



Institute for  
Interlaboratory Studies

## Results of Proficiency Test Ethanol (Food / Neutral grade) December 2022

**Organized by:** Institute for Interlaboratory Studies  
Spijkenisse, the Netherlands

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## 1 INTRODUCTION

Since 2007 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Ethanol (Food / Neutral grade) every year. During the annual proficiency testing program 2022/2023 it was decided to continue the round robin for the analysis of Ethanol (Food / Neutral grade).

In this interlaboratory study 26 laboratories in 15 countries registered for participation, see appendix 3 for the number of participants per country. In this report the results of the Ethanol (Food / Neutral grade) proficiency test are presented and discussed. This report is also electronically available through the iis website [www.iisnl.com](http://www.iisnl.com).

## 2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes 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 0.5 L labelled #22250 for regular analyzes and one bottle of 250 mL labelled #22251 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 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](http://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

For the preparation of the sample for the regular analyzes a batch of approximately 40 liters of Ethanol (Food / Neutral grade) was obtained from a local supplier. After homogenization 45 amber glass bottles of 0.5 L were filled and labelled #22250.

The homogeneity of the subsamples was checked by determination of Density at 20 °C in accordance with ASTM D4052 on 8 stratified randomly selected subsamples.

	Density at 20 °C in kg/L
sample #22250-1	0.80581
sample #22250-2	0.80583
sample #22250-3	0.80582
sample #22250-4	0.80582
sample #22250-5	0.80583
sample #22250-6	0.80585
sample #22250-7	0.80586
sample #22250-8	0.80581

Table 1: homogeneity test results of subsamples #22250

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

	Density at 20 °C in kg/L
r (observed)	0.00005
reference test method	ISO12185:96
0.3 x R (reference test method)	0.00015

Table 2: evaluation of the repeatability of subsamples #22250

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

For the preparation of the GC sample a batch of approximately 13 liters Ethanol (Food / Neutral grade) was separated from the batch for the main round and spiked with Methanol, Acetone, Benzene and Mono Ethylene glycol (MEG). After homogenization 45 amber glass bottles of 250 mL were filled and labelled #22251.

The homogeneity of the subsamples was checked by determination of Acetone and Isopropanol in accordance with an in house method on 8 stratified randomly selected subsamples.

	Acetone in mg/kg	Isopropanol in mg/kg
sample #22251-1	9.4	7.9
sample #22251-2	9.1	7.5
sample #22251-3	9.9	8.1
sample #22251-4	9.1	7.3
sample #22251-5	9.1	7.4
sample #22251-6	9.9	7.5
sample #22251-7	9.3	7.8
sample #22251-8	9.3	7.3

Table 3: homogeneity test results of subsamples #22251

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

	Acetone in mg/kg	Isopropanol in mg/kg
r (observed)	0.9	0.8
reference method	Horwitz	Horwitz
0.3 x R (reference method)	0.9	0.8

Table 4: evaluation of the repeatabilities of subsamples #22251

The calculated repeatabilities are in agreement with 0.3 times the estimated reproducibilities calculated with the Horwitz equation. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample Ethanol (Food / Neutral grade) labelled #22250 and one sample Ethanol (Food / Neutral grade) for GC only labelled #22251 was sent on November 9, 2022. 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 ANALYZES

The participants were requested to determine on sample #22250: Appearance, Density at 20 °C, Nonvolatile matter, Permanganate Time Test at 20 °C, pH<sub>e</sub> (with LiCl and KCl electrode), 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.

On sample #22251 it was requested to determine: Purity of Ethanol on dry basis, Methanol, Acetal (1,1-diethoxyethane), Acetaldehyde, Acetone, Benzene, Isopropanol, Mono Ethylene 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 reference test methods (when applicable) 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/](http://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](http://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/](http://www.kpmd.co.uk/sgs-iis/). The reported test results are tabulated per determination in appendices 1 and 2 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 reanalyses). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the 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.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

### 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 (derived from e.g. ISO or ASTM test methods), 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, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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. Therefore, the usual interpretation of z-scores is as follows:

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

## 4 EVALUATION

In this proficiency test some problems were encountered with the dispatch of the samples. Therefore, the reporting time on the data entry portal was extended with two weeks. When considering the test results of the two samples together two participants reported test results after the extended reporting date and two other participants did not report any test result. Not all participants were able to perform all analyzes requested.

In total 24 participants reported 276 numerical test results. Observed were 18 outlying test results, which is 6.5%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Not all 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 were 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 in appendix 1. 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 these tests the calculated reproducibility was compared against the estimated reproducibility calculated with the Horwitz equation.



In the iis PT reports ASTM test methods are referred to with a number (e.g. D1363) and an added designation for the year that the test method was adopted or revised (e.g. D1363:06). When a method has been reapproved an “R” will be added and the year of approval (e.g. D1363:06R19).

**sample #22250**

Appearance: This determination was not problematic. All reporting participants agreed on a test result as Pass (Clear & Bright).

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. Almost all reporting participants agreed on a test result of <1 mg/100mL. Therefore, no z-scores were calculated.

Permanganate Time Test at 20 °C: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D1363:06R19.

It is known that the pHe determined with a LiCl electrode will be lower than the pHe determined with a KCl electrode. Therefore, the test results are requested separately. Test method EN15490 describes the use of a LiCl electrode and test method ASTM D6423 describes the use of a KCl electrode.

pHe with LiCl electrode: This determination may be problematic (only four test results were reported). No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of EN15490:07.

pHe with KCl electrode: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D6423:20a.

Strength (%M/M): This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the reproducibility derived from the OIML table.

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.

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation but is not in agreement with the strict requirements of ASTM E203:16.

Regretfully, no reference test method for the determination of UV absorbance 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. Therefore, the use of a 50 mm cuvette is advised. In this PT six laboratories used a 50 mm cuvette and eight laboratories used a 10 mm cuvette. Both groups were evaluated separately.

UV - 50 mm cuvette: This determination may not be problematic. No statistical outliers were observed over all seven parameters. Five participants evaluated the sample as 'Pass'.

UV - 10 mm cuvette: This determination may not be problematic. Seven statistical outliers were observed (all from the same participant) over all seven parameters. Five participants evaluated the sample as 'Pass' while one other participant evaluated the sample as 'Fail'.

### **sample #22251**

Purity of Ethanol on dry basis: This determination may not be problematic. Two statistical outliers were observed. Regretfully, no reference test method is available that provides precision data for the determination of purity in Ethanol (Food / Neutral grade). Therefore, no z-scores could be calculated.

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

Acetone: 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 calculated with the Horwitz equation.

Benzene: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation.

Isopropanol: This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

Total impurities: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation for 7 components.

For impurities not listed above, but mentioned in paragraph 2.6, the participants agree on a concentration near or below the limit of detection. Therefore, these impurities were not further evaluated. The reported test results are given in appendix 2.

## 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility ( $2.8 \times$  standard deviation) and the target reproducibility derived from reference methods are presented in the next tables.

Parameter	unit	n	average	2.8 * sd	R(lit)
Appearance		19	Pass (C&B)	n.a.	n.a.
Density at 20 °C	kg/L	22	0.8059	0.0002	0.0005
Nonvolatile matter	mg/100mL	12	<1	n.e.	n.e.
Permanganate Time Test 20 °C	minutes	9	34.4	15.8	8.7
pHe with LiCl electrode		4	7.7	1.3	0.7
pHe with KCl electrode		7	8.0	0.4	1.0
Strength	%M/M	16	94.39	0.07	0.06
Strength	%V/V	18	96.38	0.03	0.06
Water	%M/M	15	5.59	0.22	0.48
UV – 50 mm cuvette:					
UV-absorbance 300 nm		5	0.008	0.016	n.a.
UV-absorbance 270 nm		6	0.036	0.021	n.a.
UV-absorbance 260 nm		6	0.056	0.017	n.a.
UV-absorbance 250 nm		6	0.116	0.026	n.a.
UV-absorbance 240 nm		6	0.249	0.029	n.a.
UV-absorbance 230 nm		6	0.553	0.038	n.a.
UV-absorbance 220 nm		6	1.145	0.102	n.a.
Conclusion UV-scan		5	Pass	n.a.	n.a.
UV – 10 mm cuvette:					
UV-absorbance 300 nm		7	0.0004	0.0055	n.a.
UV-absorbance 270 nm		7	0.004	0.011	n.a.
UV-absorbance 260 nm		7	0.008	0.014	n.a.
UV-absorbance 250 nm		7	0.019	0.011	n.a.
UV-absorbance 240 nm		7	0.045	0.012	n.a.
UV-absorbance 230 nm		7	0.105	0.013	n.a.
UV-absorbance 220 nm		7	0.226	0.021	n.a.
Conclusion UV-scan		5	Pass	n.a.	n.a.

Table 5: reproducibilities of tests on sample #22250

Parameter	unit	n	average	2.8 *sd	R(target)
Purity of Ethanol on dry basis	%M/M	14	99.99	0.010	n.a.
Methanol	mg/kg	12	25.6	9.2	7.0
Acetone	mg/kg	12	12.7	6.4	3.9
Benzene	mg/kg	12	13.6	3.5	4.1

Parameter	unit	n	average	2.8 *sd	R(target)
Isopropanol	mg/kg	11	8.8	3.5	2.8
Total impurities	mg/kg	10	66.4	32.3	41.9

Table 6: reproducibilities of tests on sample #22251

Without further statistical calculations it can be concluded that for many tests there is not a good compliance of the group of participants with the reference test methods. The problematic tests have been discussed in paragraph 4.1.

#### 4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2022 WITH PREVIOUS PTS

	December 2022	December 2021	December 2020	December 2019	December 2018
Number of reporting laboratories	24	27	24	25	25
Number of test results	276	311	315	337	303
Number of statistical outliers	18	8	19	19	20
Percentage of statistical outliers	6.5%	2.6%	6.0%	5.6%	6.6%

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 to the requirements of the reference test methods. The conclusions are given in the following table.

Parameter	December 2022	December 2021	December 2020	December 2019	December 2018
Density at 20 °C	++	++	++	++	++
Nonvolatile matter	n.e.	n.e.	n.e.	-	++
Permanganate Time Test 20 °C	-	-	-	+	-
pHe with LiCl electrode	-	+/-	-	-	++
pHe with KCl electrode	++	-	-	n.e.	n.e.
Strength %M/M	-	+	-	+	++
Strength %V/V	++	++	+	+/-	+
Water	++	++	+	+	-
Methanol	-	--	-	-	--
Acetal (1,1-diethoxyethane)	n.e.	n.e.	n.e.	+	n.e.
Acetaldehyde	n.e.	n.e.	n.e.	n.a.	n.a.
Acetone	-	-	-	+	+/-
Benzene	+	+/-	-	-	--
Isopropanol	-	-	-	-	+
Mono Ethylene glycol (MEG)	n.e.	n.e.	(--)	n.e.	n.e.
Other impurities	n.a.	(--)	(--)	n.a.	n.a.
Total impurities	+	--	+	-	-

Table 8: comparison determinations to the reference test methods

Results between brackets no z-scores are calculated.

The following performance categories were used:

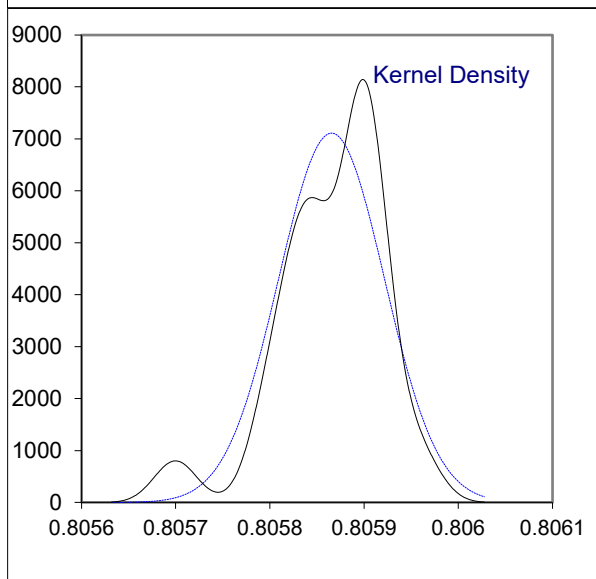
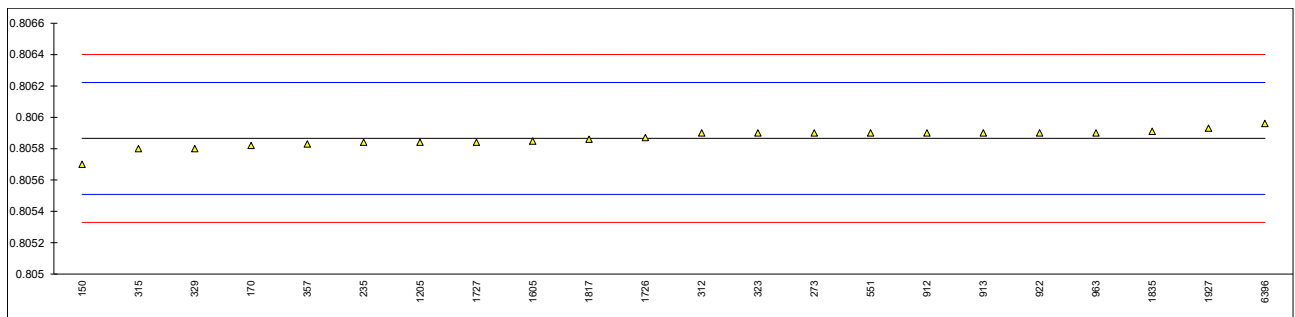
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- : group performed worse than the reference test method
- : group performed much worse than the reference test method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

**APPENDIX 1****Determination of Appearance on sample #22250;**

lab	method	value	mark	z(targ)	remarks
150	Visual	C & B		----	
170	Visual	Clear and Bright		----	
235	Visual	C&B		----	
273	Visual	Pass		----	
312	Visual	CLCOL		----	
315	E2680	pass		----	
323	Visual	clear and bright		----	
329	Visual	clear & bright		----	
357	Visual	CFSM		----	
446		----		----	
551	Visual	Pass		----	
912	E2680	Pass		----	
913	E2680	Clear&Bright		----	
922	Visual	Clear & Bright		----	
963	Visual	Pass		----	
1205		----		----	
1574		----		----	
1605		----		----	
1726	Visual	Clear&Colourless		----	
1727	Visual	Clear&Colorless		----	
1817	Visual	Pass		----	
1835	Visual	C&C		----	
1927		----		----	
6214		----		----	
6224		----		----	
6396	Visual	Clear + Bright		----	
	n	19			
	mean (n)	Clear & Bright (Pass)			

Determination of Density at 20 °C on sample #22250; results in kg/L

lab	method	value	mark	z(targ)	remarks
150	D4052	0.8057		-0.93	
170	D4052	0.80582		-0.26	
235	ISO12185	0.80584		-0.14	
273	D4052	0.8059		0.19	
312	ISO12185	0.8059		0.19	
315	D4052	0.8058		-0.37	
323	D4052	0.8059		0.19	
329	D4052	0.8058		-0.37	
357	D4052	0.80583		-0.20	
446		----		----	
551	D4052	0.8059		0.19	
912	D4052	0.8059		0.19	
913	D4052	0.8059		0.19	
922	D4052	0.8059		0.19	
963	D4052	0.8059		0.19	
1205	In house	0.805840		-0.14	
1574		----		----	
1605	D4052	0.805848		-0.10	
1726	D4052	0.80587		0.02	
1727	D4052	0.80584		-0.14	
1817	Table OIML	0.80586		-0.03	
1835	ISO12185	0.80591		0.25	
1927	D4052	0.80593		0.36	
6214		----		----	
6224		----		----	
6396	D4052	0.80596		0.53	
normality		not OK			
n		22			
outliers		0			
mean (n)		0.805866			
st.dev. (n)		0.0000561			
R(calc.)		0.000157			
st.dev.(ISO12185:96)		0.0001786			
R(ISO12185:96)		0.0005			



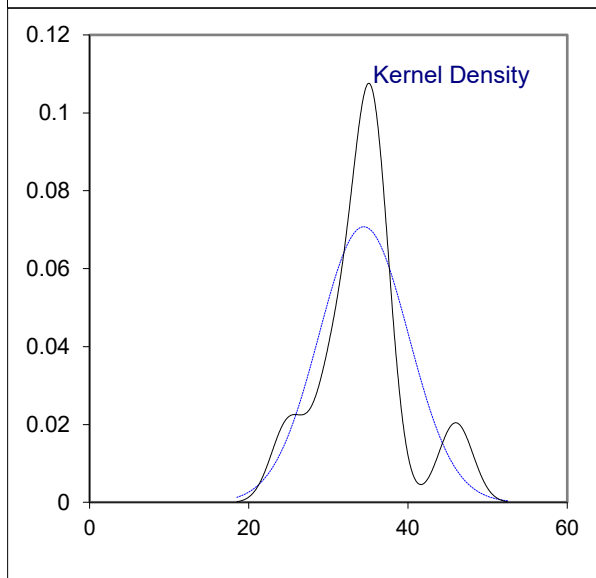
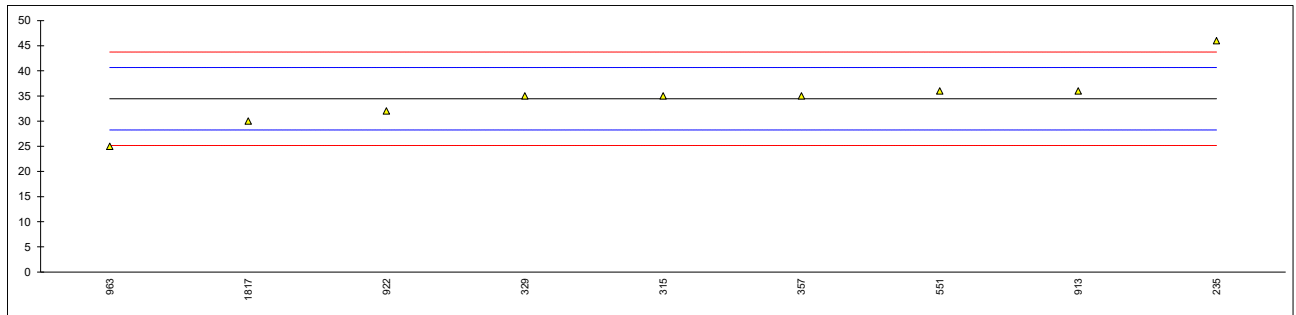
## Determination of Nonvolatile matter on sample #22250; results in mg/100 mL

lab	method	value	mark	z(targ)	remarks
150	D1353	0.00		----	
170	D1353	0.2		----	
235	D1353	0.8		----	
273		----		----	
312		----		----	
315	D1353	<1		----	
323	D1353	<1		----	
329	D1353	0.2		----	
357	D1353	< 1		----	
446		----		----	
551	D1353	0.4		----	
912	D1353	0.2		----	
913	D1353	0.3		----	
922	D1353	<1.0		----	
963		----		----	
1205		----		----	
1574		----		----	
1605		----		----	
1726	EN15691	<10		----	
1727	EN15691	nd		----	
1817	In house	0		----	
1835	EN15691	<10		----	
1927		----		----	
6214		----		----	
6224		----		----	
6396		----		----	
	n	12			
	outliers	<1			



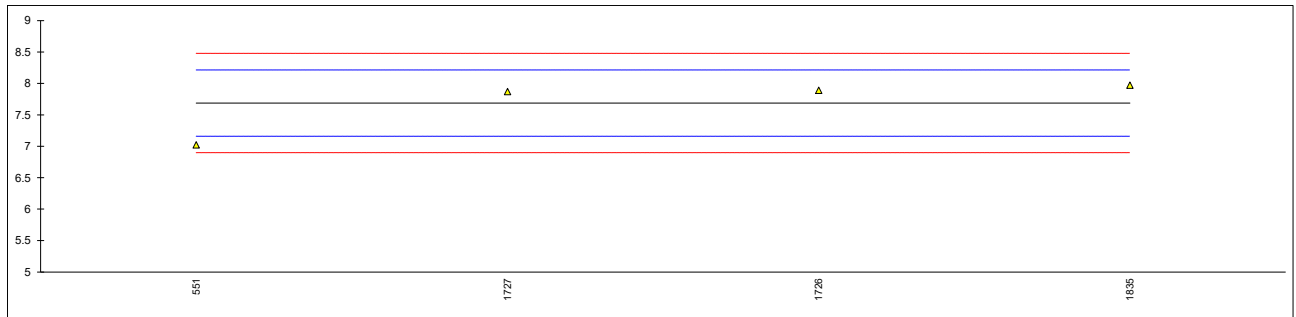
Determination of Permanganate Time Test at 20 °C on sample #22250; results in minutes

lab	method	value	mark	z(targ)	remarks
150		----		----	
170		----		----	
235	D1363	46		3.73	
273		----		----	
312		----		----	
315	D1363	35		0.18	
323	D1363	>30		----	
329	D1363	35		0.18	
357	D1363	35		0.18	
446		----		----	
551	D1363	36		0.50	
912		----		----	
913	D1363	36		0.50	
922	D1363	32		-0.79	
963	D1363	25		-3.05	
1205		----		----	
1574		----		----	
1605		----		----	
1726		----		----	
1727		----		----	
1817	In house	30		-1.43	
1835		----		----	
1927		----		----	
6214		----		----	
6224		----		----	
6396		----		----	
normality		not OK			
n		9			
outliers		0			
mean (n)		34.44			
st.dev. (n)		5.637			
R(calc.)		15.78			
st.dev.(D1363:06R19)		3.100			
R(D1363:06R19)		8.68			



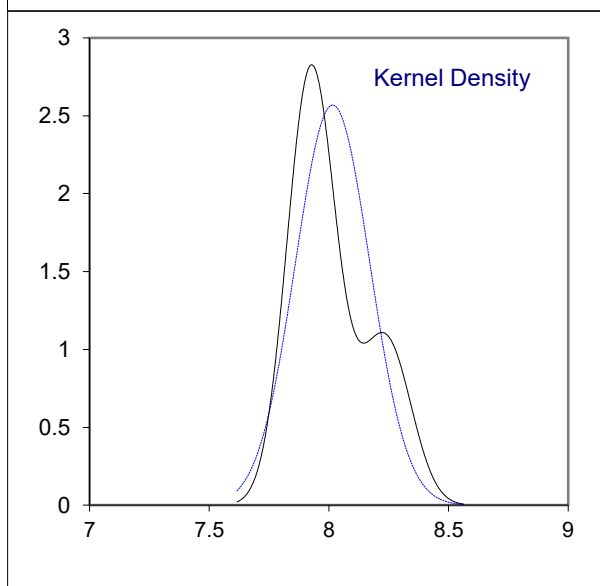
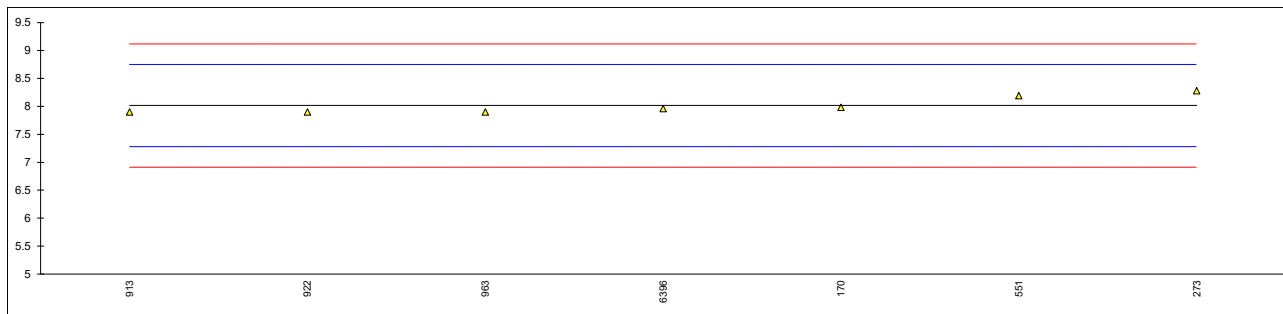
Determination of pHe with LiCl electrode on sample #22250;

lab	method	value	mark	z(targ)	remarks
150		----		----	
170		----		----	
235		----		----	
273		----		----	
312		----		----	
315		----		----	
323		----		----	
329		----		----	
357		----		----	
446		----		----	
551	NBR10891	7.02		-2.53	
912		----		----	
913		----		----	
922		----		----	
963		----		----	
1205		----		----	
1574		----		----	
1605		----		----	
1726	EN15490	7.89		0.77	
1727	EN15490	7.87		0.69	
1817		----		----	
1835	EN15490	7.97		1.07	
1927		----		----	
6214		----		----	
6224		----		----	
6396		----		----	
normality		unknown			
n		4			
outliers		0			
mean (n)		7.687			
st.dev. (n)		0.4471			
R(calc.)		1.252			
st.dev.(EN15490:07)		0.2636			
R(EN15490:07)		0.738			



Determination of pHe with KCl electrode on sample #22250;

lab	method	value	mark	z(targ)	remarks
150		----		----	
170	D6423	7.98		-0.10	
235		----		----	
273	D6423	8.28		0.72	
312		----		----	
315		----		----	
323		----		----	
329		----		----	
357		----		----	
446		----		----	
551	D6423	8.19		0.47	
912		----		----	
913	D6423	7.9		-0.31	
922	D6423	7.9		-0.31	
963	D6423	7.9		-0.31	
1205		----		----	
1574		----		----	
1605		----		----	
1726		----		----	
1727		----		----	
1817		----		----	
1835		----		----	
1927		----		----	
6214		----		----	
6224		----		----	
6396	D6423	7.96		-0.15	
normality		unknown			
n		7			
outliers		0			
mean (n)		8.016			
st.dev. (n)		0.1553			
R(calc.)		0.435			
st.dev.(D6423:20a)		0.3677			
R(D6423:20a)		1.030			

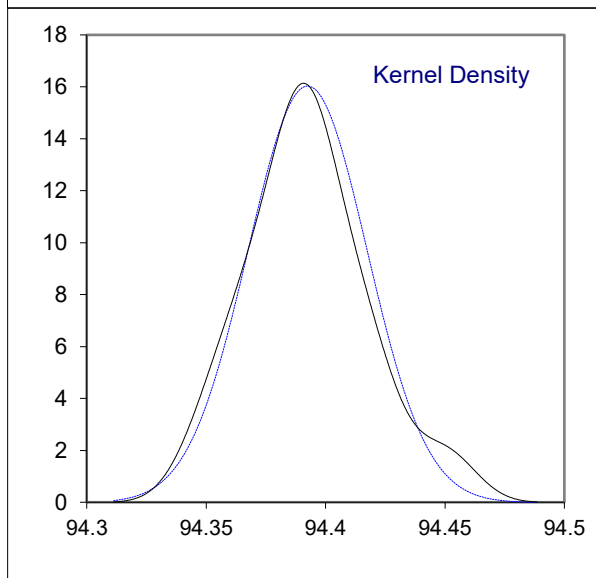
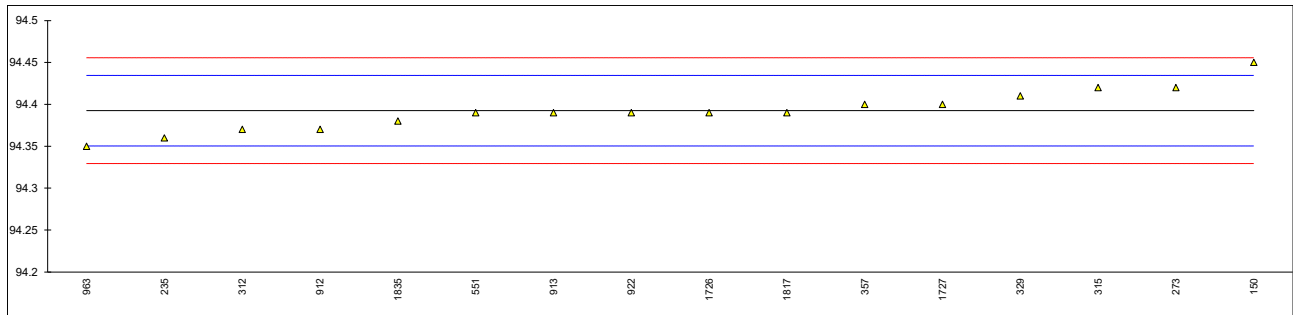


Determination of Strength on sample #22250; results in %M/M

lab	method	value	mark	z(targ)	remarks
150	Table OIML	94.45	C	2.73	first reported 78.03
170		----		----	
235	Table OIML	94.36		-1.54	
273	Table OIML	94.42	C	1.30	first reported 94.5
312	Table OIML	94.37		-1.07	
315	Table OIML	94.42		1.30	
323		----		----	
329	Table OIML	94.41		0.83	
357	Table OIML	94.40		0.36	
446		----		----	
551	Table OIML	94.39		-0.12	
912	Table OIML	94.37		-1.07	
913	Table OIML	94.39		-0.12	
922	Table OIML	94.39		-0.12	
963	Table OIML	94.35		-2.02	
1205		----		----	
1574		----		----	
1605		----		----	
1726	Table OIML	94.39		-0.12	
1727	Table OIML	94.40		0.36	
1817	Table OIML	94.39		-0.12	
1835	Table OIML	94.38		-0.59	
1927		----		----	
6214		----		----	
6224		----		----	
6396		----		----	

normality OK  
 n 16  
 outliers 0  
 mean (n) 94.393  
 st.dev. (n) 0.0249  
 R(calc.) 0.070  
 st.dev.(OIML table) 0.0211  
 R(OIML table) 0.059

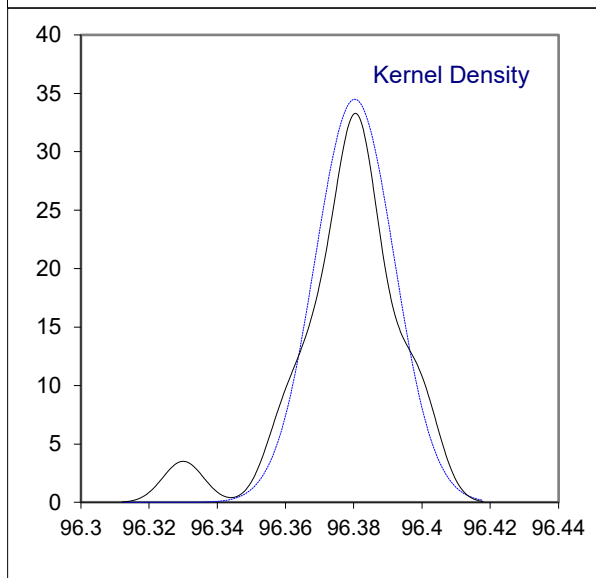
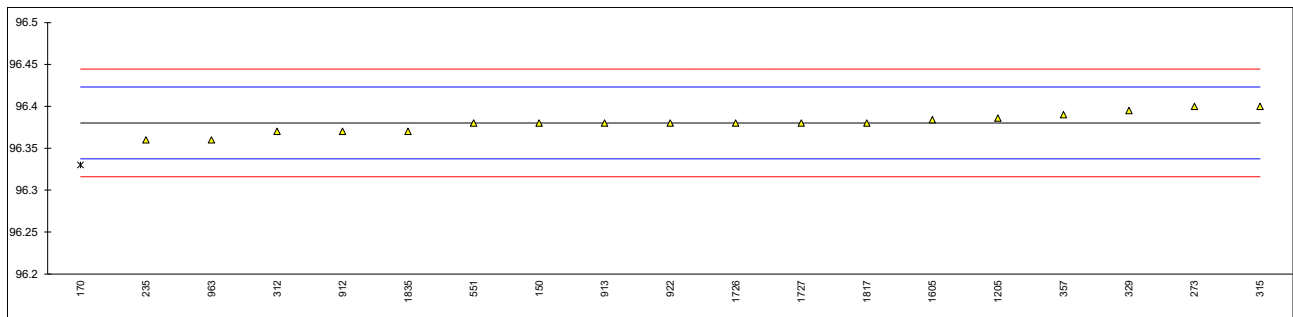
OIML R022-e75



Determination of Strength on sample #22250; results in %V/V

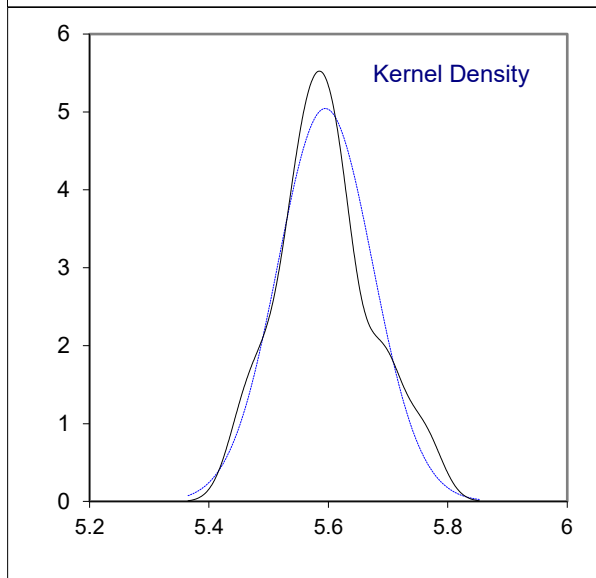
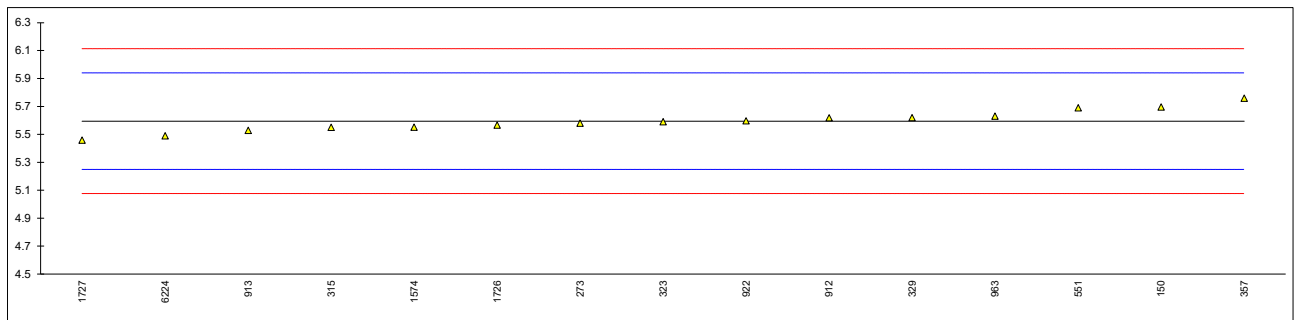
lab	method	value	mark	z(targ)	remarks
150	Table OIML	96.38		-0.01	
170	Table OIML	96.33	G(0.05)	-2.35	
235	Table OIML	96.36		-0.95	
273	Table OIML	96.4		0.92	
312	Table OIML	96.37		-0.48	
315	Table OIML	96.40		0.92	
323		----		----	
329	Table OIML	96.395		0.69	
357	Table OIML	96.39		0.45	
446		----		----	
551	Table OIML	96.38		-0.01	
912	Table OIML	96.37		-0.48	
913	Table OIML	96.38		-0.01	
922	Table OIML	96.38		-0.01	
963	Table OIML	96.36		-0.95	
1205	Table OIML	96.386		0.27	
1574		----		----	
1605	Table OIML	96.384		0.17	
1726	Table OIML	96.38		-0.01	
1727	Table OIML	96.38		-0.01	
1817	Table OIML	96.38		-0.01	
1835	Table OIML	96.37		-0.48	
1927		----		----	
6214		----		----	
6224		----		----	
6396		----		----	
normality		OK			
n		18			
outliers		1			
mean (n)		96.380			
st.dev. (n)		0.0116			
R(calc.)		0.032			
st.dev.(OIML table)		0.0214			
R(OIML table)		0.060			

OIML R022-e75



Determination of Water on sample #22250; results in %M/M

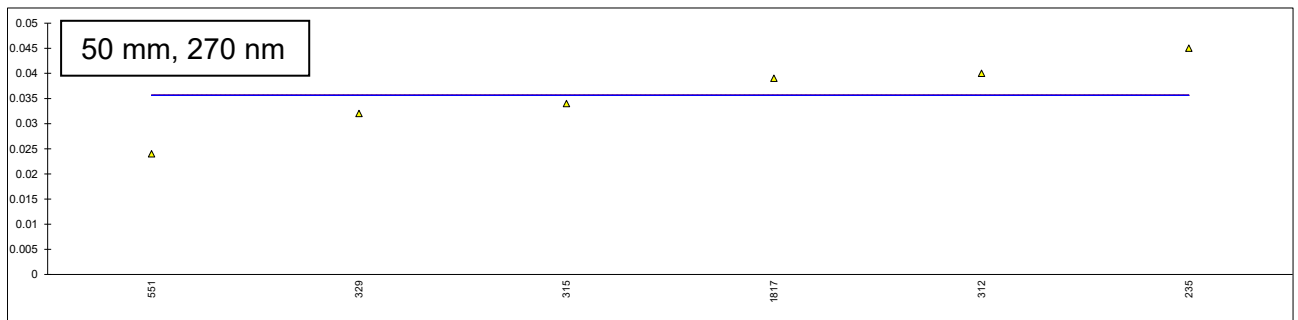
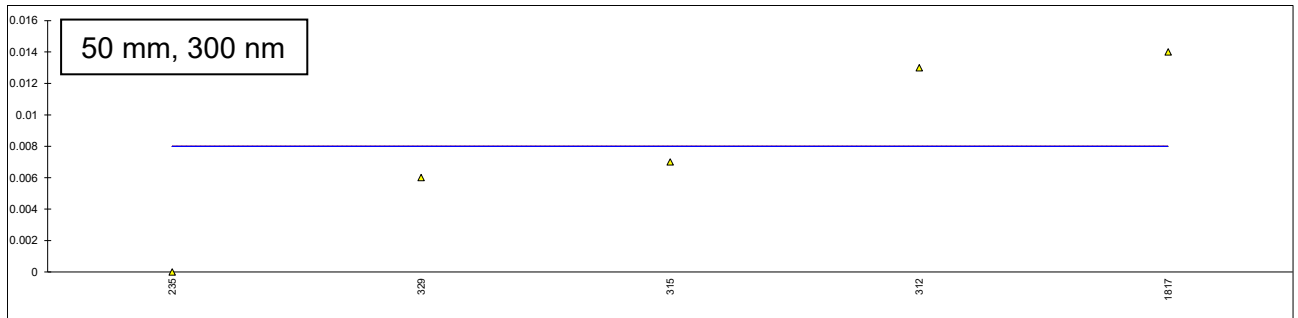
lab	method	value	mark	z(targ)	remarks
150	E203	5.696		0.59	
170		----		----	
235		----		----	
273	E203	5.58		-0.09	
312		----		----	
315	E203	5.550		-0.26	
323	E203	5.59		-0.03	
329	E203	5.6198		0.14	
357	E203	5.759		0.95	
446		----		----	
551	E203	5.690		0.55	
912	E203	5.618		0.13	
913	E203	5.528		-0.39	
922	E203	5.597		0.01	
963	E203	5.63		0.20	
1205		----		----	
1574	D1364	5.5513		-0.25	
1605		----		----	
1726	EN15691	5.5656		-0.17	
1727	EN15692	5.4587		-0.79	
1817		----		----	
1835		----		----	
1927		----		----	
6214		----		----	
6224	In house	5.49		-0.61	
6396		----		----	
normality		OK			
n		15			
outliers		0			
mean (n)		5.5949			
st.dev. (n)		0.07912			
R(calc.)		0.2215			
st.dev.(Horwitz)		0.17270			
R(Horwitz)		0.4836			
compare					
R(E203:16)		0.078			

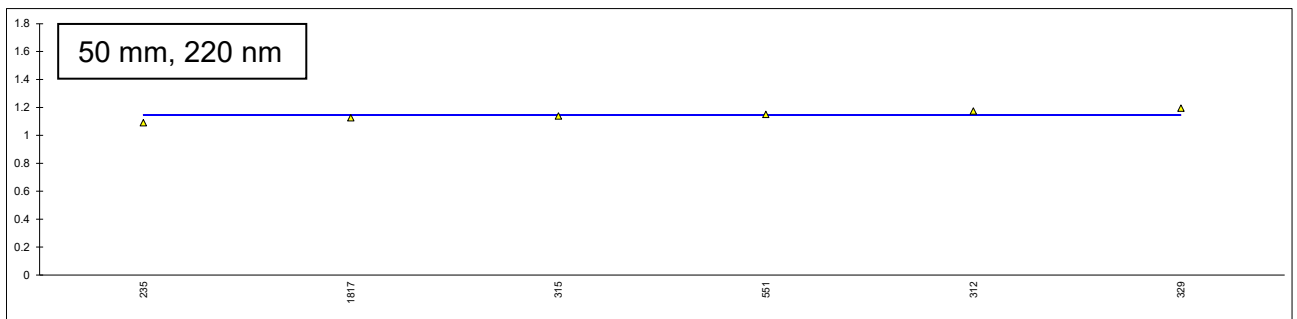
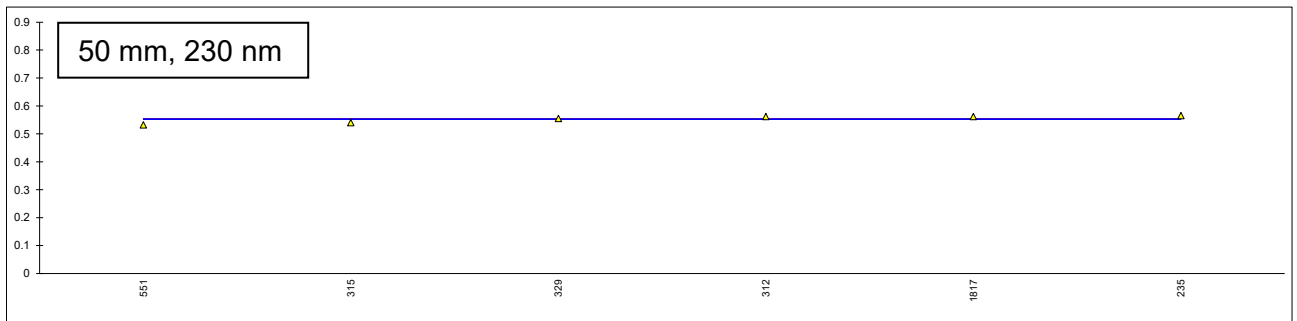
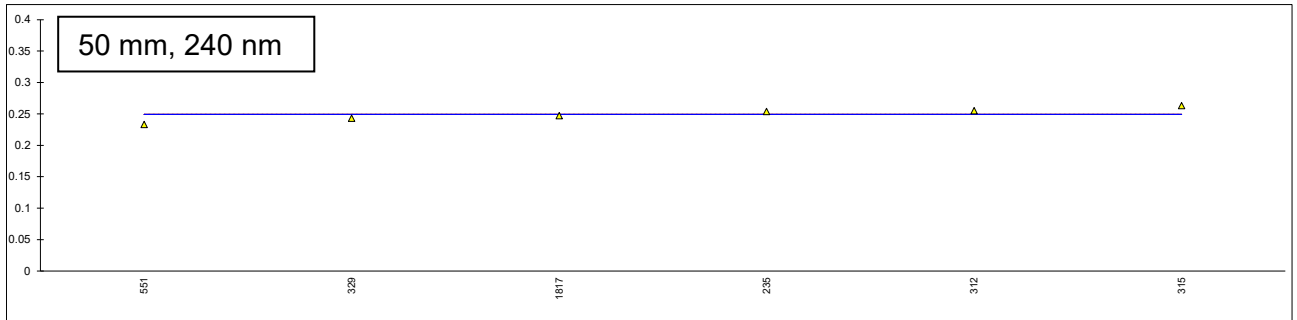
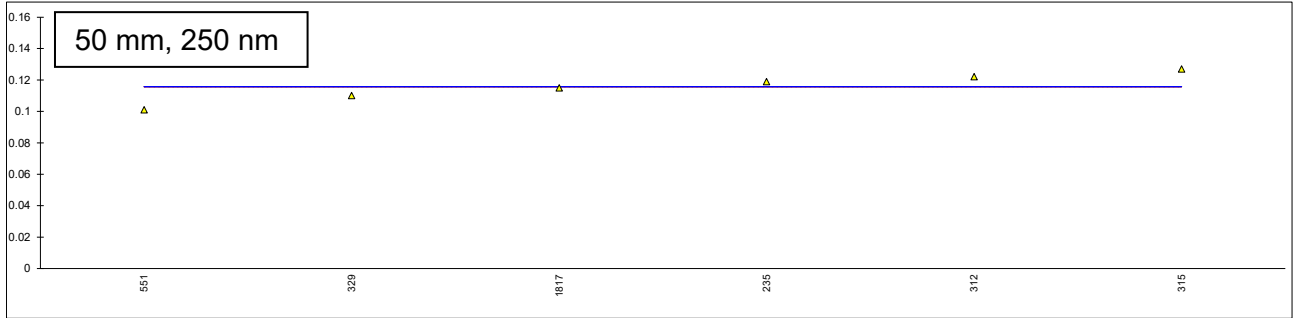
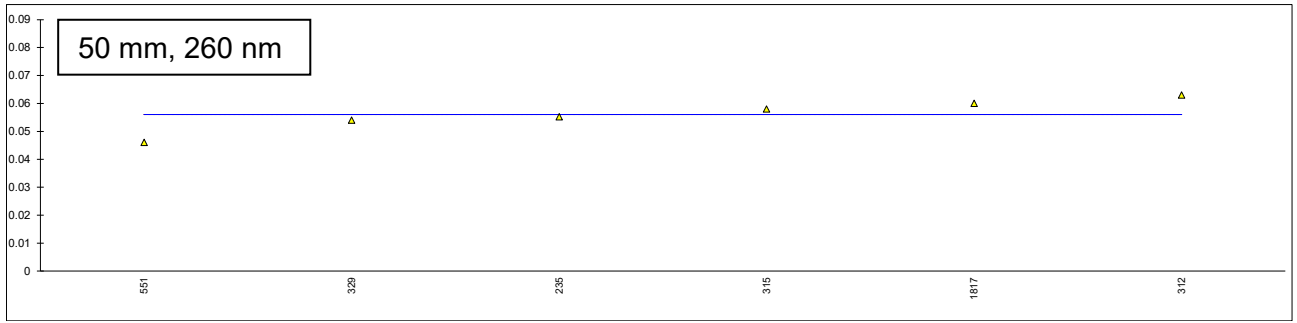


Determination of UV absorbance (50 mm cuvette) on sample #22250;

lab	method	300 nm	270 nm	260 nm	250 nm	240 nm	230 nm	220 nm	Pass/Fail
150		----	----	----	----	----	----	----	
170		----	----	----	----	----	----	----	
235	INH-13.001	0.000 C	0.0450	0.0552	0.1189	0.2539	0.5650	1.0905	Pass
273		----	----	----	----	----	----	----	
312	INH-001	0.013	0.040	0.063	0.122	0.255	0.562	1.174	Pass
315	INH-13.001	0.007	0.034	0.058	0.127	0.263	0.540	1.138	
323		----	----	----	----	----	----	----	Pass
329	INH-CM	0.006	0.032	0.054	0.110	0.243	0.555	1.194	Pass
357		----	----	----	----	----	----	----	
446		----	----	----	----	----	----	----	
551	INH-3063	<0,010	0.024	0.046	0.101	0.233	0.532	1.150	Pass
912		----	----	----	----	----	----	----	
913		----	----	----	----	----	----	----	
922		----	----	----	----	----	----	----	
963		----	----	----	----	----	----	----	
1205		----	----	----	----	----	----	----	
1574		----	----	----	----	----	----	----	
1605		----	----	----	----	----	----	----	
1726		----	----	----	----	----	----	----	
1727		----	----	----	----	----	----	----	
1817	In house	0.014	0.039	0.060	0.115	0.247	0.562	1.126	
1835		----	----	----	----	----	----	----	
1927		----	----	----	----	----	----	----	
6214		----	----	----	----	----	----	----	
6224		----	----	----	----	----	----	----	
6396		----	----	----	----	----	----	----	
normality		unknown	unknown	unknown	unknown	unknown	unknown	unknown	
n		5	6	6	6	6	6	6	5
outliers		0	0	0	0	0	0	0	
mean (n)		0.0080	0.0357	0.0560	0.1156	0.2491	0.5527	1.1454	Pass
st.dev. (n)		0.00570	0.00734	0.00589	0.00924	0.01051	0.01356	0.03646	
R(calc.)		0.0160	0.0206	0.0165	0.0259	0.0294	0.0380	0.1021	
st.dev.(lit)		unknown	unknown	unknown	unknown	unknown	unknown	unknown	
R(lit)		unknown	unknown	unknown	unknown	unknown	unknown	unknown	

Lab 235 first reported 0.0253 at 300nm

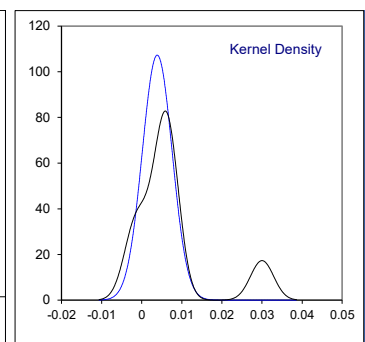
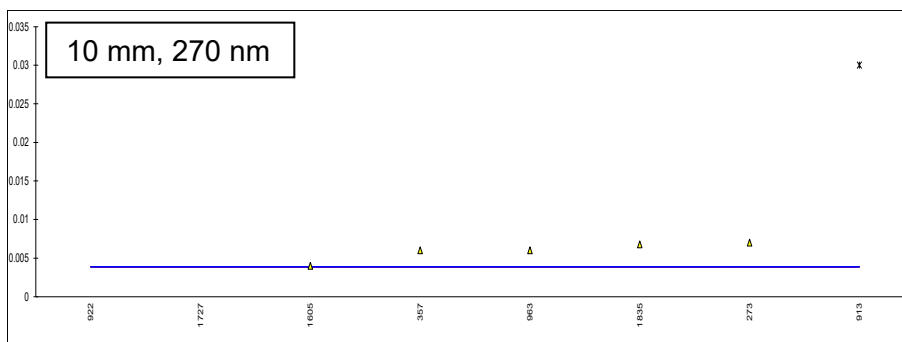
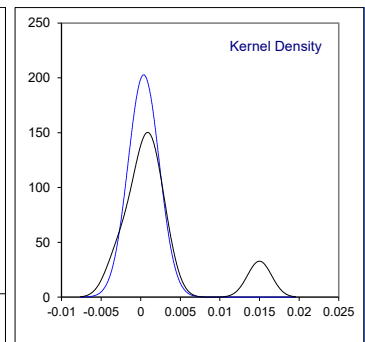
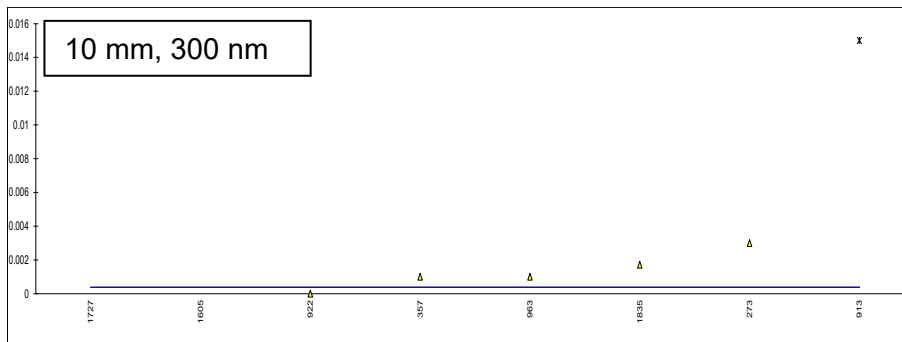


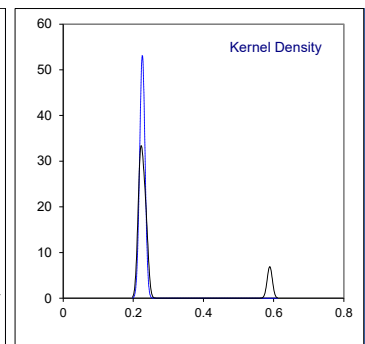
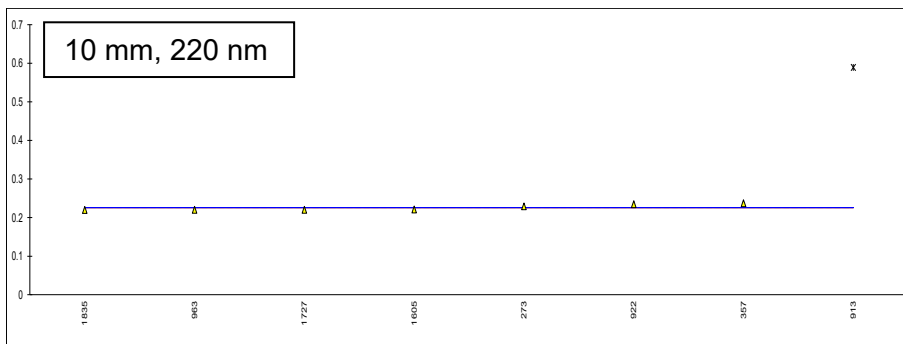
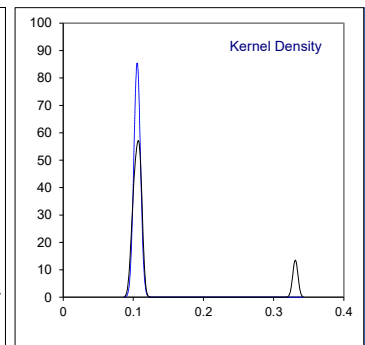
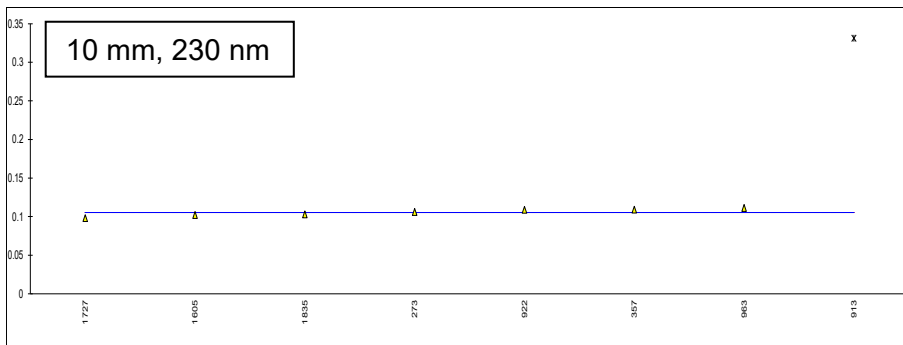
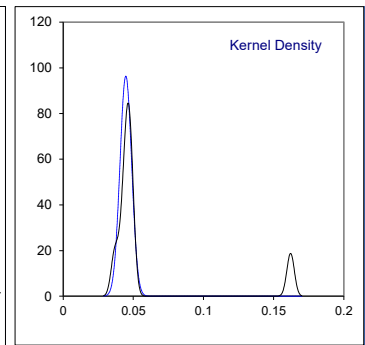
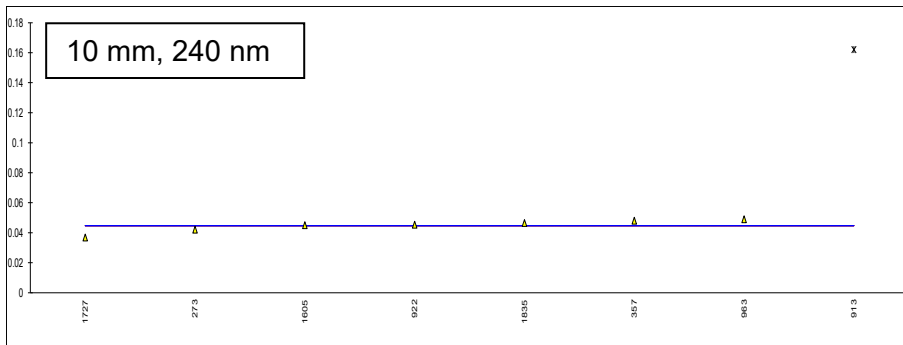
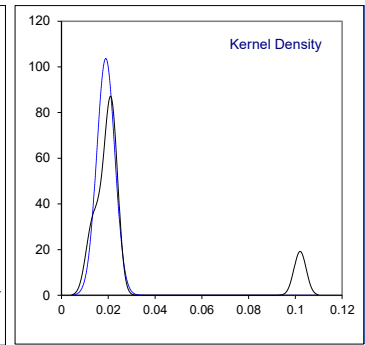
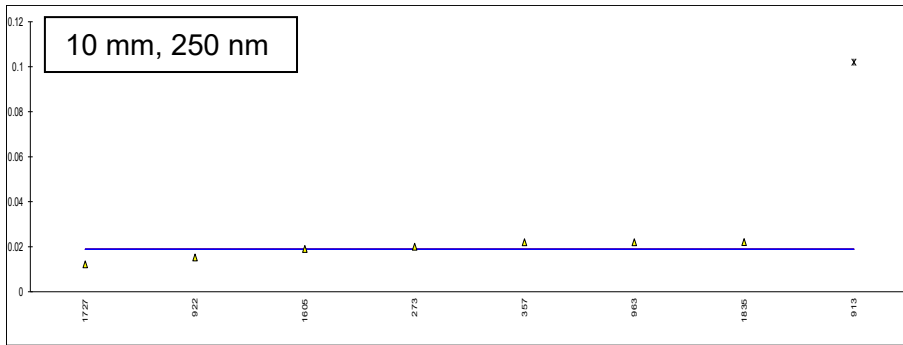
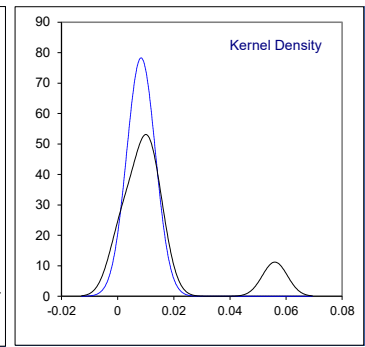
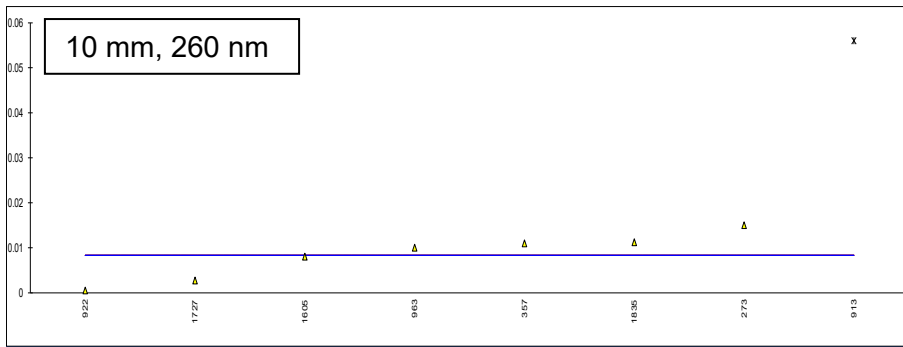




Determination of UV absorbance (10 mm cuvette) on sample #22250;

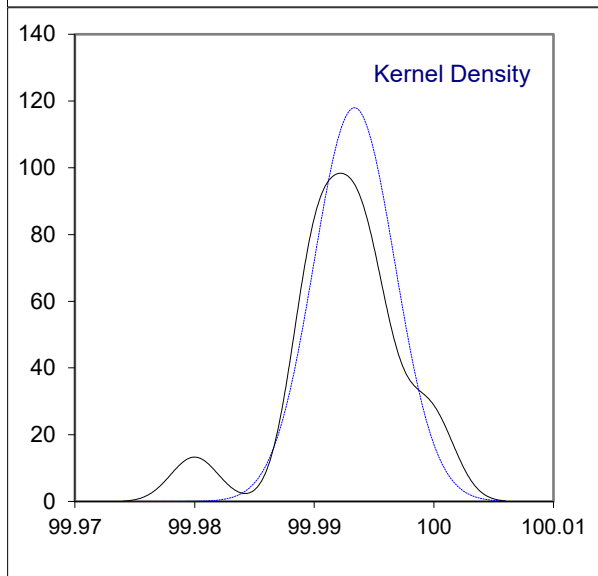
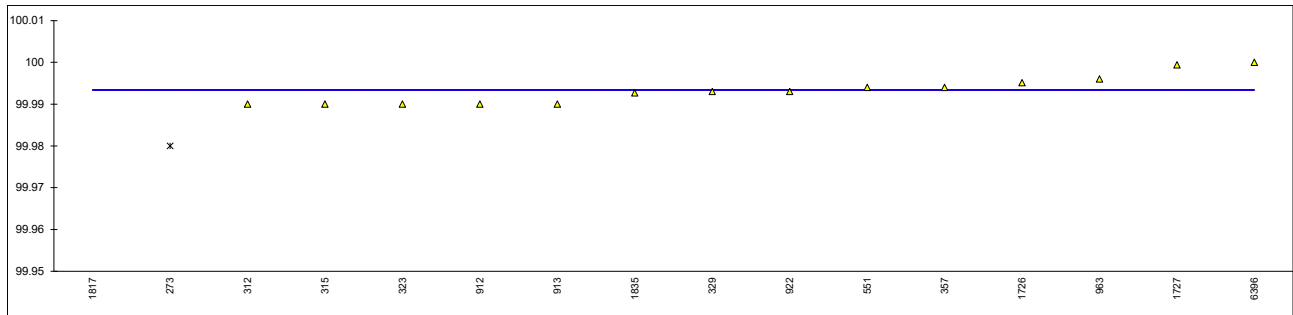
lab	method	300 nm	270 nm	260 nm	250 nm	240 nm	230 nm	220 nm	Pass/Fail
150		----	----	----	----	----	----	----	
170		----	----	----	----	----	----	----	
235		----	----	----	----	----	----	----	
273	IMPCA004	0.003	0.007	0.015	0.020	0.042	0.106	0.229	
312		----	----	----	----	----	----	----	
315		----	----	----	----	----	----	----	
323		----	----	----	----	----	----	----	
329		----	----	----	----	----	----	----	
357	INH-13-001	0.001	0.006	0.011	0.022	0.048	0.109	0.237	Pass
446		----	----	----	----	----	----	----	
551		----	----	----	----	----	----	----	
912		----	----	----	----	----	----	----	
913	IMPCA004	0.015 G1	0.03 G1	0.056 G1	0.102 G1	0.162 G1	0.331 G1	0.589 G1	Pass
922	INH-13	0.000	-0.002	0.0005	0.0152	0.0454	0.1088	0.2342	Fail
963	IMPCA004	0.001	0.006	0.010	0.022	0.049	0.111	0.220	Pass
1205		----	----	----	----	----	----	----	
1574		----	----	----	----	----	----	----	
1605		-0.001	0.004	0.008	0.019	0.045	0.102	0.221	
1726		----	----	----	----	----	----	----	
1727	IMPCA004	-0.00305	-0.000747	0.00271	0.0122	0.0368	0.098	0.22	Pass
1817		----	----	----	----	----	----	----	
1835		0.00171	0.00676	0.01125	0.02202	0.04643	0.1027	0.21945	Pass
1927		----	----	----	----	----	----	----	
6214		----	----	----	----	----	----	----	
6224		----	----	----	----	----	----	----	
6396		----	----	----	----	----	----	----	
	normality	unknown	unknown	unknown	unknown	unknown	unknown	unknown	
	n	7	7	7	7	7	7	7	5
	outliers	1	1	1	1	1	1	1	
	mean (n)	0.0004	0.0039	0.0084	0.0189	0.0447	0.1054	0.2258	Pass
	st.dev. (n)	0.00197	0.00372	0.00510	0.00385	0.00414	0.00465	0.00749	
	R(calc.)	0.0055	0.0104	0.0143	0.0108	0.0116	0.0130	0.0210	
	st.dev.(lit)	unknown	unknown	unknown	unknown	unknown	unknown	unknown	
	R(lit)	unknown	unknown	unknown	unknown	unknown	unknown	unknown	





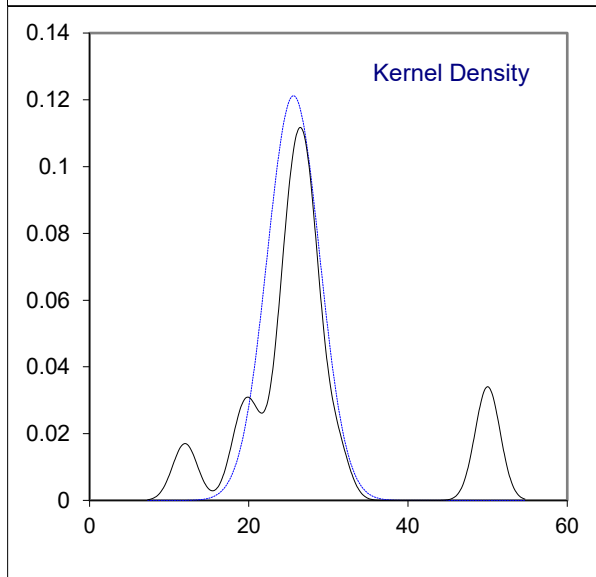
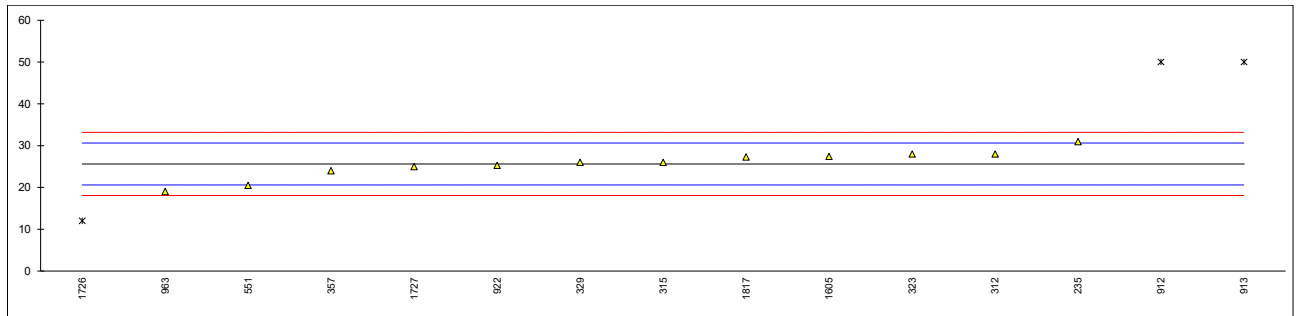
Determination of Purity of Ethanol on dry basis on sample #22251; results in %M/M

lab	method	value	mark	z(targ)	remarks
150		----		----	
170		----		----	
235		----		----	
273	IMPCA001	99.98	G(0.05)	----	
312	INH-0001	99.99		----	
315	INH-933	99.99		----	
323	INH-001	99.99		----	
329	INH-001	99.993		----	
357	INH-02	99.994		----	
446		----		----	
551	INH-1313	99.994		----	
912	IS323	99.99		----	
913	ETOH0002	99.99		----	
922	INH-02	99.993		----	
963	D5501	99.996		----	
1205		----		----	
1574		----		----	
1605		----		----	
1726	In house	99.9951		----	
1727	In house	99.9994		----	
1817	In house	94.38	G(0.01)	----	possibly a false negative test result?
1835	In house	99.9927		----	
1927		----		----	
6214		----		----	
6224		----		----	
6396		100.00		----	
normality		OK			
n		14			
outliers		2			
mean (n)		99.9934			
st.dev. (n)		0.00338			
R(calc.)		0.0095			
st.dev.(lit)		unknown			
R(lit)		unknown			



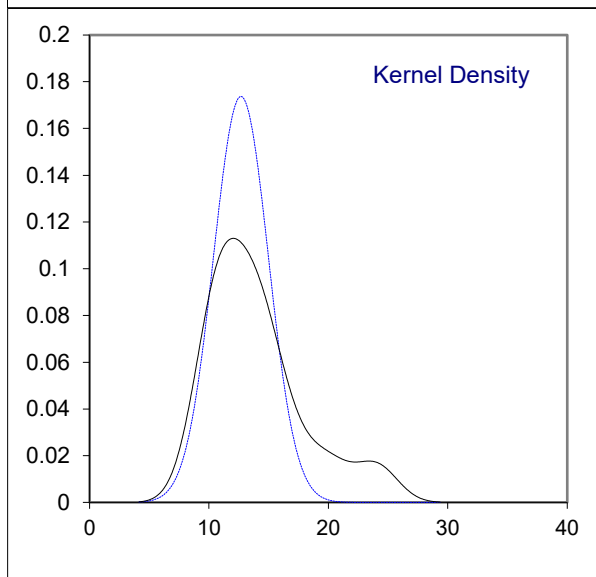
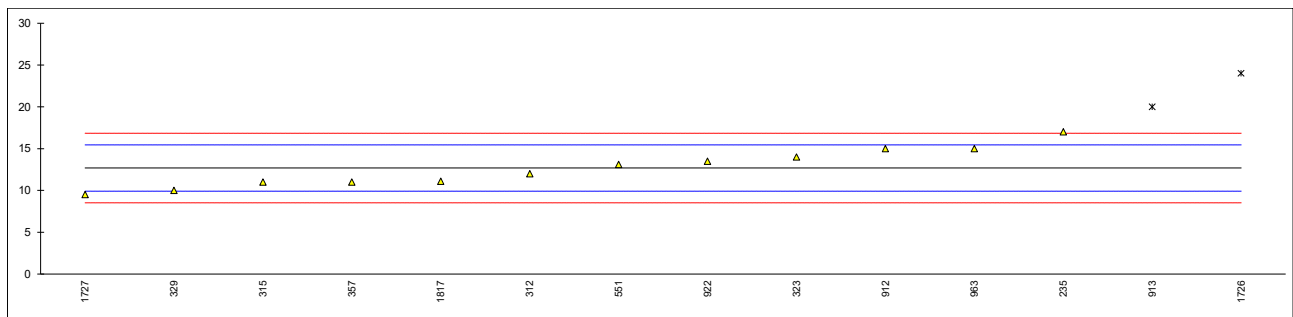
Determination of Methanol on sample #22251; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150		----		----	
170		----		----	
235	INH-0001	30.96		2.12	
273		----		----	
312	INH-0001	28		0.95	
315	INH-933	26		0.15	
323	INH-001	28		0.95	
329	INH-001	26		0.15	
357	INH-02	24		-0.64	
446		----		----	
551	INH-1313	20.5		-2.03	
912	IS323	50	DG(0.01)	9.69	
913	ETOH0002	50	DG(0.01)	9.69	
922	INH-02	25.28		-0.13	
963	D5501	19		-2.63	
1205		----		----	
1574		----		----	
1605		27.4		0.71	
1726	In house	12	G(0.05)	-5.41	
1727	In house	25		-0.25	
1817	In house	27.2573		0.65	
1835	In house	<25		----	
1927		----		----	
6214		----		----	
6224		----		----	
6396		----		----	
normality		OK			
n		12			
outliers		3			
mean (n)		25.616			
st.dev. (n)		3.2911			
R(calc.)		9.215			
st.dev.(Horwitz)		2.5156			
R(Horwitz)		7.044			



Determination of Acetone on sample #22251; results in mg/kg

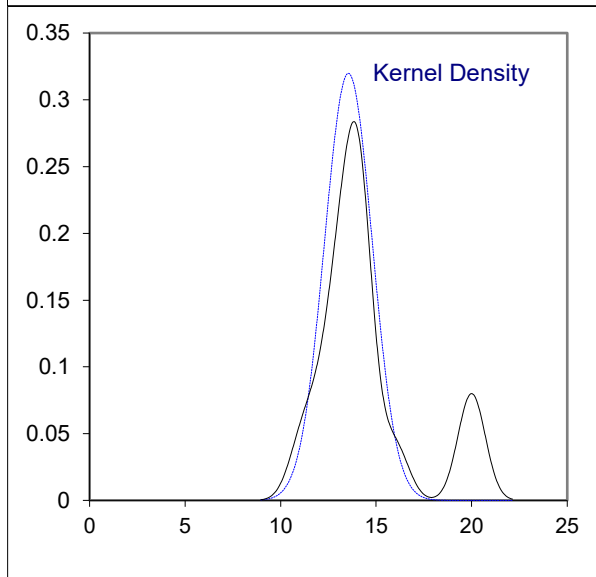
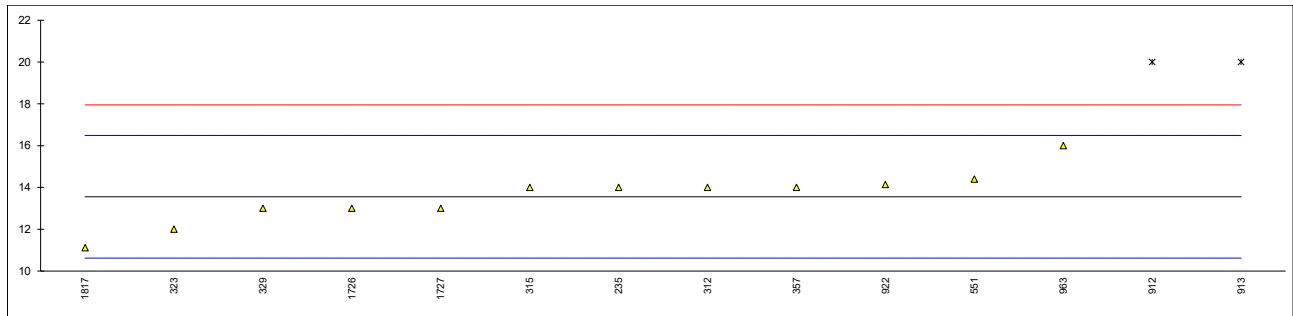
lab	method	value	mark	z(targ)	remarks
150		----		----	
170		----		----	
235	INH-0001	17.01	C	3.13	first reported 18.95
273		----		----	
312	INH-0001	12	C	-0.49	first reported 22
315	INH-933	11		-1.22	
323	INH-001	14		0.95	
329	INH-001	10		-1.94	
357	INH-02	11		-1.22	
446		----		----	
551	INH-1313	13.1		0.30	
912	IS323	15		1.67	
913	ETOH0002	20	DG(0.05)	5.29	
922	INH-02	13.48		0.58	
963	D5501	15		1.67	
1205		----		----	
1574		----		----	
1605		----		----	
1726	In house	24	DG(0.05)	8.17	
1727	In house	9.5		-2.30	
1817	In house	11.0977		-1.14	
1835	In house	<25		----	
1927		----		----	
6214		----		----	
6224		----		----	
6396		----		----	
normality		OK			
n		12			
outliers		2			
mean (n)		12.682			
st.dev. (n)		2.2958			
R(calc.)		6.428			
st.dev.(Horwitz)		1.3844			
R(Horwitz)		3.876			



Determination of Benzene on sample #22251; results in mg/kg

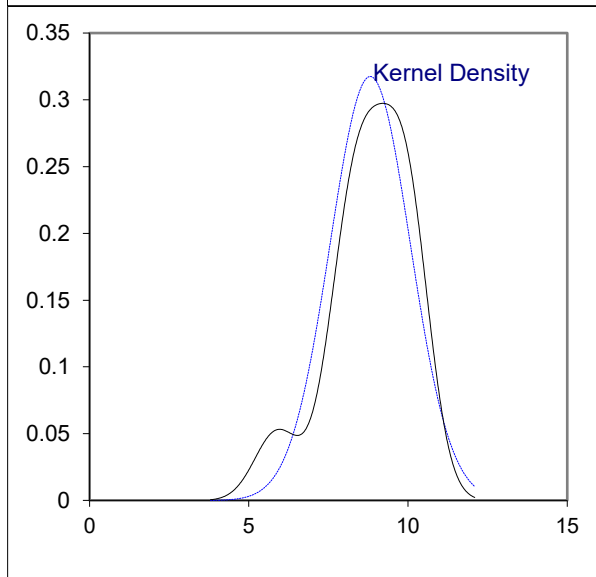
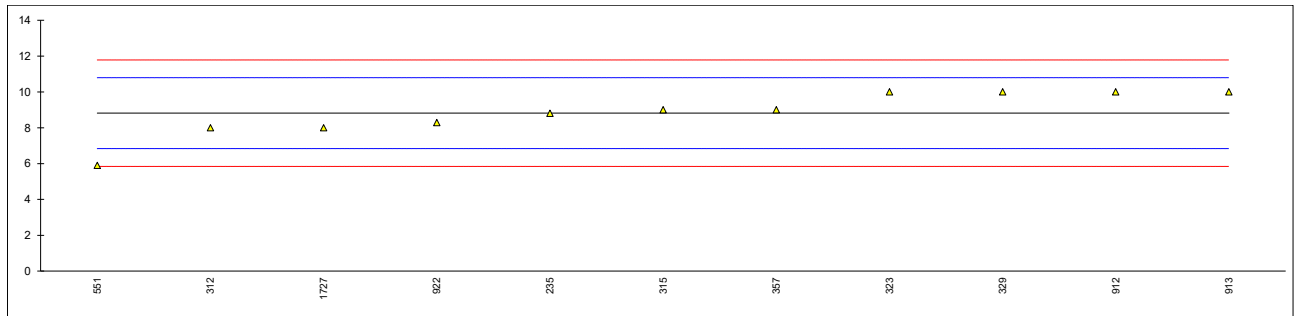
lab	method	value	mark	z(targ)	remarks
150		----		----	
170		----		----	
235	INH-0001	14		0.30	
273		----		----	
312	INH-0001	14		0.30	
315	INH-933	14		0.30	
323	INH-001	12	C	-1.06	first reported 20
329	INH-001	13		-0.38	
357	INH-02	14		0.30	
446		----		----	
551	INH-1299	14.4		0.58	
912	IS323	20	DG(0.01)	4.40	
913	ETOH0002	20	DG(0.01)	4.40	
922	INH-02	14.14		0.40	
963	D5501	16		1.67	
1205		----		----	
1574		----		----	
1605		----		----	
1726	In house	13		-0.38	
1727	In house	13		-0.38	
1817	In house	11.1177		-1.66	
1835	In house	<25		----	
1927		----		----	
6214		----		----	
6224		----		----	
6396		----		----	

normality suspect  
n 12  
outliers 2  
mean (n) 13.555  
st.dev. (n) 1.2471  
R(calc.) 3.492  
st.dev.(Horwitz) 1.4649  
R(Horwitz) 4.102



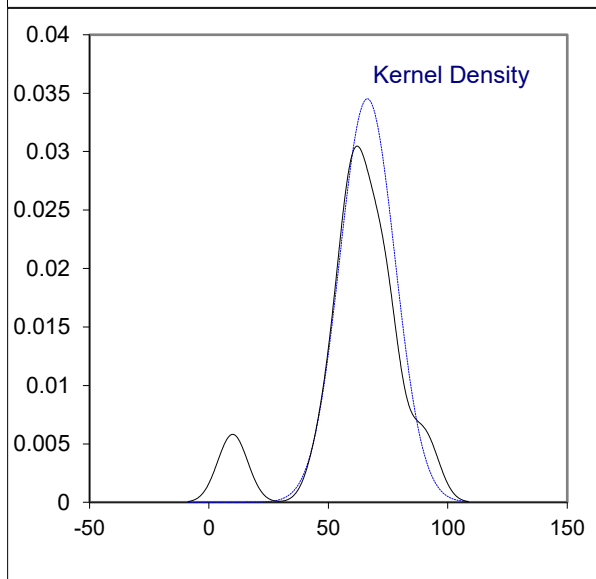
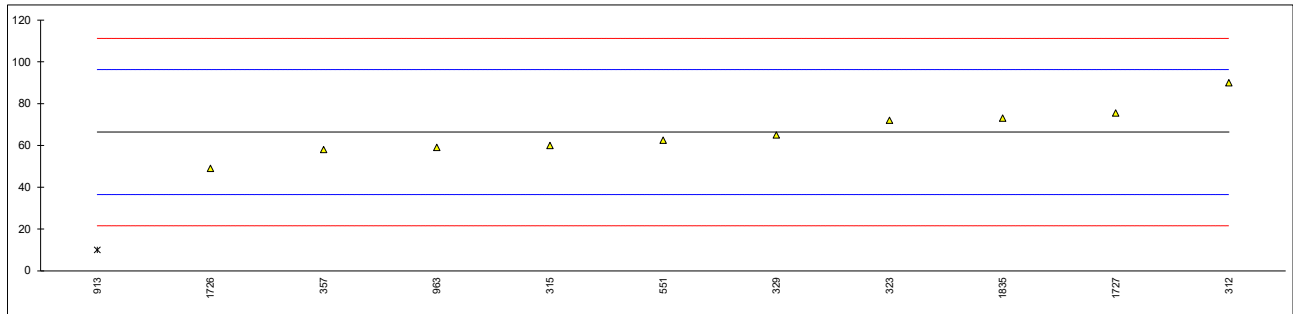
Determination of Isopropanol on sample #22251; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150		----		----	
170		----		----	
235	INH-0001	8.81		-0.01	
273		----		----	
312	INH-0001	8		-0.80	
315	INH-933	9		0.18	
323	INH-001	10		1.16	
329	INH-001	10		1.16	
357	INH-02	9		0.18	
446		----		----	
551	INH-1313	5.9		-2.87	
912	IS323	10		1.16	
913	ETOH0002	10		1.16	
922	INH-02	8.28		-0.53	
963	D5501	<10		----	
1205		----		----	
1574		----		----	
1605		----		----	
1726		----		----	
1727	In house	8		-0.80	
1817		----		----	
1835	In house	<25		----	
1927		----		----	
6214		----		----	
6224		----		----	
6396		----		----	
normality		suspect			
n		11			
outliers		0			
mean (n)		8.817			
st.dev. (n)		1.2569			
R(calc.)		3.519			
st.dev.(Horwitz)		1.0166			
R(Horwitz)		2.847			



Determination of Total impurities on sample #22251; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150		----		----	
170		----		----	
235		----		----	
273		----		----	
312	INH-0001	90		1.58	
315	INH-933	60		-0.43	
323	INH-001	72		0.37	
329	INH-001	65		-0.09	
357	INH-02	58		-0.56	
446		----		----	
551	INH-1313	62.5		-0.26	
912		----		----	
913	ETOH0002	10	G(0.05)	-3.77	
922		----		----	
963	D5501	59		-0.50	
1205		----		----	
1574		----		----	
1605		----		----	
1726	In house	49		-1.16	
1727	In house	75.5		0.61	
1817	In house	< 300		----	
1835	In house	73		0.44	
1927		----		----	
6214		----		----	
6224		----		----	
6396		----		----	
normality		OK			
n		10			
outliers		1			
mean (n)		66.400			
st.dev. (n)		11.5513			
R(calc.)		32.344			
st.dev.(Horwitz, comp:7)		14.9477			
R(Horwitz, comp:7)		41.853			





**APPENDIX 2**

Other reported impurities in sample #22251; results in mg/kg

lab	Acetal (1,1-diethoxyethane)	Acetaldehyde	Mono Ethylene glycol (MEG)	Other impurities
150	----	----	----	----
170	----	----	----	----
235	----	0	----	----
273	----	----	----	----
312	<5	<5	12	6
315	----	----	<10	<50
323	< 5	< 5	----	< 5
329	<5	<5	<5	5
357	< 5	< 5	< 30	< 5
446	----	----	----	----
551	<5	<5	29.98	----
912	<10	<10	<10	----
913	<10	<10	<10	<10
922	<2.0	<2.0	<10	----
963	----	<10	----	----
1205	----	----	----	----
1574	----	----	----	----
1605	----	----	----	----
1726	----	----	----	----
1727	<1	<1	----	19
1817	ND	ND	----	< 300
1835	<25	<10	----	<50
1927	----	----	----	----
6214	----	----	----	----
6224	----	----	----	----
6396	----	----	----	----

## **APPENDIX 3**

### **Number of participants per country**

3 labs in BELGIUM  
1 lab in BRAZIL  
1 lab in FINLAND  
1 lab in HONG KONG  
1 lab in HUNGARY  
2 labs in INDIA  
1 lab in MAURITIUS  
4 labs in NETHERLANDS  
1 lab in PAKISTAN  
1 lab in SAUDI ARABIA  
1 lab in SOUTH AFRICA  
3 labs in SPAIN  
2 labs in THAILAND  
1 lab in UNITED KINGDOM  
3 labs 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)/G1	= outlier in Grubbs' outlier test
G(0.05)/G5	= 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	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?
SDS	= Safety Data Sheet

### Literature

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
- 2 ISO5725:86
- 3 ISO5725 parts 1-6:94
- 4 ISO13528:05
- 5 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 8 J.N. Miller, Analyst, 118, 455, (1993)
- 9 Analytical Methods Committee, Technical Brief, No 4, January 2001
- 10 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
- 11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
- 12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)