

Results of Proficiency Test  
REN/Food Ethanol  
December 2009

Organised by: Institute for Interlaboratory Studies (i.i.s.)  
Spijkenisse, the Netherlands

Authors: Ing. R.J. Starink  
Correctors: Dr. R.G. Visser & Ing. M. Audier  
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## **1. INTRODUCTION**

Since 1995, a proficiency test for Ethanol is organised every year by the Institute for Interlaboratory Studies. During the planning of the annual proficiency testing program 2009/2010, it was decided to continue the round robin for the analysis of REN/Food grade Ethanol and to extend the scope on request of several laboratories with two types of alcoholic beverages.

In this international interlaboratory study 33 laboratories in 16 different countries have participated. See appendix 2 for a list of participants in alphabetical country order. In this report, the results of the proficiency test are presented and discussed.

## **2 SET-UP**

The Institute for Interlaboratory Studies (i.i.s.) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. It was decided to send three different samples (1\* 0.5 L of 95% REN/Food grade Ethanol, labelled #0994, 1\* 0.5 L of Peach liqueur, labelled #0995 and 1\* 0.5 L Crème liqueur, labelled #0996). 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 accordance with the ISO-guide G13:2007, (R007) since January 2000 by the Dutch Accreditation Council RvA (Raad voor Accreditatie). This ensures 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

### **2.2 PROTOCOL**

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'i.i.s. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (i.i.s.-protocol, version 3.2) of January 2010.

### **2.3 CONFIDENTIALITY STATEMENT**

All data present 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 of 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 #0994 was obtained from a local trader. The approximately 25 litre bulk sample was, after homogenisation in a precleaned can, divided over 48 amber glass bottles of 0.5 L and labelled #0994. The homogeneity of these subsamples was checked by determination of Density in accordance with ASTM D4052:09 and Water in accordance with ASTM D1364:07 on 8 stratified random selected samples.

Sample	Density @ 20°C in kg/L	Water in %M/M
#0994-1	0.80898	6.739
#0994-2	0.80899	6.764
#0994-3	0.80901	6.766
#0994-4	0.80901	6.783
#0994-5	0.80897	6.779
#0994-6	0.80900	6.773
#0994-7	0.80900	6.769
#0994-8	0.80900	6.754

table 1: Homogeneity tests of subsamples #0994

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)	0.00004	0.040
reference method	D4052:09	EN15489:07
0.3 * R (ref. method)	0.00015	0.047

table 2: Repeatability of subsamples #0994

The necessary bulk material (Peach Liqueur) for sample #0995 was obtained from a local producer. The approximately 25 litre bulk sample was, after homogenisation in a precleaned can, divided over 46 amber glass bottles of 0.5 L and labelled #0995. The homogeneity of these subsamples was checked by determination of Density in accordance with ASTM D4052:09 on 8 stratified random selected samples.

Sample	Density @ 20°C in kg/L
#0995-1	1.08162
#0995-2	1.08161
#0995-3	1.08162
#0995-4	1.08160
#0995-5	1.08160
#0995-6	1.08161
#0995-7	1.08160
#0995-8	1.08160

table 3: Homogeneity tests of subsamples #0995

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:

	<i>Density @ 20°C in kg/L</i>
r (Observed)	0.00004
reference method	D4052:09
0.3 * R (ref. method)	0.00015

table 4: Repeatability of subsamples #0995

The necessary bulk material (Cream Liqueur) for sample #0996 was obtained from a local producer. The approximately 25 litre bulk sample was, after homogenisation in a precleaned can, divided over 46 amber glass bottles of 0.5 L and labelled #0996. The homogeneity of these subsamples was checked by determination of Density in accordance with ASTM D4052:09 on 8 stratified random selected samples.

Sample	<i>Density @ 20°C in kg/L</i>
#0996-1	1.11683
#0996-2	1.11681
#0996-3	1.11683
#0996-4	1.11682
#0996-5	1.11680
#0996-6	1.11682
#0996-7	1.11678
#0996-8	1.11685

table 5: Homogeneity tests of subsamples #0996

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:

	<i>Density @ 20°C in kg/L</i>
r (Observed)	0.00006
reference method	D4052:09
0.3 * R (ref. method)	0.00015

table 6: Repeatability of subsamples #0996

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 #0994, 1\*0.5 L bottle of sample #0995 and 1\*0.5 L bottle of sample #0996 were sent on November 04, 2009.

## 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 #0994: Density @ 20°C, Permanganate Time Test, Water (titrimetric), Purity on dry basis, Strength (in %V/V and %M/M) and UV transmittance at 300, 270, 240, 230 and 220nm. On sample #0995 and #0996 was requested to determined: Density @ 20°C, pH and Strength (in %V/V) To get comparable results a detailed report form, on which the units were printed, was sent together with each set of samples. In addition, a letter of instructions and a SDS were added to the 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 'i.i.s. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (i.i.s.-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. 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 3, nr.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

## 4. EVALUATION

In this proficiency test, some problems were encountered with despatch of the samples. Several laboratories in Brazil, India, P.R. of China and Pakistan did receive the samples very late. No participants reported results after the final reporting date. One participant did not report any results at all.

Not all laboratories were able to perform all analysis requested. Nevertheless, the 31 reporting laboratories did send in 299 (numerical) results. Observed were 34 outlying results, which is 11.4 %. 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 are 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.

Not normal distributions were found for the following determinations: Density (#0994), Purity on dry basis, Strength #0994 (%V/V and %M/M) and Strength #0996. In these cases the statistical evaluation should be used with due care. One can see that this is justified from the Kernel Density Graphs.

#### **Sample #0994**

Density: This determination is 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 D4052:09.

Water: This determination is problematic. Two statistical outliers were observed and the calculated reproducibility, after rejection of the statistical outliers, is not at all in agreement with the requirements of ASTM D1364:07.

Permanganate Time Test: All participants reported a very short time (< 5 min.). Regrettably, no precision data are given in ASTM D1363:06 for Ethanol. Therefore, no conclusions were drawn.

Purity on dry basis: Regrettably, no standard test method with precision data exists. Therefore no conclusions were drawn. The calculated reproducibility is small in comparison with the calculated reproducibility in the previous proficiency test (iis08C11b) of December 2008 (0.0108 vs 0.0126).

Strength (%V/V): This determination is problematic for several laboratories. Three statistical outliers were observed. Regretfully, no standard method with precision data exists. Therefore, the target reproducibility is calculated from the correlation between the density and strength results. The calculated reproducibility, after rejection of the statistical outliers, is almost in agreement with the reproducibility calculated from the correlation between density and strength. Compared with the calculated reproducibility found in the previous proficiency test (iis08C11b), the calculated reproducibility is quite large (0.103 vs 0.078).

Strength(%M/M): Regretfully, no standard test method with precision data exists. Therefore no conclusions were drawn. The calculated reproducibility is small in comparison with the calculated reproducibility in the previous proficiency test (iis08C11b) of December 2008 (0.062 vs 0.129).

UV absorbance: Regretfully, no standard test method with precision data exists. Therefore no conclusions were drawn. In total 13 statistical outliers were observed. The calculated reproducibilities are all small in comparison with the calculated reproducibilities in a previous proficiency test (ii07C14) of December 2007.

#### **Liqueur samples #0995 and #0996**

Density: This determination on the peach liqueur #0995 is problematic. The determination on the cream liqueur #0996 is problematic for only several laboratories. In total five statistical outliers were observed. The calculated reproducibility for sample #0995, after rejection of the statistical outliers, does not agree with the requirements of ASTM D4052:09. However, for sample #0996 the observed spread is in agreement with the requirements of ASTM D4052:09.

pH: This determination is not problematic. In total three statistical outliers were observed and both calculated reproducibilities, after rejection of the statistical outliers, are in full agreement with the requirements of EN15490:07.

Strength (%V/V): This determination is problematic for several laboratories. In total five statistical outliers were observed. Regretfully, no standard method with precision data exists. Therefore, the precision data is calculated out of raw data from previous proficiency test report "PTS alcohol, evaluation report 39, January 2009", Both calculated reproducibilities, after rejection of the statistical outliers, are not at all in agreement compared the calculated reproducibilities from "PTS alcohol report 39" .

## 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	29	0.80898	0.00021	0.00050
Water	%M/M	16	6.73	0.26	0.18
Permanganate Time Test	min.	10	2.2	2.7	(0.6)
Purity on dry basis	%M/M	13	99.973	0.011	(0.013)
Strength	%V/V	27	95.603	0.103	0.095
Strength	%M/M	15	93.278	0.062	(0.129)
UV-absorbance 300 nm		12	0.0037	0.0060	(0.0076)
UV-absorbance 270 nm		15	0.0159	0.0110	(0.0167)
UV-absorbance 240 nm		15	0.0705	0.0179	(0.0209)
UV-absorbance 230 nm		15	0.1595	0.0306	(0.0365)
UV-absorbance 220 nm		15	0.2981	0.0334	(0.0654)

Table 7: Reproducibilities of sample #0994  
results between brackets are compared with the spread of the previous proficiency test

Parameter	Unit	n	average	2.8 *sd <sub>R</sub>	R (lit)
Density @ 20°C	kg/L	20	1.08216	0.00101	0.00050
pH		13	2.80	0.24	0.27
Strength	%V/V	8	24.038	0.149	0.086

Table 8: Reproducibilities of sample #0995

Parameter	Unit	n	average	2.8 *sd <sub>R</sub>	R (lit)
Density @ 20°C	kg/L	18	1.11663	0.00040	0.00050
pH		12	7.19	0.17	0.69
Strength	%V/V	8	17.068	0.202	0.085

Table 9: Reproducibilities of sample #0996

#### 4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2009 WITH PREVIOUS PT'S

	<i>December 2009</i>	<i>December 2008</i>	<i>December 2007</i>	<i>December 2006</i>
Number of reporting labs	31	22	40	35
Number of results reported	299	153	595	567
Statistical outliers	34	8	30	50
Percentage outliers	11.4%	5.2%	5.0%	8.8%

table 10: 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	<i>December 2009</i>	<i>December 2008</i>	<i>December 2007</i>	<i>December 2006</i>
Density @ 20°C	++	++	++	++
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 11: comparison determinations of sample #0994 against the standard results between brackets are compared with the spread of the previous round robin

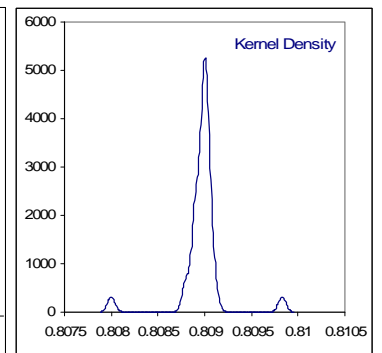
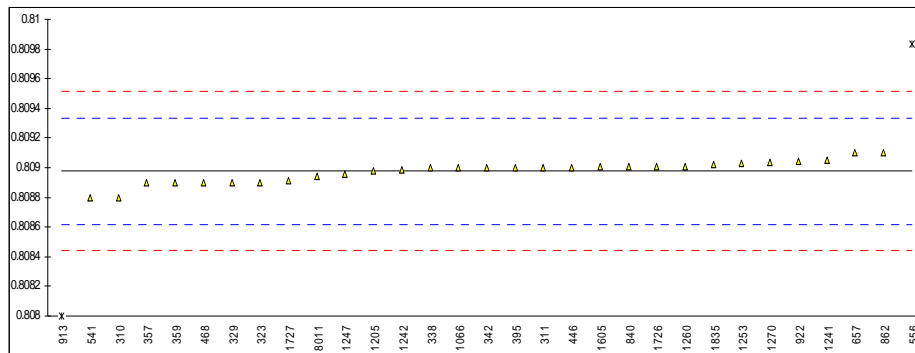
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

- ++: 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**

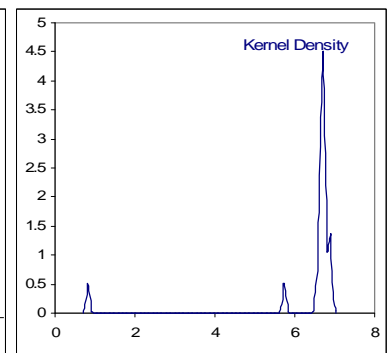
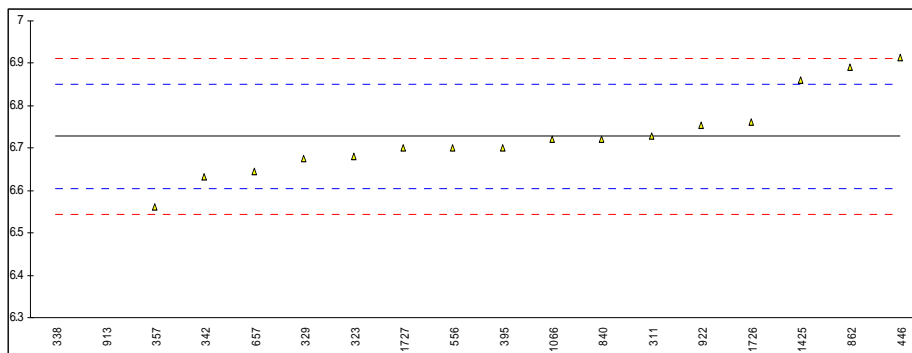
Determination of Density @ 20°C on sample #0994; results in kg/L

lab	method	value	mark	z(targ)	remarks
310	D4052	0.8088		-0.98	
311	D4052	0.8090		0.14	
323	D4052	0.8089		-0.42	
329	D4052	0.8089		-0.42	
338	ISO12185	0.8090		0.14	
342	D4052	0.8090		0.14	
357	D4052	0.8089		-0.42	
359	D4052	0.8089		-0.42	
395	D4052	0.8090		0.14	
446	D4052	0.8090		0.14	
468	D4052	0.8089	C	-0.42	
541	D4052	0.8088		-0.98	
551		-----		-----	
556	D4052	0.80983	G(0.01)	4.79	
559		-----		-----	
657	D4052	0.8091		0.70	
840	D4052	0.80901		0.19	
862	D4052	0.8091		0.70	
913	D1298	0.8080	G(0.01)	-5.46	
922	D4052	0.80904		0.36	
1066	D4052	0.8090		0.14	
1205	INH-APD	0.808979		0.02	
1241	DE50	0.80905		0.42	
1242	D4052	0.808988		0.07	
1247	D4052	0.80896		-0.09	
1253	In House	0.80903		0.31	
1260	DG45	0.80901		0.19	
1270	D4052	0.809036		0.34	
1425		-----		-----	
1605	D4052	0.80901		0.19	
1726	D4052	0.80901		0.19	
1727	D4052	0.80891		-0.37	
1835	D4052	0.80902		0.25	
8011	D4052	0.80894		-0.20	
normality		not OK			
n		29			
outliers		2			
mean (n)		0.80898			
st.dev. (n)		0.000074			
R(calc.)		0.00021			
R(D4052:09)		0.00050			



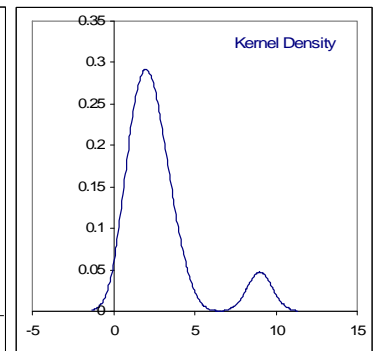
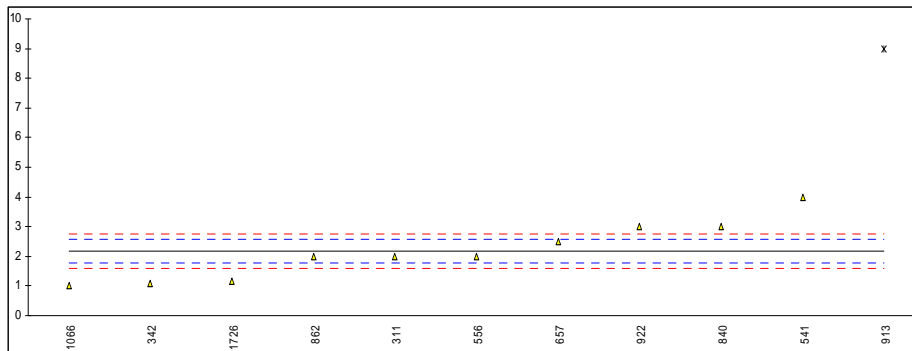
Determination of Water on sample #0994; results in %M/M

lab	method	value	mark	z(targ)	remarks
310		----		----	
311	D1364	6.728		0.00	
323	D1364	6.680		-0.74	
329	E203	6.675		-0.82	
338	D1364	0.823	CG(0.01)	-91.02	
342	E203	6.632		-1.48	
357	E203	6.561		-2.57	
359		----		----	
395	D1364	6.7017		-0.40	
446	E203	6.914		2.87	
468		----		----	
541		----		----	
551		----		----	
556	D1362	6.70		-0.43	
559		----		----	
657	D1364	6.645		-1.28	
840	D1364	6.7215		-0.10	
862	D1364	6.892		2.53	
913	D1364	5.74	G(0.01)	-15.23	
922	D1364	6.754		0.40	
1066	D1364	6.72		-0.12	
1205		----		----	
1241		----		----	
1242		----		----	
1247		----		----	
1253		----		----	
1260		----		----	
1270		----		----	
1425	In House	6.86		2.04	
1605		----		----	
1726	D1364	6.7623		0.53	
1727	D1364	6.7		-0.43	
1835		----		----	
8011		----		----	
normality		OK			
n		16			
outliers		2			
mean (n)		6.728			
st.dev. (n)		0.0939			
R(calc.)		0.263			
R(D1364:07)		0.182			



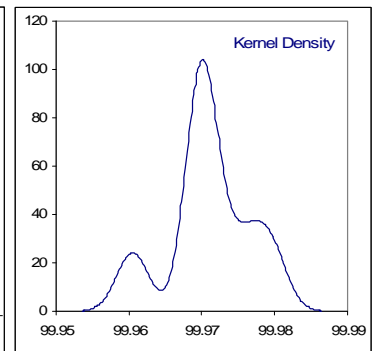
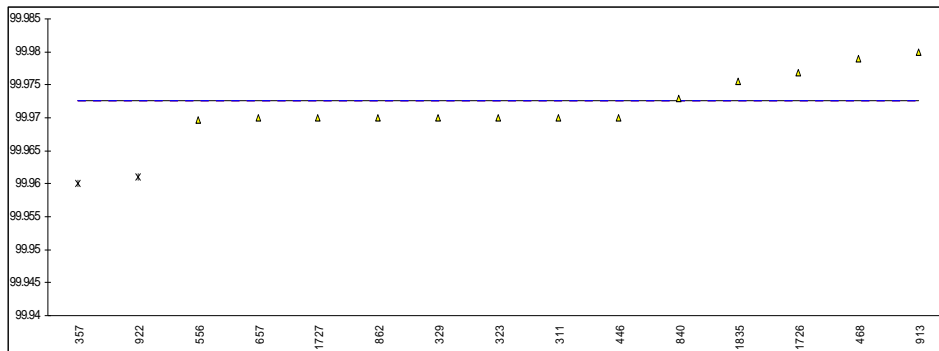
Determination of Permanganate Time Test @ 15 °C on sample #0994; results in minutes

lab	method	value	mark	z(targ)	remarks
310		----		----	
311	D1363	2		----	
323	D1363	<5		----	
329	D1363	<5		----	
338		----		----	
342	D1363	1.1		----	
357	D1363	<1		----	
359	D1363	<1		----	
395		----		----	
446		----		----	
468		----		----	
541	D1363	4		----	
551		----		----	
556	D1363	2.00		----	
559		----		----	
657	D1363	2.5		----	
840	D1363	3		----	
862	D1363	2		----	
913	D1363	9.0	G(0.01)	----	
922	D1363	3		----	
1066	D1363	1		----	
1205		----		----	
1241		----		----	
1242		----		----	
1247		----		----	
1253		----		----	
1260		----		----	
1270		----		----	
1425		----		----	
1605		----		----	
1726	D1363	1.15		----	
1727		----		----	
1835		----		----	
8011		----		----	
normality		OK			
n		10			
outliers		1			
mean (n)		2.2			
st.dev. (n)		0.97			
R(calc.)		2.7			
R(D1363:06)		(0.6)			



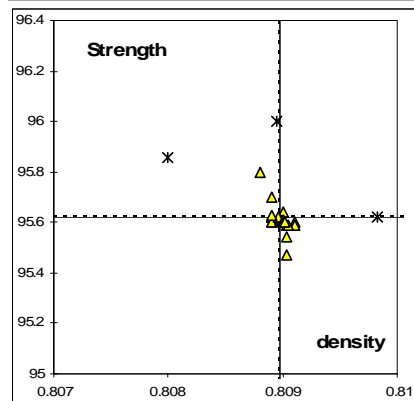
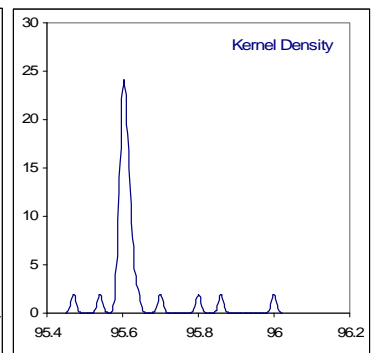
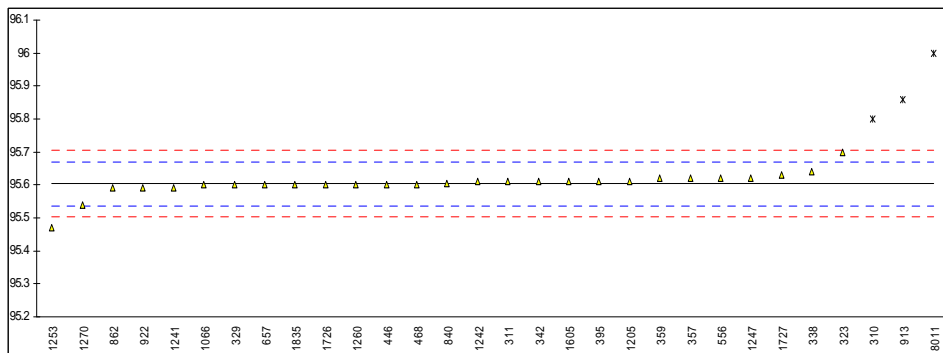
Determination of Purity on dry basis on sample #0994; results in %M/M

lab	method	value	mark	z(targ)	remarks
310		----		----	
311	INH-529	99.97		----	
323	INH-155	99.97		----	
329	INH-155	99.97		----	
338		----		----	
342		----		----	
357	INH-012	99.96	D(0.05)	----	
359		----		----	
395		----		----	
446	INH-61	99.97		----	
468		99.979		----	
541		----		----	
551		----		----	
556		99.9696		----	
559		----		----	
657	INH-01	99.97		----	
840	INH-0001	99.973		----	
862	IHN-01	99.970		----	
913	In House	99.98		----	
922	Calculated	99.961	D(0.05)	----	
1066		----		----	
1205		----		----	
1241		----		----	
1242		----		----	
1247		----		----	
1253		----		----	
1260		----		----	
1270		----		----	
1425		----		----	
1605		----		----	
1726	In House	99.9769		----	
1727		99.97		----	
1835	In House	99.9755		----	
8011		----		----	
	normality	not OK			
	n	13			
	outliers	2			
	mean (n)	99.9726			
	st.dev. (n)	0.00386			
	R(calc.)	0.0108			
	R(lit)	unknown			
				Compare R(iis08C11b) = 0.0126	



Determination of Strength on sample #0994; results in %V/V

lab	method	value	mark	z(targ)	Remarks
310	GC	95.8		5.81	
311		95.61		0.19	
323	D4052(Calc.)	95.7		2.85	
329	D4052(Calc.)	95.6		-0.10	
338		95.64		1.08	
342	OIML	95.61		0.19	
357	OIML	95.62		0.49	
359	OIML	95.62		0.49	
395	OIML	95.61		0.19	
446	OIML	95.60		-0.10	
468	OIML	95.6	CG(0.01)	-0.10	Result switched with %M/M, reported first 93.3
541		-----		-----	
551		-----		-----	
556	D4052	95.62		0.49	
559		-----		-----	
657	OIML	95.60		-0.10	
840	OIML	95.603		-0.01	
862		95.59		-0.40	
913	OIML	95.86	CG(0.01)	7.58	Result switched with %M/M, reported first 93.63
922	OIML	95.59		-0.40	
1066		95.6		-0.10	
1205	OIML	95.611		0.22	
1241	Vapodest	95.59		-0.40	
1242		95.61		0.19	
1247		95.62		0.49	
1253	In House	95.47		-3.95	
1260	OIML	95.60		-0.10	
1270		95.54		-1.88	
1425		-----		-----	
1605	OIML	95.61		0.19	
1726		95.60		-0.10	
1727		95.63		0.78	
1835	OIML	95.60		-0.10	
8011		96.00	G(0.01)	11.72	
normality		not OK			
n		27			
outliers		3			
mean (n)		95.603			
st.dev. (n)		0.0368			
R(calc.)		0.103			
R(D4052:calc)		0.095			
					Compare R(iis08C11b) = 0.078

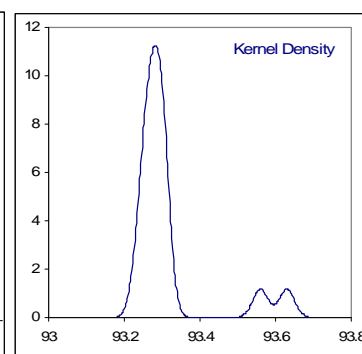
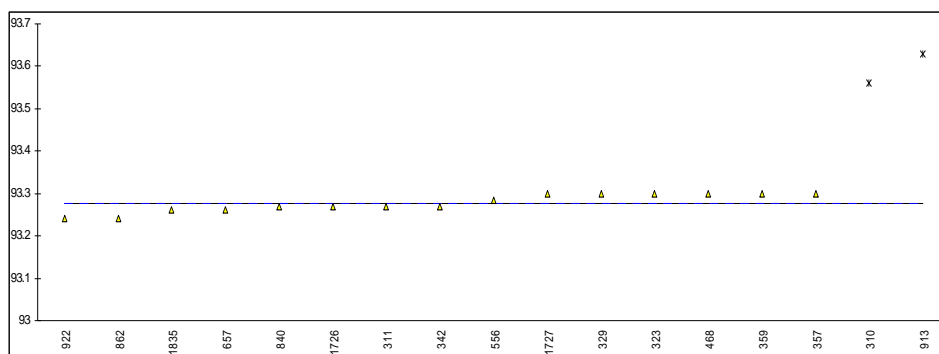


Determination of Strength on sample #0994; results in %M/M

lab	method	value	mark	z(targ)	remarks
310	GC	93.56	G(0.01)	----	
311		93.27		----	
323	D4052(Calc.)	93.3		----	
329	D4052(Calc.)	93.3		----	
338		----		----	
342	OIML	93.27		----	
357	OIML	93.30		----	
359	OIML	93.30		----	
395		----		----	
446		----		----	
468	OIML	93.3	C	----	Result switched with %M/M, reported first 95.6
541		----		----	
551		----		----	
556	D4052	93.285		----	
559		----		----	
657	OIML	93.26		----	
840	OIML	93.268		----	
862		93.24		----	
913	OIML	93.63	CG(0.01)	----	Result switched with %V/V, reported first 95.86
922	OIML	93.24		----	
1066		----		----	
1205		----		----	
1241		----		----	
1242		----		----	
1247		----		----	
1253		----		----	
1260		----		----	
1270		----		----	
1425		----		----	
1605		----		----	
1726		93.27		----	
1727		93.30		----	
1835	OIML	93.26		----	
8011		----		----	

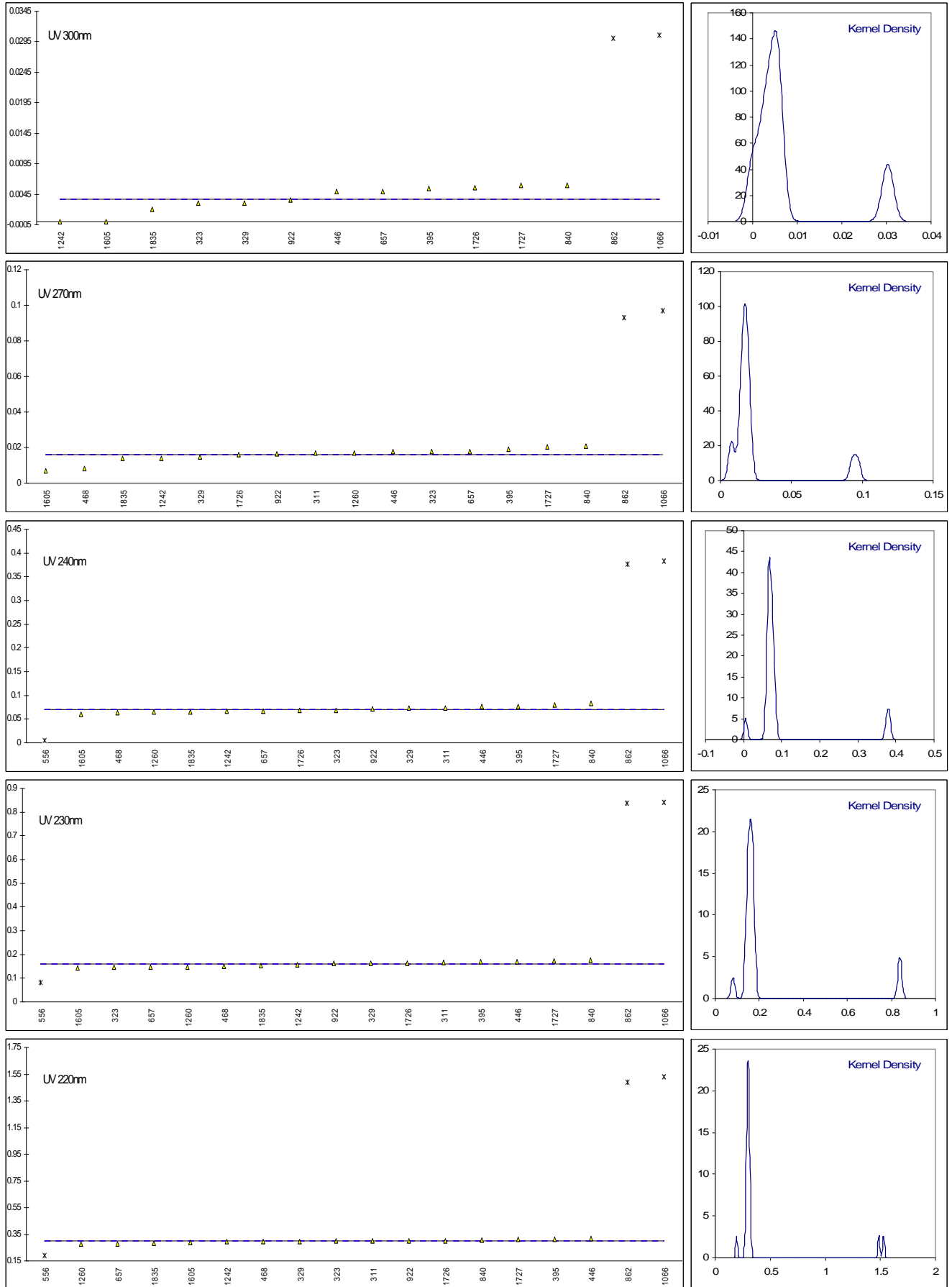
normality not OK  
n 15  
outliers 2  
mean (n) 93.278  
st.dev. (n) 0.0220  
R(calc.) 0.062  
R(lit) unknown

Compared R(iis08C11b) = 0.129



## Determination of UV absorbance on sample #0994;

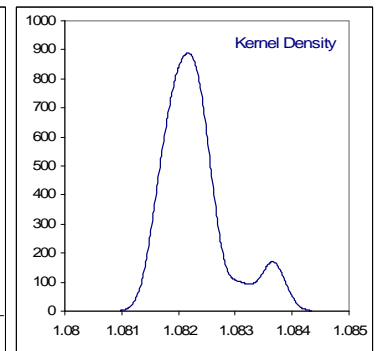
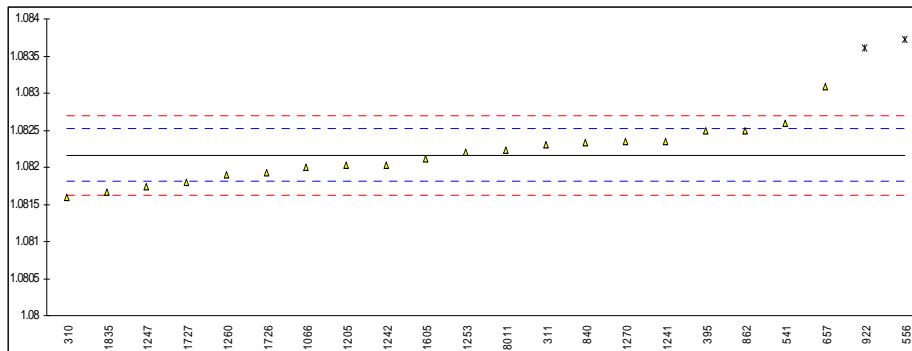
lab	method	300nm	mark	270nm	mark	240nm	mark	230nm	mark	220nm	mark
310		----		----		----		----		----	
311		<0.005		0.017		0.073		0.166		0.300	
323	INH-BP	0.003		0.018		0.068		0.146		0.298	
329	INH-BP	0.003		0.015		0.073		0.163		0.297	
338		----		----		----		----		----	
342		----		----		----		----		----	
357		----		----		----		----		----	
359		----		----		----		----		----	
395	INH-12	0.0054		0.0192		0.0763		0.1708		0.3125	
446	INH-13	0.005		0.018		0.076		0.171		0.321	
468	INH-OS	<0.001		0.0083		0.0636		0.1514		0.2938	
541		----		----		----		----		----	
551		----		----		----		----		----	
556	UV	<0.001		<0.001	False -	0.0054	G(0.01)	0.0813	G(0.01)	0.1933	G(0.01)
559		----		----		----		----		----	
657	INH-60	0.005		0.018		0.067		0.148		0.280	
840	IMPCA004	0.0060		0.0207		0.0829		0.1760		0.3065	
862		0.030	G(0.01)	0.093	G(0.01)	0.376	G(0.01)	0.837	G(0.01)	1.487	G(0.01)
913		----		----		----		----		----	
922		0.003510		0.016370		0.071458		0.162410		0.30018	
1066		0.03061	G(0.01)	0.09682	G(0.05)	0.3830	G(0.05)	0.8400	G(0.05)	1.529	G(0.05)
1205		----		----		----		----		----	
1241		----		----		----		----		----	
1242		0.000		0.014		0.067		0.158		0.293	
1247		----		----		----		----		----	
1253		----		----		----		----		----	
1260		----		0.017		0.065		0.148		0.278	
1270		----		----		----		----		----	
1425		----		----		----		----		----	
1605		0.000		0.007		0.060		0.142		0.292	
1726		0.00555		0.01625		0.06790		0.16340		0.30251	
1727		0.00593		0.02040		0.080348		0.17360		0.31164	
1835		0.0020		0.0137		0.0660		0.15281		0.28511	
8011		----		----		----		----		----	
	normality	OK		OK		OK		OK		OK	
	n	12		15		15		15		15	
	outliers	2		2		3		3		3	
	mean (n)	0.00370		0.01593		0.0705		0.1595		0.2981	
	st.dev. (n)	0.002158		0.003931		0.00640		0.01094		0.01194	
	R(calc.)	0.00604		0.01101		0.0179		0.0306		0.0334	
	R(lit)	unknown		unknown		unknown		unknown		unknown	
	R(iis08C11b)	0.00051		0.00685		0.0084		0.0204		0.0238	
	R(iis07C11b)	0.0076		0.0167		0.0209		0.0365		0.0654	



Determination of Density @ 20°C on peach liqueur: sample #0995; results in kg/L

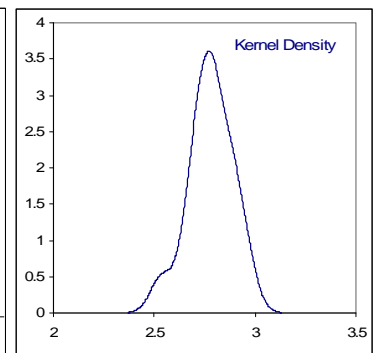
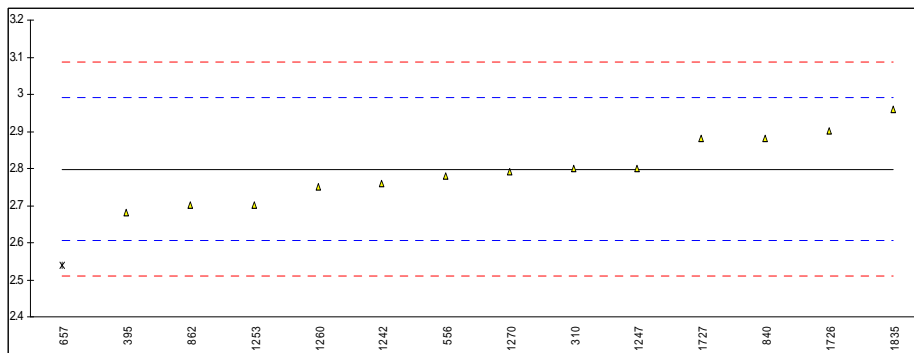
lab	method	value	mark	z(targ)	remarks
310	D4052	1.0816		-3.15	
311	D4052	1.0823		0.77	
323		-----		-----	
329		-----		-----	
338		-----		-----	
342		-----		-----	
357		-----		-----	
359		-----		-----	
395	D4052	1.0825		1.89	
446		-----		-----	
468		-----		-----	
541	D4052	1.0826		2.45	
551		-----		-----	
556	D4052	1.08372	DG(0.01)	8.72	
559		-----		-----	
657	D4052	1.08309		5.19	
840	D4052	1.08233		0.94	
862	D4052	1.0825		1.89	
913		-----		-----	
922	D4052	1.08361	DG(0.01)	8.11	
1066	D4052	1.0820		-0.91	
1205	INH-APD	1.082026		-0.76	
1241	DE50	1.08235		1.05	
1242	D4052	1.082032	C	-0.73	
1247	D4052	1.08174		-2.37	
1253	DE45	1.08221		0.27	
1260	DMA	1.08190		-1.47	
1270	D4052	1.082342		1.01	
1425		-----		-----	
1605	D4052	1.08211		-0.29	
1726	D4052	1.08193		-1.30	
1727	D4052	1.08180		-2.03	
1835	D4052	1.08166		-2.81	
8011	D4052	1.08223		0.38	

normality OK  
n 20  
outliers 2  
mean (n) 1.08216  
st.dev. (n) 0.000360  
R(calc.) 0.00101  
R(D4052:09) 0.00050



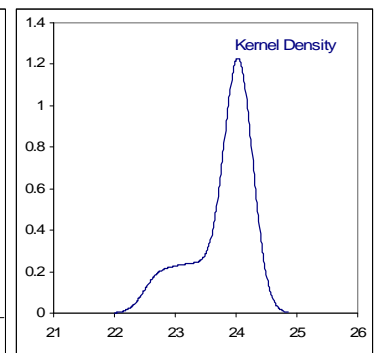
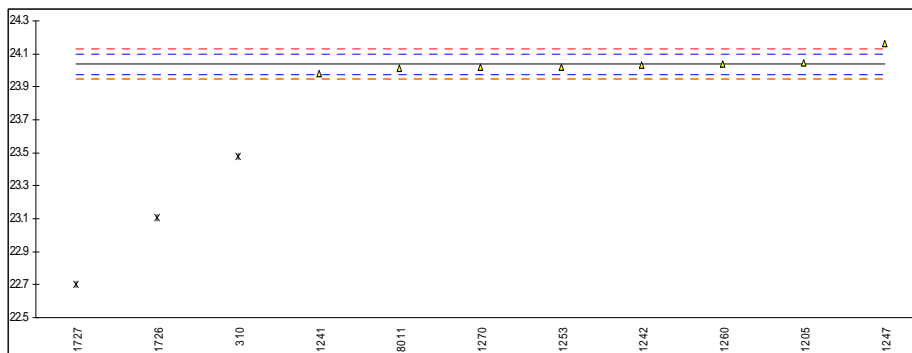
Determination of pH on peach liqueur: sample #0995;

lab	method	value	mark	z(targ)	remarks
310		2.80		0.02	
311		----		----	
323		----		----	
329		----		----	
338		----		----	
342		----		----	
357		----		----	
359		----		----	
395	D6423	2.68		-1.23	
446		----		----	
468		----		----	
541		----		----	
551		----		----	
556	INH-10891	2.78		-0.19	
559		----		----	
657	INH-56	2.54	G(0.05)	-2.69	
840	In House	2.88		0.85	
862	E70	2.70		-1.03	
913		----		----	
922		----		----	
1066		----		----	
1205		----		----	
1241		----		----	
1242		2.76		-0.40	
1247		2.8		0.02	
1253	Electrode	2.70		-1.03	
1260	Electrode	2.75		-0.51	
1270		2.79		-0.09	
1425		----		----	
1605		----		----	
1726	EN15490	2.90		1.06	
1727		2.88		0.85	
1835	EN15490	2.96		1.68	
8011		----		----	
	normality	OK			
	n	13			
	outliers	1			
	mean (n)	2.80			
	st.dev. (n)	0.085			
	R(calc.)	0.24			
	R(EN15490:07)	0.27			



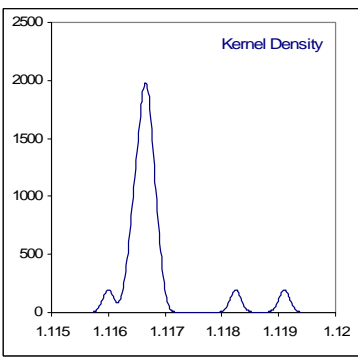
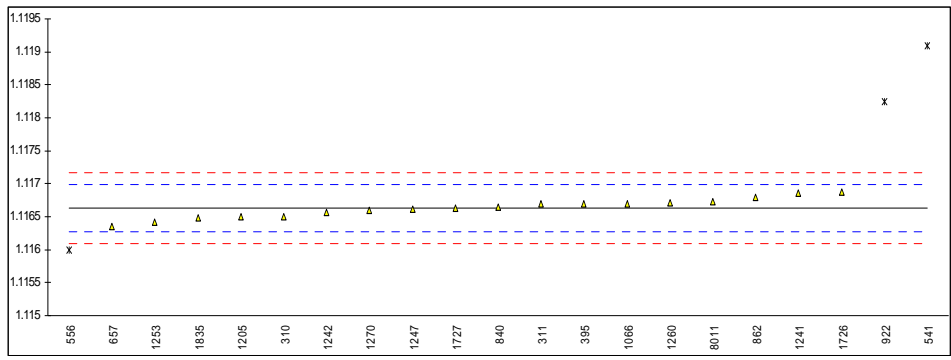
Determination of Strength on peach liqueur: sample #0995; results in %V/V

lab	method	value	mark	z(targ)	remarks
310	In House	23.48	G(0.01)	-18.17	
311		----		----	
323		----		----	
329		----		----	
338		----		----	
342		----		----	
357		----		----	
359		----		----	
395		----		----	
446		----		----	
468		----		----	
541		----		----	
551		----		----	
556		----		----	
559		----		----	
657		----		----	
840		----		----	
862		----		----	
913		----		----	
922		----		----	
1066		----		----	
1205	OIML	24.044		0.20	
1241	Vapodest	23.98		-1.89	
1242		24.03		-0.26	
1247		24.16		3.97	
1253	Buchi	24.02		-0.59	
1260	In House	24.04		0.07	
1270		24.02		-0.59	
1425		----		----	
1605		----		----	
1726	In House	23.1065	DG(0.01)	-30.33	
1727		22.7	DG(0.01)	-43.56	
1835		----		----	
8011		24.01		-0.91	
	normality	not OK			
	n	8			
	outliers	3			
	mean (n)	24.038			
	st.dev. (n)	0.0532			
	R(calc.)	0.149			
	R(PTS 39:09)	0.086			



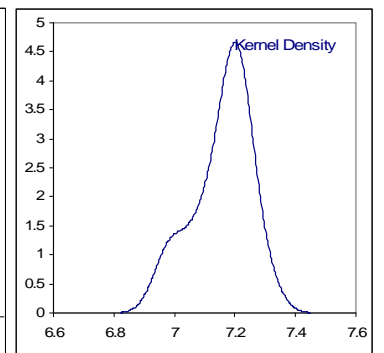
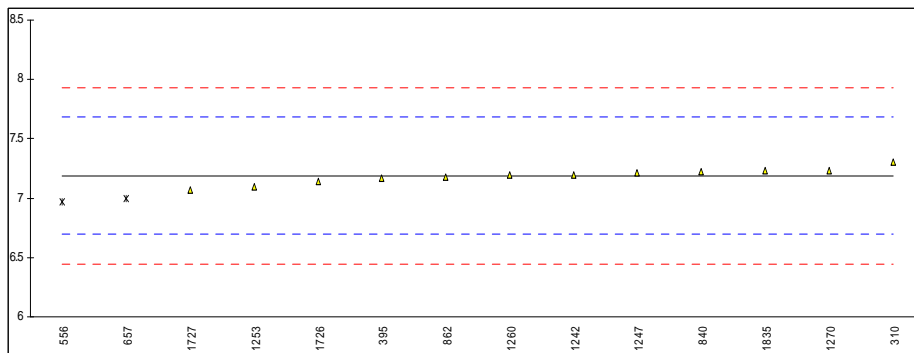
Determination of Density @ 20°C on cream liqueur: sample #0996; results in kg/L

lab	method	value	mark	z(targ)	remarks
310	D4052	1.1165		-0.75	
311	D4052	1.1167		0.37	
323		----		----	
329		----		----	
338		----		----	
342		----		----	
357		----		----	
359		----		----	
395	D4052	1.1167		0.37	
446		----		----	
468		----		----	
541	D4052	1.1191	CG(0.01)	13.81	
551		----		----	
556	D4052	1.1160	G(0.01)	-3.55	
559		----		----	
657	D4052	1.11635		-1.59	
840	D4052	1.11665		0.09	
862	D4052	1.1168		0.93	
913		----		----	
922	D4052	1.11824	G(0.01)	8.99	
1066	D4052	1.1167		0.37	
1205	INH-P	1.1165		-0.75	
1241	DE50	1.11686		1.26	
1242	D4052	1.116571		-0.35	
1247	D4052	1.11662		-0.08	
1253	DE45	1.11642		-1.20	
1260	DMA	1.11672		0.48	
1270	D4052	1.116605		-0.16	
1425		----		----	
1605		----		----	
1726	D4052	1.11687		1.32	
1727	D4052	1.11663		-0.02	
1835	D4052	1.11649		-0.81	
8011	D4052	1.11673		0.54	
normality		OK			
n		18			
outliers		3			
mean (n)		1.11663			
st.dev. (n)		0.000144			
R(calc.)		0.00040			
R(D4052:09)		0.00050			



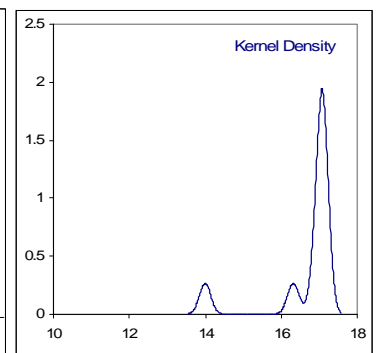
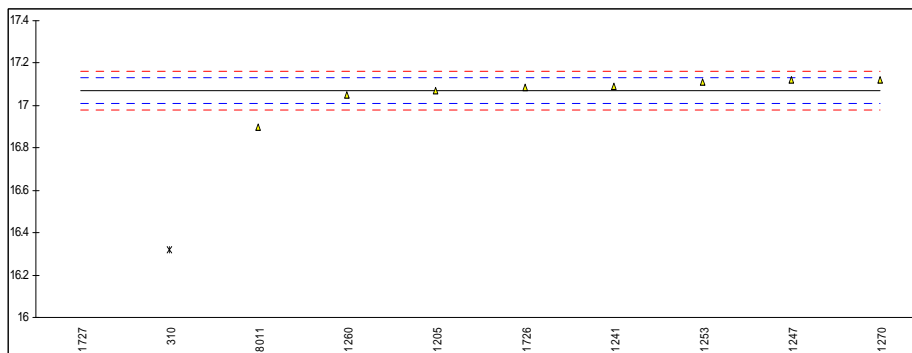
Determination of pH on cream liqueur: sample #0996;

lab	method	value	mark	z(targ)	Remarks
310		7.30		0.46	
311		----		----	
323		----		----	
329		----		----	
338		----		----	
342		----		----	
357		----		----	
359		----		----	
395		7.17		-0.07	
446		----		----	
468		----		----	
541		----		----	
551		----		----	
556	INH-10891	6.97	G(0.05)	-0.88	
559		----		----	
657	INH-56	7.00	G(0.05)	-0.76	
840	In House	7.22		0.13	
862	E70	7.18		-0.03	
913		----		----	
922		----		----	
1066		----		----	
1205		----		----	
1241		----		----	
1242		7.20		0.05	
1247	In House	7.21		0.09	
1253	Electrode	7.1		-0.36	
1260	Electrode	7.20		0.05	
1270		7.23		0.17	
1425		----		----	
1605		----		----	
1726	EN15490	7.14	C	-0.19	
1727		7.07		-0.48	
1835	EN15490	7.23		0.17	
8011		----		----	
	normality	OK			
	n	12			
	outliers	2			
	mean (n)	7.19			
	st.dev. (n)	0.062			
	R(calc.)	0.17			
	R(EN15490:07)	0.69			



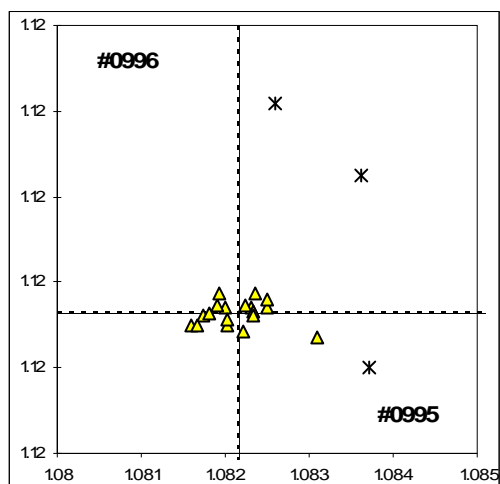
Determination of Strength on cream liqueur; sample #0996; results in %V/V

lab	method	value	mark	z(targ)	remarks
310	In House	16.32	G(0.01)	-24.65	
311		----		----	
323		----		----	
329		----		----	
338		----		----	
342		----		----	
357		----		----	
359		----		----	
395		----		----	
446		----		----	
468		----		----	
541		----		----	
551		----		----	
556		----		----	
559		----		----	
657		----		----	
840		----		----	
862		----		----	
913		----		----	
922		----		----	
1066		----		----	
1205	OIML	17.069		0.02	
1241	Vapodest	17.09		0.71	
1242		----		----	
1247	In House	17.12		1.70	
1253	Buchi	17.11		1.37	
1260	In House	17.05		-0.60	
1270		17.12		1.70	
1425		----		----	
1605		----		----	
1726	In House	17.0874	C	0.63	
1727		14.0	G(0.01)	-101.07	
1835		----		----	
8011	In House	16.90		-5.54	
	normality	OK			
	n	8			
	outliers	2			
	mean (n)	17.068			
	st.dev. (n)	0.0723			
	R(calc.)	0.202			
	R(PTS 39:09)	0.085			



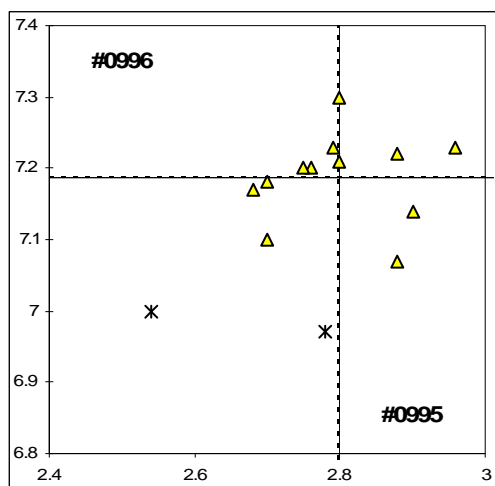
Determination of Density @ 20°C on sample #0995 and #0996; results in kg/L

lab	method	#0995	mark	#0996	mark	remarks
310	D4052	1.0816		1.1165		
311	D4052	1.0823		1.1167		
323		----		----		
329		----		----		
338		----		----		
342		----		----		
357		----		----		
359		----		----		
395	D4052	1.0825		1.1167		
446		----		----		
468		----		----		
541	D4052	1.0826		1.1191	G(0.01)	
551		----		----		
556	D4052	1.08372	G(0.01)	1.1160	G(0.01)	
559		----		----		
657	D4052	1.08309		1.11635		
840	D4052	1.08233		1.11665		
862	D4052	1.0825		1.1168		
913		----		----		
922	D4052	1.08361	G(0.01)	1.11824	G(0.05)	
1066	D4052	1.0820		1.1167		
1205	INH-P	1.08203		1.1165		
1241	DE50	1.08235		1.11686		
1242	D4052	1.08203		1.11657		
1247	D4052	1.08174		1.11662		
1253	DE45	1.08221		1.11642		
1260	DMA	1.08190		1.11672		
1270	D4052	1.08234		1.11661		
1425		----		----		
1605		1.08211		----		
1726	D4052	1.08193		1.11687		
1727	D4052	1.08180		1.11663		
1835	D4052	1.08166		1.11649		
8011	D4052	1.08223		1.11673		
normality		OK		OK		
n		20		18		
outliers		2		3		
mean (n)		1.08216		1.11663		
st.dev. (n)		0.000360		0.000144		
R(calc.)		0.00101		0.00040		
R(D4052:02e1)		0.00050		0.00050		



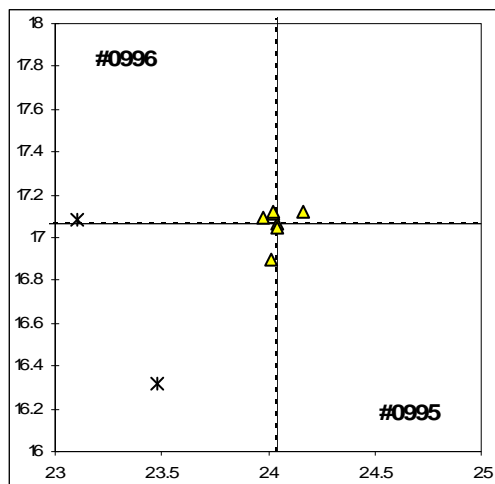
Determination of pH on sample #0995 and #0996;

lab	method	#0995	mark	#0996	mark	remarks
310		2.80		7.30		
311		----		----		
323		----		----		
329		----		----		
338		----		----		
342		----		----		
357		----		----		
359		----		----		
395	D6423	2.68		7.17		
446		----		----		
468		----		----		
541		----		----		
551		----		----		
556	INH-10891	2.78		6.97	G(0.05)	
559		----		----		
657	INH-56	2.54	G(0.05)	7.00	G(0.05)	
840	In House	2.88		7.22		
862	E70	2.70		7.18		
913		----		----		
922		----		----		
1066		----		----		
1205		----		----		
1241		----		----		
1242		2.76		7.20		
1247		2.8		7.21		
1253	Electrode	2.70		7.1		
1260	Electrode	2.75		7.20		
1270		2.79		7.23		
1425		----		----		
1605		----		----		
1726	EN15490	2.90		7.14		
1727		2.88		7.07		
1835	EN15490	2.96		7.23		
8011		----		----		
	normality	OK		OK		
	n	13		12		
	outliers	1		2		
	mean (n)	2.80		7.19		
	st.dev. (n)	0.085		0.062		
	R(calc.)	0.24		0.17		
	R(EN15490:07)	0.27		0.69		



Determination of Strength on sample #0995 and #0996; results in %V/V

lab	method	#0995	mark	#0996	mark	remarks
310	In House	23.48	G(0.01)	16.32	G(0.01)	
311		----		----		
323		----		----		
329		----		----		
338		----		----		
342		----		----		
357		----		----		
359		----		----		
395		----		----		
446		----		----		
468		----		----		
541		----		----		
551		----		----		
556		----		----		
559		----		----		
657		----		----		
840		----		----		
862		----		----		
913		----		----		
922		----		----		
1066		----		----		
1205	OIML	24.044		17.069		
1241	Vapodest	23.98		17.09		
1242		24.03		----		
1247		24.16		17.12		
1253	Buchi	24.02		17.11		
1260	In House	24.04		17.05		
1270		24.02		17.12		
1425		----		----		
1605		----		----		
1726	In House	23.1065	DG(0.01)	17.0874		
1727		22.7	DG(0.01)	14.0	G(0.01)	
1835		----		----		
8011		24.01		16.90		
	normality	not OK		OK		
	n	8		8		
	outliers	3		2		
	mean (n)	24.038		17.068		
	st.dev. (n)	0.0532		0.0723		
	R(calc.)	0.149		0.202		
	R(lit)	unknown		unknown		



## APPENDIX 2

### List of number of participants per country

1 laboratory in ARGENTINA  
4 laboratories in BELGIUM  
3 laboratories in BRAZIL  
2 laboratories in FINLAND  
1 laboratory in FRANCE  
1 laboratory in HONG KONG  
1 laboratory in INDIA  
1 laboratory in ITALY  
1 laboratory in P.R. of CHINA  
1 laboratory in PAKISTAN  
1 laboratory in SINGAPORE  
4 laboratories in SPAIN  
1 laboratory in SWEDEN  
9 laboratories in THE NETHERLANDS  
1 laboratory in UNITED KINGDOM  
1 laboratory in VIETNAM

## APPENDIX 3

### 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
U	= unit error
SDS	= safety data sheet

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