Results of Proficiency Test
Ethanol (Food/Neutral)
December 2017

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

Authors: ing. A. Lewinska
Correctors: dr. R.G. Visser & ing. A.S. Noordman-de Neef

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CONTENTS

1 INTRODUCTION

Since 2007, a proficiency test for Food/Neutral grade Ethanol is organized every year by the Institute for Interlaboratory Studies. During the planning of the annual proficiency testing program 2017/2018, it was decided to continue the round robin for the analysis of Food/Neutral grade Ethanol.
In this interlaboratory study 29 laboratories in 20 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2017 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 organizer of this proficiency test. Sample analysis 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 #17242) and a 0.25 L bottle (labelled #17243) for GC determination only. Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the

identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

The necessary bulk material for sample #17242 was obtained from a local trader. The approximately 29 litres bulk material was homogenised in a pre-cleaned drum. After homogenisation in a pre-cleaned drum, 58 amber glass bottles of 0.5 L were filled and labelled #17242. The homogeneity of these subsamples was checked by determination of Density in accordance with ASTM D4052 on 8 stratified randomly selected samples.

Sample	*Density at 20°C in kg/L*
Sample #17242-1	0.80610
Sample #17242-2	0.80610
Sample #17242-3	0.80610
Sample #17242-4	0.80610
Sample #17242-5	0.80611
Sample #17242-6	0.80610
Sample #17242-7	0.80610
Sample #17242-8	0.80611

Table 1: homogeneity test results of subsamples #17242

From the test results of table 1, the repeatability was calculated and compared with 0.3 times the corresponding target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density at 20°C in kg/L
r (observed)	0.00001
reference test method	ISO12185:96
0.3 * R (reference test method)	0.00015

Table 2: repeatability of subsamples #17242

The calculated repeatability was less than 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

The necessary bulk material for sample #17243 was obtained from an European supplier. To approximately 13 kg of this material, the following components were added:

Component	*Amount in mg/kg*
Methanol	20
Acetone	20
Isopropanol	20

Table 3: preparation table for sample #17243

After homogenisation, 64 amber glass bottles of 0.25 L were filled and labelled #17243. The homogeneity of these subsamples was checked by determination of Isopropanol on 7 stratified randomly selected samples.

	IPA mg/kg
Sample #17243-1	20.6
Sample #17243-2	20.1
Sample #17243-3	19.2
Sample #17243-4	19.4
Sample #17243-5	18.6
Sample #17243-6	19.1
Sample #17243-7	18.9

Table 4: homogeneity test results of subsamples #17243

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.

	IPA mg/kg
r (observed)	1.96
reference	Horwitz
0.3 x R (reference)	1.67

Table 5: evaluation of the repeatabilities of subsamples #17243

The calculated repeatability was almost in agreement with 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the subsamples of #17243 was assumed.

To each of the participating laboratories 1*0.5 L bottle of sample #17242 and 1*0.25 L bottle #17243 was sent on November 7, 2017. 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 #17242: 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 220nm with an evaluation of the UV-scan.

The participants were asked to determine on sample #17243: Purity Ethanol on dry basis, Methanol, Acetal, Acetaldehyde, Acetone, Benzene, Isopropanol, Mono Ethylene 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 calculations.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment.
Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalysis). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the 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 March 2017 (iis-protocol, version 3.4).
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. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.
Finally, the reproducibilities were calculated from the standard deviations by multiplying 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:

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

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

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

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

4 EVALUATION

In this proficiency test, some problems were encountered with the dispatch of the samples. Two participants reported the test results after the final reporting date. Not all laboratories were able to report all analyses requested. In total 29 laboratories reported 301 numerical results. Observed were 22 outlying results, which is 7.3%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER SAMPLE AND PER TEST

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

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

In the iis PT reports, ASTM methods are referred to with a number (e.g. 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 #17242:

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

Nonvolatile matter: The determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D1353:13.

Permanganate Time Test: The determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D1363:06(2011).

pHe: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D6423:14.

Strength (%M/M): This determination may not be problematic. No statistical outlier was observed. The calculated reproducibility is in agreement with the reproducibility derived from the OIML table and ISO12185:96.

Strength (%V/V): This determination may not be problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the reproducibility derived from the OIML table and ISO12185:96.

Water: This determination was problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D1364:02(2012).

UV absorbance: Regretfully, no Standard Test Method for this determination exists. Some participants reported results obtained with a 50 mm cuvette, others with a 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 is preferable. Unfortunately, only seven laboratories used a 50 mm cuvette and nine laboratories used a 10 mm cuvette. Both groups were evaluated separately.

UV - 50 mm cuvette: In total, one statistical outlier was observed. All laboratories evaluated the sample as ‘Pass’.

UV - 10 mm cuvette: In total, eleven statistical outliers were observed and three test results were excluded. All laboratories evaluated the sample as ‘Pass’.

Sample #17243:

Purity on dry basis: Regretfully, no Standard Method is available that gives a clear definition of purity in Ethanol Food/Neutral grade. Therefore, no significant conclusions could be drawn. No statistical outliers were observed. The calculated reproducibility is smaller than the calculated reproducibility in the previous proficiency tests iis16C11 and iis15C15.

Methanol: This determination may be problematic. Two statistical outliers were observed and one possible false negative test result was reported. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility using the Horwitz equation.

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

Isopropanol (IPA): This determination may not be problematic. One statistical outlier was observed, but two possible false negative test results were reported. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility using the Horwitz equation.

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

Other impurities: For Acetal, Acetaldehyde, Benzene, Monoethylene glycol (MEG) and Other impurities the majority of participants reported a result <10 mg/kg and <25 mg/kg.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant reference test method and the reproducibility as found for the group of participating laboratories. The target reproducibilities derived from literature reference test method or previous proficiency tests are compared in the next tables, the UV results can be found on the next page.

Parameter	unit	n	average	2.8 *sd	R (lit)
Density at 20°C	kg/L	26	0.8061	0.0001	0.0005
Nonvolatile matter	mg/100mL	10	0.5	1.0	2.1
Permanganate Time Test	min.	12	32.5	9.4	8.2
pHe		11	7.6	1.9	1.1
Strength	%M/M	17	94.30	0.04	0.06
Strength	%V/V	24	96.32	0.03	0.06
Water (titrimetric)	%M/M	16	5.70	0.24	0.14
UV – 50 mm cuvette:					
UV-absorbance 300 nm		5	0.011	0.028	n.a.
UV-absorbance 270 nm		6	0.025	0.029	n.a.
UV-absorbance 260 nm		7	0.043	0.035	n.a.
UV-absorbance 250 nm		7	0.097	0.033	n.a.
UV-absorbance 240 nm		7	0.222	0.040	n.a.
UV-absorbance 230 nm		6	0.490	0.077	n.a.
UV-absorbance 220 nm		5	0.982	0.052	n.a.
Conclusion UV-scan	Pass/Fail	8	Pass	n.a.	n.a.
UV – 10 mm cuvette:					
UV-absorbance 300 nm		4	0.0002	0.0009	n.a.
UV-absorbance 270 nm		8	0.002	0.003	n.a.
UV-absorbance 260 nm		9	0.007	0.004	n.a.
UV-absorbance 250 nm		9	0.017	0.005	n.a.
UV-absorbance 240 nm		9	0.042	0.008	n.a.
UV-absorbance 230 nm		9	0.096	0.013	n.a.
UV-absorbance 220 nm		9	0.188	0.024	n.a.
Conclusion UV-scan	Pass/Fail	8	pass	n.a.	n.a.

Table 6: reproducibilities of tests on sample #17242

Parameter	unit	n	average	2.8 *sd	R (lit)
Purity EtOH on dry basis	%M/M	15	99.99	0.01	n.a.
Methanol	mg/kg	12	13.91	5.0	4.2
Acetone	mg/kg	12	11.9	6.5	3.7
Isopropanol	mg/kg	12	15.3	3.8	4.5
Total impurities	mg/kg	9	45.4	32.6	19.9

Table 7: reproducibilities of UV tests on sample #17243

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2017 WITH PREVIOUS PTS

	December 2017	*December 2016*	*November 2015*	*November 2014*	*November 2013*
Number of reporting labs	29	26	32	25	24
Number of results reported	301	329	254	210	160
Number of statistical outliers	22	16	11	13	9
Percentage outliers	7.3%	4.9%	4.3%	6.2%	5.6%

Table 8: 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 2017	December 2016	November 2015	November 2014	November 2013
Density at 20°C	++	++	++	++	++
Nonvolatile matter	++	++	++	n.e.	++
Permanganate Time Test	-	(+)	-	+	--
pHe	-	(-)	--	n.e.	n.e.
Strength %M/M	++	(+/-)	(+)	(+)	(--)
Strength %V/V	++	++	+	+	--
Water (titrimetric)	-	-	-	--	--
Purity EtOH on dry basis	(--)	(--)	(-)	(-)	(+)
Methanol	-	n.e.	n.e.	n.e.	n.e.
Acetal	n.e.	n.e.	n.e.	n.e.	n.e.
Benzene	n.e.	n.e.	n.e.	n.e.	n.e.
Mono Ethylene Glycol	n.e.	n.e.	n.e.	n.e.	n.e.
UV-absorbance 300 nm	n.e.	(+/-)	(-)	(++)	(-)
UV-absorbance 270 nm	n.e.	n.e.	(++)	(+/-)	(--)
UV-absorbance 260 nm	n.e.	n.e.	n.e.	n.e.	n.e.
UV-absorbance 250 nm	n.e.	n.e.	n.e.	n.e.	n.e.
UV-absorbance 240 nm	n.e.	n.e.	(-)	(++)	(+/-)
UV-absorbance 230 nm	n.e.	n.e.	(+)	(-)	(++)
UV-absorbance 220 nm	n.e.	n.e.	(++)	(--)	(--)

Table 9: comparison determinations of sample #17242 and #17243 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:

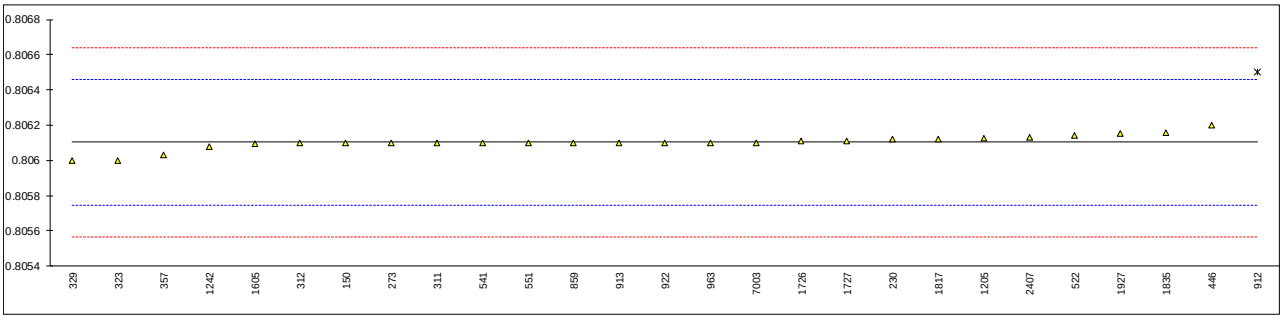
++:	group performed much better than the reference test method
+ :	group performed better than the reference test method
+/-:	group performance equals the reference test method
- :	group performed worse than the reference test method
-- :	group performed much worse than the reference test method
n.e.: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

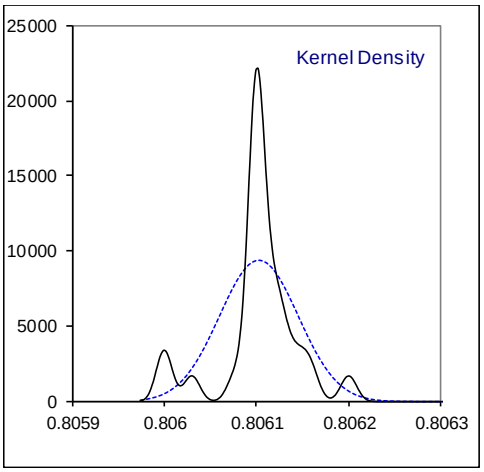
APPENDIX 1

Determination of Density at 20°C on sample #17242; results in kg/L

lab	method	value	mark	z(targ)	remarks
150	D4052	0.8061		-0.01	
230	D4052	0.80612		0.10	
273	D4052	0.8061		-0.01	
311	D4052	0.8061		-0.01	
312	ISO12185	0.8061		-0.01	
323	D4052	0.8060		-0.57	
329	D4052	0.8060		-0.57	
357	D4052	0.80603		-0.41	
446	D4052	0.8062		0.55	
522	D4052	0.80614		0.21	
541	D4052	0.80610		-0.01	
551	D4052	0.8061		-0.01	
859	D4052	0.8061		-0.01	
912	D4052	0.8065	D(0.01)	2.23	
913	D4052	0.8061		-0.01	
922	D4052	0.8061		-0.01	
963	D4052	0.8061		-0.01	
1205	In house	0.806125		0.13	
1242	In house	0.806077		-0.14	
1438		-----		-----	
1574		-----		-----	
1605	D4052	0.806094		-0.05	
1726	D4052	0.80611		0.04	
1727	D4052	0.80611		0.04	
1817	In house	0.80612		0.10	
1835	ISO12185	0.80616		0.32	
1927	D4052	0.80615		0.27	
2407	ISO12185	0.80613		0.15	
7003	D4052	0.8061		-0.01	

normality	not OK
n	26
outliers	1
mean (n)	0.806103
st.dev. (n)	0.0000427
R(calc.)	0.000119
st.dev.(ISO12185:96)	0.0001786
R(ISO12185:96)	0.0005

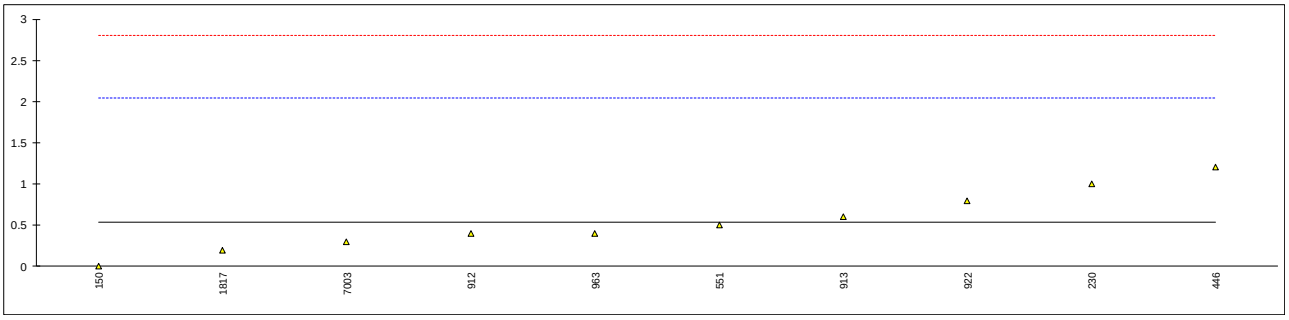


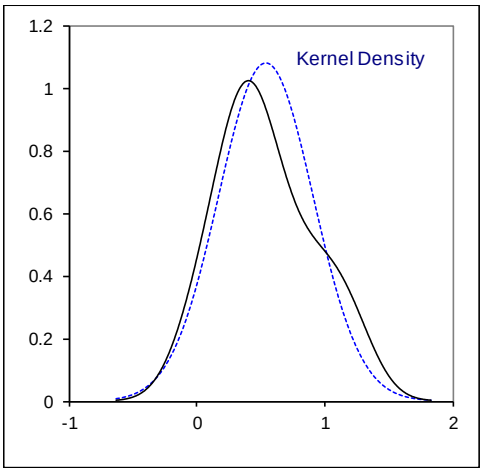


Determination of Nonvolatile matter on sample #17242; results in mg/100mL

lab	method	value	mark	z(targ)	remarks
150	D1353	0.0		-0.72	
230	D1353	1.0		0.61	
273		-----		-----	
311	D1353	<1		-----	
312	INH-90	<1.0		-----	
323	D1353	< 1		-----	
329	D1353	<1		-----	
357	D1353	<1		-----	
446	D1353	1.2		0.88	
522		-----		-----	
541	D1353	<0.1		-----	
551	D1353	0.5		-0.05	
859	D1353	<1		-----	
912	D1353	0.4		-0.19	
913	D1353	0.6		0.08	
922	D1353	0.8		0.35	
963	D1353	0.4		-0.19	
1205		-----		-----	
1242		-----		-----	
1438		-----		-----	
1574		-----		-----	
1605		-----		-----	
1726	EN15691	<10		-----	
1727	EN15691	<1		-----	
1817		0.2		-0.45	
1835	EN15691	<10		-----	
1927		-----		-----	
2407		-----		-----	
7003	D1353	0.3		-0.32	

normality	OK
n	10
outliers	0
mean (n)	0.54
st.dev. (n)	0.369
R(calc.)	1.03
st.dev.(D1353:13)	0.754
R(D1353:13)	2.11

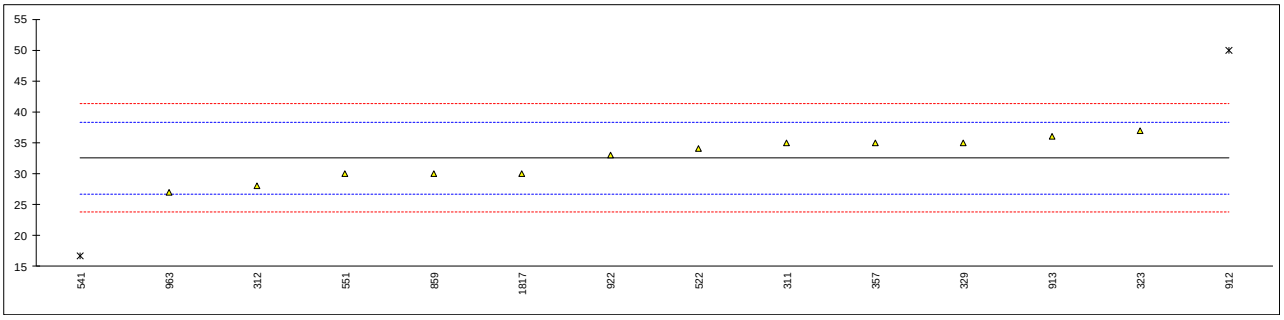


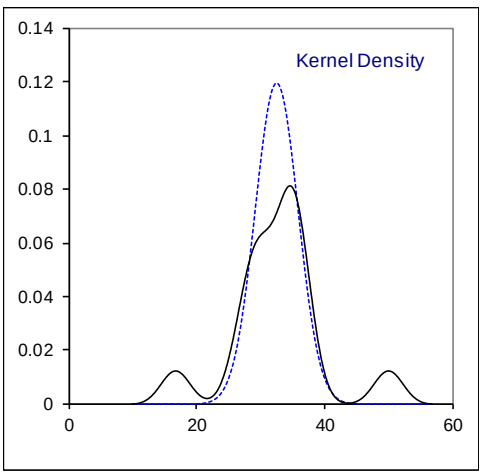


Determination of Permanganate Time Test at 20°C on sample #17242; results in minutes

lab	method	value	mark	z(targ)	remarks
150	D1363	<35		-----	
230	D1363	<50		-----	
273		-----		-----	
311	D1363	35		0.85	
312	INH-90	28		-1.54	
323	D1363	37		1.54	
329	D1363	35		0.85	
357	D1363	35		0.85	
446	BS6392-9	>30		-----	
522	D1363	34		0.51	
541	D1363	16.7	D(0.05)	-5.40	
551	D1363	30		-0.85	
859	D1363	30		-0.85	
912	D1363	50	D(0.05)	5.98	
913	D1363	36	C	1.20	first reported 50
922	D1363	33		0.17	
963	D1363	27		-1.88	
1205		-----		-----	
1242		-----		-----	
1438		-----		-----	
1574		-----		-----	
1605		-----		-----	
1726		-----		-----	
1727		-----		-----	
1817		30		-0.85	
1835		-----		-----	
1927		-----		-----	
2407		-----		-----	
7003		-----		-----	

normality	OK
n	12
outliers	2
mean (n)	32.50
st.dev. (n)	3.344
R(calc.)	9.36
st.dev.(D1363:06)	2.925
R(D1363:06)	8.19

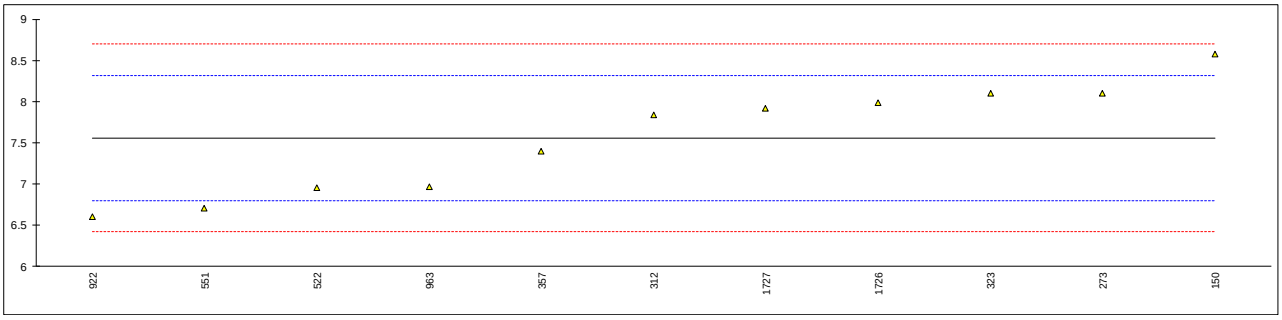


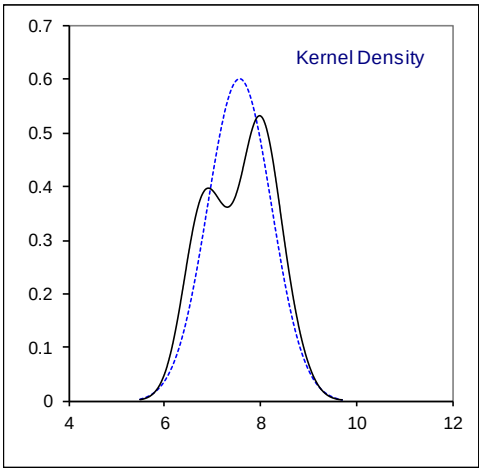


Determination of pHe on sample #17242;

lab	method	Electrode	value	mark	z(targ)	remarks
150	D6423	KCl	8.58		2.70	
230			-----		-----	
273	D6423		8.1		1.43	
311			-----		-----	
312	D6423	Orion	7.84		0.74	
323	D6423	LiCl	8.1		1.43	
329			-----		-----	
357	D6423	LiCl	7.4		-0.42	
446			-----		-----	
522	D6423	KCl	6.95		-1.61	
541			-----		-----	
551	NBR10891	LiCl	6.7		-2.27	
859			-----		-----	
912			-----		-----	
913			-----		-----	
922	D6423	KCl	6.6		-2.53	
963	D6423	KCl	6.97		-1.55	
1205			-----		-----	
1242			-----		-----	
1438			-----		-----	
1574			-----		-----	
1605			-----		-----	
1726	EN15490	LiCl	7.98		1.11	
1727	EN15490	LiCl	7.92		0.96	
1817			-----		-----	
1835			-----		-----	
1927			-----		-----	
2407			-----		-----	
7003			-----		-----	

normality	OK
n	11
outliers	0
mean (n)	7.558
st.dev. (n)	0.6645
R(calc.)	1.860
st.dev.(D6423:14)	0.3788
R(D6423:14)	1.0607

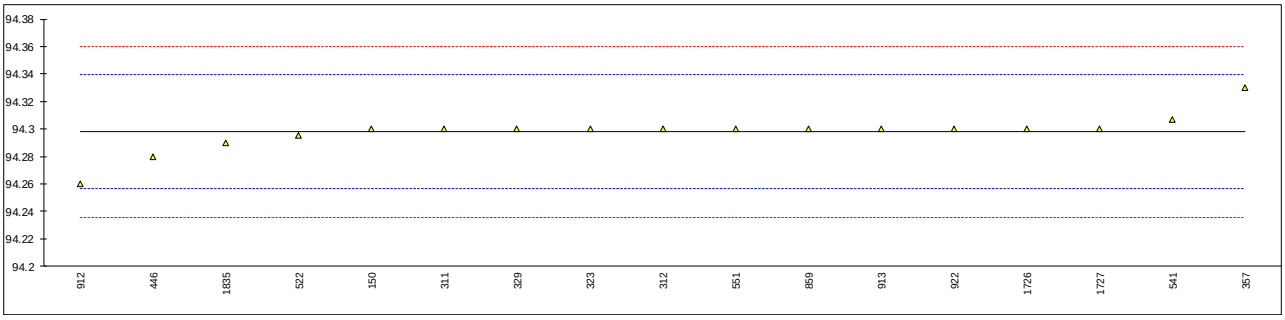


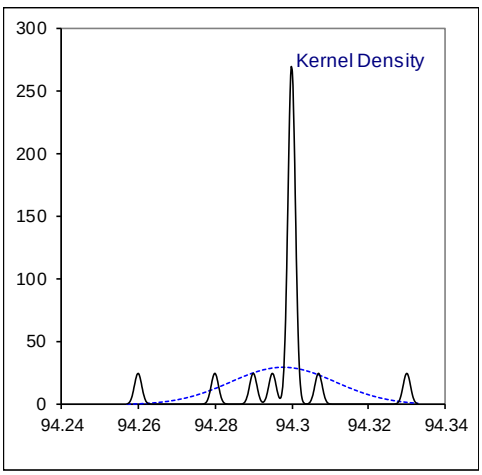


Determination of Strength on sample #17242; results in %M/M

lab	method	value	mark	z(targ)	remarks
150	Table OIML	94.3		0.11	
230		-----		-----	
273		-----		-----	
311	Table OIML	94.30		0.11	
312	Table OIML	94.30		0.11	
323	Table OIML	94.30		0.11	
329	Table OIML	94.30		0.11	
357	Table OIML	94.33		1.56	
446	Table OIML	94.28		-0.86	
522	Table OIML	94.295		-0.13	
541	Table OIML	94.307		0.45	
551	NBR15639	94.3		0.11	
859	Table OIML	94.30		0.11	
912	Table OIML	94.26	C	-1.82	first reported 94.15
913	Table OIML	94.30		0.11	
922	Table OIML	94.30		0.11	
963		-----		-----	
1205		-----		-----	
1242		-----		-----	
1438		-----		-----	
1574		-----		-----	
1605		-----		-----	
1726	Table OIML	94.30		0.11	
1727	Table OIML	94.30		0.11	
1817		-----		-----	
1835	Table OIML	94.29		-0.38	
1927		-----		-----	
2407		-----		-----	
7003		-----		-----	

normality	not OK
n	17
outliers	0
mean (n)	94.298
st.dev. (n)	0.0137
R(calc.)	0.038
st.dev.(OIML table)	0.0207
R(OIML table)	0.058

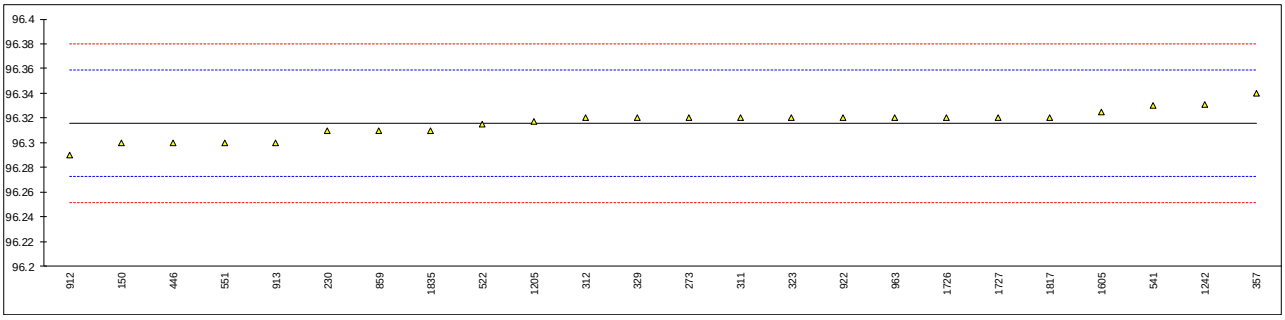


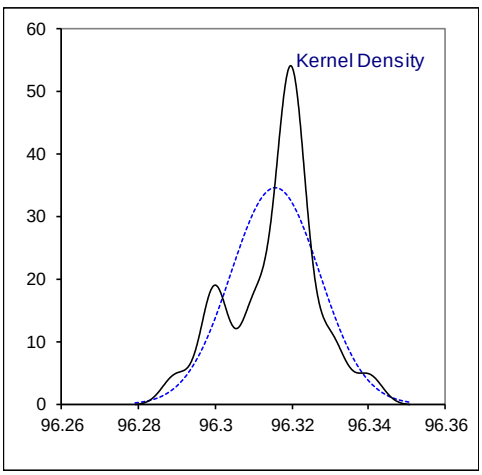


Determination of Strength on sample #17242; results in %V/V

lab	method	value	mark	z(targ)	remarks
150	Table OIML	96.3		-0.73	
230	Table OIML	96.31		-0.27	
273	Table OIML	96.32		0.20	
311	Table OIML	96.32		0.20	
312	Table OIML	96.32		0.20	
323	Table OIML	96.32		0.20	
329	Table OIML	96.32		0.20	
357	Table OIML	96.34		1.13	
446	Table OIML	96.30		-0.73	
522	Table OIML	96.315		-0.03	
541	Table OIML	96.330		0.67	
551	NBR15639	96.3		-0.73	
859	Table OIML	96.31		-0.27	
912	Table OIML	96.29	C	-1.20	first reported 96.23
913	Table OIML	96.30		-0.73	
922	Table OIML	96.32		0.20	
963	Table OIML	96.32		0.20	
1205	Table OIML	96.317		0.06	
1242	In house	96.331		0.71	
1438		-----		-----	
1574		-----		-----	
1605	Table OIML	96.325		0.43	
1726	Table OIML	96.32		0.20	
1727	Table OIML	96.32		0.20	
1817	Table OIML	96.32		0.20	
1835	Table OIML	96.31		-0.27	
1927		-----		-----	
2407		-----		-----	
7003		-----		-----	

normality	OK
n	24
outliers	0
mean (n)	96.316
st.dev. (n)	0.0115
R(calc.)	0.032
st.dev.(OIML table)	0.0214
R(OIML table)	0.060

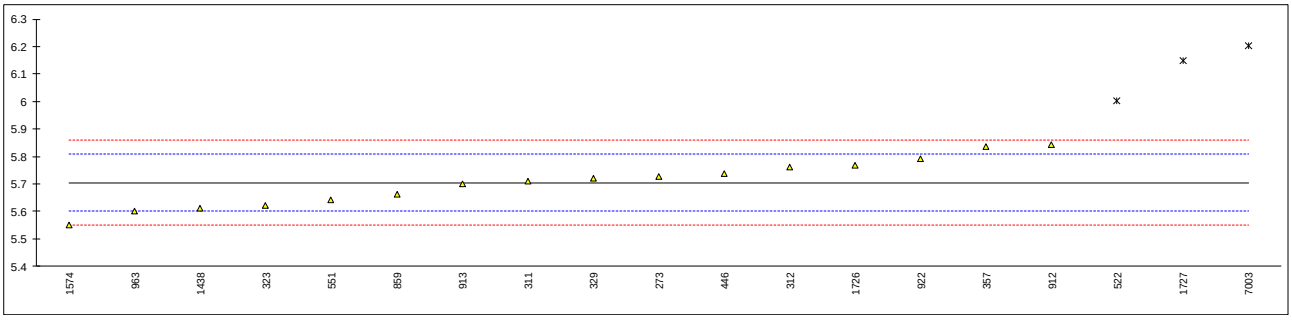


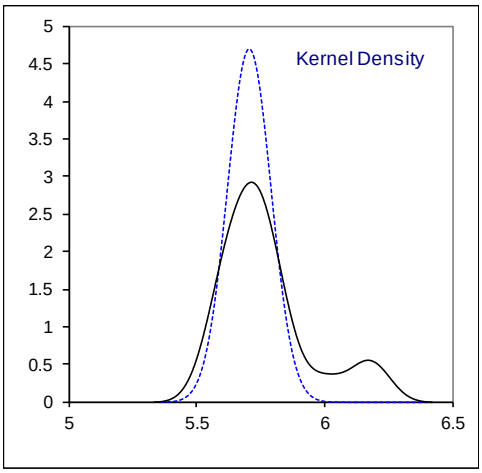


Determination of Water (Titrimetric) on sample #17242; results in %M/M

lab	method	value	mark	z(targ)	remarks
150		-----		-----	
230		-----		-----	
273	E203	5.727		0.43	
311	D1364	5.711		0.12	
312	E203	5.76		1.08	
323	D1364	5.62		-1.66	
329	E203	5.721		0.31	
357	E203	5.835		2.54	
446	E203	5.737		0.63	
522	D1364	6.002	DG(0.05)	5.81	
541		-----		-----	
551	E203	5.641		-1.25	
859	D1364	5.664		-0.80	
912	E203	5.842		2.68	
913	E203	5.70		-0.10	
922	E203	5.79		1.66	
963	D1364	5.60		-2.05	
1205		-----		-----	
1242		-----		-----	
1438	D1364	5.61		-1.85	
1574	D4017	5.5509		-3.01	
1605		-----		-----	
1726	EN15692	5.7692		1.26	
1727		6.15	DG(0.05)	8.70	
1817		-----		-----	
1835		-----		-----	
1927		-----		-----	
2407		-----		-----	
7003	E203	6.2035	DG(0.05)	9.74	

normality	OK
n	16
outliers	3
mean (n)	5.7049
st.dev. (n)	0.08511
R(calc.)	0.2383
st.dev.(D1364:02)	0.05118
R(D1364:02)	0.1433

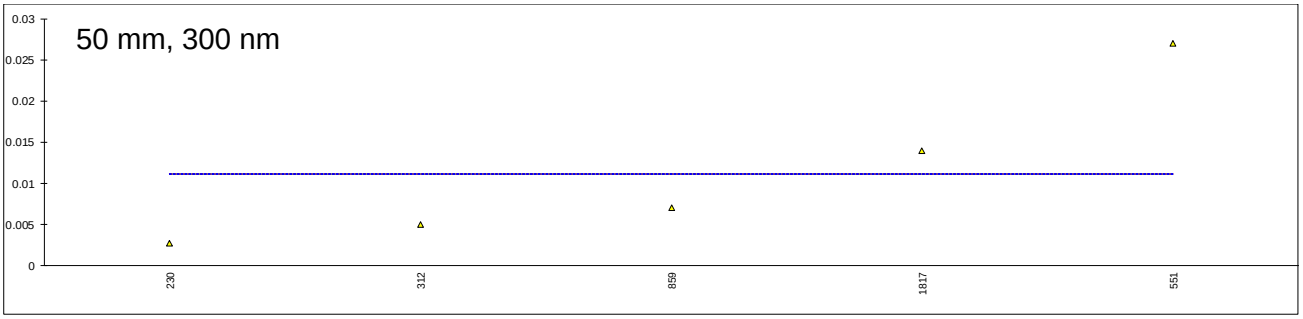


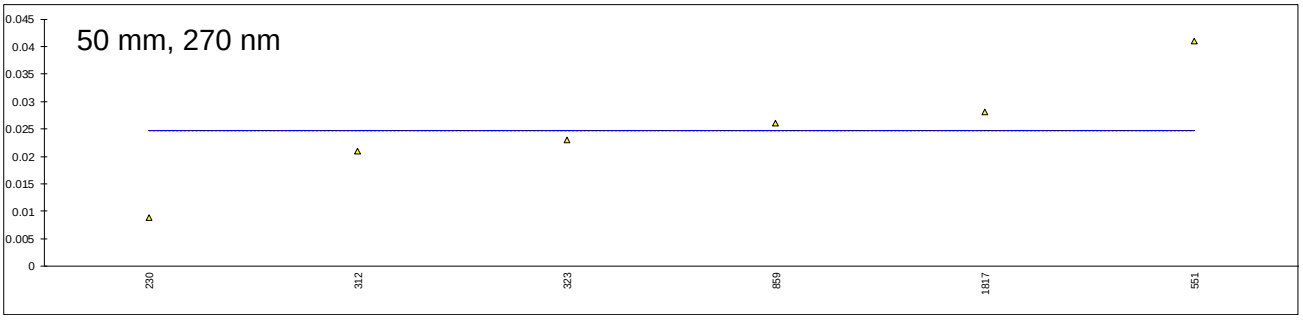


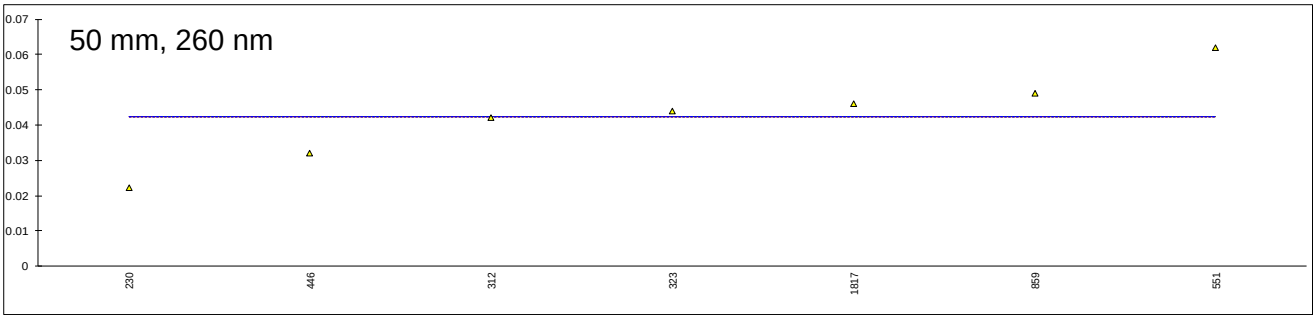
Determination of UV absorbance (50 mm cuvette) on sample #17242;

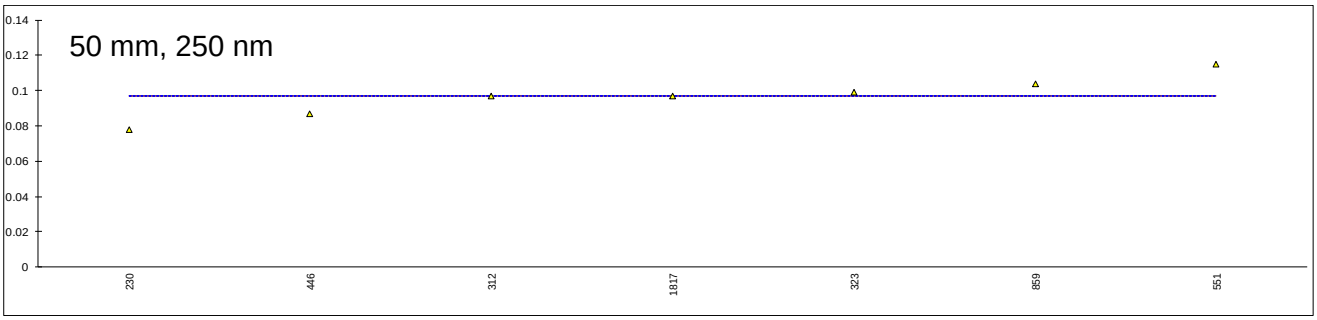
lab	method	300nm	270nm	260nm	250nm	240nm	230nm	220nm	Pass/Fail
150	IMPCA004	-----	-----	-----	-----	-----	-----	-----	Pass
230	INH-13	0.0027	0.0089	0.02225	0.07775	0.1972	0.4468	**0.82055**	Pass
273	IMPCA004	-----	-----	-----	-----	-----	-----	-----	-----
311	INH-094	-----	-----	-----	-----	-----	-----	-----	-----
312	INH-001	0.005	0.021	0.042	0.097	0.222	0.496	0.972	Pass
323	IMPCA004	< 0.01	0.023	0.044	0.099	0.224	0.480	0.991	pass
329	INH-13	-----	-----	-----	-----	-----	-----	-----	-----
357	INH-13	-----	-----	-----	-----	-----	-----	-----	-----
446	INH-UV	<0.01	<0.01	0.032	0.087	0.218	0.481	0.954	Pass
522		-----	-----	-----	-----	-----	-----	-----	-----
541		-----	-----	-----	-----	-----	-----	-----	-----
551	INH-3063	0.027	0.041	0.062	0.115	0.243	0.519	0.994	pass
859	IMPCA004	0.007	0.026	0.049	0.104	0.233	0.519	0.998	Pass
912		-----	-----	-----	-----	-----	-----	-----	-----
913		-----	-----	-----	-----	-----	-----	-----	-----
922	In house	-----	-----	-----	-----	-----	-----	-----	-----
963		-----	-----	-----	-----	-----	-----	-----	-----
1205		-----	-----	-----	-----	-----	-----	-----	-----
1242		-----	-----	-----	-----	-----	-----	-----	-----
1438		-----	-----	-----	-----	-----	-----	-----	-----
1574		-----	-----	-----	-----	-----	-----	-----	-----
1605		-----	-----	-----	-----	-----	-----	-----	-----
1726	In house	-----	-----	-----	-----	-----	-----	-----	-----
1727	IMPCA004	-----	-----	-----	-----	-----	-----	-----	-----
1817		0.014	0.028	0.046	0.097	0.217	-----	-----	Pass
1835		-----	-----	-----	-----	-----	-----	-----	-----
1927		-----	-----	-----	-----	-----	-----	-----	-----
2407		-----	-----	-----	-----	-----	-----	-----	-----
7003	D2008	-----	-----	-----	-----	-----	-----	-----	-----
	normality	unknown	unknown	unknown	unknown	unknown	unknown	unknown	n.a.
	n	5	6	7	7	7	6	5	8
	outliers	0	0	0	0	0	0	1	0
	mean (n)	0.0111	0.0247	0.0425	0.0967	0.2220	0.4903	0.9818	pass
	st.dev. (n)	0.00982	0.01044	0.01263	0.01188	0.01427	0.02744	0.01847	n.a.
	R(calc.)	0.0275	0.0292	0.0354	0.0333	0.0400	0.0768	0.0517	n.a.

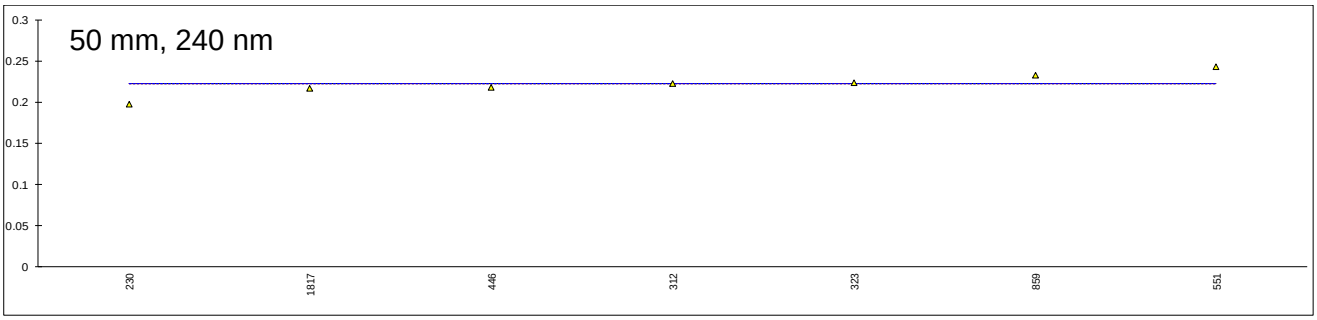
Statistical outliers are marked in **bold and underlined** text

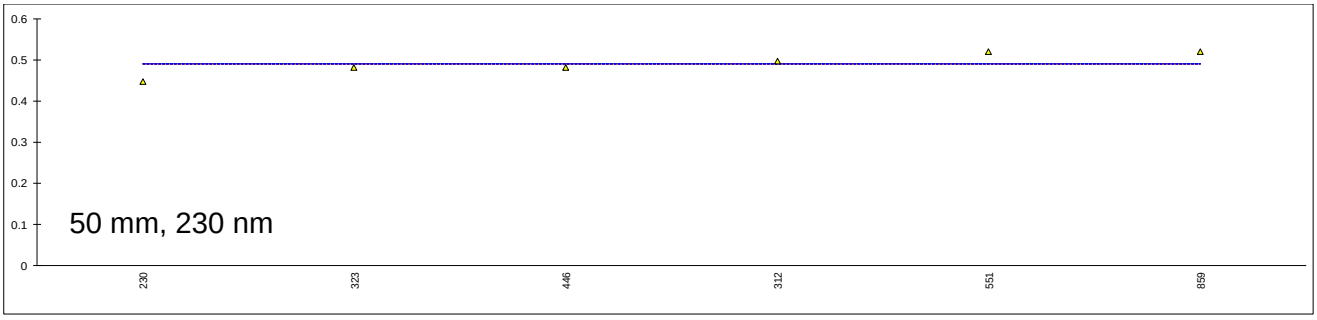


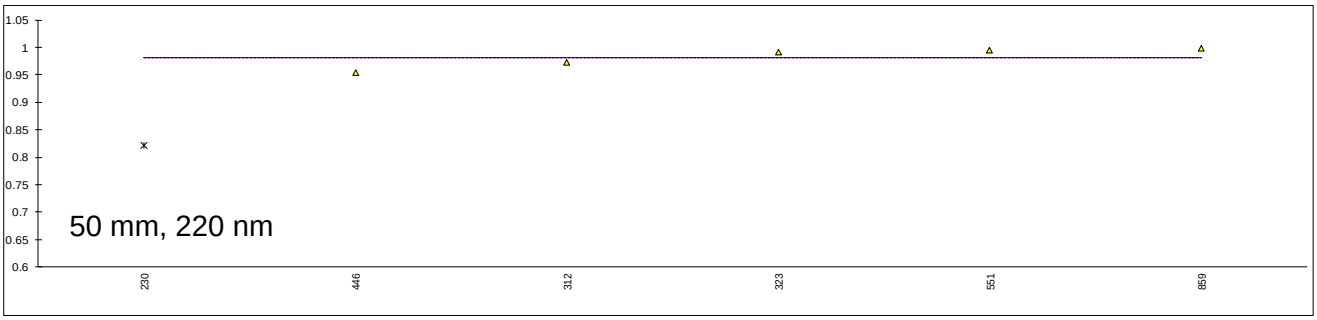



50 mm, 260 nm
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
230
446
312
323
1817
859
551


50 mm, 250 nm
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
230
446
312
1817
323
859
551


50 mm, 240 nm
0.3
0.25
0.2
0.15
0.1
0.05
0
230
1817
446
312
323
859
551

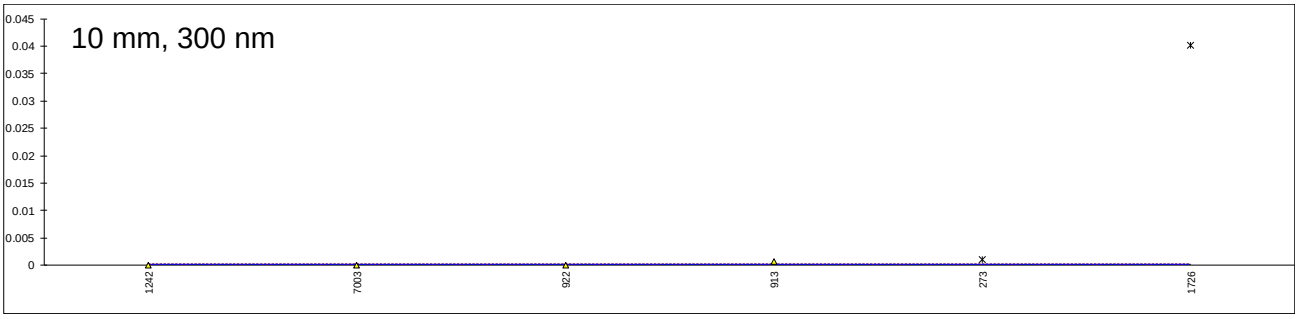

50 mm, 230 nm
0.6
0.5
0.4
0.3
0.2
0.1
0
230
323
446
312
551
859

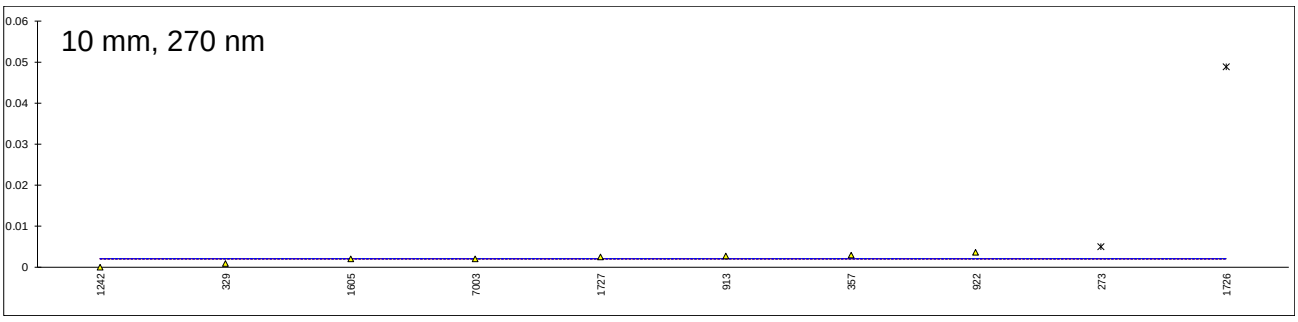

50 mm, 220 nm
1.05
1
0.95
0.9
0.85
0.8
0.75
0.7
0.65
0.6
230
446
312
323
551
859

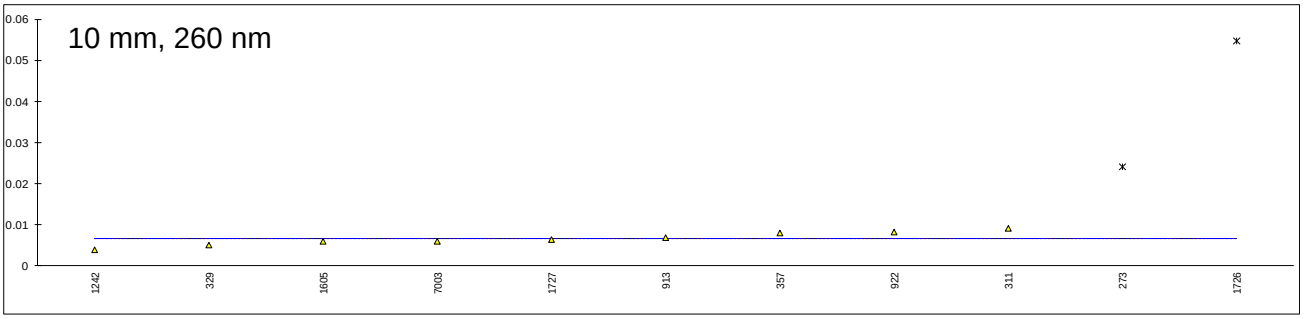
Determination of UV absorbance (10 mm cuvette) on sample #17242;

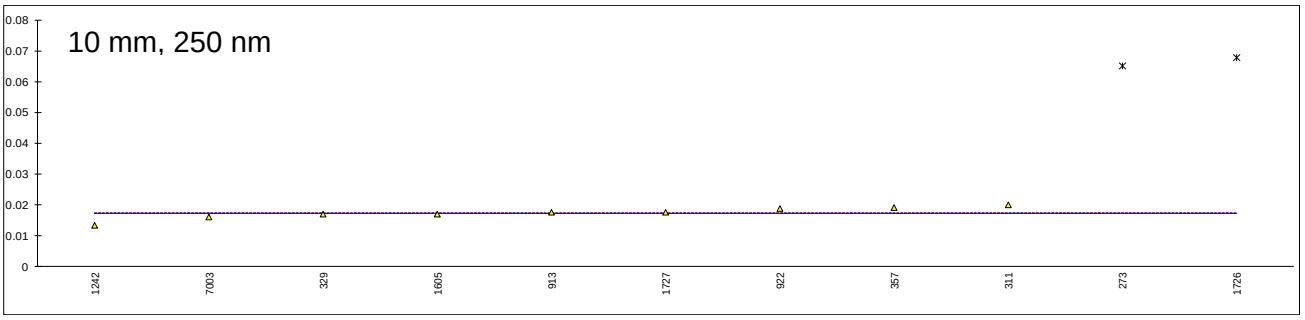
lab	method	300nm	270nm	260nm	250nm	240nm	230nm	220nm	Pass/Fail
150	IMPCA004	-----	-----	-----	-----	-----	-----	-----	Pass
230	INH-13	-----	-----	-----	-----	-----	-----	-----	-----
273	IMPCA004	0.001 ex, C	0.005 ex, C	**0.024** C	**0.065** C	**0.088** C	**0.115** C	0.189 ex, C	-----
311	INH-094	<0.005	<0.005	0.009	0.020	0.046	0.101	0.198	pass
312	INH-001	-----	-----	-----	-----	-----	-----	-----	-----
323	IMPCA004	-----	-----	-----	-----	-----	-----	-----	-----
329	INH-13	<0,001	0.001	0.005	0.017	0.043	0.097	0.190	pass
357	INH-13	<0,001	0.003	0.008	0.019	0.044	0.099	0.194	Pass
446	INH-UV	-----	-----	-----	-----	-----	-----	-----	-----
522		-----	-----	-----	-----	-----	-----	-----	-----
541		-----	-----	-----	-----	-----	-----	-----	-----
551	INH-3063	-----	-----	-----	-----	-----	-----	-----	-----
859	IMPCA004	-----	-----	-----	-----	-----	-----	-----	-----
912		-----	-----	-----	-----	-----	-----	-----	-----
913		0.0007	0.0028	0.0068	0.0176	0.0420	0.0973	0.1877	Pass
922	In house	0.0001	0.0037	0.0081	0.0188	0.0449	0.0995	0.1948	pass
963		-----	-----	-----	-----	-----	-----	-----	-----
1205		-----	-----	-----	-----	-----	-----	-----	-----
1242		0.0000	0.000 C	0.0040	0.0135	0.0380	0.0925	0.1855	-----
1438		-----	-----	-----	-----	-----	-----	-----	-----
1574		-----	-----	-----	-----	-----	-----	-----	-----
1605		< 0.001	0.002	0.006	0.017	0.041	0.092	0.177	-----
1726	In house	**0.0402**	**0.048693**	**0.054615**	**0.06789**	**0.095086**	**0.15102**	**0.24812**	PASS
1727	IMPCA004	-----	0.0025	0.0063	0.0177	0.0438	0.098	0.194	Pass
1817		-----	-----	-----	-----	-----	-----	-----	-----
1835		-----	-----	-----	-----	-----	-----	-----	-----
1927		-----	-----	-----	-----	-----	-----	-----	-----
2407		-----	-----	-----	-----	-----	-----	-----	-----
7003	D2008	0	0.002	0.006	0.016	0.038	0.087	0.172	-----
	normality	unknown	OK	OK	suspect	OK	OK	OK	n.a.
	n	4	8	9	9	9	9	9	8
	outliers	1 (+1ex)	1 (+1ex)	2	2	2	2	1 (+1ex)	0
	mean (n)	0.0002	0.0021	0.0066	0.0174	0.0423	0.0959	0.1881	pass
	st.dev. (n)	0.00034	0.00117	0.00159	0.00190	0.00285	0.00450	0.00870	n.a.
	R(calc.)	0.0009	0.0033	0.0044	0.0053	0.0080	0.0126	0.0244	n.a.

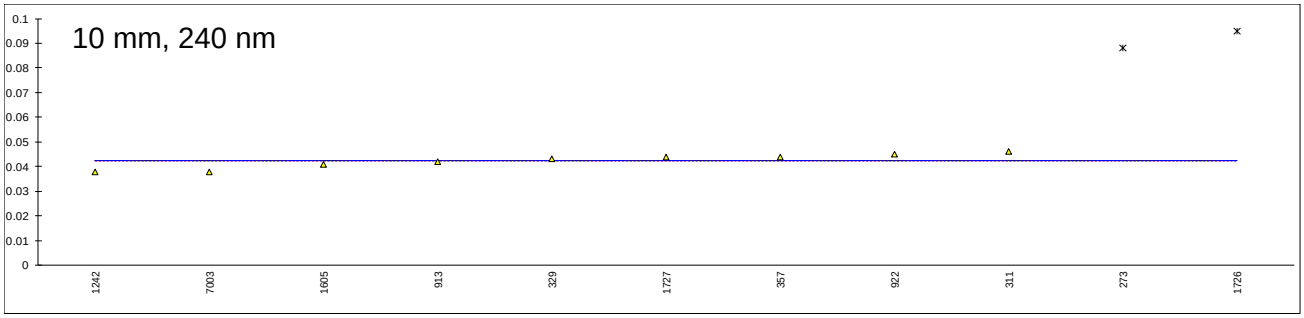
Lab 273 first reported for 300nm 0.189, for 270nm 0.115, for 260nm 0.088, for 250nm 0.065, for 240nm 0.024, for 230nm <0.01, for 220nm <0.01.
The test results of lab 273 were excluded due to outliers in the other determination of UV absorbance (10 mm cuvette)
Lab 1242 first reported for 270nm 0.0010
Statistical outliers are marked in **bold and underlined** text

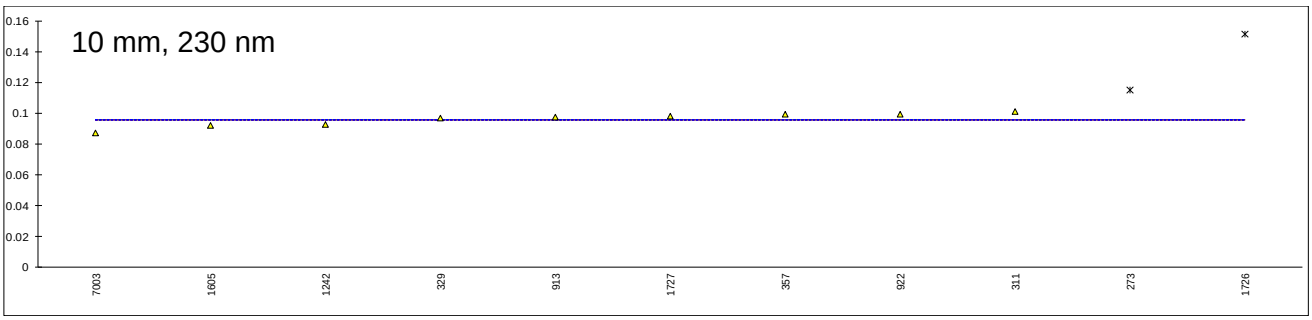


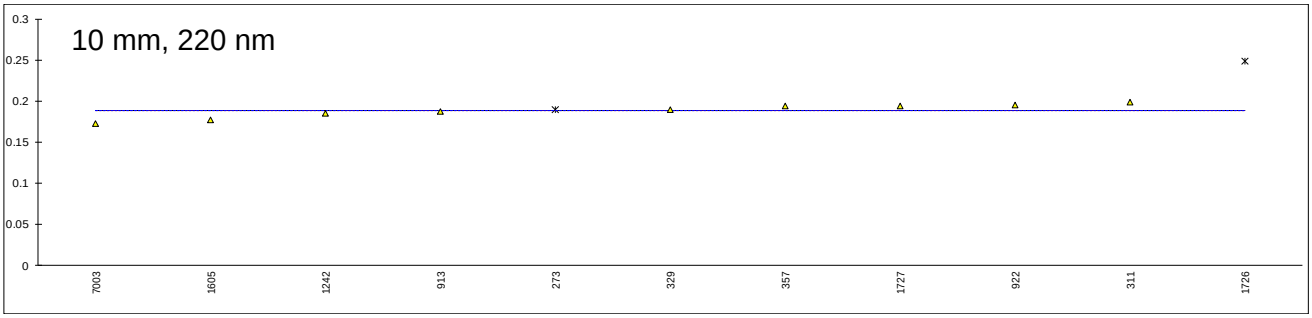



10 mm, 260 nm
0.06
0.05
0.04
0.03
0.02
0.01
0
1242
329
1605
7003
1727
913
357
922
311
273
1726


10 mm, 250 nm
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
1242
7003
329
1605
913
1727
922
357
311
273
1726

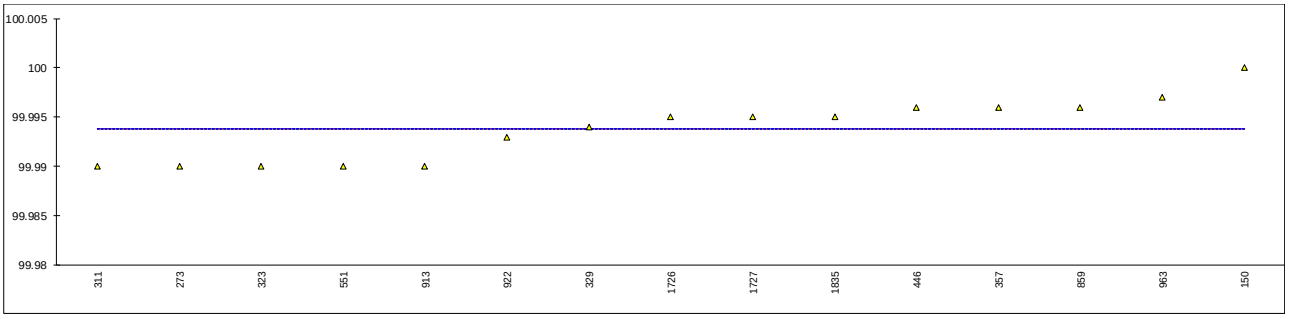

10 mm, 240 nm
0.1
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
1242
7003
1605
913
329
1727
357
922
311
273
1726

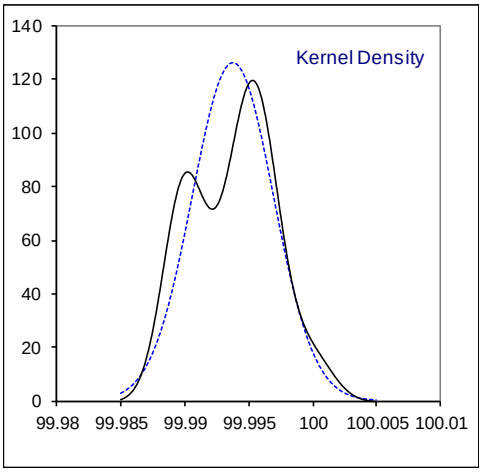

10 mm, 230 nm
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
7003
1605
1242
329
913
1727
357
922
311
273
1726


10 mm, 220 nm
0.3
0.25
0.2
0.15
0.1
0.05
0
7003
1605
1242
913
273
329
357
1727
922
311
1726

Determination of Purity of Ethanol on dry basis on sample #17243; results in %M/M

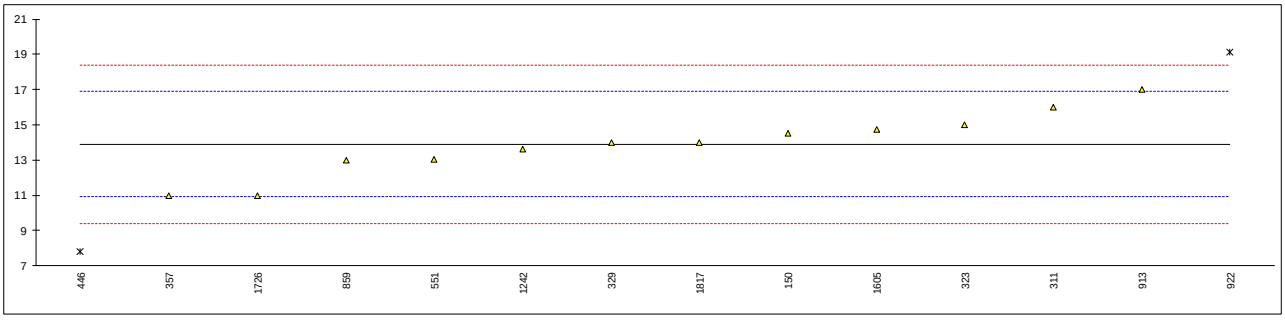
lab	method	value	mark	z(targ)	remarks
150	INH-02	100.00		-----	
230		-----		-----	
273	In house	99.99		-----	
311	INH-529	99.99		-----	
312		N.A.		-----	
323	INH-EtOH	99.99		-----	
329	INH-02	99.994		-----	
357	INH-0002	99.996		-----	
446	INH-EtOH	99.996		-----	
522		-----		-----	
541		-----		-----	
551	INH-1313	99.99		-----	
859	EN15721	99.996		-----	
912		-----		-----	
913	INH-0001	99.99		-----	
922	INH-0001	99.993		-----	
963	EN15721	99.997		-----	
1205		-----		-----	
1242		-----		-----	
1438		-----		-----	
1574		-----		-----	
1605		-----		-----	
1726	In house	99.995		-----	
1727		99.995		-----	
1817		-----		-----	
1835	In house	99.995		-----	
1927		-----		-----	
2407		-----		-----	
7003		-----		-----	
	normality	OK			
	n	15			
	outliers	0			
	mean (n)	99.9938			
	st.dev. (n)	0.00317			
	R(calc.)	0.0089		R(iis16C11) = 0.0181 or R(iis15C15) = 0.1291	
	st.dev.(lit.)	n.a.			
	R(lit.)	n.a.			

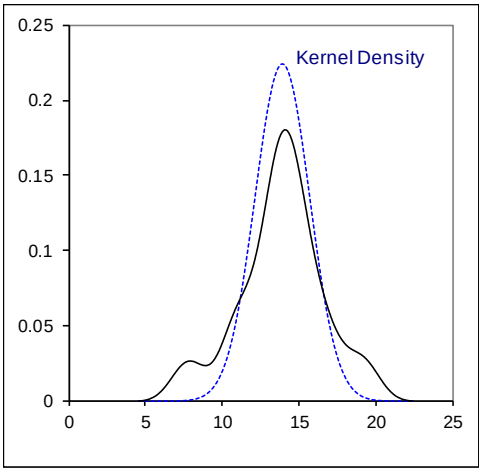




Determination of Methanol on sample #17243; results in mg/kg

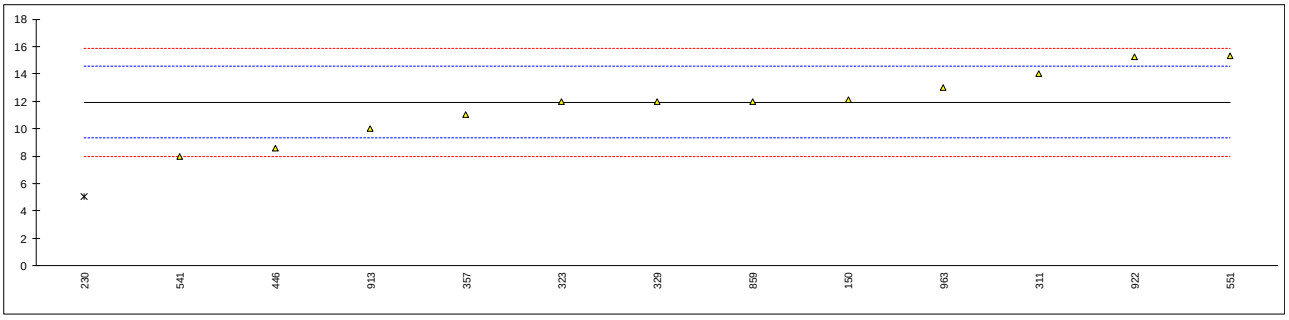
lab	method	value	mark	z(targ)	remarks
150	INH-02	14.5		0.40	
230	INH-001	<5		<-5.95	Possibly a false negative test result?
273		-----		-----	
311	INH-529	16		1.40	
312		-----		-----	
323	INH-EtOH	15		0.73	
329	INH-02	14		0.06	
357	INH-0002	11		-1.94	
446	INH-EtOH	7.8	G(0.01)	-4.08	
522		-----		-----	
541		-----		-----	
551	INH-1313	13.055		-0.57	
859	EN15721	13		-0.61	
912		-----		-----	
913	INH-0001	17.0		2.07	
922	INH-0001	19.14	G(0.01)	3.49	
963	EN15721	<10		-----	
1205		-----		-----	
1242		13.6091		-0.20	
1438		-----		-----	
1574		-----		-----	
1605		14.72		0.54	
1726	In house	11		-1.94	
1727		<10	C	-----	first reported 7
1817	In house	14.0042		0.06	
1835	In house	<25		-----	
1927		-----		-----	
2407		-----		-----	
7003		-----		-----	
	normality	OK			
	n	12			
	outliers	2	Spike		
	mean (n)	13.907	20		Recovery <69.6%
	st.dev. (n)	1.7764			
	R(calc.)	4.974			
	st.dev.(Horwitz)	1.4972			
	R(Horwitz)	4.192			

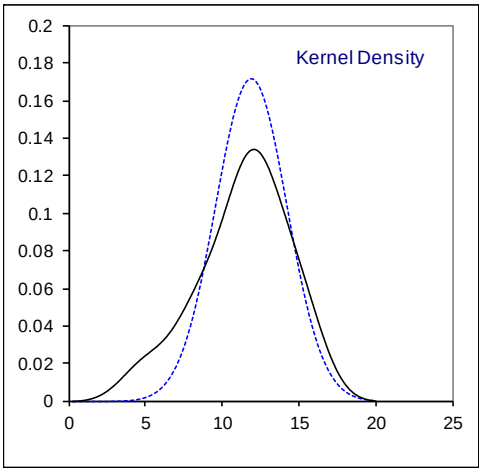




Determination of Acetone on sample #17243; results in mg/kg

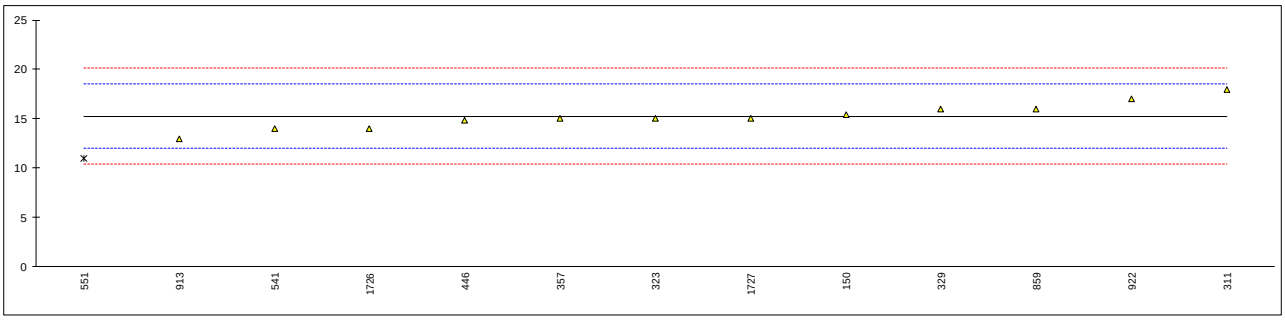
lab	method	value	mark	z(targ)	remarks
150	INH-02	12.1		0.12	
230	INH-001	5.05	G(0.01)	-5.24	
273		-----		-----	
311	INH-529	14		1.57	
312		-----		-----	
323	INH-EtOH	12		0.05	
329	INH-02	12		0.05	
357	INH-0002	11		-0.72	
446	INH-EtOH	8.6		-2.54	
522		-----		-----	
541		8		-3.00	
551	INH-1313	15.318		2.57	
859	EN15721	12		0.05	
912		-----		-----	
913	INH-0001	10.0		-1.48	
922	INH-0001	15.27		2.53	
963	EN15721	13		0.81	
1205		-----		-----	
1242		-----		-----	
1438		-----		-----	
1574		-----		-----	
1605		-----		-----	
1726		-----		-----	
1727		-----		-----	
1817		-----		-----	
1835	In house	<50		-----	
1927		-----		-----	
2407		-----		-----	
7003		-----		-----	
	normality	OK			
	n	12			
	outliers	1	Spike		
	mean (n)	11.941	20		Recovery < 59.7%
	st.dev. (n)	2.3205			
	R(calc.)	6.497			
	st.dev.(Horwitz)	1.3153			
	R(Horwitz)	3.683			

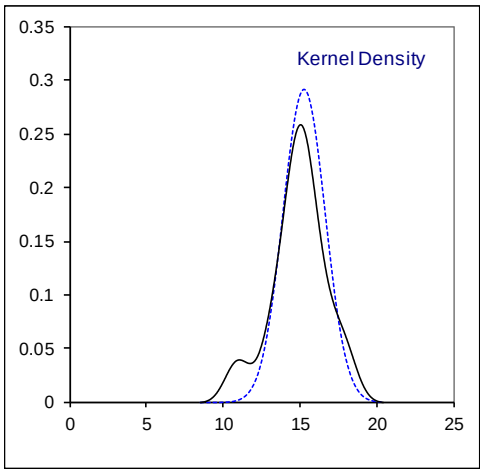




Determination of Isopropanol on sample #17243; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	INH-02	15.4		0.08	
230	INH-001	<5	C	< -5.80	first reported 7.72. Possibly a false negative test result?
273		-----		-----	
311	INH-529	18		1.68	
312		-----		-----	
323	INH-EtOH	15		-0.17	
329	INH-02	16		0.45	
357	INH-0002	15		-0.17	
446	INH-EtOH	14.8		-0.29	
522		-----		-----	
541		14		-0.78	
551	INH-1313	10.968	D(0.05)	-2.65	
859	EN15721	16		0.45	
912		-----		-----	
913	INH-0001	13.0		-1.40	
922	INH-0001	17.05		1.10	
963	EN15721	<10		<-2.97	Possibly a false negative test result?
1205		-----		-----	
1242		-----		-----	
1438		-----		-----	
1574		-----		-----	
1605		-----		-----	
1726	In house	14		-0.78	
1727		15		-0.17	
1817		-----		-----	
1835	In house	<25		-----	
1927		-----		-----	
2407		-----		-----	
7003		-----		-----	
	normality	OK			
	n	12			
	outliers	1	Spike		
	mean (n)	15.271	20		Recovery < 74.7%
	st.dev. (n)	1.3659			
	R(calc.)	3.824			
	st.dev.(Horwitz)	1.6210			
	R(Horwitz)	4.539			

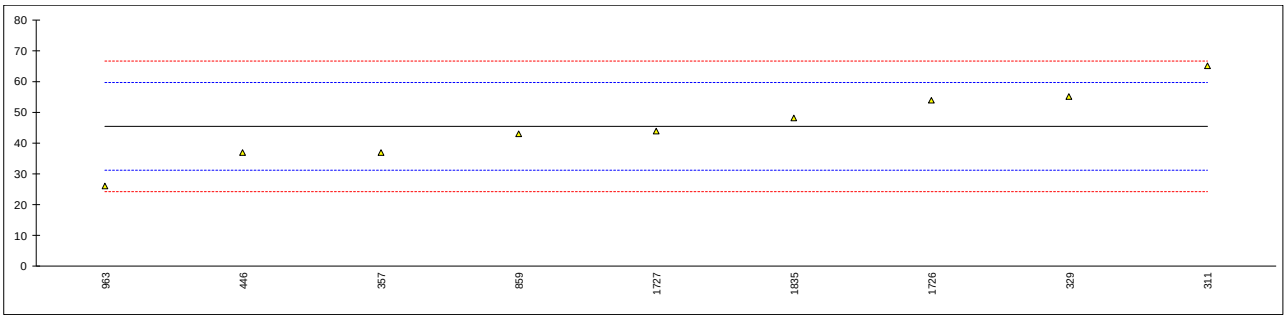


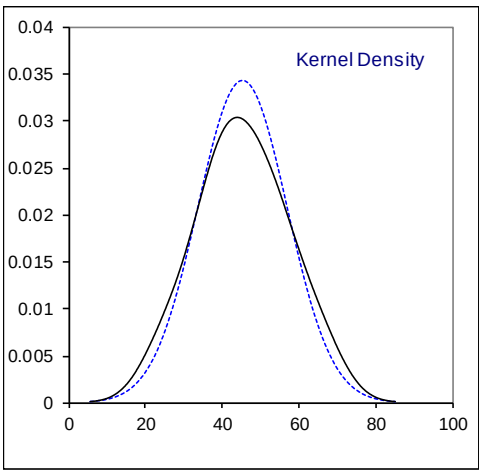


Determination of Total impurities on sample #17243; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150		-----		-----	
230		-----		-----	
273		-----		-----	
311	INH-529	65		2.76	
312		-----		-----	
323		-----		-----	
329	INH-02	55		1.35	
357	INH-0002	37		-1.19	
446	INH-EtOH	36.9		-1.20	
522		-----		-----	
541		-----		-----	
551		-----		-----	
859	EN15721	43		-0.34	
912		-----		-----	
913		-----		-----	
922		-----		-----	
963	EN15721	26		-2.74	
1205		-----		-----	
1242		-----		-----	
1438		-----		-----	
1574		-----		-----	
1605		-----		-----	
1726	In house	54		1.21	
1727		44		-0.20	
1817	In house	<300		-----	
1835	In house	48		0.36	
1927		-----		-----	
2407		-----		-----	
7003		-----		-----	

normality	OK
n	9
outliers	0
mean (n)	45.433
st.dev. (n)	11.6400
R(calc.)	32.592
st.dev.(Horwitz, comp:3)	7.0892
R(Horwitz, comp:3)	19.850





Determination of Acetal (1,1-diethoxyethane), Acetaldehyde, Benzene, Monoethylene glycol (MEG) and Other impurities on sample #17243; results in mg/kg

lab	method	Acetal	Acetaldehyde	Benzene	MEG	Other impurities	remarks
150	INH-02	<2	5.6	<2	-----	<2	
230	INH-0001	<5	<5	<5	-----	-----	
273		-----	-----	-----	-----	-----	
311	INH-529	<5	<5	5	-----	<5	
312		-----	-----	-----	-----	-----	
323	INH-EtOH	< 5	< 5	< 5	-----	-----	
329	INH-02	<2	<2	<2	-----	<5	
357	INH-0002	<5	<5	<5	<30	<5	
446	INH-EtOH	<5	<5	<5	<5	5.7	
522		-----	-----	-----	-----	-----	
541		-----	<5	<5	-----	-----	
551	INH-1299	<6	<6	<0,1	-----	-----	
859	EN15721	<5	<5	<5	<5	<5	
912		-----	-----	-----	-----	-----	
913	INH-0001	<5.0	<5.0	<5.0	<5.0	-----	
922	INH-0001	<5.0	<5.0	<2.0	15.71	-----	
963	EN15721	-----	2	<5	-----	11	
1205		-----	-----	-----	-----	-----	
1242		-----	-----	-----	-----	-----	
1438		-----	-----	-----	-----	-----	
1574		-----	-----	-----	-----	-----	
1605		-----	-----	-----	-----	-----	
1726	In house	<10	<10	<10	<10	29	
1727		<10	<10	-----	-----	-----	
1817	In house	ND	ND	ND	-----	<300	
1835	In house	<25	<25	<10	-----	<25	
1927		-----	-----	-----	-----	-----	
2407		-----	-----	-----	-----	-----	
7003		-----	-----	-----	-----	-----	

APPENDIX 2

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	IRAN, Islamic Republic of
1 lab in	ISRAEL
1 lab in	MAURITIUS
1 lab in	MEXICO
3 labs in	NETHERLANDS
1 lab in	P.R. of CHINA
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
1 lab in	UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations:

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= probably an error in calculations
U	= test result probably reported in a different unit
W	= test result withdrawn on request participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= Safety Data Sheet

Literature:

1 iis, Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, March 2017
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4 ASTM E1301:95(2003)
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6 ISO 5725, parts 1-6, 1994
7 ISO 13528
8 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
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14 Analytical Methods Committee Technical brief, No 4.January 2001
15 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364 (2002).
16 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), 165-172, (1983)