1064	0.3153		0.30	
663	E1064	0.3125		-0.02	
823	E1064	0.3111		-0.18	
840	E1064	0.2867		-2.95	
913		-----		-----	
922	D6304	0.30		-1.44	
1040	ISO12937	0.3358		2.62	
1201	EN15489	0.3126		-0.01	
1300	EN15489	0.3059		-0.77	
1320	EN15489	0.3054		-0.83	
1359	EN15489	0.314663		0.23	
1397		-----		-----	
1438		-----		-----	
1446		-----		-----	
1523		-----		-----	
1563		-----		-----	
1656	EN15489	0.3279		1.73	
1710	EN15489	0.256	R(0.01)	-6.43	
1712	EN15489	0.313		0.04	
1726	EN15489	0.313		0.04	
1727	EN15489	0.3258		1.49	
1817	In house	0.32		0.83	
1835	EN15489	0.3114		-0.14	
1852	ISO12937	0.3127		0.00	
1919	EN15489	0.32523		1.42	
6072	E1064	0.308		-0.53	
6214		0.313		0.04	

|		
normality	suspect	
n	38	
outliers	3	
mean (n)	0.31267	
st.dev. (n)	0.009792	
R(calc.)	0.02742	
st.dev.(EN15489:07)	0.00881	
R(EN15489:07)	0.02468	
Compare:		
R(E1064:16)	0.05347	
R(D6304:16e1)	0.21122	
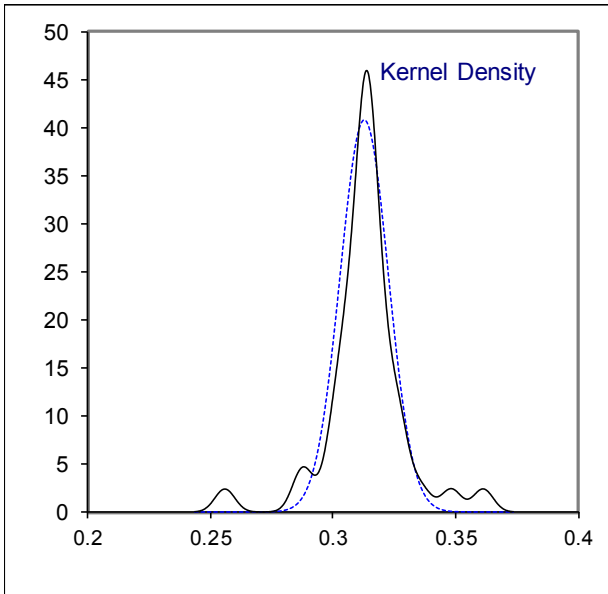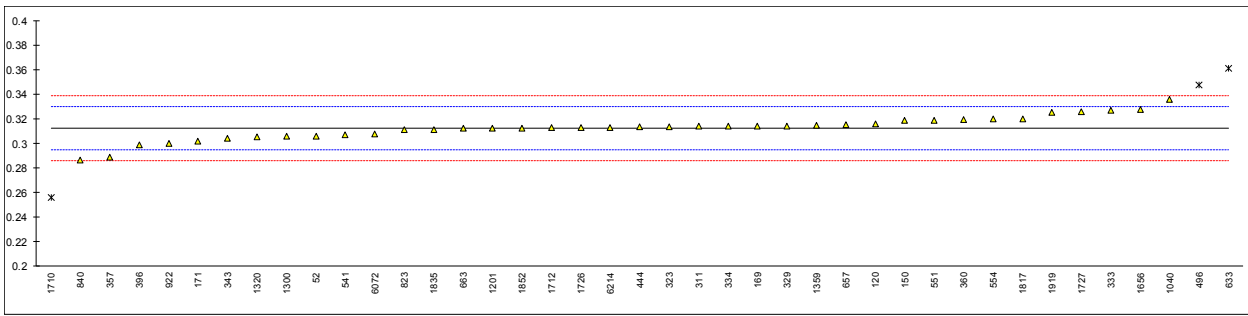
Determination of Water (titrimetric KF) on sample #18240; results in %M/M

lab	method	value	mark	z(targ)	remarks
52		-----		-----	
120		-----		-----	
150	E203	0.3171		0.34	
169	E203	0.3291		0.77	
171	E203	0.300		-0.28	
174	E203	0.292		-0.56	
175		-----		-----	
194	E203	0.3046		-0.11	
230		-----		-----	
311	EN15692	0.301		-0.24	
323	E203	0.312		0.15	
329	E203	0.3040		-0.13	
333		-----		-----	
334	E203	0.304		-0.13	
337		-----		-----	
343	E203	0.311		0.12	
357	E203	0.2998		-0.28	
360		-----		-----	
391		-----		-----	
396		-----		-----	
444	E203	0.3280		0.73	
496	E203	0.328		0.73	
541		-----		-----	
551	E203	0.302		-0.21	
554	E203	0.305		-0.10	
631	E203	0.310		0.08	
633		-----		-----	
657	E203	0.3142		0.23	
663	E203	0.3158		0.29	
823	E203	0.302		-0.21	
840	E203	0.2919		-0.57	
913	E203	0.27		-1.35	
922	E203	0.283		-0.89	
1040		-----		-----	
1201	E203	0.309		0.05	
1300		-----		-----	
1320	E203	0.3025		-0.19	
1359		-----		-----	
1397		-----		-----	
1438		-----		-----	
1446	ISO760	0.2959		-0.42	
1523	E203	0.34		1.16	
1563		-----		-----	
1656	E203	0.3257		0.65	
1710		-----		-----	
1712		-----		-----	
1726	EN15692	0.3095		0.06	
1727	EN15692	0.3167		0.32	
1817		-----		-----	
1835		-----		-----	
1852		-----		-----	
1919		-----		-----	
6072		-----		-----	
6214		-----		-----	

|		
normality	OK	
n	29	
outliers	0	
mean (n)	0.30772	
st.dev. (n)	0.014560	
R(calc.)	0.04077	
st.dev.(E203:16)	0.027857	
R(E203:16)	0.078	
Compare:		
R(EN15692:09)	0.09915	
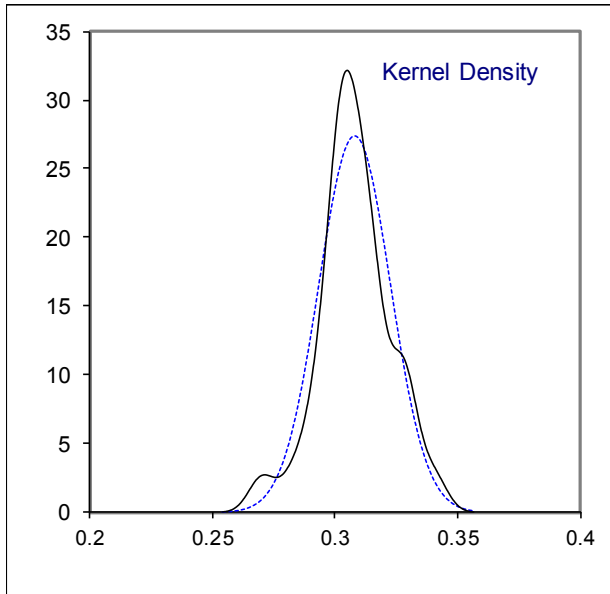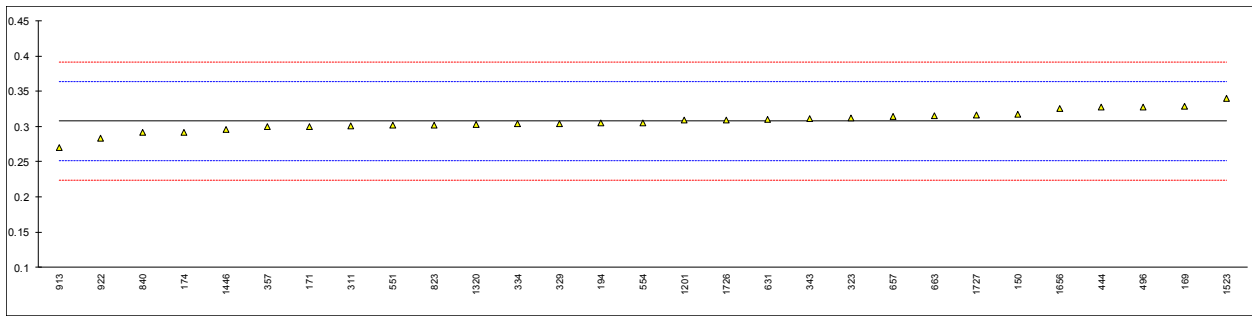
Determination of Ethanol incl. Higher Alcohols acc. to EN15721 on sample #18240 in %M/M

lab	method	value	mark	z(targ)	remarks
52		-----		-----	
120		-----		-----	
150		-----		-----	
169		-----		-----	
171		-----		-----	
174		-----		-----	
175		-----		-----	
194		-----		-----	
230		-----		-----	
311	EN15721	99.41		-1.86	
323	EN15721	99.467		-0.90	
329	EN15721	99.481		-0.67	
333	EN15721	99.84		5.36	
334	EN15721	99.590		1.16	
337		-----		-----	
343	EN15721	99.069		-7.59	
357	EN15721	99.436		-1.42	
360	EN15721	99.4375		-1.40	
391		-----		-----	
396		-----		-----	
444	EN15721	99.391		-2.18	
496	EN15721	99.3607		-2.69	
541		-----		-----	
551	INH-1313	99.40358		-1.97	
554		99.45		-1.19	
631		-----		-----	
633		-----		-----	
657	INH-2	99.6015		1.36	
663		-----		-----	
823		-----		-----	
840		-----		-----	
913		-----		-----	
922		-----		-----	
1040	EN15721	99.460		-1.02	
1201	EN15721	99.451		-1.17	
1300	EN15721	99.344		-2.97	
1320	EN15721	99.1306		-6.55	
1359	EN15721	99.72713		3.47	
1397		-----		-----	
1438		-----		-----	
1446	EN15721	99.94		7.04	
1523		-----		-----	
1563		-----		-----	
1656	EN15721	99.72		3.35	
1710	EN15721	99.733		3.56	
1712		-----		-----	
1726	EN15721	99.494		-0.45	
1727	EN15721	99.521		0.00	
1817		-----		-----	
1835	EN15721	99.5996		1.32	
1852	EN15721	99.5334		0.21	
1919	EN15721	99.90		6.37	
6072		-----		-----	
6214		99.57		0.83	
	normality	OK			
	n	27			
	outliers	0			
	mean (n)	99.52077			
	st.dev. (n)	0.201707			
	R(calc.)	0.56478			
	st.dev.(EN15721:13)	0.059543			
	R(EN15721:13)	0.16672			

Kernel Density
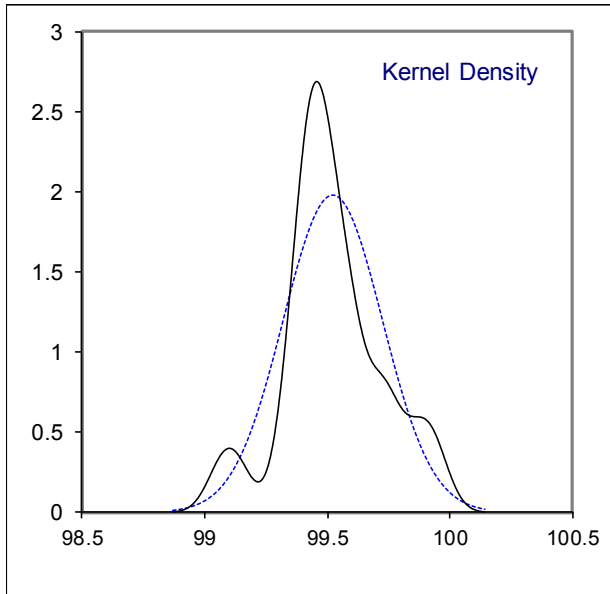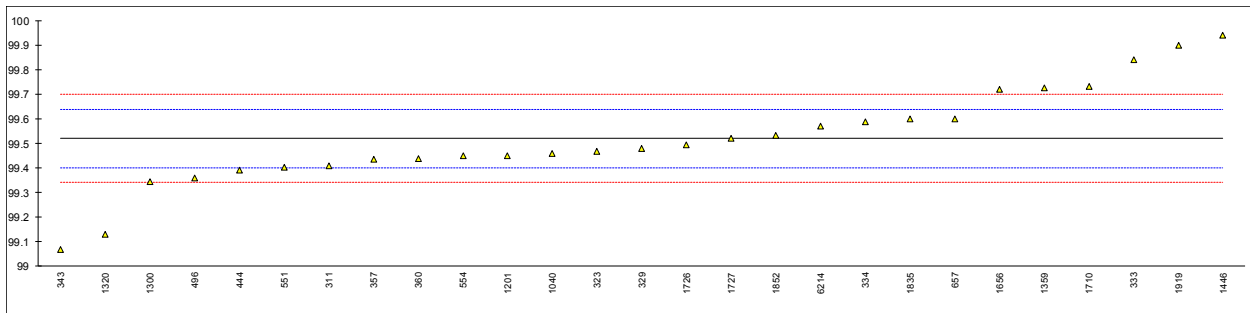
Determination of Higher Alcohols acc. to EN15721 on sample #18240; results in %M/M

lab	method	value	mark	z(targ)	remarks
52		-----		-----	
120		-----		-----	
150		-----		-----	
169		-----		-----	
171		-----		-----	
174		-----		-----	
175		-----		-----	
194		-----		-----	
230		-----		-----	
311	EN15721	0.196		-2.57	
323	EN15721	0.280		0.56	
329	EN15721	0.2695		0.17	
333	EN15721	0.245		-0.75	
334	EN15721	0.2513		-0.51	
337		-----		-----	
343		-----		-----	
357	EN15721	0.278		0.48	
360	EN15721	0.2822		0.64	
391		-----		-----	
396		-----		-----	
444	EN15721	0.267		0.07	
496	EN15721	0.2252		-1.48	
541		-----		-----	
551	OGC1313	0.256025		-0.33	
554		0.256		-0.34	
631		-----		-----	
633		-----		-----	
657	INH-2	0.2707		0.21	
663		-----		-----	
823		-----		-----	
840		-----		-----	
913		-----		-----	
922	INH-0001	0.2825		0.65	
1040	EN15721	0.2661		0.04	
1201	EN15721	0.231		-1.27	
1300	EN15721	0.2328	C	-1.20	first reported: 0.1682
1320	EN15721	0.2551		-0.37	
1359	EN15721	0.27676667		0.44	
1397		-----		-----	
1438		-----		-----	
1446	EN15721	0.2852		0.75	
1523		-----		-----	
1563		-----		-----	
1656	EN15721	0.32		2.05	
1710	EN15721	0.280		0.56	
1712		-----		-----	
1726	EN15721	0.2786		0.51	
1727	EN15721	0.2597		-0.20	
1817		-----		-----	
1835	EN15721	0.3059		1.52	
1852	EN15721	0.2486		-0.61	
1919	EN15721	0.3230		2.16	
6072		-----		-----	
6214		0.2335		-1.17	

	normality	OK	
	n	27	
	outliers	0	
	mean (n)	0.26503	
	st.dev. (n)	0.028248	
	R(calc.)	0.07909	
	st.dev.(EN15721:13)	0.026872	
	R(EN15721:13)	0.07524	

Kernel Density
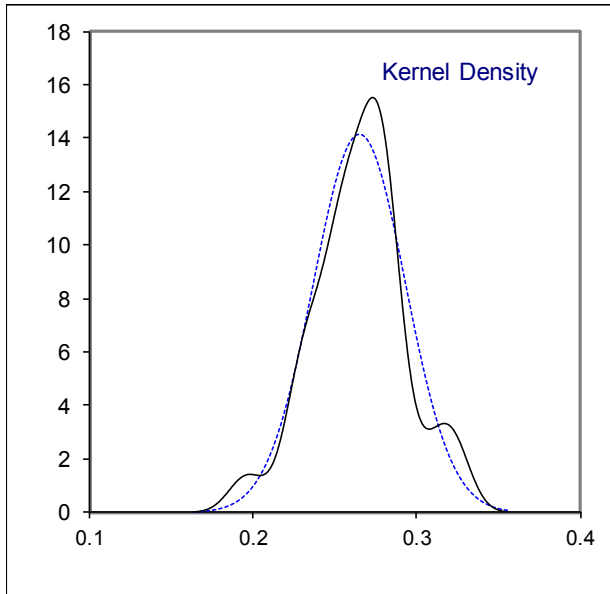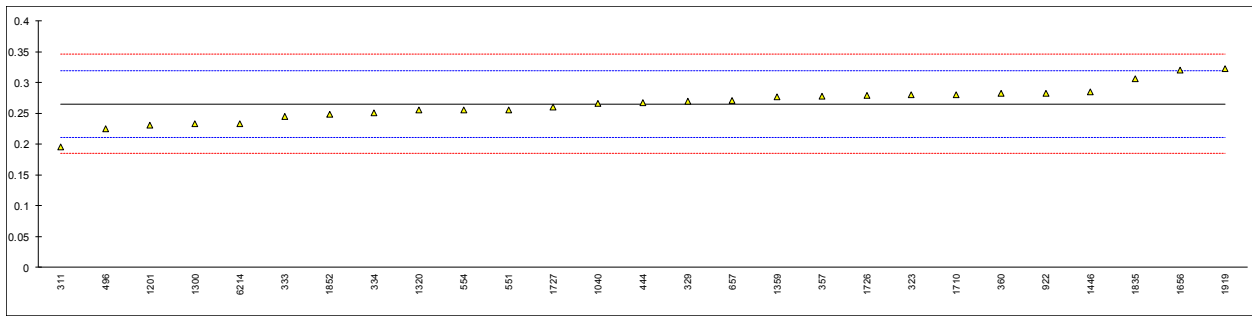
Determination of Impurities acc. to EN15721 on sample #18240; results in %M/M

lab	method	value	mark	z(targ)	remarks
52		-----		-----	
120		-----		-----	
150		-----		-----	
169		-----		-----	
171		-----		-----	
174		-----		-----	
175		-----		-----	
194		-----		-----	
230		-----		-----	
311	EN15721	0.540	ex	0.42	test result excluded: sum of components <100%
323	EN15721	0.518		0.10	
329	EN15721	0.505		-0.09	
333	EN15721	0.163	ex	-5.13	test result excluded: no numeric value for MeOH
334	EN15721	0.1686	ex	-5.05	test result excluded: sum of components <100%
337		-----		-----	
343		-----		-----	
357	EN15721	0.550		0.57	
360	EN15721	0.5499		0.57	
391		-----		-----	
396		-----		-----	
444	EN15721	0.595		1.24	
496	EN15721	0.6133	ex	1.51	test result excluded: outlier in MeOH
541		-----		-----	
551	OGC1313	0.580203		1.02	
554		0.60	ex	1.31	test result excluded: sum of components >100%
631		-----		-----	
633		-----		-----	
657	INH-2	0.3832		-1.89	
663		-----		-----	
823		-----		-----	
840		-----		-----	
913		-----		-----	
922		-----		-----	
1040	EN15721	0.1905	ex	-4.73	test result excluded: sum of components <100%
1201	EN15721	0.534		0.34	
1300	EN15721	0.6415		1.92	
1320	EN15721	0.6004	ex	1.32	test result excluded: sum of components <100%
1359	EN15721	0.179106667	ex	-4.89	test result excluded: sum of components <100%
1397		-----		-----	
1438		-----		-----	
1446		-----		-----	
1523		-----		-----	
1563		-----		-----	
1656	EN15721	0.20	ex	-4.59	test result excluded: no numeric value for MeOH
1710	EN15721	0.231	ex	-4.13	test result excluded: no numeric value for MeOH
1712		-----		-----	
1726	EN15721	0.4911		-0.30	
1727	EN15721	0.462		-0.72	
1817		-----		-----	
1835	EN15721	0.3854		-1.85	
1852	EN15721	0.4498		-0.90	
1919	EN15721	0.0893	G(0.01)	-6.22	
6072		-----		-----	
6214		0.4027	ex	-1.60	test result excluded: outlier in MeOH

normality	OK		
n	13		
outliers	1 +11ex		
mean (n)	0.51116		
st.dev. (n)	0.077014		
R(calc.)	0.21564		
st.dev.(Horwitz (n=9))	0.067859		
R(Horwitz (n=9))	0.19000		
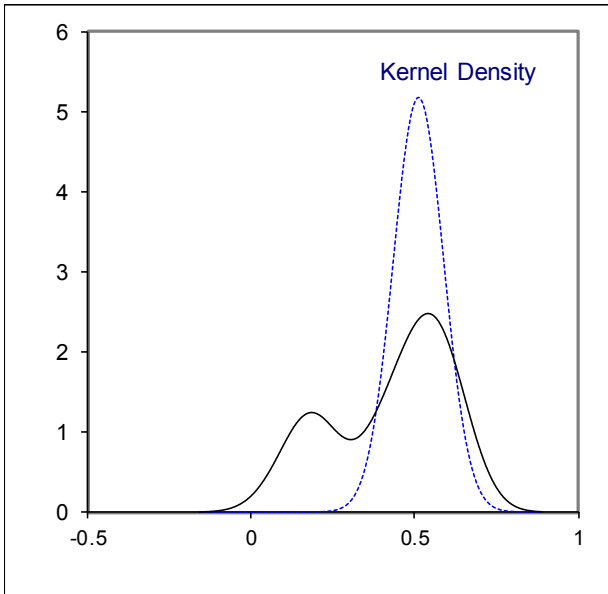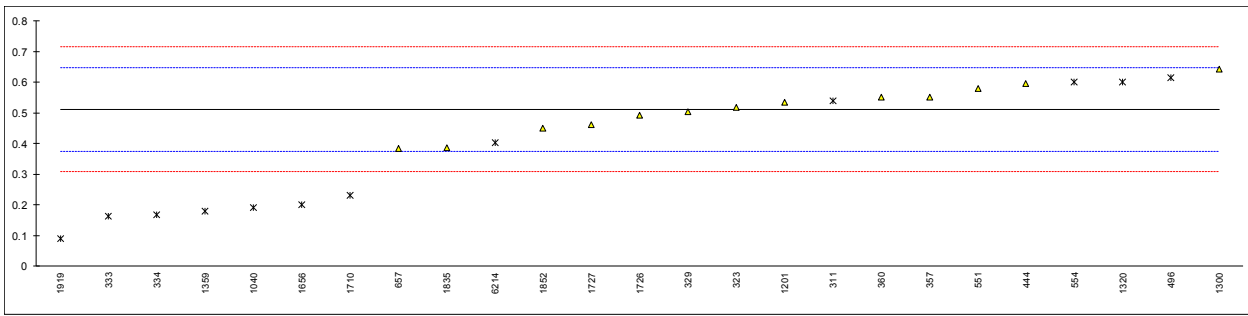
Determination of Methanol on sample #18240; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	D5501	0.02		4.29	
120	D5501	0.050033	R(0.01)	30.71	
150		-----		-----	
169		-----		-----	
171		-----		-----	
174	D5501	0.02		4.29	
175		-----		-----	
194		-----		-----	
230		-----		-----	
311	EN15721	0.013		-1.87	
323	EN15721	0.017		1.65	
329	EN15721	0.0138	C	-1.16	first reported 0.138
333	EN15721	<0.100		-----	
334	EN15721	0.0149		-0.19	
337		-----		-----	
343	EN15721	0.013		-1.87	
357	EN15721	0.014		-0.99	
360	EN15721	0.0126		-2.22	
391		-----		-----	
396		-----		-----	
444	EN15721	0.014		-0.99	
496	EN15721	0.0260	R(0.01)	9.57	
541		-----		-----	
551	OGC1313	0.01622		0.97	
554		0.0157		0.51	
631	D5501	0.02	C	4.29	first reported 0.038
633		-----		-----	
657	INH-2	0.01532		0.18	
663	INH-0002	0.014		-0.99	
823	D5501	0.0121	C	-2.66	first reported 0.052
840		-----		-----	
913		-----		-----	
922	INH-0001	0.0232	R(0.05)	7.11	
1040	EN15721	0.0143		-0.72	
1201	EN15721	0.0149		-0.19	
1300	EN15721	0.0146		-0.46	
1320	EN15721	0.0140		-0.99	
1359	EN15721	0.015616667		0.44	
1397		-----		-----	
1438		-----		-----	
1446	EN15721	0.0149		-0.19	
1523	D5501	0.01363		-1.31	
1563		-----		-----	
1656	EN15721	<0.01	C	-----	first reported 0.06
1710		-----	W	-----	first reported 0.036
1712		-----		-----	
1726	EN15721	0.0150		-0.11	
1727	EN15721	0.015		-0.11	
1817		-----		-----	
1835	EN15721	0.0150		-0.11	
1852	EN15721	0.0168		1.48	
1919	EN15721	0.0140		-0.99	
6072		-----	W	-----	first reported 0.0494
6214		0.0276	R(0.01)	10.98	

normality	not OK			
n	28			
outliers	4			
mean (n)	0.01512			
st.dev. (n)	0.002068			
R(calc.)	0.00579			
st.dev.(Horwitz)	0.001137			
R(Horwitz)	0.00318			
Compare:				
R(D5501:12)	0.014365		application range: 0.01 - 0.6 %M/M	
R(EN15721:13)	-0.00124		application range: 0.1 - 3 %M/M	
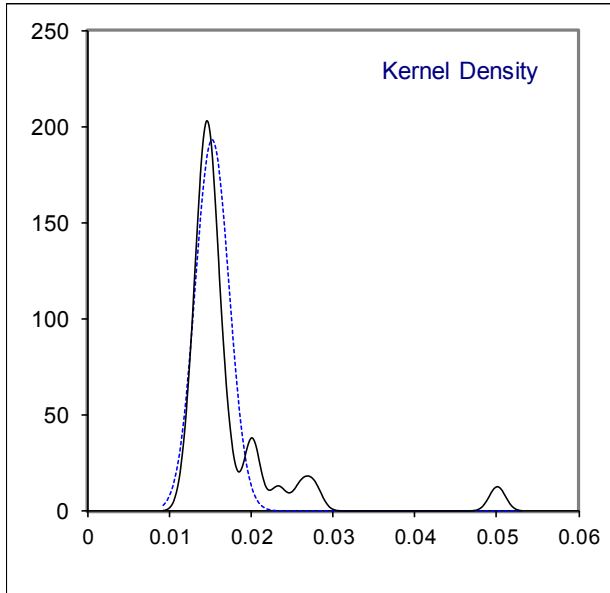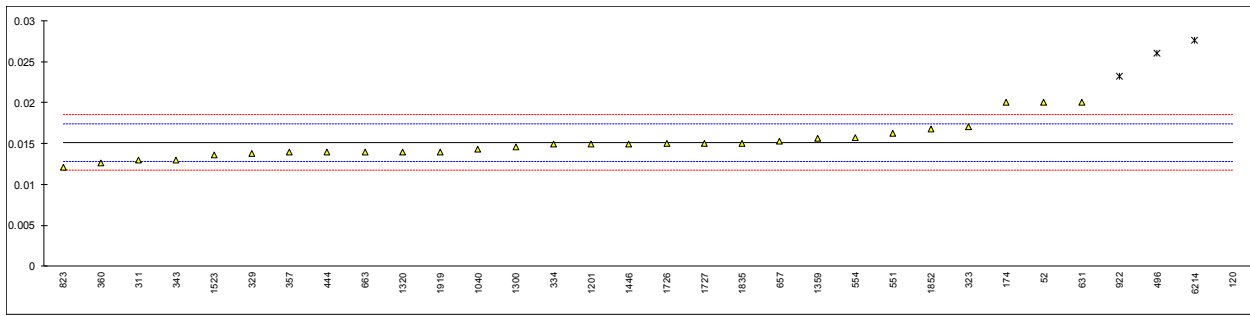
Kernel Density

Determination of Ethanol acc. to ASTM D5501 on sample #18240; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	D5501	99.18		-0.19	
120	D5501	99.467573	ex	0.62	test result excluded as Ethanol %M/M is > Ethanol %V/V
150	D5501	99.10		-0.41	
169		-----		-----	
171		-----		-----	
174	D5501	99.53	ex	0.79	test result excluded as Ethanol %M/M is > Ethanol %V/V
175		-----		-----	
194		-----		-----	
230		-----		-----	
311	D5501	99.18		-0.19	
323		-----		-----	
329		-----		-----	
333		-----		-----	
334		-----		-----	
337		-----		-----	
343		-----		-----	
357		-----		-----	
360		-----		-----	
391		-----		-----	
396		-----		-----	
444		-----		-----	
496		-----		-----	
541		-----		-----	
551	D5501	98.70		-1.54	
554	D5501	99.67		1.19	
631	D5501	99.638		1.10	
633		-----		-----	
657	D5501	99.2564		0.03	
663	D5501	99.190		-0.16	
823	D5501	99.2567		0.03	
840	D5501	99.183		-0.18	
913		-----		-----	
922		-----		-----	
1040		-----		-----	
1201		-----		-----	
1300	D5501	99.674		1.20	
1320		-----		-----	
1359	D5501	99.67		1.19	
1397		-----		-----	
1438	In house	99.1		-0.41	
1446		-----		-----	
1523		-----		-----	
1563		-----		-----	
1656		-----		-----	
1710		-----		-----	
1712		-----		-----	
1726		-----		-----	
1727		-----		-----	
1817		-----		-----	
1835	D5501	99.253		0.02	
1852		-----		-----	
1919		-----		-----	
6072	D5501	98.6597		-1.65	
6214		-----		-----	

normality	OK	
n	15	
outliers	0 +2ex	
mean (n)	99.24739	
st.dev. (n)	0.315884	
R(calc.)	0.88448	
st.dev.(D5501:12)	0.355575	
R(D5501:12)	0.99561	

Kernel Density
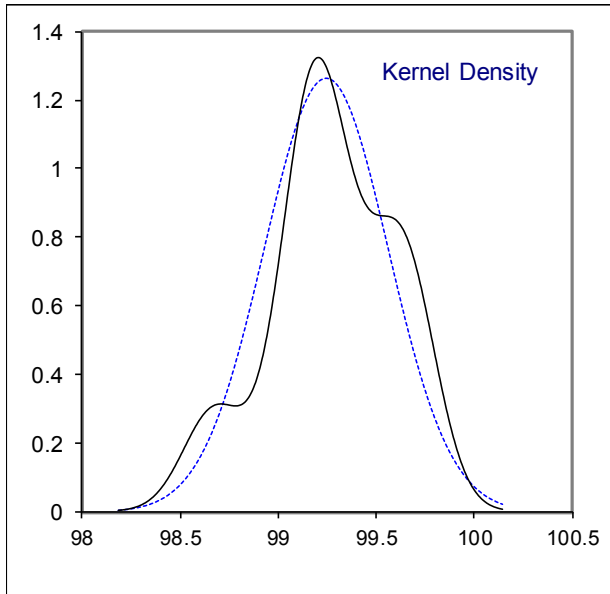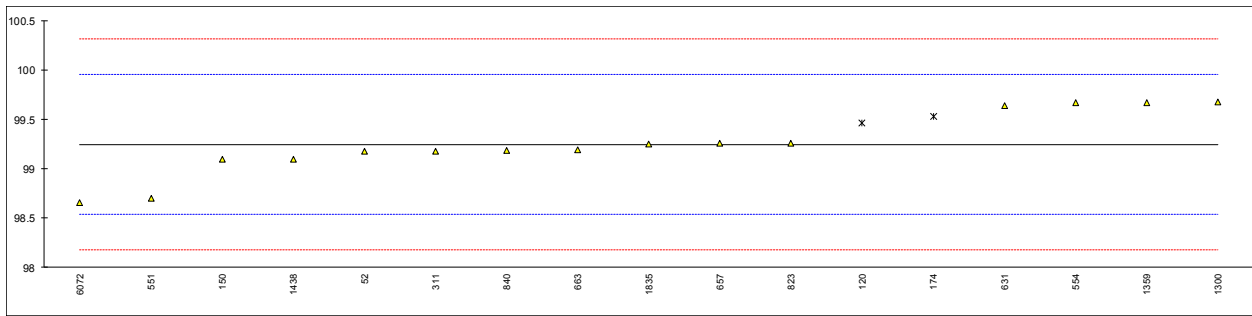
Determination of Ethanol acc. to ASTM D5501 on sample #18240; results in %V/V

lab	method	value	mark	z(targ)	remarks
52	D5501	99.30		-0.21	
120	D5501	99.24	ex	-0.38	test result excluded as Ethanol %M/M is > Ethanol %V/V
150	D5501	99.21		-0.47	
169		-----		-----	
171		-----		-----	
174	D5501	99.34	ex	-0.10	test result excluded as Ethanol %M/M is > Ethanol %V/V
175		-----		-----	
194		-----		-----	
230		-----		-----	
311	D5501	99.34		-0.10	
323		-----		-----	
329		-----		-----	
333		-----		-----	
334		-----		-----	
337		-----		-----	
343		-----		-----	
357		-----		-----	
360		-----		-----	
391		-----		-----	
396		-----		-----	
444		-----		-----	
496		-----		-----	
541		-----		-----	
551	D5501	98.81		-1.59	
554	D5501	99.77		1.11	
631	D5501	99.715		0.96	
633		-----		-----	
657	D5501	99.4200		0.13	
663	D5501	99.354		-0.06	
823	D5501	99.2687		-0.30	
840	D5501	99.321		-0.15	
913		-----		-----	
922		-----		-----	
1040		-----		-----	
1201		-----		-----	
1300	D5501	99.834		1.29	
1320		-----		-----	
1359	D5501	99.80		1.19	
1397		-----		-----	
1438		-----		-----	
1446		-----		-----	
1523		-----		-----	
1563		-----		-----	
1656		-----		-----	
1710		-----		-----	
1712		-----		-----	
1726		-----		-----	
1727		-----		-----	
1817		-----		-----	
1835	D5501	99.384		0.02	
1852		-----		-----	
1919		-----		-----	
6072	D5501	98.7306		-1.82	
6214		-----		-----	

normality	OK	
n	14	
outliers	0 +2ex	
mean (n)	99.37553	
st.dev. (n)	0.333771	
R(calc.)	0.93456	
st.dev.(D5501:12)	0.355300	
R(D5501:12)	0.99484	
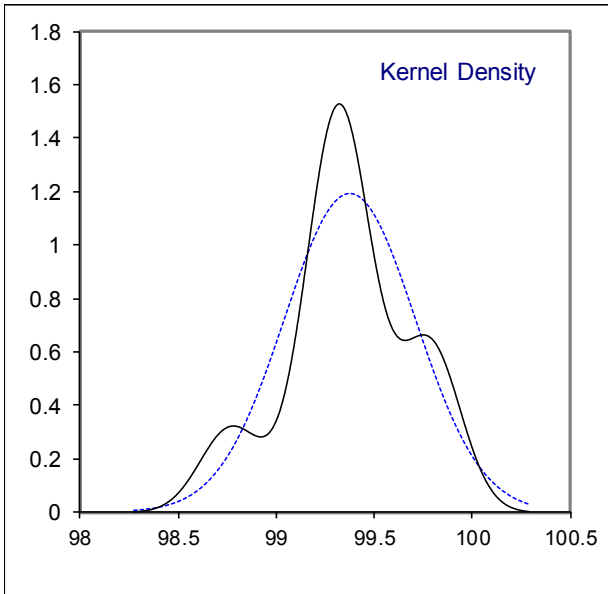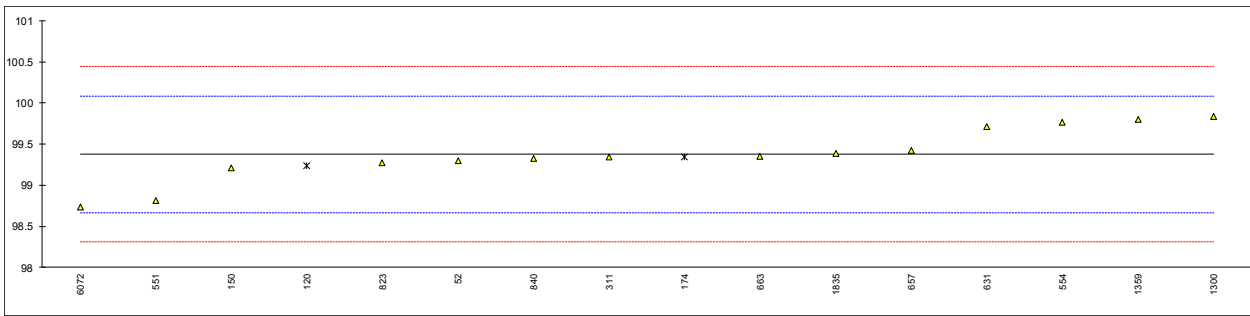
Determination of Chlorides, Inorganic as Cl on sample #18241; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52		-----		-----	
120	D7319	5.522		-0.04	
150	D7328	6.50		1.38	
169	D7319	5.90749		0.52	
171	EN15492	6.5		1.38	
174	D7319	6.6		1.53	
175	D7319	4.24		-1.91	
194		-----		-----	
230	INH-23	<1	C	<-6.63	first reported <2; possibly a false negative test result?
311	EN15492	4.8		-1.09	
323	EN15492	6.0		0.66	
329	EN15492	4.6	ex	-1.39	reported 4,6 mg/l
333	EN15492	5.6		0.07	
334		-----		-----	
337		-----		-----	
343	EN15492	4.4		-1.68	
357		-----		-----	
360	EN15492	5.73		0.26	
391		-----		-----	
396		-----		-----	
444		-----		-----	
496	EN15492	6.46		1.33	
541	D7328	6.223		0.98	
551	D7319	4.93		-0.90	
554		-----		-----	
631		-----		-----	
633		-----		-----	
657	D7328	5.967		0.61	
663		-----		-----	
823		-----		-----	
840	D7319	6.71		1.69	
913		-----		-----	
922		-----		-----	
1040		-----		-----	
1201	EN15492	4.6	C	-1.39	first reported 2.6
1300	EN15492	6.25		1.02	
1320	EN15492	3.76		-2.61	
1359	EN15492	5.255		-0.43	
1397	EN15492	5.0		-0.80	
1438		-----		-----	
1446		-----		-----	
1523		-----		-----	
1563		-----		-----	
1656		-----		-----	
1710		-----		-----	
1712	EN15484	<5,1		-----	
1726		-----		-----	
1727	EN15492	6.2		0.95	
1817		-----		-----	
1835	EN15492	6.07		0.76	
1852		-----		-----	
1919		-----		-----	
6072		-----		-----	
6214		3.986		-2.28	

					EN15492 only
	normality	OK			OK
	n	24			14
	outliers	0 +1ex	spike:		0 +1ex
	mean (n)	5.550	6.2 mg/kg recovery <90%		5.473
	st.dev. (n)	0.8892			0.8526
	R(calc.)	2.490			2.387
	st.dev.(Horwitz)	0.6861			0.6780
	R(Horwitz)	1.921			
	Compare:				
	R(EN15492:12)	0.916	application range: 1 - 30 mg/kg		0.910
	R(D7319:17)	1.385	application range: 0.75 - 50 mg/kg		

Kernel Density
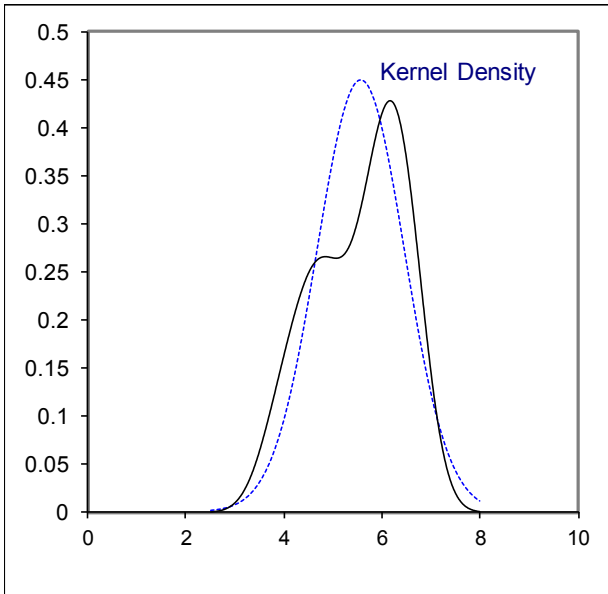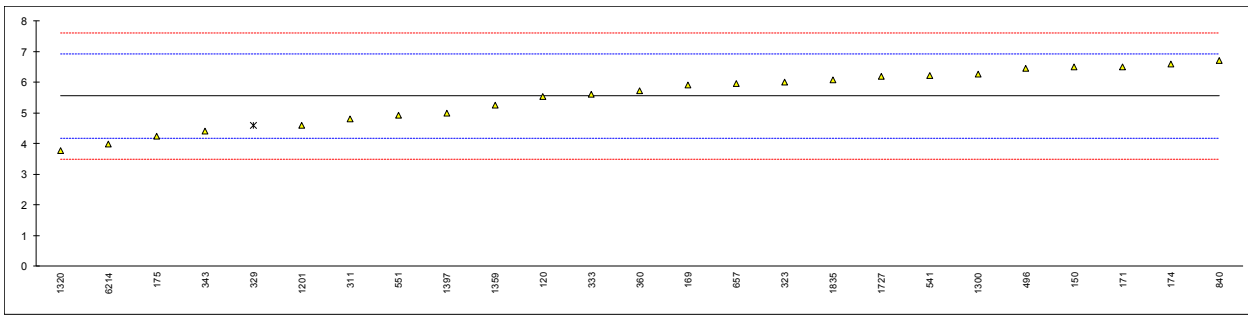
Determination of Sulfate as SO_4 on sample #18241; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52		-----		-----	
120	D7319	3.182		-0.76	
150	D7328	5.21		0.81	
169	D7319	8.18756		3.12	
171	EN15492	8.7		3.51	
174	D7319	2.1		-1.59	
175		-----		-----	
194		-----		-----	
230		-----		-----	
311	EN15492	4.7		0.42	
323	EN15492	6.2		1.58	
329	EN15492	4.1	ex	-0.05	reported 4,1 mg/l
333	EN15492	4.2		0.03	
334		-----		-----	
337		-----		-----	
343	EN15492	5.3		0.88	
357		-----		-----	
360	EN15492	2.47		-1.31	
391		-----		-----	
396		-----		-----	
444		-----		-----	
496	EN15492	5.07		0.70	
541	D7328	4.055		-0.08	
551	D7319	0.63		-2.73	
554		-----		-----	
631		-----		-----	
633		-----		-----	
657	D7328	6.212		1.59	
663		-----		-----	
823		-----		-----	
840	D7319	3.76		-0.31	
913		-----		-----	
922		-----		-----	
1040		-----		-----	
1201	EN15492	2.6		-1.21	
1300	EN15492	2.479		-1.30	
1320	EN15492	3.78		-0.29	
1359	EN15492	4.7009		0.42	
1397	EN15492	3.5		-0.51	
1438		-----		-----	
1446		-----		-----	
1523		-----		-----	
1563		-----		-----	
1656		-----		-----	
1710		-----		-----	
1712		-----		-----	
1726		-----		-----	
1727	EN15492	1.5		-2.06	
1817		-----		-----	
1835	EN15492	4.61		0.35	
1852		-----		-----	
1919		-----		-----	
6072		-----		-----	
6214		2.5375		-1.26	

normality	OK			
n	23			
outliers	0 +1ex		spike:	
mean (n)	4.160		4.3 mg/kg recovery <97%	
st.dev. (n)	1.9709			
R(calc.)	5.519			
st.dev.(D7319:17)	1.2928			
R(D7319:17)	3.620		application range: 1 - 20 mg/kg	
Compare:				
R(EN15492:12)	1.014		application range: 1 - 20 mg/kg	
R(D7328:16)	1.858		application range: 0.55 - 20 mg/kg	

Kernel Density
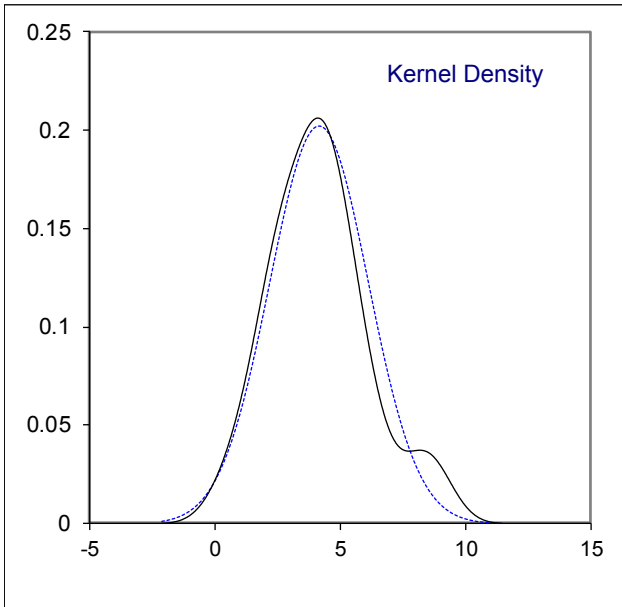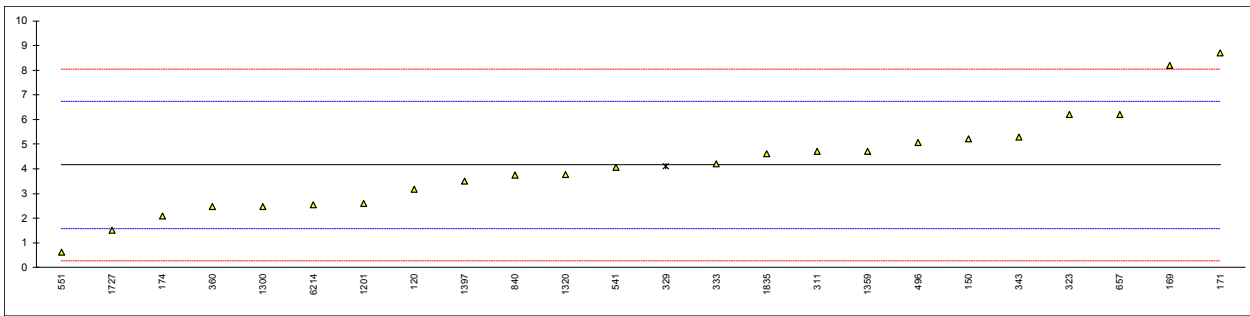
Determination of Sulfur on sample #18241; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	D5453	3.7		0.34	
120	D5453	3.652		0.30	
150	D5453	2.6		-0.54	
169	D5453	2.88		-0.32	
171	EN15485	4.82		1.24	
174		-----		-----	
175	D5453	2.62		-0.53	
194		-----		-----	
230		-----		-----	
311	EN15486	<5.0		-----	
323	D5453	3		-0.22	
329	D5453	2.3		-0.78	
333	EN15485	3.9		0.50	
334	EN15485	3.0		-0.22	
337	ISO20846	3.9		0.50	
343		-----		-----	
357	D5453	2.49		-0.63	
360	EN15486	3.1		-0.14	
391		-----		-----	
396		-----		-----	
444	D5453	3.64		0.29	
496		-----		-----	
541	D5453	2.13		-0.92	
551	D5453	2.775		-0.40	
554		-----		-----	
631		-----		-----	
633		-----		-----	
657	D5453	2.695		-0.47	
663	D5453	4.59		1.05	
823	D5453	1.91		-1.10	
840	D5453	4.35		0.86	
913		-----		-----	
922	D5453	3.885		0.49	
1040	ISO20846	3.89		0.49	
1201	EN15486	0.77		-2.01	
1300	EN15485	4.516		1.00	
1320		-----		-----	
1359	In house	4.547		1.02	
1397	D5453	4.5		0.98	
1438		-----		-----	
1446		-----		-----	
1523		-----		-----	
1563		-----		-----	
1656	EN15486	3.8		0.42	
1710	ISO20846	2.7		-0.46	
1712	EN15486	2.17		-0.89	
1726		-----		-----	
1727		-----		-----	
1817		-----		-----	
1835	EN15486	3.5		0.18	
1852	ISO20846	3.22		-0.04	
1919		-----		-----	
6072		-----		-----	
6214		-----		-----	
	normality	OK			
	n	31			
	outliers	0			
	mean (n)	3.276			
	st.dev. (n)	0.9421			
	R(calc.)	2.638			
	st.dev.(EN15485:07)	1.2462			
	R(EN15485:07)	3.489			application range: 7 - 20 mg/kg
	Compare:				
	R(EN15486:07)	2.194			application range: 5 - 20 mg/kg
	R(D5453:16e1)	1.412			at a concentration < 400 mg/kg; application range: 1 - 8000 mg/kg

Kernel Density
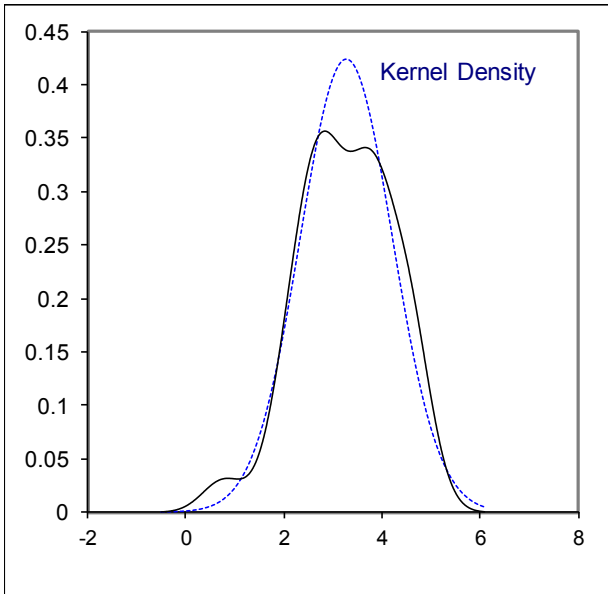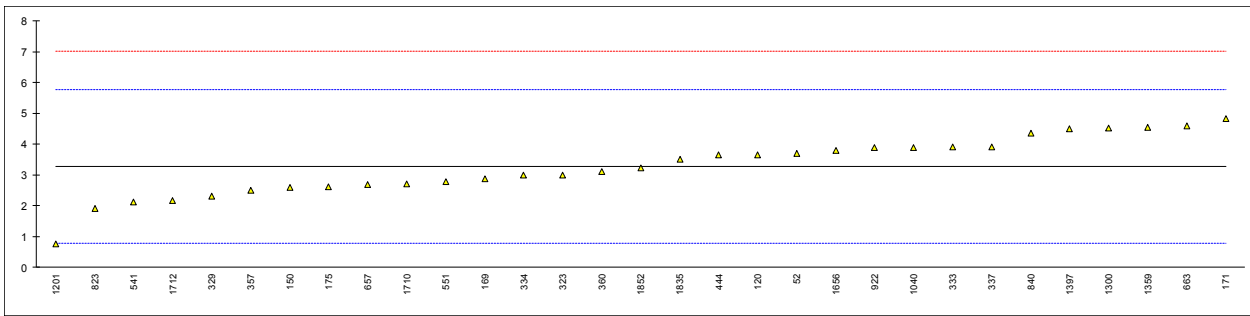
APPENDIX 2

Number of participating laboratories per country:

1 lab in   ARGENTINA
3 labs in BELGIUM
2 labs in BRAZIL
1 lab in   BULGARIA
1 lab in   CANADA
1 lab in   COLOMBIA
1 lab in   CROATIA
1 lab in   CZECH REPUBLIC
1 lab in   ESTONIA
1 lab in   FINLAND
3 labs in FRANCE
3 labs in GERMANY
2 labs in HUNGARY
1 lab in   INDIA
1 lab in   ISRAEL
2 labs in ITALY
1 lab in   MAURITIUS
3 labs in NETHERLANDS
1 lab in   PAKISTAN
2 labs in PHILIPPINES
1 lab in   POLAND
1 lab in   SINGAPORE
1 lab in   SLOVAKIA
1 lab in   SOUTH KOREA
4 labs in SPAIN
1 lab in   SWEDEN
2 labs in THAILAND
3 labs in UNITED KINGDOM
7 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			= possibly an error in calculations
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, June 2018
2	W. Horwitz and R. Albert, J. AOAC Int., 79, 3, 589, (1996)
3	ASTM E178:02
4	ASTM E1301:95(2003)
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, Analyst, 2002, 127, 1359-1364 (2002).
16	Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)
17	M.A. Gonçalves et.al., Sensors and Actuators, B158, 327-332 (2011)