

Results of Proficiency Test
Isopropanol
(Isopropyl alcohol)
November 2009

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2003, the Institute for Interlaboratory Studies organises a proficiency test for the analysis of Isopropanol. As part of the annual proficiency test program of 2009/2010 the Institute decided to continue this proficiency test on Isopropanol. The proficiency test of Isopropanol has been organised in accordance with the latest applicable version of the ASTM D770:05 specifications and a number of additional tests requested by some participants. In this international interlaboratory study, 16 laboratories out of 13 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. Analyses for fit-for-use and homogeneity testing were subcontracted. It was decided to send one sample (1* 500 mL, labelled #0997) to the participants. The participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO guide 43 and ILAC-G13:2007, (R007), since January 2000, by the Dutch Accreditation Council (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 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

Approximately 25 litre of very pure Isopropanol was obtained from a local trader. This material was spiked with 13.1 mg Sodium Chloride. After homogenisation, the material was divided over 35 amber glass bottles of 500 mL with inner and outer caps and labelled #0997. The homogeneity of subsample #0997 was checked by determination Density in accordance with ASTM D4052:02e1, Water in accordance with ASTM D1364:07 and Chloride as Cl in accordance with an in house test method on 8 stratified random selected samples.

	Density at 20°C in kg/L	Water content in %M/M	Chloride in mg/kg
sample #0997-1	0.78522	0.059	0.6
sample #0997-2	0.78519	0.060	0.5
sample #0997-3	0.78522	0.061	0.5
sample #0997-4	0.78523	0.061	0.5
sample #0997-5	0.78524	0.061	0.5
sample #0997-6	0.78523	0.061	0.5
sample #0997-7	0.78524	0.060	0.5
sample #0997-8	0.78524	0.060	0.5

Table 1: homogeneity test of subsample #0997

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities of the target methods or with the reproducibility calculate using the Horwitz equation in agreement with the procedure of ISO 13528, Annex B2 in the next table

	Density at 20°C in kg/L	Water in %M/M	Chloride in mg/kg
r (Observed)	0.00002	0.001	0.10
reference method	ASTM D4052:02e1	ASTM D1364:07	IMPCA002:98
0.3 * R (ref method)	0.00015	0.002	0.10

Table 2: repeatabilities of subsamples #0997

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibility of the target method. Therefore, homogeneity of the samples was assumed.

To each of the participating laboratories 1* 0.5 litre (labelled #0997) was sent on November 04, 2009.

2.5 STABILITY OF THE SAMPLES

The stability of Isopropanol, packed in a amber glass bottle, was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were asked to determine Acidity, Anorganic Chloride, Appearance, Colour Pt/Co, Density 20°C, Distillation (IBP, 50% evaporated & DP), Nonvolatile Matter, Peroxide, Specific Gravity 20/20°C, Water, Purity (both "as it is" and on dry basis), Ethanol, n-Propanol, n-Butanol, Methyl-Ethyl-Ketone and Other Impurities on sample #0997. To get comparable results, a detailed report form on which the units and the preferred test methods were printed, was sent together with each of the samples. Also 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 gathered. The original data are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder fax was sent to the laboratories that had not reported 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

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 of the results should be used with due care.

In accordance to 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 them with a factor of 2.8.

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

3.2 GRAPHICS

In order to visualise 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 method is for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nr.12 and 13).

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. The z-scores were calculated in accordance with:

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

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. The participants in India and P.R. of China received the samples after the formal final reporting date. The participant in Mexico did not receive the samples at all due to custom clearance problems. From the 15 reporting participants, five participants did report in the results after the final reporting date. The 15 reporting laboratories submitted 165 numerical results. Observed were 5 outlying results, which is 3.0 %. In proficiency studies, outlier percentages of 3 % - 7.5 % are quite normal.

4.1 EVALUATION PER TEST

In this section, the results are discussed per test. When no literature reproducibility was available, other target values were used. In those cases, the Horwitz equation is used to estimate the target reproducibility.

Not normal distributions were found for the following determinations: Acidity, Colour Pt/Co, Distillation (IBP, 50% evaporated, DP), Water and Methyl-Ethyl-Ketone. In these cases the statistical evaluation should be used with due care.

Acidity: This determination is not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D 1613:06.

Anorg. Chloride: This determination may be problematic. Although no statistical outliers were observed, the calculated reproducibility is not in agreement with the requirements of IMPCA002:98. The large spread may be partly explained by the small numbers of results.

Appearance: No analytical problems were observed. All labs agreed about the appearance of sample #0997, which is bright, clear and free of suspended matter. The uniformity of reporting can be improved. A new standardized method is available for Appearance since 2009, being ASTM E2680. According this method the appearance should be reported as 'pass' (or 'fail').

Colour Pt/Co: No analytical problems were observed. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D1209:05.

Density @ 20 °C: No analytical problems were observed. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D4052:02e1.

Distillation: This determination is not problematic. No statistical outliers were observed and all three calculated reproducibilities are in good agreement with the requirements of ASTM D1078:05 for the automated and the manual mode.

NVM: This determination is not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D1353:03.

Peroxide: It is hard to draw any conclusions, because only two participants reported a (numerical) test result.

- Specific Gravity @ 20/20 °C: This determination is problematic for one laboratory. Only one statistical outlier was observed and the calculated reproducibility is, after rejection of the statistical outlier, in good agreement with the requirements of ASTM D4052:02e1.
- Water: No analytical problems were observed. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D1364:07.
- Purity "as is": Regretfully, the methods used do not provide any reproducibility value, therefore no significant conclusions were drawn. However, the calculated reproducibility, after rejection of the statistical outlier, is smaller than the calculated reproducibility found in the previous proficiency test (iis07C15) of December 2007 (0.062 vs 0.072)
- Purity on dry basis: Regretfully, the methods used do not provide any reproducibility value, therefore no significant conclusions were drawn. However, the calculated reproducibility is somewhat then the calculated reproducibility found in the previous proficiency test (iis07C15) of December 2007 (0.052 vs 0.045).
- Ethanol: Too few numerical results were reported for meaningful conclusions. Only four participants reported a numerical test result.
- n-Propanol: This determination is problematic for one laboratory. Only one statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier, is in full agreement with the estimated reproducibility calculated using the Horwitz equation.
- n-Butanol: This determination may be very problematic. One statistical outlier and three false negatives were observed. The calculated reproducibility, after rejection of the statistical outliers, is not at all in agreement with the estimated reproducibility calculated using the Horwitz equation.
- MEK: This determination may be very problematic. Only one statistical outlier was observed. However, the calculated reproducibility, after rejection of the statistical outlier, is not at all in agreement with the estimated reproducibility, calculated using the Horwitz equation.
- Other Imp.: The results reported for this parameter vary over a rather large range: between 391 – 800 mg/kg. This might be explained by the large variety of methods used and the definition of the term "other impurities". To obtain optimum results each component should be identified and calculated using the appropriate response factor.

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 that participated. The reproducibilities derived from literature standards (in casu ASTM standards) and the calculated reproducibilities of sample #0790 are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acidity as Acetic Acid	%M/M	13	0.00078	0.00075	0.00140
Anorganic Chloride	mg/kg	7	0.48	0.41	0.30
Appearance		12	B&C	--	--
Colour	Pt/Co	7	3.4	3.2	7.0
Density @ 20°C	kg/L	13	0.78521	0.00029	0.00050
Initial Boiling Point	°C	11	82.12	0.24	1.28
50% evaporated	°C	11	82.25	0.19	0.56
Dry Point	°C	11	82.48	0.27	0.88
Nonvolatile Matter	mg/100mL	7	0.51	0.56	2.40
Peroxide	mg/kg	2	16.7	unknown	1.4
Specific Gravity @ 20/20°C		10	0.78654	0.00043	0.00050
Water	%M/M	13	0.0623	0.0118	0.0150
Purity	%M/M	9	99.868	0.062	unknown
Purity on dry basis	%M/M	11	99.929	0.052	unknown
Ethanol	mg/kg	4	10.3	5.5	(3.3)
n-Propanol	mg/kg	10	106.0	24.8	23.5
n-Butanol	mg/kg	5	13.3	17.9	4.0
Methyl-Ethyl-Ketone	mg/kg	7	12.0	11.4	3.7
Other Impurities	mg/kg	8	542.8	375.4	unknown

Table 3: Reproducibilities for sample #0790

Without further statistical calculations it can be concluded that for many tests there is a not a good compliance of the group of participants with the relevant standards. The tests that are problematic have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2009 WITH THE PREVIOUS PTS.

	<i>November 2009</i>	<i>November 2007</i>	<i>November 2006</i>	<i>December 2005</i>
Number of reporting labs	15	15	14	13
Number of results reported	165	148	139	135
Statistical outliers	5	10	9	7
Percentage outliers	3.0%	6.8%	6.5%	5.2%

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:

Determination	<i>November 2009</i>	<i>November 2007</i>	<i>November 2006</i>	<i>December 2005</i>
Acidity as acetic acid	++	++	++	++
Anorganic Chlorides as Cl	-	++	++	n.e.
Colour Pt/Co	++	++	++	++
Density @ 20°C	++	++	++	++
Initial Boiling Point	++	++	++	++
50% evaporated	++	n.e.	n.e.	n.e.
Dry Point	++	++	++	++
Nonvolatile Matter	++	++	++	++
Specific Gravity 20/20 °C	++	++	n.d.	n.d.
Water	++	++	++	++
Purity	(+)	(--)	(-)	(++)
Purity on dry basis	(-)	(+)	(-)	(++)
Ethanol	(--)	n.e.	n.e.	--
n-Propanol	+/-	+/-	++	++
n-Butanol	--	++	--	-
Methyl-Ethyl-Ketone	--	++	--	++

Table 5: comparison determinations against the standard requirements

Results between brackets are compared with reproducibility of the previous round robin, due to the lack of target data.

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

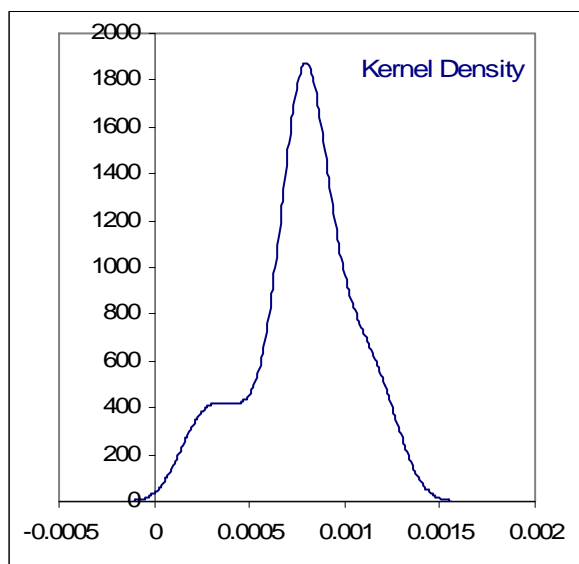
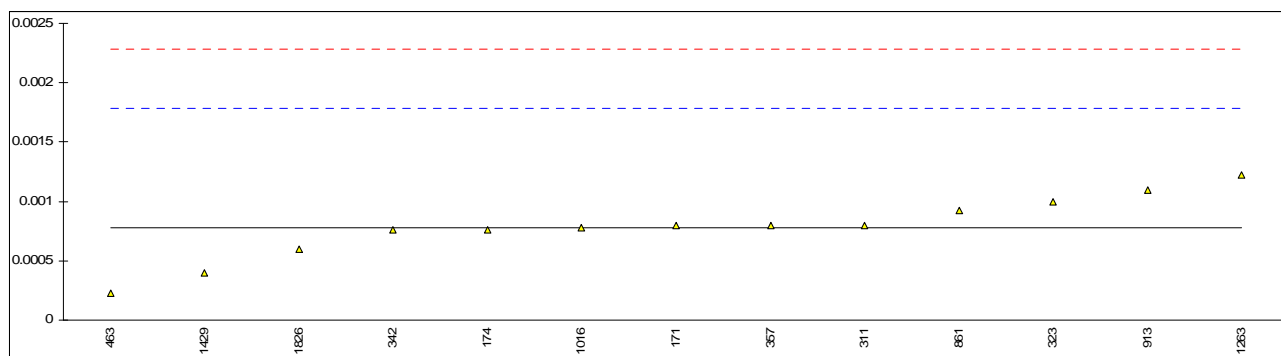
++:	group performed much better than the standard
+	group performed better than the standard
+/-:	group performance equals the standard
-	group performed worse than the standard
--:	group performed much worse than the standard
n.d.:	not determined
n.e.:	not evaluated

APPENDIX 1

Determination of Acidity as Acetic Acid on sample #0997; results in %M/M.

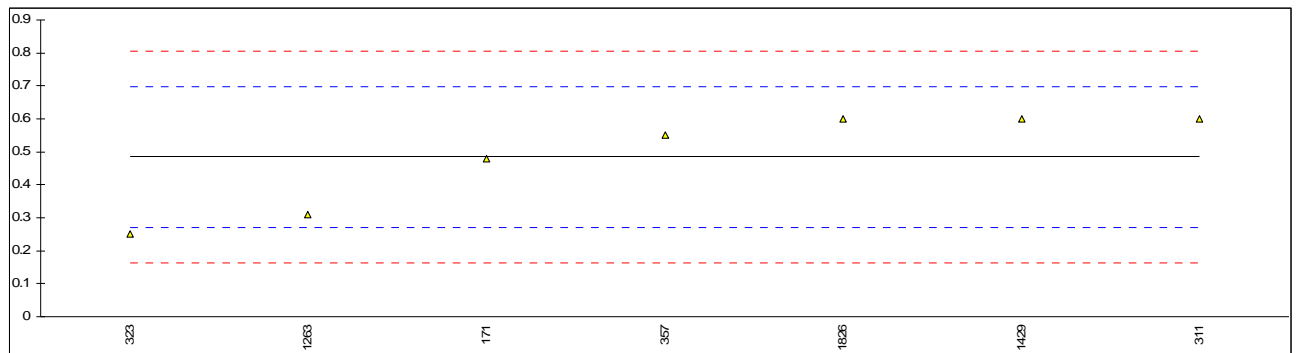
lab	method	value	mark	z(targ)	remarks
171	D1613	0.0008		0.04	
174	D1613	0.00076		-0.04	
311	D1613	0.0008		0.04	
323	D1613	0.0010		0.44	
342	D1613	0.00076		-0.04	
357	D1613	0.0008		0.04	
463	D1613	0.000227		-1.11	
522		----		----	
613		----		----	
861	D1613	0.00092		0.28	
913	D1613	0.0011		0.64	
1016	D1613	0.00078		0.00	
1255		----		----	
1263	D1613	0.00122		0.88	
1429	D1613	0.0004		-0.76	
1826	D1613	0.0006		-0.36	

normality not OK
n 13
outliers 0
mean (n) 0.00078
st.dev. (n) 0.000266
R(calc.) 0.00075
R(D1613:06) 0.00140



Determination of Anorganic Chloride as Cl on sample #0997; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
171	D5808	0.48		-0.04	
174		----		----	
311	INH-158	0.6		1.08	
323	IMPCA002	0.25		-2.19	
342		----		----	
357	IMPCA002	0.55		0.61	
463		----		----	
522		----		----	
613		----		----	
861		----		----	
913		----		----	
1016		----		----	
1255		----		----	
1263	EN14077	0.310		-1.63	
1429		0.6		1.08	
1826		0.60		1.08	
normality	OK				
n	7				
outliers	0		<u>Spike:</u>		
mean (n)	0.48		0.50		Recovery <104%
st.dev. (n)	0.147				
R(calc.)	0.41				
R(IMPCA002:98)	0.30				



Determination of Appearance on sample #0997;

lab	method	value	mark	z(targ)	remarks
171	E2680	PASS		----	
174	E2680	PASS		----	
311	E2680	PASS		----	
323	INH-001	CFFSM		----	
342		----		----	
357	E2680	PASS		----	
463		----		----	
522		----		----	
613		----		----	
861	E2680	C&B		----	
913	E2680	CFFSM		----	
1016	E2680	PASS		----	
1255		----		----	
1263		----		----	
1429	E2680	C&B		----	
1826	E2680	PASS		----	

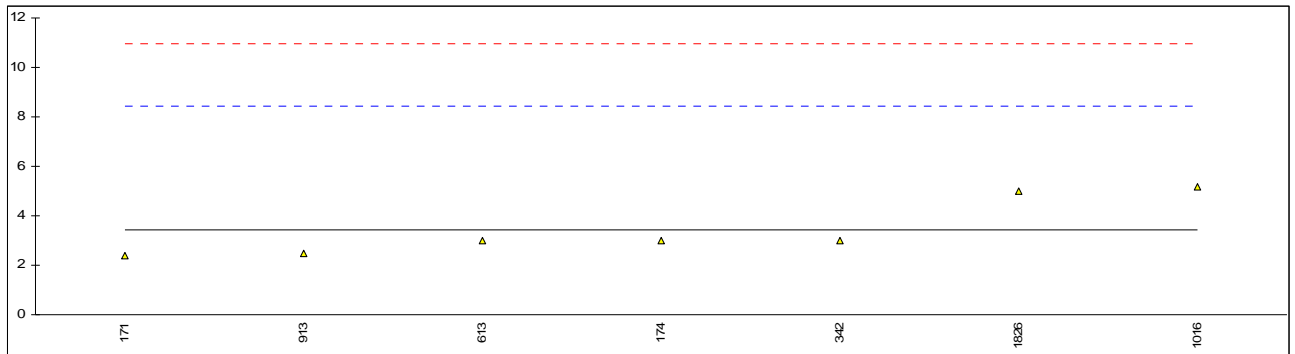
C&B = Clear and Bright

CFFSM = Clear and free from Suspended Matter

Determination of Colour Pt/Co scale on sample #0997;

lab	method	value	mark	z(targ)	remarks
171	D1209	2.4		-0.42	
174	D1209	3		-0.18	
311	D1209	<5		----	
323	D1209	<5		----	
342	D1209	3		-0.18	
357	D1209	<5		----	
463	D1209	<3		----	
522		----		----	
613	D1209	3		-0.18	
861	D1209	<5		----	
913	D1209	2.5		-0.38	
1016	D1209	5.18		0.70	
1255		----		----	
1263		----		----	
1429	D1209	<5		----	
1826	D1209	5		0.62	

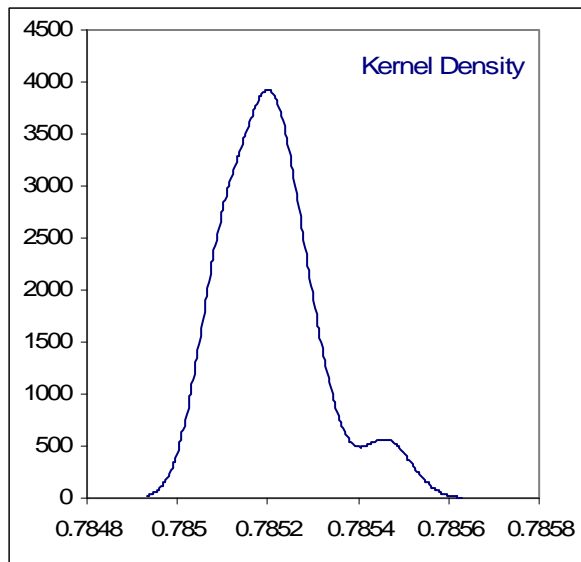
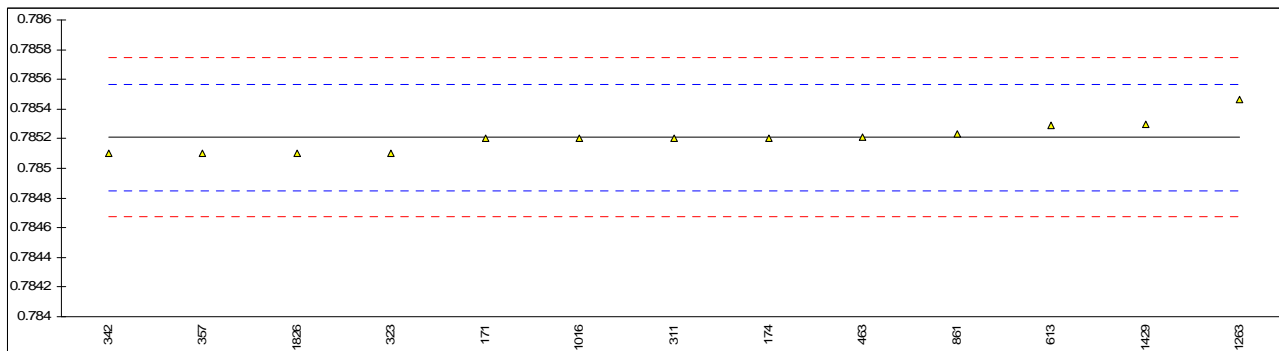
normality not OK
n 7
outliers 0
mean (n) 3.4
st.dev. (n) 1.16
R(calc.) 3.2
R(D1209:05) 7.0



Determination of Density 20 °C on sample #0997; results in kg/L.

lab	method	value	mark	z(targ)	remarks
171	D4052	0.7852		-0.04	
174	D4052	0.7852		-0.04	
311	D4052	0.7852		-0.04	
323	D4052	0.7851		-0.60	
342	D4052	0.7851		-0.60	
357	D4052	0.7851		-0.60	
463	D4052	0.78521		0.02	
522		-----		-----	
613	D4052	0.78529		0.46	
861	D4052	0.78523		0.13	
913		-----		-----	
1016	D4052	0.7852		-0.04	
1255		-----		-----	
1263	ISO12185	0.785462		1.43	
1429	D4052	0.7853		0.52	
1826	D4052	0.7851		-0.60	

normality OK
n 13
outliers 0
mean (n) 0.78521
st.dev. (n) 0.000103
R(calc.) 0.00029
R(D4052:02e1) 0.00050

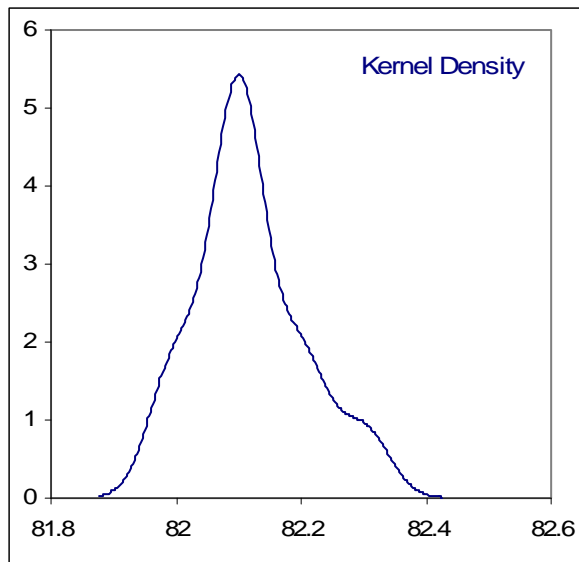
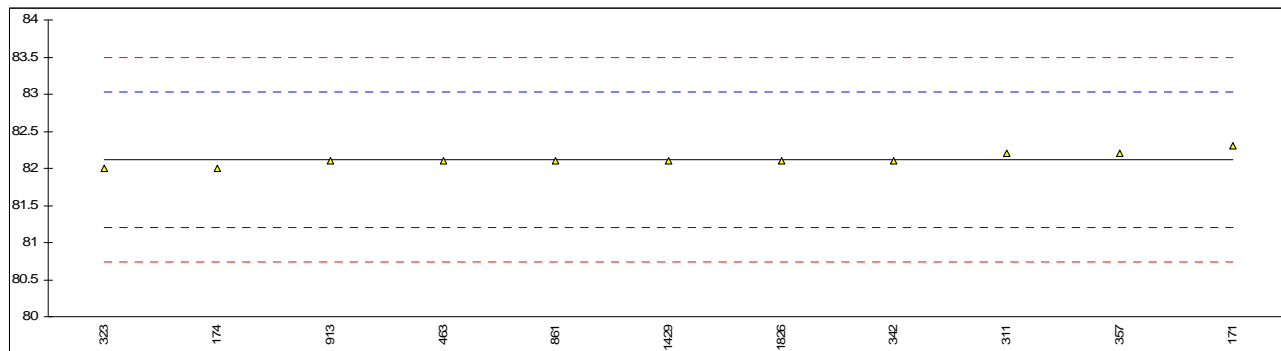


Determination of Distillation: Initial Boiling Point @ 760 mmHg on sample #0997; results in °C.

lab	method	value	mark	z(targ)	remarks
171	D1078-A	82.3		0.40	
174	D1078-A	82.0		-0.26	
311	D1078-A	82.2		0.18	
323	D1078-M	82.0		-0.26	
342	D1078-M	82.1		-0.04	
357	D1078-A	82.2		0.18	
463	D1078-A	82.1		-0.04	
522		----		----	
613		----		----	
861	D1078-M	82.1		-0.04	
913	D1078-M	82.1		-0.04	
1016		----		----	
1255		----		----	
1263		----		----	
1429	D1078-A	82.1		-0.04	
1826	D1078-M	82.1		-0.04	

normality not OK
n 11
outliers 0
mean (n) 82.12
st.dev. (n) 0.087
R(calc.) 0.24
R(D1078:05-A) 1.28

Compare R(D1078:05-M) = 0.88

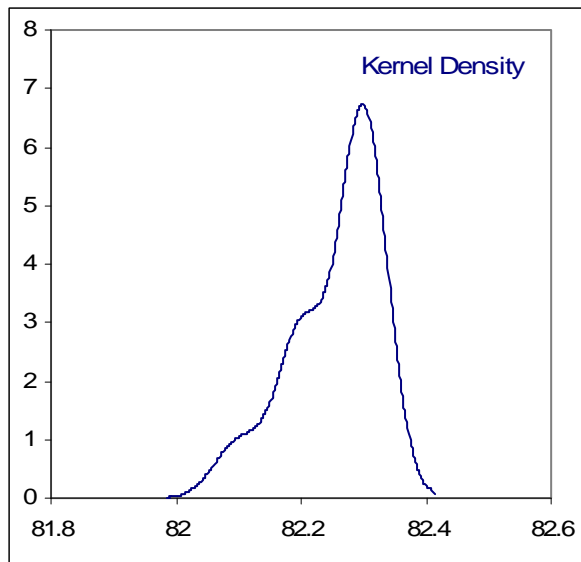
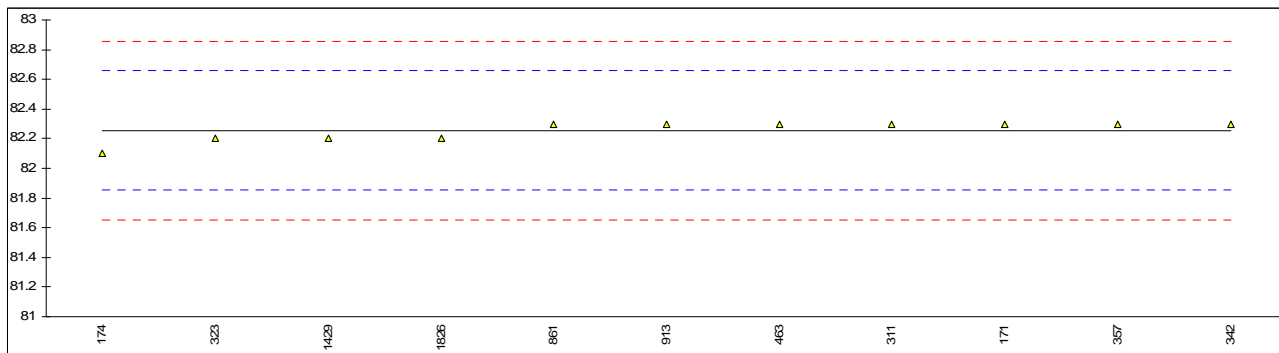


Determination of Distillation: 50% evaporated @ 760 mmHg on sample #0997; results in °C.

lab	method	value	mark	z(targ)	remarks
171	D1078-A	82.3		0.23	
174	D1078-A	82.1		-0.77	
311	D1078-A	82.3		0.23	
323	D1078-M	82.2		-0.27	
342	D1078-M	82.3		0.23	
357	D1078-A	82.3		0.23	
463	D1078-A	82.3		0.23	
522		----		----	
613		----		----	
861	D1078-M	82.3		0.23	
913	D1078-M	82.3		0.23	
1016		----		----	
1255		----		----	
1263		----		----	
1429	D1078-A	82.2		-0.27	
1826	D1078-M	82.2		-0.27	

normality not OK
n 11
outliers 0
mean (n) 82.25
st.dev. (n) 0.069
R(calc.) 0.19
R(D1078:05-A) 0.56

Compare R(D1078:05-M) = 0.55

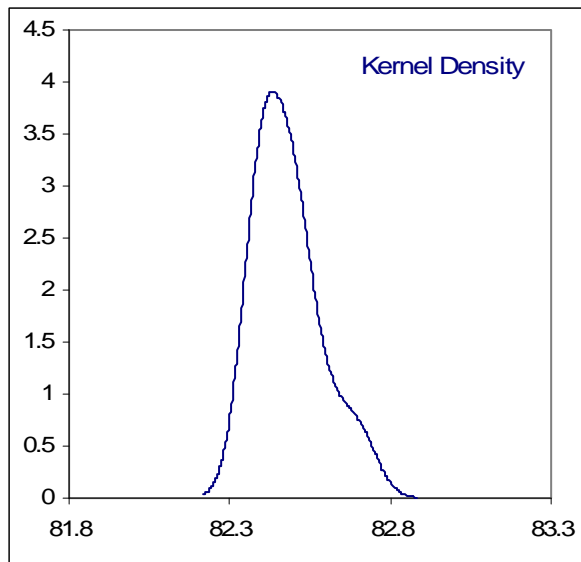
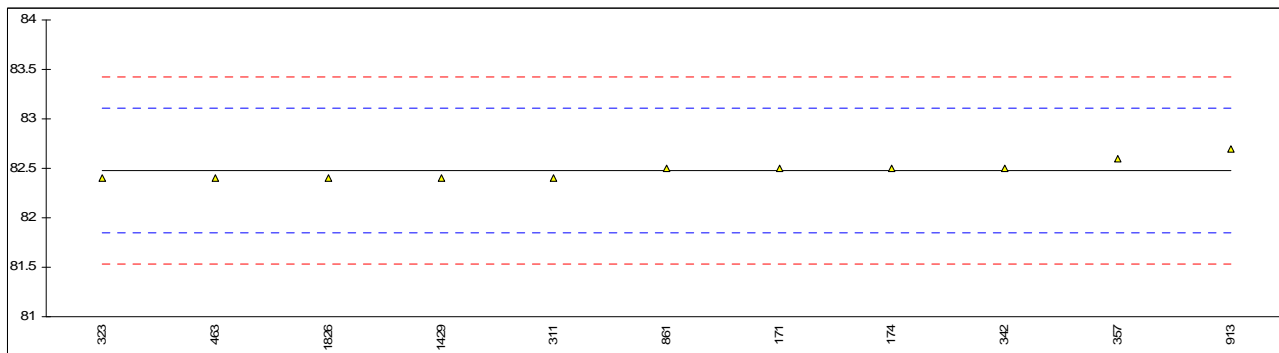


Determination of Distillation: Dry Point @ 760 mmHg on sample #0997; results in °C.

lab	method	value	mark	z(targ)	remarks
171	D1078-A	82.5		0.06	
174	D1078-A	82.5		0.06	
311	D1078-A	82.4		-0.26	
323	D1078-M	82.4		-0.26	
342	D1078-M	82.5		0.06	
357	D1078-A	82.6		0.37	
463	D1078-A	82.4		-0.26	
522		----		----	
613		----		----	
861	D1078-M	82.5		0.06	
913	D1078-M	82.7		0.69	
1016		----		----	
1255		----		----	
1263		----		----	
1429	D1078-A	82.4		-0.26	
1826	D1078-M	82.4		-0.26	

normality not OK
n 11
outliers 0
mean (n) 82.48
st.dev. (n) 0.098
R(calc.) 0.27
R(D1078:05-A) 0.88

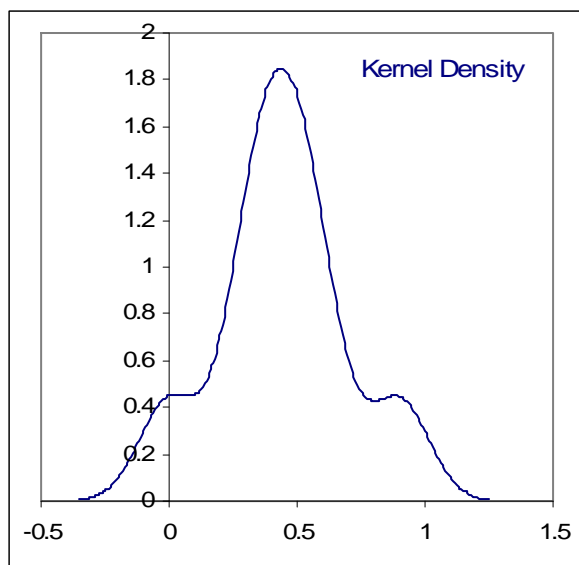
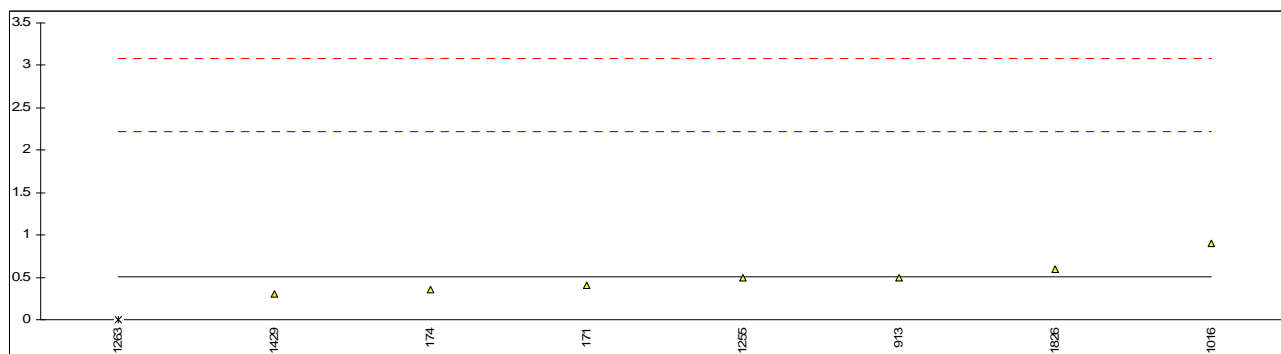
Compare R(D1078:05-M) = 1.07



Determination of Nonvolatile Matter on sample #0997; results in mg/100 mL.

lab	method	value	mark	z(targ)	remarks
171	D1353	0.4		-0.13	
174	D1353	0.35		-0.18	
311		----		----	
323	D1353	<1		----	
342		----		----	
357	D1353	<1		----	
463		----		----	
522		----		----	
613		----		----	
861	D1353	<1		----	
913	D1353	0.5		-0.01	
1016	D1353	0.9		0.46	
1255	ISO6353	0.5		-0.01	
1263	D1353	0.0	ex	-0.59	Result excluded from statistical evaluation, not a real result
1429	D1353	0.3		-0.24	
1826	D1353	0.6		0.11	

normality OK
n 7
outliers 0
mean (n) 0.51
st.dev. (n) 0.201
R(calc.) 0.56
R(D1353:03) 2.40



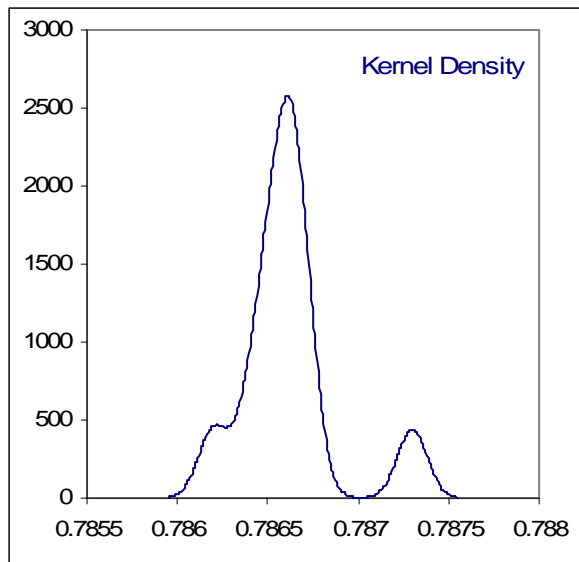
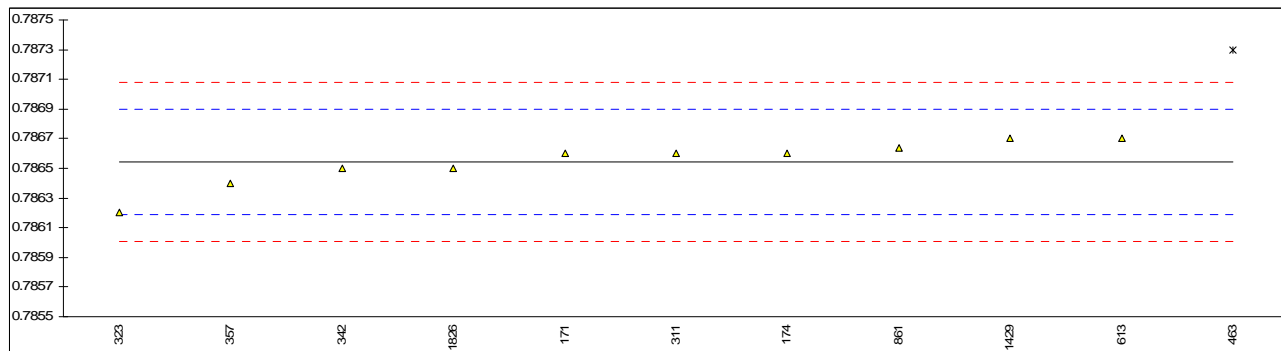
Determination of Peroxide as O on sample #0997; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
171	E299	14.3		----	
174		----		----	
311		----		----	
323		----		----	
342		----		----	
357	E299	19		----	
463		----		----	
522		----		----	
613		----		----	
861		----		----	
913		----		----	
1016		----		----	
1255		----		----	
1263		----		----	
1429		----		----	
1826		----		----	
	normality	n.a.			
	n	2			
	outliers	0			
	mean (n)	16.65			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(E299:08)	1.4			

Determination of Specific Gravity 20/20°C on sample #0997;

lab	method	value	mark	z(targ)	remarks
171	D4052	0.7866		0.31	
174	D4052	0.7866		0.31	
311	D4052	0.7866		0.31	
323	D4052	0.7862		-1.93	
342	D4052	0.7865		-0.25	
357	CALC	0.7864		-0.81	
463		0.7873	C,G(0.05)	4.23	First reported 0.7913 (at 20°C)
522		-----		-----	
613	D4052	0.7867		0.87	
861	D4052	0.78664		0.54	
913		-----		-----	
1016		-----		-----	
1255		-----		-----	
1263		-----		-----	
1429		0.7867		0.87	
1826		0.7865		-0.25	

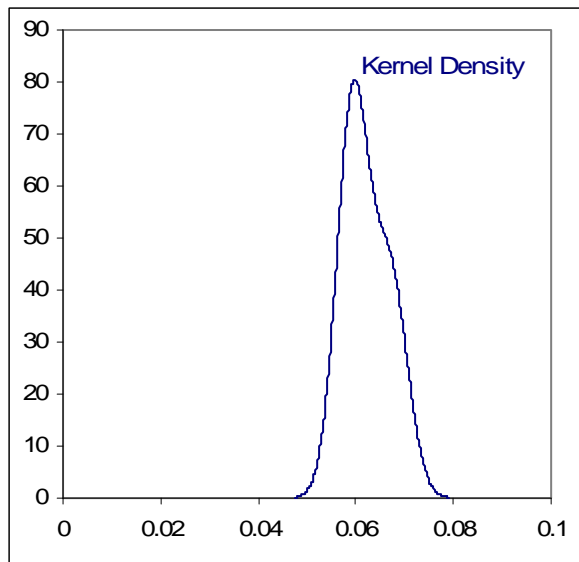
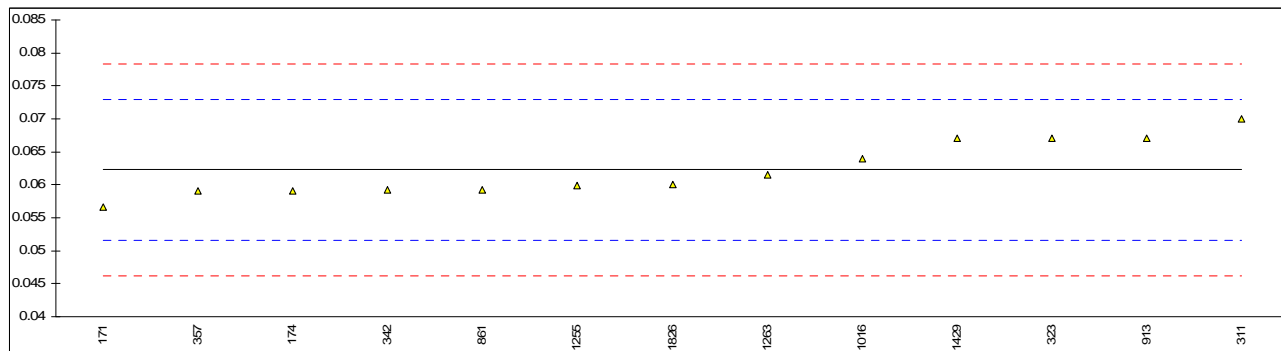
normality OK
 n 10
 outliers 1
 mean (n) 0.78654
 st.dev. (n) 0.000153
 R(calc.) 0.00043
 R(D4052:02e1) 0.00050



Determination of Water content on sample #0997; results in %M/M.

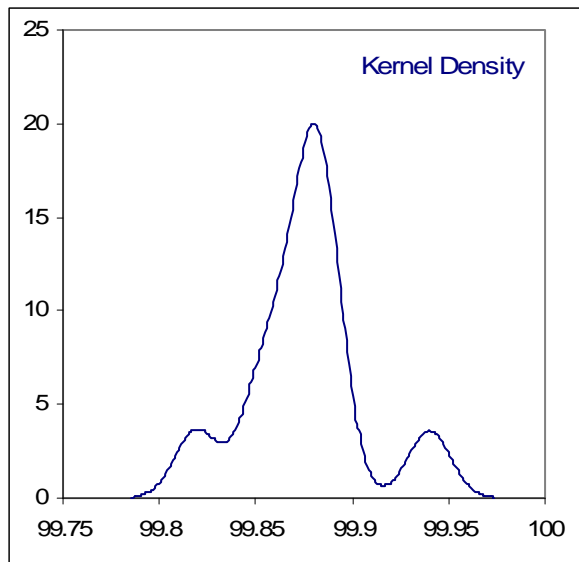
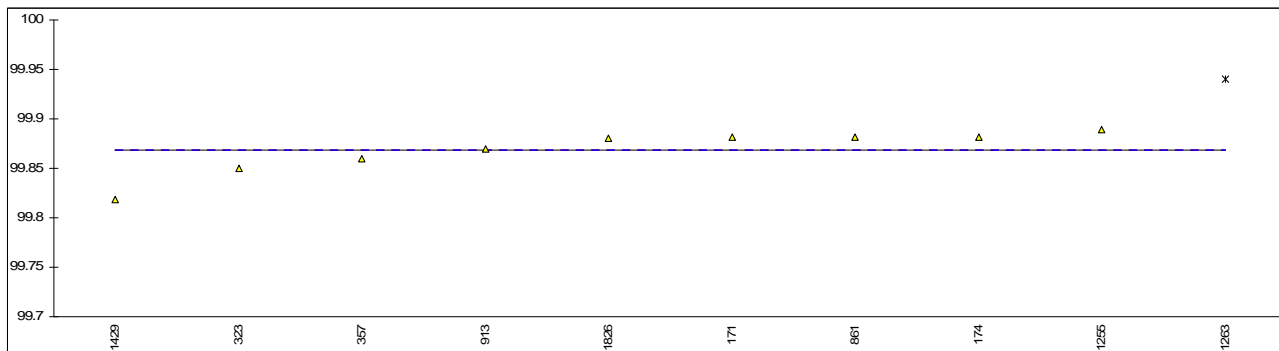
lab	method	value	mark	z(targ)	remarks
171	D1364	0.0566		-1.06	
174	D1364	0.059		-0.61	
311	D1364	0.070		1.45	
323	E203	0.067		0.89	
342	E1064	0.0592		-0.57	
357	E1064	0.059		-0.61	
463		----		----	
522		----		----	
613		----		----	
861	E1064	0.0593		-0.55	
913	D1364	0.067		0.89	
1016	D1364	0.064		0.32	
1255	ISO760	0.0599	C	-0.44	First reported 0.044
1263	ISO12937	0.06147		-0.15	
1429	D1364	0.0670	C	0.89	First reported 0.084
1826	D1364	0.060		-0.42	

normality not OK
n 13
outliers 0
mean (n) 0.0623
st.dev. (n) 0.00421
R(calc.) 0.0118
R(D1364:07) 0.0150



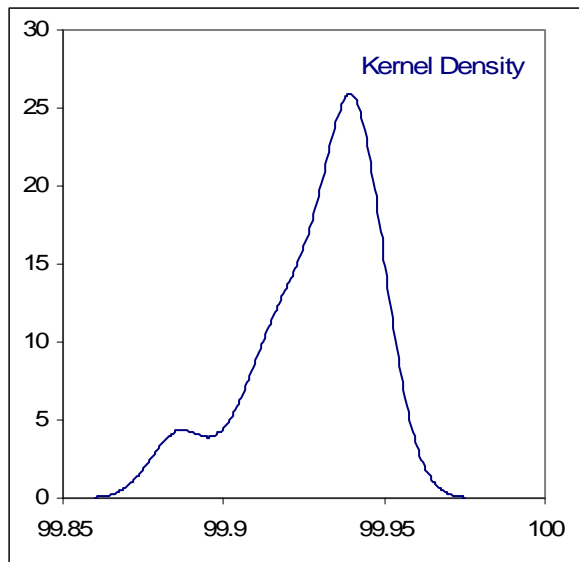
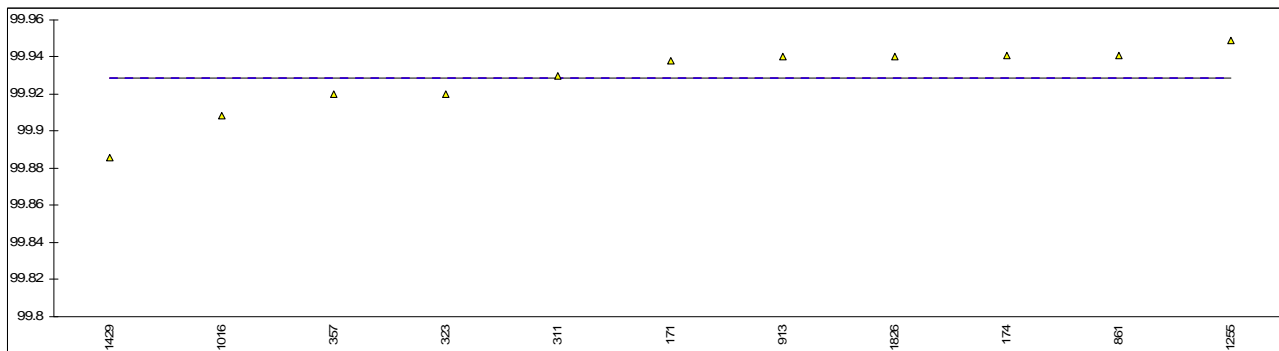
Determination of Purity "as is" on sample #0997; results in %M/M.

lab	method	value	mark	z(targ)	remarks
171	D5501	99.881		----	
174	D5501	99.882		----	
311		----		----	
323	INH-060	99.85		----	
342		----		----	
357	INH-012	99.86		----	
463		----		----	
522		----		----	
613		----		----	
861	INH-041	99.882		----	
913	GC	99.87		----	
1016		----		----	
1255	GC	99.889	C	----	First reported 99.9049
1263	D5501	99.94	D(0.05)	----	
1429		99.8190		----	
1826	in house	99.88		----	
normality		OK			
n		9			
outliers		1			
mean (n)		99.8681			
st.dev. (n)		0.02219			
R(calc.)		0.0621			
R(lit.)		unknown			Compare R(iis07C15) = 0.0721



Determination of Purity on dry basis on sample #0997, results in %M/M.

lab	method	value	mark	z(targ)	remarks
171	D5501	99.938		----	
174	D5501	99.941		----	
311	INH-082	99.93		----	
323	INH-060	99.92		----	
342		----		----	
357	INH-012	99.92		----	
463		----		----	
522		----		----	
613		----		----	
861	INH-041	99.941		----	
913	GC	99.94		----	
1016		99.9082	C	----	First reported 99.9286
1255	GC	99.9489		----	
1263		----		----	
1429		99.8860	C	----	First reported 99.97
1826	in house	99.94		----	
normality		OK			
n		11			
outliers		0			
mean (n)		99.9285			
st.dev. (n)		0.01857			
R(calc.)		0.0520			
R(lit.)		unknown			Compare R(iis07C15) = 0.045

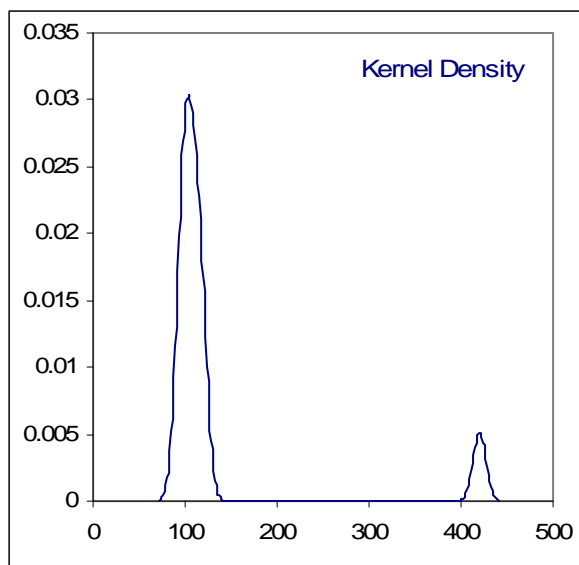
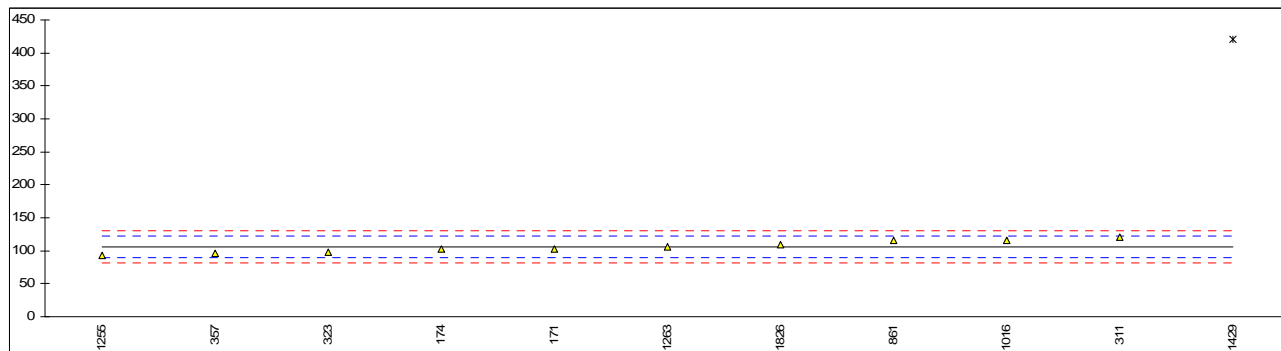


Determination of Ethanol content on sample #0997; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
171	D5501	11.0		----	
174	D5501	10.8		----	
311	INH-082	12		----	
323	INH-060	<5		----	
342		----		----	
357	INH-012	<10		----	
463		----		----	
522		----		----	
613		----		----	
861	INH-041	<10		----	
913		----		----	
1016		----		----	
1255	GC	7.5		----	
1263		----		----	
1429		<10		----	
1826	in house	<100		----	
	normality	unknown			
	n	4			
	outliers	0			
	mean (n)	10.33			
	st.dev. (n)	1.955			
	R(calc.)	5.47			
	R(Horwitz)	(3.26)			

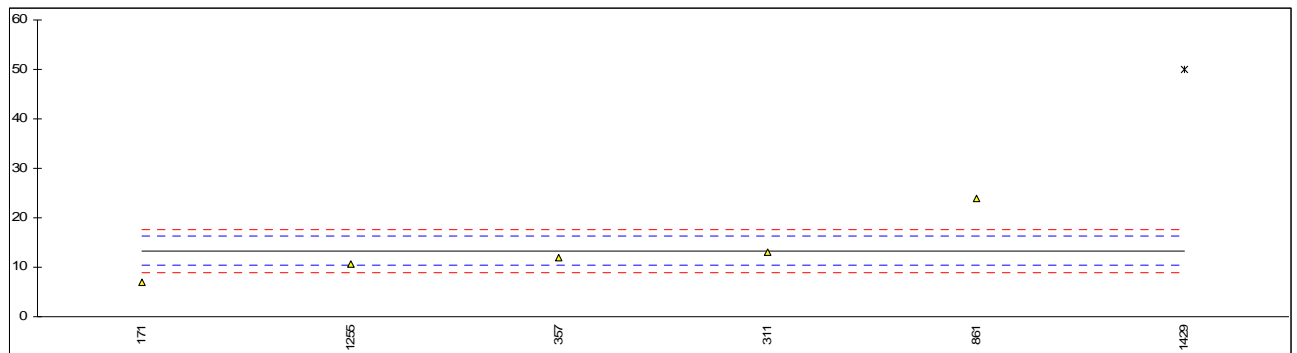
Determination of n-Propanol on sample #0997; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
171	D5501	103.1		-0.34	
174	D5501	102.4		-0.43	
311	INH-082	120		1.67	
323	INH-060	98		-0.95	
342		----		----	
357	INH-012	97		-1.07	
463		----		----	
522		----		----	
613		----		----	
861	INH-041	115		1.07	
913		----		----	
1016		115.1		1.09	
1255	GC	93.2		-1.52	
1263	D5501	106.0		0.00	
1429		420	CG(0.01)	37.36	First reported <10
1826	in house	110		0.48	
normality		OK			
n		10			
outliers		1			
mean (n)		105.98			
st.dev. (n)		8.857			
R(calc.)		24.80			
R(Horwitz)		23.53			



Determination of n-Butanol on sample #0997; results in mg/kg.

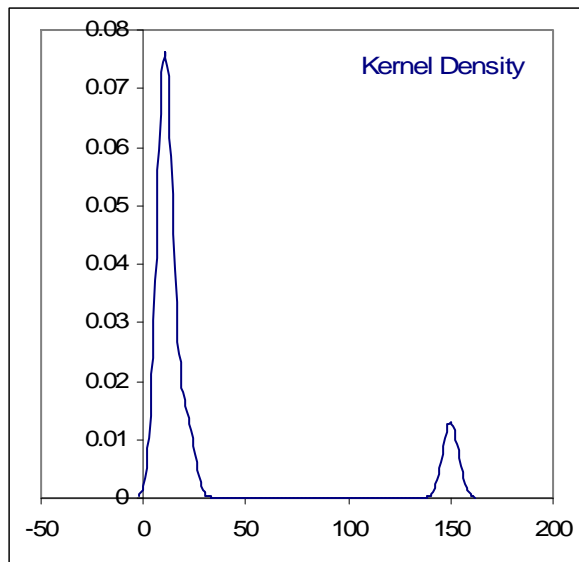
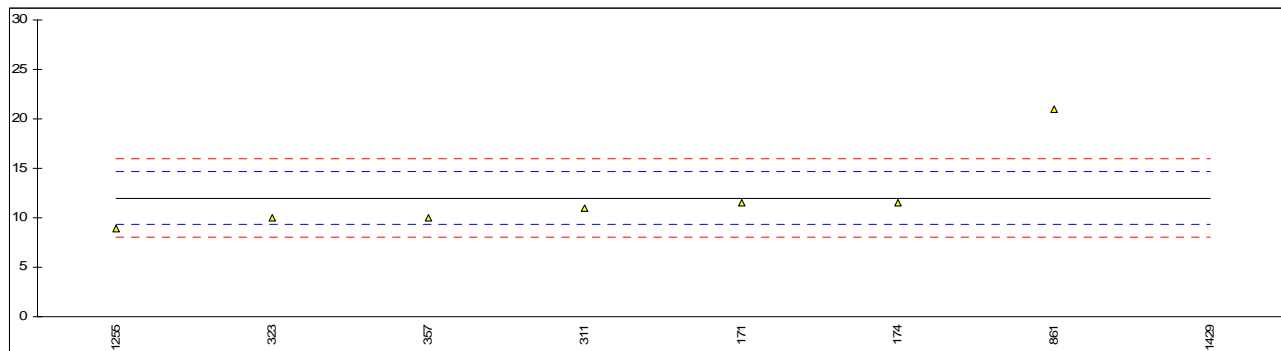
lab	method	value	mark	z(targ)	remarks
171	D5501	6.9		-4.45	
174	D5501	<10		<-2.30	
311	INH-082	13		-0.22	
323	INH-060	<5		<-5.77	False negative?
342		-----		-----	
357	INH-012	12		-0.91	
463		-----		-----	
522		-----		-----	
613		-----		-----	
861	INH-041	24		7.40	
913		-----		-----	
1016		-----		-----	
1255	GC	10.7		-1.82	
1263	D5501	<5		<-5.77	False negative?
1429		50	CG(0.05)	25.41	First reported <10
1826	in house	<100		-----	
	normality	unknown			
	n	5			
	outliers	1			
	mean (n)	13.32			
	st.dev. (n)	6.403			
	R(calc.)	17.93			
	R(Horwitz)	4.04			



Determination of Methyl-Ethyl-Ketone on sample #0997; results in mg/kg.

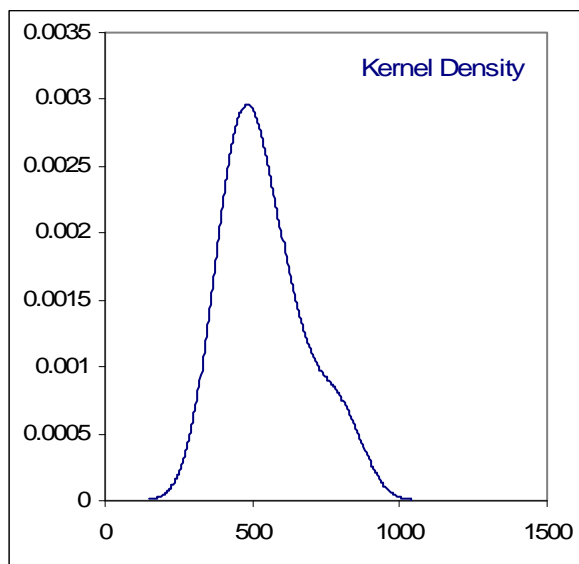
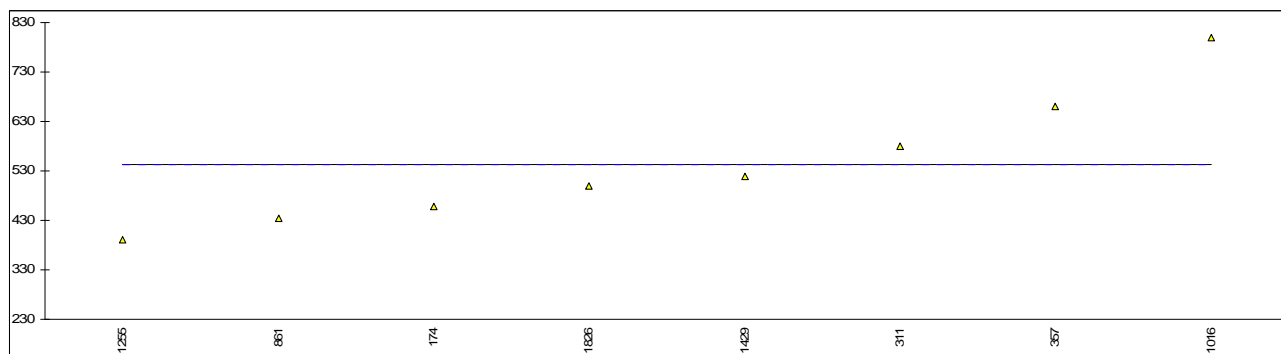
lab	method	value	mark	z(targ)	remarks
171	D5501	11.5		-0.37	
174	D5501	11.5		-0.37	
311	INH-082	11		-0.75	
323	INH-060	10		-1.50	
342		----		----	
357	INH-012	10		-1.50	
463		----		----	
522		----		----	
613		----		----	
861	INH-041	21		6.83	
913		----		----	
1016		----		----	
1255	GC	8.9		-2.34	
1263		----		----	
1429		150	CG(0.01)	104.59	First reported 594
1826		----		----	

normality not OK
n 7
outliers 1
mean (n) 11.99
st.dev. (n) 4.085
R(calc.) 11.44
R(Horwitz) 3.69



Determination of Other Impurities on sample #0997; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
171		----		----	
174	D5501	458		----	
311	INH-082	580		----	
323		----		----	
342		----		----	
357	INH-012	660		----	
463		----		----	
522		----		----	
613		----		----	
861	INH-041	434		----	
913		----		----	
1016		800	C	----	First reported 540.4
1255	GC	390.6		----	
1263		----		----	
1429		520		----	
1826	in house	500		----	
normality		OK			
n		8			
outliers		0			
mean (n)		542.8			
st.dev. (n)		134.07			
R(calc.)		375.4			
R(lit.)		unknown			



APPENDIX 2**List of number of participants**

1 laboratory in	AUSTRALIA
1 laboratory in	AUSTRIA
1 laboratory in	BELGIUM
1 laboratory in	FINLAND
1 laboratory in	INDIA
1 laboratory in	MEXICO
1 laboratory in	P.R. of CHINA
1 laboratory in	ROMANIA
1 laboratory in	SPAIN
1 laboratory in	SWEDEN
3 laboratories in	THE NETHERLANDS
2 laboratories in	U.S.A.
1 laboratory in	UNITED KINGDOM

APPENDIX 3

Abbreviations:

C	= final result after checking of first reported suspect result
U	= reported in wrong unit
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
EX	= excluded from calculations
E	= error in calculations
n.a.	= not applicable
W	= withdrawn on request participant
SDS	= Safety Data Sheet

Literature:

- 1 i.i.s. Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, January 2010
- 2 ASTM E178-02
- 3 ASTM E1301-03
- 4 ISO 13528-05
- 5 ISO 5725, parts 1-6, 1994
- 6 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 7 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 8 IP 367/84
- 9 DIN 38402 T41/42
- 10 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 11 J.N. Miller, Analyst, 118, 455, (1993)
- 12 Analytical Methods Committee Technical Brief, No4 January 2001
- 13 The Royal Society of Chemistry 2002, Analyst 2002, 127 page1359-1364, P.J. Lowthian and M. Thompson. (see <http://www.rsc.org/suppdata/an/b2/b205600n/>)