Results of Proficiency Test
Ethanol (Food/Neutral)
December 2016

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

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CONTENTS

1 INTRODUCTION

Since 2007, a proficiency test for Food/Neutral grade Ethanol is organised every year by the Institute for Interlaboratory Studies. During the planning of the annual proficiency testing program 2016/2017, it was decided to continue the round robin for the analysis of Food/Neutral grade Ethanol.
In this interlaboratory study, 31 laboratories in 18 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the 2016 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. Analysis for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one sample (1* 0.5 L of Food/Neutral grade Ethanol, labelled #16262). 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. Also customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3).
The protocol can be downloaded from 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 #16262 was obtained from a local trader. To 36.3 kg of this material 0.8 grams of each of the following chemicals was added: Acetal, Benzene, Methanol and Mono Ethylene Glycol. After homogenisation in a pre-cleaned drum, 50 amber glass bottles of 0.5 L were filled and labelled #16262. 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 #16262-1	0.80643
Sample #16262-2	0.80643
Sample #16262-3	0.80642
Sample #16262-4	0.80643
Sample #16262-5	0.80642
Sample #16262-6	0.80643
Sample #16262-7	0.80642
Sample #16262-8	0.80642

Table 1: Homogeneity test results of subsamples #16262

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 #16262

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

To each of the participating laboratories 1*0.5 L bottle of sample #16262 was sent on November 9, 2016. An SDS of the product was added to the samples.

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 #16262: Density at 20°C, Non-volatile matter, Permanganate Time Test at 20°C, pHe, Strength (in %M/M and %V/V), Water (titrimetric), Purity Ethanol on dry basis, Methanol, Acetal, Acetaldehyde, Acetone

Benzene, iso-Propanol, Mono Ethylene Glycol and UV Absorbance at 300, 270, 260, 250, 240, 230 and 220nm with an evaluation of the UV-scan.

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 results, but report as much significant figures as possible. It was also requested not to report 'less than' results, which are above the detection limit, because such results cannot be used for meaningful statistical evaluations.

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

3.1 STATISTICS

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded 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 and/or Grubbs' and/or Rosner’s outlier tests. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ test and by R(0.01) for the Rosner’s test. Stragglers are marked by D(0.05) for the Dixon’s test, by G(0.05) or DG(0.05) for the Grubbs’ test and by R(0.05) for the Rosner’s test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

Finally, the reproducibilities were calculated from the standard deviations by multiplying these with a factor of 2.8.

3.2 GRAPHICS

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

3.3 Z-SCORES

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

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

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

The $z_{(target)}$ scores are listed in the result tables of 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:

	$\lvert z \rvert < 1$	good
$1 <$	$\lvert z \rvert < 2$	satisfactory
$2 <$	$\lvert z \rvert < 3$	questionable
$3 <$	$\lvert z \rvert$	unsatisfactory

4 EVALUATION

In this proficiency test, some problems were encountered with the dispatch of the samples. Five participants did not report any test results at all and all other participants reported test results before the PT deadline. Not all laboratories were able to report all analyses requested. In total 26 laboratories reported 329 numerical results. Observed were 16 outlying results, which is 4.9%. 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 TEST

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

Unfortunately, a suitable test method providing precision data is not available for all determinations. For the tests 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 e.g. D1363:06 will be used.

Density: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers 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 was not in agreement with the requirements of ASTM D1363:06(2011).
Two participants reported a result of >30 min., which is the ‘on spec’ limit for ethanol. This means the two participants would approve this sample, whereas the majority of the group would have rejected it for being ‘off spec’.

pHe: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility was in agreement with the requirements of ASTM D6423:14. There is a small difference in results for the laboratories using KCl and LiCl electrodes (see lit. 16), but all test results are well within the reproducibility limits of the method.

Strength (%M/M): This determination may not be problematic. One statistical outlier was observed. Regretfully, no standard test method with precision data exists for this determination. The calculated reproducibility after rejection of the statistical outlier is small in comparison to the calculated reproducibility in the previous proficiency test iis15C15.

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 very problematic. No statistical outliers were observed. The calculated reproducibility is not at all in agreement with the requirements of ASTM D1364:02(2012).

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 ten times smaller than the calculated reproducibility in the previous proficiency test iis15C15 and in line with the proficiency test iis14C11.

Methanol: 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.

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

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

Mono Ethylene Glycol: This determination may be very problematic. No statistical outliers were observed, but two possible false negative test results were reported. The calculated reproducibility is not at all in agreement with the estimated reproducibility using the Horwitz equation.

Other impurities: For Acetaldehyde, Acetone and iso-Propanol, the majority of participants reported a result <10 mg/kg or less.

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 twelve

laboratorories used a 10 mm cuvette. Both groups were evaluated separately.

UV - 50 mm cuvette: This determination may be problematic. In total, four statistical outliers were observed. For laboratory 1817 all test results were statistical outliers, except one. This test result was excluded. All laboratories evaluated the sample as 'Fail'.

UV - 10 mm cuvette: This determination may be problematic. In total, six statistical outliers were observed. The calculated reproducibility at 300nm is in line with the calculated reproducibility observed in the previous proficiency test iis15C15 (R(calc)=0.0067 at mean Absorbance=0.0181).
Three laboratories evaluated the sample as a 'Pass', whereas all other laboratories evaluated the sample as a 'Fail'. This may be a result of the use of a 10 mm cuvette and not enlarging the resulting graph well enough to judge the impurities properly.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	2.8 *sd	R (lit)
Density at 20°C	kg/L	23	0.8064	0.0002	0.0005
Nonvolatile matter	mg/100mL	9	0.4	1.0	2.1
Permanganate Time Test	min.	11	25.6	8.1	6.4
pHe		11	7.8	0.9	1.0
Strength	%M/M	15	94.19	0.03	(0.04)
Strength	%V/V	24	96.24	0.07	0.06
Water (titrimetric)	%M/M	17	5.81	0.34	0.14
Purity EtOH on dry basis	%M/M	13	99.98	0.02	n.a.
Methanol	mg/kg	14	24.7	9.1	6.8
Acetal	mg/kg	13	20.8	6.1	5.9
Acetaldehyde	mg/kg	14	≤10	n.a.	n.a.
Acetone	mg/kg	12	<10	n.a.	n.a.
Benzene	mg/kg	12	19.7	3.0	5.6
iso-Propanol	mg/kg	14	<10	n.a.	n.a.
Mono Ethylene Glycol	mg/kg	5	16.8	14.1	4.9

Table 3: Reproducibilities of tests on sample #16262
Results between brackets are compared with the observed reproducibility of the previous proficiency test

Parameter	unit	n	average	2.8 *sd	R (lit)
UV – 50 mm cuvette:					
UV-absorbance 300 nm		6	0.024	0.014	n.a.
UV-absorbance 270 nm		6	0.478	0.104	n.a.
UV-absorbance 260 nm		6	1.114	0.124	n.a.
UV-absorbance 250 nm		6	1.436	0.061	n.a.
UV-absorbance 240 nm		6	1.334	0.022	n.a.
UV-absorbance 230 nm		6	1.380	0.029	n.a.
UV-absorbance 220 nm		6	3.257	0.846	n.a.
Conclusion UV-scan	Pass/Fail	6	Fail	n.a.	n.a.
UV – 10 mm cuvette:					
UV-absorbance 300 nm		10	0.003	0.005	(0.007)
UV-absorbance 270 nm		11	0.090	0.013	n.a.
UV-absorbance 260 nm		10	0.216	0.015	n.a.
UV-absorbance 250 nm		9	0.283	0.023	n.a.
UV-absorbance 240 nm		11	0.264	0.019	n.a.
UV-absorbance 230 nm		11	0.271	0.030	n.a.
UV-absorbance 220 nm		12	1.287	0.149	n.a.
Conclusion UV-scan	Pass/Fail	7	Fail	n.a.	n.a.

Table 4: Reproducibilities of UV tests on sample #16262
Results between brackets are compared with the observed reproducibility of the previous proficiency test

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 2016 WITH PREVIOUS PTS

	December 2016	*November 2015*	*November 2014*	*November 2013*	*November 2012*
Number of reporting labs	26	32	25	24	24
Number of results reported	329	254	210	160	169
Number of statistical outliers	16	11	13	9	5
Percentage outliers	4.9%	4.3%	6.2%	5.6%	3.0%

Table 5: Comparison with previous proficiency tests

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

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

Parameter	December 2016	November 2015	November 2014	November 2013	November 2012
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	n.e.	(--)	(-)	(-)	(+)
Methanol	-	n.e.	n.e.	n.e.	n.e.
Acetal	+/-	n.e.	n.e.	n.e.	n.e.
Benzene	+	n.e.	n.e.	n.e.	n.e.
Mono Ethylene Glycol	--	n.e.	n.e.	n.e.	n.e.
UV-absorbance 300 nm	(--)	(+/-)	(-)	(++)	(-)
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 6: Comparison determinations of sample #16262 against the standard
Results between brackets are compared with the observed reproducibility of the previous proficiency test

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

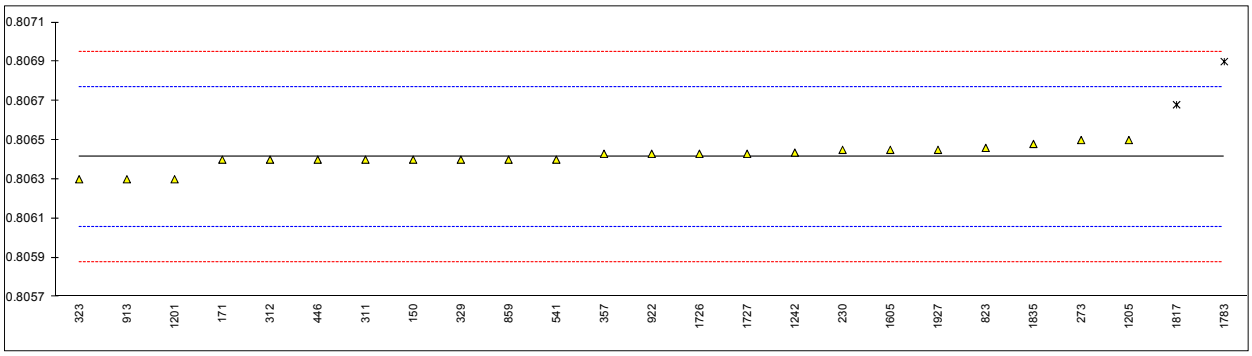
- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- - : group performed worse than the standard
- -- : group performed much worse than the standard
- n.e.: not evaluated

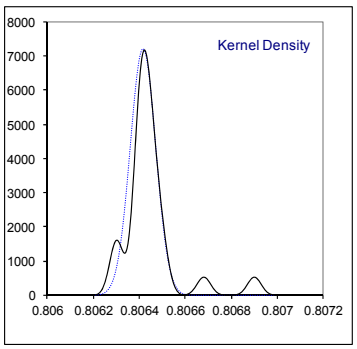
APPENDIX 1

Determination of Density at 20°C on sample #16262; results in kg/L

lab	method	value	mark	z(targ)	remarks
150	D4052	0.8064		-0.08	
171	D4052	0.8064		-0.08	
174		-----		-----	
230	D4052	0.80645		0.20	
273	D4052	0.8065	C	0.48	first reported: 0.8060
311	D4052	0.8064		-0.08	
312	ISO12185	0.8064		-0.08	
323	D4052	0.8063		-0.64	
329	D4052	0.8064		-0.08	
357	D4052	0.80643		0.08	
446	D4052	0.8064		-0.08	
541	ISO12185	0.8064		-0.08	
551		-----		-----	
558		-----		-----	
823	ISO12185	0.80646		0.25	
859	D4052	0.8064		-0.08	
912		-----		-----	
913	D4052	0.8063		-0.64	
922	D4052	0.80643		0.08	
963		-----		-----	
1201	ISO12185	0.8063		-0.64	
1205	In house	0.806500		0.48	
1242		0.806436		0.12	
1574		-----		-----	
1605	D4052	0.806450		0.20	
1726	D4052	0.80643		0.08	
1727	D4052	0.80643		0.08	
1783	D4052	0.8069	R(0.01)	2.72	
1817		0.80668	R(0.01)	1.48	
1835	ISO12185	0.80648		0.36	
1927	D4052	0.80645		0.20	

normality	OK
n	23
outliers	2
mean (n)	0.806415
st.dev. (n)	0.0000553
R(calc.)	0.000155
R(ISO12185:96)	0.000500

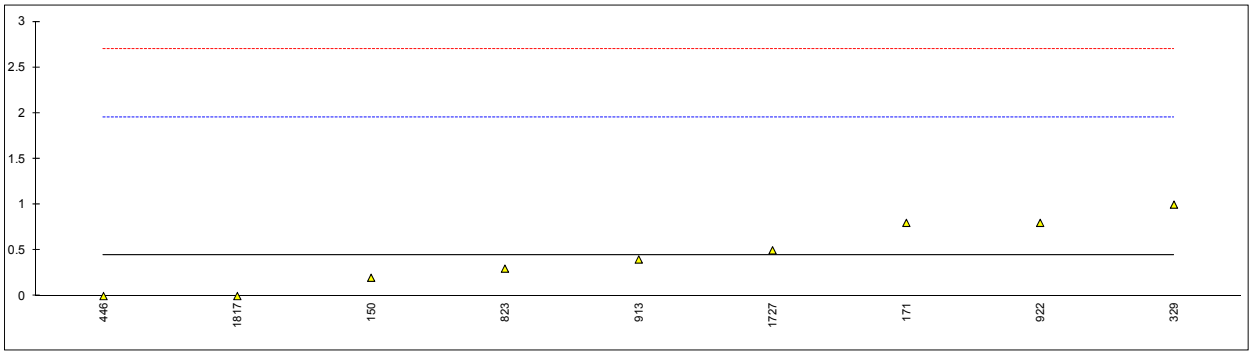


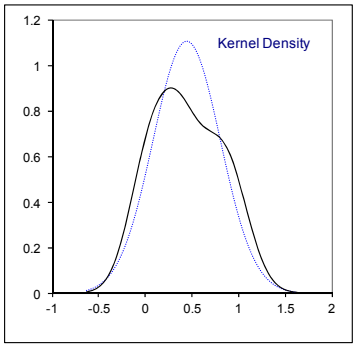


Determination of Nonvolatile matter on sample #16262; results in mg/100mL

lab	method	value	mark	z(targ)	remarks
150	D1353	0.2		-0.32	
171	D1353	0.8		0.47	
174		-----		-----	
230	D1353	<1		-----	
273		-----		-----	
311		-----		-----	
312	D1353	<1		-----	
323	D1353	<1		-----	
329	EN15691	1		0.74	
357	D1353	< 1		-----	
446	D1353	0.0		-0.59	
541	D1353	<1		-----	
551		-----		-----	
558		-----		-----	
823	D1353	0.3		-0.19	
859	D1353	<1		-----	
912		-----		-----	
913	D1353	0.4		-0.06	
922	D1353	0.80		0.47	
963		-----		-----	
1201	D1353	<0.1		-----	
1205		-----		-----	
1242		-----		-----	
1574		-----		-----	
1605		-----		-----	
1726	EN15691	ND		-----	
1727	EN15691	0.5		0.07	
1783		-----		-----	
1817		0		-0.59	
1835	EN15691	<10		-----	
1927		-----		-----	

normality	OK
n	9
outliers	0
mean (n)	0.44
st.dev. (n)	0.361
R(calc.)	1.01
R(D1353:13)	2.11

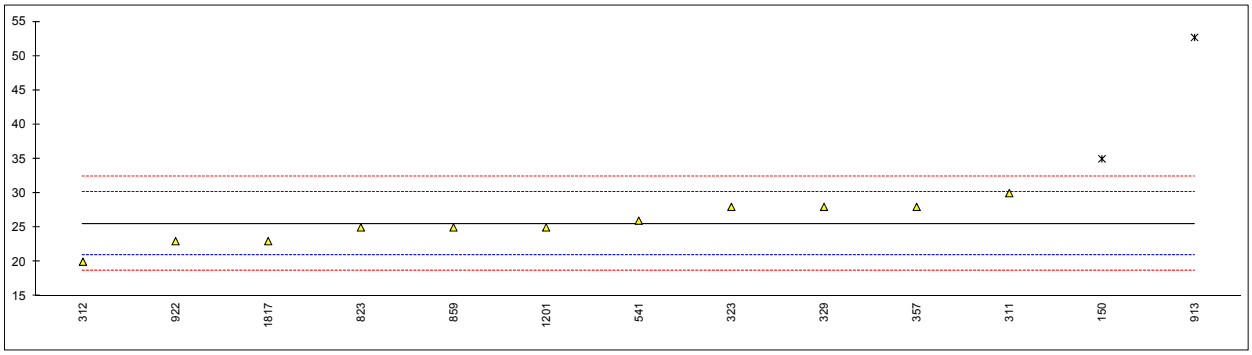


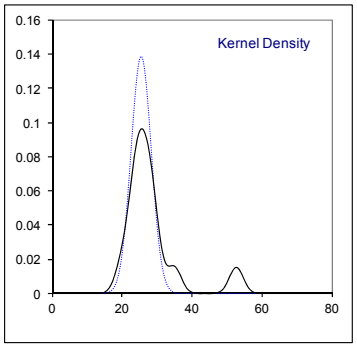


Determination of Permanganate Time Test at 20°C on sample #16262; results in minutes

lab	method	value	mark	z(targ)	remarks
150	D1363	35	G(0.05)	4.11	
171	D1363	>30		-----	
174		-----		-----	
230	D1363	<50		-----	
273		-----		-----	
311	D1363	30		1.94	
312	INH-90-3	20		-2.41	
323	D1363	28		1.07	
329	D1363	28		1.07	
357	D1363	28		1.07	
446		-----		-----	
541	D1363	26		0.20	
551		-----		-----	
558		-----		-----	
823	D1363	25		-0.24	
859	D1363	25		-0.24	
912		-----		-----	
913	D1363	52.7	G(0.01)	11.81	
922	D1363	23		-1.11	
963		-----		-----	
1201	D1363	25		-0.24	
1205		-----		-----	
1242		-----		-----	
1574		-----		-----	
1605		-----		-----	
1726		-----		-----	
1727		-----		-----	
1783		-----		-----	
1817		23		-1.11	
1835	D1363	>30	C	-----	first reported: 40
1927		-----		-----	

normality	OK
n	11
outliers	2
mean (n)	25.55
st.dev. (n)	2.876
R(calc.)	8.05
R(D1363:06)	6.44

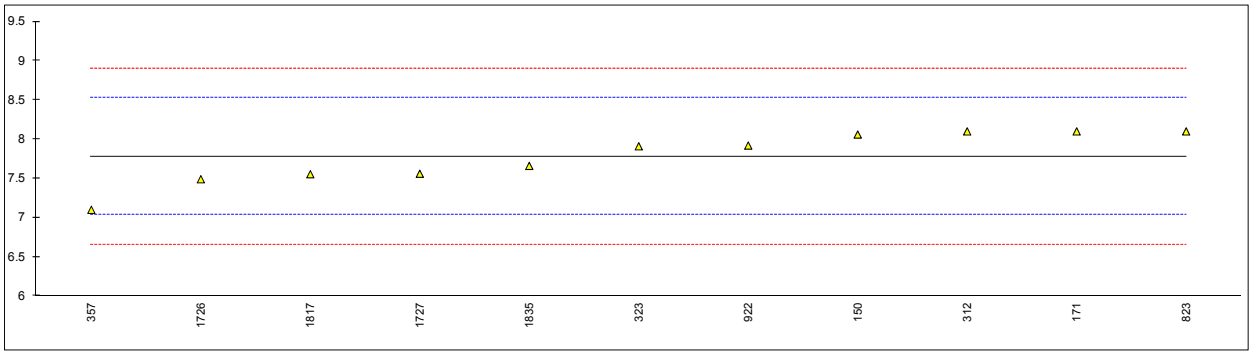


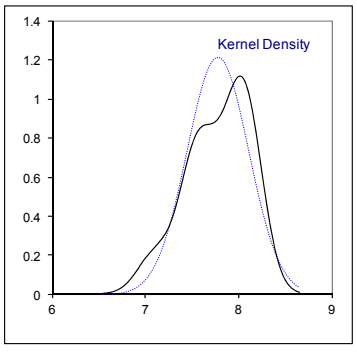


Determination of pHe on sample #16262;

lab	method	Electrode	value	mark	z(targ)	remarks
150	D6423	KCl	8.06		0.76	
171	D6423	Other	8.1		0.86	Electrode with long-life gel
174			-----		-----	
230			-----		-----	
273			-----		-----	
311			-----		-----	
312	D6423	KCl	8.1		0.86	
323	EN15490	LiCl	7.91		0.35	
329			-----		-----	
357	D6423	LiCl	7.1		-1.81	
446			-----		-----	
541			-----		-----	
551			-----		-----	
558			-----		-----	
823	D6423	KCl	8.1		0.86	
859			-----		-----	
912			-----		-----	
913			-----		-----	
922	D6423	KCl	7.92		0.38	
963			-----		-----	
1201			-----		-----	
1205			-----		-----	
1242			-----		-----	
1574			-----		-----	
1605			-----		-----	
1726	EN15490	LiCl	7.49		-0.77	
1727	EN15490	LiCl	7.56		-0.58	
1783			-----		-----	
1817	D6423	KCl	7.554		-0.60	
1835	EN15490	LiCl	7.66		-0.31	
1927			-----		-----	

normality	OK
n	11
outliers	0
mean (n)	7.778
st.dev. (n)	0.3290
R(calc.)	0.921
R(D6423:14)	1.046

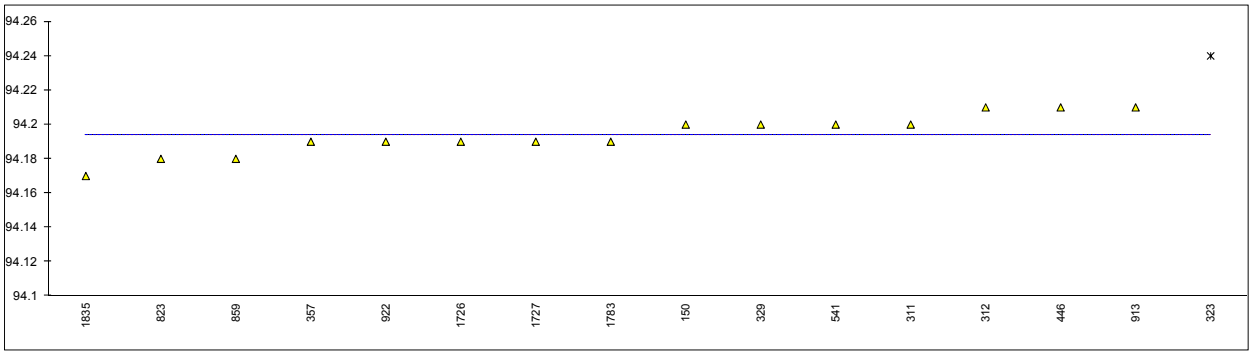


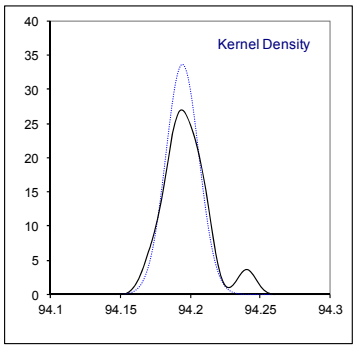


Determination of Strength on sample #16262; results in %M/M

lab	method	value	mark	z(targ)	remarks
150	Table OIML	94.2		-----	
171		-----		-----	
174		-----		-----	
230		-----		-----	
273		-----		-----	
311	Table OIML	94.20		-----	
312	Table OIML	94.21		-----	
323	Table OIML	94.24	G(0.05)	-----	
329	Table OIML	94.20		-----	
357	Table OIML	94.19		-----	
446	Table OIML	94.21		-----	
541	Table OIML	94.20		-----	
551		-----		-----	
558		-----		-----	
823	Table OIML	94.18		-----	
859	Table OIML	94.18		-----	
912		-----		-----	
913	Table OIML	94.21		-----	
922	Table OIML	94.19		-----	
963		-----		-----	
1201		-----		-----	
1205		-----		-----	
1242		-----		-----	
1574		-----		-----	
1605		-----		-----	
1726	Table OIML	94.19		-----	
1727	Table OIML	94.19		-----	
1783	Table OIML	94.19		-----	
1817		-----		-----	
1835	Table OIML	94.17		-----	
1927		-----		-----	

normality	OK	
n	15	
outliers	1	
mean (n)	94.194	
st.dev. (n)	0.01183	
R(calc.)	0.0331	
R(lit)	n.a.	Compare R(iis15C15) = 0.042

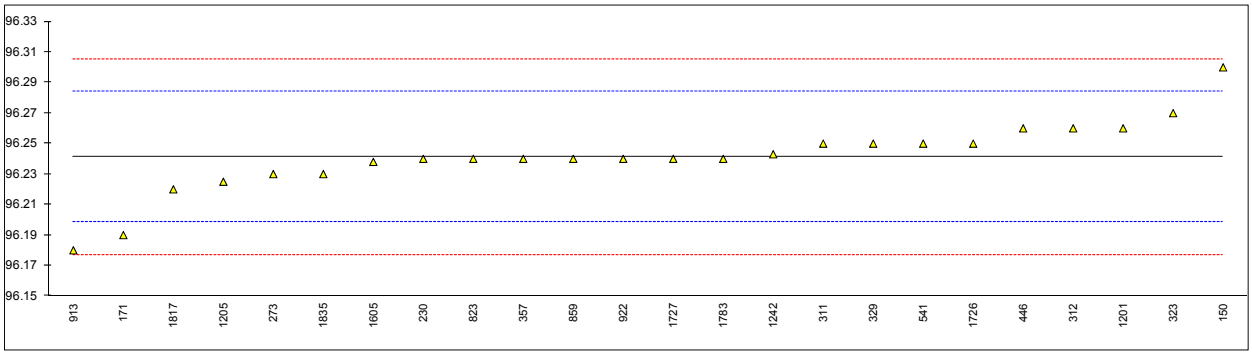


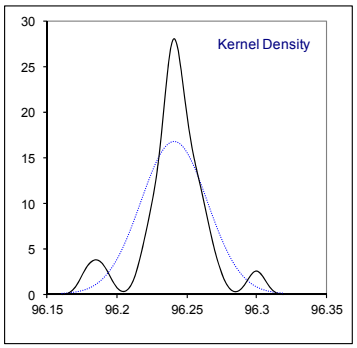


Determination of Strength on sample #16262; results in %V/V

lab	method	value	mark	z(targ)	remarks
150	Table OIML	96.3		2.75	
171	Table OIML	96.19		-2.38	
174		-----		-----	
230	Table OIML	96.24		-0.05	
273	Table OIML	96.23	C	-0.52	first reported: 96.35
311	Table OIML	96.25		0.42	
312	Table OIML	96.26		0.88	
323	Table OIML	96.27		1.35	
329	Table OIML	96.25		0.42	
357	Table OIML	96.24		-0.05	
446	Table OIML	96.26		0.88	
541	Table OIML	96.25		0.42	
551		-----		-----	
558		-----		-----	
823	Table OIML	96.24		-0.05	
859	Table OIML	96.24		-0.05	
912		-----		-----	
913	Table OIML	96.18		-2.85	
922	Table OIML	96.24		-0.05	
963		-----		-----	
1201	Table OIML	96.26		0.88	
1205	Table OIML	96.225		-0.75	
1242		96.243		0.09	
1574		-----		-----	
1605	Table OIML	96.238		-0.14	
1726	Table OIML	96.25		0.42	
1727	Table OIML	96.24		-0.05	
1783	Table OIML	96.24		-0.05	
1817	Table OIML	96.22	C	-0.98	first reported: 96.18
1835	Table OIML	96.23		-0.52	
1927		-----		-----	

normality	not OK
n	24
outliers	0
mean (n)	96.241
st.dev. (n)	0.0238
R(calc.)	0.067
R(OIML table)	0.060

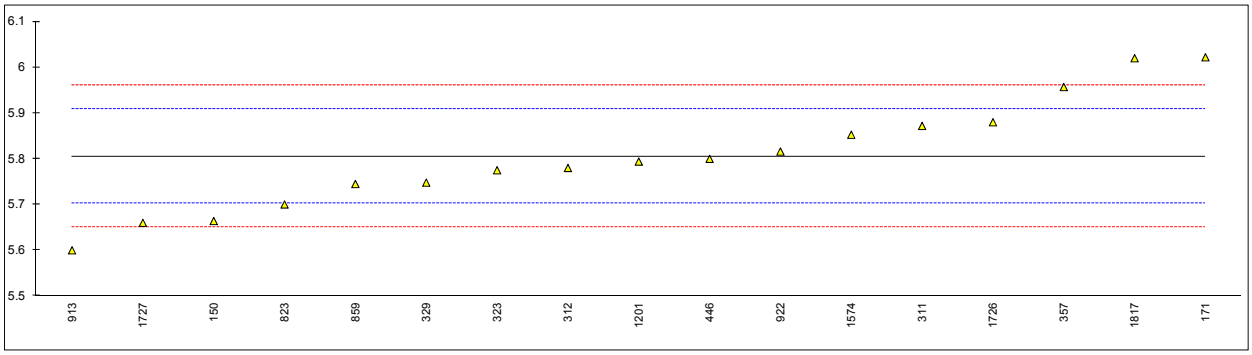


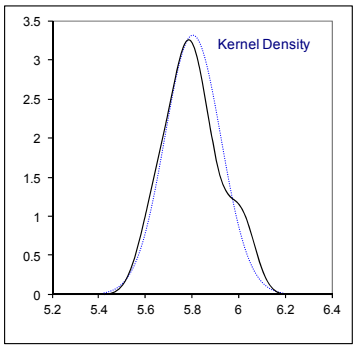


Determination of Water (Titrimetric) on sample #16262; results in %M/M

lab	method	value	mark	z(targ)	remarks
150	E203	5.664		-2.73	
171	E203	6.022		4.20	
174		-----		-----	
230		-----		-----	
273		-----		-----	
311	D1364	5.872		1.30	
312	E203	5.78	C	-0.49	
323	D1364	5.775		-0.58	
329	E203	5.748		-1.11	
357	E203	5.957		2.94	
446	E203	5.8		-0.10	
541		-----		-----	
551		-----		-----	
558		-----		-----	
823	D1364	5.7003		-2.03	
859	D1364	5.745		-1.16	
912		-----		-----	
913	D1364	5.60		-3.97	
922	D1364	5.816		0.21	
963		-----		-----	
1201	D1364	5.794		-0.21	
1205		-----		-----	
1242		-----		-----	
1574		5.8527		0.92	
1605		-----		-----	
1726	E203	5.88		1.45	
1727	D1364	5.66		-2.81	
1783		-----		-----	
1817		6.02		4.16	
1835		-----		-----	
1927		-----		-----	

normality	OK
n	17
outliers	0
mean (n)	5.8051
st.dev. (n)	0.12024
R(calc.)	0.3367
R(D1364:02)	0.1445

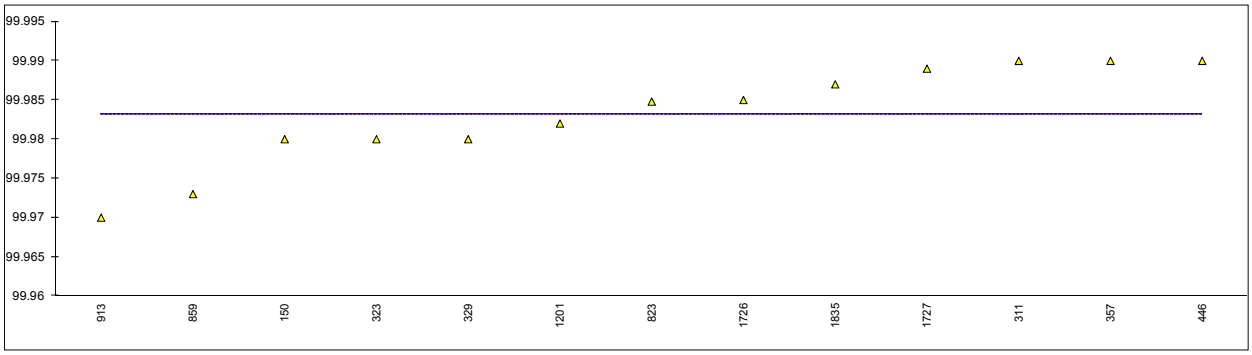


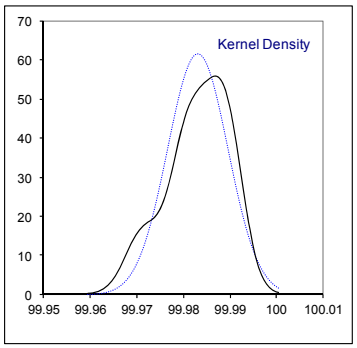


Determination of Purity of Ethanol on dry basis on sample #16262; results in %M/M

lab	method	value	mark	z(targ)	remarks
150	INH-0001	99.98		-----	
171		-----		-----	
174		-----		-----	
230		-----		-----	
273		-----		-----	
311	INH-529	99.99		-----	
312		-----		-----	
323	INH-0001	99.98		-----	
329		99.98		-----	
357	INH-0001	99.99		-----	
446		99.99		-----	
541		-----		-----	
551		-----		-----	
558		-----		-----	
823	INH-0001	99.9848		-----	
859	EN15721	99.973		-----	
912		-----		-----	
913	D5501	99.97		-----	
922		-----		-----	
963		-----		-----	
1201	In house	99.982		-----	
1205		-----		-----	
1242		-----		-----	
1574		-----		-----	
1605		-----		-----	
1726		99.985		-----	
1727		99.989		-----	
1783		-----		-----	
1817		-----		-----	
1835	In house	99.987		-----	
1927		-----		-----	

normality	OK	
n	13	
outliers	0	
mean (n)	99.9831	
st.dev. (n)	0.00647	
R(calc.)	0.0181	
R(lit.)	n.a.	R(iis15C15) = 0.1291 or R(iis14C11) = 0.0176

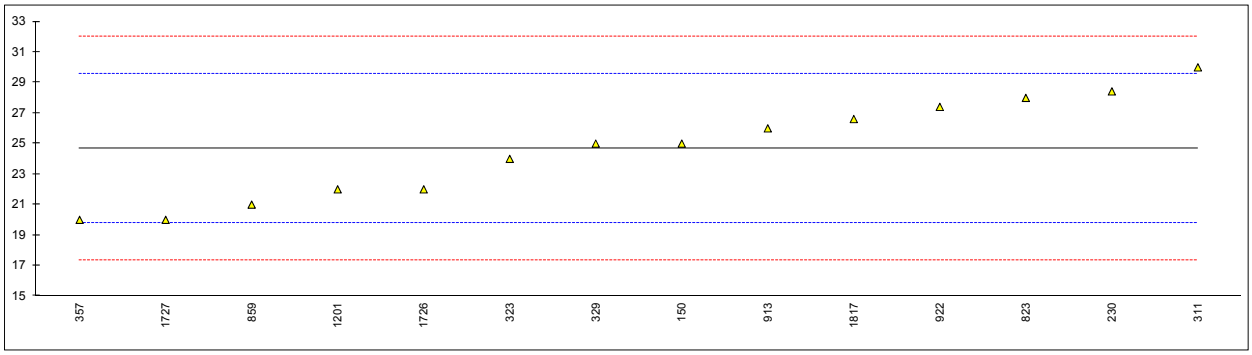


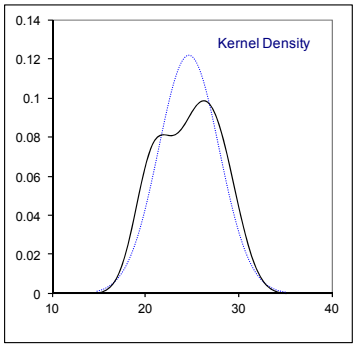


Determination of Methanol on sample #16262; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	INH-0001	25	C	0.13	first reported: 34
171		-----		-----	
174		-----		-----	
230		28.423		1.54	
273		-----		-----	
311	INH-529	30	C	2.19	first reported: 35
312		-----		-----	
323	INH-0001	24		-0.28	
329		25		0.13	
357	INH-0001	20		-1.92	
446		<30		-----	
541		-----		-----	
551		-----		-----	
558		-----		-----	
823	INH-0001	28		1.36	
859	EN15721	21		-1.51	
912		-----		-----	
913	INH-0001	26		0.54	
922	INH-0001	27.41		1.12	
963		-----		-----	
1201	In house	22		-1.10	
1205		-----		-----	
1242		-----		-----	
1574		-----		-----	
1605		-----		-----	
1726		22		-1.10	
1727		20		-1.92	
1783		-----		-----	
1817		26.61179		0.79	
1835	In house	<50		-----	
1927		-----		-----	

normality	OK		
n	14		
outliers	0	Spike:	
mean (n)	24.675	22.06	recovery <112%
st.dev. (n)	3.2657		
R(calc.)	9.144		
R(Horwitz)	6.823		

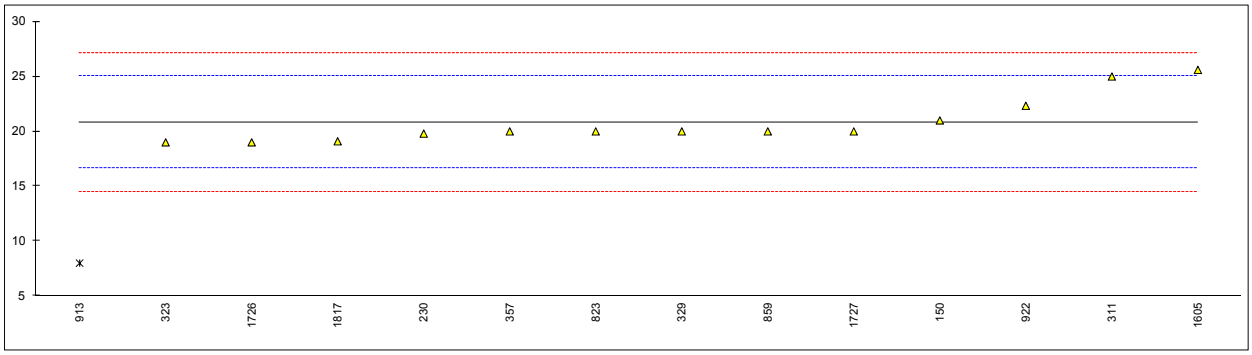


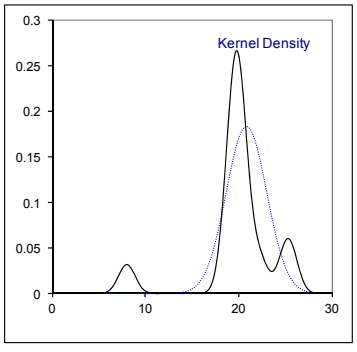


Determination of Acetal on sample #16262; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	INH-0001	21	C	0.08	first reported: 28
171		-----		-----	
174		-----		-----	
230		19.800		-0.49	
273		-----		-----	
311	INH-529	25		1.97	
312		-----		-----	
323	INH-0001	19		-0.87	
329		20		-0.39	
357	INH-0001	20		-0.39	
446		<10		<-10.43	possible false negative test result?
541		-----		-----	
551		-----		-----	
558		-----		-----	
823	INH-0001	20		-0.39	
859	EN15721	20		-0.39	
912		-----		-----	
913	INH-0001	8.0	G(0.01)	-6.08	
922	INH-0001	22.336		0.71	
963		-----		-----	
1201	In house	<0.5	C	<-19.57	first reported: 0, possible false negative test result?
1205		-----		-----	
1242		-----		-----	
1574		-----		-----	
1605		25.6		2.26	
1726		19		-0.87	
1727		20		-0.39	
1783		-----		-----	
1817		19.09981		-0.82	
1835	In house	<50		-----	
1927		-----		-----	

	normality	not OK	
	n	13	
	outliers	1	Spike:
	mean (n)	20.836	22.61 recovery <92%
	st.dev. (n)	2.1725	
	R(calc.)	6.083	
	R(Horwitz)	5.909	

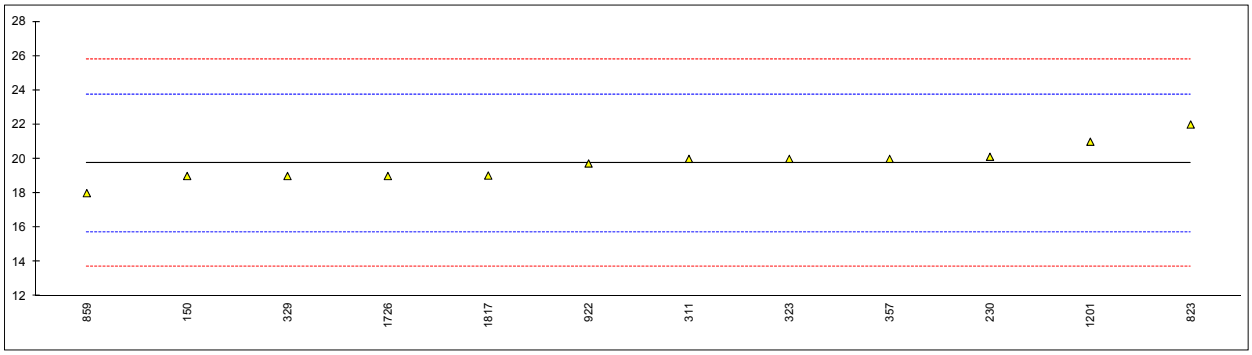


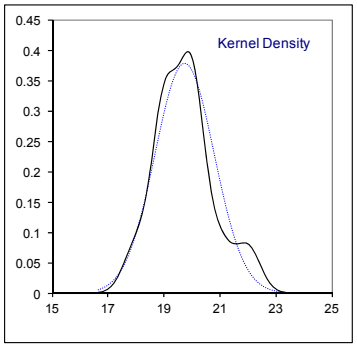


Determination of Benzene on sample #16262; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	INH-0001	19		-0.37	
171		-----		-----	
174		-----		-----	
230		20.121		0.19	
273		-----		-----	
311	INH-529	20		0.13	
312		-----		-----	
323	INH-0001	20		0.13	
329		19		-0.37	
357	INH-0001	20		0.13	
446		<20		-----	
541		-----		-----	
551		-----		-----	
558		-----		-----	
823	INH-0001	22		1.12	
859	EN15721	18		-0.86	
912		-----		-----	
913		-----		-----	
922	INH-0001	19.73		0.00	
963		-----		-----	
1201	In house	21	C	0.63	first reported: 0
1205		-----		-----	
1242		-----		-----	
1574		-----		-----	
1605		-----		-----	
1726		19		-0.37	
1727		-----		-----	
1783		-----		-----	
1817		19.02668		-0.35	
1835	In house	<50		-----	
1927		-----		-----	

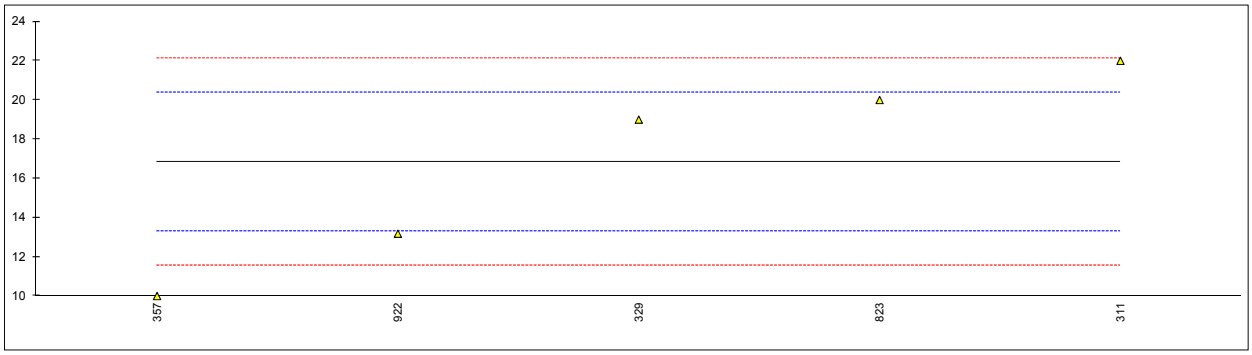
normality	OK		
n	12		
outliers	0	Spike:	
mean (n)	19.740	22.61	recovery <87%
st.dev. (n)	1.0541		
R(calc.)	2.951		
R(Horwitz)	5.645		





Determination of Mono Ethylene Glycol on sample #16262; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	INH-0001	<2		<-8.43	possible false negative test result?
171		-----		-----	
174		-----		-----	
230		-----		-----	
273		-----		-----	
311	INH-270	22		2.93	
312		-----		-----	
323		-----		-----	
329		19		1.23	
357	INH-0001	10	C	-3.88	first reported: <10
446		-----		-----	
541		-----		-----	
551		-----		-----	
558		-----		-----	
823	INH-0001	20		1.80	
859	EN15721	<10		<-3.88	possible false negative test result?
912		-----		-----	
913		-----		-----	
922	INH-0001	13.18		-2.08	
963		-----		-----	
1201		-----		-----	
1205		-----		-----	
1242		-----		-----	
1574		-----		-----	
1605		-----		-----	
1726		-----		-----	
1727		-----		-----	
1783		-----		-----	
1817		-----		-----	
1835		-----		-----	
1927		-----		-----	
	normality	unknown			
	n	5			
	outliers	0	Spike:		
	mean (n)	16.84	22.33		recovery <75%
	st.dev. (n)	5.036			
	R(calc.)	14.10			
	R(Horwitz)	4.93			

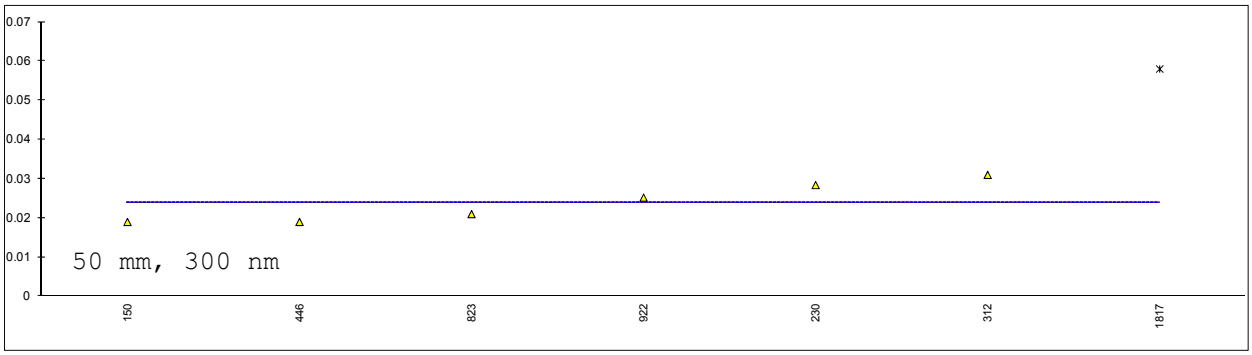


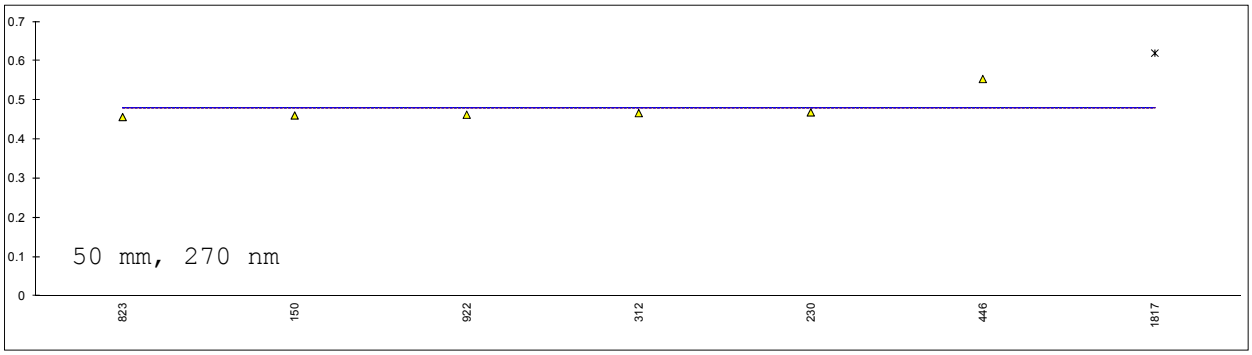
Determination of Acetaldehyde, Acetone and Isopropanol on sample #16262; results in mg/kg

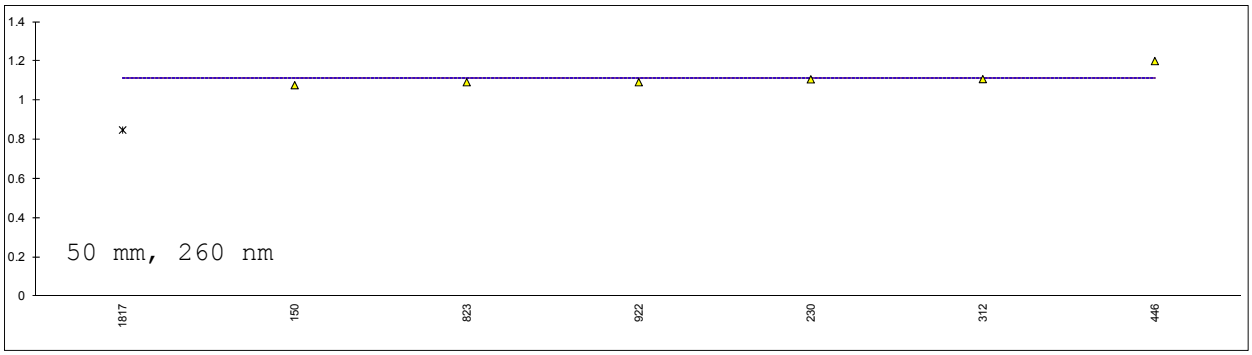
lab	method	Acetaldehyde		Acetone	i-Propanol	remarks
150	INH-0001	<2	C	<2	<2	first reported for Acetaldehyde: 14
171		-----		-----	-----	
174		-----		-----	-----	
230		<5		<2	2.255	
273		-----		-----	-----	
311	INH-529	10		<5	<5	
312		-----		-----	-----	
323	INH-0001	11	C	<5	<5	first reported for Acetaldehyde: 15
329		10	C	<5	<5	first reported for Acetaldehyde: 12
357	INH-0001	< 10		< 10	< 10	
446		<10		-----	<10	
541		-----		-----	-----	
551		-----		-----	-----	
558		-----		-----	-----	
823	INH-0001	5		<5	<5	
859	EN15721	<10		<10	<10	
912		-----		-----	-----	
913	INH-0001	<5		<5	<5	
922	INH-0001	<5		<2	<5	
963		-----		-----	-----	
1201	In house	<1		<1	0.7	
1205		-----		-----	-----	
1242		-----		-----	-----	
1574		-----		-----	-----	
1605		9.30		-----	-----	
1726		1.4		ND	1.2	
1727		<10		-----	<10	
1783		-----		-----	-----	
1817		ND		2.52116	-----	
1835	In house	<50		<50	<50	
1927		-----		-----	-----	
	n	14		12	14	
	mean (n)	≤10		<10	<10	

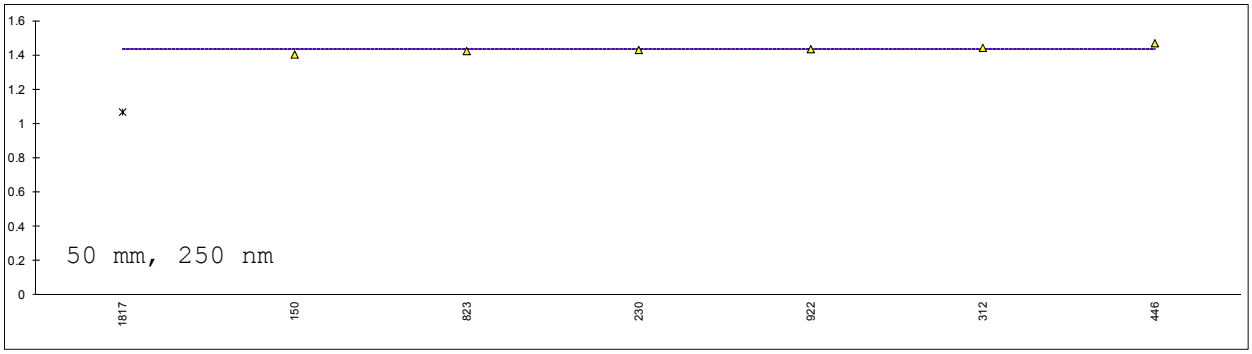
Determination of UV absorbance (50 mm cuvette) on sample #16262;

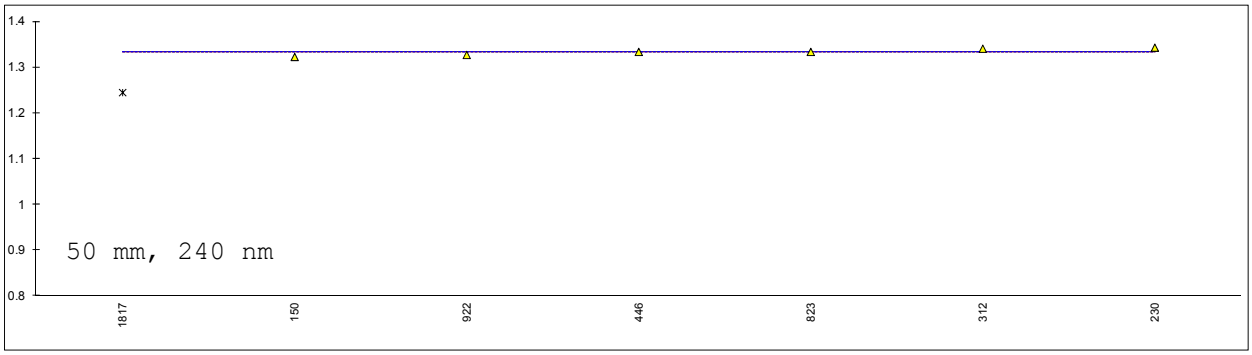
lab	method	300nm	270nm	260nm	250nm	240nm	230nm	220nm	Pass/Fail
150	IMPCA004	0.019	0.461	1.078	1.405	1.323	1.373	2.761	Fail
171		-----	-----	-----	-----	-----	-----	-----	-----
174		-----	-----	-----	-----	-----	-----	-----	-----
230	INH-13	0.0284	0.4688	1.1077	1.4316	1.3433	1.3815	3.1905	Fail
273		-----	-----	-----	-----	-----	-----	-----	-----
311		-----	-----	-----	-----	-----	-----	-----	-----
312	In house	0.031	0.467	1.109	1.444	1.341	1.396	3.469	Fail
323		-----	-----	-----	-----	-----	-----	-----	-----
329		-----	-----	-----	-----	-----	-----	-----	-----
357		-----	-----	-----	-----	-----	-----	-----	-----
446		0.019	0.554	1.201	1.471	1.334	1.365	3.228	Fail
541		-----	-----	-----	-----	-----	-----	-----	-----
551		-----	-----	-----	-----	-----	-----	-----	-----
558		-----	-----	-----	-----	-----	-----	-----	-----
823	INH-13	0.021	0.457	1.093	1.425	1.334	1.380	3.239	FAIL
859		-----	-----	-----	-----	-----	-----	-----	-----
912		-----	-----	-----	-----	-----	-----	-----	-----
913		-----	-----	-----	-----	-----	-----	-----	-----
922	In house	0.0252	0.4629	1.0931	1.4362	1.3273	1.3815	3.6568	Fail
963		-----	-----	-----	-----	-----	-----	-----	-----
1201		-----	-----	-----	-----	-----	-----	-----	-----
1205		-----	-----	-----	-----	-----	-----	-----	-----
1242		-----	-----	-----	-----	-----	-----	-----	-----
1574		-----	-----	-----	-----	-----	-----	-----	-----
1605		-----	-----	-----	-----	-----	-----	-----	-----
1726		-----	-----	-----	-----	-----	-----	-----	-----
1727		-----	-----	-----	-----	-----	-----	-----	-----
1783		-----	-----	-----	-----	-----	-----	-----	-----
1817		**0.058**	0.620 ex	**0.849**	**1.069**	**1.245**	-----	-----	-----
1835		-----	-----	-----	-----	-----	-----	-----	-----
1927		-----	-----	-----	-----	-----	-----	-----	-----
	normality	unknown	unknown	unknown	unknown	unknown	unknown	unknown	n.a.
	n	6	6	6	6	6	6	6	6
	outliers	1	0 (+1ex)	1	1	1	0	0	0
	mean (n)	0.0239	0.4784	1.1136	1.4355	1.3338	1.3795	3.2574	Fail
	st.dev. (n)	0.00507	0.03725	0.04429	0.02186	0.00776	0.01032	0.30212	n.a.
	R(calc.)	0.0142	0.1043	0.1240	0.0612	0.0217	0.0289	0.8459	n.a.

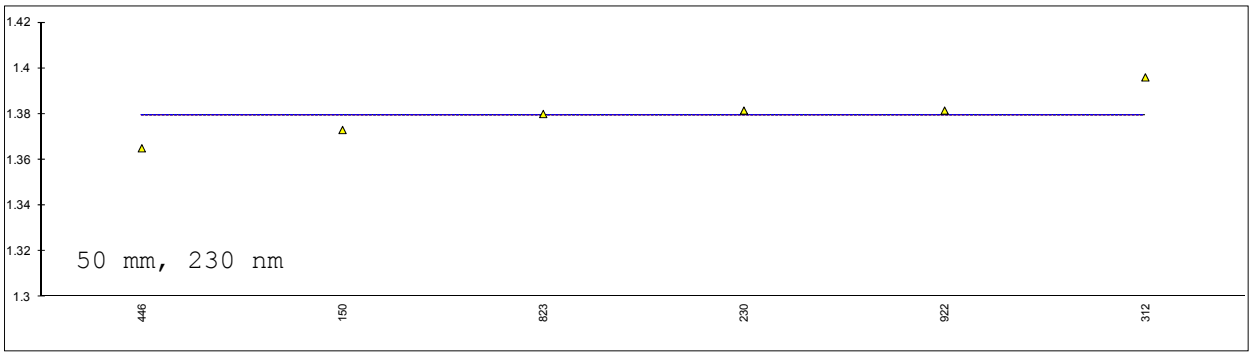


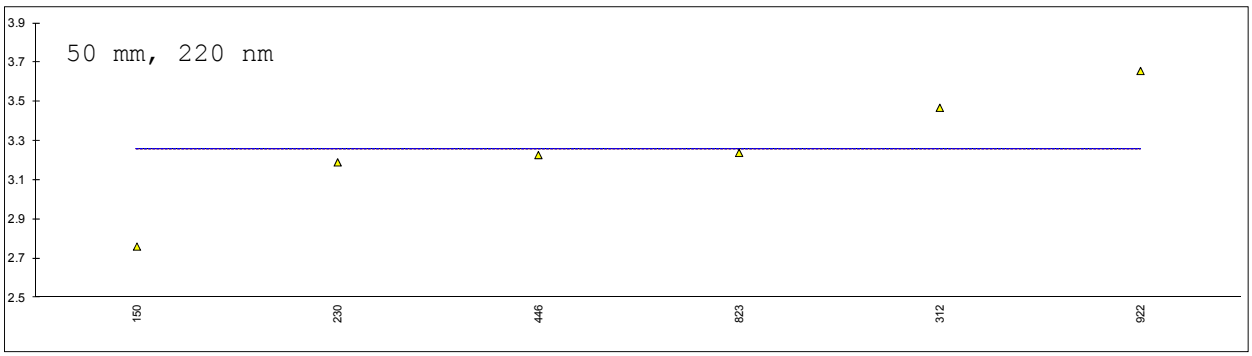



50 mm, 260 nm
1817
150
823
922
230
312
446


50 mm, 250 nm
1817
150
823
230
922
312
446


50 mm, 240 nm
1817
150
922
446
823
312
230

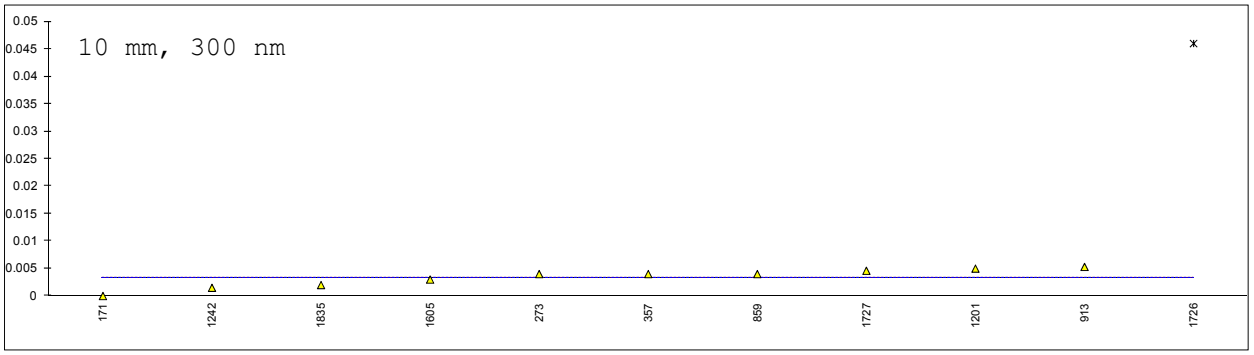

50 mm, 230 nm
446
150
823
230
922
312

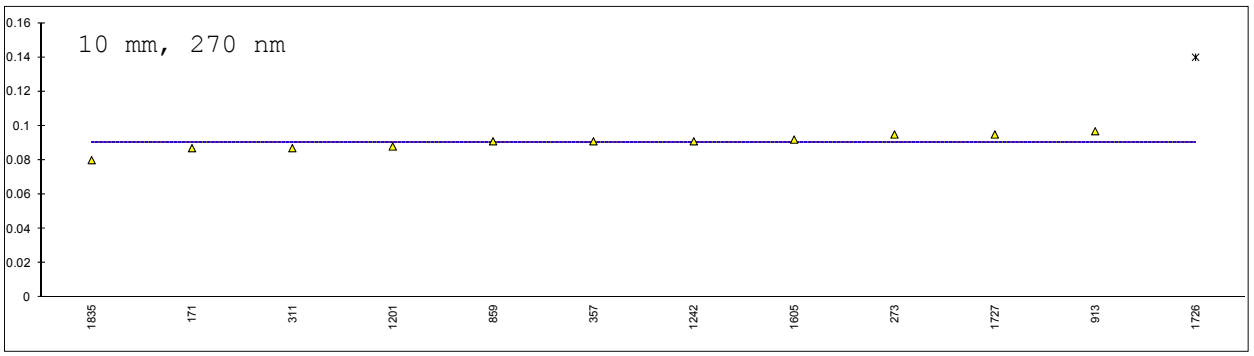

50 mm, 220 nm
150
230
446
823
312
922

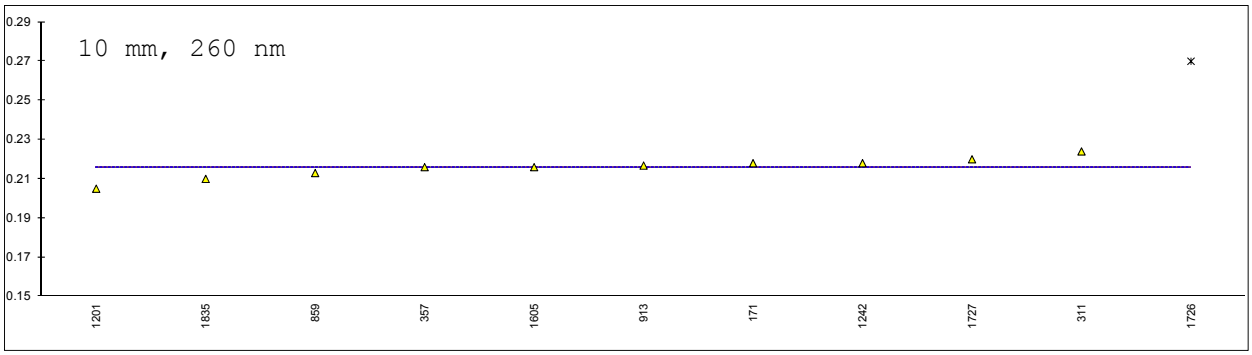
Determination of UV absorbance (10 mm cuvette) on sample #16262;

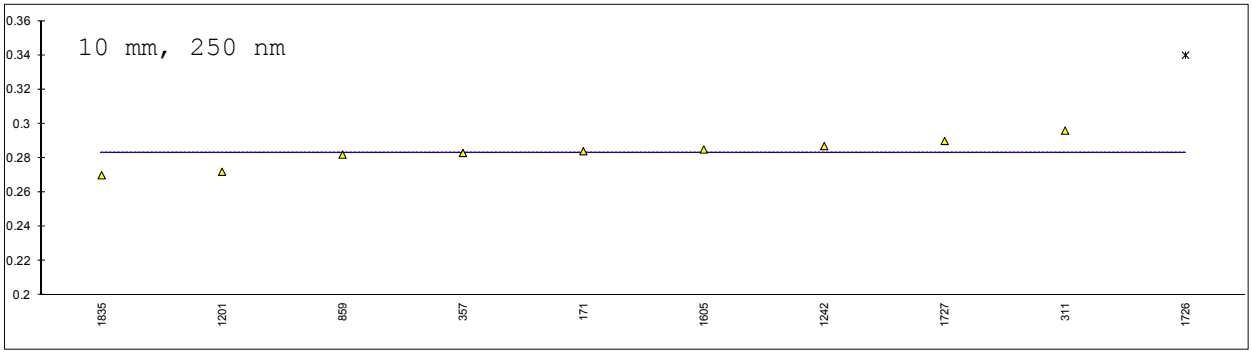
lab	method	300nm	270nm	260nm	250nm	240nm	230nm	220nm	Pass/Fail
150		-----	-----	-----	-----	-----	-----	-----	-----
171		0.000	0.087	0.218	0.284	0.270	0.281	1.301	Fail
174		-----	-----	-----	-----	-----	-----	-----	-----
230		-----	-----	-----	-----	-----	-----	-----	-----
273	IMPCA004	0.004 C	0.095 C	-----	-----	0.264 C	0.287 C	1.313 C	-----
311	INH-094	<0.005	0.087	0.224 C	0.296 C	0.276 C	0.254	1.288	Fail
312		-----	-----	-----	-----	-----	-----	-----	-----
323		-----	-----	-----	-----	-----	-----	-----	-----
329		-----	-----	-----	-----	-----	-----	-----	FAIL
357	INH-13	0.004	0.091	0.216	0.283	0.265	0.274	1.287	Fail
446		-----	-----	-----	-----	-----	-----	-----	-----
541		-----	-----	-----	-----	-----	-----	-----	-----
551		-----	-----	-----	-----	-----	-----	-----	-----
558		-----	-----	-----	-----	-----	-----	-----	-----
823		-----	-----	-----	-----	-----	-----	-----	-----
859	INH-13	0.004	0.091	0.213	0.282	0.264	0.274	1.286	Fail
912		-----	-----	-----	-----	-----	-----	-----	-----
913	IPMCA	0.0053	0.0969	0.2168	-----	0.2625	0.2771	1.3631	Fail
922		-----	-----	-----	-----	-----	-----	-----	-----
963		-----	-----	-----	-----	-----	-----	-----	-----
1201	IMPCA004	0.005	0.088	0.205	0.272	0.249	0.255	1.220	Fail
1205		-----	-----	-----	-----	-----	-----	-----	-----
1242		0.0015	0.0910	0.2180	0.2870	0.2635	0.2730	1.2875	-----
1574		-----	-----	-----	-----	-----	-----	-----	-----
1605		0.003	0.092	0.216	0.285	0.263	0.270	1.223	-----
1726		**0.046**	**0.14**	**0.27**	**0.34**	**0.32**	**0.33**	1.36	Pass fp+?
1727		0.0046	0.095	0.22	0.29	0.27	0.28	1.32	Pass fp+?
1783		-----	-----	-----	-----	-----	-----	-----	-----
1817		-----	-----	-----	-----	-----	-----	-----	-----
1835	In house	0.002	0.08	0.21	0.27	0.26	0.26	1.19	Pass fp+?
1927		-----	-----	-----	-----	-----	-----	-----	-----
	normality	OK	suspect	OK	OK	not OK	OK	OK	n.a.
	n	10	11	10	9	11	11	12	7
	outliers	1	1	1	1	1	1	0	3 (Pass)
	mean (n)	0.0033	0.0904	0.2157	0.2832	0.2643	0.2714	1.2865	Fail
	st.dev. (n)	0.00170	0.00475	0.00531	0.00814	0.00679	0.01078	0.05315	n.a.
	R(calc.)	0.0048	0.0133	0.0149	0.0228	0.0190	0.0302	0.1488	n.a.
	R(iis15C15)	0.0067 at abs = 0.0181							

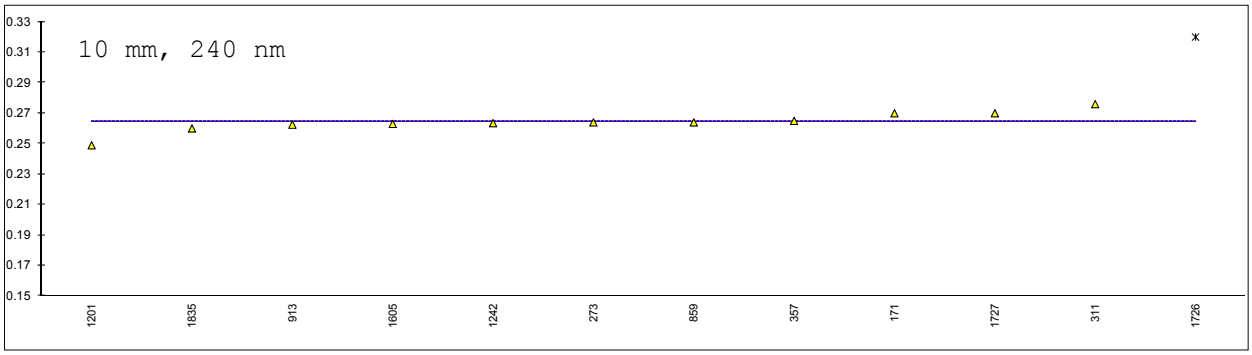
Lab 273 first reported for 300 nm 0.036, for 270 nm 0.133, for 240 nm 0.307, for 230 nm 0.380, for 220 nm 1.441
Lab 311 first reported for 260 nm 0.197, for 250 nm 0.238, for 240 nm 0.254

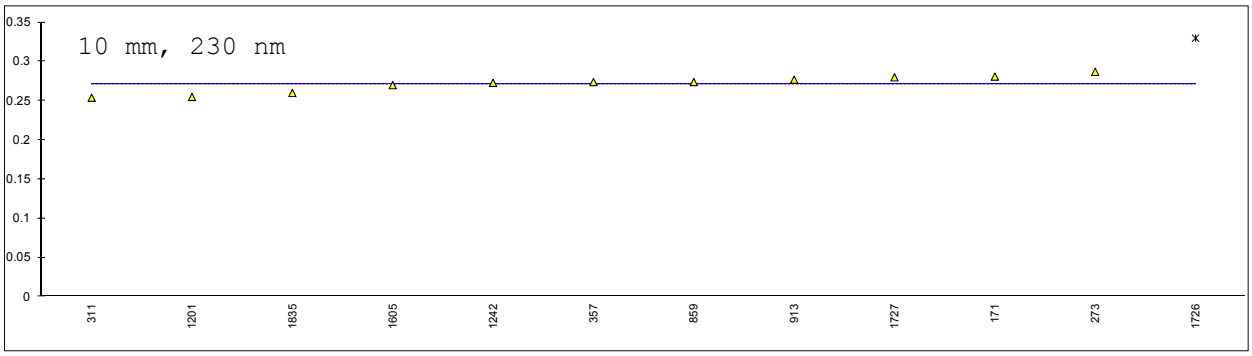


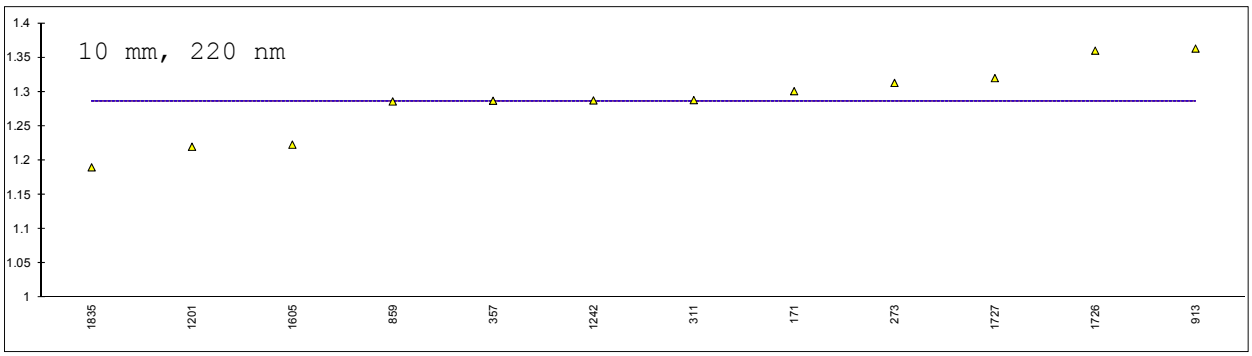



10 mm, 260 nm
0.29
0.27
0.25
0.23
0.21
0.19
0.17
0.15
1201
1835
859
357
1605
913
171
1242
1727
311
1726


10 mm, 250 nm
0.36
0.34
0.32
0.3
0.28
0.26
0.24
0.22
0.2
1835
1201
859
357
171
1605
1242
1727
311
1726


10 mm, 240 nm
0.33
0.31
0.29
0.27
0.25
0.23
0.21
0.19
0.17
0.15
1201
1835
913
1605
1242
273
859
357
171
1727
311
1726


10 mm, 230 nm
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
311
1201
1835
1605
1242
357
859
913
1727
171
273
1726


10 mm, 220 nm
1.4
1.35
1.3
1.25
1.2
1.15
1.1
1.05
1
1835
1201
1605
859
357
1242
311
171
273
1727
1726
913

APPENDIX 2

Number of participants per country

1 lab in ARGENTINA
1 lab in AUSTRALIA
4 labs in BELGIUM
2 labs 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
4 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
3 labs 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
ex	= test result excluded from statistical evaluations
n.a.	= not applicable
OILM	= International Organization of Legal Metrology
U	= test result probably reported in a different unit
SDS	= safety data sheet

Literature:

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4	ASTM E1301:2003
5	ISO13528:2005
5	ISO 5725:1986
6	ISO 5725, parts 1-6, 1994
7	M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
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9	IP 367/84
10	DIN 38402 T41/42
11	P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
12	J.N. Miller, Analyst, 118, 455, (1993)
13	Analytical Methods Committee Technical brief, No 4.February 2001
14	P.J. Lowthian and M. Thompson. The Royal Society of Chemistry 2002, Analyst, 2002, 127, page 1359-1364, (see http://www.rsc.org/suppdata/an/b2/b205600n/)
15	Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), pages 165-172, (1983)
16	M.A. Gonçalves et.al., Sensors and Actuators B158 (2011) pages 327-332