Results of Proficiency Test
Acetone
September 2011

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 1999, the Institute for Interlaboratory Studies organizes on regular basis a proficiency test for the analysis of Acetone. As part of the annual proficiency test program of 2011/2012, the Institute decided to continue this proficiency test on Acetone. In this interlaboratory study, 21 laboratories from 15 different countries have participated. See appendix 2 for the number of participants per country.
In this report, the results of the Acetone PT are presented and discussed.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. The analyses for fit-for-use and homogeneity determination were subcontracted. It was decided to send one sample Acetone (1*1L bottle, labelled #11062) 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, has implemented a quality system based on ISO guide 43, ILAC-G13:2007 and ISO 17043:2010. This ensures 100% confidentiality of participant's data. Also, customer's satisfaction is measured on a regular basis by sending out questionnaires

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2), which can be downloaded from www.iisnl.com.

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

The necessary bulk material of Acetone was purchased from a local Chemical supplier. The approx. 35 litre bulk sample was homogenised in a pre-cleaned drum. After homogenisation, the bulk sample was divided over 32 brown glass bottles of one litre (labelled #11062). The homogeneity of the subsamples #11062 was checked by

determination of Water content in accordance with ASTM D1364:07 and Density in accordance with ASTM D4052:09 on 4 stratified randomly selected samples.

	Density @20ºC in kg/L	Water in %M/M
sample #11062-1	0.79040	0.1860
sample #11062-2	0.79040	0.1860
sample #11062-3	0.79040	0.1860
sample #11062-4	0.79040	0.1870

Table 1: homogeneity test results of subsample #11062

From the above test results, the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density @20ºC in kg/L	Water in %M/M
r (observed)	0.00000	0.0014
reference method	D 4052:09	D 1364:07
0.3 x R (ref. method)	0.00015	0.0081

Table 2: repeatabilities of subsample #11062

The repeatability of the test results for Density and for Water on sample #11062 were in full agreement with the repeatability as required by the respective standards. Therefore, homogeneity of subsample #11062 was assumed.

One sample of acetone (a bottle of 1L , labelled #11062) was sent to each of the participating laboratories on August 24, 2011.

2.5 STABILITY OF THE SAMPLES

The stability of the Acetone, packed in the brown 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: Acidity, Aldehydes, Appearance, Chloride as Cl, Colour as Pt/Co, Density @ 20 ^{o}C, Distillation (IBP, MBP and DP), Water Miscibility, Nonvolatile Matter, Purity “as is”, Purity on dry basis, Diacetonalcohol, Mesityloxide, methanol, Permanganate Time Test @ 25 ^{o}C, Refractive Index @ 20 ^{o}C and Water on sample #11062 in accordance with specification ASTM D329:07e1.
To get comparable results, a detailed report form on which the units and the standard methods were printed, was sent together with each set of 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 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

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

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. In case a data set does not have a normal distribution, the (results of the) statistical evaluation 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 and by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test and by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of the averages and the 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 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 is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; no.14 and 15).

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, 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 target reproducibility (preferably taken from a standardized test method) by division with 2.8.
The z-scores were calculated in accordance with:

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

The $z_{(target)}$ scores are listed in the result tables in appendix 1.

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

Absolute values for z<2 are very common and absolute values for z>3 are very rare. Therefore, the usual interpretation of z-scores is as follows:

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

4 Evaluation

In this proficiency test, problems were encountered with the dispatch of the samples to the Brazil and India, due to custom clearance problems. From the total of 21 participants, only three participants did not report any result. In total 198 numerical results were reported. Observed were 3 outlying results, which is 1.5%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

Not all original data sets proved to have a normal distribution. For Colour, Density, IBP and MBP (automated and manual mode) non-Gaussian distributions were found and therefore the statistical evaluation for these determinations should be used with care. In this section, the results are discussed per test.

Acidity: No analytical problems were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D1613:06.

Aldehydes: No conclusions could be drawn. Only two participants reported a result. One laboratory reported a numerical result in accordance an in house test method. The other laboratory used test method ASTM D329:07, that describes a pass/fail test.

Appearance: No analytical problems were observed. All labs agreed about the appearance of sample #11062, which is bright, clear and free of suspended matter. The uniformity of reporting can be improved.
A standardized method is available for Appearance since 2009, being ASTM E2680. According this method the appearance should be reported as 'pass' (or 'fail').

Chloride: It is hard to draw conclusions, because the chloride concentration is near or below the detection limit and only three participants reported a numerical result.

Colour: This determination was not problematic. The calculated reproducibility is in good agreement with the requirements of ASTM D1209:05e1.

Density @ 20°C: This determination was not problematic. The calculated reproducibility is in good agreement with the requirements of ASTM D4052:09.

Distillation: This determination was not problematic for both the manual and automated mode. All calculated reproducibilities for both operating modes (for IBP, MBP and DP as well) were in agreement with the requirements of ASTM D1078:05. From the reported results of the Mid Boiling Point (automated), it was concluded that all participating laboratories may have corrected the results properly for barometric pressure and thermometer accuracy as described in ASTM D1078.

Water Miscibility: This determination was not problematic. All laboratories reported this test pass(es). The analytical method described in ASTM D1722:09 is a pass/fail test.

NVM: This determination was not problematic. The calculated reproducibility is in good agreement with the requirements of ASTM D1353:09.

Purity as is: For this determination only one statistical outlier was noticed. The calculated reproducibility is large in comparison with the 2009 PT “iis09C08” (0.049 vs 0.025).

Purity on DB: No statistical outliers were observed. The calculated reproducibility is somewhat large in comparison with the calculated reproducibility of the 2009 PT “iis09C08” (0.010 vs 0.008).

Diacetonalcohol: This determination may have been problematic. The calculated reproducibility is not in agreement with the estimated requirements, calculated using the strict Horwitz equation.

Mesityloxide: It is hard to draw conclusions, because only three participants reported a numerical result. All reporting laboratories agreed on a value below 10 mg/kg.

Methanol: This determination was problematic for only one laboratory. Only one statistical outlier was observed. The calculated reproducibility, after rejection of the statistical outlier, is in full agreement with the estimated requirements, calculated using the strict Horwitz equation.

PTT: This determination was not problematic. Only one statistical outlier was observed. All participants agreed on a result far above 30 minutes. When a statistical evaluation is performed on the actually reported results, the calculated reproducibility is not in agreement with the requirements of ASTM D1363:11. However, as it is unknown whether a Permanganate Time Test result of >100 minutes is in the applicability range, it is therefore difficult to draw any conclusions. No z-scores were calculated.

Refractive index: This test was not problematic. The calculated reproducibility is in full agreement with the requirements of ASTM D1218:07.

Water: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D1364:07.

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 standards) are compared in the next tables.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acidity as acetic acid	%M/M	17	0.0012	0.0012	0.0014
Aldehydes	---		n.a.	n.a.	n.a.
Appearance	---	17	pass	n.a.	n.a.
Chloride as Cl	mg/kg	3	0.08	n.a.	n.a.
Colour Pt/Co	---	13	3.0	2.8	7.0
Density @ 20 °C	kg/L	16	0.7904	0.0002	0.0005
Initial Boiling Point (automated)	°C	7	55.97	0.27	0.87
Mid Boiling Point (automated)	°C	7	56.09	0.11	0.38
Dry Point (automated)	°C	7	56.23	0.21	0.60
Initial Boiling Point (manual)	°C	7	55.94	0.15	0.60
Mid Boiling Point (manual)	°C	7	56.09	0.11	0.36
Dry Point (manual)	°C	7	56.39	0.34	0.73
Miscibility with water	---	14	pass	n.a.	n.a.
Nonvolatile Matter	mg/100 mL	10	0.73	1.79	2.40
Purity as is	%M/M	12	99.7831	0.0485	unknown
Purity on dry basis	%M/M	15	99.9794	0.0099	unknown
Diacetonalcohol	mg/kg	11	26.72	13.54	7.30
Mesityloxide	mg/kg	3	0.23	n.a.	n.a.
Methanol	mg/kg	13	165.6	31.9	34.4
Permanganate Time Test	min	11	274	159	(69)
Refractive Index	---	11	1.35871	0.00049	0.00050
Water	mg/kg	17	1936	375	270

Table 3: Reproducibilities for sample #11062

Without further statistical calculations, it can be concluded that for many tests there is a good compliance of the group of participating laboratories with the relevant standards. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF SEPTEMBER 2011 WITH PREVIOUS PT'S

	September 2011	September 2009	September 2007	October 2006
Number of reporting labs	18	19	18	21
Number of results reported	198	216	157	178
Statistical outliers	3	7	8	12
Percentage outliers	1.5%	3.2%	5.1%	6.7%

Table 4: comparison of summary data 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	September 2011	September 2009	September 2007	October 2006
Acidity as acetic acid	+	++	++	++
Chloride as Cl	n.e.	n.e.	n.e.	n.e.
Colour Pt/Co	++	++	++	++
Density @ 20 ^{o}C	++	++	++	++
Distillation (automated)	++	++	++	++
Distillation (manual)	++	++	++	++
Nonvolatile Matter	++	++	++	++
Diacetonalcohol	--	--	--	--
Mesityloxide	n.e.	+	n.e.	n.e.
Methanol	+	++	n.e.	n.e.
Permanganate Time Test	n.e.	n.e.	n.e.	--
Refractive Index	+/-	-	--	-
Water	--	++	++	++

Table 6: comparison determinations against the standard

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

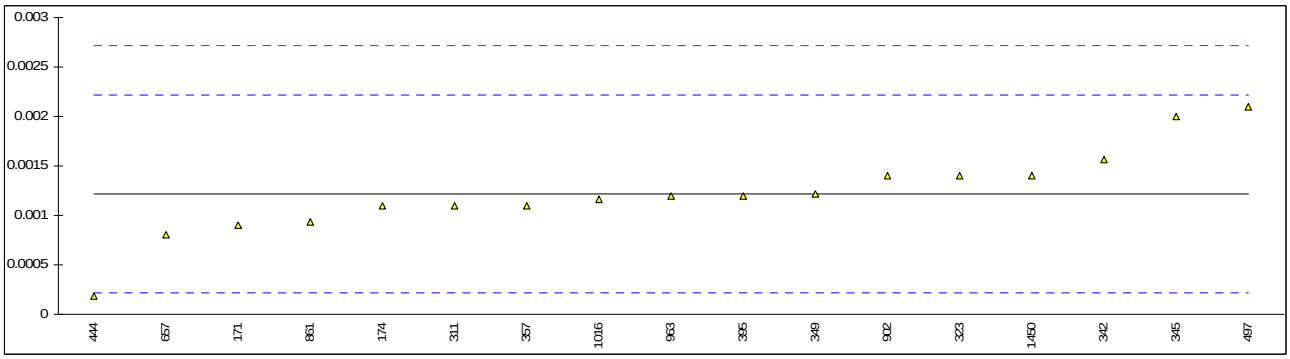
- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- - : group performed worse than the standard
- -- : group performed much worse than the standard
- n.e.: not evaluated

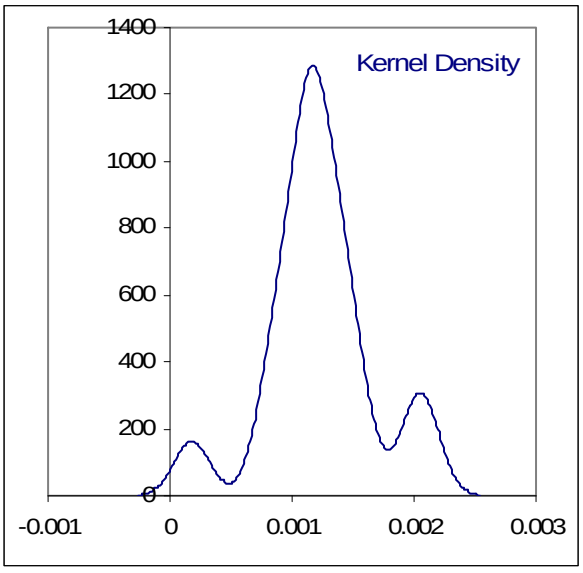
APPENDIX 1

Determination of Acidity on sample #11062; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D1613	0.0009		-0.64	
174	D1613	0.0011		-0.24	
311	D1613	0.0011	C	-0.24	First reported 0.011
323	D1613	0.0014		0.36	
342	D1613	0.00157		0.70	
345	D1613	0.002		1.56	
349	D1613	0.00122		0.00	
357	D1613	0.0011		-0.24	
395	D1613	0.0012		-0.04	
444	D1613	0.00018		-2.08	
497	D1613	0.0021		1.76	
551		-----		-----	
657	D1613	0.0008		-0.84	
861	D1613	0.00094		-0.56	
902	D1613	0.0014		0.36	
912		-----		-----	
913		-----		-----	
963	D1613	0.0012		-0.04	
1016	D1613	0.001167	C	-0.11	First reported 11.67
1438		-----		-----	
1450	D1613	0.0014		0.36	

normality	OK
n	17
outliers	0
mean (n)	0.00122
st.dev. (n)	0.000439
R(calc.)	0.00123
R(D1613:06)	0.00140





Determination of Aldehydes on sample #11062;

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174		-----		-----	
311		-----		-----	
323		-----		-----	
342		-----		-----	
345	INH-4714	32.2		-----	
349		-----		-----	
357	D329	pass		-----	
395		-----		-----	
444		-----		-----	
497		-----		-----	
551		-----		-----	
657		-----		-----	
861		-----		-----	
902		-----		-----	
912		-----		-----	
913		-----		-----	
963		-----		-----	
1016		-----		-----	
1438		-----		-----	
1450		-----		-----	
	normality	n.a.			
	n	1			
	outliers	0			
	mean (n)	n.a.			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D329:07e1)	n.a.			

Determination of Appearance on sample #11062;

lab	method	value	mark	z(targ)	remarks
171	E2680	C&F		-----	
174	E2680	pass		-----	
311	INH-402	C&F		-----	
323	E2680	CFFSIM		-----	
342	E2680	pass		-----	
345	visuel	pass		-----	
349	E2680	pass		-----	
357	E2680	pass		-----	
395	E2680	CFSM		-----	
444	E2680	pass		-----	
497	visual	B&C		-----	
551		-----		-----	
657	E2680	pass		-----	
861	E2680	B&C		-----	
902	E2680	pass		-----	
912		-----		-----	
913		-----		-----	
963	E2680	pass		-----	
1016	In house	pass		-----	
1438		-----		-----	
1450	E2680	B&C		-----	
	normality	n.a.			
	n	17			
	outliers	n.a.			
	mean (n)	pass			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(E2680:09)	n.a.			

Abbreviations:

B&C:	bright and clear
CFFSIM:	clear and free from matter in suspension
C&F:	clear and free
CFSM:	clear from suspended matter

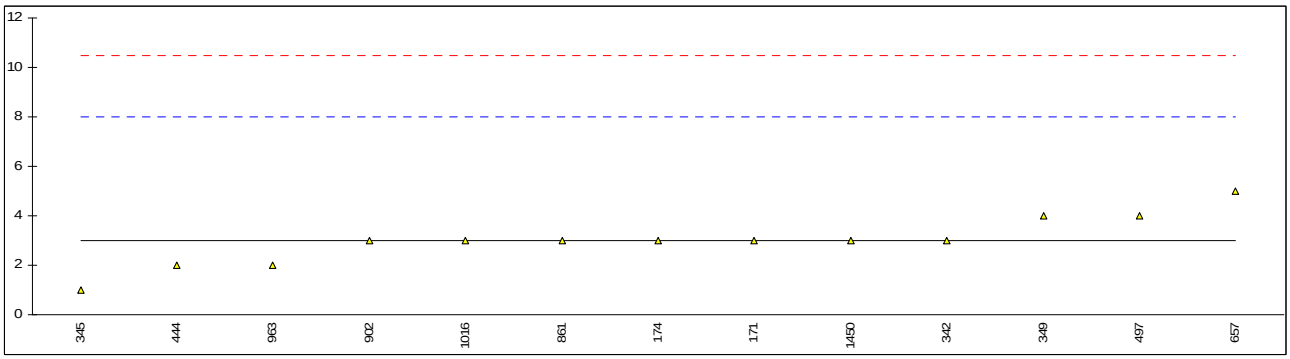
Determination of Chloride as Cl on sample #11062; results in mg/kg

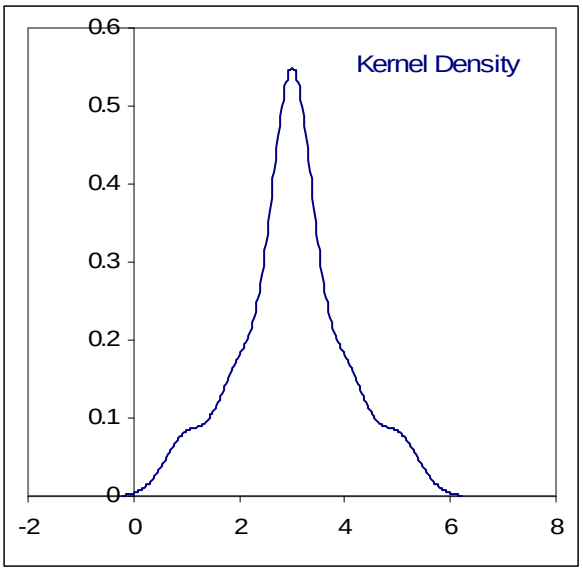
lab	method	value	mark	z(targ)	remarks
171	Titrimetric	<0.1		-----	
174	E2469	0.16		-----	
311	INH158	<0.2		-----	
323	IMPA002	<0.25		-----	
342		-----		-----	
345		-----		-----	
349		-----		-----	
357	IMPA002	<0.5		-----	
395		-----		-----	
444		-----		-----	
497	IMPA002	0.04		-----	
551		-----		-----	
657	INH 0055	<0.5		-----	
861		-----		-----	
902		-----		-----	
912		-----		-----	
913		-----		-----	
963	INH-turb	0.03		-----	
1016		-----		-----	
1438		-----		-----	
1450		-----		-----	
	normality	n.a.			
	n	3			
	outliers	0			
	mean (n)	0.077			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(Horwitz)	n.a.			

Determination of Colour as Pt/Co on sample #11062;

lab	method	value	mark	z(targ)	remarks
171	D1209	3		0.00	
174	D1209	3.0		0.00	
311	D1209	<5		-----	
323	D1209	<5		-----	
342	D5386	3		0.00	
345	D1209	1		-0.80	
349	D1209	4		0.40	
357	D1209	<5		-----	
395	D1209	<5		-----	
444	D5386	2		-0.40	
497	D1209	4		0.40	
551		-----		-----	
657	D1209	5		0.80	
861	D1209	3		0.00	
902	D5386	3		0.00	
912		-----		-----	
913		-----		-----	
963	D1209	2		-0.40	
1016	D1209	3	C	0.00	First reported 8
1438		-----		-----	
1450	D1209	3		0.00	

normality	not OK
n	13
outliers	0
mean (n)	3.0
st.dev. (n)	1.00
R(calc.)	2.8
R(D1209:05e1)	7.0

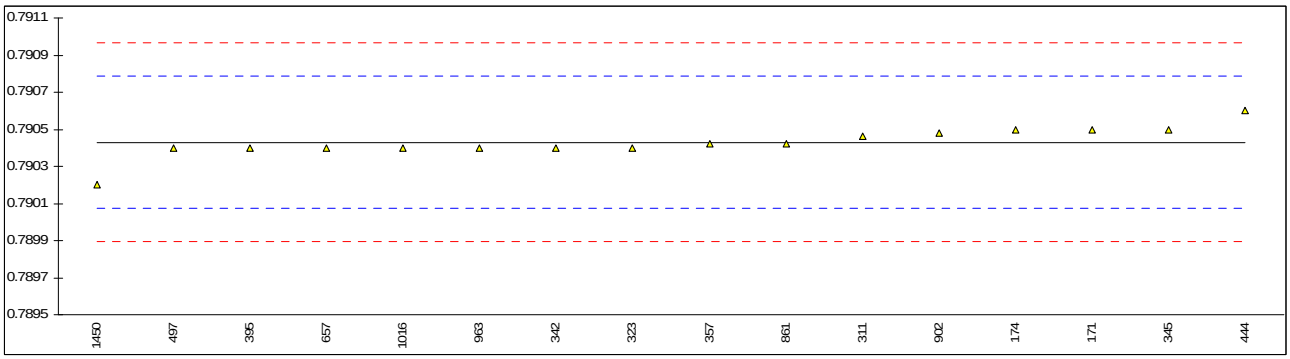


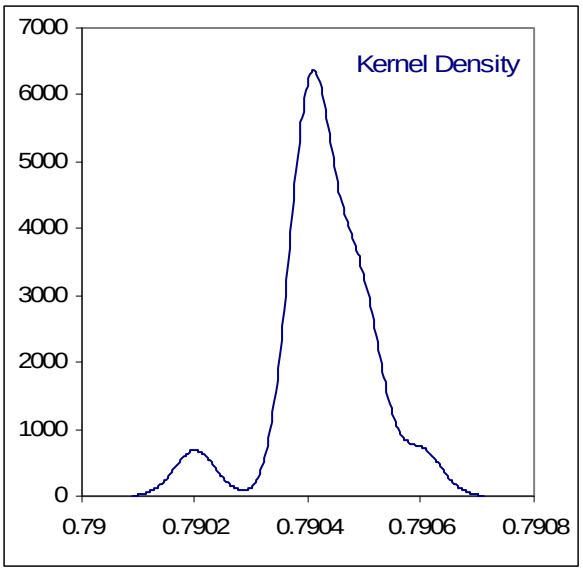


Determination of Density @ 20°C on sample #11062; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.7905		0.39	
174	D4052	0.7905		0.39	
311	D4052	0.79046		0.17	
323	D4052	0.7904		-0.17	
342	D4052	0.7904		-0.17	
345	D4052	0.7905		0.39	
349		-----		-----	
357	D4052	0.79042		-0.06	
395	D4052	0.7904		-0.17	
444	D4052	0.7906	C	0.95	First reported 0.7920
497	D4052	0.7904		-0.17	
551		-----		-----	
657	D4052	0.7904		-0.17	
861	D4052	0.79042		-0.06	
902	D4052	0.79048		0.28	
912		-----		-----	
913		-----		-----	
963	D4052	0.7904		-0.17	
1016	D4052	0.7904	C	-0.17	First reported 0.7942
1438		-----		-----	
1450	D4052	0.7902		-1.29	

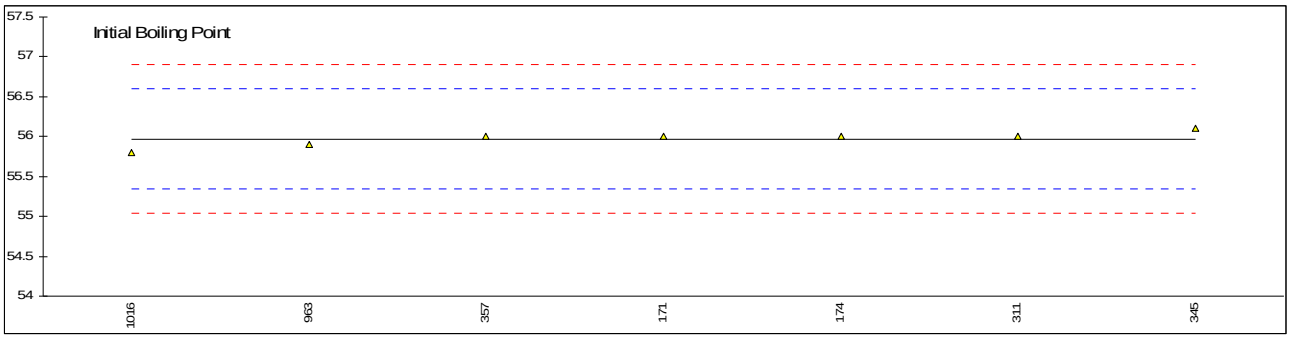
normality	not OK
n	16
outliers	0
mean (n)	0.79043
st.dev. (n)	0.000084
R(calc.)	0.00024
R(D4052:09)	0.00050

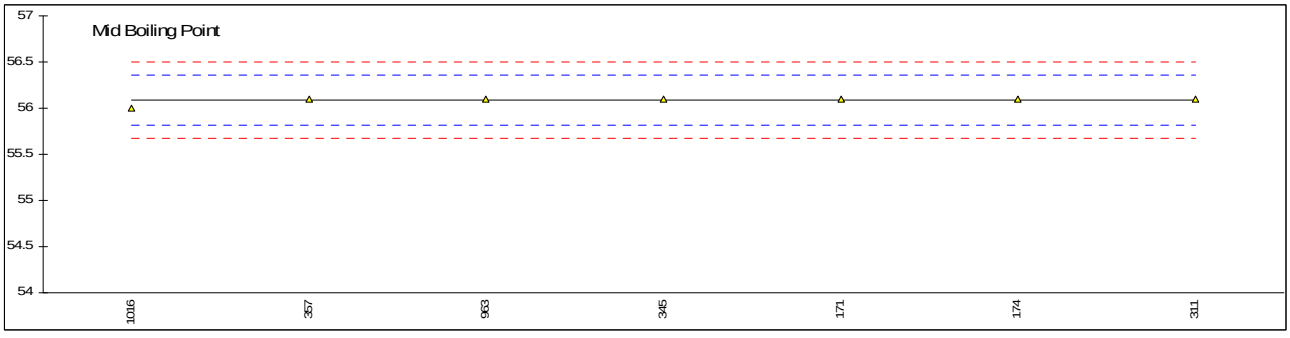


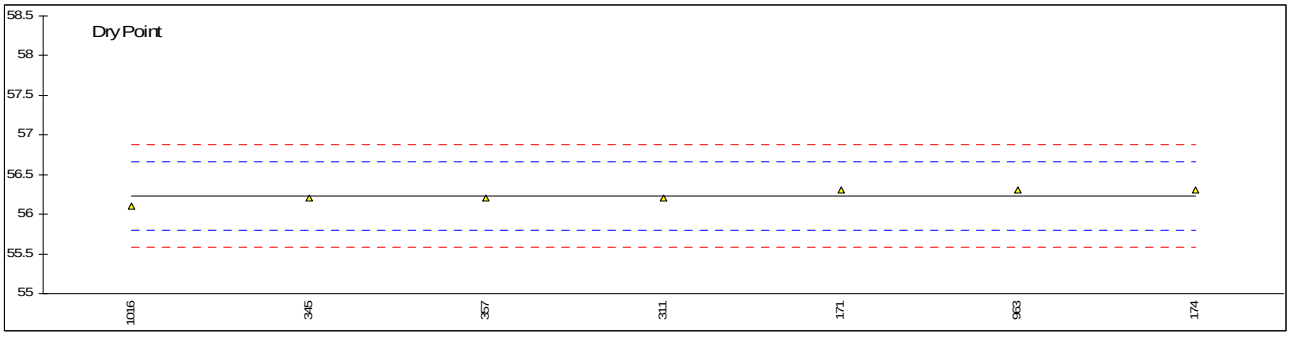


Determination of IBP, MBP and DP (automated) @ 760 mmHg on sample #11062; results in °C

lab	method	IBP	mark	z(targ)	MBP	mark	z(targ)	DP	mark	z(targ)	remarks
171	D1078	56.0		0.09	56.1		0.10	56.3		0.33	
174	D1078	56.0		0.09	56.1		0.10	56.3		0.33	
311	D1078	56.0		0.09	56.1		0.10	56.2		-0.13	
323		-----		-----	-----		-----	-----		-----	
342		-----		-----	-----		-----	-----		-----	
345	D1078	56.1		0.41	56.1		0.10	56.2		-0.13	
349		-----		-----	-----		-----	-----		-----	
357	D1078	56.0		0.09	56.1		0.10	56.2		-0.13	
395		-----		-----	-----		-----	-----		-----	
444		-----		-----	-----		-----	-----		-----	
497		-----		-----	-----		-----	-----		-----	
551		-----		-----	-----		-----	-----		-----	
657		-----		-----	-----		-----	-----		-----	
861		-----		-----	-----		-----	-----		-----	
902		-----		-----	-----		-----	-----		-----	
912		-----		-----	-----		-----	-----		-----	
913		-----		-----	-----		-----	-----		-----	
963	D1078	55.9		-0.23	56.1		0.10	56.3		0.33	
1016	D1078	55.8		-0.55	56.0		-0.63	56.1		-0.60	
1438		-----		-----	-----		-----	-----		-----	
1450		-----		-----	-----		-----	-----		-----	
	normality	not OK			not OK			OK			
	n	7			7			7			
	outliers	0			0			0			
	mean (n)	55.97			56.09			56.23			
	st.dev. (n)	0.095			0.038			0.076			
	R(calc.)	0.27			0.11			0.21			
	R(D1078:05-A)	0.87			0.38			0.60			

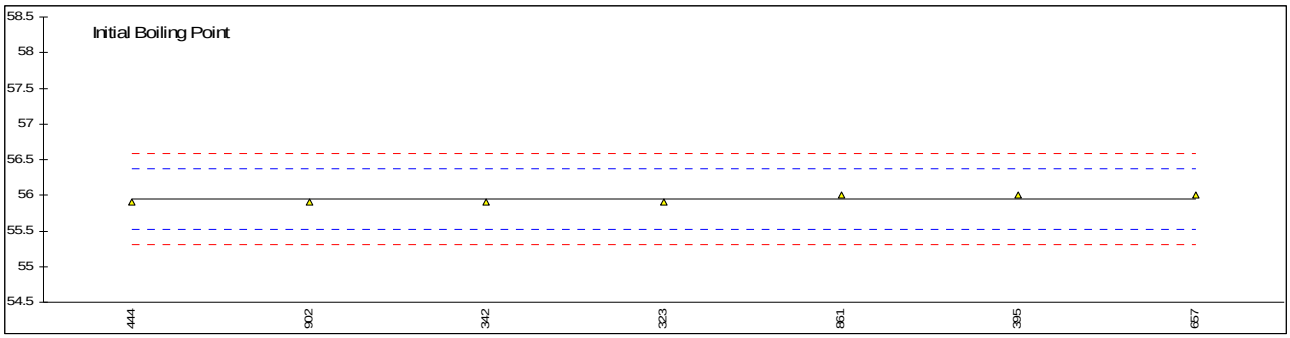


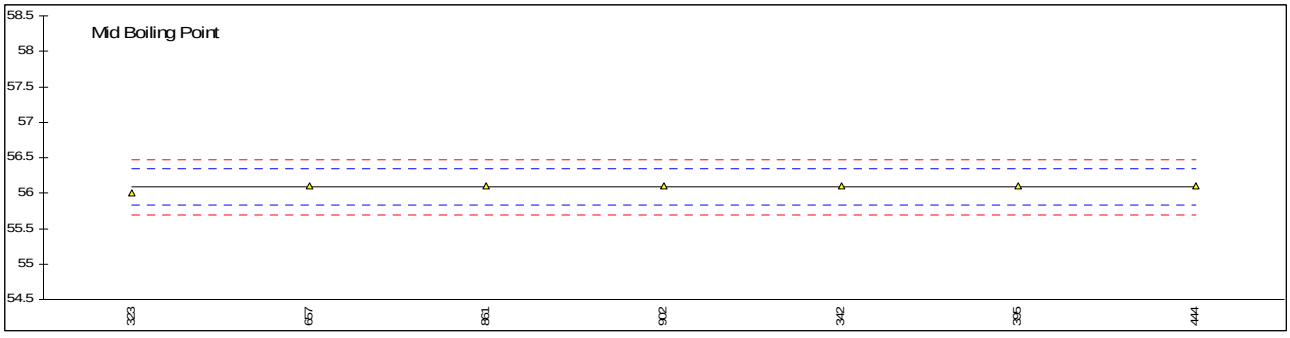


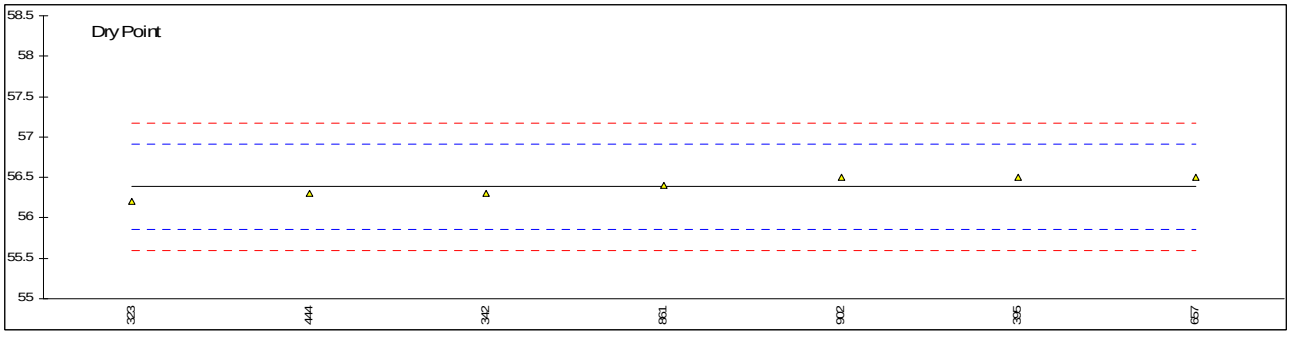


Determination of IBP, MBP and DP (manual) @ 760 mmHg on sample #11062; results in °C

lab	method	IBP	mark	z(targ)	MBP	mark	z(targ)	DP	mark	z(targ)	remarks
171		-----		-----	-----		-----	-----		-----	
174		-----		-----	-----		-----	-----		-----	
311		-----		-----	-----		-----	-----		-----	
323	D1078	55.9		-0.20	56.0		-0.66	56.2		-0.71	
342	D1078	55.9		-0.20	56.1		0.11	56.3		-0.33	
345		-----		-----	-----		-----	-----		-----	
349		-----		-----	-----		-----	-----		-----	
357		-----		-----	-----		-----	-----		-----	
395	D1078	56.0		0.27	56.1		0.11	56.5		0.44	
444	D1078	55.9		-0.20	56.1		0.11	56.3		-0.33	
497		-----		-----	-----		-----	-----		-----	
551		-----		-----	-----		-----	-----		-----	
657	D1078	56.0		0.27	56.1		0.11	56.5		0.44	
861	D1078	56.0		0.27	56.1		0.11	56.4		0.05	
902	D1078	55.9		-0.20	56.1		0.11	56.5		0.44	
912		-----		-----	-----		-----	-----		-----	
913		-----		-----	-----		-----	-----		-----	
963		-----		-----	-----		-----	-----		-----	
1016		-----		-----	-----		-----	-----		-----	
1438		-----		-----	-----		-----	-----		-----	
1450		-----		-----	-----		-----	-----		-----	
	normality	not OK			not OK			OK			
	n	7			7			7			
	outliers	0			0			0			
	mean (n)	55.94			56.09			56.39			
	st.dev. (n)	0.053			0.038			0.121			
	R(calc.)	0.15			0.11			0.34			
	R(D1078:05-M)	0.60			0.36			0.73			







Determination of Miscibility with Water on sample #11062;

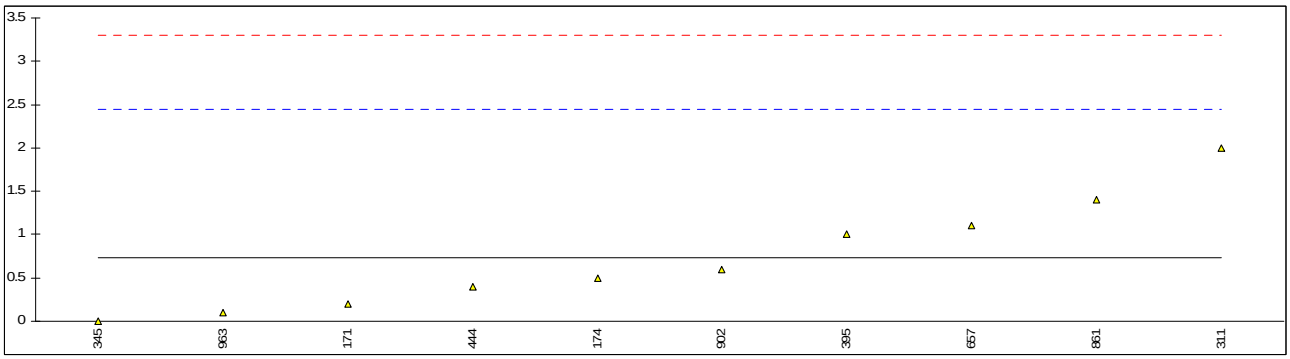
lab	method	value	mark	z(targ)	remarks
171	D1722	pass		-----	
174	D1722	pass		-----	
311	D1722	pass		-----	
323	D1722	pass		-----	
342	D1722	pass		-----	
345	D1722	pass		-----	
349		-----		-----	
357	D1722	pass		-----	
395	D1722	pass		-----	
444		-----		-----	
497	D1722	pass		-----	
551		-----		-----	
657	D1722	pass		-----	
861	D1722	pass		-----	
902	D1722	pass		-----	
912		-----		-----	
913		-----		-----	
963	D1722	pass		-----	
1016	D1722	pass		-----	
1438		-----		-----	
1450		-----		-----	

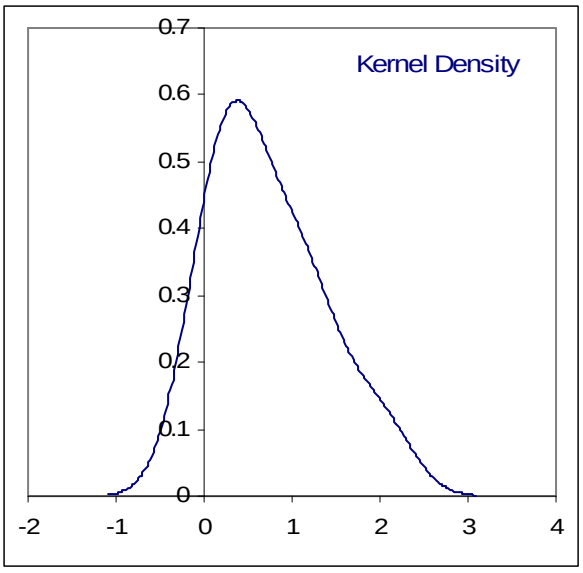
normality	n.a.
n	14
outliers	n.a.
mean (n)	pass
st.dev. (n)	n.a.
R(calc.)	n.a.
R(D1722:09)	n.a.

Determination of Nonvolatile Matter on sample #11062; results in mg/100 mL

lab	method	value	mark	z(targ)	remarks
171	D1353	0.2		-0.62	
174	D1353	0.5		-0.27	
311	D1353	2		1.48	
323	D1353	<1		-----	
342		-----		-----	
345	D1353	0		-0.85	
349		-----		-----	
357	D1353	<1		-----	
395	D1353	1.0		0.31	
444	D1353	0.4		-0.39	
497	D1353	<1		-----	
551		-----		-----	
657	D1353	1.1		0.43	
861	D1353	1.4		0.78	
902	D1353	0.6		-0.15	
912		-----		-----	
913		-----		-----	
963	D1353	0.1		-0.74	
1016	D1353	<0.2		-----	
1438		-----		-----	
1450		-----		-----	

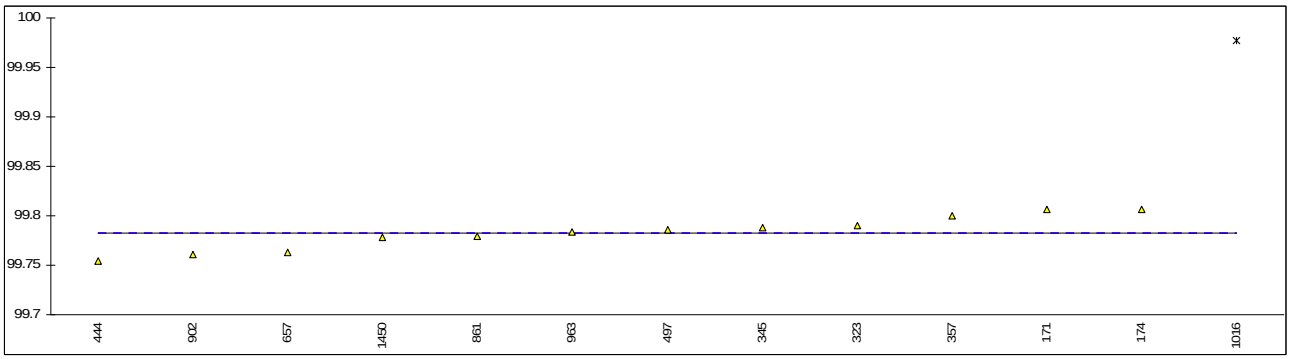
normality	OK
n	10
outliers	0
mean (n)	0.73
st.dev. (n)	0.638
R(calc.)	1.79
R(D1353:09)	2.40

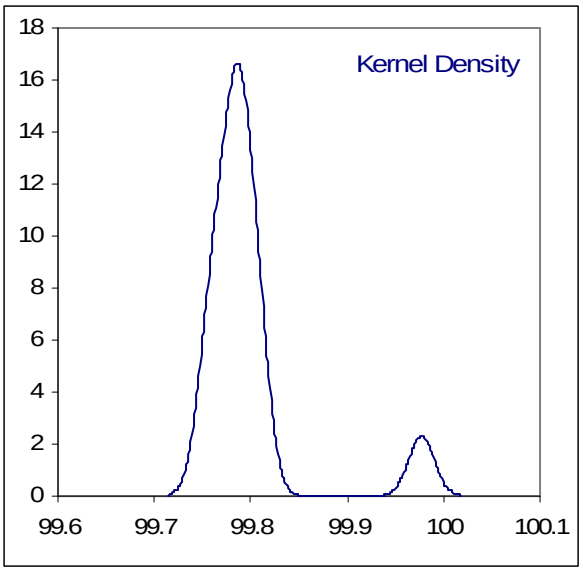




Determination of Purity “as is” on sample #11062, results in %M/M

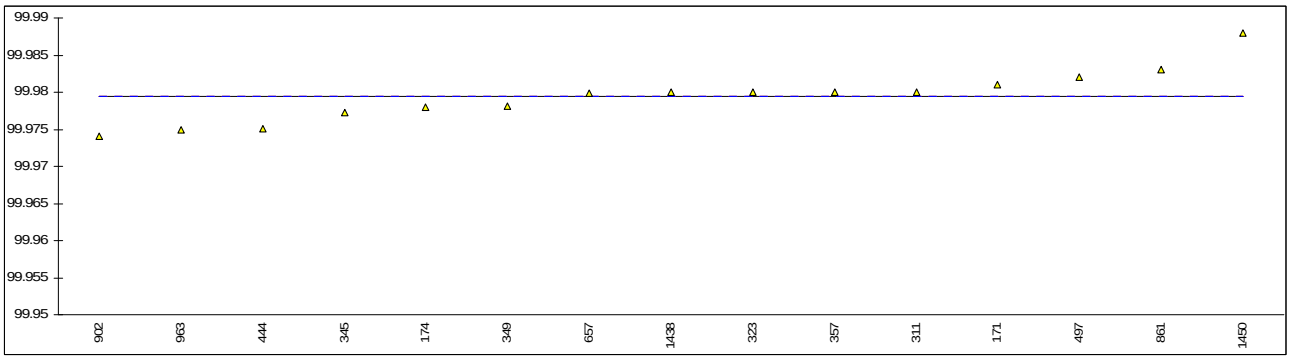
lab	method	value	mark	z(targ)	remarks
171	In house	99.807		-----	
174	D6438	99.807		-----	
311		-----		-----	
323	INH-022	99.79		-----	
342		-----		-----	
345	INH-4714	99.7882		-----	
349	INH-09	-----		-----	
357	INH-054	99.80		-----	
395		-----		-----	
444	INH-001	99.7541		-----	
497		99.786	C	-----	First reported 99.982
551		-----		-----	
657	calc.	99.7626	C	-----	First reported 99.7048
861	INH-001	99.779		-----	
902	INH-125	99.761		-----	
912		-----		-----	
913		-----		-----	
963	In house	99.784		-----	
1016		99.9773	G(0.01)	-----	
1438		-----		-----	
1450	INH-6026	99.778		-----	
	normality	OK			
	n	12			
	outliers	1			
	mean (n)	99.7831			
	st.dev. (n)	0.01733			
	R(calc.)	0.0485			
	R(lit)	unknown			Compare R(iis09C08) = 0.0248

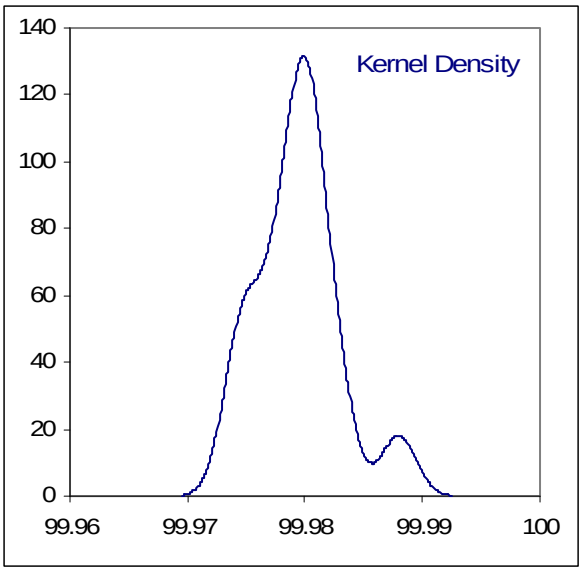




Determination of Purity on Dry Basis on sample #11062, results in %M/M

lab	method	value	mark	z(targ)	Remarks
171	in house	99.981		-----	
174	D6438	99.978		-----	
311	INH-168	99.98		-----	
323	INH-022	99.98		-----	
342		-----		-----	
345	INH-4714	99.9772		-----	
349	INH-09	99.9781		-----	
357	INH-054	99.98		-----	
395		-----		-----	
444	INH-001	99.9751		-----	
497		99.982	C	-----	First reported 99.786
551		-----		-----	
657	INH-009	99.9798		-----	
861	INH-001	99.983		-----	
902	INH-125	99.974	C	-----	First reported 99.955
912		-----		-----	
913		-----		-----	
963	In house	99.975		-----	
1016		-----		-----	
1438	USP34-NF29	99.98		-----	
1450	INH-6324	99.988		-----	
	normality	OK			
	n	15			
	outliers	0			
	mean (n)	99.9794			
	st.dev. (n)	0.00352			
	R(calc.)	0.0099			
	R(lit)	unknown			Compare R(iis09C08) = 0.0080

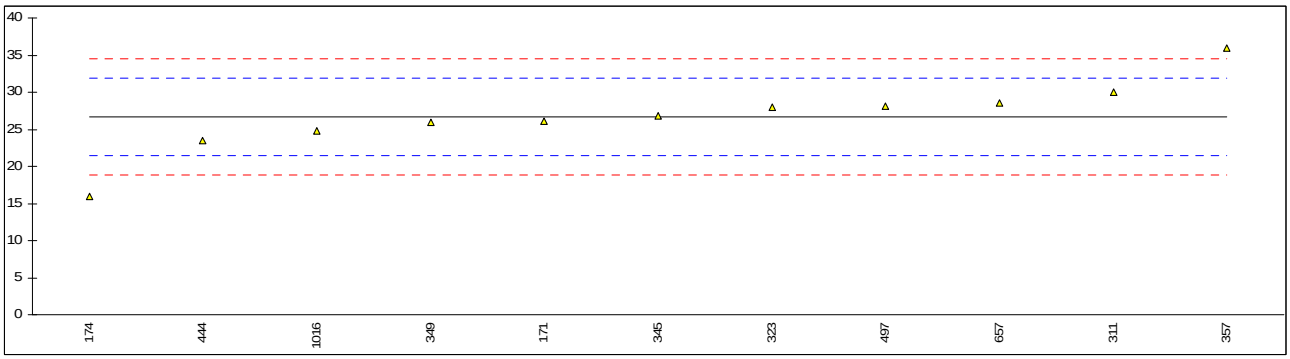


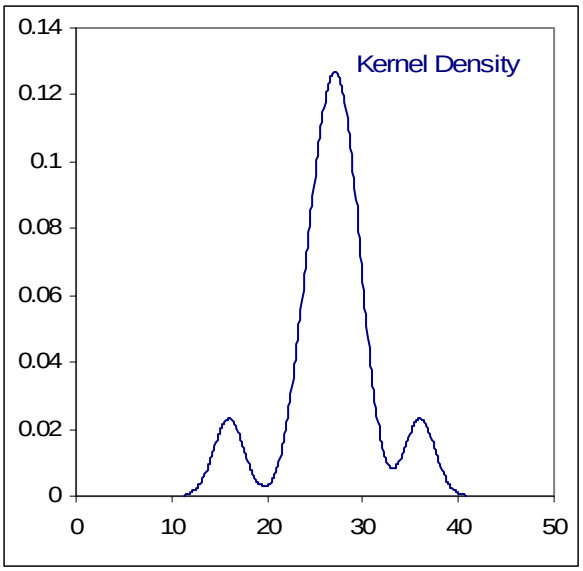


Determination of Diacetonalcohol on sample #11062, results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	in house	26.1		-0.24	
174	D6438	16		-4.11	
311	INH-168	30		1.26	
323	INH-022	28		0.49	
342		-----		-----	
345	INH-4714	26.8		0.03	
349	INH-09	26		-0.28	
357	INH-054	36		3.56	
395		-----		-----	
444	INH-001	23.5		-1.24	
497		28.1	C	0.53	First reported 281
551		-----		-----	
657	INH-009	28.6	C	0.72	First reported 6.5
861		-----		-----	
902		-----		-----	
912		-----		-----	
913		-----		-----	
963		-----		-----	
1016		24.833		-0.72	
1438		-----		-----	
1450		-----		-----	

normality	OK
n	11
outliers	0
mean (n)	26.72
st.dev. (n)	4.834
R(calc.)	13.54
R(Horwitz)	7.30





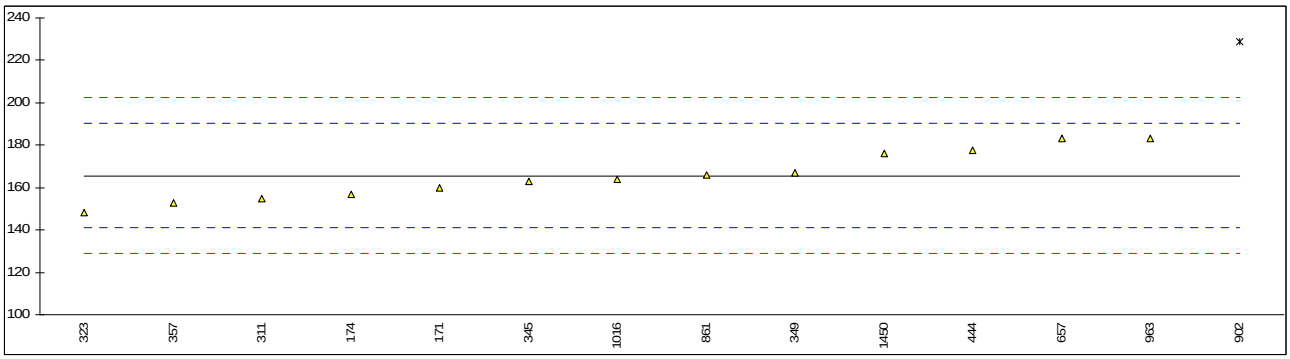
Determination of Mesityloxide on sample #11062, results in mg/kg

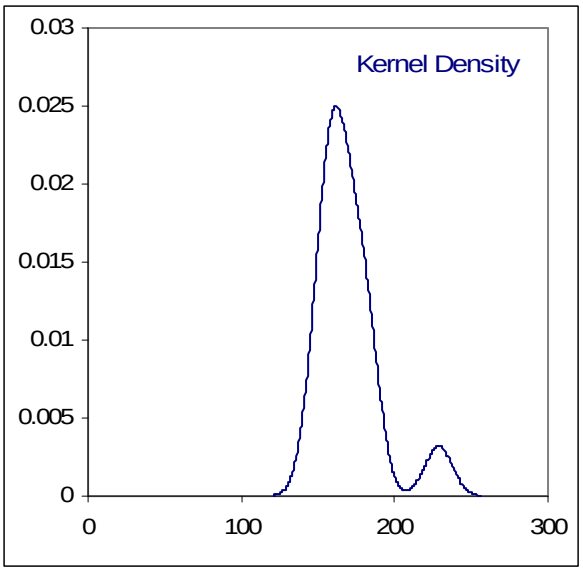
lab	method	value	mark	z(targ)	remarks
171	in house	<5		-----	
174	D6438	<10		-----	
311	INH-168	<5		-----	
323	INH-022	<5		-----	
342		-----		-----	
345	INH-4714	0		-----	
349	INH-09	<1		-----	
357	INH-054	<10		-----	
395		-----		-----	
444	INH-001	0.4		-----	
497		<10		-----	
551		-----		-----	
657	INH-009	n.d.		-----	
861		-----		-----	
902		-----		-----	
912		-----		-----	
913		-----		-----	
963	In house	<1		-----	
1016		0.294		-----	
1438		-----		-----	
1450		-----		-----	
	normality	n.a.			
	n	3			
	outliers	0			
	mean (n)	0.23			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit)	n.a.			

Determination of Methanol on sample #11062, results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	in house	160.1		-0.45	
174	D6438	157		-0.70	
311	INH-168	155		-0.86	
323	INH-022	148		-1.43	
342		-----		-----	
345	INH-4714	163		-0.21	
349	INH-09	167		0.11	
357	INH-054	153		-1.03	
395		-----		-----	
444	INH-001	177.7		0.99	
497		-----		-----	
551		-----		-----	
657	INH-009	183		1.42	
861	INH-001	166		0.03	
902	INH-125	228.9	C,G(0.01)	5.16	First reported 252.4
912		-----		-----	
913		-----		-----	
963	In house	183		1.42	
1016		163.929		-0.14	
1438		-----		-----	
1450	INH-6026	176		0.85	

normality	OK
n	13
outliers	1
mean (n)	165.59
st.dev. (n)	11.390
R(calc.)	31.89
R(Horwitz)	34.38

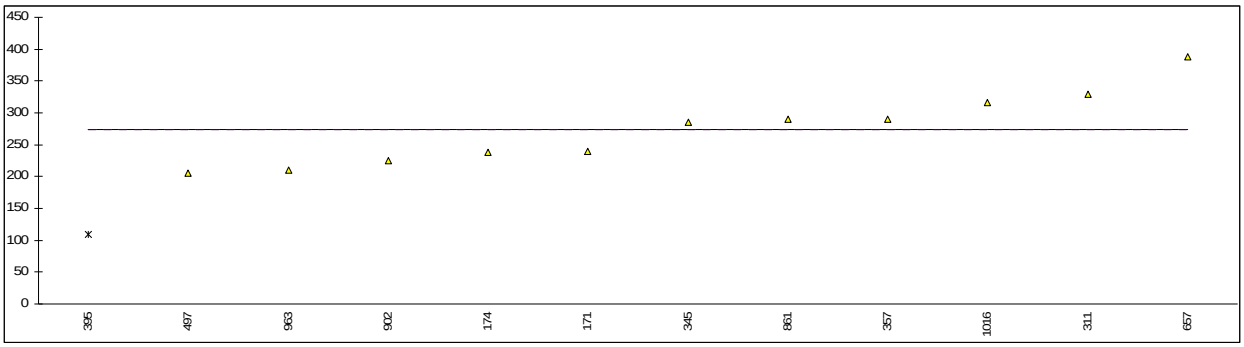


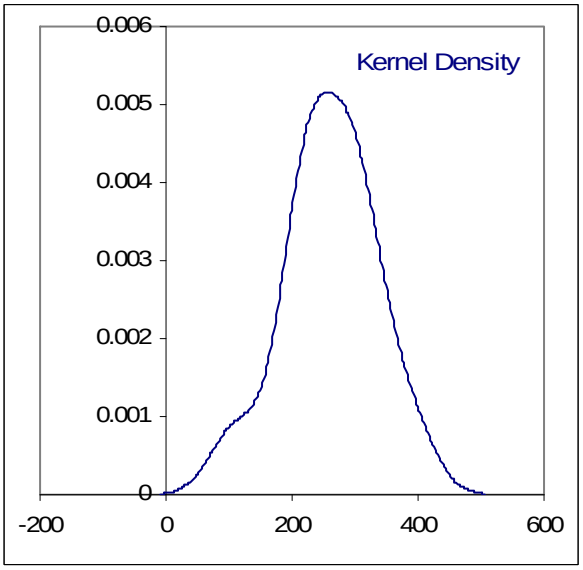


Determination of Permanganate Time Test @ 25°C on sample #11062; results in minutes

lab	method	value	mark	z(targ)	remarks
171	D1363	240		-----	
174	D1363	238		-----	
311	D1363	330		-----	
323	D1363	>150		-----	
342		-----		-----	
345	D1363	285		-----	
349		-----		-----	
357	D1363	290		-----	
395	D1363	110	G(0.05)	-----	False positive result?
444	D1363	>180		-----	
497	D1363	205		-----	
551		-----		-----	
657	D1363	388		-----	
861	D1363	290		-----	
902	D1363	225		-----	
912		-----		-----	
913		-----		-----	
963	D1363	210		-----	
1016	D1363	317		-----	
1438		-----		-----	
1450		-----		-----	

normality	OK
n	11
outliers	1
mean (n)	274
st.dev. (n)	56.9
R(calc.)	159
R(D1363:11)	(69)

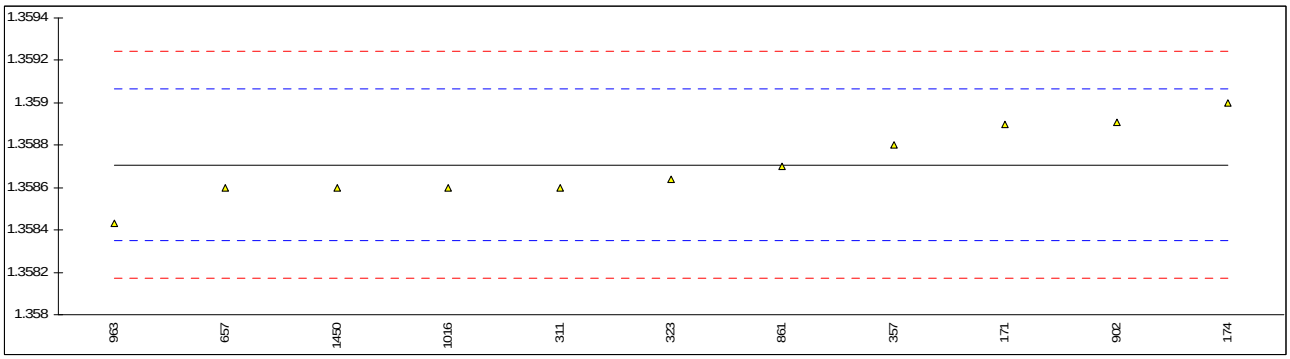


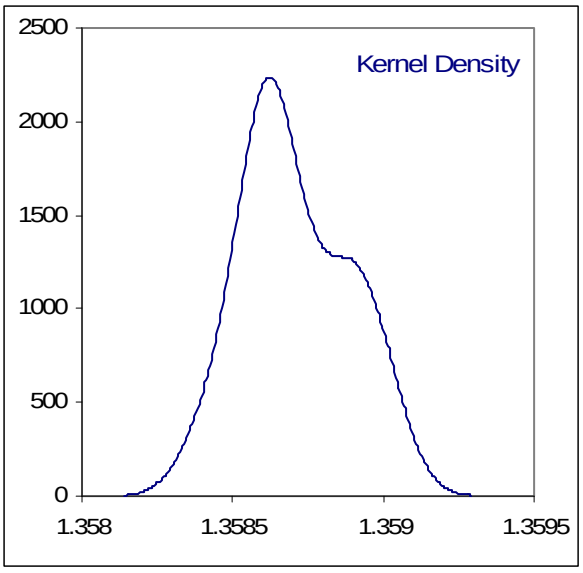


Determination of Refractive Index @ 20 °C on sample #11062;

lab	method	value	mark	z(targ)	remarks
171	D1218	1.3589		1.08	
174	D1218	1.3590		1.64	
311	D1218	1.3586		-0.60	
323	D1218	1.35864		-0.38	
342		-----		-----	
345		-----		-----	
349		-----		-----	
357	D1218	1.3588		0.52	
395		-----		-----	
444		-----		-----	
497		-----		-----	
551		-----		-----	
657	D1218	1.35860		-0.60	
861	D1218	1.3587		-0.04	
902	D1218	1.35891		1.14	
912		-----		-----	
913		-----		-----	
963	D1218	1.35843		-1.55	
1016	D1218	1.3586		-0.60	
1438		-----		-----	
1450	D1218	1.3586		-0.60	

normality	OK
n	11
outliers	0
mean (n)	1.35871
st.dev. (n)	0.000173
R(calc.)	0.00049
R(D1218:07)	0.00050

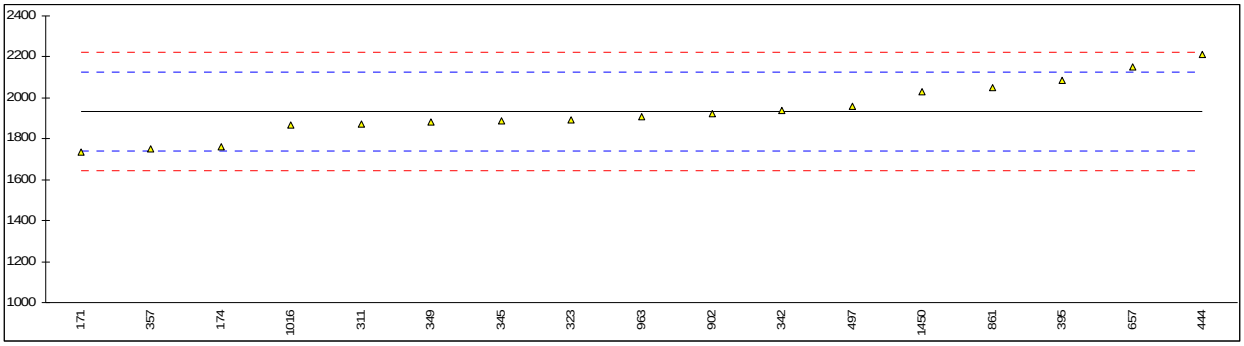


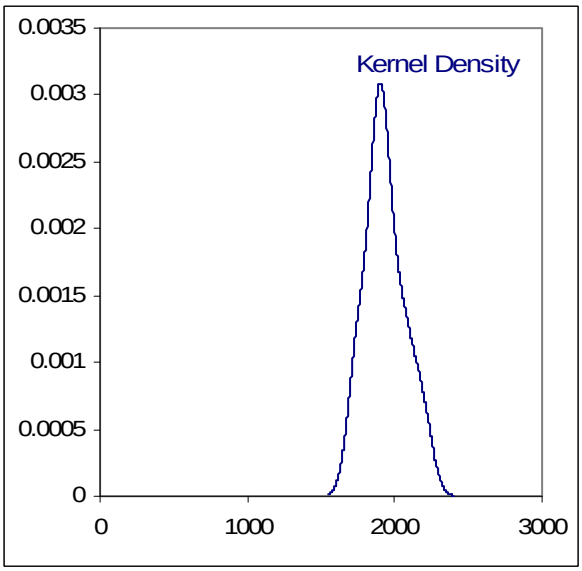


Determination of Water on sample #11062; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D1364	1738		-2.05	
174	D1364	1761		-1.81	
311	D1364	1870		-0.68	
323	D1364	1895		-0.42	
342	E203	1938		0.02	
345	D1364	1890		-0.47	
349	D1364	1881	C	-0.57	First reported 1801
357	D1364	1750		-1.92	
395	D1364	2085		1.55	
444	E203	2210	C	2.85	First reported 0.2210
497	D1364	1960		0.25	
551		-----		-----	
657	E203	2150	C	2.22	First reported 2750
861	E1064	2048		1.17	
902	D1364	1924		-0.12	
912		-----		-----	
913		-----		-----	
963	D1364	1910		-0.27	
1016	D1364	1865.4		-0.73	
1438		-----		-----	
1450	INH-6026	2030		0.98	

normality	OK
n	17
outliers	0
mean (n)	1935.6
st.dev. (n)	133.80
R(calc.)	374.6
R(D1364:07)	270.0





APPENDIX 2

Number of participants per country

1 lab in BELGIUM
1 lab in BRAZIL
1 lab in FINLAND
1 lab in GERMANY
2 labs in INDIA
1 lab in ISRAEL
1 lab in ITALY
2 labs in P.R. of CHINA
1 lab in SAUDI ARABIA
1 lab in SINGAPORE
3 labs in SPAIN
2 labs in THE NETHERLANDS
1 lab in TURKEY
2 labs in U.S.A.
1 lab in UNITED KINGDOM

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
U	= reported in wrong unit
ex	= excluded from calculations
n.a.	= not applicable
n.d.	= not detected

Literature:

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