

Results of Proficiency Test  
REN/Food Ethanol  
November 2013

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

Authors: ing. C. Nijssen-Wester  
Correctors: dr. R.G. Visser & ing. R. Starink  
Report: iis13C13

February 2014

**CONTENTS**

1	INTRODUCTION .....	3
2	SET UP .....	3
2.1	ACCREDITATION .....	3
2.2	PROTOCOL.....	3
2.3	CONFIDENTIALITY STATEMENT .....	3
2.4	SAMPLES .....	4
2.5	STABILITY .....	4
2.6	ANALYSES .....	5
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 2013 WITH PREVIOUS PTS.....	10

## Appendices:

1.	Data and statistical results .....	11
2.	Analytical Details .....	20
3.	Number of participants per country .....	22
4.	Abbreviations and literature .....	23

## 1. INTRODUCTION

Since 2007, a proficiency test for REN/Food grade Ethanol is organised every year by the Institute for Interlaboratory Studies. During the planning of the annual proficiency testing program 2013/2014, it was decided to continue the round robin for the analysis of REN/Food grade Ethanol.

In this interlaboratory study, 29 laboratories in 15 different countries have participated. See appendix 3 for the number of participants per country. In this report, the results of the 2013 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 #13222). 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/IEC 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% 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' (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 #13222 was obtained from a local trader. The approximately 50 litre bulk sample was, after homogenisation in a precleaned drum, divided over 50 amber glass bottles of 0.5 L and labelled #13222. The homogeneity of these subsamples was checked by determination of Density in accordance with ASTM D4052:02e1 and Water in accordance with ASTM D1364:12 on 8 stratified randomly selected samples.

Sample	Density @ 20°C in kg/L	Water in %M/M
Sample #13222-1	0.80597	5.636
Sample #13222-2	0.80596	5.637
Sample #13222-3	0.80597	5.658
Sample #13222-4	0.80597	5.610
Sample #13222-5	0.80597	5.636
Sample #13222-6	0.80597	5.641
Sample #13222-7	0.80597	5.653
Sample #13222-8	0.80597	5.661

table 1: Homogeneity test results of subsamples #13222

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)	0.00001	0.046
reference method	ASTM D4052:02e1	ASTM D1364:12
0.3 * R (ref. method)	0.00015	0.043

table 2: Repeatability of subsamples #13222

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 #13222 was sent on October 30, 2013.

## 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 #13222: 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](http://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 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 (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:

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

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 in order to evaluate the fit-for-useness of the reported test result.

#### 4. EVALUATION

In this proficiency test, some problems were encountered with the despatch of the samples.

Four participants reported results after the final reporting date and five participants did not report any results at all. Not all laboratories were able to perform all analysis requested. In total 24 laboratories reported 160 numerical results. Observed were 9 outlying results, which is 5.6%. 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. Twenty-one laboratories answered the questions fully or partially (See Appendix 2). Based on these analytical details none of the participants performed a distillation before the strength determination and five participants reported the use of a density meter for the strength determination. Based on the analytical details of the UV Absorbance test, all reporting laboratories used water to measure against and all, but one, used a 10 mm cuvette.

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

Density: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D4052:02e1.

Nonvolatile matter: Almost all participants reported a "less than" result. Therefore no significant conclusions could be drawn.

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

- Permanganate Time Test: This determination may be problematic. One statistical outlier was observed. However, the calculated reproducibility, after rejection of the statistical outlier, is 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 large in comparison to the calculated reproducibility of the previous proficiency test iis12C12 of November 2012 (0.012 vs 0.008).
- Strength (%V/V): This determination may be problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the reproducibility derived from the OIML table and ASTM D4052:02e1. The spread found is small in comparison to the calculated reproducibility of the previous proficiency test iis12C12 of November 2012 (0.041 vs. 0.062).
- Strength(%M/M): This determination may be problematic. One statistical outlier was observed. Regretfully, no standard test method with precision data exists. The calculated reproducibility, after rejection of the statistical outlier, is small in comparison to the calculated reproducibility in the previous proficiency test iis12C12 of November 2012 (0.075 vs 0.107).
- UV absorbance: Regretfully, no standard test method with precision data exists. Therefore no significant conclusions were drawn. For every UV absorbance test, except 230 nm, one statistical outlier was observed. For the test at 300 nm, 270 nm and 240 nm, the calculated reproducibilities are small or the same in comparison with the calculated reproducibilities from the previous proficiency test iis12C12 of November 2012. For the test at 230 nm and 220 nm, the calculated reproducibilities are much higher than in the PT of 2012. 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 1242. This participant used 5 mm 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 <sub>R</sub>	R (lit)
Density @ 20°C	kg/L	23	0.8060	0.0003	0.0005
Nonvolatile matter	mg/100mL	3	<1	n.a.	n.a.
Water	%M/M	15	5.62	0.24	0.14
Permanganate Time Test	min.	11	24.3	5.6	6.1
Purity on dry basis	%M/M	10	99.986	0.012	(0.008)
Strength	%V/V	17	99.365	0.041	0.060
Strength	%M/M	10	94.374	0.075	(0.107)
UV-absorbance 300 nm		9	0.001	0.002	(0.008)
UV-absorbance 270 nm		12	0.140	0.018	(0.017)
UV-absorbance 240 nm		12	0.121	0.011	(0.019)
UV-absorbance 230 nm		13	0.184	0.035	(0.027)
UV-absorbance 220 nm		12	2.092	0.456	(0.162)

Table 3: Reproducibilities of sample #13222

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

#### 4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2013 WITH PREVIOUS PT'S

	<i>November 2013</i>	<i>November 2012</i>	<i>November 2011</i>	<i>November 2010</i>
Number of reporting labs	24	24	23	28
Number of results reported	160	169	151	189
Number of statistical outliers	9	5	9	13
Percentage outliers	5.6%	3.0%	6.0%	6.9%

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 in the following table:

Parameter	<i>November 2013</i>	<i>November 2012</i>	<i>November 2011</i>	<i>November 2010</i>
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 #13222 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:

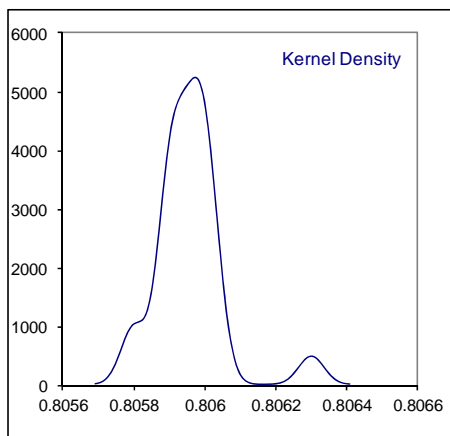
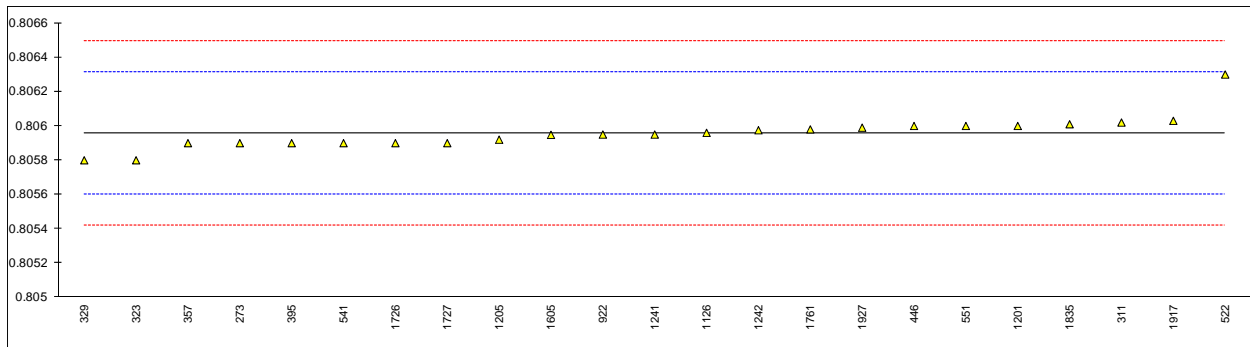
- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- : group performed worse than the standard
- : group performed much worse than the standard
- n.e.: not evaluated

**APPENDIX 1**

Determination of Density @ 20°C on sample #13222; results in kg/L

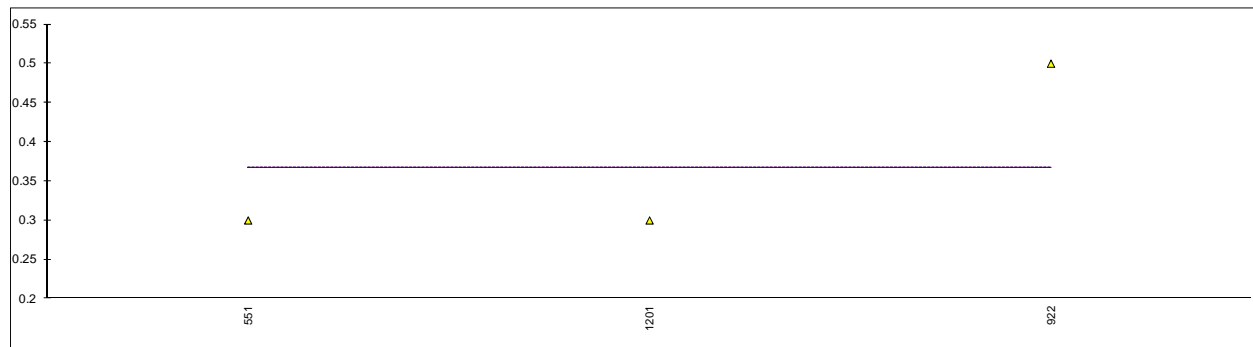
lab	method	value	mark	z(targ)	remarks
273	D4052	0.8059		-0.32	
311	D4052	0.80602		0.35	
323	D4052	0.8058		-0.88	
329	D4052	0.8058		-0.88	
357	D4052	0.8059		-0.32	
395	D4052	0.8059		-0.32	
446	D4052	0.8060		0.24	
522	D4052	0.8063		1.92	
529		----		----	
541	D4052	0.8059		-0.32	
551	D4052	0.8060		0.24	
554		----		----	
556		----		----	
559		----		----	
922	D4052	0.80595	C	-0.04	First reported: 805.95
1067		----		----	
1126	D4052	0.80596		0.01	
1201	D4052	0.8060		0.24	
1205	in house	0.80592		-0.21	
1241	INH-50	0.80595		-0.04	
1242	D4052	0.805975		0.10	
1574		----		----	
1605	D4052	0.805948		-0.06	
1726	D4052	0.80590		-0.32	
1727	D4052	0.8059		-0.32	
1761	D4052	0.80598		0.12	
1835	D4052	0.80601		0.29	
1917	D4052	0.80603		0.40	
1927	in house	0.80599		0.18	

normality not OK  
n 23  
outliers 0  
mean (n) 0.805958  
st.dev. (n) 0.0000971  
R(calc.) 0.000272  
R(D4052:02e1) 0.000500



Determination of Nonvolatile matter on sample #13222; results in mg/100mL

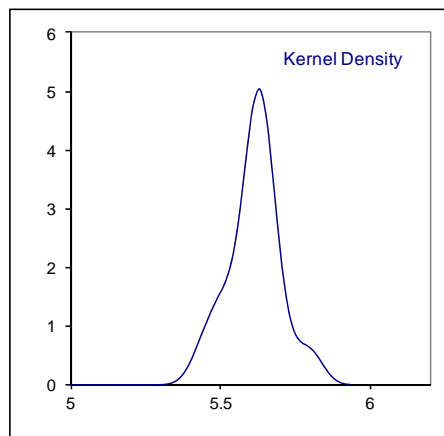
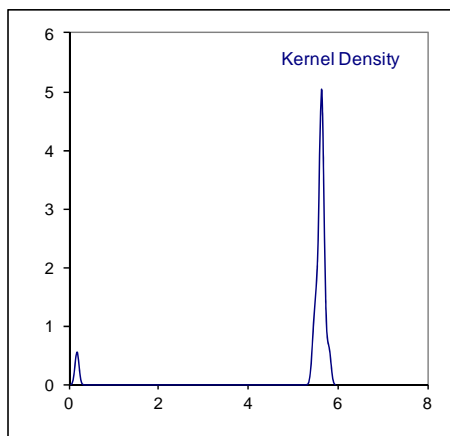
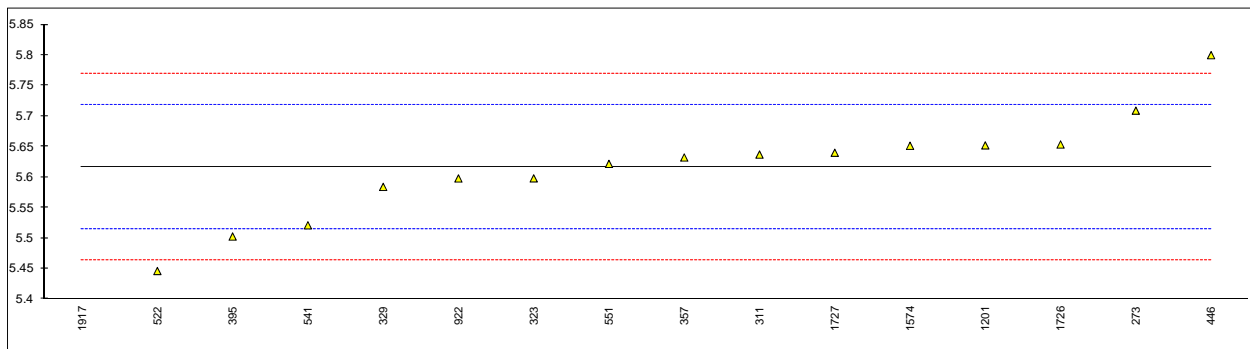
lab	method	value	mark	z(targ)	remarks
273		----		----	
311	D1353	<1		----	
323	D1353	<1		----	
329	D1353	<1		----	
357	D1353	<1		----	
395		----		----	
446	D1353	<1		----	
522		----		----	
529		----		----	
541	D1353	<1		----	
551	D1353	0.3		----	
554		----		----	
556		----		----	
559		----		----	
922	D1353	0.50		----	
1067		----		----	
1126		----		----	
1201	D1353	0.3		----	
1205		----		----	
1241		----		----	
1242		----		----	
1574		----		----	
1605		----		----	
1726	D1353	<10		----	
1727	D1353	<0.5		----	
1761		----		----	
1835	EN15691	<10		----	
1917		----		----	
1927		----		----	
	normality	n.a.			
	n	3			
	outliers	n.a.			
	mean (n)	<1			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D1353:13)	n.a.			



Determination of Water (Titrimetric) on sample #13222; results in %M/M

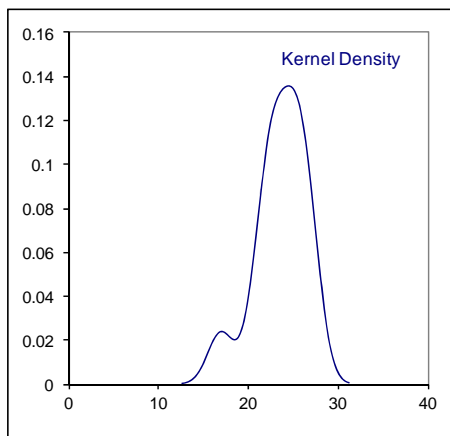
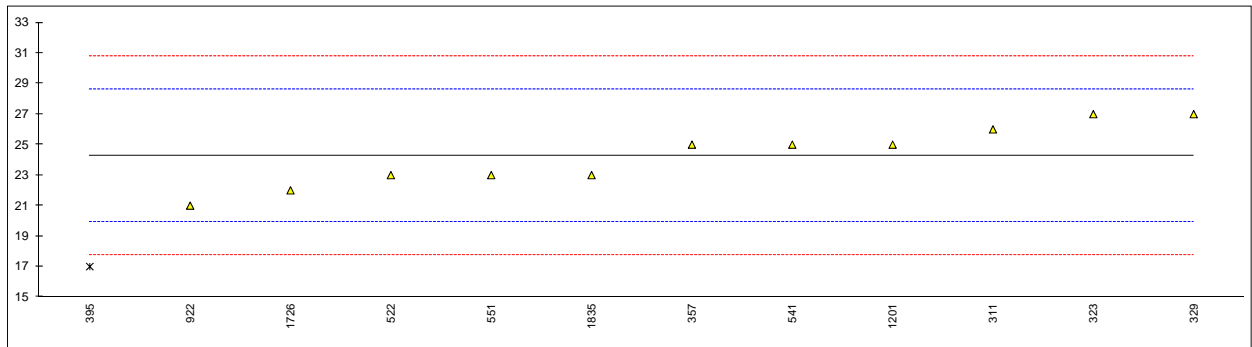
lab	method	value	mark	z(targ)	remarks
273	E203	5.709		1.82	
311	D1364	5.637		0.41	
323	D1364	5.598		-0.36	
329	E203	5.584		-0.64	
357	D1364	5.6322		0.31	
395	D1364	5.5026		-2.24	
446	D1364	5.80		3.62	
522	E203	5.446		-3.36	
529		----		----	
541	E1064	5.521		-1.88	
551	D1364	5.6217		0.10	
554		----		----	
556		----		----	
559		----		----	
922	E203	5.5978		-0.37	
1067		----		----	
1126		----		----	
1201	D1364	5.652		0.70	
1205		----		----	
1241		----		----	
1242		----		----	
1574	INH-76	5.6514		0.69	
1605		----		----	
1726	D1364	5.6535		0.73	
1727	D1364	5.64		0.46	
1761		----		----	
1835		----		----	
1917	D1364	0.18	C,G(0.01)	-107.05	First reported: 5.33
1927		----		----	

normality OK  
n 15  
outliers 1  
mean (n) 5.6164  
st.dev. (n) 0.08461  
R(calc.) 0.2369  
R(D1364:12) 0.1422



Determination of Permanganate Time Test @ 15 °C on sample #13222; results in minutes

lab	method	value	mark	z(targ)	remarks
273		----		----	
311	D1363	26		0.79	
323	D1363	27		1.25	
329	D1363	27		1.25	
357	D1363	25		0.33	
395	D1363	17	G(0.05)	-3.33	
446		----		----	
522	D1363	23		-0.58	
529		----		----	
541	D1363	25		0.33	
551	D1363	23		-0.58	
554		----		----	
556		----		----	
559		----		----	
922	D1363	21		-1.50	
1067		----		----	
1126		----		----	
1201	D1363	25		0.33	
1205		----		----	
1241		----		----	
1242		----		----	
1574		----		----	
1605		----		----	
1726	D1363	22		-1.04	
1727		----		----	
1761		----		----	
1835	D1363	23		-0.58	
1917	D1363	<20		< -1.95	
1927		----		----	
normality		OK			
n		11			
outliers		1			
mean (n)		24.27			
st.dev. (n)		2.005			
R(calc.)		5.61			
R(D1363:11)		6.12			

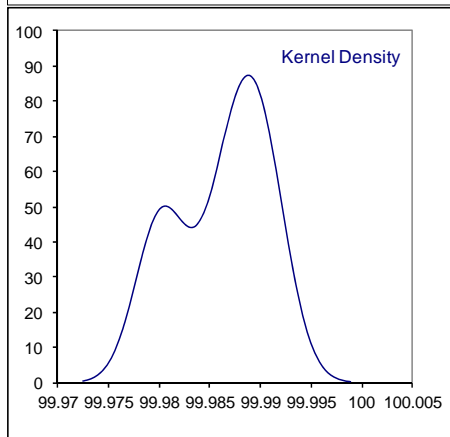
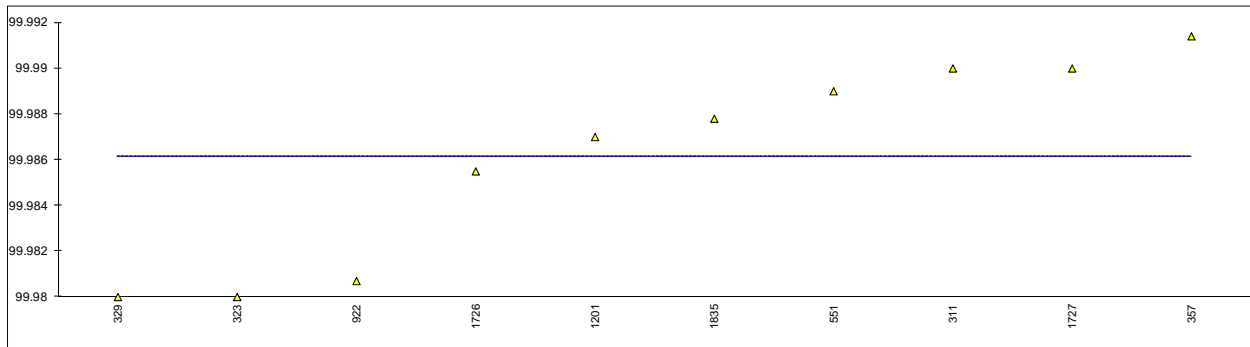


Determination of Purity on dry basis on sample #13222; results in %M/M

lab	method	value	mark	z(targ)	remarks
273		----		----	
311	INH-529	99.99		----	
323		99.98		----	
329		99.98		----	
357	EN15721	99.9914		----	
395		----		----	
446		----		----	
522		----		----	
529		----		----	
541		----		----	
551	INH-1313	99.989		----	
554		----		----	
556		----		----	
559		----		----	
922	INH-0001	99.9807		----	
1067		----		----	
1126		----		----	
1201		99.987		----	
1205		----		----	
1241		----		----	
1242		----		----	
1574		----		----	
1605		----		----	
1726		99.9855		----	
1727	EN15721	99.99		----	
1761		----		----	
1835	in house	99.9878		----	
1917		----		----	
1927		----		----	

normality OK  
n 10  
outliers 0  
mean (n) 99.9861  
st.dev. (n) 0.00440  
R(calc.) 0.0123  
R(lit.) Unknown

Compare R(iis12C12) = 0.0082

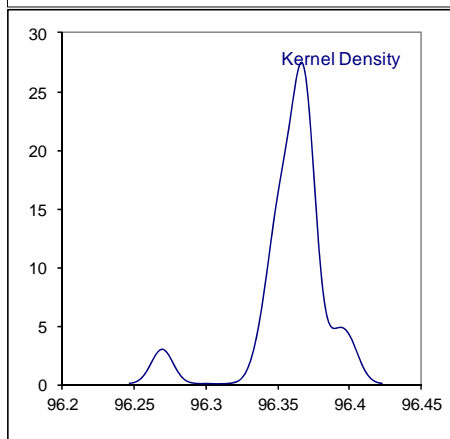
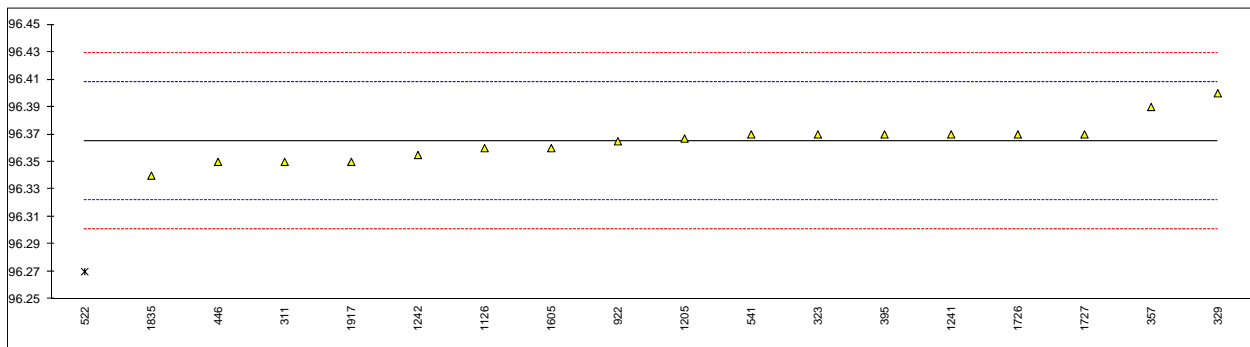


Determination of Strength on sample #13222; results in %V/V

lab	method	value	mark	z(targ)	remarks
273		-----		-----	
311	OIML	96.35		-0.71	
323		96.37		0.23	
329		96.40		1.63	
357		96.39		1.16	
395	OIML	96.37		0.23	
446	OIML	96.35		-0.71	
522	D4052	96.27	G(0.01)	-4.44	
529		-----		-----	
541	OIML	96.37		0.23	
551		-----		-----	
554		-----		-----	
556		-----		-----	
559		-----		-----	
922	OIML	96.365		-0.01	
1067		-----		-----	
1126	in house	96.36		-0.24	
1201		-----		-----	
1205	in house	96.367		0.09	
1241	Alc.table	96.37		0.23	
1242		96.355		-0.47	
1574		-----		-----	
1605		96.36		-0.24	
1726	OIML	96.37		0.23	
1727	D4052	96.37		0.23	
1761		-----		-----	
1835	OIML	96.34		-1.17	
1917		96.35		-0.71	
1927		-----		-----	

normality not OK  
n 17  
outliers 1  
mean (n) 96.365  
st.dev. (n) 0.0147  
R(calc.) 0.041  
R(OIML table) 0.060

Compare R(iis12C12) = 0.062



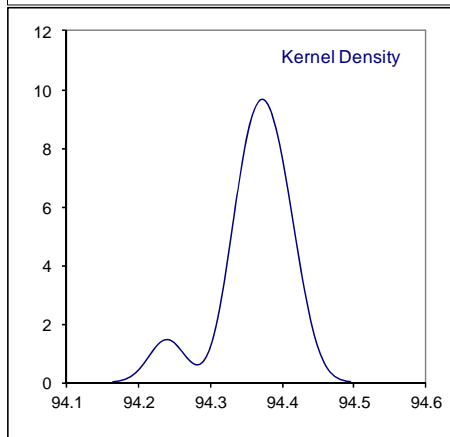
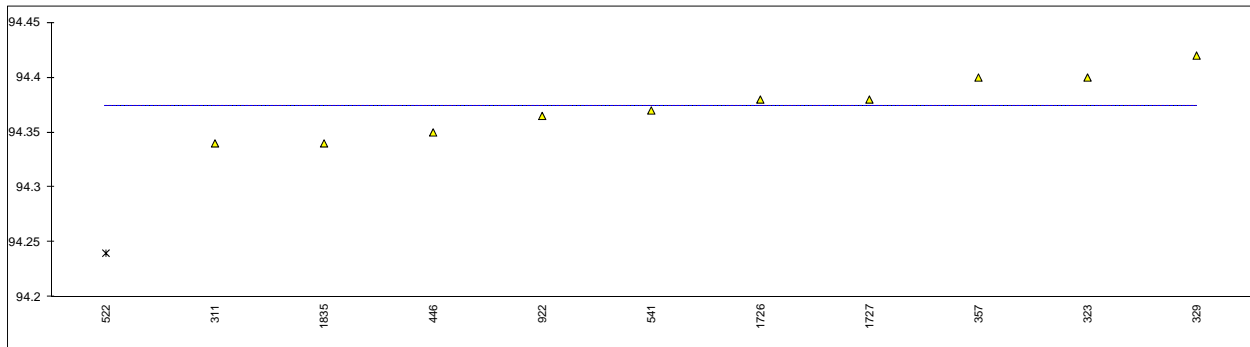


Determination of Strength on sample #13222; results in %M/M

lab	method	value	mark	z(targ)	remarks
273		----		----	
311	OIML	94.34		----	
323		94.40		----	
329		94.42		----	
357		94.40		----	
395		----		----	
446	OIML	94.35	C	----	First reported: 96.35
522	D4052	94.24	G(0.05)	----	
529		----		----	
541	OIML	94.37		----	
551		----		----	
554		----		----	
556		----		----	
559		----		----	
922	OIML	94.365		----	
1067		----		----	
1126		----		----	
1201		----		----	
1205		----		----	
1241		----		----	
1242		----		----	
1574		----		----	
1605		----		----	
1726	OIML	94.38		----	
1727	D4052	94.38		----	
1761		----		----	
1835	OIML	94.34		----	
1917		----		----	
1927		----		----	

normality OK  
n 10  
outliers 1  
mean (n) 94.374  
st.dev. (n) 0.0269  
R(calc.) 0.075  
R(lit.) Unknown

Compare R(iis12C12) = 0.107



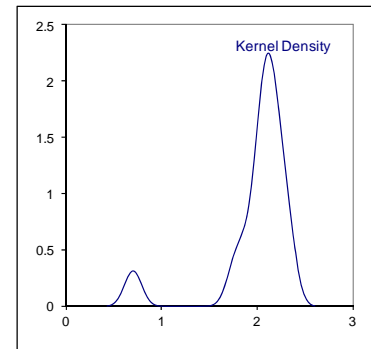
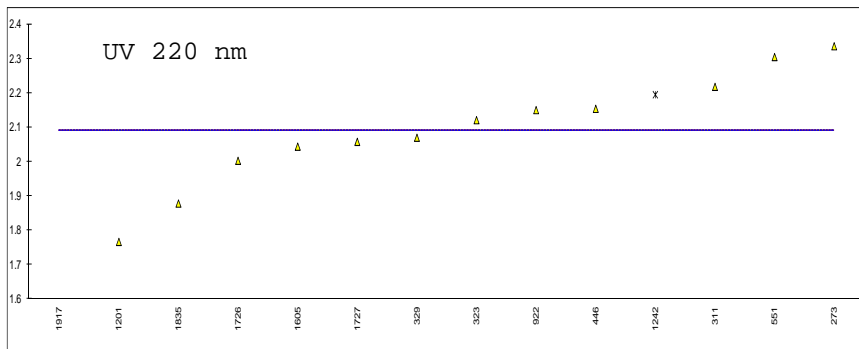
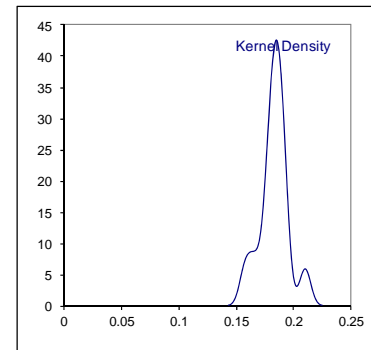
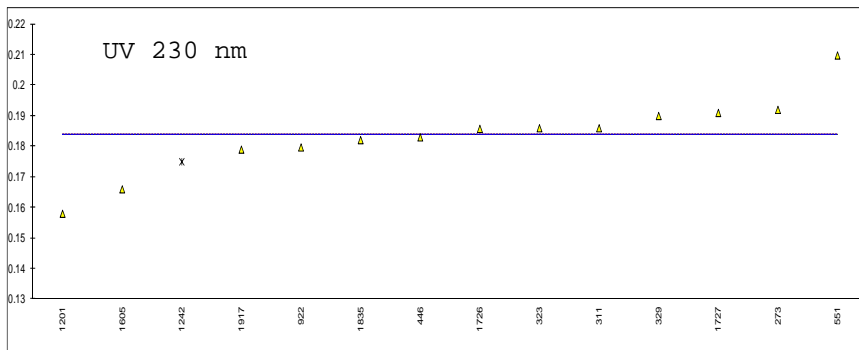
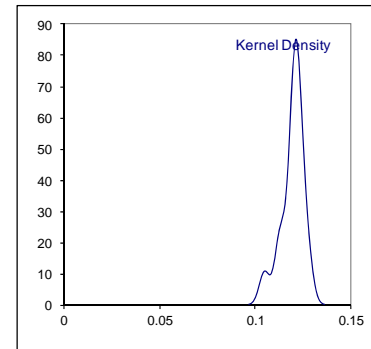
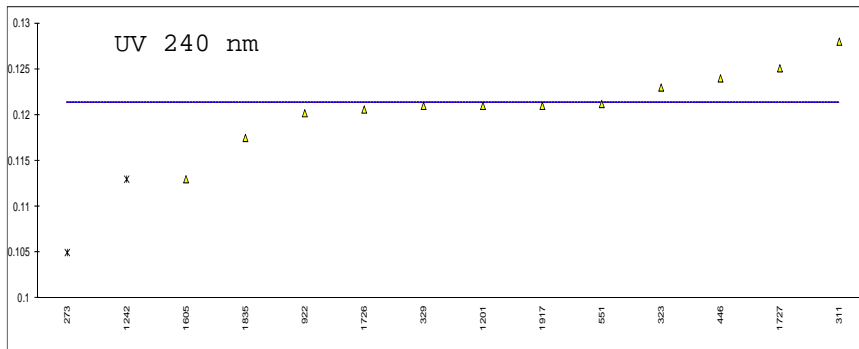
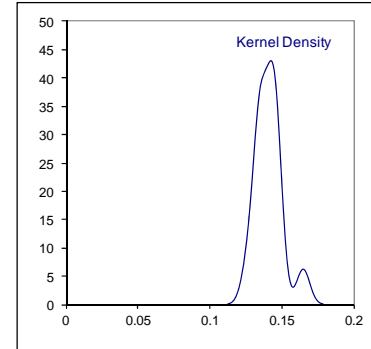
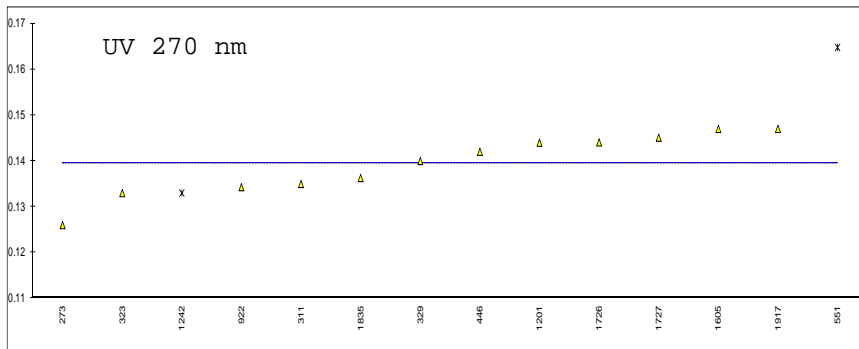
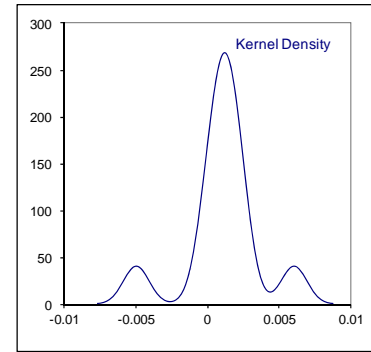
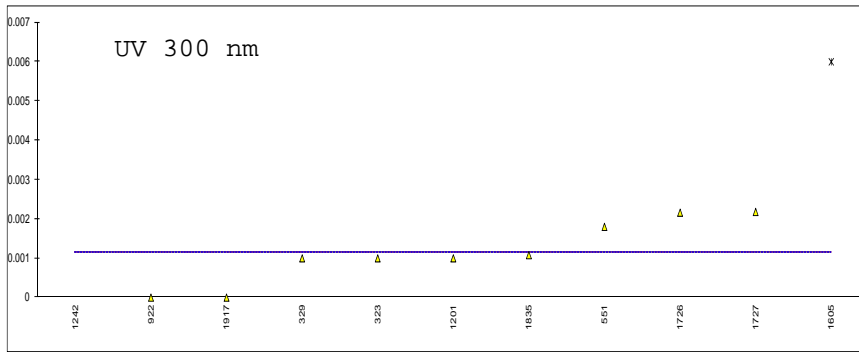
## Determination of UV absorbance on sample #13222;

lab	method	300nm	mark	270nm	mark	240nm	mark	230nm	mark	220nm	mark
273	IMPCA004	<0.001		0.126		0.105	C,G(0.05)	0.192		2.336	
311	INH-94	<0.005		0.135		0.128		0.186		2.218	
323		0.001		0.133		0.123		0.186		2.121	
329	INH-CM	0.001		0.140		0.121	C	0.190		2.070	
357		----		----		----		----		----	
395		----		----		----		----		----	
446	INH-13	<0.01		0.142		0.124		0.183		2.154	
522		----		----		----		----		----	
529		----		----		----		----		----	
541		----		----		----		----		----	
551	INH-1519	0.0018		0.1648	G(0.05)	0.1212		0.2098		2.3046	
554		----		----		----		----		----	
556		----		----		----		----		----	
559		----		----		----		----		----	
922	in house	0.000		0.1343		0.1202		0.1797		2.1503	
1067		----		----		----		----		----	
1126		----		----		----		----		----	
1201		0.001		0.144		0.121		0.158		1.767	
1205		----		----		----		----		----	
1241		----		----		----		----		----	
1242		-0.005	ex	0.133	ex	0.113	ex	0.175	ex	2.195	ex
1574		----		----		----		----		----	
1605		0.006	G(0.01)	0.147		0.113		0.166		2.044	
1726		0.00216183		0.14406		0.12060		0.18578		2.00293	
1727		0.00218		0.1451		0.1251		0.1910		2.058	
1761		----		----		----		----		----	
1835		0.00108		0.1363		0.1175		0.1821		1.8785	
1917		0		0.147		0.121		0.179		0.701	G(0.01)
1927		----		----		----		----		----	
	normality	OK		OK		OK		OK		OK	
	n	9		12		12		13		12	
	outliers	1 (+ 1 ex)		1 (+ 1 ex)		1 (+ 1 ex)		0 (+ 1 ex)		1 (+ 1 ex)	
	mean (n)	0.0011		0.1395		0.1213		0.1837		2.0920	
	st.dev. (n)	0.00081		0.00656		0.00375		0.01249		0.16287	
	R(calc.)	0.0023		0.0183		0.0105		0.0350		0.4560	
	R(lit)	Unknown		Unknown		Unknown		Unknown		Unknown	
	R(iis12C12)	0.0082		0.0173		0.0192		0.0272		0.1619	

**Results for Lab 1242 were excluded as a 5 mm cuvette was used.**

Corrected results at 240nm: Lab 273 first reported: 0.165 and Lab 329 first reported: 0.141

Gauss plots and Kernel Density graphs can be found on the next page.



**APPENDIX 2a****Analytical details regarding Strength determination.**

Lab	Distillation	Equipment	Strength		Other details
			used sample in ml	how much distillate was obtained in ml	
273					
311	No				
323	No				
329	No				
357	No				
395	No				
446	No				
522		Digital densimeter			
529					
541	No				
551					
554					
556					
559					
922	No	Anton Paar DMA4500			
1067					
1126	No				
1201	No				
1205	No	Oscillating tube densimeter			
1241	No				
1242	No				
1574					
1605	No				
1726	No	Anton Paar densimeter			
1727	No				
1761					
1835					
1917	No	Densimeter			
1927					

**APPENDIX 2b****Analytical details regarding UV absorbance.**

Lab	UV absorbance		Other details
	cuvette (mm)	measured against:	
273	10	H <sub>2</sub> O	
311	10	H <sub>2</sub> O	
323	10	H <sub>2</sub> O	
329	10	H <sub>2</sub> O demin.	
357			
395			
446	10	H <sub>2</sub> O demin.	
522			
529			
541			
551	10	H <sub>2</sub> O	
554			
556			
559			
922	10	H <sub>2</sub> O	
1067			
1126			
1201	10	H <sub>2</sub> O	
1205			
1241			
1242	5	H <sub>2</sub> O	
1574			
1605	10	H <sub>2</sub> O	
1726	10	H <sub>2</sub> O	
1727	10	H <sub>2</sub> O	
1761			
1835	10	H <sub>2</sub> O	
1917	10	H <sub>2</sub> O	
1927			

## **APPENDIX 3**

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

1 lab in ARGENTINA  
4 labs in BELGIUM  
4 labs in BRAZIL  
1 lab in FINLAND  
1 lab in FRANCE  
1 lab in HONG KONG  
1 lab in ITALY  
1 lab in KOREA  
2 labs in MEXICO  
6 labs in NETHERLANDS  
1 lab in PAKISTAN  
1 lab in SOUTH AFRICA  
3 labs in SPAIN  
1 lab in THAILAND  
1 lab in UNITED KINGDOM

## APPENDIX 4

### Abbreviations:

C	= final result after checking of first reported suspect 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
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:

- 1 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, 76, 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, 331, 513, (1988)
- 12 J.N. Miller, Analyst, 118, 455, (1993)
- 13 Analytical Methods Committee Technical brief, No 4. February 2001
- 14 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/>)