Results of Proficiency Test
Isopropanol
(Isopropyl alcohol)
November 2011

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 2011/2012 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 D770:11 specifications and a number of additional tests requested by some participants. In this interlaboratory study, 16 laboratories out of 11 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the proficiency test are presented and discussed.

2 SET UP

The Institute for Interlaboratory studies (iis) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. Analyses for fit-for-use and homogeneity testing were subcontracted. It was decided to send one 500 ml bottle with Isopropanol, labelled #11125 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/IEC 17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially 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 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (iis-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 high purity Isopropanol was obtained from a local trader. After homogenisation, the material was divided over 25 amber glass bottles of 500 ml with inner and outer caps and labelled #11125. The homogeneity of subsample #11125 was checked by determination Density in accordance with ASTM D4052:11 and Water in accordance with ASTM D1364:07 on 4 stratified randomly selected samples.

	Density at 20ºC in kg/L	Water content in %M/M
sample #11125-1	0.78509	0.024
sample #11125-2	0.78509	0.023
sample #11125-3	0.78509	0.024
sample #11125-4	0.78509	0.025

Table 1: homogeneity test of subsample #11125

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

	Density at 20ºC in kg/l	Water in %M/M
r (Observed)	0.00000	0.002
reference method	ASTM D4052:11	ASTM D1364:07
0.3 * R (ref method)	0.00015	0.003

Table 2: repeatabilities of subsamples #11125

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 bottle, labelled #11125 was sent on November 02, 2011.

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 20ºC, Distillation (IBP, 50% evaporated & DP), Nonvolatile Matter, Peroxide, Specific Gravity 20/20ºC (= relative density 20ºC), Water, Purity (both “as received” and on dry basis), Ethanol, n-Propanol, n-Butanol, Methylethylketone and Other Impurities on sample #11125. 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

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).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.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them 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 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:

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

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. 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, some problems were encountered with despatch of the samples. The participants in India and U.S.A. received the samples after the final reporting date. The participant in Mexico did not receive the samples at all due to custom clearance problems. From the 13 reporting participants, five participants did report the results after the final reporting date. The 13 reporting laboratories submitted 143 numerical results. Observed were 10 outlying results, which is 7.0%. 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: Density and Water. In these cases the statistical evaluation should be used with due care.

Acidity: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D1613:06.

Appearance: No analytical problems were observed. All labs, except one, agreed about the appearance of sample #11125, which is bright, clear and free of suspended matter. According to ASTM E2680, one should report 'pass" or "fail".

Anorg. Chloride: Only one participant reported a numerical test result. Therefore, no significant conclusions were drawn.

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 D1209:11.

Density @ 20 ^{o}C: No analytical problems were observed. Only one statistical outlier was observed and the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D4052:11.

Specific Gravity @ 20/20 ^{o}C: The determination of the Relative Density @20°C was not problematic. Two statistical outliers were observed and the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D4052:11.

Distillation: This determination was not problematic. No statistical outliers were observed and all three calculated reproducibilities are in good agreement with the requirements of ASTM D1078:11 for the automated and the manual mode. Two laboratories apparently did not correct the results for barometric pressure.

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

Water: This determination was problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D1364:07.

Purity “as received”: Regretfully, the methods used do not provide any reproducibility limit. Therefore no significant conclusions were drawn.
However, the calculated reproducibility is larger then the calculated reproducibility found in the previous proficiency test (iis09C14) of November 2009 (0.087 vs 0.062)

Purity on dry basis: Regretfully, the methods used do not provide any reproducibility limit. Therefore no significant conclusions were drawn.
However, the calculated reproducibility after rejection of the statistical outlier is smaller then the calculated reproducibility found in the previous proficiency test (iis09C14) of November 2009 (0.025 vs 0.052).

Ethanol: No serious analytical problems were observed. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the estimated reproducibility calculated using the Horwitz equation. The low number of reported test results may partly explain the relative large spread.

n-Propanol: This determination is problematic for one laboratory that reported a false negative test result. No statistical outliers were observed. The calculated reproducibility is in full agreement with the estimated reproducibility calculated using the Horwitz equation.

n-Butanol: Only four participants reported a numerical test result. Therefore, no significant conclusions were drawn. One laboratory reported a false positive test result.

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. The low concentration, which is near or below the lower detection limit may explain the large spread.

Other Imp.: This determination seems problematic for only one laboratory. Only one statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the estimated reproducibility calculated using the Horwitz equation (for 4 components).

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 #11125 are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acidity as Acetic Acid	%M/M	10	0.0009	0.0008	0.0014
Appearance		11	Pass	n.a.	n.a.
Anorganic Chloride	mg/kg	4	<0.5	n.a.	n.a.
Colour	Pt/Co	8	5.0	3.3	7.0
Density @ 20ºC	kg/L	12	0.7851	0.0002	0.0005
Specific Gravity @ 20/20ºC		9	0.7865	0.0002	0.0005
Initial Boiling Point	°C	11	82.10	0.27	1.28
50% evaporated	°C	11	82.24	0.26	0.56
Dry Point	°C	11	82.39	0.44	0.88
Nonvolatile Matter	mg/100mL	6	0.64	1.79	2.40
Water	%M/M	10	0.025	0.014	0.010
Purity	%M/M	8	99.911	0.087	n.a.
Purity on dry basis	%M/M	8	99.930	0.025	n.a.
Ethanol	mg/kg	6	9.7	4.2	3.1
n-Propanol	mg/kg	8	99.0	24.8	22.2
n-Butanol	mg/kg	3	10.3	14.4	n.a.
Methylethylketone	mg/kg	5	8.9	6.2	2.9
Other Impurities	mg/kg	6	597.2	170.2	204.4

Table 3: Reproducibilities for sample #11125

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 2011 WITH THE PREVIOUS PTS.

	November 2011	*November 2009*	*November 2007*	*November 2006*
Number of reporting labs	13	15	15	14
Number of results reported	143	165	148	139
Statistical outliers	10	5	10	9
Percentage outliers	7.0%	3.0%	6.8%	6.5%

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	*November 2011*	*November 2009*	*November 2007*	*November 2006*
Acidity as acetic acid	++	++	++	++
Anorganic Chlorides as Cl	n.e.	-	++	++
Colour Pt/Co	++	++	++	++
Density @ 20°C	++	++	++	++
Specific Gravity 20/20 ºC	++	++	++	n.d.
Initial Boiling Point	++	++	++	++
50% evaporated	++	++	n.e.	n.e.
Dry Point	++	++	++	++
Nonvolatile Matter	++	++	++	++
Water	--	++	++	++
Purity	(--)	(+)	(--)	(-)
Purity on dry basis	(++)	(-)	(+)	(-)
Ethanol	-	(--)	n.e.	n.e.
n-Propanol	+/-	+/-	+/-	++
n-Butanol	n.e.	--	++	--
Methylethylketone	--	--	++	--
Other impurities	+	n.e.	n.e.	n.e.

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:

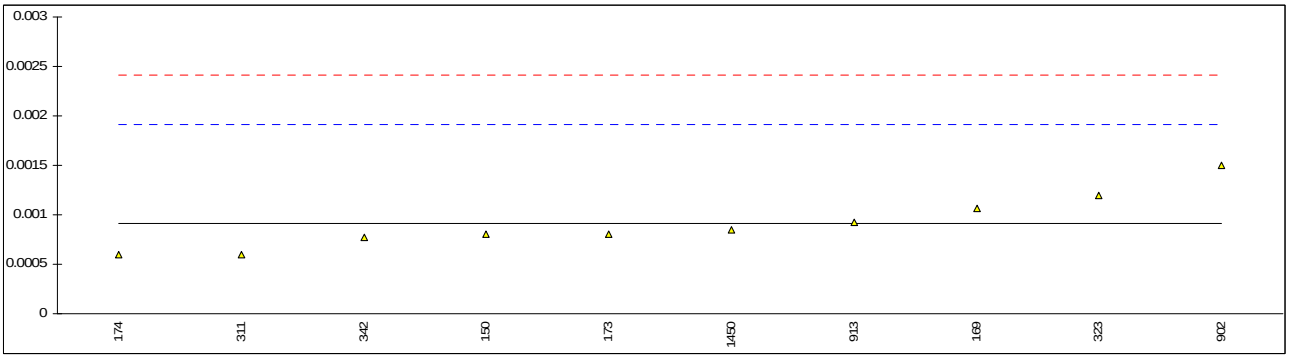
++:	group performed much better than the standard
+ :	group performed better than the standard
+/-:	group performance equals the standard
- :	group performed worse than the standard
-- :	group performed much worse than the standard
n.e.:	not evaluated

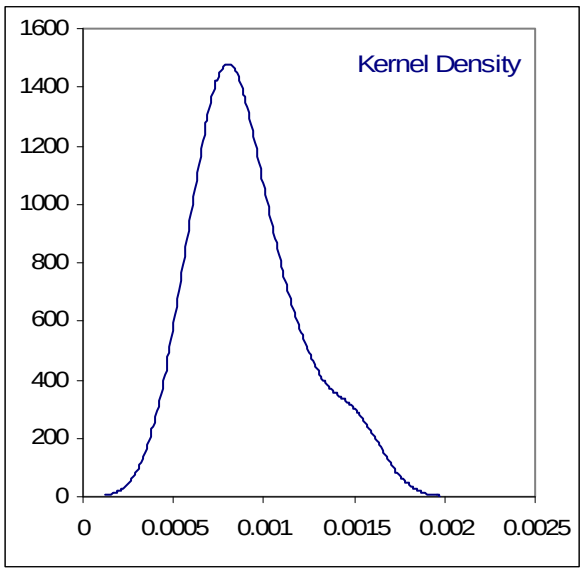
APPENDIX 1

Determination of Acidity as Acetic Acid on sample #11125; results in %M/M.

lab	method	value	mark	z(targ)	remarks
150	D1613	0.0008		-0.22	
169	D1613	0.001068		0.31	
171		-----		-----	
173	D1613	0.0008		-0.22	
174	D1613	0.0006		-0.62	
311	D1613	0.0006		-0.62	
323	D1613	0.0012		0.58	
342	D1613	0.00077		-0.28	
522		-----		-----	
902	D1613	0.0015		1.18	
913	D1613	0.00092		0.02	
1016		-----		-----	
1373		-----		-----	
1429	D1613	<0.0005		<-0.82	
1438		-----		-----	
1450	D1613	0.00085		-0.12	

normality	OK
n	10
outliers	0
mean (n)	0.00091
st.dev. (n)	0.000278
R(calc.)	0.00078
R(D1613:06)	0.00140





Determination of Appearance on sample #11125;

lab	method	value	mark	z(targ)	remarks
150	E2680	pass		-----	
169	E2680	pass		-----	
171		-----		-----	
173	visual	CFFSM		-----	
174	E2680	pass		-----	
311	E2680	pass		-----	
323	E2680	pass		-----	
342		-----		-----	
522		-----		-----	
902	E2680	pass		-----	
913	E2680	CFFSM		-----	
1016	E2680	SM		-----	
1373		-----		-----	
1429	E2680	C&B		-----	
1438		-----		-----	
1450	E2680	pass		-----	

C&B = Clear and Bright
CFFSM = Clear and free from Suspended Matter
SM = Suspended Matter

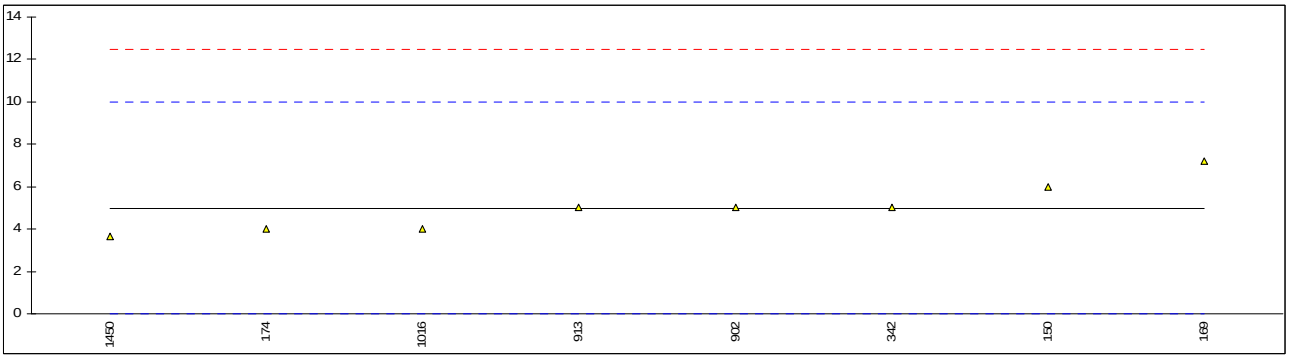
Determination of Anorganic Chloride as Cl on sample #11125; results in mg/kg.

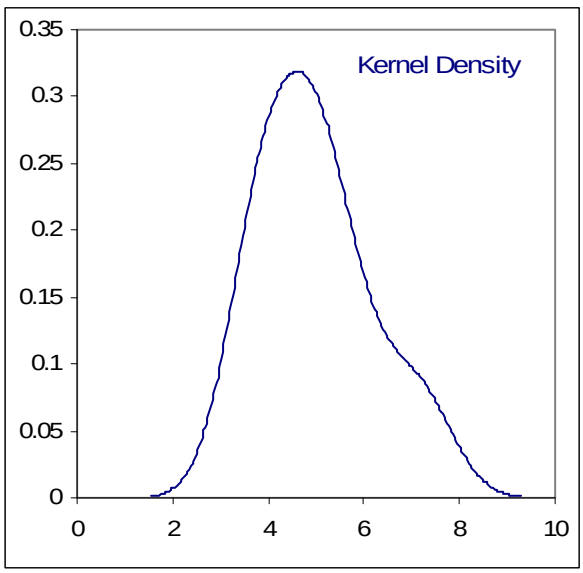
lab	method	value	mark	z(targ)	remarks
150	D7359	<0.5		-----	
169		-----		-----	
171		-----		-----	
173		-----		-----	
174	E2469	<0.1		-----	
311	INH-158	<0.2		-----	
323	INH-007	0.4		-----	
342		-----		-----	
522		-----		-----	
902		-----		-----	
913		-----		-----	
1016		-----		-----	
1373		-----		-----	
1429		-----		-----	
1438		-----		-----	
1450		-----		-----	
	normality	n.a.			
	n	1			
	outliers	n.a.			
	mean (n)	n.a.			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit.)	n.a.			

Determination of Colour Pt/Co scale on sample #11125;

lab	method	value	mark	z(targ)	remarks
150	D5386	6		0.41	
169	D5386	7.2		0.89	
171		-----		-----	
173	D1209	<5		-----	
174	D1209	4		-0.39	
311		-----		-----	
323	D1209	<5		-----	
342	D5386	5		0.01	
522		-----		-----	
902	D5386	5		0.01	
913	D5386	5		0.01	
1016	D1209	4		-0.39	
1373		-----		-----	
1429	D1209	<5		-----	
1438		-----		-----	
1450	D1209	3.65		-0.53	

normality	OK
n	8
outliers	0
mean (n)	5.0
st.dev. (n)	1.17
R(calc.)	3.3
R(D1209:11)	7.0

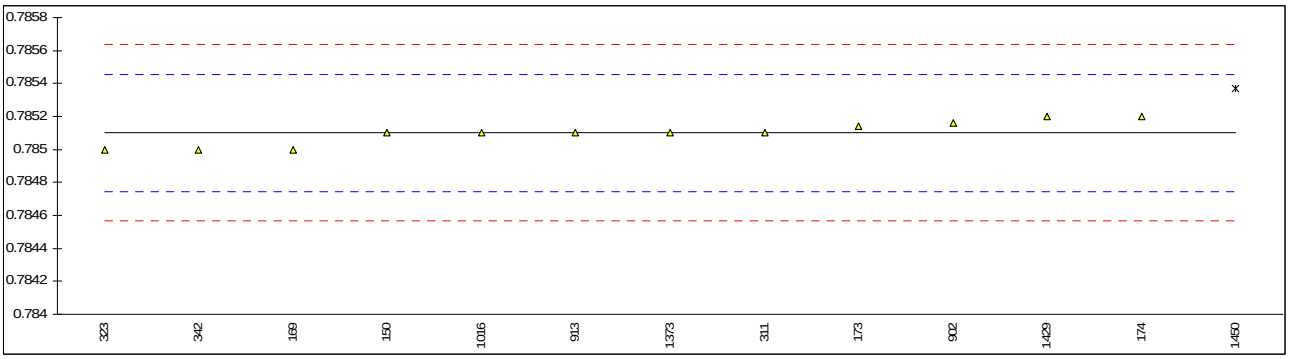


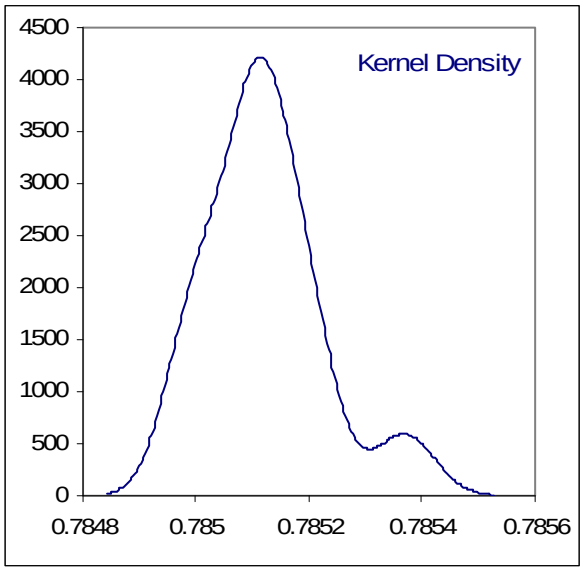


Determination of Density 20 ºC on sample #11125; results in kg/L.

lab	method	value	mark	z(targ)	remarks
150	D4052	0.7851		0.00	
169	D4052	0.7850		-0.56	
171		-----		-----	
173	D4052	0.78514		0.22	
174	D4052	0.7852		0.56	
311	D4052	0.7851		0.00	
323	D4052	0.7850		-0.56	
342	D4052	0.7850		-0.56	
522		-----		-----	
902	D4052	0.78516		0.34	
913	D4052	0.7851		0.00	
1016	D4052	0.7851		0.00	
1373	INH-007	0.7851		0.00	
1429	D4052	0.7852		0.56	
1438		-----		-----	
1450	D4052	0.78537	G(0.05)	1.51	

normality	not OK
n	12
outliers	1
mean (n)	0.78510
st.dev. (n)	0.000070
R(calc.)	0.00020
R(D4052:11)	0.00050

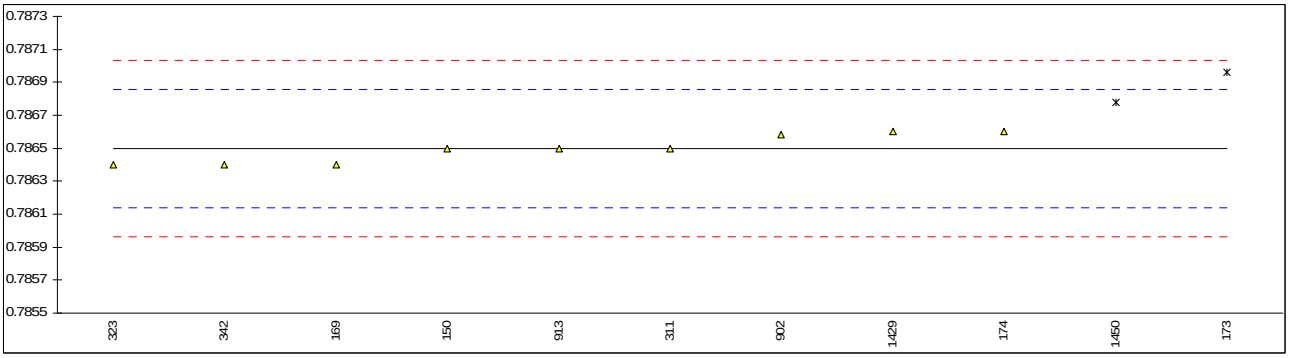


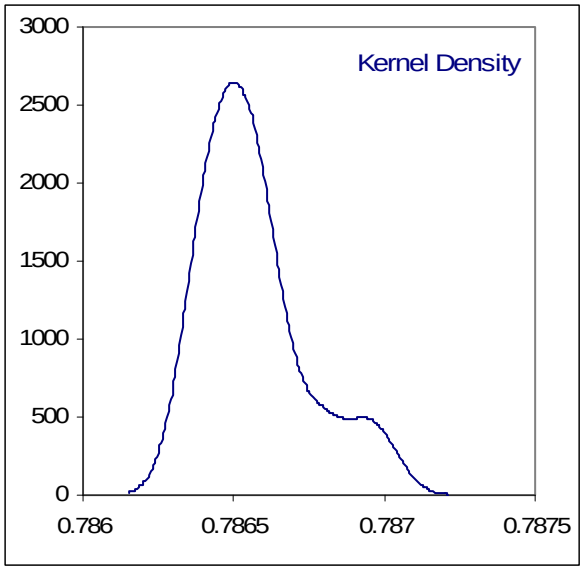


Determination of Specific Gravity 20/20ºC (relative Density 20ºC) on sample #11125;

lab	method	value	mark	z(targ)	remarks
150	D4052	0.7865		0.01	
169	D4052	0.7864		-0.55	
171		-----		-----	
173	D4052	0.78696	DG(0.05)	2.59	
174	D4052	0.7866		0.57	
311		0.7865		0.01	
323	D4052	0.7864		-0.55	
342	D4052	0.7864		-0.55	
522		-----		-----	
902	D4052	0.78658		0.46	
913	D4052	0.7865		0.01	
1016		-----		-----	
1373		-----		-----	
1429		0.7866		0.57	
1438		-----		-----	
1450		0.78678	DG(0.05)	1.58	

normality	OK
n	9
outliers	2
mean (n)	0.78650
st.dev. (n)	0.000084
R(calc.)	0.00024
R(D4052:11)	0.00050



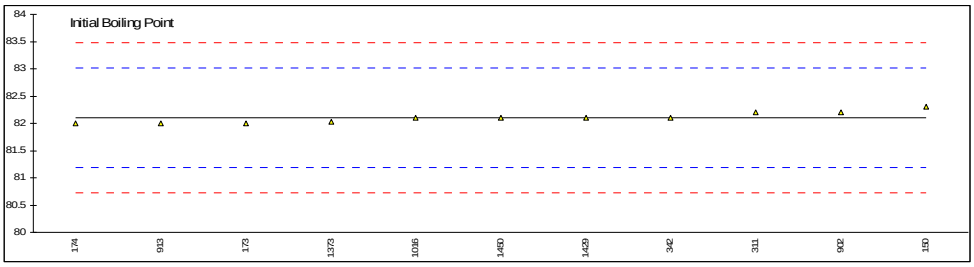


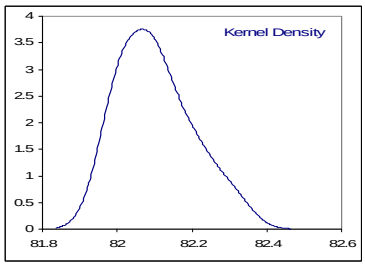
Determination of Distillation @ 760 mmHg on sample #11125; results in °C.

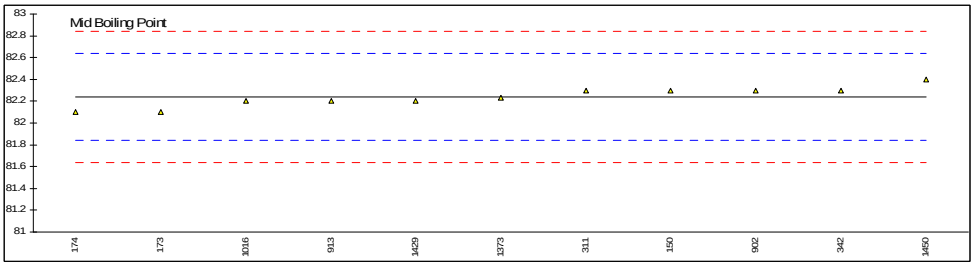
lab	method	IBP	mark	z(targ)	MBP	mark	z(targ)	DP	mark	z(targ)	remarks
150	D1078-A	82.3		0.43	82.3		0.30	82.5		0.34	
169		-----		-----	-----		-----	-----		-----	
171		-----		-----	-----		-----	-----		-----	
173	D1078-A	82.0		-0.22	82.1		-0.69	82.2		-0.61	
174	D1078-A	82.0		-0.22	82.1		-0.69	82.3		-0.30	
311	D1078-A	82.2		0.21	82.3		0.30	82.3		-0.30	
323		-----		-----	-----		-----	-----		-----	
342	D1078-M	82.1		-0.01	82.3		0.30	82.5		0.34	
522		-----		-----	-----		-----	-----		-----	
902	D1078-M	82.2		0.21	82.3		0.30	82.5		0.34	
913	D1078-M	82.0		-0.22	82.2		-0.19	82.4		0.02	
1016	D1078-A	82.1		-0.01	82.2		-0.19	82.3		-0.30	
1373	D1078-M	82.03		-0.16	82.23		-0.05	82.73		1.07	
1429	D1078-A	82.1		-0.01	82.2		-0.19	82.2		-0.61	
1438		-----		-----	-----		-----	-----		-----	
1450	D1078-A	82.1		-0.01	82.4		0.80	82.4		0.02	
	normality	OK			OK			OK			
	n	11			11			11			
	outliers	0			0			0			
	mean (n)	82.10			82.24			82.39			
	st.dev. (n)	0.097			0.092			0.158			
	R(calc.)	0.27			0.26			0.44			
	R(D1078:11-A)	1.28			0.56			0.88			
	R(D1078:11-M)	0.88			0.53			1.07			

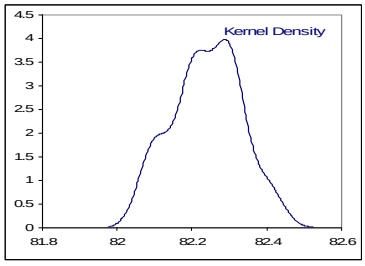
After manual correction for barometric pressure

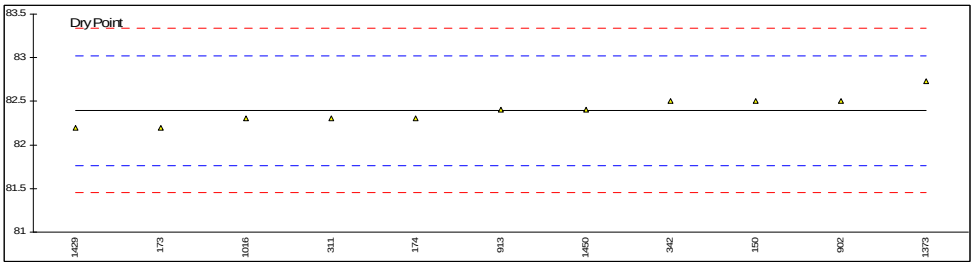
173	D1078-A	82.2		0.13	82.3		0.12	82.4		-0.07	
174	D1078-A	82.2		0.13	82.3		0.12	82.4		-0.07	
	normality	OK			OK			OK			
	n	11			11			11			
	outliers	0			0			0			
	mean (n)	82.14			82.28			82.42			
	st.dev. (n)	0.088			0.061			0.139			
	R(calc.)	0.25			0.17			0.39			
	R(D1078:11-A)	1.28			0.56			0.88			
	R(D1078:11-M)	0.88			0.53			1.07			

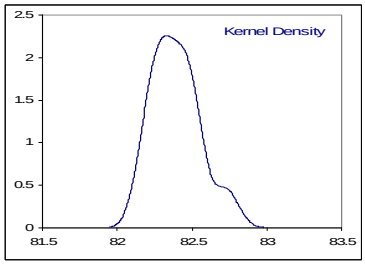








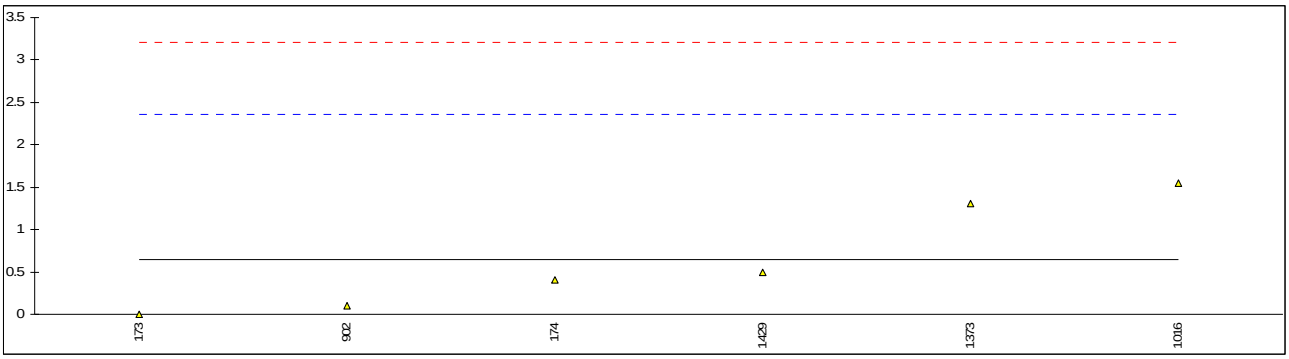




Determination of Nonvolatile Matter on sample #11125; results in mg/100 mL.

lab	method	value	mark	z(targ)	remarks
150		-----		-----	
169		-----		-----	
171		-----		-----	
173	D1353	0.0025	U	-0.75	Possibly an unit error?
174	D1353	0.4		-0.28	
311	D1353	<1		-----	
323		-----		-----	
342		-----		-----	
522		-----		-----	
902	D1353	0.1		-0.63	
913		-----		-----	
1016	D1353	1.55		1.06	
1373	D1353	1.3		0.77	
1429	D1353	0.5		-0.17	
1438		-----		-----	
1450		-----		-----	

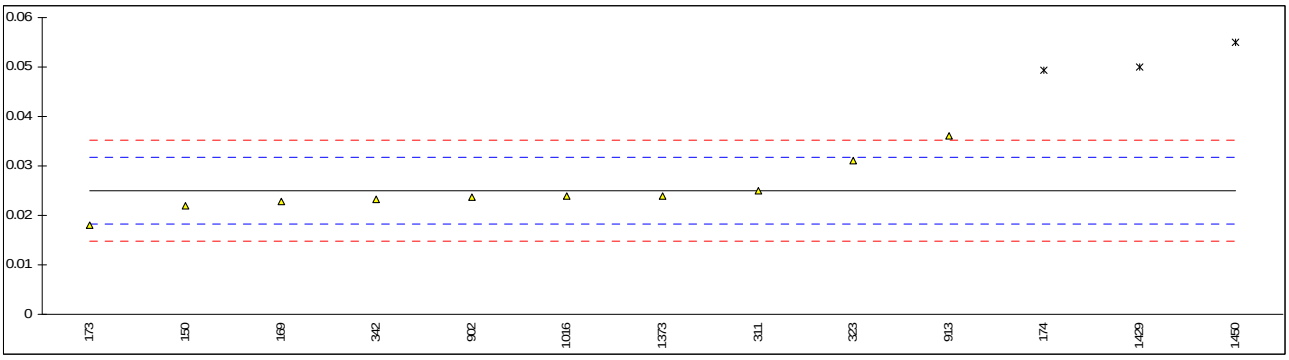
normality	OK
n	6
outliers	0
mean (n)	0.64
st.dev. (n)	0.639
R(calc.)	1.79
R(D1353:09)	2.40

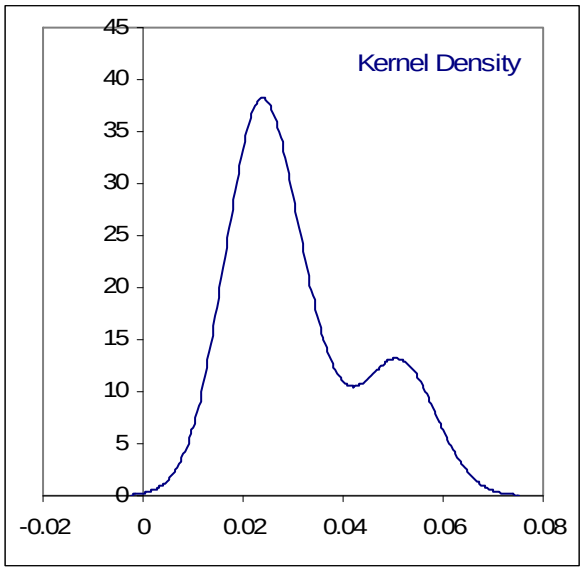


Determination of Water content on sample #11125; results in %M/M.

lab	method	value	mark	z(targ)	remarks
150	D1364	0.022		-0.88	
169	E1064	0.0228		-0.64	
171		-----		-----	
173	E1064	0.01794		-2.08	
174	D1364	0.0494	G(0.05)	7.21	
311	D1364	0.025		0.01	
323	D1364	0.031		1.78	
342	E1064	0.0232		-0.52	
522		-----		-----	
902	D1364	0.0238		-0.35	
913	D1364	0.036		3.26	
1016	D1364	0.024		-0.29	
1373	INH-001	0.024		-0.29	
1429	D1364	0.050	DG(0.01)	7.39	
1438		-----		-----	
1450	D1364	0.0551	DG(0.01)	8.90	

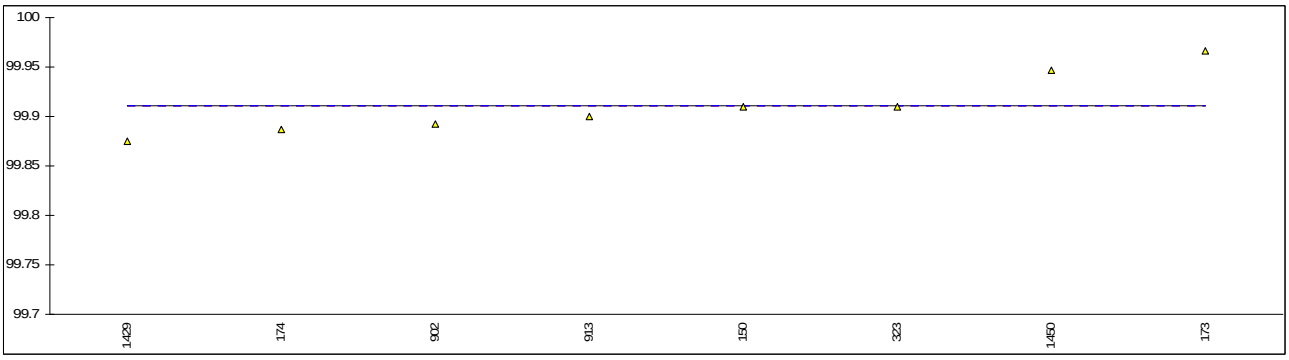
normality	not OK
n	10
outliers	3
mean (n)	0.0250
st.dev. (n)	0.00503
R(calc.)	0.0141
R(D1364:07)	0.0095

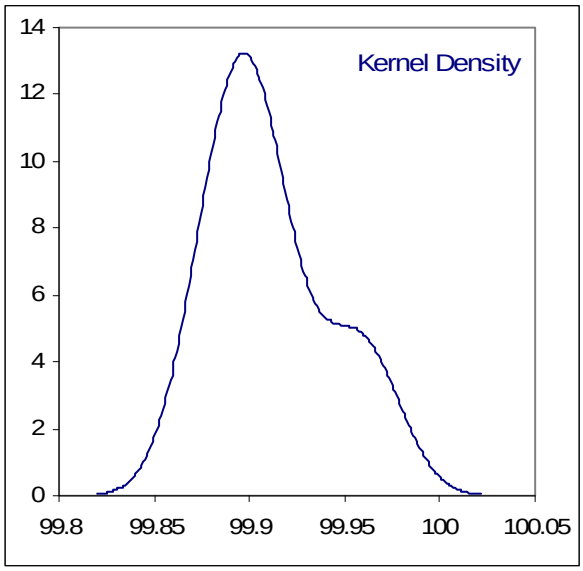




Determination of Purity “as received” on sample #11125; results in %M/M.

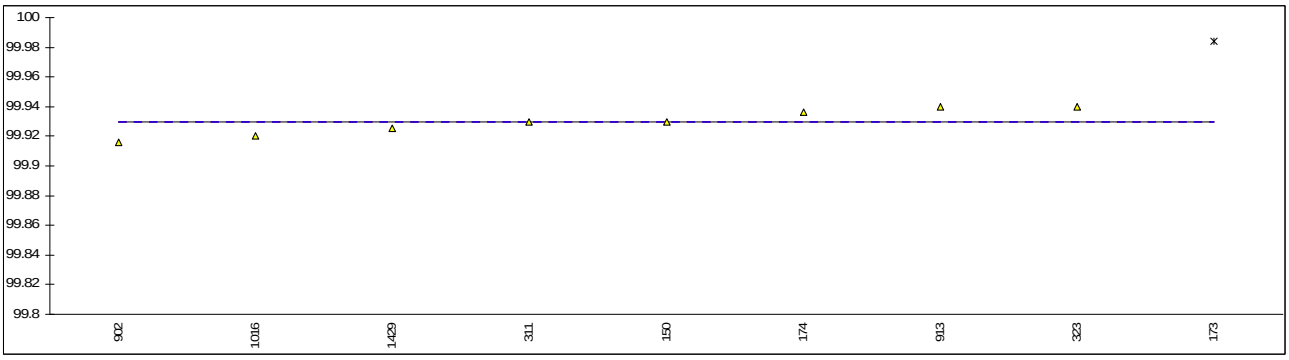
lab	method	value	mark	z(targ)	remarks
150	DIN55635Mod.	99.91		-----	
169		-----		-----	
171		-----		-----	
173	INH-6012	99.9665		-----	
174	D5501	99.887		-----	
311		-----		-----	
323	INH-006	99.91		-----	
342		-----		-----	
522		-----		-----	
902	INH-129	99.892		-----	
913	D5501	99.90		-----	
1016		-----		-----	
1373		-----		-----	
1429	DIN55685	99.875	C	-----	First reported 99.894
1438		-----		-----	
1450		99.947		-----	
	normality	OK			
	n	8			
	outliers	0			
	mean (n)	99.9109			
	st.dev. (n)	0.03101			
	R(calc.)	0.0868			
	R(lit.)	unknown			Compare R(iis09C14) = 0.0621

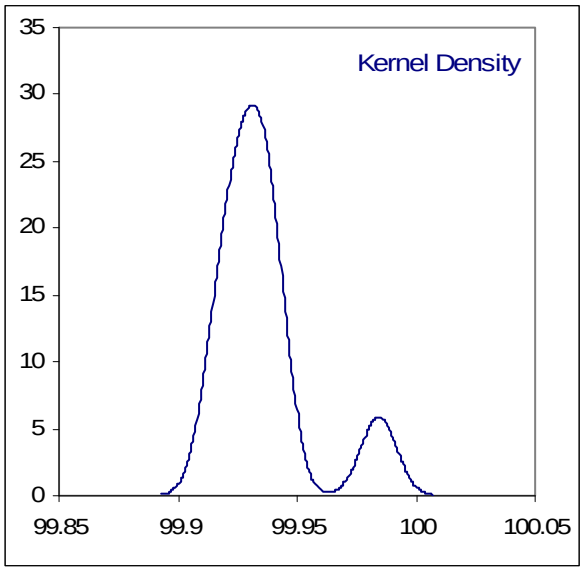




Determination of Purity on dry basis on sample #11125, results in %M/M.

lab	method	value	mark	z(targ)	remarks
150	DIN55635Mod.	99.93		-----	
169		-----		-----	
171		-----		-----	
173	INH-6012	99.9844	G(0.01)	-----	
174	D5501	99.936		-----	
311	INH-002	99.93		-----	
323	INH-006	99.94		-----	
342		-----		-----	
522		-----		-----	
902	INH-129	99.916		-----	
913	D5501	99.94		-----	
1016		99.92		-----	
1373		-----		-----	
1429	DIN55685	99.925	C	-----	First reported 99.944
1438		-----		-----	
1450		-----		-----	
	normality	OK			
	n	8			
	outliers	1			
	mean (n)	99.9296			
	st.dev. (n)	0.00891			
	R(calc.)	0.0250			
	R(lit.)	unknown			Compare R(iis09C14) = 0.0520

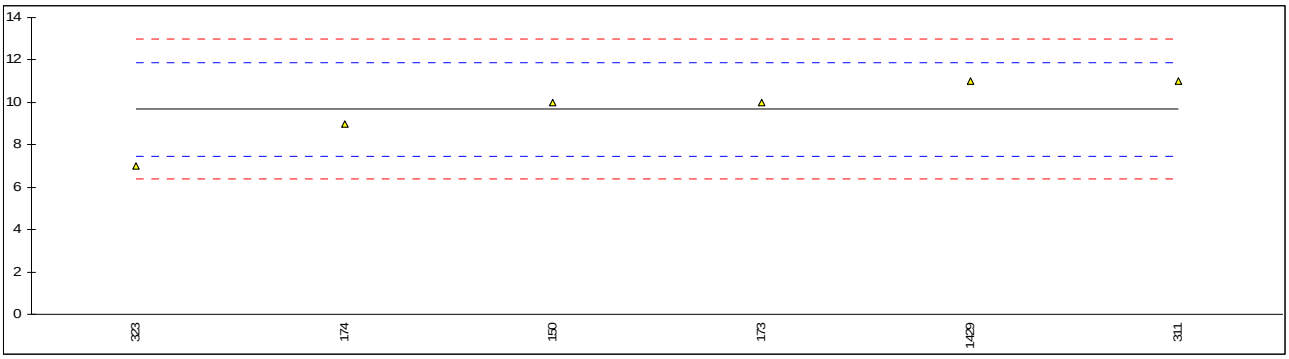




Determination of Ethanol content on sample #11125; results in mg/kg.

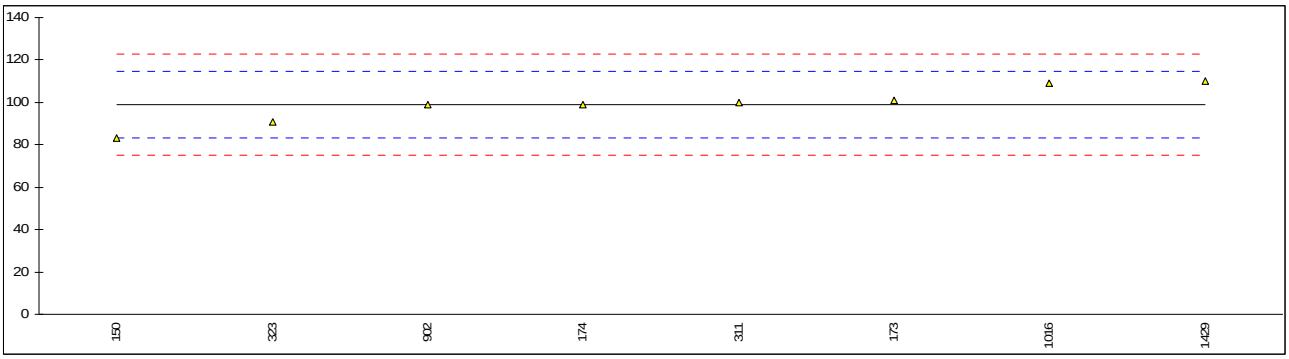
lab	method	value	mark	z(targ)	remarks
150	DIN55635Mod.	10		0.30	
169		-----		-----	
171		-----		-----	
173	INH-6012	10		0.30	
174	D5501	9		-0.61	
311	INH-002	11		1.21	
323	INH-006	7		-2.43	
342		-----		-----	
522		-----		-----	
902		-----		-----	
913		-----		-----	
1016		-----		-----	
1373		-----		-----	
1429	DIN55685	11		1.21	
1438		-----		-----	
1450	INH-7814	<10		<0.30	

normality	OK
n	6
outliers	0
mean (n)	9.7
st.dev. (n)	1.51
R(calc.)	4.2
R(Horwitz)	3.1



Determination of n-Propanol on sample #11125; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	DIN55635Mod.	83		-2.02	
169		-----		-----	
171		-----		-----	
173	INH-6012	101		0.25	
174	D5501	99		0.00	
311	INH-002	100		0.13	
323	INH-006	91		-1.01	
342		-----		-----	
522		-----		-----	
902	INH-129	98.8		-0.03	
913		-----		-----	
1016		109.2		1.29	
1373		-----		-----	
1429	DIN55685	110		1.39	
1438		-----		-----	
1450	INH-7814	<10		<-11.23	False negative?
	normality	OK			
	n	8			
	outliers	0			
	mean (n)	99.0			
	st.dev. (n)	8.86			
	R(calc.)	24.8			
	R(Horwitz)	22.2			

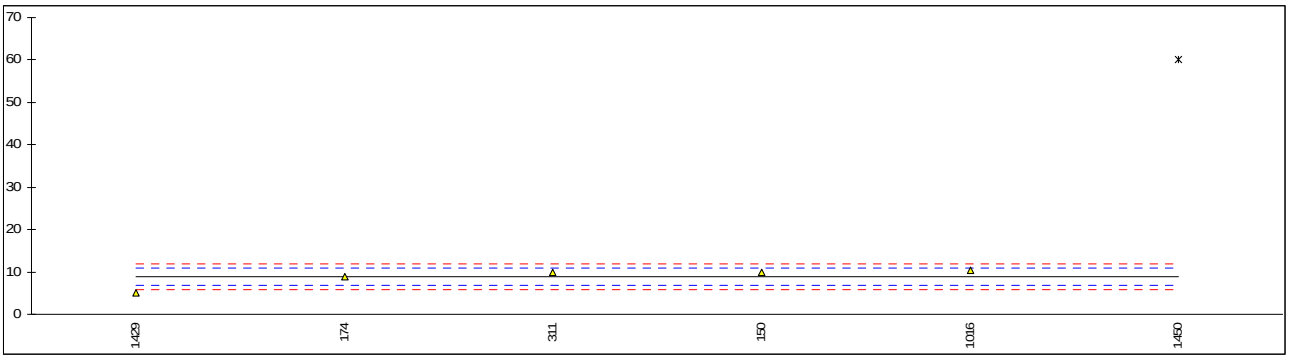


Determination of n-Butanol on sample #11125; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	DIN55635Mod.	6		-----	
169		-----		-----	
171		-----		-----	
173		-----		-----	
174	D5501	<10		-----	
311	INH-002	16		-----	
323		-----		-----	
342		-----		-----	
522		-----		-----	
902		-----		-----	
913		-----		-----	
1016		54.8	G(0.01)	-----	False positive?
1373		-----		-----	
1429	DIN55685	9		-----	
1438		-----		-----	
1450	INH-7814	<10		-----	
	normality	n.a.			
	n	3			
	outliers	1			
	mean (n)	10.3			
	st.dev. (n)	5.13			
	R(calc.)	14.4			
	R(lit)	unknown			

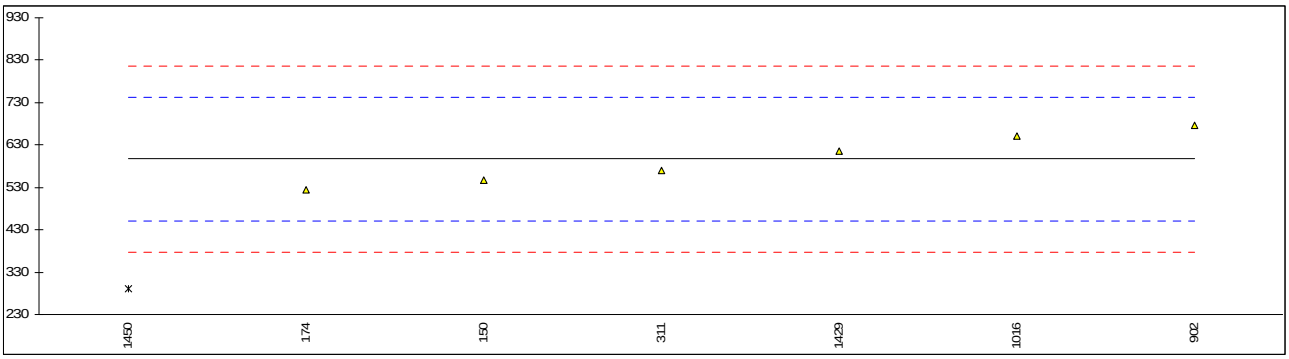
Determination of Methylethylketone on sample #11125; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	DIN55635Mod.	10		1.12	
169		-----		-----	
171		-----		-----	
173	INH-6012	<100		-----	
174	D5501	9		0.14	
311	INH-002	10		1.12	
323		-----		-----	
342		-----		-----	
522		-----		-----	
902	INH-129	<10		-----	
913		-----		-----	
1016		10.3		1.41	
1373		-----		-----	
1429	DIN55685	5		-3.78	
1438		-----		-----	
1450	INH-7814	60	G(0.01)	50.10	
	normality	OK			
	n	5			
	outliers	1			
	mean (n)	8.9			
	st.dev. (n)	2.21			
	R(calc.)	6.2			
	R(Horwitz)	2.9			



Determination of Other Impurities on sample #11125; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	DIN55635Mod.	546		-0.70	
169		-----		-----	
171		-----		-----	
173		-----		-----	
174	D5501	523		-1.02	
311	INH-002	570		-0.37	
323		-----		-----	
342		-----		-----	
522		-----		-----	
902	INH-129	677		1.09	
913		-----		-----	
1016		651		0.74	
1373		-----		-----	
1429	DIN55685	616	C	0.26	First reported 923
1438		-----		-----	
1450	INH-7814	292	G(0.05)	-4.18	
	normality	OK			
	n	6			
	outliers	1			
	mean (n)	597.2			
	st.dev. (n)	60.80			
	R(calc.)	170.2			
	R(Horwitz)	204.4			



APPENDIX 2

Number of participants per country

1 laboratory in	BELGIUM
1 laboratory in	INDIA
1 laboratory in	ISRAEL
1 laboratory in	MEXICO
1 laboratory in	P.R. of CHINA
1 laboratory in	PORTUGAL
1 laboratory in	SPAIN
2 laboratories in	THE NETHERLANDS
1 laboratory in	TURKEY
5 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/)