Results of Proficiency Test REN/Food Ethanol November 2012

Organised by: Institute for Interlaboratory Studies (iis)

Spijkenisse, the Netherlands

ing. R.J. Starink

Authors: Correctors: Report: dr. R.G. Visser & ing. N. Boelhouwer

Report: iis12C12

February 2013

CONTENTS

1	INTRODUCTION	3
2	SET UP	3
2.1	ACCREDITATION	3
2.2	PROTOCOL	3
2.3	CONFIDIENTIALITY STATEMENT	3
2.4	SAMPLES	4
2.5	STABILITY	4
2.6	ANALYSES	4
3	RESULTS	5
3.1	STATISTICS	5
3.2	GRAPHICS	6
3.3	Z-SCORES	6
4	EVALUATION	7
4.1	EVALUATION PER TEST	7
4.2	PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES	9
4.3	COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2012 WITH PREVIOUS PTS	10

Appendices:

1.	Data and statistical results	11
2.	Analytical Details	20
3.	Number of participants per country	21
4	Abbreviations and literature	22

1. INTRODUCTION

Since 2007, a proficiency test for REN/Food Ethanol is organised every year by the Institute for Interlaboratory Studies. During the planning of the annual proficiency testing program 2012/2013, it was decided to continue the round robin for the analysis of REN/Food grade Ethanol.

In this interlaboratory study, 27 laboratories in 15 different countries have participated. See appendix 3 for the number of participants per country. In this report, the results of the 2012 proficiency test are presented and discussed.

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. It was decided to send one sample (1* 0.5 L of 95% REN/Food grade Ethanol, labelled #12152). Participants were requested to report rounded and unrounded results. The unrounded 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 17043:10, (R007) since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially 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' (iis-protocol, version 3.2) of January 2010.

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 #12152 was obtained from a local trader. The approximately 50 litre bulk sample was, after homogenisation in a precleaned can, divided over 50 amber glass bottles of 0.5 L and labelled #12152. The homogeneity of these subsamples was checked by determination of Density in accordance with ASTM D4052:02e1 and Water in accordance with ASTM D1364:07 on 8 stratified randomly selected samples.

Sample	Density @ 20°C in kg/L	Water in %M/M		
Sample #12152-1	0.80559	5.483		
Sample #12152-2	0.80557	5.471		
Sample #12152-3	0.80558	5.482		
Sample #12152-4	0.80556	5.475		
Sample #12152-5	0.80558	5.474		
Sample #12152-6	0.80559	5.479		
Sample #12152-7	0.80558	5.483		
Sample #12152-8	0.80558	5.491		

table 1: Homogeneity test results of subsamples #12152

From the test results of table 1, the repeatabilities were 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 @ 20°C in kg/L	Water in %M/M		
r (Observed)	0.00003	0.018		
reference method	ASTM D4052:02e1	ASTM D1364:07		
0.3 * R (ref. method)	0.00015	0.042		

table 2: Repeatability of subsamples #12152

The repeatabilities of the results from the homogeneity test were in agreement with the requirements of the respective standards. Therefore, homogeneity of all the prepared subsamples was assumed.

To each of the participating laboratories 1*0.5 L bottle of sample #12152 was sent on October 31, 2012.

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 #12152: Density @ 20°C, Nonvolatile matter, Permanganate Time Test, Purity on dry basis, Water (titrimetric), Strength (in %V/V and %M/M) and UV transmittance at 300, 270, 240, 230 and 220nm.

To get comparable results a detailed report form, on which the units were prescribed as well as some of the required standards and a letter of instructions were prepared and made available for download on the iis website (www.iisnl.com).

A SDS and a form to confirm receipt of the samples were added to the sample package.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were received. The original reported results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after deadline, a reminder fax was sent to those laboratories that had not yet reported any results at that moment.

Shortly after the deadline, the available results were screened for suspect data. A 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 results. Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (iis.-protocol, version 3.2) of January 2010.

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. 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. After removal of outliers, this check was repeated. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation should be used with due care.

In accordance with ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs 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 under 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 (see appendix 4, nos.13-14).

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 spread of 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, literature repeatability is available; in other cases, a reproducibility of a former iis proficiency test could be used and the Horwitz equation can be used to estimate target reproducibility.

The z-scores were calculated according to:

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

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 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 the fit-for-useness of the reported test result.

4. EVALUATION

In this proficiency test, some problems were encountered with despatch of the samples. Three participants reported results after the final reporting date and three participants did not report any results at all. Not all laboratories were able to perform all analysis requested. Finally, the 24 reporting laboratories did send in 169 (numerical) results. Observed were 5 outlying results, which is 3.0%. In proficiency studies, outlier percentages of 3% - 7.5% are normal.

4.1 EVALUATION PER TEST

In this section, the 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 4.

On the registration form the participants were asked to fill out the analytical details regarding the strength determination and UV absorbance. Nineteen laboratories answered the questions fully or partially (See Appendix 2). From the analytical details none of the participants did perform a distillation before the strength determination and all participants used the density meter for the strength determination.

A not normal distribution was found for the following determinations: Purity, Strength %V/V and UV300nm, 240nm and 220nm. In this case the statistical evaluations should be used with due care.

Density:

This determination was problematic for two laboratories. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D4052:02e1.

Nonvolatile matter: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D1353:09.

Water:

This determination was very problematic. Two statistical outliers were observed. The calculated reproducibility, after rejection of the statistical outliers, is not at all in agreement with the requirements of ASTM D1364:12.

- <u>Permanganate Time Test:</u> This determination was problematic. No statistical outliers were observed and the calculated reproducibility is not in agreement with the requirements of ASTM D1363:11.
- Purity on dry basis: Regretfully, no standard test method with precision data exists.

 Therefore no conclusions were drawn. No statistical outliers were observed. The calculated reproducibility is small in comparison with the calculated reproducibility of the previous proficiency test (iis11C13a) of November 2011 (0.008 vs 0.011). The large spread may partly be explained by rounding of the test results.
- Strength (%V/V): This determination may be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the reproducibility derived from the OIML table and ASTM D4052:02e1 and large in comparison with iis11C13a of November 2011 (0.062 vs. 0.038).
- Strength(%M/M): This determination may be problematic. Regretfully, no standard test method with precision data exists. The calculated reproducibility is large in comparison with the calculated reproducibility in the previous proficiency test (iis11C13a) of November 2011 (0.107 vs 0.044).
- UV absorbance: Regretfully, no standard test method with precision data exists.

 Therefore no significant conclusions were drawn.

 No statistical outliers were observed. The calculated reproducibilities are all large in comparison with the calculated reproducibilities from the previous proficiency test (iis11C13a) of November 2011, except for 230nm.

From the analytical details, it is clear that almost all participants measured the UV absorbance against water and used a 10 mm cuvette, except laboratories 1067, 1817 and 1917. These participants used respectively a 50 mm cuvette. Laboratory used a 5mm cuvette.

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 average results per sample, calculated reproducibilities and reproducibilities derived from literature standards (in casu ASTM, EN standards) or previous proficiency tests are compared in the next table.

Parameter	unit	n	average	2.8 *sd _R	R (lit)
Density @ 20°C	kg/L	21	0.8055	0.0002	0.0005
Nonvolatile matter	mg/100mL	5	1.0	1.5	2.4
Water	%M/M	14	5.49	0.25	0.14
Permanganate Time Test	min.	11	22.5	7.1	5.7
Purity on dry basis	%M/M	12	99.991	0.008	(0.011)
Strength	%V/V	19	96.459	0.062	0.040
Strength	%M/M	11	94.515	0.107	(0.044)
UV-absorbance 300 nm		8	<0.001	0.008	(0.005)
UV-absorbance 270 nm		12	0.083	0.017	(800.0)
UV-absorbance 240 nm		12	0.105	0.019	(0.018)
UV-absorbance 230 nm		12	0.158	0.027	(0.035)
UV-absorbance 220 nm		12	1.355	0.162	(0.059)

Table 3: Reproducibilities of sample #12152

Results between brackets are compared with the spread of the previous proficiency test.

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2012 WITH PREVIOUS PT'S

	November 2012	November 2011	November 2010	December 2009
Number of reporting labs	24	23	28	31
Number of results reported	169	151	189	299
Number of statistical outliers	5	9	13	34
Percentage outliers	3.0%	6.0%	6.9%	11.4%

Table 4: 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 the following table:

Parameter	November 2012	November 2011	November 2010	December 2009
Density @ 20°C	++	++	++	++
Nonvolatile matter	++	n.e	++	n.e.
Water		+/-	++	
Permanganate Time Test		()	()	
Purity on dry basis	(+)	(+)	()	(++)
Strength %V/V		++	++	-
Strength %M/M	()	++	()	(++)
UV-absorbance 300 nm	(-)	()	(++)	(++)
UV-absorbance 270 nm	()	(-)	(++)	(++)
UV-absorbance 240 nm	(+/-)	(-)	(++)	(++)
UV-absorbance 230 nm	(++)	(-)	(+)	(++)
UV-absorbance 220 nm	()	(-)	(-)	(++)

Table 5: comparison determinations of sample #12152 against the standard

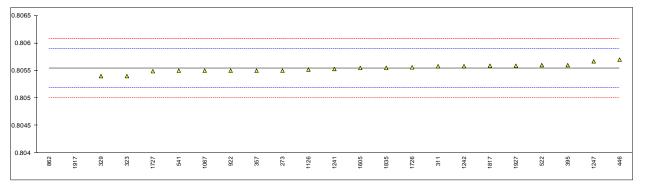
results between brackets are compared with the spread of the previous round robin

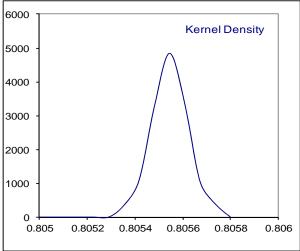
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

- ++: 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

Determination of Density @ 20°C on sample #12152; results in kg/L

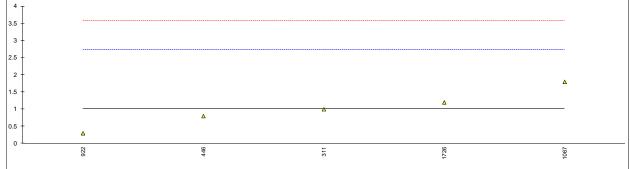
lab	method	value	mark	z(targ)	remarks
273	D4052	0.8055		-0.24	
311	D4052	0.80558		0.21	
323	D4052	0.8054		-0.80	
329	D4052	0.8054		-0.80	
357	D4052	0.8055		-0.24	
395	D4052	0.8056		0.32	
446	D4052	0.8057		0.88	
522	D4052	0.8056		0.32	
541	D4052	0.8055		-0.24	
551					
556					
559					
862	D4052	0.78967	G(0.01)	-88.89	
922	D4052	0.8055		-0.24	
1067	D4052	0.8055		-0.24	
1126	ISO12185	0.80552	С	-0.13	
1241	D4052	0.805530		-0.07	
1242	D4052	0.805581		0.21	
1247	INH-4500	0.80567		0.71	
1574	_				
1605	D4052	0.805548		0.03	
1726	D4052	0.80556		0.09	
1727	D4052	0.80549		-0.30	
1817	INH-26	0.80559		0.26	
1835	D4052	0.80555		0.04	
1917	B. 10=0	0.79	CG(0.01)	-87.04	
1927	D4052	0.80559		0.26	
	normality	ОК			
	n	21			
	outliers	2			
	mean (n)	0.80554			
	st.dev. (n)	0.000074			
	R(calc.)	0.00021			
	R(D4052:02e1)	0.00050			
	(= :::2:0201)				





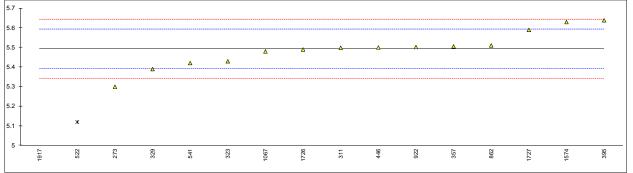
Determination of Nonvolatile matter on sample #12152; results in mg/100mL

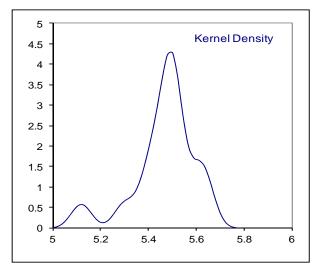
lab	method	value	mark	z(targ)	remarks
273					
311	D1353	1.0		-0.02	
323	D1353	<1			
329	D1353	<1			
357	D1353	<1			
395					
446	D1353	8.0		-0.26	
522					
541	EN15691	<10			
551					
556					
559					
862					
922	D1353	0.30		-0.84	
1067	D1353	1.8		0.91	
1126					
1241					
1242					
1247					
1574					
1605					
1726	D1353	1.2		0.21	
1727					
1817					
1835	EN15691	<10			
1917					
1927					
	normality	OK			
	n	5			
	outliers	0			
	mean (n)	1.02			
	st.dev. (n)	0.550			
	R(calc.)	1.54			
	R(D1353:09)	2.40			
	(/	-			
4 T					
3.5 -					
5.5					



Determination of Water (Titrimetric) on sample #12152; results in %M/M

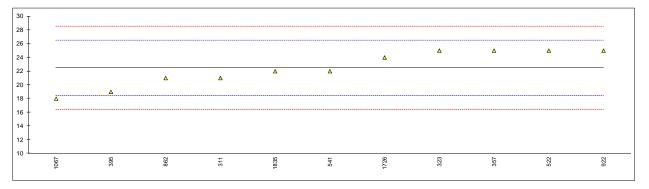
lab	method	value	mark	z(targ)	remarks
273	E203	5.30		-3.82	
311	D1364	5.499		0.14	
323	D1364	5.43		-1.24	
329	E203	5.39		-2.03	
357	D1364	5.506		0.28	
395	D1364	5.639		2.93	
446	E203	5.5005		0.17	
522	E203	5.12	G(0.05)	-7.41	
541	D1364	5.421		-1.41	
551					
556					
559					
862	D1364	5.5108		0.37	
922	E203	5.5025		0.21	
1067	D1364	5.48	С	-0.24	First reported 4.48
1126					
1241					
1242					
1247					
1574	INH-76	5.6298		2.74	
1605					
1726	D1364	5.4901		-0.04	
1727	D1364	5.59		1.95	
1817					
1835					
1917		0.2446	G(0.01)	-104.49	
1927					
					Only ASTM D1364 data:
	normality	OK			OK
	n	14			9
	outliers	2			0
	mean (n)	5.4921			5.5073
	st.dev. (n)	0.09084			0.06958
	R(calc.)	0.2544			0.1948
	R(D1364:12)	0.1406			0.1408
	,				
5.7 _T					
." [
.6					<u>~</u> _

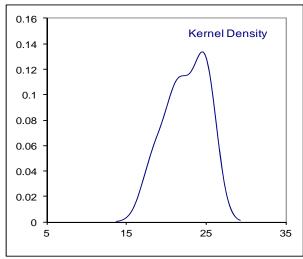




Determination of Permanganate Time Test @ 15 °C on sample #12152; results in minutes

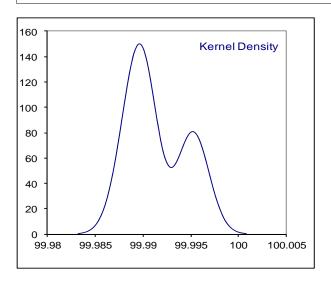
lab	method	value	mark	z(targ)	remarks
273					
311	D1363	21		-0.72	
323	D1363	25		1.26	
329	D1363	>20		>-1.42	
357	D1363	25		1.26	
395	D1363	19		-1.71	
446					
522	D1363	25		1.26	
541	D1363	22		-0.22	
551					
556					
559					
862	D1613	21		-0.72	
922	D1363	25		1.26	
1067	D1363	18		-2.20	
1126					
1241					
1242					
1247					
1574					
1605					
1726	D1363	24		0.76	
1727					
1817					
1835	D1363	22		-0.22	
1917					
1927					
	normality	OK			
	n	11			
	outliers	0			
	mean (n)	22.5			
	st.dev. (n)	2.54			
	R(calc.)	7.1			
	R(D1363:11)	5.7			
	. ,				





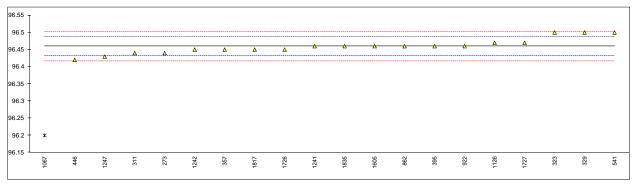
Determination of Purity on dry basis on sample #12152; results in %M/M

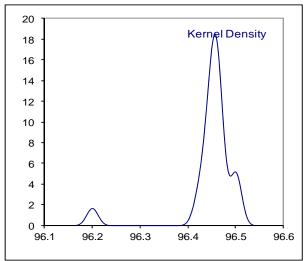
lab	method		value	mark	z(targ)	ren	narks					
273												
311	INH-529		99.99									
323	INH-001		99.99									
329			99.99									
357	EN15721		99.996									
395												
446	INH-17		99.995									
522	INH-GC		99.995									
541 551												
556												
559												
862	INH-001		99.99									
922	INH-001		99.988									
1067			99.99									
1126												
1241												
1242												
1247												
1574												
1605												
1726	in house		99.9884									
1727			99.9950									
1817												
1835	in house		99.9896									
1917												
1927												
	normality		not OK									
	n		12									
	outliers		0									
	mean (n)		99.9914									
	st.dev. (n		0.00292									
	R(calc.)	,	0.0082									
	R(lit)		unknown			Cor	npare R(iis	11C13a) =	0.011			
99.998 _T												
99.996 -												Δ
									Δ	Δ	Δ	-
99.994 -												
99.992 -												
99.99 -			Δ	Δ	Δ	Δ	Δ	Δ				
99.988 -	Δ	Δ										
99.986 -												
99.984 -												
99.982 -												
99.98	922	1726	1835	862	311	323	329	- 28	1727	9446	225	327
	8	4	₩	₩	'n	ನ	%	1067	17.	4	22	76



Determination of Strength on sample #12152; results in %V/V

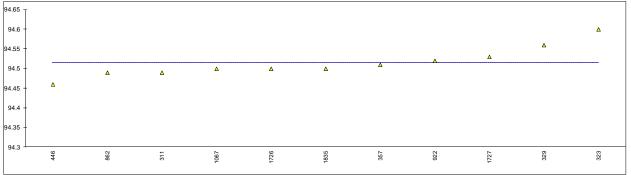
lab	method	value	mark	z(targ)	remarks
273	OIML	96.44		-1.36	
311	OIML	96.44		-1.36	
323	CalcD4052	96.5		2.84	
329		96.5		2.84	
357		96.45		-0.66	
395	OIML	96.46		0.04	
446	OIML	96.42		-2.76	
522					
541	ISO758	96.5		2.84	
551					
556					
559					
862	AlcTable	96.46		0.04	
922	OIML	96.46		0.04	
1067		96.2	G(0.01)	-18.16	
1126		96.47		0.74	
1241	AlcTable	96.460		0.04	
1242		96.45		-0.66	
1247	OIML	96.43		-2.06	
1574					
1605		96.46		0.04	
1726	OIML	96.45		-0.66	
1727		96.47		0.74	
1817	INH-26	96.45		-0.66	
1835	OIML	96.46		0.04	
1917					
1927					
	normality	not OK			
	n	19			
	outliers	1			
	mean (n)	96.459			
	st.dev. (n)	0.0220			
	R(calc.)	0.062			
	R(OIML table)	0.040			Compare R(iis11C13a) = 0.038
		3.0.0			

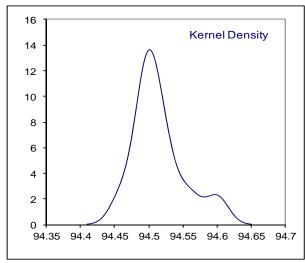




Determination of Strength on sample #12152; results in %M/M

lab	method	value	mark	z(targ)	remarks
273					
311	OIML	94.49			
323	CalcD4052	94.6			
329		94.56			
357		94.51			
395					
446	OIML	94.46			
522					
541					
551					
556					
559					
862	AlcTable	94.49			
922	OIML	94.52			
1067		94.5			
1126					
1241					
1242					
1247					
1574					
1605					
1726	OIML	94.50			
1727		94.53			
1817					
1835	OIML	94.50			
1917					
1927					
	normality	OK			
	n	11			
	outliers	0			
	mean (n)	94.515			
	st.dev. (n)	0.0380			
	R(calc.)	0.107			
	R(lit)	unknown			Compare R(iis11C13a) = 0.044

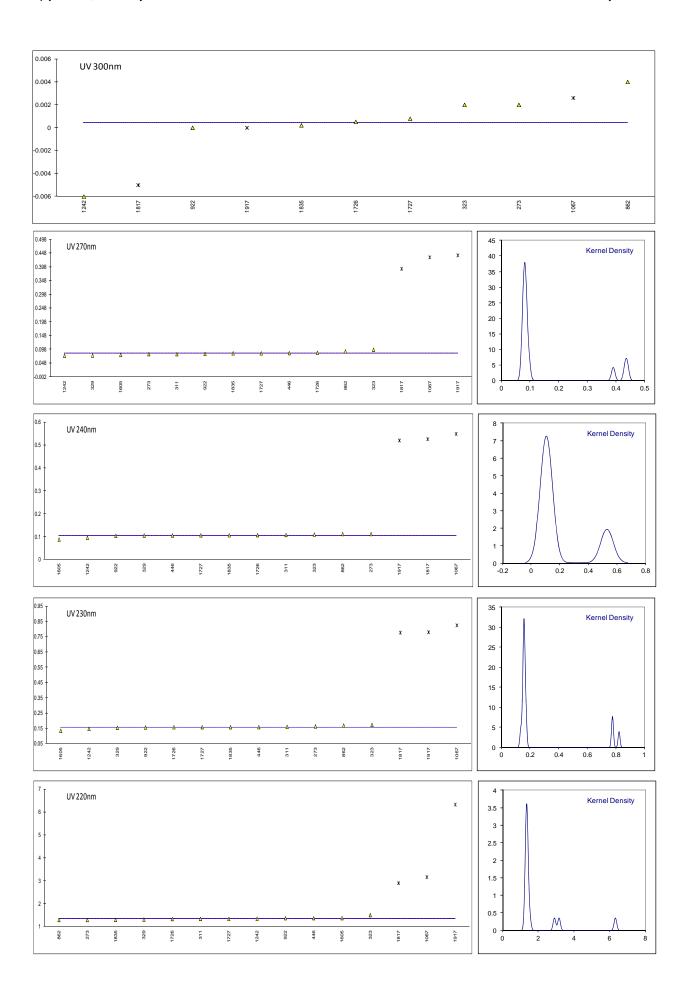




Determination of UV absorbance on sample #12152;

lab	method	300nm	mark	270nm	mark	240nm	mark	230nm	mark	220nm	mark
273	IMPCA 004	0.002		0.081		0.112		0.164		1.298	
311	INH-094	< 0.005		0.081		0.108		0.161		1.347	
323	INH-CM	0.002		0.097		0.110		0.174		1.511	
329	INH-CM	< 0.001		0.076		0.106		0.155		1.320	
357											
395											
446	INH-13	< 0.001		0.0847		0.1062		0.1590		1.3787	
522											
541											
551											
556											
559											
862	IMPCA 004	0.004		0.091		0.112		0.170		1.298	
922	in house	0.0000		0.0820		0.1043		0.1569		1.3762	
1067		0.0026	ex	0.4327	ex	0.5486	ex	0.8246	ex	3.164	ex
1126											
1241											
1242		-0.006		0.075		0.096		0.148		1.358	
1247											
1574											
1605		< 0.005		0.078		0.088		0.136		1.379	
1726		0.00051841		0.086192		0.10725		0.15837		1.33950	
1727		0.0008		0.0841		0.1067		0.1586		1.3494	
1817	INH-2008	-0.005	ex	0.391	ex	0.526	ex	0.777	ex	2.903	ex
1835		0.000212		0.0841		0.1068		0.1587		1.302	
1917		0.000	ex	0.440	ex	0.520	ex	0.780	ex	6.330	ex
1927											
	normality	not OK		OK		not OK		OK		not OK	
	n	8		12		12		12		12	
	outliers	0		0		0		0		0	
	mean (n)	0.0004		0.0833		0.1053		0.1583		1.3547	
	st.dev. (n)	0.00292		0.00619		0.00684		0.00972		0.05784	
	R(calc.)	0.0082		0.0173		0.0192		0.0272		0.1619	
	R(lit)	Unknown		Unknown		Unknown		Unknown		Unknown	
	R(iis11C13a)	0.00492		0.0079		0.0177		0.0354		0.0592	
	11(1101101104)									000=	

Results for Lab. 1067, 1817 and 1917 were excluded as a 50 mm cuvette was used.



Analytical details regarding Strength determination and UV absorbance.

Lab			Str	ength	UV abso	orbance	Other details
	Distillation	Equipment	used sample in ml	how much distillate was obtained in ml	cuvette(mm)	measured aginst:	
273	no	Density meter			10	H ₂ O	
311	no	Density meter			10	H ₂ O	
323	no	Density meter			10	H ₂ O	
329	no	Density meter			10	H ₂ O	
357	no	Density meter					
395	no						
446	no				10	H ₂ O	
522							
541							
551							
556							
559							
862	no	Density meter			10	H ₂ O	
922	no	Density meter			10	H ₂ O	
1067	no				50	H ₂ O	
1126	no						
1241	no						
1242	no				5	H ₂ O	
1247	no	Density meter					
1574							
1605	no				10	H ₂ O	
1726	no	Density meter			10	H ₂ O	
1727	no				10	H ₂ O	
1817	no	AOAC table			50	H ₂ O	
1835	no				10	H ₂ O	
1917					50		
1927							

Number of participants per country

- 1 lab in ARGENTINA
- 3 labs in BELGIUM
- 3 labs in BRAZIL
- 1 lab in FINLAND
- 1 lab in FRANCE
- 1 lab in HONG KONG
- 1 lab in ITALY
- 1 lab in MEXICO
- 1 lab in P.R. of CHINA
- 1 lab in PAKISTAN
- 1 lab in SOUTH AFRICA
- 3 labs in SPAIN
- 2 labs in THAILAND
- 6 labs in THE NETHERLANDS
- 1 lab in UNITED KINGDOM

Abbreviations:

C = final result after checking of first reported suspect result

 $\begin{array}{ll} D(0.01) &= \text{outlier in Dixon's outlier test} \\ D(0.05) &= \text{straggler in Dixon's outlier test} \\ G(0.01) &= \text{outlier in Grubbs' outlier test} \\ G(0.05) &= \text{straggler in Grubbs' outlier test} \\ DG(0.01) &= \text{outlier in Double Grubbs' outlier test} \end{array}$

DG(0.05) = straggler in Double Grubbs' outlier test

E = error in calculations

ex = excluded from calculations

n.a. = not applicable

OILM = International Organization of Legal Metrology

U = unit error

SDS = safety data sheet

Literature:

- i.i.s. Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, February 2010
- 2 W. Horwitz and R. Albert, J. AOAC Int., Vol. 79, 3, p. 589, (1996)
- 3 ASTM E178-02
- 4 ASTM E1301-03
- 5 ISO13528-05
- 5 ISO 5725-86
- 6 ISO 5725, parts 1-6, 1994
- 7 M. Thompson and R. Wood, J. AOAC Int, <u>76</u>, 926, (1993)
- 8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 9 IP 367/84
- 10 DIN 38402 T41/42
- 11 P.L. Davies, Fr. Z. Anal. Chem, <u>331</u>, 513, (1988)
- 12 J.N. Miller, Analyst, <u>118</u>, 455, (1993)
- 13 Analytical Methods Committee Technical brief, No 4.February 2001
- The Royal Society of Chemistry 2002, Analyst, 2002, 127, page 1359-1364, P.J. Lowthian and M. Thompson. (see http://www.rsc.org/suppdata/an/b2/b205600n/)