

Results of Proficiency Test
Ethanol (Food/Neutral grade)
December 2019

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2007, a proficiency test for Ethanol (Food/Neutral grade) is organized by the Institute for Interlaboratory Studies every year. During the annual proficiency testing program of 2019/2020, it was decided to continue the round robin for the analysis of Ethanol (Food/Neutral grade).

In this interlaboratory study 26 laboratories in 17 different countries registered for participation. See appendix 3 for the number of participants per country.

In this report, the results of the 2019 proficiency test 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/IEC17025 accredited laboratory. It was decided to send two different samples of Ethanol (Food/Neutral grade), one bottle of 1L labelled #19252 and one bottle of 0.25L labelled #19253 for GC determination only.

The 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/IEC17043:2010 (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 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). 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

Preparation of samples for PT on Ethanol (Food/Neutral grade), regular analysis

A batch of approximately 35 liters of Ethanol (Food/Neutral grade) was obtained from a local supplier. After homogenisation 79 amber glass bottles of 0.5 L were filled and labelled #19252. The homogeneity of the subsamples was checked by determination of Density at 20°C in accordance with ASTM D4052 and Water in accordance with ASTM E203 on 8 stratified randomly selected samples.

	Density at 20°C in kg/L	Water in %M/M
Sample #19252-1	0.80588	5.697
Sample #19252-2	0.80589	5.704
Sample #19252-3	0.80589	5.695
Sample #19252-4	0.80589	5.693
Sample #19252-5	0.80588	5.695
Sample #19252-6	0.80588	5.698
Sample #19252-7	0.80589	5.701
Sample #19252-8	0.80589	5.697

Table 1: homogeneity test results of subsamples #19252

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

	Density at 20°C in kg/L	Water in %M/M
r (observed)	0.00001	0.010
reference test method	ISO12185:96	EN203:16
0.3 x R (ref. test method)	0.00015	0.023

Table 2: evaluation of the repeatabilities of subsamples #19252

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibility of the reference test methods. Therefore, homogeneity of the subsamples was assumed.

Preparation of samples for PT on Ethanol (Food/Neutral grade), GC determination only

A batch of approximately 15 liters Ethanol (Food/Neutral grade) was separated from the batch for the main round and spiked with Methanol (approx. 25 mg/kg), Acetone (approx. 15 mg/kg), Benzene (approx. 15 mg/kg), Isopropanol (approx. 20 mg/kg), Acetal (approx. 20 mg/kg) and Monoethylene Glycol (approx. 25 mg/kg). After homogenization 58 amber glass bottles of 0.25 L were filled and labelled #19253. The homogeneity of the subsamples was checked by determination of Benzene on 8 stratified randomly selected samples.

	Benzene in mg/kg
Sample #19253-1	17.4
Sample #19253-2	17.7
Sample #19253-3	17.3
Sample #19253-4	18.2
Sample #19253-5	16.9
Sample #19253-6	17.4
Sample #19253-7	16.3
Sample #19253-8	17.2

Table 3: homogeneity test results of subsamples #19253

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

	Benzene in mg/kg
r (observed)	1.56
reference method	Horwitz
0.3 x R (ref. method)	1.51

Table 4: evaluation of the repeatability of subsamples #19253

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

To each of the participating laboratories one bottle of 0.5 L labelled #19252 and one bottle of 0.25 L labelled #19253 was sent on November 6, 2019. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYSES

The participants were asked to determine on sample #19252: Density at 20°C, Nonvolatile matter, Permanganate Time Test at 20°C, pHe, Strength (in %M/M and %V/V), Water and UV Absorbance at 300, 270, 260, 250, 240, 230 and 220 nm with an evaluation of the UV-scan.

The participants were asked to determine on sample #19253: Purity Ethanol on dry basis, Methanol, Acetal (1,1-diethoxyethane), Acetaldehyde, Acetone, Benzene, Isopropanol, Monoethylene glycol (MEG), Other impurities and Total impurities.

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 evaluations.

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 appropriate 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 reanalyzes). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of 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, 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 them with a factor of 2.8.

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis, the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

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

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

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

Absolute values for $z < 2$ are very common and absolute values for $z > 3$ are very rare. The usual interpretation of z-scores is as follows:

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

4 EVALUATION

In this proficiency test, some problems were encountered with the dispatch of the samples. One participant did not report any test results at all and one participant reported after the final reporting date. Not all participants were able to report results for all analyses requested. In total 25 laboratories reported 337 numerical results. Observed were 19 outlying results, which is 5.6%. 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, which were used by the various laboratories, are taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data. The abbreviations, used in these tables, are explained in appendix 4.

Unfortunately, a suitable reference 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. 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 will be used.

Sample #19252

Density at 20°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ISO12185:96.

Nonvolatile matter: This 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 at 20°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D1363:06(2011).

pHe: 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 D6423:19.

Strength (%M/M): This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the reproducibility derived from the OIML table and ISO12185:96.

Strength (%V/V): This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the reproducibility derived from the OIML table and ISO12185:96.

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility using the Horwitz equation.

UV absorbance: Regretfully, no reference test method for this determination exists. Some participants reported test results obtained with a 50 mm cuvette, others with a 10 mm cuvette. In order to determine a Pass or Fail based on the sample UV-graph, it is important that even the smallest deviation is detected visually. Therefore, the use of a 50 mm cuvette is preferable. Eight laboratories used a 50 mm cuvette and ten laboratories used a 10 mm cuvette. Both groups were evaluated separately.

UV - 50 mm cuvette: In total over seven parameters (UV absorbance in nm), two statistical outliers were observed. All laboratories evaluated the sample as 'Pass'.

UV - 10 mm cuvette: In total over seven parameters (UV absorbance in nm), seven statistical outliers were observed. All laboratories evaluated the sample as 'Pass'.

Sample #19253

Purity of Ethanol on dry basis: Regretfully, no reference test method is available that gives a clear definition of purity in Ethanol Food/Neutral grade. Therefore, no z-scores could be calculated. Three statistical outliers were observed. The calculated reproducibility is smaller than the calculated reproducibility in the previous proficiency tests iis18C11 and iis17C16.

Methanol: This determination may be problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility using the Horwitz equation.

Acetal: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility using the Horwitz equation.

Acetone: This determination may be problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the estimated reproducibility using the Horwitz equation.

Benzene: This determination may be problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the estimated reproducibility using the Horwitz equation.

Isopropanol: This determination may be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility using the Horwitz equation.

Monoethylene glycol: Four laboratories reported a numeric test result and three other laboratories reported a "less than" test result. Therefore, no z-scores were calculated.

Total impurities: This determination may be problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the estimated reproducibility using the Horwitz equation calculated for 6 components.

The impurities which were not detected are listed in appendix 2.

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 or as declared by the estimated target reproducibility using the Horwitz equation and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average result, calculated reproducibilities ($2.8 \cdot$ standard deviation) and the target reproducibilities derived from literature reference test methods (in casu ASTM and ISO test methods) or the estimated target reproducibility are presented in the next table.

Parameter	unit	n	average	$2.8 \cdot$ sd	R (lit)
Density at 20°C	kg/L	23	0.8059	0.0002	0.0005
Nonvolatile matter	mg/100mL	7	0.5	1.1	2.1
Permanganate Time Test	minutes	13	33.3	4.8	8.4
pHe		9	7.9	1.7	1.0
Strength	%M/M	13	94.37	0.05	0.06
Strength	%V/V	20	96.36	0.06	0.06
Water	%M/M	18	5.63	0.33	0.49
UV – 50 mm cuvette:					
UV-absorbance 300 nm		7	0.009	0.021	n.a.
UV-absorbance 270 nm		7	0.025	0.038	n.a.
UV-absorbance 260 nm		7	0.041	0.035	n.a.
UV-absorbance 250 nm		7	0.101	0.043	n.a.
UV-absorbance 240 nm		8	0.243	0.087	n.a.
UV-absorbance 230 nm		7	0.549	0.057	n.a.
UV-absorbance 220 nm		7	1.03	0.111	n.a.
Conclusion UV-scan	Pass/Fail	6	Pass	n.a.	n.a.
UV – 10 mm cuvette:					
UV-absorbance 300 nm		7	-0.001	0.009	n.a.
UV-absorbance 270 nm		7	0.002	0.011	n.a.
UV-absorbance 260 nm		8	0.006	0.010	n.a.
UV-absorbance 250 nm		8	0.018	0.012	n.a.
UV-absorbance 240 nm		8	0.045	0.013	n.a.
UV-absorbance 230 nm		8	0.106	0.015	n.a.
UV-absorbance 220 nm		8	0.201	0.030	n.a.
Conclusion UV-scan	Pass/Fail	10	pass	n.a.	n.a.

Table 5: reproducibilities of tests on sample #19252

Parameter	unit	n	average	2.8 *sd	R (lit)
Purity of Ethanol on dry basis	%M/M	13	99.99	0.002	n.a.
Methanol	mg/kg	15	25.03	8.2	6.9
Acetal (1,1-diethoxyethane)	mg/kg	14	19.2	4.9	5.5
Acetone	mg/kg	14	19.3	4.2	5.5
Benzene	mg/kg	16	15.3	6.3	4.5
Isopropanol	mg/kg	14	30.8	12.3	8.2
Monoethylene glycol (MEG)	mg/kg	4	24.9	20.8	n.a.
Total impurities	mg/kg	12	124.0	78.5	65.9

Table 6: reproducibilities of tests on sample #19253

Without further statistical calculations, it could be concluded that for several tests there is a good compliance of the group of laboratories with the relevant reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2019 WITH PREVIOUS PTS

	December 2019	December 2018	December 2017	December 2016	November 2015
Number of reporting laboratories	25	25	29	26	32
Number of results reported	337	303	301	329	254
Number of statistical outliers	19	20	22	16	11
Percentage statistical outliers	5.6%	6.6%	7.3%	4.9%	4.3%

Table 7: comparison with previous proficiency tests

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

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

Parameter	December 2019	December 2018	December 2017	December 2016	November 2015
Density at 20°C	++	++	++	++	++
Nonvolatile matter	-	++	++	++	++
Permanganate Time Test	+	-	-	(+)	-
pHe	-	++	-	(-)	--
Strength %M/M	+	++	++	(+/-)	(+)
Strength %V/V	+/-	+	++	++	+
Water	+	-	-	-	-
Purity Ethanol on dry basis	(++)	(+)	(+)	(+)	(-)
Methanol	-	--	-	++	n.e.
Acetal (1,1-diethoxyethane)	+	n.e.	n.e.	+/-	n.e.
Acetone	+	+/-	--	n.e.	n.e.
Benzene	-	--	n.e.	. ++	n.e.

Parameter	December 2019	December 2018	December 2017	December 2016	November 2015
Isopropanol	-	+	+	n.e.	n.e.
Monoethylene glycol (MEG)	n.e.	n.e.	n.e.	+/-	n.e.
Total impurities	-	-	-	n.e.	n.e.

Table 8: comparison determinations of samples #19252 and #19253 against the reference test methods

Results between brackets are compared with the observed reproducibility of the previous proficiency test

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

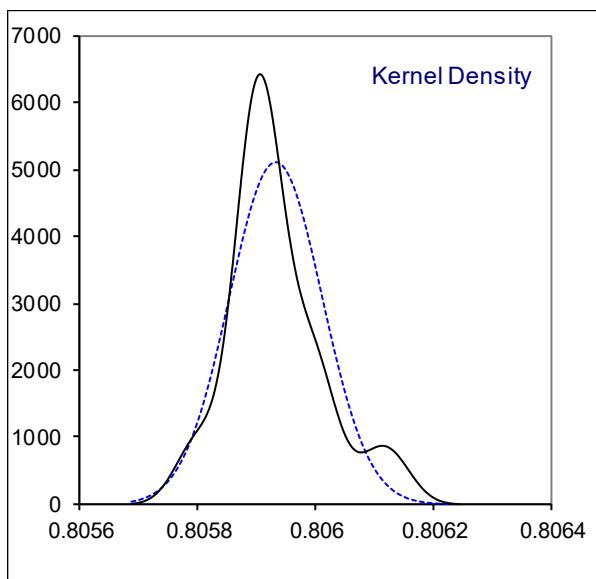
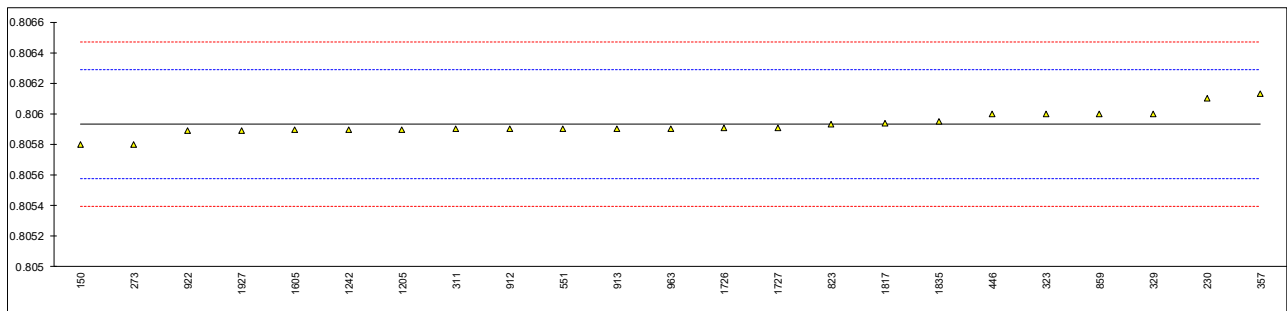
- ++: group performed much better than the reference test method
- + : group performed better than the reference test method
- +/-: group performance equals the reference test method
- : group performed worse than the reference test method
- : group performed much worse than the reference test method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

APPENDIX 1

Determination of Density at 20°C on sample #19252; results in kg/L

lab	method	value	mark	z(targ)	remarks
150	D4052	0.8058		-0.74	
230	D4052	0.80610		0.94	
273	D4052	0.8058		-0.74	
311	D4052	0.8059		-0.18	
323	ISO12185	0.8060		0.38	
329	D4052	0.8060		0.38	
357	D4052	0.80613		1.11	
446	D4052	0.8060		0.38	
541		----		----	
551	D4052	0.8059		-0.18	
823	ISO12185	0.80593		-0.01	
859	D4052	0.8060		0.38	
912	D4052	0.8059		-0.18	
913	D4052	0.8059		-0.18	
922	D4052	0.80589		-0.24	
963	ISO12185	0.8059		-0.18	
1205	In house	0.805898		-0.19	
1242	In house	0.805896		-0.20	
1574		----		----	
1605	D4052	0.805893		-0.22	
1726	D4052	0.80591		-0.12	
1727	D4052	0.80591		-0.12	
1817	Table OIML	0.80594		0.04	
1835	ISO12185	0.80595		0.10	
1927	D4052	0.80589		-0.24	
6224		----		----	

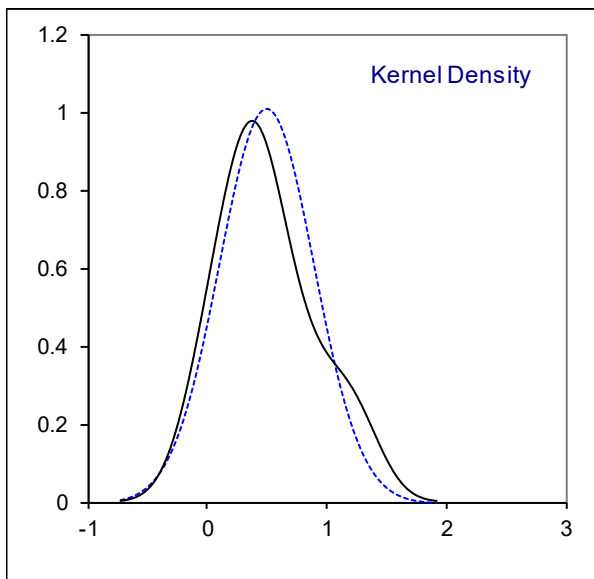
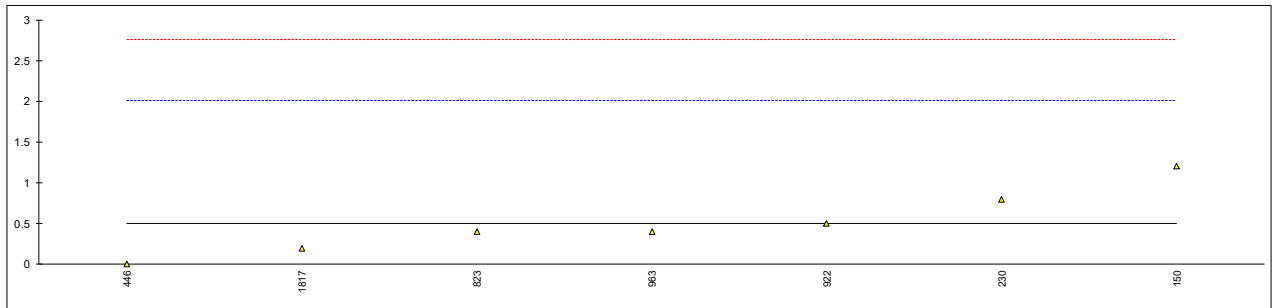
normality suspect
n 23
outliers 0
mean (n) 0.805932
st.dev. (n) 0.0000783
R(calc.) 0.000219
st.dev.(ISO12185:96) 0.0001786
R(ISO12185:96) 0.0005



Determination of Nonvolatile matter on sample #19252; results in mg/100mL

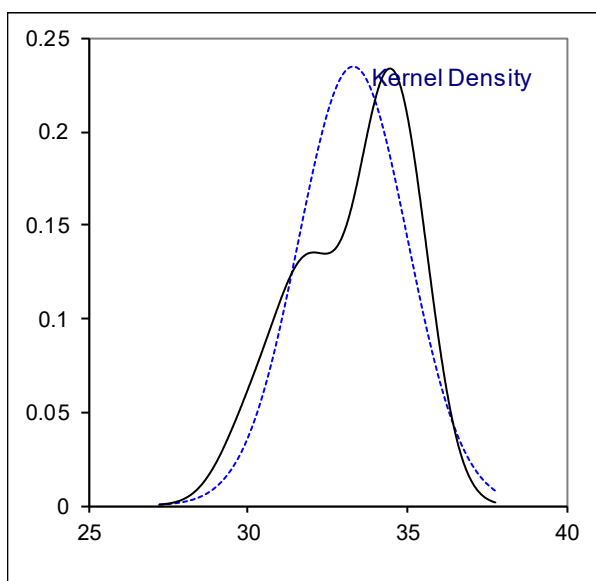
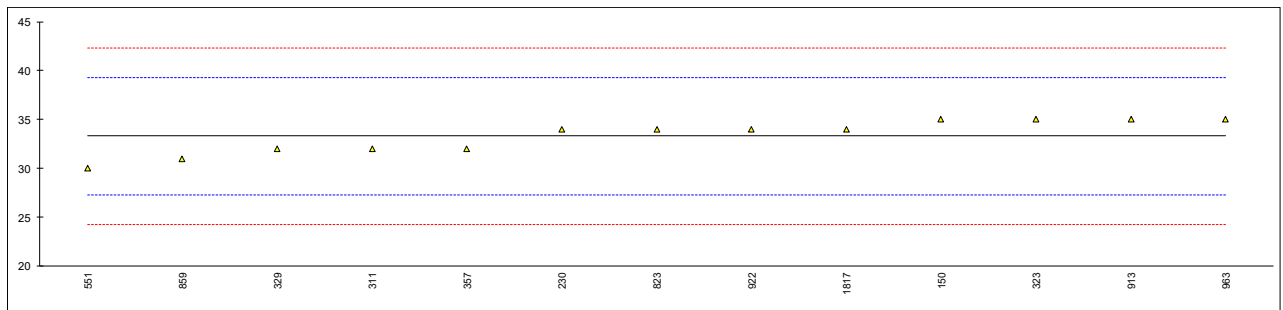
lab	method	value	mark	z(targ)	remarks
150	D1353	1.2		0.93	
230	D1353	0.8	C	0.40	first reported 3
273		----		----	
311	D1353	<1		----	
323	D1353	<1		----	
329	D1353	<1		----	
357	D1353	< 1		----	
446	BS4524	0		-0.66	
541		----		----	
551	D1353	<0.1		----	
823	D1353	0.4		-0.13	
859	D1353	<1		----	
912		----		----	
913	D1353	<1		----	
922	D1353	0.5		0.00	
963	D1353	0.4		-0.13	
1205		----		----	
1242		----		----	
1574		----		----	
1605		----		----	
1726	EN15691	<10		----	
1727	EN15691	<1		----	
1817	In house	0.2		-0.40	
1835	EN15691	<10		----	
1927		----		----	
6224		----		----	

normality unknown
n 7
outliers 0
mean (n) 0.50
st.dev. (n) 0.396
R(calc.) 1.11
st.dev.(D1353:13) 0.754
R(D1353:13) 2.11



Determination of Permanganate Time Test at 20°C on sample #19252; results in minutes

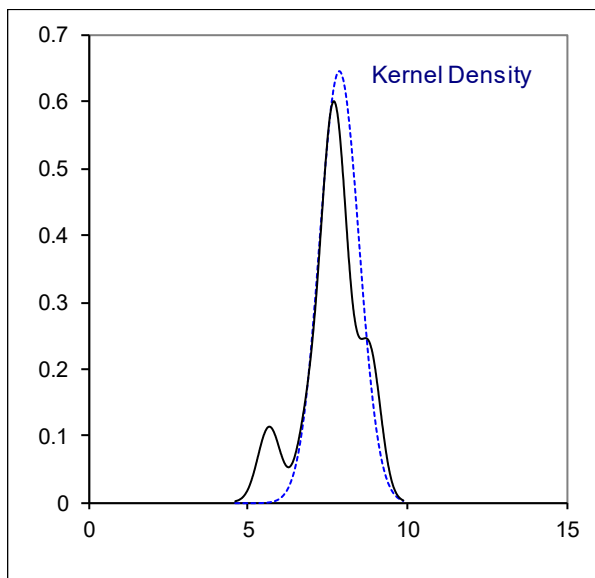
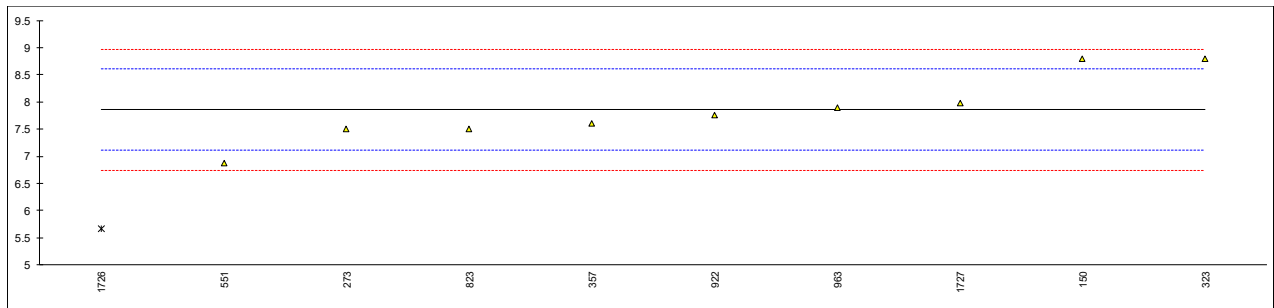
lab	method	value	mark	z(targ)	remarks
150	D1363	35		0.56	
230	D1363	34	C	0.23	first reported 44
273		----			
311	D1363	32		-0.44	
323	D1363	35		0.56	
329	D1363	32		-0.44	
357	D1363	32		-0.44	
446	BS6392	>20		----	
541		----			
551	D1363	30		-1.10	
823	D1363	34		0.23	
859	D1363	31		-0.77	
912		----			
913	D1363	35		0.56	
922	D1363	34		0.23	
963	D1363	35		0.56	
1205		----			
1242		----			
1574		----			
1605		----			
1726		----			
1727		----			
1817	In house	34		0.23	
1835		----			
1927		----			
6224		----			
normality		OK			
n		13			
outliers		0			
mean (n)		33.31			
st.dev. (n)		1.702			
R(calc.)		4.77			
st.dev.(D1363:06)		2.998			
R(D1363:06)		8.39			



Determination of pHe on sample #19252;

lab	method	electrode	value	mark	z(targ)	remarks
150	D6423	KCl	8.79		2.51	
230			----		----	
273	D6423	KCl	7.5		-0.96	
311			----		----	
323	D6423	LiCl	8.8		2.54	
329			----		----	
357	D6423	KCl	7.6		-0.69	
446			----		----	
541			----		----	
551	D6423	KCl	6.88		-2.62	
823	D6423	KCl	7.5		-0.96	
859			----		----	
912			----		----	
913			----		----	
922	D6423	KCl	7.75		-0.28	
963	D6423	KCl	7.9		0.12	
1205			----		----	
1242			----		----	
1574			----		----	
1605			----		----	
1726	EN15490	LiCl	5.66	G(0.01)	-5.91	
1727	EN15490	LiCl	7.98		0.33	
1817			----		----	
1835			----		----	
1927			----		----	
6224			----		----	

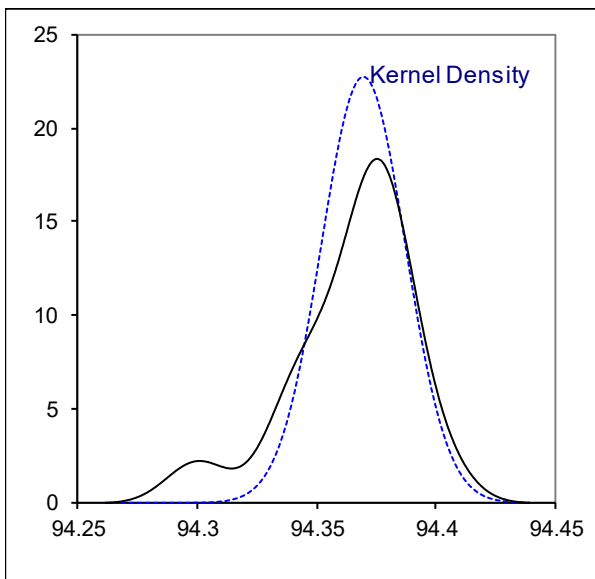
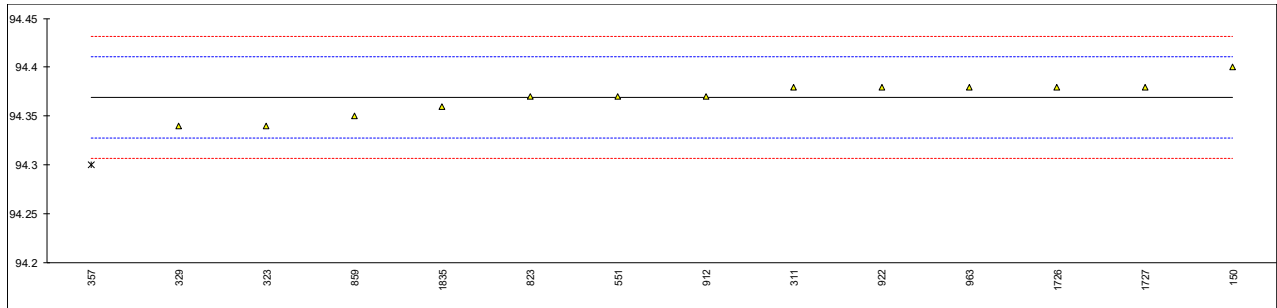
normality OK
n 9
outliers 1
mean (n) 7.856
st.dev. (n) 0.6188
R(calc.) 1.733
st.dev.(D6423:19) 0.3718
R(D6423:19) 1.041



Determination of Strength on sample #19252; results in %M/M

lab	method	value	mark	z(targ)	remarks
150	Table OIML	94.4		1.49	
230		----		----	
273		----		----	
311	Table OIML	94.38		0.52	
323	Table OIML	94.34		-1.41	
329	Table OIML	94.34		-1.41	
357	Table OIML	94.30	G(0.05)	-3.34	
446		----		----	
541		----		----	
551	NBR 15639	94.37		0.04	
823	Table OIML	94.37		0.04	
859	Table OIML	94.35		-0.93	
912	Table OIML	94.37		0.04	
913		----		----	
922	Table OIML	94.38		0.52	
963	Table OIML	94.38		0.52	
1205		----		----	
1242		----		----	
1574		----		----	
1605		----		----	
1726	Table OIML	94.38		0.52	
1727	Table OIML	94.38		0.52	
1817		----		----	
1835	Table OIML	94.36		-0.45	
1927		----		----	
6224		----		----	
normality		OK			
n		13			
outliers		1			
mean (n)		94.369			
st.dev. (n)		0.0175			
R(calc.)		0.049			
st.dev.(OIML table)		0.0207			
R(OIML table)		0.058			

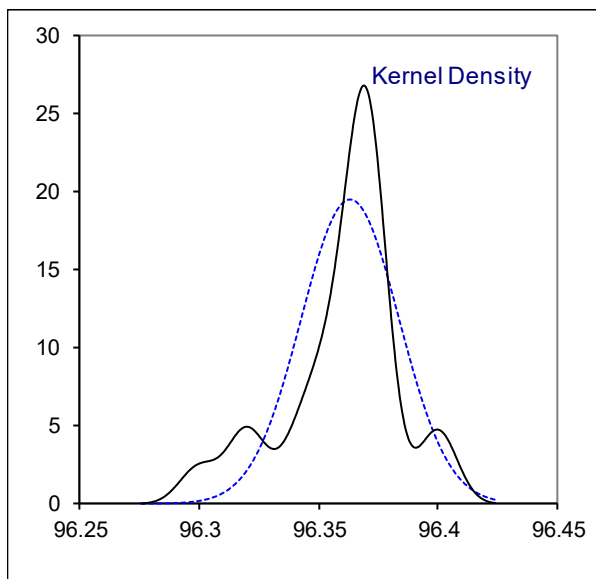
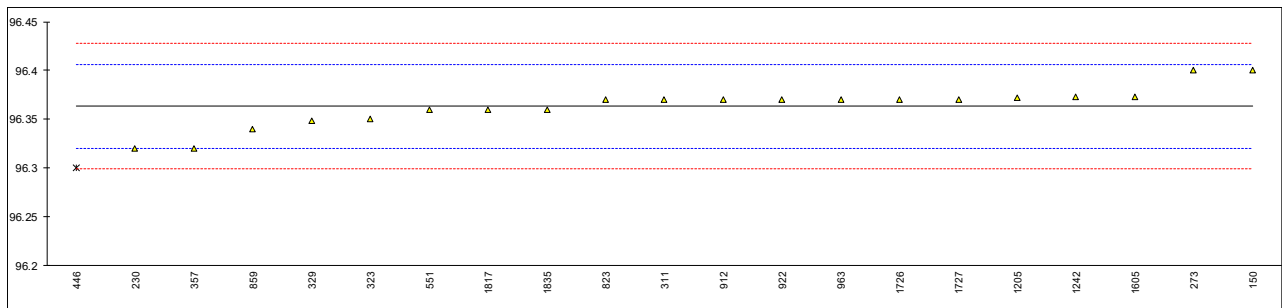
OIML R022-e75



Determination of Strength on sample #19252; results in %V/V

lab	method	value	mark	z(targ)	remarks
150	Table OIML	96.4		1.71	
230	Table OIML	96.32		-2.02	
273	Table OIML	96.4		1.71	
311	Table OIML	96.37		0.31	
323	Table OIML	96.35		-0.62	
329	Table OIML	96.348		-0.71	
357	Table OIML	96.32		-2.02	
446	Table OIML	96.3	G(0.05)	-2.95	
541		----		----	
551	NBR 15639	96.36		-0.15	
823	Table OIML	96.37		0.31	
859	Table OIML	96.34		-1.09	
912	Table OIML	96.37		0.31	
913		----		----	
922	Table OIML	96.37		0.31	
963	Table OIML	96.37		0.31	
1205	Table OIML	96.372		0.41	
1242	In house	96.373		0.45	
1574		----		----	
1605	Table OIML	96.373		0.45	
1726	Table OIML	96.37		0.31	
1727	Table OIML	96.37		0.31	
1817	Table OIML	96.36		-0.15	
1835	Table OIML	96.36		-0.15	
1927		----		----	
6224		----		----	
normality		OK			
n		20			
outliers		1			
mean (n)		96.363			
st.dev. (n)		0.0205			
R(calc.)		0.057			
st.dev.(OIML table)		0.0214			
R(OIML table)		0.060			

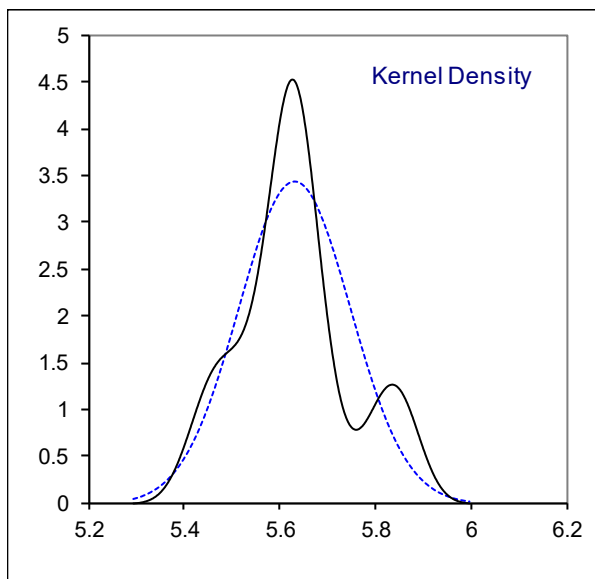
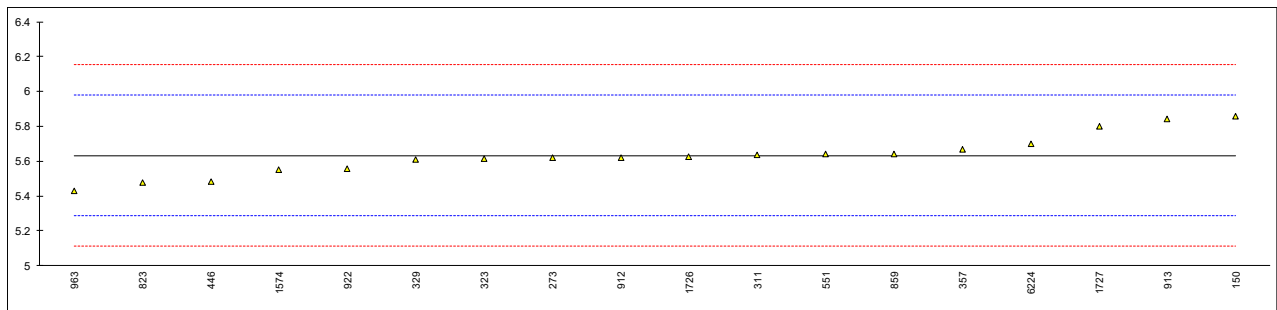
OIML R022-e75



Determination of Water on sample #19252; results in %M/M

lab	method	value	mark	z(targ)	remarks
150	E203	5.86	C	1.31	first reported 6.00
230		----		----	
273	E203	5.62		-0.07	
311	E203	5.637		0.03	
323	E203	5.615		-0.10	
329	E203	5.611		-0.12	
357	E203	5.665		0.19	
446	E203	5.484		-0.85	
541		----		----	
551	D1364	5.642		0.06	
823	D1364	5.477		-0.89	
859	D1364	5.642		0.06	
912	E203	5.62		-0.07	
913	E203	5.842		1.21	
922	E203	5.555		-0.44	
963	D1364	5.432		-1.15	
1205		----		----	
1242		----		----	
1574		5.5490		-0.48	
1605		----		----	
1726	EN15692	5.6251		-0.04	
1727	EN15692	5.80	C	0.97	first reported 5.92
1817		----		----	
1835		----		----	
1927		----		----	
6224	In house	5.7		0.39	
normality		OK			
n		18			
outliers		0			
mean (n)		5.6320			
st.dev. (n)		0.11633			
R(calc.)		0.3257			
st.dev.(Horwitz)		0.17367			
R(Horwitz)		0.4863			

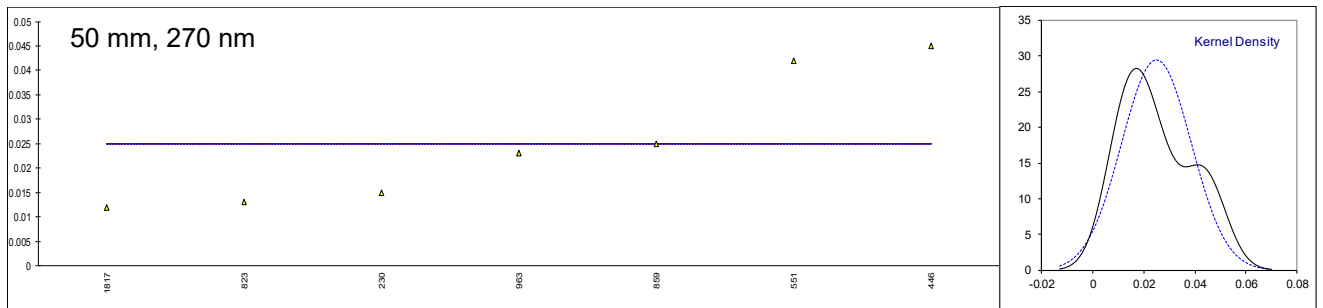
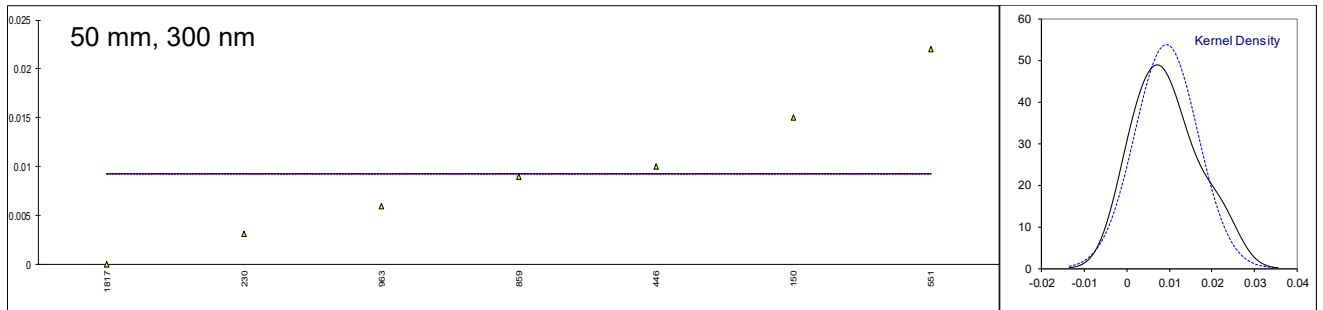
Compare R(E203:16) = 0.0780 range: 660 – 960 mg/kg

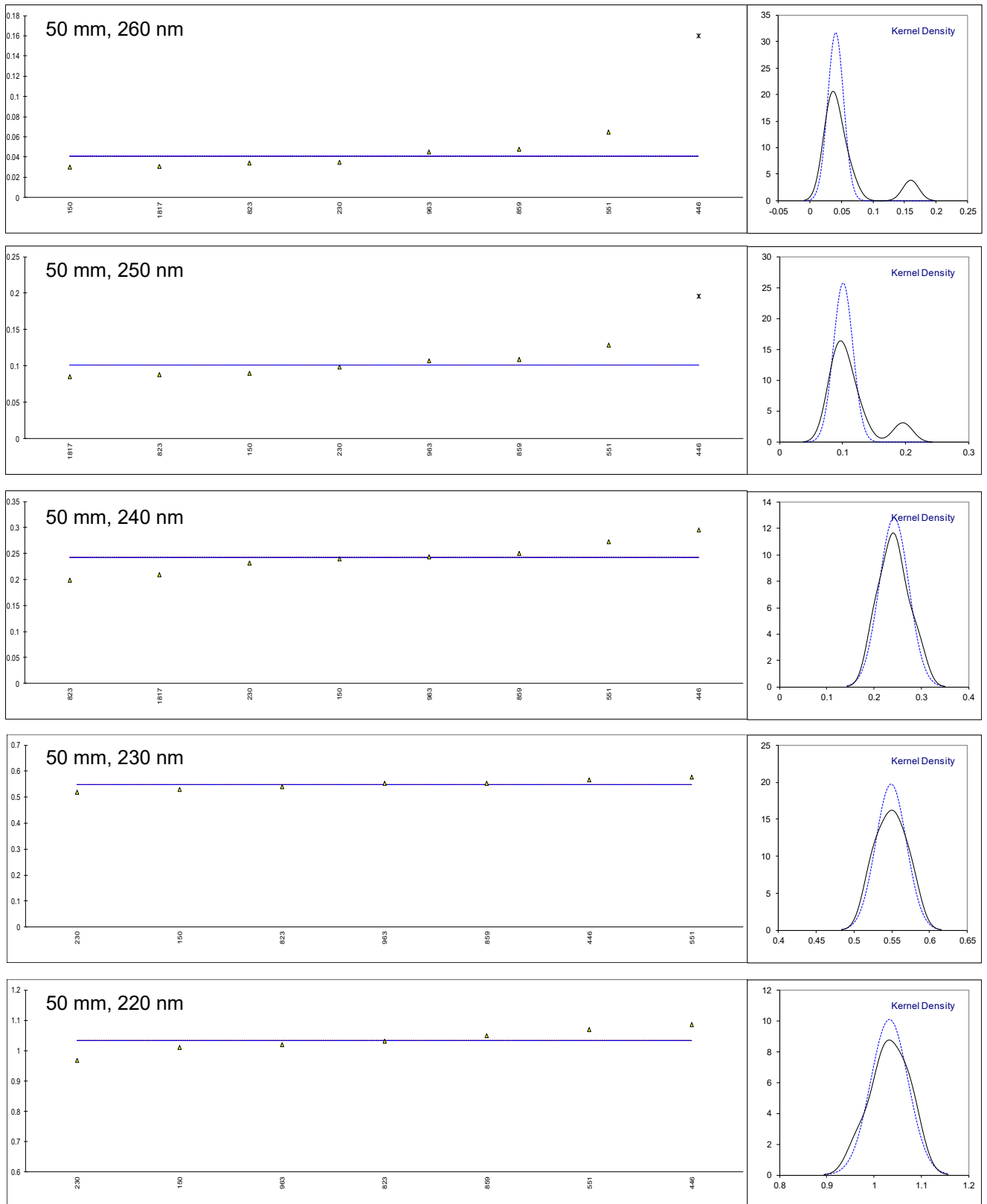


Determination of UV absorbance (50 mm cuvette) on sample #19252;

lab	method	300 nm	270 nm	260 nm	250 nm	240 nm	230 nm	220 nm	Pass/Fail
150	IMPCA004	0.015	----	0.03	0.09	0.24	0.53	1.01	Pass
230	INH-13	0.0031	0.0150	0.0346	0.0985	0.2320	0.5201	0.9669	Pass
273		----	----	----	----	----	----	----	----
311		----	----	----	----	----	----	----	----
323		----	----	----	----	----	----	----	----
329		----	----	----	----	----	----	----	----
357		----	----	----	----	----	----	----	----
446	INH-CM	0.010	0.045	0.160	0.196	0.295	0.567	1.085	Pass
541		----	----	----	----	----	----	----	----
551	OGC 3063	0.022	0.042	0.065	0.129	0.272	0.578	1.069	----
823	IMPCA004	<0.001	0.013	0.034	0.088	0.199	0.541 C	1.030 C	Pass
859	IMPCA004	0.009	0.025	0.048	0.109	0.250	0.553	1.050	Pass
912		----	----	----	----	----	----	----	----
913		----	----	----	----	----	----	----	----
922		----	----	----	----	----	----	----	----
963	IMPCA004	0.006	0.023	0.045	0.107	0.244	0.552	1.019	Pass
1205		----	----	----	----	----	----	----	----
1242		----	----	----	----	----	----	----	----
1574		----	----	----	----	----	----	----	----
1605		----	----	----	----	----	----	----	----
1726		----	----	----	----	----	----	----	----
1727		----	----	----	----	----	----	----	----
1817		0	0.012	0.031	0.085	0.209	----	----	----
1835		----	----	----	----	----	----	----	----
1927		----	----	----	----	----	----	----	----
6224		----	----	----	----	----	----	----	----
	normality	unknown	unknown	unknown	unknown	unknown	unknown	unknown	
	n	7	7	7	7	8	7	7	6
	outliers	0	0	1	1	0	0	0	
	mean (n)	0.0093	0.025	0.0411	0.1009	0.2426	0.5487	1.0328	Pass
	st.dev. (n)	0.00742	0.01358	0.01260	0.01547	0.03120	0.02021	0.03956	
	R(calc.)	0.0208	0.0380	0.0353	0.0433	0.0874	0.0566	0.1108	

The reported test results underlined and bold are statistical outliers
 Lab 823 first reported 0.302 and 0.752 respectively

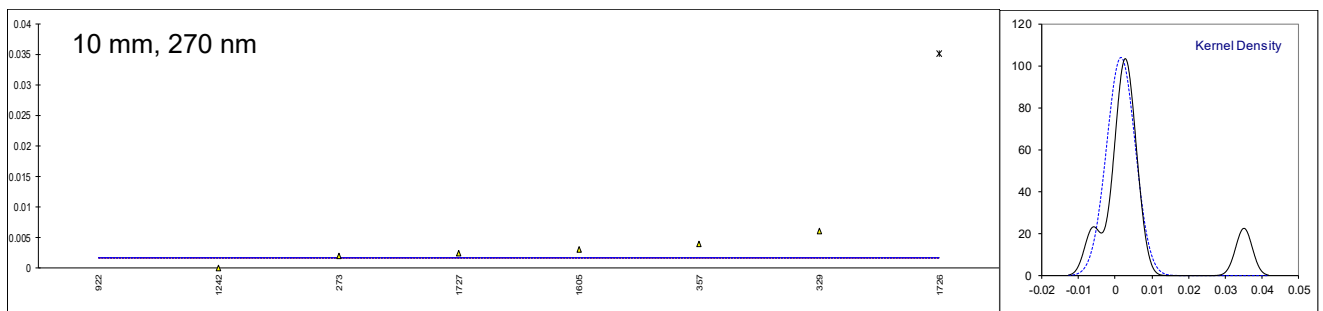
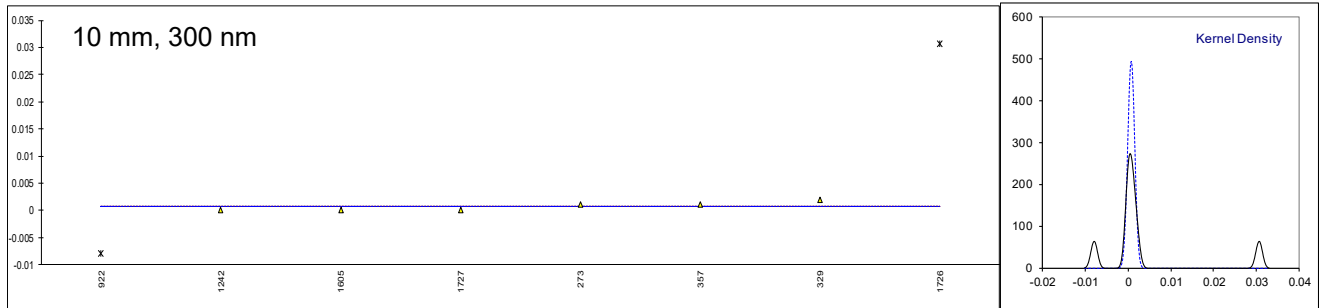


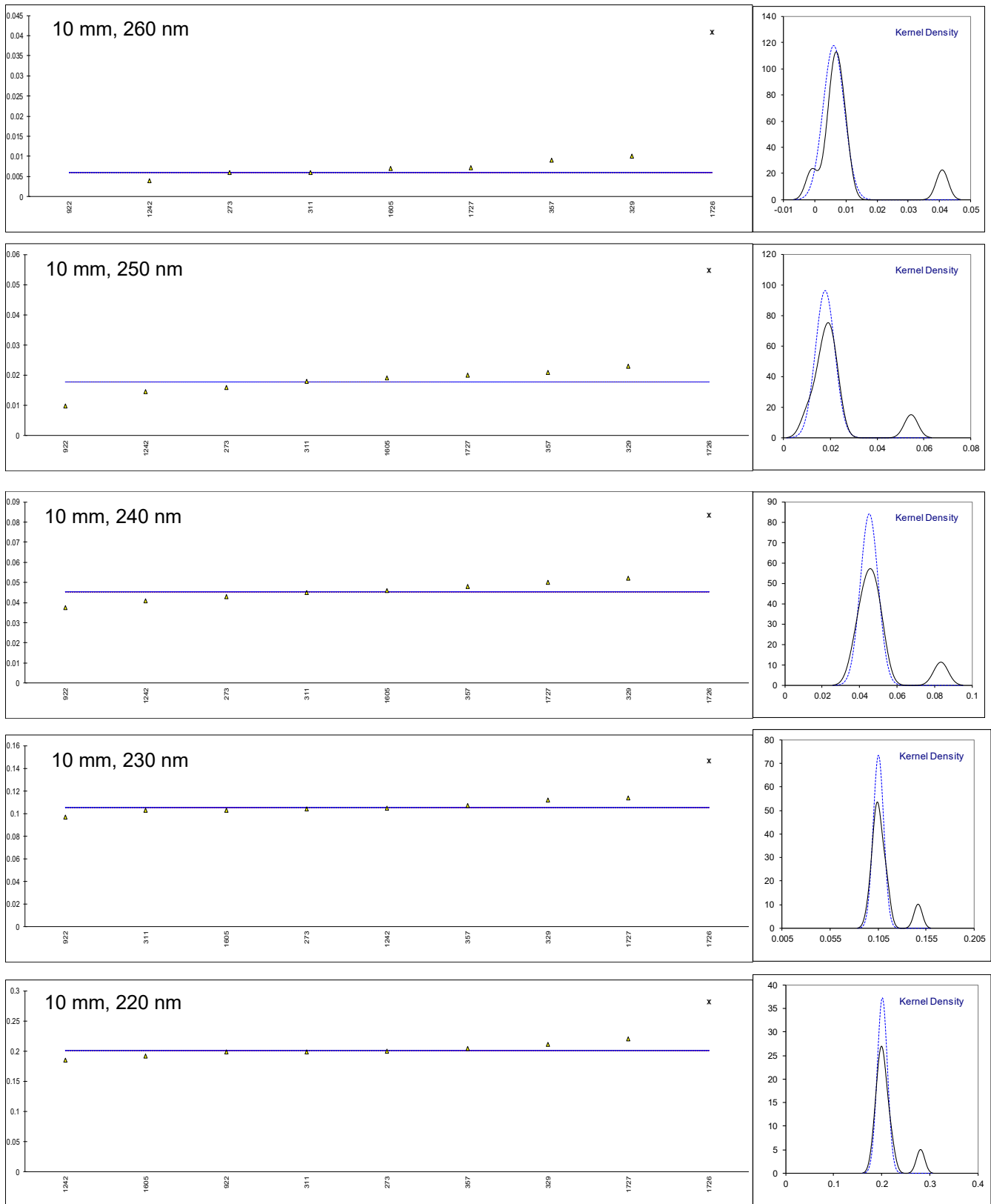


Determination of UV absorbance (10 mm cuvette) on sample #19252;

lab	method	300 nm	270 nm	260 nm	250 nm	240 nm	230 nm	220 nm	Pass/Fail
150		----	----	----	----	----	----	----	----
230		----	----	----	----	----	----	----	----
273	IMPCA004	0.001	0.002	0.006	0.016	0.043	0.104	0.200	Pass
311	INH-094	<0.005	<0.005	0.006	0.018	0.045	0.103	0.199	Pass
323		----	----	----	----	----	----	----	Pass
329	INH-CM	0.002	0.006	0.010	0.023	0.052	0.112	0.211	Pass
357	INH-13	0.001	0.004	0.009	0.021	0.048	0.107	0.204	Pass
446		----	----	----	----	----	----	----	----
541		----	----	----	----	----	----	----	----
551		----	----	----	----	----	----	----	----
823		----	----	----	----	----	----	----	----
859		----	----	----	----	----	----	----	----
912		----	----	----	----	----	----	----	----
913		----	----	----	----	----	----	----	----
922	INH-13	-0.008	-0.006	-0.001	0.0099	0.0376	0.0969	0.1985	Pass
963		----	----	----	----	----	----	----	----
1205		----	----	----	----	----	----	----	----
1242	In house	0.0000	0.0000	0.0040	0.0145	0.0410	0.1045	0.1855	Pass
1574		----	----	----	----	----	----	----	----
1605	In house	0.000	0.003	0.007	0.019	0.046	0.103	0.192	Pass
1726	In house	0.03061	0.03516	0.040915	0.054595	0.083454	0.14643	0.28155	Pass
1727	IMPCA004	<u>0.0000439</u>	<u>0.0025</u>	<u>0.0072</u>	<u>0.02</u>	<u>0.05</u>	<u>0.114</u>	<u>0.22</u>	Pass
1817		----	----	----	----	----	----	----	----
1835		----	----	----	----	----	----	----	----
1927		----	----	----	----	----	----	----	----
6224		----	----	----	----	----	----	----	----
	normality	unknown	unknown	not OK	OK	OK	OK	OK	
	n	7	7	8	8	8	8	8	10
	outliers	1	1	1	1	1	1	1	
	mean (n)	-0.0006	0.0016	0.0060	0.0177	0.0453	0.1056	0.2012	Pass
	st.dev. (n)	0.00336	0.00384	0.00339	0.00414	0.00475	0.00543	0.01070	
	R(calc.)	0.0094	0.0107	0.0095	0.0116	0.0133	0.0152	0.0300	

The reported test results underlined and bold are statistical outliers

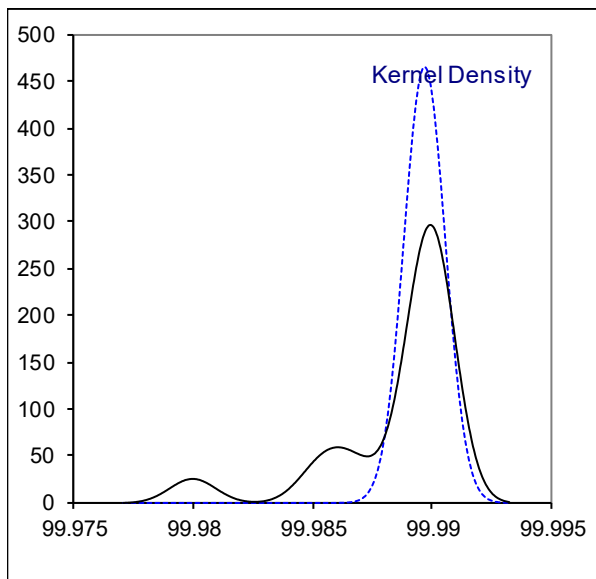
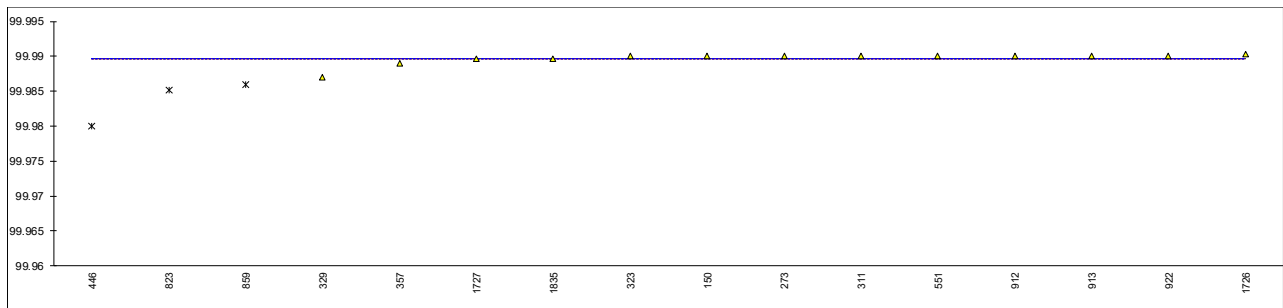




Determination of Purity of Ethanol on dry basis on sample #19253; results in %M/M

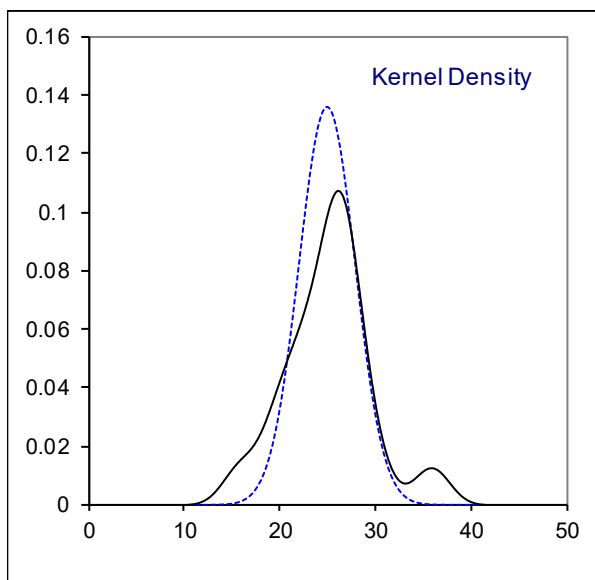
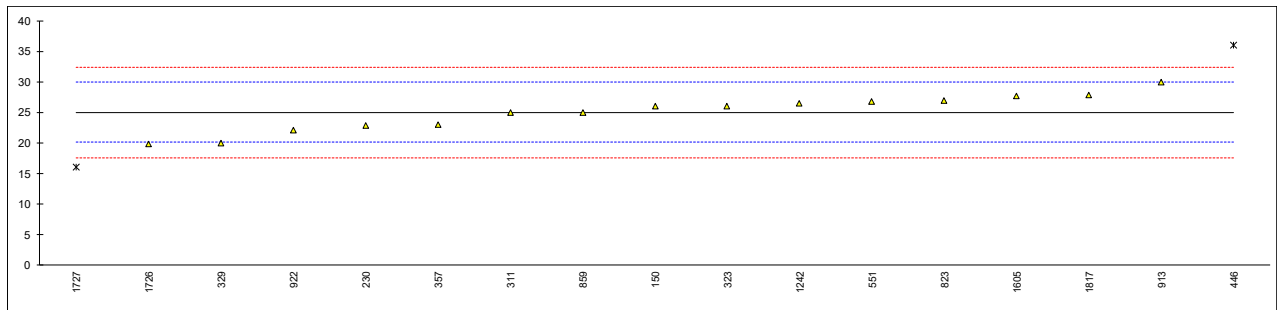
lab	method	value	mark	z(targ)	remarks
150	INH-0002	99.99		----	
230		----		----	
273	INH-0001	99.99		----	
311	INH-933	99.99		----	
323	INH-001	99.99		----	
329	INH-EtOH	99.987		----	
357	INH-02	99.989		----	
446	INH-EtOH	99.98	G(0.01)	----	
541		----		----	
551	OGC 1313	99.99		----	
823	INH-0002	99.9852	DG(0.01)	----	
859	GB/T394.2	99.986	DG(0.01)	----	
912	INH-EtOH	99.99		----	
913	INH-0002	99.99		----	
922	INH-02	99.99		----	
963		----		----	
1205		----		----	
1242		----		----	
1574		----		----	
1605		----		----	
1726	In house	99.9903		----	
1727		99.9896		----	
1817		----		----	
1835	In house	99.9897		----	
1927		----		----	
6224		----		----	
	normality	not OK			
	n	13			
	outliers	3			
	mean (n)	99.9897			
	st.dev. (n)	0.00086			
	R(calc.)	0.0024			
	st.dev.(lit)	n.a.			
	R(lit)	n.a.			

compare R(iis18C11) = 0.0076 or R(iis17C16) = 0.0089



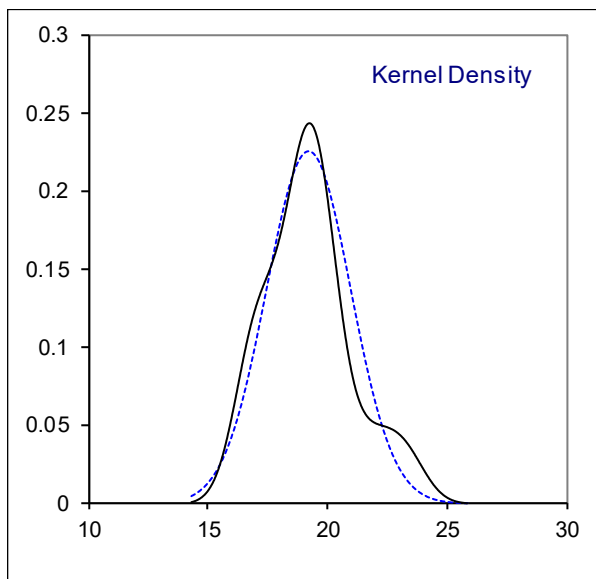
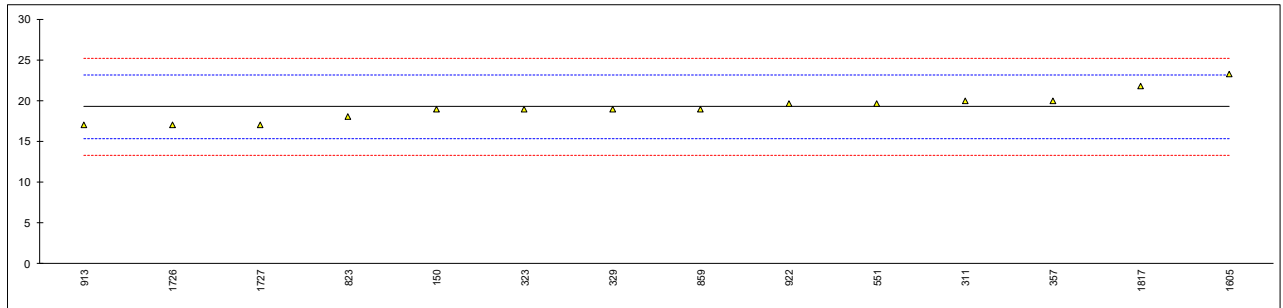
Determination of Methanol on sample #19253; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	INH-0002	26		0.39	
230	INH-0001	22.82		-0.90	
273		----		----	
311	INH-933	25		-0.01	
323	INH-001	26		0.39	
329	INH-EtOH	20		-2.04	
357	INH-02	23		-0.82	
446	INH-EtOH	36	D(0.01)	4.45	
541		----		----	
551	OGC 1313	26.82		0.73	
823	INH-0002	27		0.80	
859	GB/T394.2	25		-0.01	
912		----		----	
913	INH-0002	30		2.01	
922	INH-02	22.1		-1.19	
963		----		----	
1205		----		----	
1242		26.4054		0.56	
1574		----		----	
1605	In house	27.7		1.08	
1726	In house	19.8		-2.12	
1727		16	C,D(0.01)	-3.66	first reported 13
1817	In house	27.8129		1.13	
1835	In house	<25		----	
1927		----		----	
6224		----		----	
normality		OK			
n		15			
outliers		2			
mean (n)		25.031			
st.dev. (n)		2.9360			
R(calc.)		8.221			
st.dev.(Horwitz)		2.4666			
R(Horwitz)		6.906			



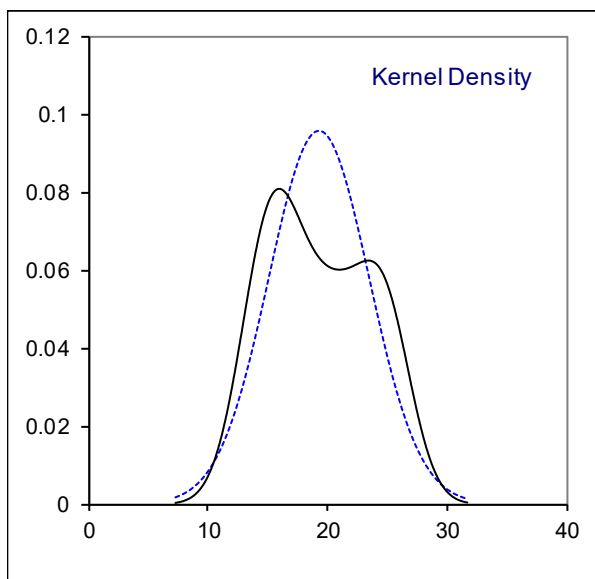
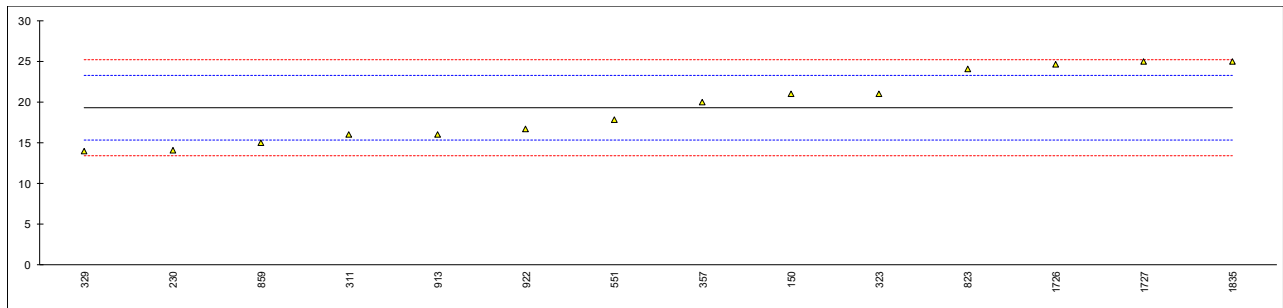
Determination of Acetal (1,1-diethoxyethane) on sample #19253; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	INH-0002	19		-0.12	
230		----		----	
273		----		----	
311	INH-933	20		0.39	
323	INH-001	19		-0.12	
329	INH-EtOH	19		-0.12	
357	INH-02	20		0.39	
446		----		----	
541		----		----	
551	OGC 1313	19.65		0.21	
823	INH-0002	18	C	-0.63	first reported 26
859	GB/T394.2	19		-0.12	
912		----		----	
913	INH-0002	17	C	-1.13	first reported <5
922	INH-02	19.6		0.19	
963		----		----	
1205		----		----	
1242		----		----	
1574		----		----	
1605	In house	23.2		2.01	
1726	In house	17.0		-1.13	
1727		17		-1.13	
1817	In house	21.8162		1.31	
1835	In house	<25		----	
1927		----		----	
6224		----		----	
normality		OK			
n		14			
outliers		0			
mean (n)		19.233			
st.dev. (n)		1.7670			
R(calc.)		4.948			
st.dev.(Horwitz)		1.9720			
R(Horwitz)		5.522			



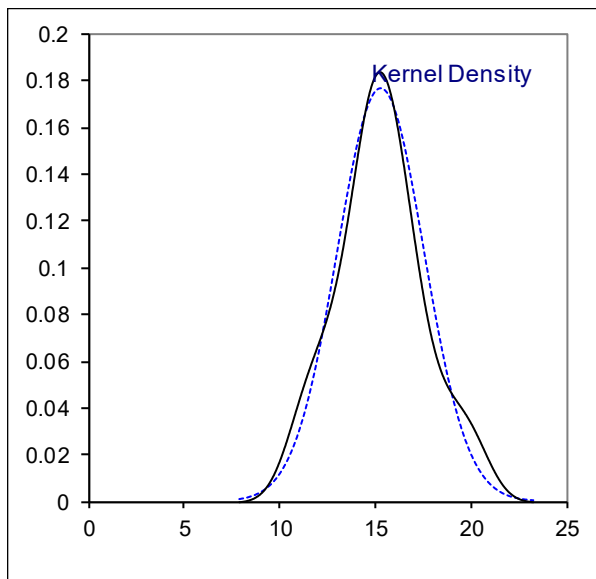
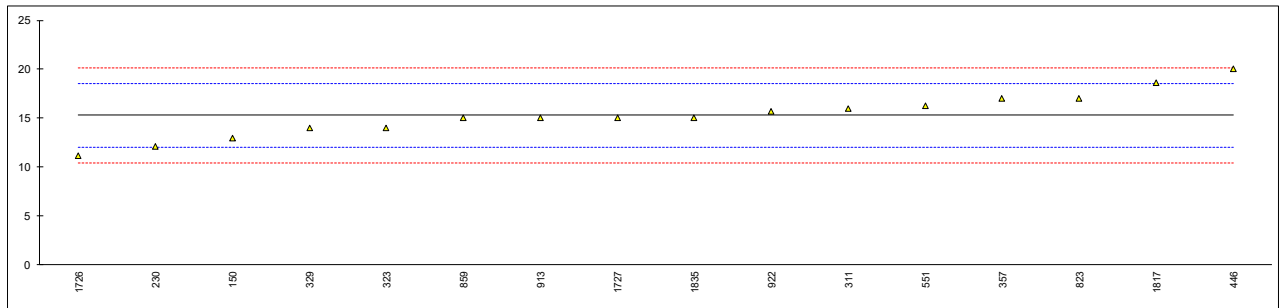
Determination of Acetone on sample #19253; results in mg/kg

lab	method	value	mark	z(target)	remarks
150	INH-0002	21		0.86	
230	INH-0001	14.09		-2.63	
273		----		----	
311	INH-933	16		-1.67	
323	INH-001	21		0.86	
329	INH-EtOH	14		-2.68	
357	INH-02	20		0.35	
446		----		----	
541		----		----	
551	OGC 1313	17.83		-0.74	
823	INH-0002	24		2.38	
859	GB/T394.2	15		-2.17	
912		----		----	
913	INH-0002	16		-1.67	
922	INH-02	16.7		-1.32	
963		----		----	
1205		----		----	
1242		----		----	
1574		----		----	
1605		----		----	
1726	In house	24.6		2.68	
1727		25		2.88	
1817		----		----	
1835	In house	25		2.88	
1927		----		----	
6224		----		----	
normality		OK			
n		14			
outliers		0			
mean (n)		19.301			
st.dev. (n)		4.1681			
R(calc.)		11.671			
st.dev.(Horwitz)		1.9779			
R(Horwitz)		5.538			



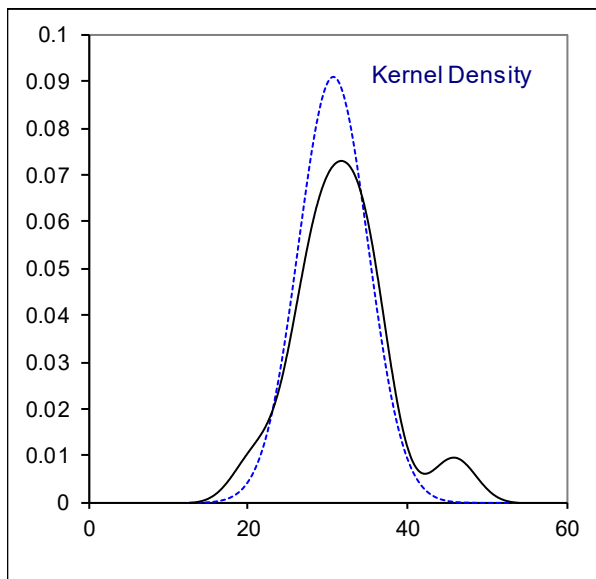
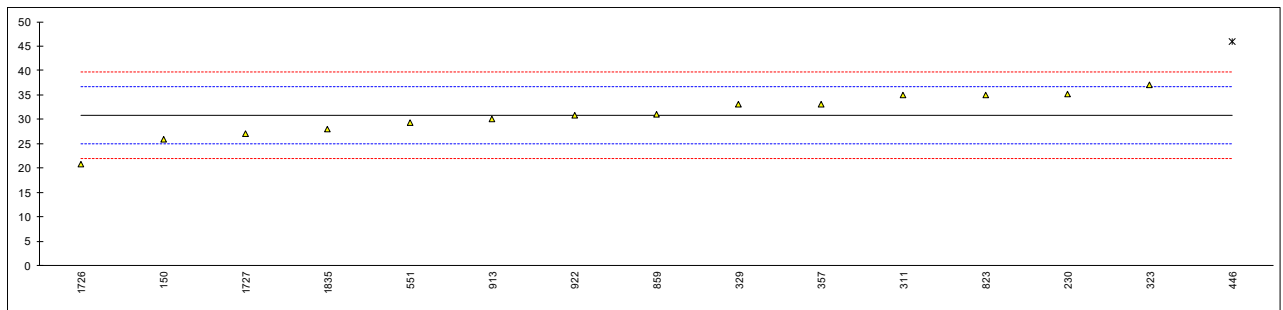
Determination of Benzene on sample #19253; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	INH-0002	13		-1.42	
230	INH-0001	12.10		-1.97	
273		----		----	
311	INH-933	16		0.43	
323	INH-001	14		-0.80	
329	INH-EtOH	14		-0.80	
357	INH-02	17		1.05	
446	INH-EtOH	20		2.89	
541		----		----	
551	OGC 1313	16.24		0.58	
823	INH-0002	17		1.05	
859	GB/T394.2	15		-0.19	
912		----		----	
913	INH-0002	15	C	-0.19	first reported 7
922	INH-02	15.7		0.25	
963		----		----	
1205		----		----	
1242		----		----	
1574		----		----	
1605		----		----	
1726	In house	11.2		-2.53	
1727		15		-0.19	
1817	In house	18.5924		2.03	
1835	In house	15		-0.19	
1927		----		----	
6224		----		----	
normality	OK				
n	16				
outliers	0				
mean (n)	15.302				
st.dev. (n)	2.2557				
R(calc.)	6.316				
st.dev.(Horwitz)	1.6239				
R(Horwitz)	4.547				



Determination of Isopropanol on sample #19253; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	INH-0002	26		-1.63	
230	INH-0001	35.19		1.49	
273		----		----	
311	INH-933	35		1.43	
323	INH-001	37		2.11	
329	INH-EtOH	33		0.75	
357	INH-02	33		0.75	
446	INH-EtOH	46	D(0.05)	5.16	
541		----		----	
551	OGC 1313	29.28		-0.52	
823	INH-0002	35		1.43	
859	GB/T394.2	31		0.07	
912		----		----	
913	INH-0002	30		-0.27	
922	INH-02	30.9		0.03	
963		----		----	
1205		----		----	
1242		----		----	
1574		----		----	
1605		----		----	
1726	In house	20.9		-3.37	
1727		27		-1.29	
1817		----		----	
1835	In house	28		-0.95	
1927		----		----	
6224		----		----	
normality		OK			
n		14			
outliers		1			
mean (n)		30.805			
st.dev. (n)		4.3765			
R(calc.)		12.254			
st.dev.(Horwitz)		2.9423			
R(Horwitz)		8.238			

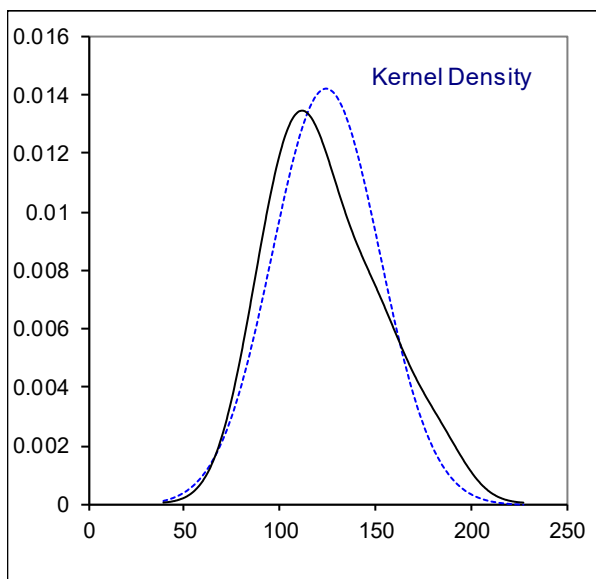
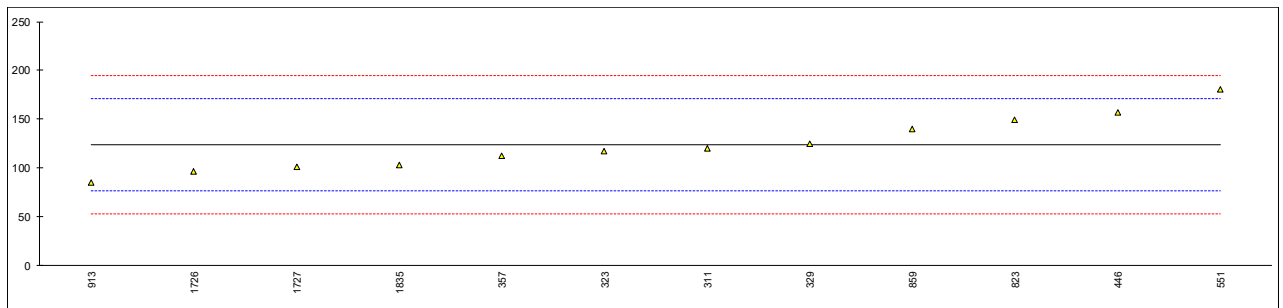


Determination of Monoethylene glycol (MEG) on sample #19253; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150		----		----	
230		----		----	
273		----		----	
311		----		----	
323		----		----	
329	INH-EtOH	25		----	
357	INH-02	< 30		----	
446		----		----	
541		----		----	
551	OGC 1313	26.46		----	
823	INH-0002	<15		----	
859	GB/T394.2	33		----	
912		----		----	
913	INH-0002	<10		----	
922	INH-02	15.0		----	
963		----		----	
1205		----		----	
1242		----		----	
1574		----		----	
1605		----		----	
1726		----		----	
1727		----		----	
1817		----		----	
1835		----		----	
1927		----		----	
6224		----		----	
	normality	unknown			
	n	4			
	outliers	0			
	mean (n)	24.865			
	st.dev. (n)	7.4400			
	R(calc.)	20.832			
	st.dev.(lit)	n.a.			
	R(lit)	n.a.			

Determination of Total impurities on sample #19253; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150		----		----	
230		----		----	
273		----		----	
311	INH-933	120		-0.17	
323	INH-001	117		-0.30	
329	INH-EtOH	125		0.04	
357	INH-02	113		-0.47	
446	INH-EtOH	157		1.40	
541		----		----	
551	OGC 1313	180.98		2.42	
823	INH-0002	149		1.06	
859	GB/T394.2	140		0.68	
912		----		----	
913	INH-0002	85		-1.66	
922		----		----	
963		----		----	
1205		----		----	
1242		----		----	
1574		----		----	
1605		----		----	
1726	In house	96.7		-1.16	
1727		101		-0.98	
1817		----		----	
1835	In house	103		-0.89	
1927		----		----	
6224		----		----	
normality		OK			
n		12			
outliers		0			
mean (n)		123.973			
st.dev. (n)		28.0260			
R(calc.)		78.473			
st.dev.(Horwitz, 6 components)		23.5205			
R(Horwitz, 6 components)		65.857			



APPENDIX 2

Other reported impurities in sample #19253; results in mg/kg

lab	Acetaldehyde	Other impurities
150	4	----
230	16.87	----
273	----	----
311	<5	<5
323	<5	----
329	<5	<5
357	< 5	< 5
446	----	----
541	----	----
551	1.72	42.98
823	9	----
859	<5	<5
912	----	----
913	<5	<5
922	2.1	----
963	----	----
1205	----	----
1242	----	----
1574	----	----
1605	1.77	----
1726	1.2	2
1727	<10	2
1817	ND	< 300
1835	<10	<25
1927	----	----
6224	----	----

Lab 823 first reported 16

APPENDIX 3

Number of participants per country

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

APPENDIX 4

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 of participant
ex	= test result excluded from statistical evaluation
fr.	= first reported
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
SDS	= Safety Data Sheet

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