

Results of Proficiency Test
Ethanol (Food/Neutral)
December 2018

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis18C11

March 2019

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1 INTRODUCTION

Since 2007, a proficiency test for Food/Neutral grade Ethanol is organized by the Institute for Interlaboratory Studies every year. During the planning of the annual proficiency testing program 2018/2019, it was decided to continue the round robin for the analysis of Food/Neutral grade Ethanol.

In this interlaboratory study 25 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 2018 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/IEC 17025 accredited laboratory. It was decided to send two different samples of Ethanol (Food & Neutral grade), a 0.5 L bottle (labelled #18242) and a 0.25 L bottle (labelled #18243) 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/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires

2.2 PROTOCOL

The protocol followed in the 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), main round

Approximately 55 litre of Ethanol (Food & Neutral grade) was obtained from a local supplier. After homogenisation, 48 amber glass bottles of 0.5 L were filled and labelled #18242. 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 D1364 on 8 stratified randomly selected samples.

	Density at 20°C in kg/L	Water in %M/M
Sample #18242-1	0.80612	5.707
Sample #18242-2	0.80611	5.702
Sample #18242-3	0.80611	5.698
Sample #18242-4	0.80611	5.707
Sample #18242-5	0.80611	5.700
Sample #18242-6	0.80611	5.699
Sample #18242-7	0.80611	5.703
Sample #18242-8	0.80611	5.717

Table 1: homogeneity test results of subsamples #18242

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 ISO 13528, Annex B2 in the next table:

	Density at 20°C in kg/L	Water in %M/M
r (observed)	0.00001	0.017
reference test method	ISO12185:96	D1364:02
0.3 * R (ref. test method)	0.00015	0.043

Table 2: evaluation of the repeatabilities of subsamples #18242

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibility of the reference test methods. Therefore, homogeneity of the subsamples #18242 was assumed.

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

A batch of approximately 17 litre Ethanol (Food & Neutral grade) was separated from the batch for the main round and spiked with Methanol (approx. 15 mg/kg), Acetone (approx. 25 mg/kg), Benzene (approx. 10 mg/kg), Isopropanol (approx. 30 mg/kg) and Monoethylene Glycol (approx. 30 mg/kg). After homogenisation, out of the batch 48 amber glass bottles of 0.25 L were filled and labelled #18243. The homogeneity of the subsamples was checked by determination of Acetone, Benzene and Isopropanol on 8 stratified randomly selected samples.

	Acetone in mg/kg	Benzene in mg/kg	Isopropanol in mg/kg
Sample #18243-1	13.7	7.0	24.3
Sample #18243-2	13.6	7.0	22.6
Sample #18243-3	13.8	6.6	23.1
Sample #18243-4	12.9	6.7	24.0
Sample #18243-5	12.9	7.2	23.9
Sample #18243-6	13.8	6.7	22.7
Sample #18243-7	13.5	6.9	23.2
Sample #18243-8	14.2	6.7	23.0

Table 3: homogeneity test results of subsamples #18243

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

	Acetone in mg/kg	Benzene in mg/kg	Isopropanol in mg/kg
r (observed)	1.26	0.58	1.78
reference method	Horwitz	Horwitz	Horwitz
0.3 * R (ref. method)	1.23	0.69	1.95

Table 4: evaluation of the repeatabilities of subsamples #18243

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the subsamples #18243 was assumed.

To each of the participating laboratories 1*0.5 L bottle of sample #18242 and 1*0.25 L bottle #18243 was sent on November 7, 2018. An SDS of the product was added to the sample package.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYSES

The participants were asked to determine on sample #18242: Density at 20°C, Non-volatile matter, Permanganate Time Test at 20°C, pHe, Strength (in %M/M and %V/V), Water (titrimetric) 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 #18243: Purity Ethanol on dry basis, Methanol, Acetal, Acetaldehyde, Acetone, Benzene, Isopropanol, Monoethylene glycol, 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 reanalyses). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). For the statistical evaluation, the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<...>' or '>...>' were not used in the statistical evaluation.

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

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

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. 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, no major problems were encountered with the dispatch of the samples. One participant reported the results after the final reporting date. Not all participants were able to report results for all analyses requested. In total 25 laboratories reported 303 numerical results. Observed were 20 outlying results, which is 6.6%. 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 listed 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 #18242:

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

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 problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D1363:06(2011).

- pHe: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D6423:14.
- Strength (%M/M): This determination may not be problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the reproducibility derived from the OIML table and ISO12185:96.
- Strength (%V/V): This determination may not be problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the reproducibility derived from the OIML table and ISO12185:96.
- Water (titrimetric): This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D1364:02(2012).
- UV absorbance: Regretfully, no 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. Nine laboratories used a 50 mm cuvette and seven laboratories used a 10 mm cuvette. Both groups were evaluated separately.
- UV - 50 mm cuvette: In total over seven parameters (UV absorbance in nm), eight statistical outliers were observed and two other test results were excluded. One laboratory evaluated the sample as 'Fail', whereas all other laboratories evaluated the sample as 'Pass'.
- UV - 10 mm cuvette: No statistical outliers were observed. All laboratories evaluated the sample as 'Pass'.

Sample #18243:

Purity 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. Two statistical outliers were observed. The calculated reproducibility is smaller than the calculated reproducibility in the previous proficiency tests iis17C16 and iis16C11.

Methanol: This determination may be very problematic. No statistical outlier was observed. However, the calculated reproducibility is not in agreement with the estimated reproducibility using the Horwitz equation. The average recovery of Methanol (theoretical increment of 15.2 mg Methanol/kg) may be insufficient: "< 55%" (the actual blank Methanol content is not known).

Acetone: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in full agreement with the estimated reproducibility using the Horwitz equation. The average recovery of Acetone (theoretical increment of 24.6 mg Acetone/kg) may be insufficient: "< 54%" (the actual blank Acetone content is not known).

Benzene: This determination may be very problematic. No statistical outlier was observed. However, the calculated reproducibility is not in agreement with the estimated reproducibility using the Horwitz equation. The average recovery of Benzene (theoretical increment of 9.8 mg Benzene/kg) may be sufficient: "< 81%" (the actual blank Benzene content is not known).

Isopropanol: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility using the Horwitz equation.

Monoethylene glycol: Four laboratories reported a numeric test result and two other laboratories reported a "less than" test result. Therefore, no z-scores were calculated. The average recovery of Monoethylene glycol (theoretical increment of 29.9 mg Monoethylene glycol/kg) may be good: "< 93%" (the actual blank Monoethylene glycol content is not known).

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 for 5 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 ($2.8 \times$ 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 * sd	R (lit)
Density at 20°C	kg/L	23	0.8061	0.0002	0.0005
Nonvolatile matter	mg/100mL	11	0.4	1.0	2.1
Permanganate Time Test	min.	11	36.4	14.8	9.2
pHe		9	7.9	0.5	1.0
Strength	%M/M	14	94.30	0.02	0.06
Strength	%V/V	21	96.32	0.04	0.06
Water (titrimetric)	%M/M	16	5.67	0.16	0.14
UV – 50 mm cuvette:					
UV-absorbance 300 nm		7	0.010	0.006	n.a.
UV-absorbance 270 nm		7	0.028	0.009	n.a.
UV-absorbance 260 nm		7	0.050	0.011	n.a.
UV-absorbance 250 nm		8	0.105	0.021	n.a.
UV-absorbance 240 nm		7	0.233	0.027	n.a.
UV-absorbance 230 nm		7	0.514	0.065	n.a.
UV-absorbance 220 nm		7	1.00	0.125	n.a.
Conclusion UV-scan	Pass/Fail	7	Pass	n.a.	n.a.
UV – 10 mm cuvette:					
UV-absorbance 300 nm		6	-0.002	0.013	n.a.
UV-absorbance 270 nm		6	0.0003	0.0172	n.a.
UV-absorbance 260 nm		7	0.004	0.016	n.a.
UV-absorbance 250 nm		7	0.015	0.017	n.a.
UV-absorbance 240 nm		7	0.041	0.020	n.a.
UV-absorbance 230 nm		7	0.094	0.020	n.a.
UV-absorbance 220 nm		7	0.186	0.028	n.a.
Conclusion UV-scan	Pass/Fail	5	pass	n.a.	n.a.

Table 5: reproducibilities of tests on sample #18242

Parameter	unit	n	average	2.8 *sd	R (lit)
Purity of Ethanol on dry basis	%M/M	14	99.99	0.01	n.a.
Methanol	mg/kg	15	8.3	5.8	2.7
Acetone	mg/kg	10	13.3	4.1	4.0
Benzene	mg/kg	13	7.9	5.2	2.6
Isopropanol	mg/kg	12	20.5	4.9	5.8
Monoethylene glycol (MEG)	mg/kg	3	27.8	18.2	n.a.
Total impurities	mg/kg	11	56.1	47.2	30.6

Table 6: reproducibilities of tests on sample #18243

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 2018 WITH PREVIOUS PTS

	December 2018	December 2017	December 2016	November 2015	November 2014
Number of reporting labs	25	29	26	32	25
Number of results reported	303	301	329	254	210
Number of statistical outliers	20	22	16	11	13
Percentage outliers	6.6%	7.3%	4.9%	4.3%	6.2%

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 2018	December 2017	December 2016	November 2015	November 2014
Density at 20°C	++	++	++	++	++
Nonvolatile matter	++	++	++	++	n.e.
Permanganate Time Test	-	-	(+)	-	+
pHe	++	-	(-)	--	n.e.
Strength %M/M	++	++	(+/-)	(+)	(+)
Strength %V/V	+	++	++	+	+
Water (titrimetric)	-	-	-	-	--
Purity Ethanol on dry basis	(+)	(+)	(+)	(-)	(-)
Methanol	--	-	++	n.e.	n.e.
Acetone	+/-	--	n.e.	n.e.	n.e.
Acetal	n.e.	n.e.	+/-	n.e.	n.e.
Benzene	--	n.e.	. ++	n.e.	n.e.

Parameter	December 2018	December 2017	December 2016	November 2015	November 2014
Isopropanol	+	+	n.e.	n.e.	n.e.
Monoethylene glycol (MEG)	n.e.	n.e.	+/-	n.e.	n.e.
Total impurities	-	-	n.e.	n.e.	n.e.

Table 8: comparison determinations of sample #18242 and #18243 against the standard

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

The performance of the determinations against the requirements of the respective reference test methods is listed in the above table.

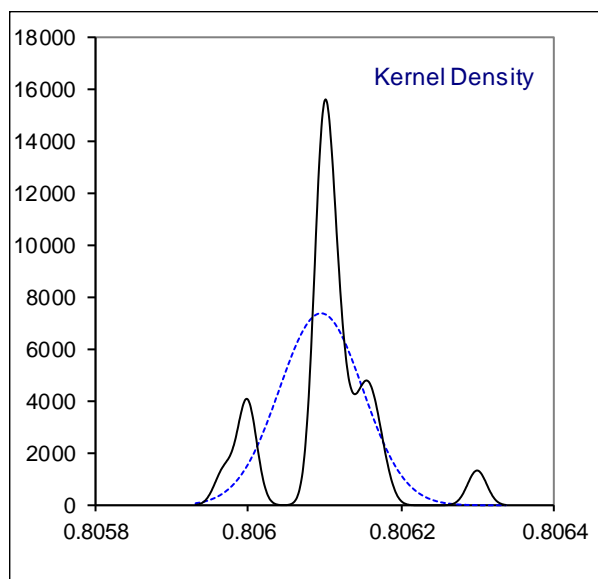
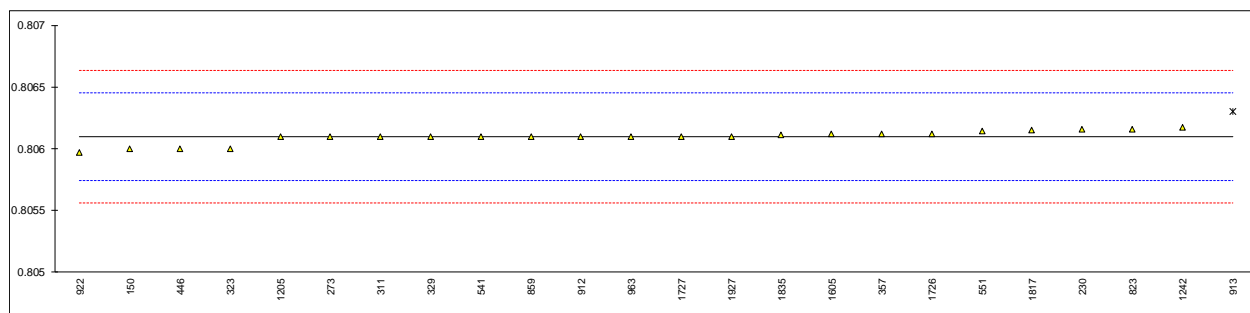
The following performance categories were used:

- ++: 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

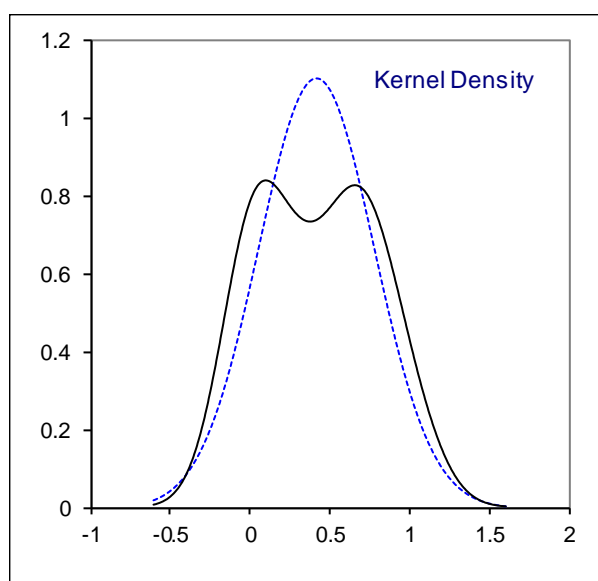
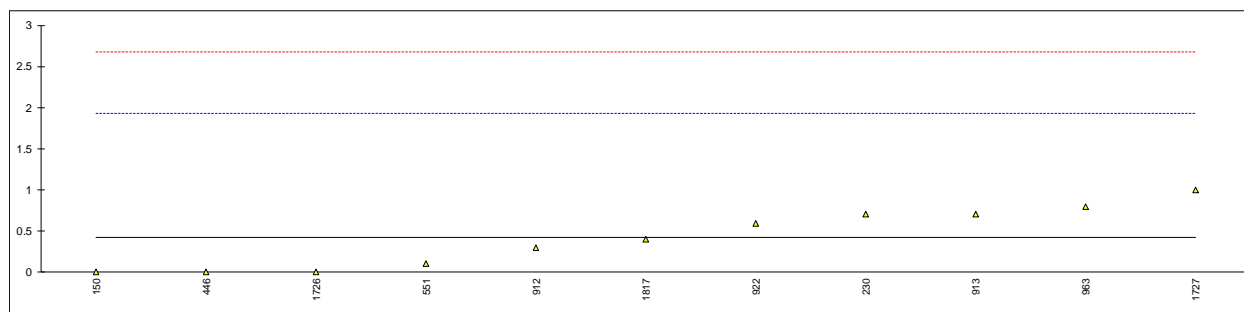
Determination of Density at 20°C on sample #18242; results in kg/L

lab	method	value	mark	z(targ)	remarks
150	D4052	0.8060		-0.54	
230	D4052	0.80616		0.36	
273	D4052	0.8061		0.02	
311	D4052	0.8061		0.02	
323	D4052	0.8060		-0.54	
329	D4052	0.8061		0.02	
357	D4052	0.80612		0.13	
446	D4052	0.8060		-0.54	
541	D4052	0.80610		0.02	
551	D4052	0.80614		0.24	
823	D4052	0.80616		0.36	
859	D4052	0.8061		0.02	
912	D4052	0.8061		0.02	
913	D4052	0.8063	C,R(0.05)	1.14	first reported 806.3 kg/L
922	D4052	0.80597		-0.71	
963	D4052	0.8061		0.02	
1205	In house	0.806095		-0.01	
1242	In house	0.806175		0.44	
1574		-----		-----	
1605	D4052	0.806119		0.13	
1726	D4052	0.80612		0.13	
1727	D4052	0.80610		0.02	
1817	Table OIML	0.80615		0.30	
1835	ISO12185	0.80611		0.08	
1927	D4052	0.80610		0.02	
normality		OK			
n		23			
outliers		1			
mean (n)		0.806096			
st.dev. (n)		0.0000543			
R(calc.)		0.000152			
st.dev.(ISO12185:96)		0.0001786			
R(ISO12185:96)		0.0005			



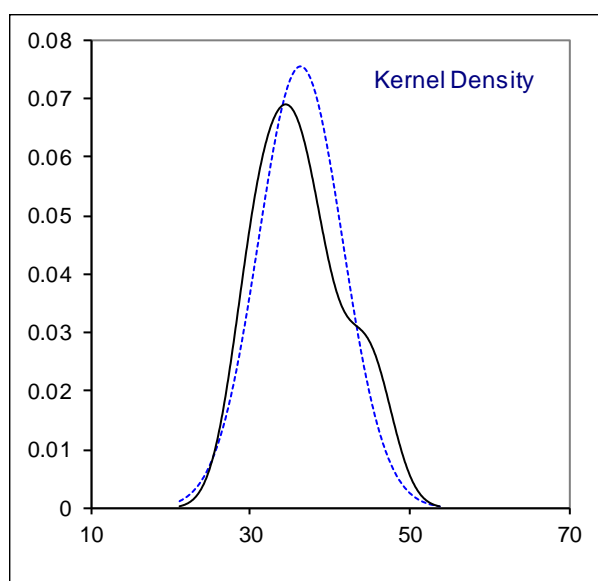
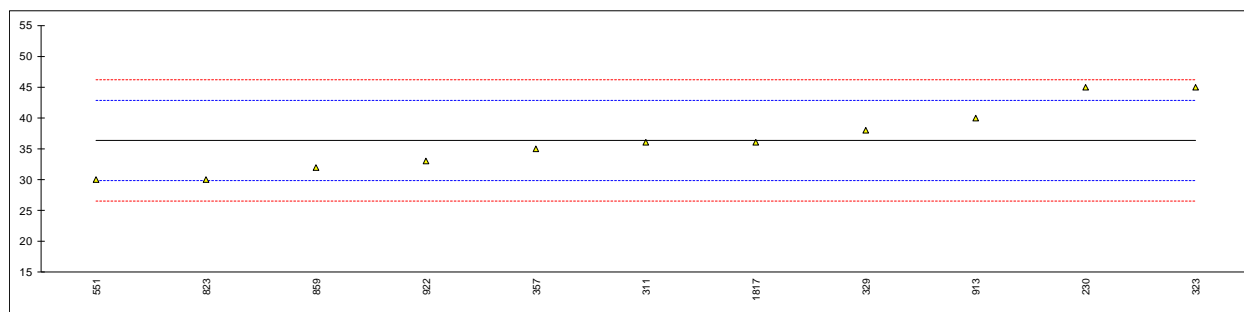
Determination of Nonvolatile matter on sample #18242; results in mg/100mL

lab	method	value	mark	z(targ)	remarks
150	D1353	0		-0.55	
230	D1353	0.7		0.38	
273		----		----	
311	D1353	<1		----	
323	D1353	<1		----	
329	D1353	<1		----	
357	D1353	< 1		----	
446	D1353	0.0		-0.55	
541	D1353	<0.1		----	
551	D1353	0.1		-0.42	
823		----		----	
859	D1353	<1		----	
912	D1353	0.3		-0.16	
913	D1353	0.7		0.38	
922	D1353	0.59		0.23	
963	D1353	0.8		0.51	
1205		----		----	
1242		----		----	
1574		----		----	
1605		----		----	
1726	EN15691	0		-0.55	
1727	EN15691	1		0.77	
1817	In house	0.4		-0.02	
1835	EN15691	<10		----	
1927		----		----	
normality		OK			
n		11			
outliers		0			
mean (n)		0.42			
st.dev. (n)		0.362			
R(calc.)		1.01			
st.dev.(D1353:13)		0.754			
R(D1353:13)		2.11			



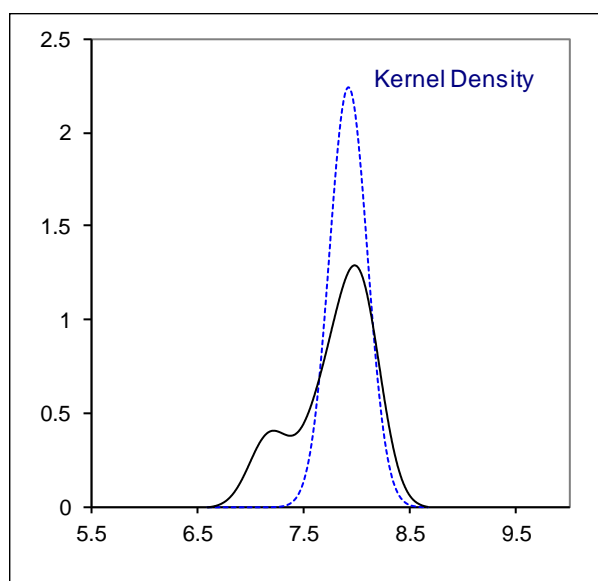
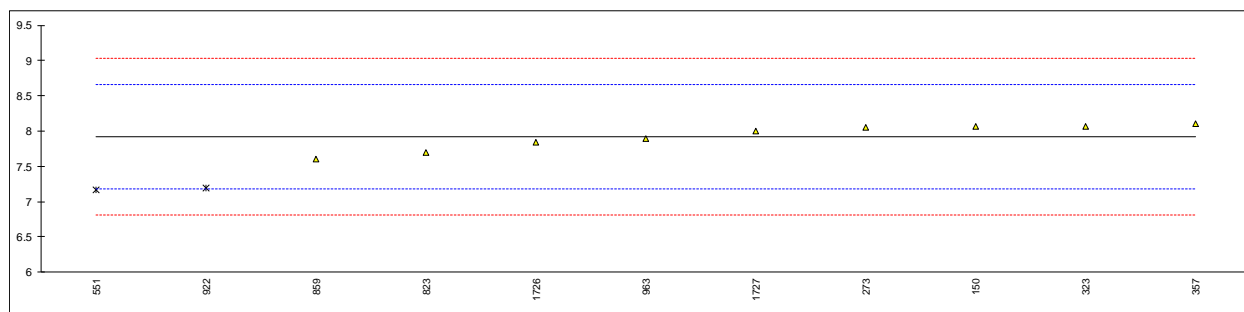
Determination of Permanganate Time Test at 20°C on sample #18242; results in minutes

lab	method	value	mark	z(targ)	remarks
150		----		----	
230	D1363	45		2.64	
273		----		----	
311	D1363	36		-0.11	
323	D1363	45		2.64	
329	D1363	38		0.50	
357	D1363	35		-0.42	
446	BS6392	>30		----	
541		----		----	
551	D1363	30		-1.94	
823	D1363	30		-1.94	
859	D1363	32		-1.33	
912		----		----	
913	D1363	40		1.11	
922	D1363	33		-1.03	
963		----		----	
1205		----		----	
1242		----		----	
1574		----		----	
1605		----		----	
1726		----		----	
1727		----		----	
1817	In house	36		-0.11	
1835		----		----	
1927		----		----	
normality		OK			
n		11			
outliers		0			
mean (n)		36.36			
st.dev. (n)		5.278			
R(calc.)		14.78			
st.dev.(D1363:06)		3.273			
R(D1363:06)		9.16			



Determination of pHe on sample #18242;

lab	method	electrode	value	mark	z(targ)	remarks
150	D6423	KCl	8.06		0.37	
230			----		----	
273	D6423	KCl	8.05		0.34	
311			----		----	
323	D6423	LiCl	8.06		0.37	
329			----		----	
357	D6423	KCl	8.1		0.48	
446			----		----	
541			----		----	
551	D6423	LiCl	7.17	DG(0.05)	-2.04	
823	D6423	KCl	7.7		-0.60	
859	D6423	LiCl	7.6		-0.87	
912			----		----	
913			----		----	
922	D6423	KCl	7.199	DG(0.05)	-1.96	
963	D6423		7.9		-0.06	
1205			----		----	
1242			----		----	
1574			----		----	
1605			----		----	
1726	EN15490	LiCl	7.84		-0.23	
1727	EN15490	LiCl	8.00		0.21	
1817			----		----	
1835			----		----	
1927			----		----	
normality			OK			
n			9			
outliers			2			
mean (n)			7.923			
st.dev. (n)			0.1776			
R(calc.)			0.497			
st.dev.(D6423:14)			0.3701			
R(D6423:14)			1.036			

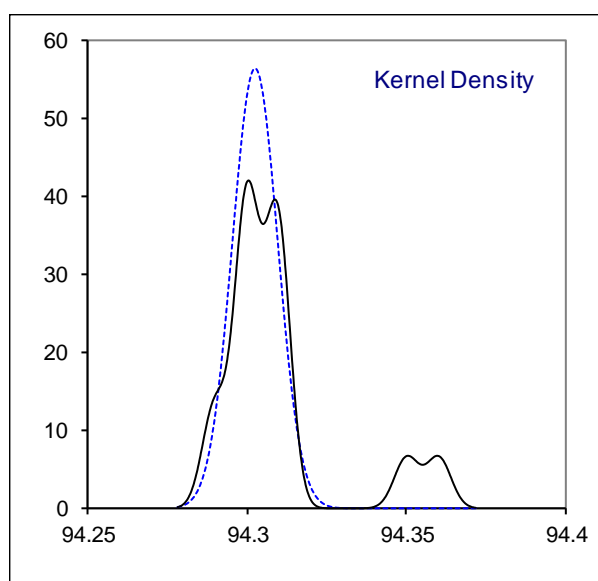
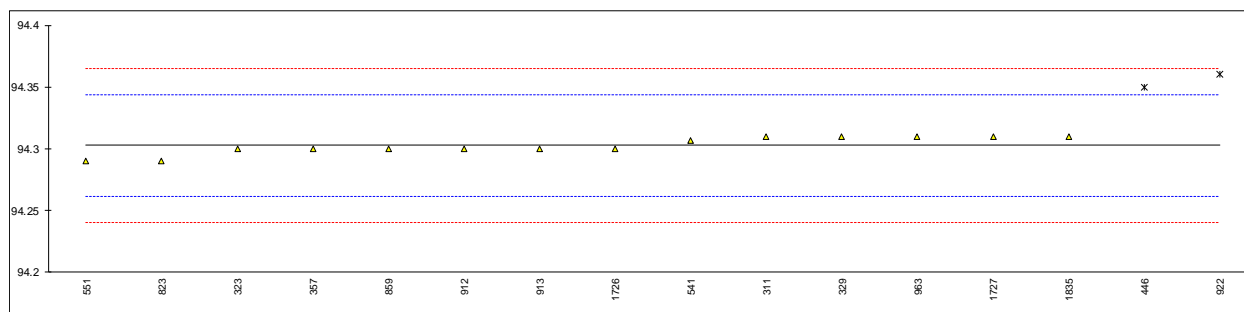


Determination of Strength on sample #18242; results in %M/M

lab	method	value	mark	z(targ)	remarks
150		----		----	
230		----		----	
273		----		----	
311	Table OIML	94.31		0.35	
323	Table OIML	94.30	E	-0.13	iis calculated 94.34
329	Table OIML	94.31		0.35	
357	Table OIML	94.30		-0.13	
446	Table OIML	94.35	G(0.01)	2.29	
541	Table OIML	94.307		0.21	
551	NBR15639	94.29		-0.61	
823	Table OIML	94.29		-0.61	
859	Table OIML	94.30		-0.13	
912	Table OIML	94.30		-0.13	
913	Table OIML	94.30	C	-0.13	first reported 94.24
922	Table OIML	94.36	G(0.05)	2.77	
963	Table OIML	94.31		0.35	
1205		----		----	
1242		----		----	
1574		----		----	
1605		----		----	
1726	Table OIML	94.30		-0.13	
1727	Table OIML	94.31		0.35	
1817		----		----	
1835	Table OIML	94.31		0.35	
1927		----		----	

normality OK
 n 14
 outliers 2
 mean (n) 94.303
 st.dev. (n) 0.0071
 R(calc.) 0.020
 st.dev.(OIML table) 0.0207
 R(OIML table) 0.058

OIML R022-e75

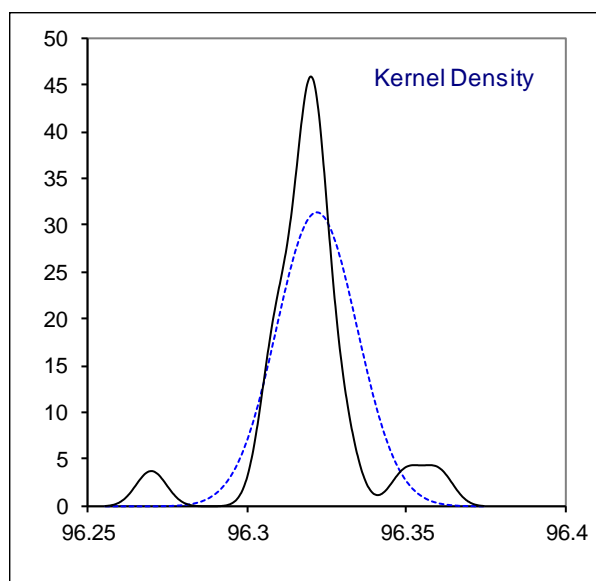
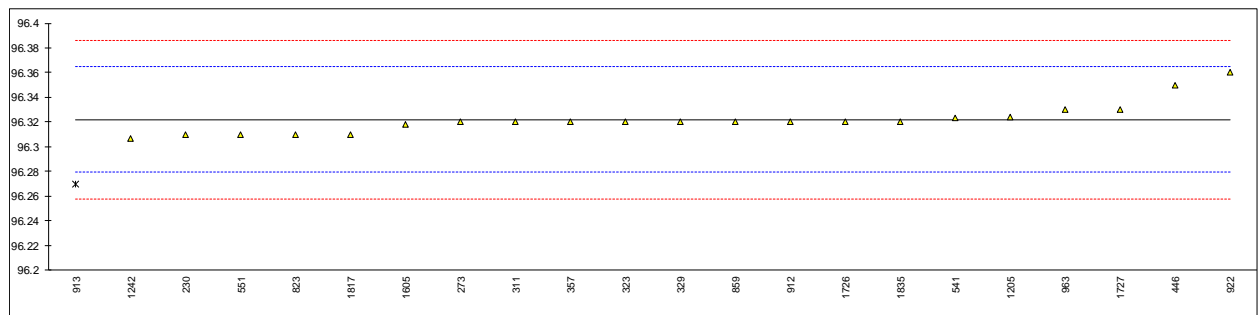


Determination of Strength on sample #18242; results in %V/V

lab	method	value	mark	z(targ)	remarks
150		----		----	
230	Table OIML	96.31		-0.56	
273	Table OIML	96.32		-0.09	
311	Table OIML	96.32		-0.09	
323	Table OIML	96.32		-0.09	
329	Table OIML	96.32		-0.09	
357	Table OIML	96.32		-0.09	
446	Table OIML	96.35		1.31	
541	Table OIML	96.323		0.05	
551	NBR15639	96.31		-0.56	
823	Table OIML	96.31		-0.56	
859	Table OIML	96.32		-0.09	
912	Table OIML	96.32		-0.09	
913	Table OIML	96.27	R(0.05)	-2.43	
922	Table OIML	96.36		1.77	
963	Table OIML	96.33		0.37	
1205	Table OIML	96.324		0.09	
1242	In house	96.307		-0.70	
1574		----		----	
1605	Table OIML	96.318		-0.19	
1726	Table OIML	96.32		-0.09	
1727	Table OIML	96.33		0.37	
1817	Table OIML	96.31		-0.56	
1835	Table OIML	96.32		-0.09	
1927		----		----	

normality not OK
n 21
outliers 1
mean (n) 96.322
st.dev. (n) 0.0127
R(calc.) 0.035
st.dev.(OIML table) 0.0214
R(OIML table) 0.060

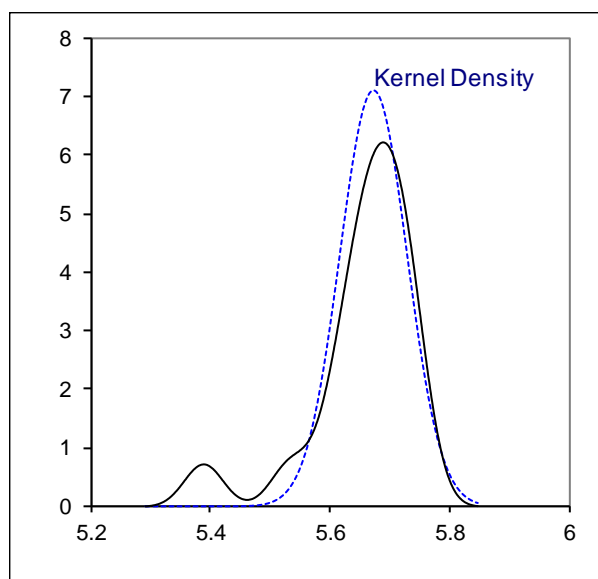
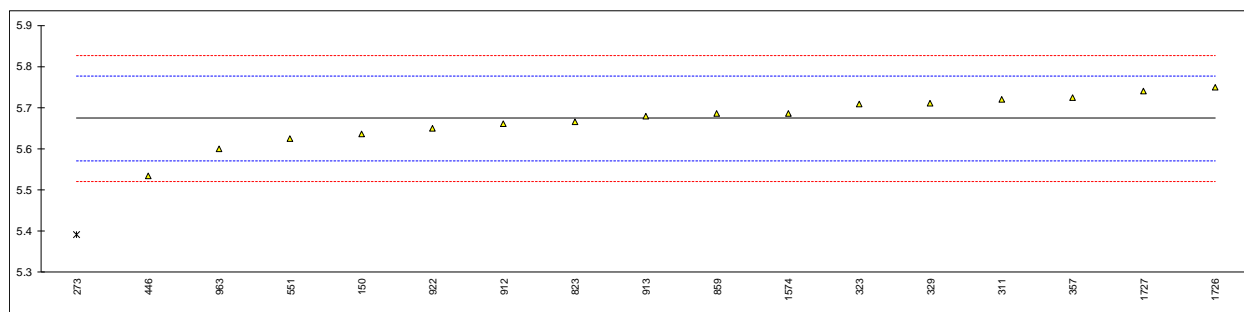
OIML R022-e75



Determination of Water (titrimetric) on sample #18242; results in %M/M

lab	method	value	mark	z(targ)	remarks
150	E203	5.635		-0.75	
230		-----		-----	
273	E203	5.39	G(0.01)	-5.55	
311	E203	5.719		0.89	
323	D1364	5.708		0.68	
329	E203	5.710		0.72	
357	E203	5.725		1.01	
446	D1364	5.535		-2.71	
541		-----		-----	
551	D1364	5.624		-0.97	
823	D1364	5.666		-0.14	
859	D1364	5.685		0.23	
912	E203	5.66		-0.26	
913	E203	5.68		0.13	
922	E203	5.65		-0.46	
963	D1364	5.60		-1.44	
1205		-----		-----	
1242		-----		-----	
1574	INH-76	5.6863		0.25	
1605		-----		-----	
1726	EN15692	5.7502		1.51	
1727	EN15692	5.74	C	1.31	first reported 5.44
1817		-----		-----	
1835		-----		-----	
1927		-----		-----	

normality suspect
n 16
outliers 1
mean (n) 5.6733
st.dev. (n) 0.05609
R(calc.) 0.1571
st.dev.(D1364:02) 0.05104
R(D1364:02) 0.1429



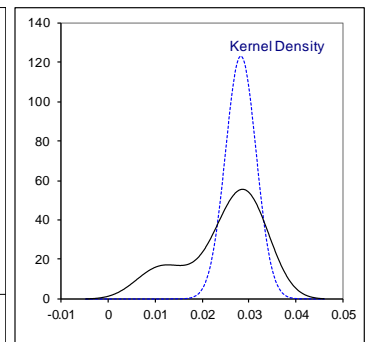
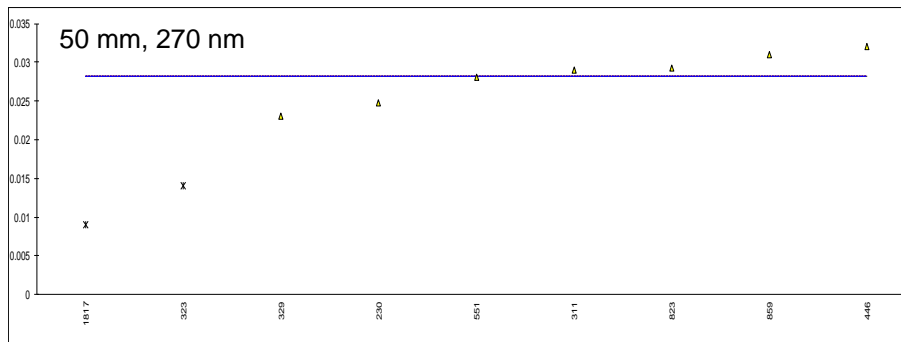
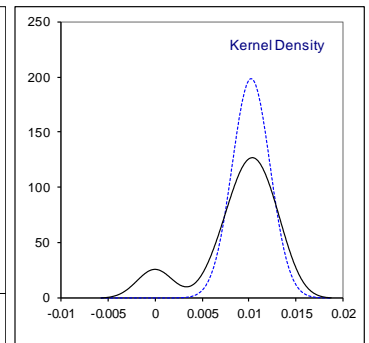
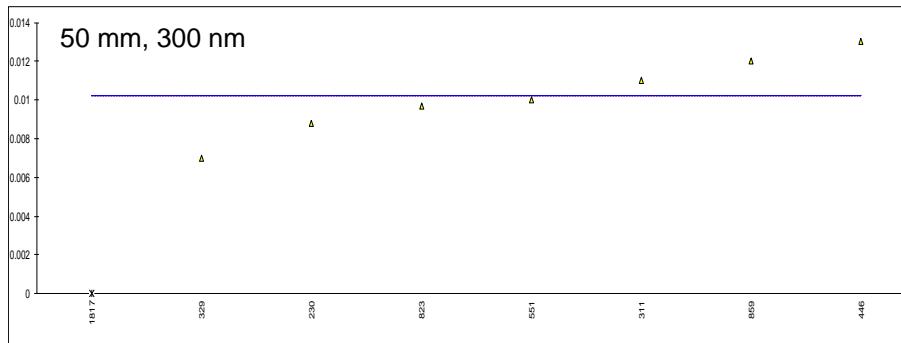
Determination of UV absorbance (50 mm cuvette) on sample #18242;

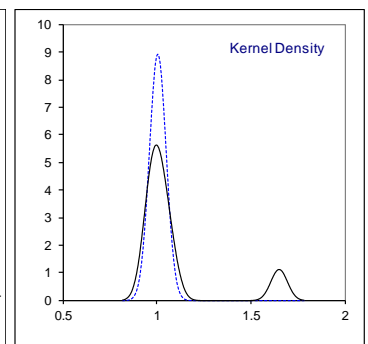
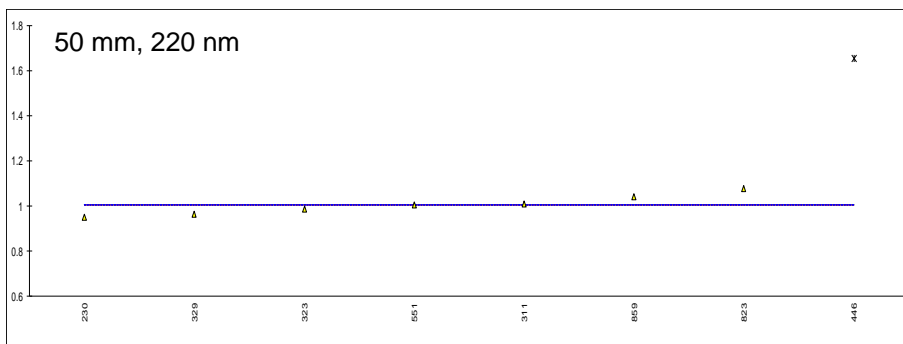
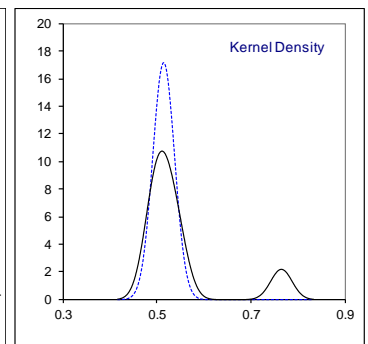
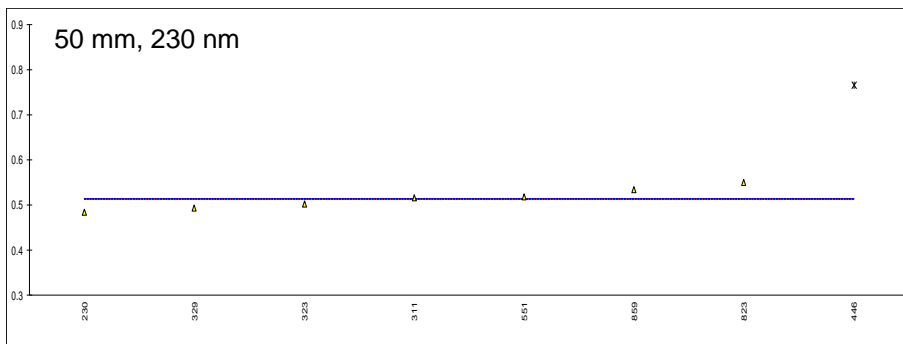
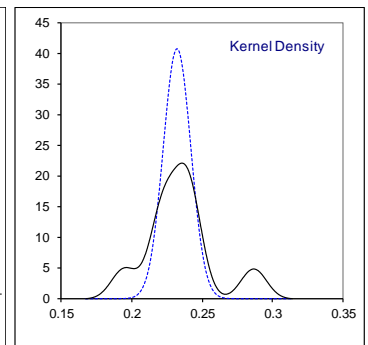
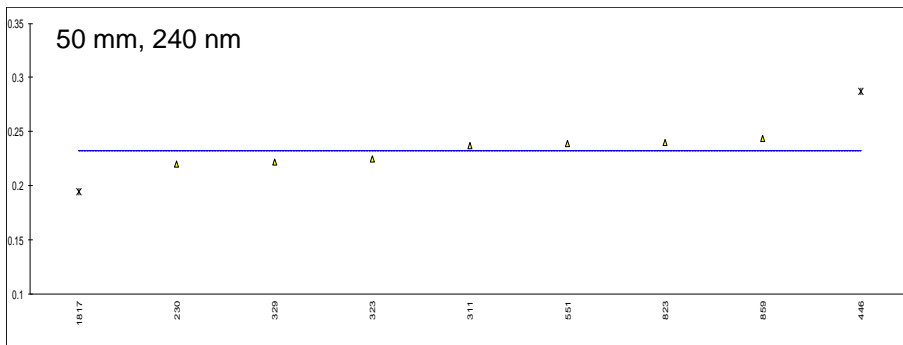
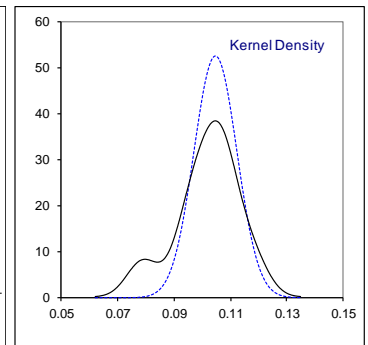
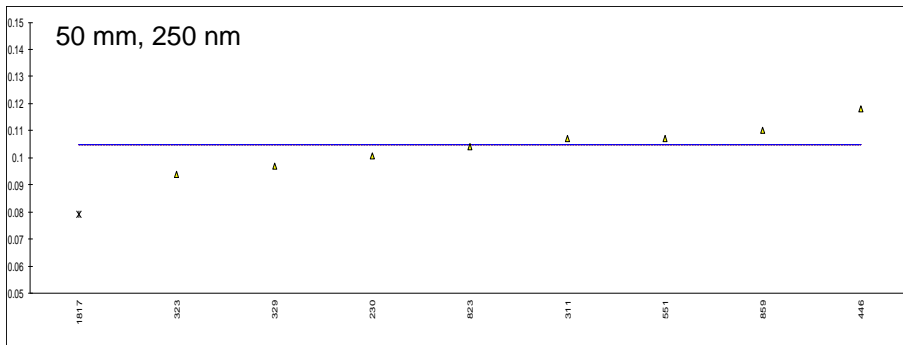
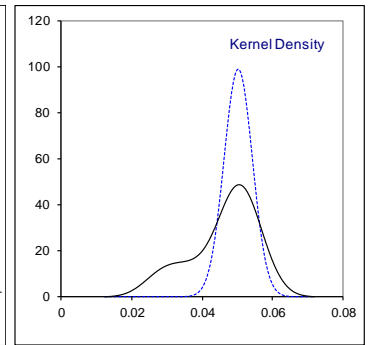
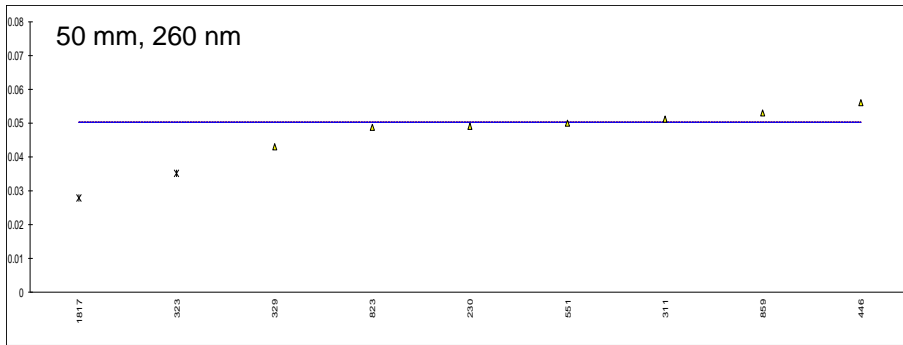
lab	method	300 nm	270 nm	260 nm	250 nm	240 nm	230 nm	220 nm	Pass/Fail
150		----	----	----	----	----	----	----	----
230	INH-13	0.0088	0.0247	0.0490	0.10075	0.2202	0.48405	0.9478	Pass
273		----	----	----	----	----	----	----	----
311	INH-13	0.011	0.029	0.051	0.107	0.237	0.515	1.007	Pass
323	IMPCA004	<0.01	0.014 DG5	0.035 DG5	0.094	0.225	0.503	0.987	Pass
329	INH-CM	0.007	0.023	0.043	0.097	0.222	0.492	0.962	Pass
357		----	----	----	----	----	----	----	----
446	INH-13	0.013	0.032	0.056	0.118	0.287 D5	0.764 G1	1.651 G1	Pass
541		----	----	----	----	----	----	----	----
551	INH-3063	0.010	0.028	0.050	0.107	0.239	0.517	1.005	Pass
823	IMPCA004	0.0097	0.0292	0.0488	0.1041	0.2403	0.5498	1.0769	Pass
859	IMPCA004	0.012	0.031	0.053	0.110	0.244	0.535	1.042	Fail f-?
912		----	----	----	----	----	----	----	----
913		----	----	----	----	----	----	----	----
922		----	----	----	----	----	----	----	----
963		----	----	----	----	----	----	----	----
1205		----	----	----	----	----	----	----	----
1242		----	----	----	----	----	----	----	----
1574		----	----	----	----	----	----	----	----
1605		----	----	----	----	----	----	----	----
1726		----	----	----	----	----	----	----	----
1727		----	----	----	----	----	----	----	----
1817		0 G5	0.009 DG5	0.028 DG5	0.079 ex	0.195 ex	----	----	----
1835		----	----	----	----	----	----	----	----
1927		----	----	----	----	----	----	----	----
normality		unknown	OK	suspect	OK	OK	unknown	unknown	
n		7	7	7	8	7	7	7	7
outliers		1	2	2	0 (+1 ex)	1 (+1 ex)	1	1	1 (Fail)
mean (n)		0.0102	0.0281	0.0501	0.1047	0.2325	0.5137	1.0040	Pass
st.dev. (n)		0.00201	0.00325	0.00403	0.00761	0.00978	0.02322	0.04468	
R(calc.)		0.0056	0.0091	0.0113	0.0213	0.0274	0.0650	0.1251	

Please note: G1, G5, D5 and DG5 means G(0.01), G(0.05), D(0.05) and DG(0.05) respectively

Lab 859: possibly a false negative test result (f-)?

Lab 1817: two test results excluded as three other test results are statistical outliers

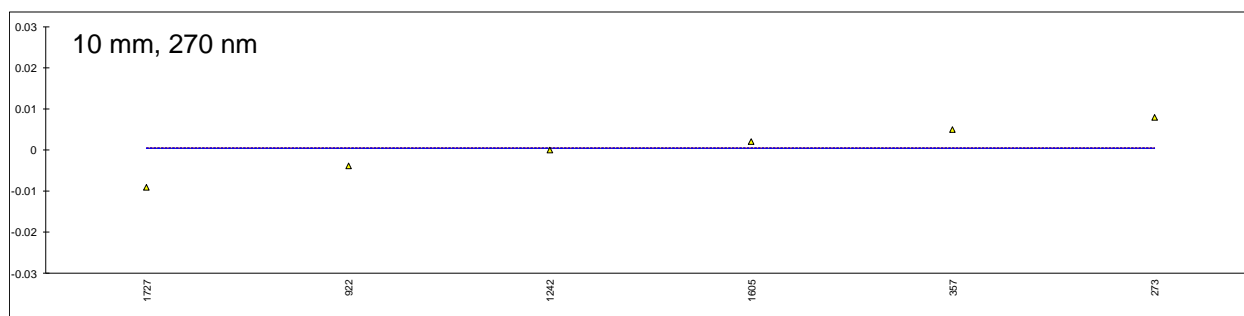
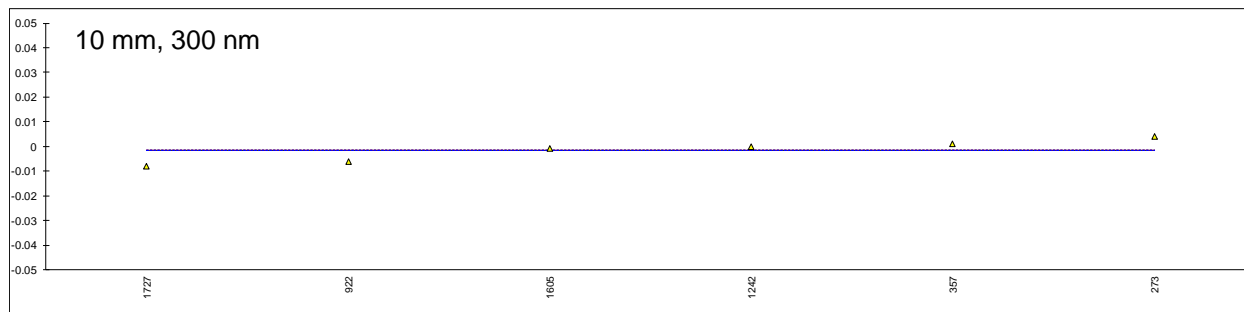


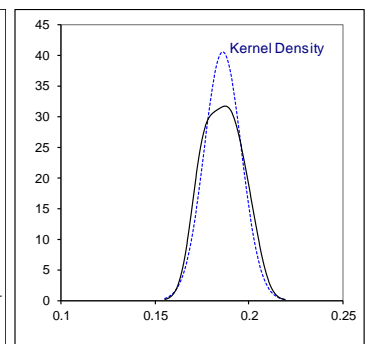
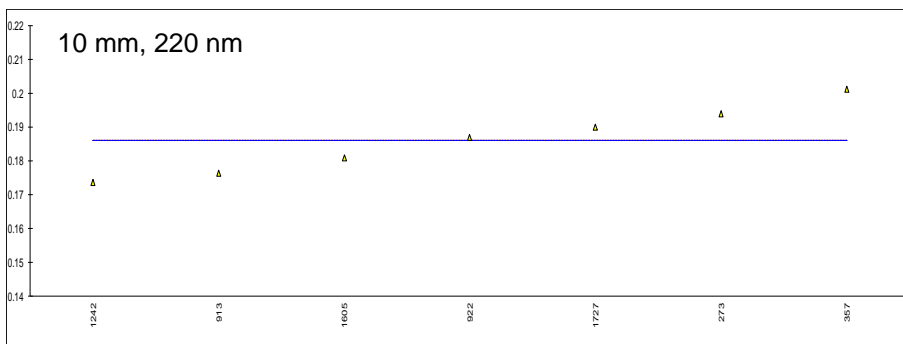
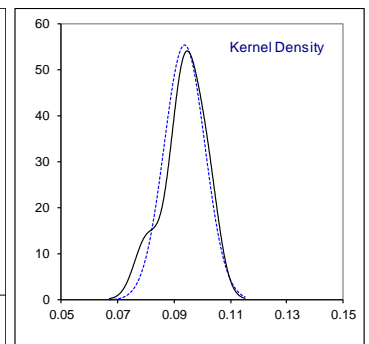
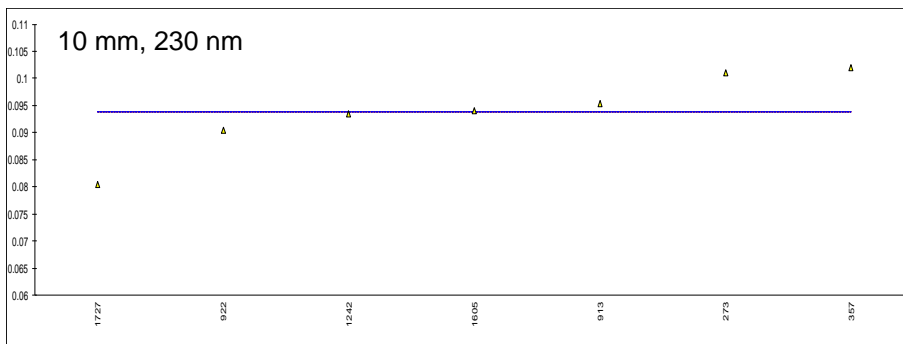
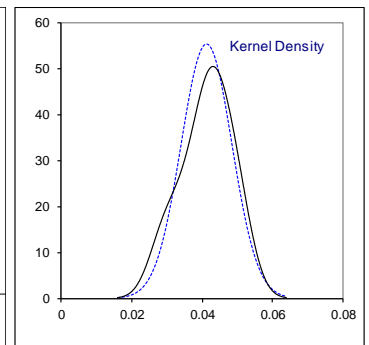
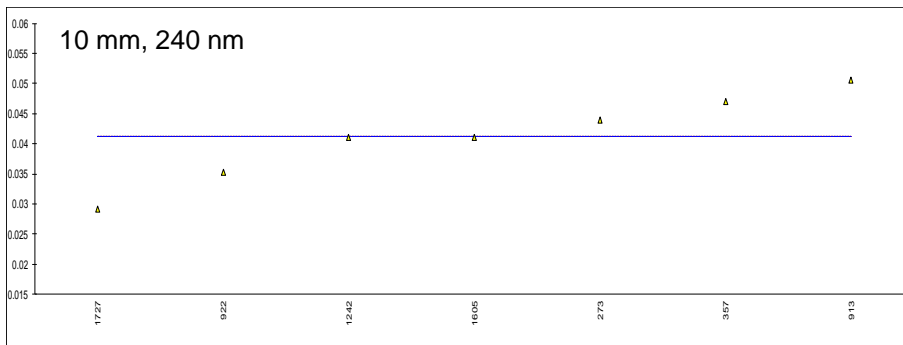
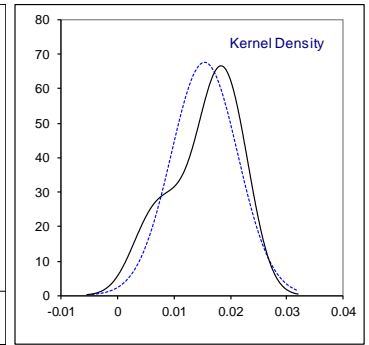
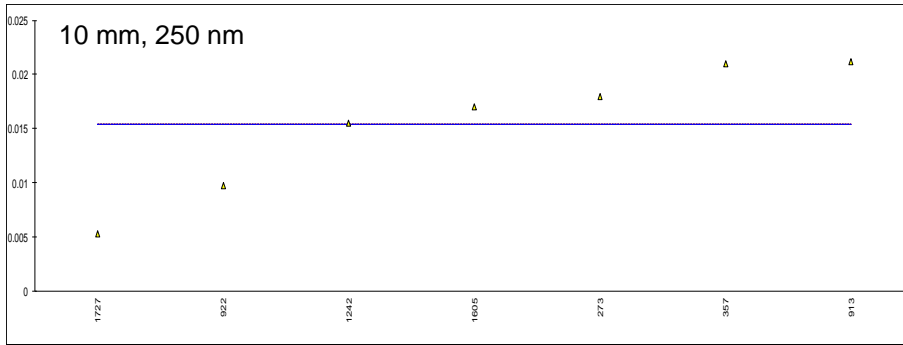
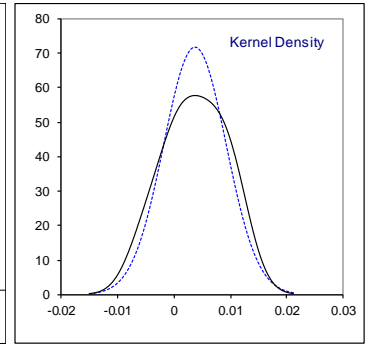
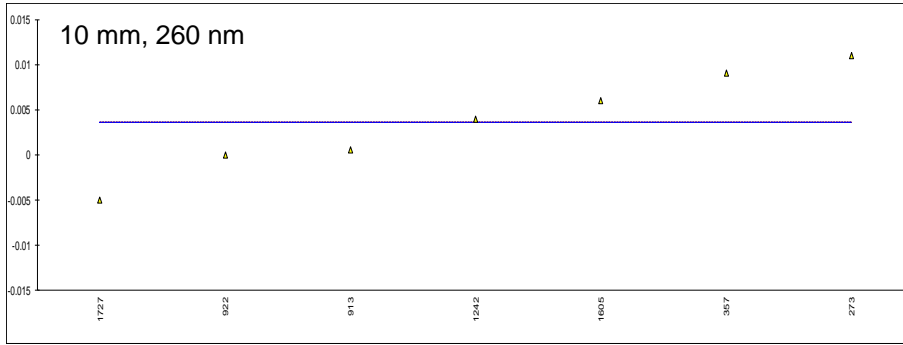


Determination of UV absorbance (10 mm cuvette) on sample #18242;

lab	method	300 nm	270 nm	260 nm	250 nm	240 nm	230 nm	220 nm	Pass/Fail
150		----	----	----	----	----	----	----	----
230		----	----	----	----	----	----	----	----
273	IMPCA004	0.004	0.008	0.011	0.018	0.044	0.101	0.194	----
311		----	----	----	----	----	----	----	----
323		----	----	----	----	----	----	----	----
329		----	----	----	----	----	----	----	----
357	INH-13	0.001	0.005	0.009	0.021	0.047	0.102	0.201	Pass
446		----	----	----	----	----	----	----	----
541		----	----	----	----	----	----	----	----
551		----	----	----	----	----	----	----	----
823		----	----	----	----	----	----	----	----
859		----	----	----	----	----	----	----	----
912		----	----	----	----	----	----	----	----
913	IMPCA004	<0.0001	<0.0001	0.0006	0.0212	0.0506	0.0953	0.1762	Pass
922	In house	-0.006	-0.004	0.000	0.0097	0.0353	0.0904	0.1869	Pass
963		----	----	----	----	----	----	----	----
1205		----	----	----	----	----	----	----	----
1242	In house	0.0000	0.0000	0.0040	0.0155	0.0410	0.0935	0.1735	Pass
1574		----	----	----	----	----	----	----	----
1605		-0.001	0.002	0.006	0.017	0.041	0.094	0.181	----
1726		----	----	----	----	----	----	----	----
1727	IMPCA004	-0.008090	-0.009000	-0.004950	0.005350	0.0291 C	0.0804 C	0.19	Pass
1817		----	----	----	----	----	----	----	----
1835		----	----	----	----	----	----	----	----
1927		----	----	----	----	----	----	----	----
	normality	unknown	unknown	unknown	unknown	unknown	unknown	unknown	
	n	6	6	7	7	7	7	7	5
	outliers	0	0	0	0	0	0	0	
	mean (n)	-0.0017	0.0003	0.0037	0.0154	0.0411	0.0938	0.1861	Pass
	st.dev. (n)	0.00453	0.00615	0.00555	0.00589	0.00720	0.00722	0.00986	
	R(calc.)	0.0127	0.0172	0.0155	0.0165	0.0202	0.0202	0.0276	

Lab 1727: first reported 0.00291, 0.00804 respectively

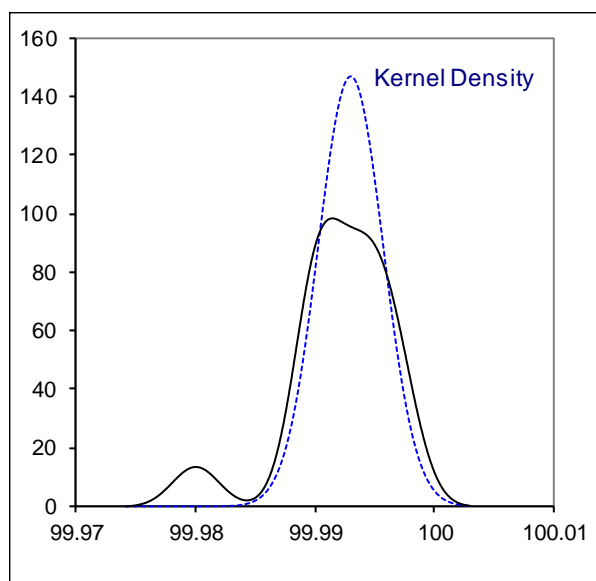
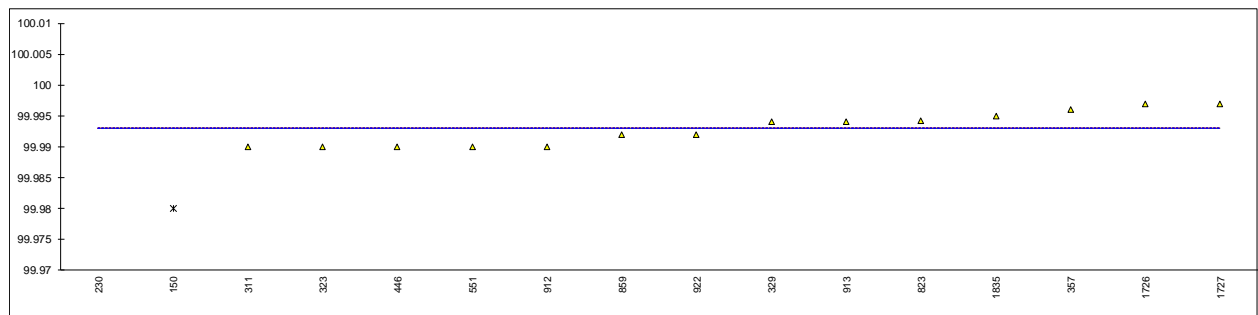




Determination of Purity of Ethanol on dry basis on sample #18243; results in %M/M

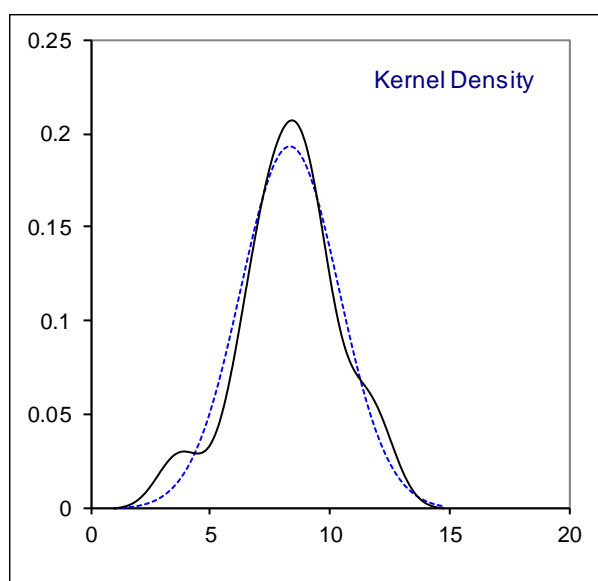
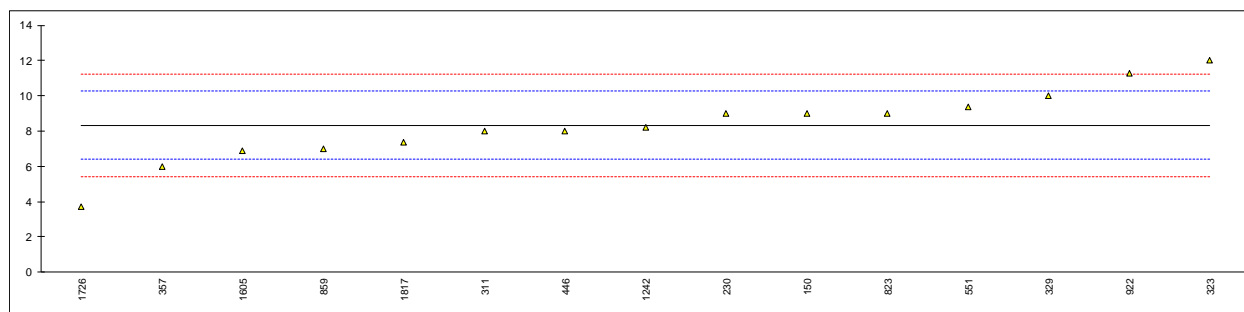
lab	method	value	mark	z(targ)	remarks
150		99.98	G(0.01)	----	
230	INH-0001	94.31	G(0.01)	----	possibly test result not on dry basis?
273		----		----	
311	INH-529	99.99		----	
323	INH-0001	99.99		----	
329	INH-EtOH	99.994		----	
357	INH-0002	99.996		----	
446	INH-CM	99.99		----	
541		----		----	
551	D5501	99.99		----	
823	OINH-0002	99.9942		----	
859	GB/T18350	99.992		----	
912	INH-EtOH	99.99		----	
913		99.994		----	
922	INH-0001	99.992		----	
963		----		----	
1205		----		----	
1242		----		----	
1574		----		----	
1605		----		----	
1726	In house	99.997		----	
1727	EN15721	99.997		----	
1817		----		----	
1835	In house	99.9950		----	
1927		----		----	
	normality	OK			
	n	14			
	outliers	2			
	mean (n)	99.9929			
	st.dev. (n)	0.00271			
	R(calc.)	0.00759			
	st.dev.(lit)	n.a.			
	R(lit)	n.a.			

compare R(iis17C16) = 0.0089 or R(iis16C11) = 0.0181



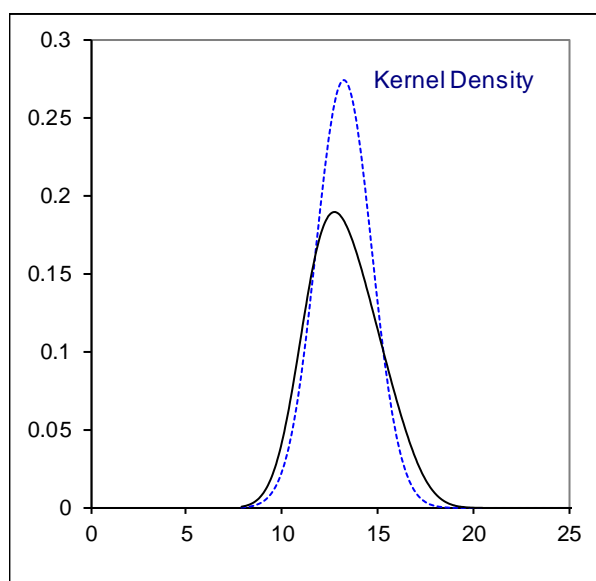
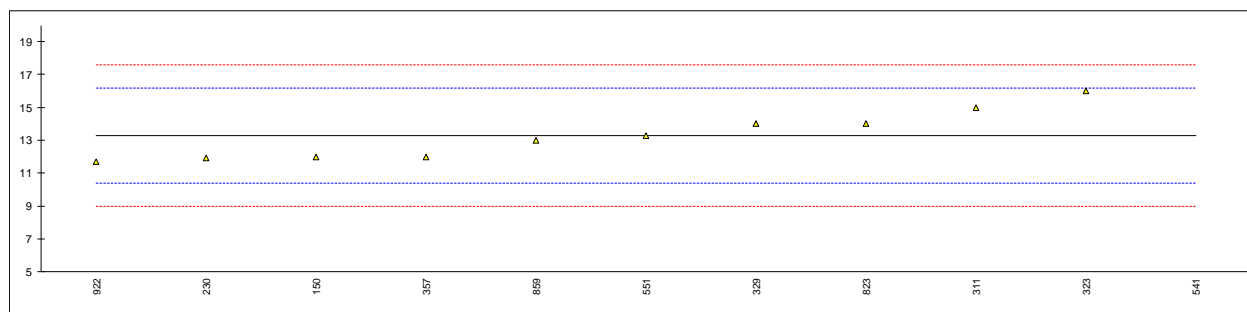
Determination of Methanol on sample #18243; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150		9		0.70	
230	INH-0001	8.995		0.70	
273		----		----	
311	INH-529	8		-0.33	
323	INH-0001	12		3.80	
329	INH-EtOH	10		1.73	
357	INH-0002	6		-2.40	
446	INH-CM	8		-0.33	
541	INH-0002	<5		<-3.43	possibly a false negative test result?
551	INH-1313	9.364		1.08	
823	INH-0002	9		0.70	
859	GB/T18350	7		-1.37	
912		----		----	
913		<5		<-3.43	possibly a false negative test result?
922	INH-0001	11.30	C	3.08	first reported 14.92
963		----		----	
1205		----		----	
1242		8.2240		-0.10	
1574		----		----	
1605		6.89		-1.48	
1726	In house	3.7		-4.78	
1727	EN15721	<10		----	
1817		7.3520		-1.00	
1835	In house	<25		----	
1927		----		----	
				0.70	
	normality	OK			
	n	15			
	outliers	0			
	mean (n)	8.322		spike 15.2	Recovery < 55%
	st.dev. (n)	2.0595			
	R(calc.)	5.767			
	st.dev.(Horwitz)	0.9679			
	R(Horwitz)	2.710			



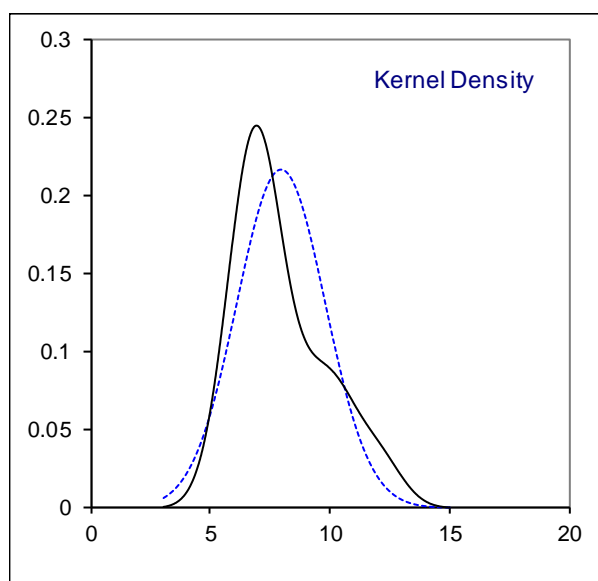
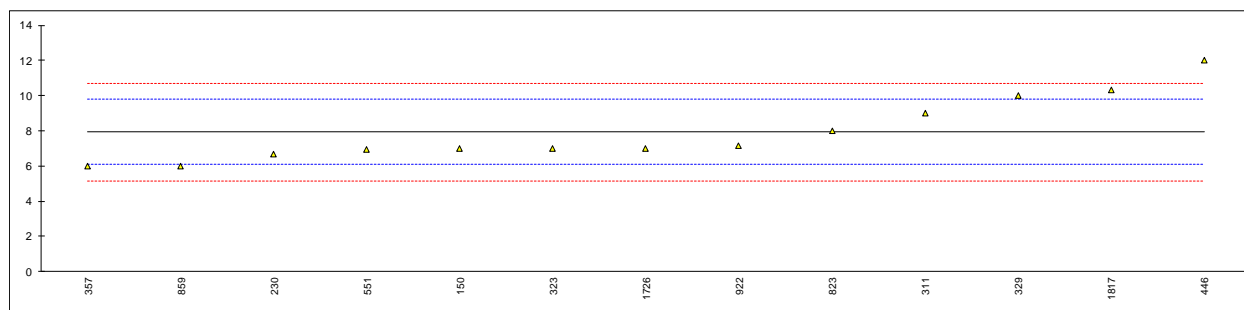
Determination of Acetone on sample #18243; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150		12		-0.90	
230	INH-0001	11.95		-0.93	
273		----		----	
311	INH-529	15		1.19	
323	INH-0001	16		1.88	
329	INH-EtOH	14		0.49	
357	INH-0002	12		-0.90	
446	INH-CM	<5		<-5.75	possibly a false negative test result?
541	INH-0002	35	D(0.01)	15.07	
551	INH-1313	13.261		-0.02	
823	INH-0002	14		0.49	
859	GB/T18350	13		-0.20	
912		----		----	
913		----		----	
922	INH-0001	11.69		-1.11	
963		----		----	
1205		----		----	
1242		----		----	
1574		----		----	
1605		----		----	
1726		----		----	
1727		----		----	
1817		----		----	
1835	In house	<50		----	
1927		----		----	
normality		OK			
n		10			
outliers		1		spike	
mean (n)		13.290		24.6	Recovery < 54%
st.dev. (n)		1.4547			
R(calc.)		4.0731			
st.dev.(Horwitz)		1.4406			
R(Horwitz)		4.034			



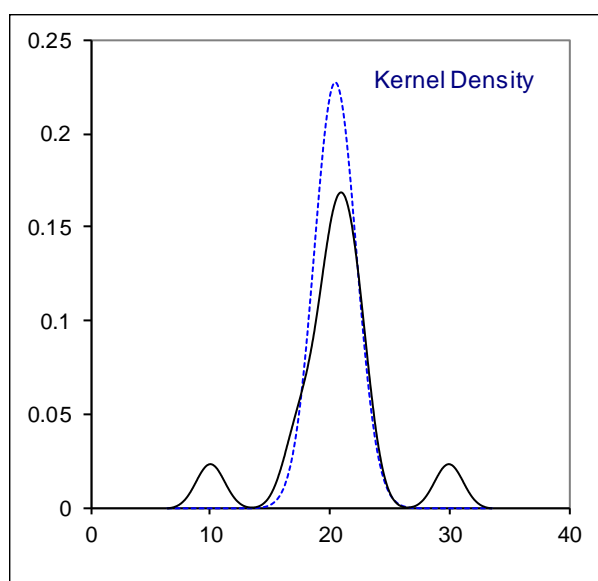
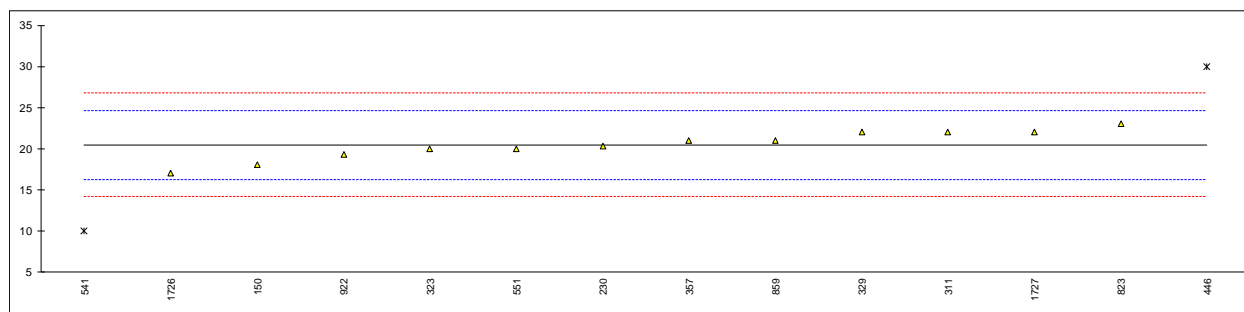
Determination of Benzene on sample #18243; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150		7		-1.00	
230	INH-0001	6.685		-1.34	
273		----		----	
311	INH-529	9		1.15	
323	INH-0001	7		-1.00	
329	INH-EtOH	10		2.23	
357	INH-0002	6		-2.08	
446	INH-CM	12		4.38	
541	INH-0002	<5		<-3.15	possibly a false negative test result?
551	INH-1299	6.92		-1.09	
823	INH-0002	8		0.07	
859	GB/T18350	6		-2.08	
912		----		----	
913		<5		<-3.15	possibly a false negative test result?
922	INH-0001	7.166		-0.82	
963		----		----	
1205		----		----	
1242		----		----	
1574		----		----	
1605		----		----	
1726	In house	7		-1.00	
1727		----		----	
1817		10.3236		2.58	
1835	In house	<10		----	
1927		----		----	
normality		suspect			
n		13			
outliers		0		spike	
mean (n)		7.93		9.8	Recovery < 81%
st.dev. (n)		1.847			
R(calc.)		5.17			
st.dev.(Horwitz)		0.929			
R(Horwitz)		2.60			



Determination of Isopropanol on sample #18243; results in mg/kg

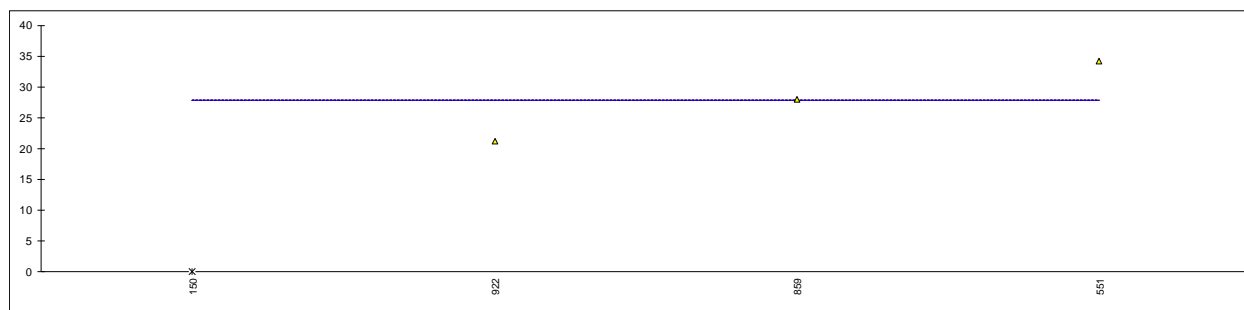
lab	method	value	mark	z(targ)	remarks
150		18		-1.19	
230	INH-0001	20.37		-0.05	
273		----		----	
311	INH-529	22		0.73	
323	INH-0001	20		-0.23	
329	INH-EtOH	22		0.73	
357	INH-0002	21		0.25	
446	INH-CM	30	G(0.01)	4.58	
541	INH-0002	10	G(0.01)	-5.04	
551	INH-1313	20.035		-0.21	
823	INH-0002	23		1.21	
859	GB/T18350	21		0.25	
912		----		----	
913		----		----	
922	INH-0001	19.30		-0.57	
963		----		----	
1205		----		----	
1242		----		----	
1574		----		----	
1605		----		----	
1726	In house	17		-1.67	
1727	EN15721	22		0.73	
1817		----		----	
1835	In house	<25		----	
1927		----		----	
normality		OK			
n		12			
outliers		2		spike	
mean (n)		20.475		29.7	Recovery < 69%
st.dev. (n)		1.7559			
R(calc.)		4.917			
st.dev.(Horwitz)		2.0797			
R(Horwitz)		5.823			



Determination of Monoethylene glycol (MEG) on sample #18243; results in mg/kg

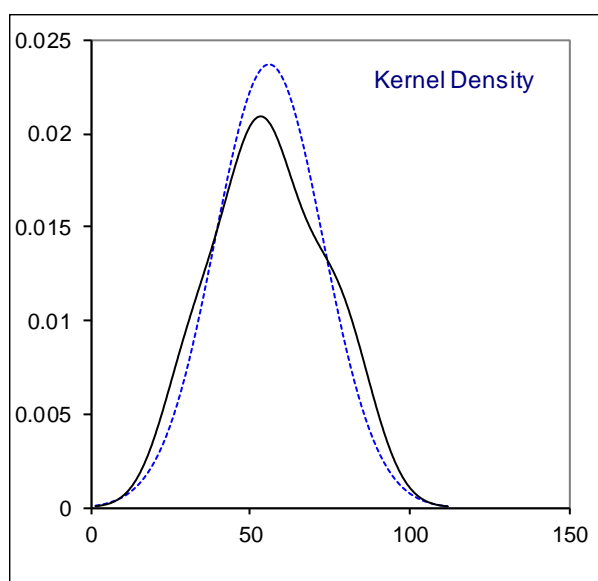
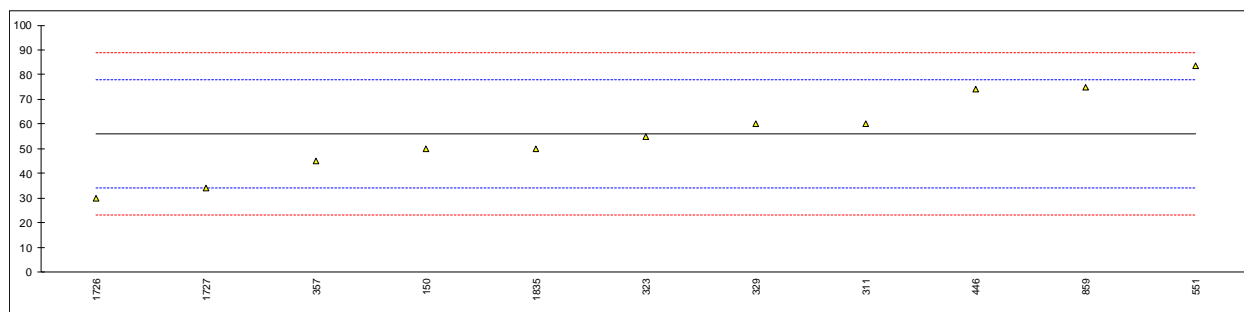
lab	method	value	mark	z(targ)	remarks
150		0	ex	----	possibly a false negative test result? excluded as 0 is not a real test result
230		----		----	
273		----		----	
311		----		----	
323	INH-0001	<5		----	possibly a false negative test result?
329		----		----	
357	INH-0002	< 30		----	
446		----		----	
541		----		----	
551	INH-1379	34.141		----	
823		----		----	
859	GB/T18350	28		----	
912		----		----	
913		----		----	
922	INH-0001	21.146		----	
963		----		----	
1205		----		----	
1242		----		----	
1574		----		----	
1605		----		----	
1726		----		----	
1727		----		----	
1817		----		----	
1835		----		----	
1927		----		----	

normality unknown
 n 3
 outliers 0 (+1 ex) spike
 mean (n) 27.762 29.9 Recovery < 93%
 st.dev. (n) 6.5008
 R(calc.) 18.202
 st.dev.(lit) n.a.
 R(lit) n.a.



Determination of Total impurities on sample #18243; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150		50		-0.55	
230		-----		-----	
273		-----		-----	
311	INH-529	60		0.36	
323	INH-0001	55		-0.10	
329	INH-EtOH	60		0.36	
357	INH-0002	45		-1.01	
446	INH-CM	74		1.64	
541		-----		-----	
551	INH-1313	83.721		2.53	
823		-----		-----	
859	GB/T18350	75		1.73	
912		-----		-----	
913		-----		-----	
922		-----		-----	
963		-----		-----	
1205		-----		-----	
1242		-----		-----	
1574		-----		-----	
1605		-----		-----	
1726	In house	30		-2.38	
1727	EN15721	34		-2.02	
1817		-----		-----	
1835	In house	50		-0.55	
1927		-----		-----	
normality		OK			
n		11			
outliers		0			
mean (n)		56.066			
st.dev. (n)		16.8505			
R(calc.)		47.182			
st.dev.(Horwitz, comp:5)		10.9420			
R(Horwitz, comp:5)		30.638			



APPENDIX 2**Other reported impurities in sample #18243; results in mg/kg**

lab	Acetal	Acetaldehyde	Other impurities
150	0	4	----
230	----	0.0	----
273	----	----	----
311	<1	<1	<5
323	<5	<5	<5
329	<2	<2	<5
357	< 5	< 5	< 5
446	<5	<5	23
541	----	<5	----
551	<6	<6	<6
823	<5	<5	----
859	<5	<5	<5
912	----	----	----
913	<5	<5	----
922	<5.0	<5.0	----
963	----	----	----
1205	----	----	----
1242	----	----	----
1574	----	----	----
1605	< 0.1	< 0.1	----
1726	----	----	2
1727	<10	<10	12
1817	ND	<1	<300
1835	<25	<25	<50
1927	----	----	----

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
2 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
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= Safety Data Sheet

Literature:

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- 3 ASTM E178:02
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- 7 ISO 13528
- 8 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
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- 10 IP 367/84
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- 14 Analytical Methods Committee Technical brief, No 4. January 2001
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- 16 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), 165-172, (1983)