

**Results of Proficiency Test
Ethanol (REN & Food)
November 2015**

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

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1 INTRODUCTION

Since 2007, a proficiency test for REN/Food grade Ethanol is organised every year by the Institute for Interlaboratory Studies. During the planning of the annual proficiency testing program 2015/2016, it was decided to continue the round robin for the analysis of REN/Food grade Ethanol.

In this interlaboratory study, 32 laboratories in 16 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the 2015 proficiency test are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET-UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. Analysis for fit-for-use and homogeneity testing were subcontracted to an accredited laboratory. It was decided to send one sample (1* 0.5 L of REN/Food grade Ethanol, labelled #15232). Participants were requested to report rounded and unrounded results. The unrounded 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 17043:10, (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. Also customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

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 April 2014 (iis-protocol, version 3.3). The protocol can be downloaded from 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 #15232 was obtained from a local trader. The approximately 50 litre bulk sample was homogenised in a pre-cleaned drum and 50 amber glass bottles of 0.5 L were filled and labelled #15232. The homogeneity of these subsamples was checked by determination of Density in accordance with ASTM D4052 and Water in accordance with ASTM D1364 on 8 stratified randomly selected samples.

Sample	Density at 20°C in kg/L	Water in %M/M
Sample #15232-1	0.80597	5.671
Sample #15232-2	0.80596	5.680
Sample #15232-3	0.80597	5.666
Sample #15232-4	0.80597	5.651
Sample #15232-5	0.80597	5.655
Sample #15232-6	0.80598	5.681
Sample #15232-7	0.80596	5.644
Sample #15232-8	0.80597	5.654

Table 1: Homogeneity test results of subsamples #15232

From the test results of table 1, the repeatabilities were calculated and compared with 0.3 times the corresponding target reproducibility 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	0.039
reference method	ISO12185:96	ASTM D1364:02(2012)
0.3 * R (reference method)	0.00015	0.043

Table 2: Repeatabilities of subsamples #15232

The calculated repeatabilities were less than 0.3 times the corresponding reproducibilities of the reference test methods. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories 1*0.5 L bottle of sample #15232 was sent on November 11, 2015.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYSES

The participants were asked to determine on sample #15232: Density at 20°C, Non-volatile matter, Permanganate Time Test at 20°C, pHe, Purity Ethanol on dry basis, Strength (in %M/M and %V/V), Water (by KF) and UV Absorbance at 300, 270, 260, 250, 240, 230 and 220nm with an evaluation of the UV-scan.

To get comparable results a detailed report form, on which the units were prescribed as well as the required standards and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/.

A SDS and a form to confirm receipt of the samples were added to the samples.

3 RESULTS

During four weeks after sample dispatch, the results of the individual laboratories were received via the data entry portal www.kpmd.co.uk/sgs-iis/. The original reported 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 results at that moment.

Shortly after the deadline, the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1. 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 April 2014 (iis-protocol, version 3.3).

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

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test 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. Not all data sets proved to have a normal

distribution, in which cases the statistical evaluation of the results should be used with due care.

According to ISO 5725 the original 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. 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 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 analysis 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 standard. 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 spread of 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 of a former iis proficiency test 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, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

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

The z(target) scores are listed 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 with the dispatch of the samples. Six participants reported results after the final reporting date. In total 32 laboratories reported 254 numerical results. Observed were 11 outlying results, which is 4.3%. 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 methods, which were used by the various laboratories, are 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.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D1363) and an added designation for the year that the method was adopted or revised (e.g. D1363:06). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D1363:06(2011)). In the results tables of Appendix 1 only the method number and year of adoption or revision e.g. D1363:06 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, see also paragraph 3.1.

- Density: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ISO12185:96.
- Nonvolatile matter: The determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D1353:13.
- Permanganate Time Test: Sample #15232 contained impurities that reduced the Permanganate solution rather quick (For 'on spec' ethanol the Permanganate Time Test will give a test result > 30 minutes). All participants agreed on the time equal or less than 5 minutes, except one participant (reported <50min). Due to this low value for the Permanganate Time Test the precision requirements of ASTM D1363:06(2011) became unrealistic small. Therefore it was decided not to calculate z-scores for this PT.
- pHe: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers was not in agreement with the requirements of ASTM D6423:14.
It is known (see ref. 16) that in general in a pHe determination with a LiCl electrode the observed values are lower than when other types of electrodes are used. In last year PT (iis14C11) a bimodal distribution was observed. Therefore, in this PT the type of electrode used in the determination was reported by the participants. Unfortunately, the number of observations is too small to be able to underpin this phenomenon.
- Purity on dry basis: Regretfully, no Standard Method does exist that gives a clear definition of purity in Ethanol REN/Food grade. Six of the 13 participants reported to have used method EN15721. This method describes the purity in Ethanol based on the content of Methanol and higher Alcohols as determined by GC. This means that other components (for example Aldehydes, Ketones or other impurities like Aromatics) are not taken into account.
Therefore no significant conclusions could be drawn. No statistical outliers were observed. The calculated reproducibility is large in comparison to the calculated reproducibility of the previous proficiency test iis14C11. When the EN15721 test results were evaluated separately, the reproducibility is not in agreement with the requirements of EN15721:13.

- Strength (%M/M): This determination may not be problematic. Two statistical outliers were observed. Regretfully, no standard test method with precision data exists. The calculated reproducibility after rejection of the statistical outliers is small in comparison to the calculated reproducibility in the previous proficiency test iis14C11.
- Strength (%V/V): This determination may not be problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the reproducibility derived from the OIML table and ISO12185:96.
- Water: 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 ASTM D1364:02(2012).
- UV absorbance: Regretfully, no Standard Test Method for this determination exists. Some participants reported results obtained with a 50 mm cuvette, others with a 10mm cuvette. For the PT evaluation, the results at 300 nm were converted to 10 mm cuvette by dividing the original values of 50 mm by 5 according to the Law of Lambert-Beer. The calculated reproducibility at 300nm is in line with the calculated reproducibility ($R(\text{calc})=0.0052$ at mean Absorbance=0.0190) observed in the previous proficiency test iis14C11. This conversion from 50 to 10 mm cuvette could not be done for measurements at 270 till 220 nm as the linearity is not longer valid at Absorbencies > 0.8. Therefore the reported results from 50 mm cuvettes are excluded for the evaluation at 270 nm. From 260 till 220 nm no difference exists any longer between 10 and 50 mm as the Absorbencies are >3. Therefore the results of the statistical evaluations in appendix 1 are listed between brackets. Sample #15232 was clearly contaminated as shown by the high absorbencies. Remarkable, two participants reported "Pass" as conclusion of the UV absorbance determination.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories. The target reproducibilities derived from literature standards (in casu ASTM, EN standards) or previous proficiency tests are compared in the next table.

Parameter	unit	n	average	2.8 *sd	R (lit)
Density at 20°C	kg/L	31	0.8060	0.0002	0.0005
Nonvolatile matter	mg/100mL	12	0.6	1.1	2.1
Permanganate Time Test	min.	18	≤ 5	(3.5)	n.a.
pHe		12	8.1	1.5	1.0
Purity EtOH on dry basis	%M/M	13	99.92	0.13	(0.02)
Strength	%M/M	19	94.34	0.04	(0.05)
Strength	%V/V	27	96.35	0.03	0.06
Water (titrimetric)	%M/M	19	5.60	0.20	0.14
UV-absorbance 300 nm		14	0.0181	0.0067	(0.0052)
UV-absorbance 270 nm		11	1.54	0.08	n.a.
UV-absorbance 260 nm		14	(3.5)	(1.0)	n.a.
UV-absorbance 250 nm		14	(3.6)	(0.8)	n.a.
UV-absorbance 240 nm		14	(3.6)	(0.9)	n.a.
UV-absorbance 230 nm		14	(3.7)	(0.8)	n.a.
UV-absorbance 220 nm		12	(3.8)	(0.5)	n.a.
Conclusion UV-scan	Pass/Fail	12	Fail	n.a.	n.a.

Table 3: Reproducibilities of sample #15232

Results between brackets under R (lit) column are compared with the observed reproducibility of the previous proficiency test iis14C11.

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2015 WITH PREVIOUS PTS

	<i>November 2015</i>	<i>November 2014</i>	<i>November 2013</i>	<i>November 2012</i>	<i>November 2011</i>
Number of reporting labs	32	25	24	24	23
Number of results reported	254	210	160	169	151
Number of statistical outliers	11	13	9	5	9
Percentage outliers	4.3%	6.2%	5.6%	3.0%	6.0%

Table 4: Comparison with previous proficiency tests

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

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

Parameter	November 2015	November 2014	November 2013	November 2012	November 2011
Density at 20°C	++	++	++	++	++
Nonvolatile matter	++	++	n.e.	++	n.e.
Permanganate Time Test	(+)	-	+	--	(--)
pHe	(-)	--	n.e.	n.e.	n.e.
Purity EtOH on dry basis	(--)	(-)	(-)	(+)	(+)
Strength %M/M	(+/-)	(+)	(+)	(--)	++
Strength %V/V	++	+	+	--	++
Water (titrimetric)	-	-	--	--	+/-
UV-absorbance 300 nm	(+/-)	(-)	(++)	(-)	(--)
UV-absorbance 270 nm	n.e.	(++)	(+/-)	(--)	(-)
UV-absorbance 260 nm	n.e.	n.e.	n.e.	n.e.	n.e.
UV-absorbance 250 nm	n.e.	n.e.	n.e.	n.e.	n.e.
UV-absorbance 240 nm	n.e.	(-)	(++)	(+/-)	(-)
UV-absorbance 230 nm	n.e.	(+)	(-)	(++)	(-)
UV-absorbance 220 nm	n.e.	(++)	(--)	(--)	(-)

Table 5: Comparison determinations of sample #15232 against the standard
Results between brackets are compared with the observed reproducibility of the previous proficiency test

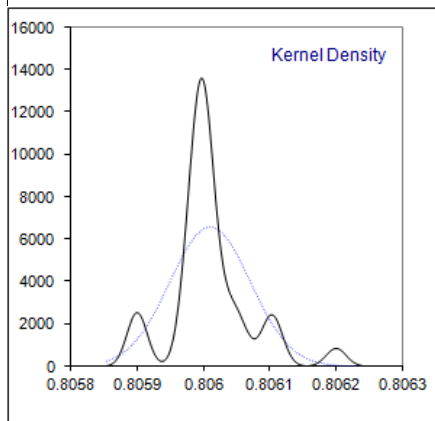
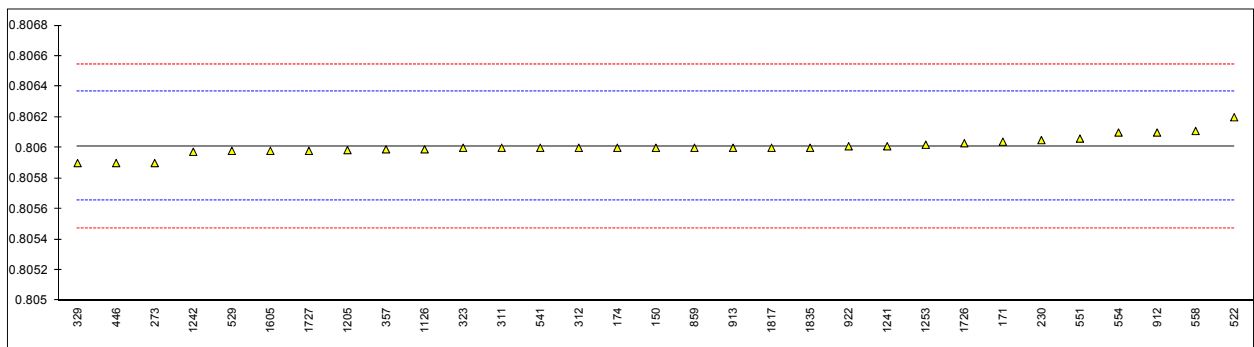
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

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

APPENDIX 1

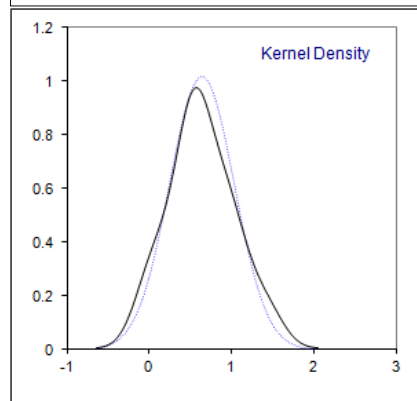
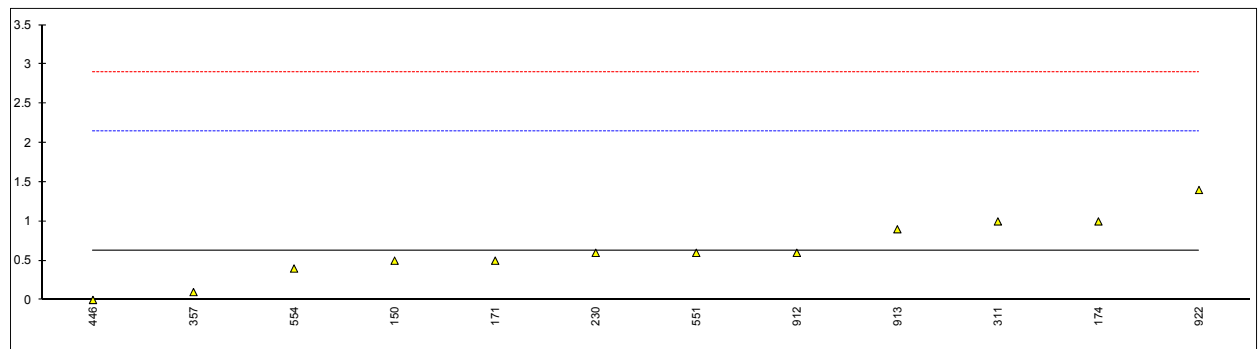
Determination of Density at 20°C on sample #15232; results in kg/L

lab	method	value	mark	z(targ)	remarks
150	D4052	0.8060		-0.06	
171	D4052	0.80604		0.17	
174	D4052	0.8060		-0.06	
230	D4052	0.80605	C	0.22	first reported: 0.80495
273	D4052	0.8059		-0.62	
311	D4052	0.8060		-0.06	
312	ISO12185	0.8060		-0.06	
323	D4052	0.8060		-0.06	
329	D4052	0.8059		-0.62	
357	D4052	0.80599		-0.11	
446	D4052	0.8059		-0.62	
522	D4052	0.8062		1.06	
529	D4052	0.80598		-0.17	
541	ISO12185	0.8060		-0.06	
551	D4052	0.80606		0.28	
554		0.8061		0.50	reported: 806.1 kg/L
558	D4052	0.80611		0.56	
859	D4052	0.8060		-0.06	
912	D4052	0.8061		0.50	
913	D4052	0.8060		-0.06	
922	D4052	0.80601		0.00	
1126	ISO12185	0.80599		-0.11	
1205	in house	0.805985		-0.14	
1241	in house	0.80601		0.00	
1242		0.805974		-0.20	
1253	ISO12185	0.80602		0.06	
1574		----		----	
1605	D4052	0.805980		-0.17	
1726	D4052	0.80603		0.11	
1727	D4052	0.80598		-0.17	
1817		0.80600		-0.06	
1835	D4052	0.80600		-0.06	
normality		not OK			
n		31			
outliers		0			
mean (n)		0.806010			
st.dev. (n)		0.0000607			
R(calc.)		0.000170			
R(ISO12185:96)		0.000500			



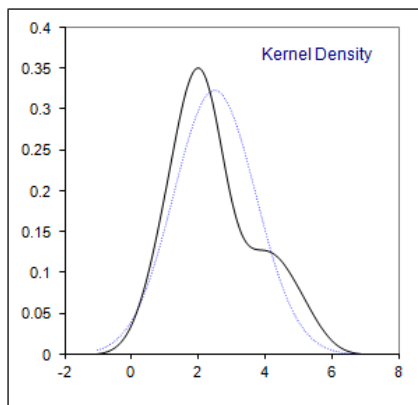
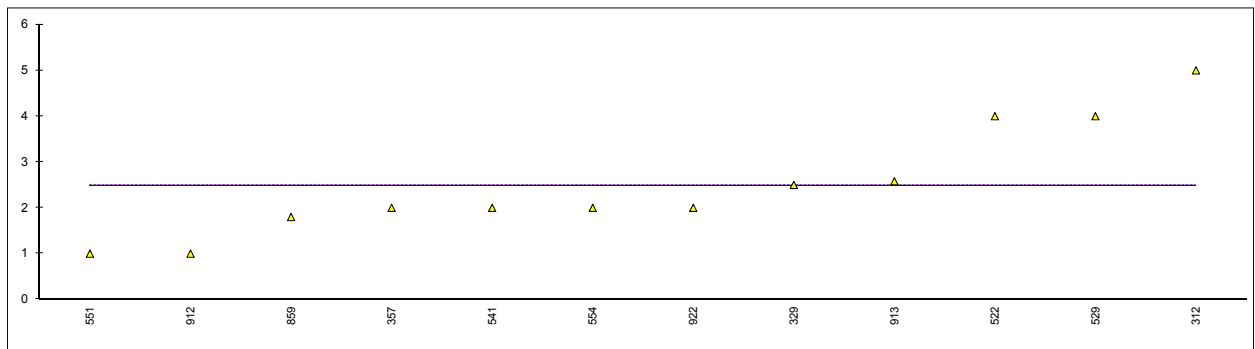
Determination of Nonvolatile matter on sample #15232; results in mg/100mL

lab	method	value	mark	z(targ)	remarks
150	D1353	0.5		-0.18	
171	D1353	0.5		-0.18	
174	D1353	1.0		0.49	
230	D1353	0.6		-0.04	
273		----		----	
311	D1353	1		0.49	
312	D1353	<1		----	
323	D1353	<1		----	
329	EN15691	<1		----	
357	D1353	0.1		-0.71	
446	D1353	0.0		-0.84	
522		----		----	
529		----		----	
541	D1353	< 1		----	
551	D1353	0.6		-0.04	
554		0.4		-0.31	
558		----		----	
859	D1353	<1		----	
912	D1353	0.6		-0.04	
913	D1353	0.9		0.35	
922	D1353	1.40		1.02	
1126		----		----	
1205		----		----	
1241		----		----	
1242		----		----	
1253		----		----	
1574		----		----	
1605		----		----	
1726	EN15691	<1		----	
1727	EN15691	<1		----	
1817		----		----	
1835	EN15691	<10		----	
	normality	OK			
	n	12			
	outliers	0			
	mean (n)	0.63			
	st.dev. (n)	0.394			
	R(calc.)	1.10			
	R(D1353:13)	2.11			



Determination of Permanganate Time Test at 20°C on sample #15232; results in minutes

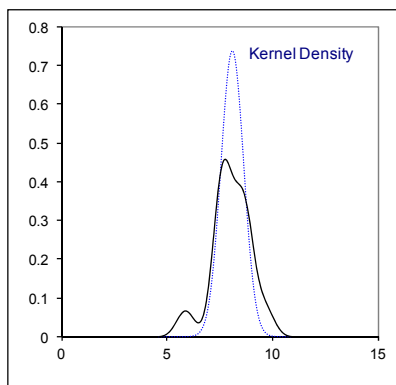
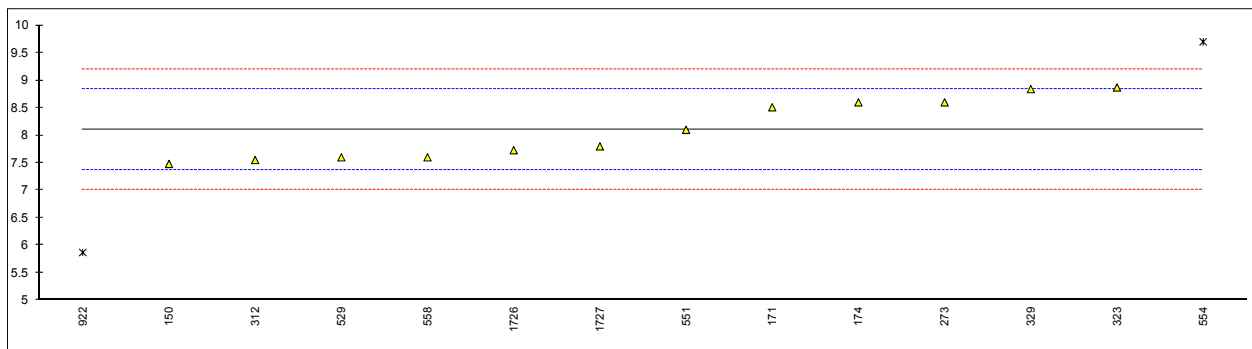
lab	method	value	mark	z(targ)	remarks
150	D1363	<5		----	
171	D1363	<3		----	
174	D1363	<3		----	
230	D1363	<50		----	
273		----		----	
311	D1363	<5		----	
312	D1363	5		----	
323	D1363	<5		----	
329	D1363	2.5		----	
357	D1363	2		----	
446		----		----	
522	D1363	4		----	
529	D1363	4.0		----	
541	D1363	2		----	
551	D1363	1		----	
554		2		----	
558		----		----	
859	D1363	1.8		----	
912	D1363	1		----	
913	D1363	2.58		----	
922	D1363	2		----	
1126		----		----	
1205		----		----	
1241		----		----	
1242		----		----	
1253		----		----	
1574		----		----	
1605		----		----	
1726		----		----	
1727		----		----	
1817		----		----	
1835	D1363	<2		----	
	normality	OK			
	n	18			
	outliers	0			
	mean (n)	≤ 5			
	st.dev. (n)	(1.2338)			
	R(calc.)	(3.455)			
	R(D1363:06)	n.a.			



Determination of pHe on sample #15232;

lab	method	electrode	value	mark	z(targ)	remarks
150	D6423	KCl	7.48		-1.72	
171	D6423	KCl	8.51		1.10	
174	D6423	KCl	8.6		1.35	
230			----		----	
273	D6423		8.6		1.35	
311			----		----	
312	D6423	KCl	7.55		-1.52	
323	EN15490	LiCl	8.87		2.09	
329	EN15490	LiCl	8.84		2.01	
357			----		----	
446			----		----	
522			----		----	
529	D6423	KCl	7.60		-1.39	
541			----		----	
551	D6423	KCl	8.1		-0.02	
554		KCl	9.7	D(0.01)	4.36	
558	NBR10841		7.60		-1.39	
859			----		----	
912			----		----	
913			----		----	
922	D6423	KCl	5.87	C, D(0.01)	-6.12	first reported: 6.87
1126			----		----	
1205			----		----	
1241			----		----	
1242			----		----	
1253			----		----	
1574			----		----	
1605			----		----	
1726	EN15490	LiCl	7.73		-1.03	
1727	EN15490	LiCl	7.80		-0.84	
1817			----		----	
1835			----		----	

normality OK
n 12
outliers 2
mean (n) 8.107
st.dev. (n) 0.5412
R(calc.) 1.515
R(D6423:14) 1.023



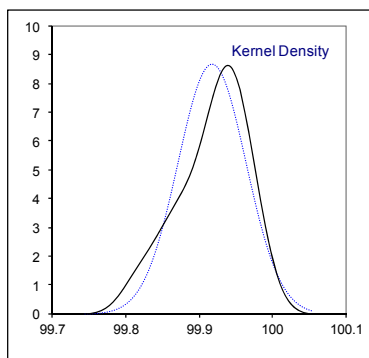
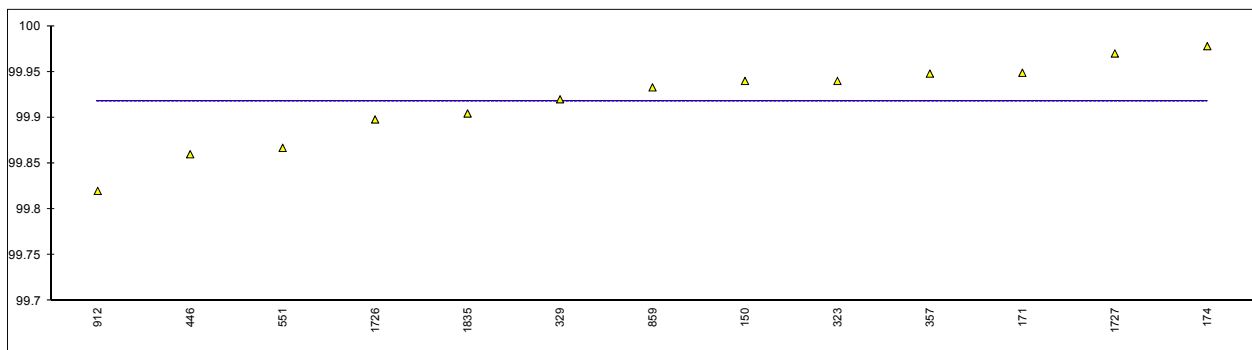
Determination of Purity of Ethanol on dry basis on sample #15232; results in %M/M

lab	method	value	mark	z(targ)	remarks
150	INH-0001	99.94		----	
171	EN15721	99.9489		----	
174	D5501	99.978		----	
230		----		----	
273		----		----	
311		----		----	
312		----		----	
323	EN15721	99.94		----	
329	in house	99.92		----	reported: gas chromatographic analysis
357	EN15721	99.948		----	
446	INH-1316	99.86		----	
522		----		----	
529		----		----	
541		----		----	
551	INH-1313	99.867		----	
554		----		----	
558		----		----	
859	GB/T394	99.933		----	
912		99.82		----	
913		----		----	
922		----		----	
1126		----		----	
1205		----		----	
1241		----		----	
1242		----		----	
1253		----		----	
1574		----		----	
1605		----		----	
1726	EN15721	99.898		----	
1727	EN15721	99.97		----	
1817		----		----	
1835	EN15721	99.9045		----	

EN15721 results only

normality	OK	OK
n	13	6
outliers	0	0
mean (n)	99.9175	99.9349
st.dev. (n)	0.04610	0.02797
R(calc.)	0.1291	0.0783
R(target)	n.a.	0.0468 (R(EN15721:13))

Compare R(iis14C11) = 0.0176

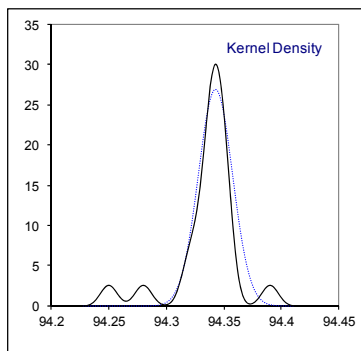
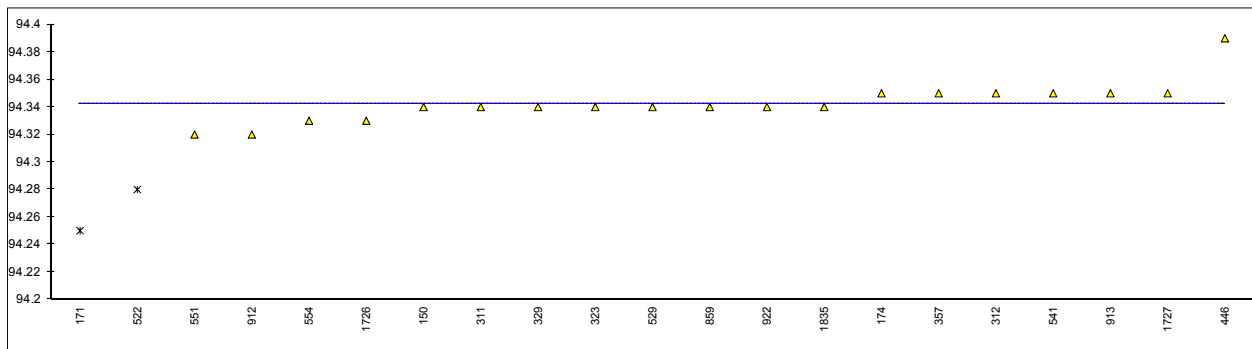


Determination of Strength on sample #15232; results in %M/M

lab	method	value	mark	z(target)	remarks
150	Table OIML	94.34		----	
171	Table AOAC	94.25	R(0.01)	----	
174	Table OIML	94.35		----	
230		----		----	
273		----		----	
311	Table OIML	94.34		----	
312	Table OIML	94.35		----	
323	Table OIML	94.34	C	----	first reported: 94.39
329	Table OIML	94.34	C	----	first reported: 94.39
357	Table OIML	94.35		----	
446	Table OIML	94.39		----	
522	D4052 - OIML	94.28	R(0.05)	----	
529	Table OIML	94.34		----	
541	Table OIML	94.35		----	
551	NBR15639	94.32		----	
554		94.33		----	
558		----		----	
859		94.34		----	
912	D4052 - OIML	94.32		----	
913	Table OIML	94.35		----	
922	Table OIML	94.34		----	
1126		----		----	
1205		----		----	
1241		----		----	
1242		----		----	
1253		----		----	
1574		----		----	
1605		----		----	
1726	Table OIML	94.33		----	
1727	Table OIML	94.35		----	
1817		----		----	
1835	Table OIML	94.34		----	

normality not OK
n 19
outliers 2
mean (n) 94.343
st.dev. (n) 0.0148
R(calc.) 0.042
R(target) n.a.

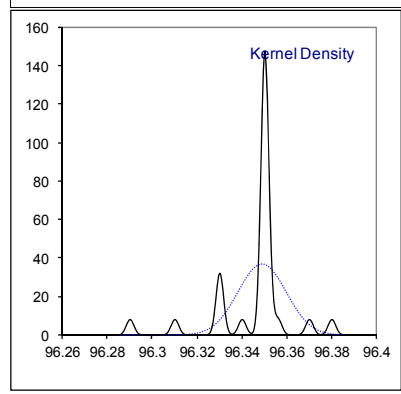
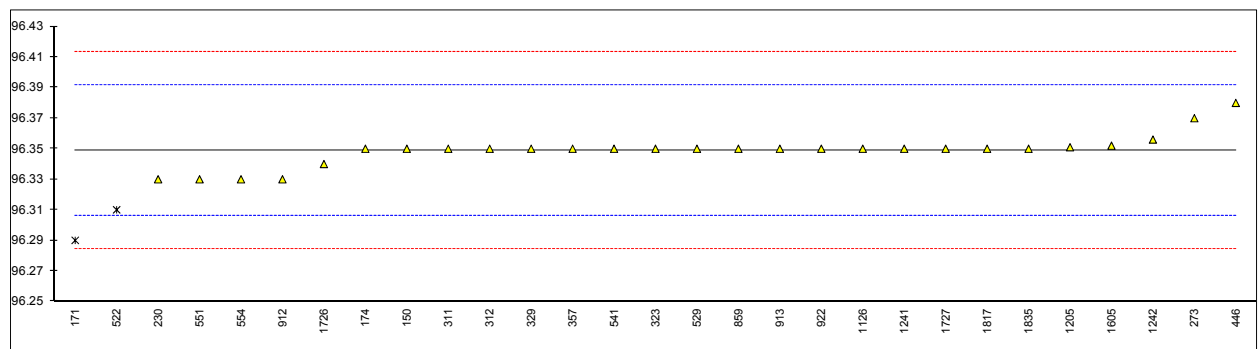
Compare R(iis14C11) = 0.053



Determination of Strength on sample #15232; results in %V/V

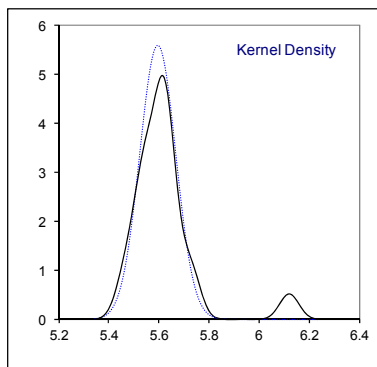
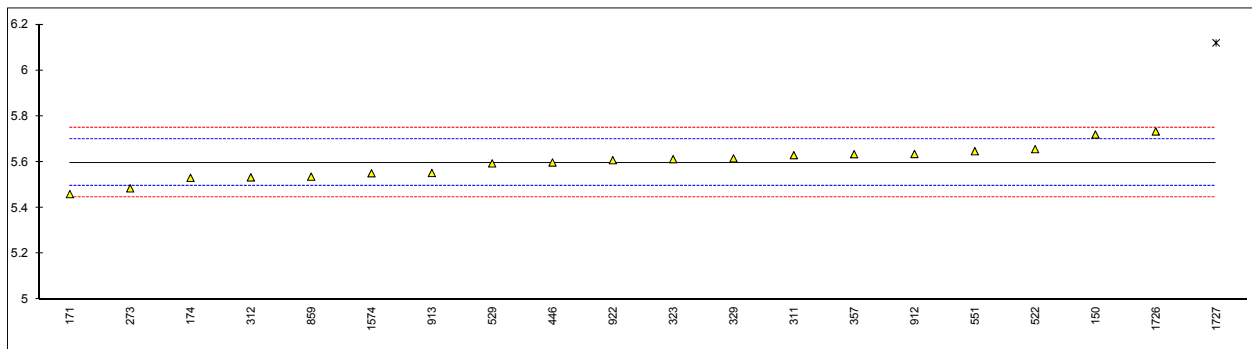
lab	method	value	mark	z(targ)	remarks
150	Table OIML	96.35		0.05	
171	Table AOAC	96.29	R(0.01)	-2.75	
174	Table OIML	96.35		0.05	
230	Table OIML	96.33		-0.88	
273	Table OIML	96.37		0.99	
311	Table OIML	96.35		0.05	
312	Table OIML	96.35		0.05	
323	Table OIML	96.35	C	0.05	first reported: 96.40
329	Table OIML	96.35	C	0.05	first reported: 96.40
357	Table OIML	96.35		0.05	
446	Table OIML	96.38		1.45	
522	D4052 - OIML	96.31	R(0.05)	-1.81	
529	Table OIML	96.35		0.05	
541	Table OIML	96.35		0.05	
551	NBR15639	96.33		-0.88	
554		96.33		-0.88	
558		----		----	
859	Table OIML	96.35		0.05	
912	D4052 - OIML	96.33		-0.88	
913	Table OIML	96.35		0.05	
922	Table OIML	96.35		0.05	
1126	Table OIML	96.35		0.05	
1205	Table OIML	96.351		0.10	
1241	Table OIML	96.35		0.05	
1242		96.356		0.33	
1253		----		----	
1574		----		----	
1605	Table OIML	96.352		0.15	
1726	Table OIML	96.34		-0.41	
1727	Table OIML	96.35		0.05	
1817	Table OIML	96.35		0.05	
1835	Table OIML	96.35		0.05	

normality not OK
n 27
outliers 2
mean (n) 96.3488
st.dev. (n) 0.01075
R(calc.) 0.0301
R(OIML table) 0.0600



Determination of Water (Titrimetric) on sample #15232; results in %M/M

lab	method	value	mark	z(targ)	remarks
150	E1064	5.72		2.44	
171	E203	5.4599		-2.69	
174	D1364	5.531	C	-1.29	reported 5.531 mg/kg
230		----		----	
273	E203	5.485	C	-2.20	first reported: 6.145
311	E203	5.63		0.66	
312	E203	5.533		-1.25	
323	D1364	5.6124		0.32	reported 56124 mg/kg
329	E203	5.616		0.39	
357	E203	5.634		0.74	
446	E203	5.598		0.03	
522	E203	5.656		1.18	
529	D1364	5.594	C	-0.05	reported 5.594 mg/kg
541		----		----	
551	D1364	5.647		1.00	
554		----		----	
558		----		----	
859	D1364	5.536		-1.19	
912	E203	5.635		0.76	
913	D1364	5.552		-0.88	
922	D1364	5.608		0.23	
1126		----		----	
1205		----		----	
1241		----		----	
1242		----		----	
1253		----		----	
1574		5.5509		-0.90	
1605		----		----	
1726	D1364	5.7335		2.70	
1727	EN15692	6.12	R(0.01)	10.33	
1817		----		----	
1835		----		----	
normality		OK			
n		19			
outliers		1			
mean (n)		5.5964			
st.dev. (n)		0.07150			
R(calc.)		0.2002			
R(D1364:02)		0.1419			



Determination of UV absorbance on sample #15232;

lab	method	cuvette	300nm	270nm	260nm	250nm	240nm	230nm	220nm	Pass/Fail
150	INH-001	10 mm	----	----	----	----	----	----	----	Fail
171	IMPCA004	10 mm	0.0368	1.5432	3.3468	3.4704	3.6128	3.6415	3.7290	Fail
174		10 mm	----	----	----	----	----	----	----	----
230	INH-001	50 mm	0.0178 *)	3.4186 (ex)	3.6438	3.6421	3.1868	3.3947	3.4538	Fail
273	IMPCA004	10 mm	0.013	1.545	3.065	3.130	3.172	3.297	2.984	Fail
311			----	----	----	----	----	----	----	----
312	INH-001	50 mm	0.019 *)	3.681 (ex)	3.996	3.835	3.219	3.912	3.674	Fail
323	INH-I	10 mm	0.0165	1.582	>3	>3	>3	>3	>3	Fail
329	in house	10 mm	0.0155	1.538	3.617	3.717	3.528	3.824	3.610	Fail
357	INH-001	10 mm	0.019	1.541	> 3	> 3	> 3	> 3	> 3	Fail (> 3)
446	INH-CM	50 mm	0.020 *)	3.476 (ex)	3.599	3.753	3.830	3.794	3.840	Fail
522			----	----	----	----	----	----	----	----
529		10 mm	0.0152	1.4979	3.6326	3.7886	3.6554	3.7177	3.7584	Fail
541			----	----	----	----	----	----	----	----
551	INH-3063	50 mm	0.022 *)	3.603 (ex)	3.711	3.804	3.867	3.856	3.909	Fail
554			----	----	----	----	----	----	----	----
558			----	----	----	----	----	----	----	----
859	INH-001	50 mm	0.019 *)	3.947 (ex)	3.958	3.982	3.984	3.961	3.973	Fail
912			----	----	----	----	----	----	----	----
913			----	----	----	----	----	----	----	----
922	in house	10 mm	0.0179	1.5529	3.4296	3.6477	3.9070	4.0858	4.0065	Fail
1126			----	----	----	----	----	----	----	----
1205			----	----	----	----	----	----	----	----
1241			----	----	----	----	----	----	----	----
1242		10 mm	0.0185	1.5515	3.4060	3.4930	3.5470	3.5650	3.6650	Fail
1253			----	----	----	----	----	----	----	----
1574			----	----	----	----	----	----	----	----
1605		10 mm	0.021	1.532	2.751	2.932	2.985	3.036	3.002	----
1726			----	----	----	----	----	----	----	----
1727	in house	10 mm	0.075	1.47	3.32	3.41	3.5	3.95	3.95	Pass
1817			----	----	----	----	----	----	----	----
1835		10 mm	0.019	1.53	2.95	3.43	3.74	3.65	3.90	Pass
	normality		OK	suspect	OK	OK	OK	OK	OK	n.a.
	n		14	11	14	14	14	14	12	12
	outliers		2	0+5ex	0	0	0	0	2	2 (Pass)
	mean (n)		0.0181	1.535	(3.46)	(3.57)	(3.55)	(3.69)	(3.79)	Fail
	st.dev. (n)		0.00239	0.0294	(0.357)	(0.287)	(0.310)	(0.290)	(0.169)	n.a.
	R(calc.)		0.0067	0.082	(1.00)	(0.80)	(0.87)	(0.81)	(0.47)	n.a.
	R(target)		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	R(iis14C11) =		0.0052	at abs=0.0190						

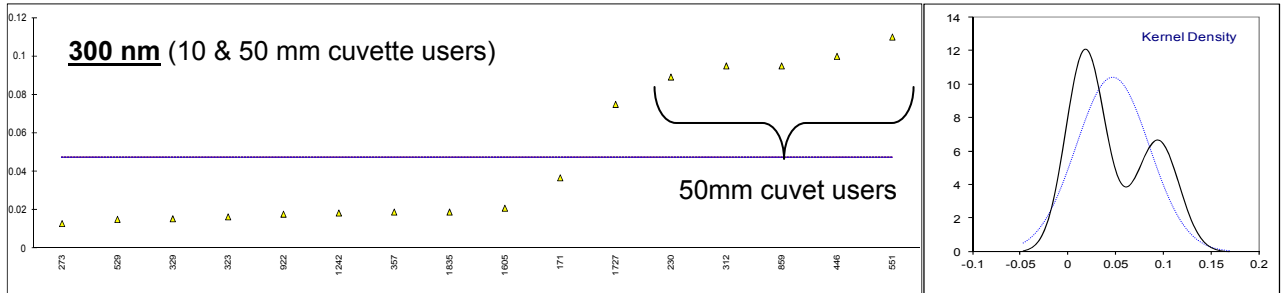
Bold and underlined results: outliers according to D(0.01)

Bold results: outliers according to DG(0.01)

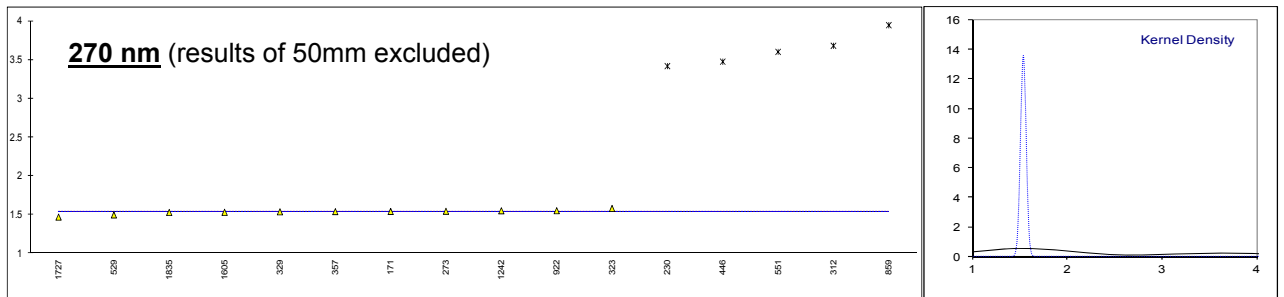
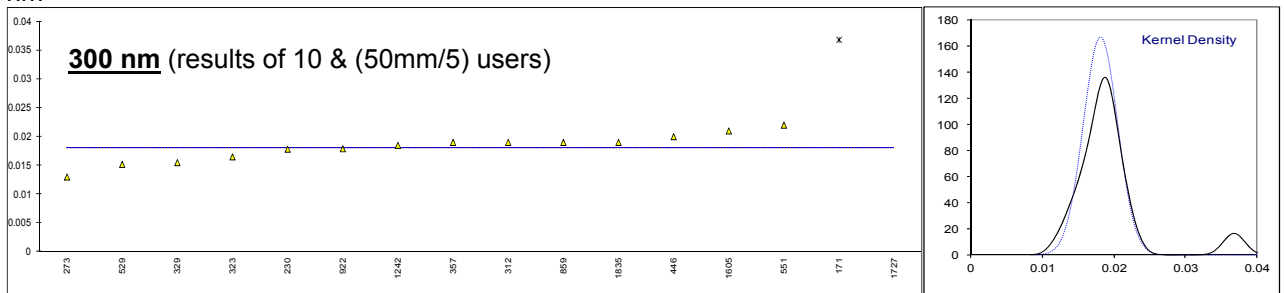
Lab 311 reported: UV measurement not possible due to absorbencies out of range

Lab 329 reported: UV absorbance: against pure demi-water

*) Labs 230; 312; 446; 551; 859 used a 50 mm cuvette. For the evaluation the reported values were divided by 5. The original reported values are respectively; 0.0892; 0.095; 0.100; 0.110; 0.095 (ex)=reported results from 50 mm are excluded from the evaluation, see § 4.1 for more discussion



For evaluation; reported values of 50mm users divided by 5 acc. to Law of Lambert-Beer, see next fig. of 300 nm



APPENDIX 2

Number of participants per country

3 labs in UNITED STATES OF AMERICA
1 lab in MAURITIUS
1 lab in SOUTH AFRICA
6 labs in NETHERLANDS
4 labs in BELGIUM
1 lab in FINLAND
1 lab in UNITED KINGDOM
2 labs in MEXICO
1 lab in ARGENTINA
3 labs in BRAZIL
1 lab in CHINA, People's Republic
2 labs in INDIA
1 lab in PAKISTAN
1 lab in HONG KONG
3 labs in SPAIN
1 lab in THAILAND

APPENDIX 3

Abbreviations:

C	= final result after checking of first reported suspect result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner outlier test
R(0.05)	= straggler in Rosner outlier test
E	= probably error in calculations
ex	= test result excluded from calculations
n.a.	= not applicable
OILM	= International Organization of Legal Metrology
U	= reported in a deviating unit
SDS	= safety data sheet

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- 3 ASTM E178-02
- 4 ASTM E1301-03
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- 10 DIN 38402 T41/42
- 11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 12 J.N. Miller, Analyst, 118, 455, (1993)
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- 15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), pages 165-172, (1983)
- 16 M.A. Gonçalves et.al., Sensors and Actuators B158 (2011) pages 327-332