

Results of Proficiency Test
Isopropanol
(Isopropyl alcohol)
December 2015

Organised by: Institute for Interlaboratory Studies
Spijkensisse, 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 2015/2016 the Institute for Interlaboratory Studies decided to continue this proficiency test on Isopropanol. The proficiency test on Isopropanol has been organised in accordance with the latest applicable version of the ASTM D770 specification and a number of additional tests requested by some participants. In this interlaboratory study, 17 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 2015 proficiency test are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET UP

The Institute for Interlaboratory studies (iis) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an accredited laboratory. It was decided to send one 0.5 L bottle with Isopropanol, labelled #15240 to the participants. The participants were requested to report the analytical results using the indicated units and 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/IEC 17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 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 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (iis-protocol, version 3.3) of April 2014. This protocol is electronically available through the iis internet site www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

Approximately 20 litre of Isopropanol was obtained from a local chemical supplier. After homogenisation, 25 amber glass bottles of 0.5 L with inner and outer caps were filled and labelled #15240. The homogeneity of subsample #15240 was checked by determination of the Density in accordance with ISO12185 and Water in accordance with ASTM D1364 on 8 stratified randomly selected samples.

	Density at 20°C in kg/L	Water content in %M/M
sample #15240-1	0.78505	0.028
sample #15240-2	0.78504	0.028
sample #15240-3	0.78505	0.029
sample #15240-4	0.78504	0.030
sample #15240-5	0.78505	0.029
sample #15240-6	0.78506	0.029
sample #15240-7	0.78505	0.029
sample #15240-8	0.78505	0.029

Table 1: homogeneity tests of subsamples #15240

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 20°C in kg/L	Water content in %M/M
r (observed)	0.00002	0.002
reference method	ISO12185:96	D1364:02(2012)
0.3 * R (reference method)	0.00015	0.003

Table 2: evaluation of the repeatabilities of subsamples #15240

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

To each of the participating laboratories 1* 0.5 litre bottle, labelled #15240 was sent on November 11, 2015.

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, Inorganic Chloride, Appearance, Colour Pt/Co, Density at 20°C, Distillation (IBP, 50% evaporated & DP), Nonvolatile Matter, Specific Gravity at 20/20°C, Water, Purity (both “as received” and on dry basis), Ethanol, n-Propanol, n-Butanol and Other Impurities on sample #15240.

To get comparable results, a detailed report form on which the units were prescribed as well as the preferred test methods and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sqs-iis/. A SDS and a form to confirm receipt of the samples were added to the package.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sqs-iis/. 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 was sent to those 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

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' of April 2014 (iis-protocol, version 3.3).

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 a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. 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.

According to ISO 5725 the original results per determination were submitted to Dixon's and/or Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test (ref. 14). Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the

Grubbs' test and by $R(0.05)$ for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

Finally the reproducibilities were calculated from the standard deviations by multiplying these with a factor of 2.8.

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are 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. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

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:

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

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 whether the reported test result is fit-for-use.

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. Participants in Brazil and India received the sample late. The 17 participating laboratories submitted 192 numerical results. Observed were 8 outlying results, which is 4.2%. 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.

Unfortunately, a suitable standard test method, providing the precision data, is not available for all determinations. For the tests, that have no available precision data, the spreads were compared against the spreads estimated from the Horwitz equation.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as "not OK" or "suspect". The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D1613) and an added designation for the year that the method was adopted or revised (e.g. 1613:06). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D1613:06 (2012)). In the results tables of appendix 1 only the method number and year of adoption or revision will be used.

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(2012).

Appearance: No analytical problems were observed. For appearance, it is advised to use method ASTM E2680:09(2015). All participants agreed about the appearance of sample #15240 to be 'clear and bright' or 'pass'. Participants who used ASTM E2680 should report the appearance as 'pass' or as 'fail' dependent on the appearance of the product.

Chloride, inorganic: All reported test results were near or below the detection limit. Therefore, no significant conclusions were drawn.

- Colour Pt/Co: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D1209:05(2011).
- Density at 20°C: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ISO12185:96.
- Specific Gravity at 20/20 °C: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ISO12185:96.
- Distillation: This determination was not problematic. In total one statistical outlier was observed. All three calculated reproducibilities after rejection of the statistical outlier are in good agreement with the requirements of ASTM D1078:11 for the automated and the manual mode. Remarkably, two laboratories probably did not correct for barometric pressure and thermometer readings. Although the theoretical mid boiling point is 82.3 °C (see table 3 of ASTM D1078), test results of 82.0 and 82.1 °C were reported by two laboratories.
- NVM: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D1353:09(2013).
- Purity on dry basis: This determination is problematic as the group of reported test results appears to be bimodal divided. Obviously various ways of calculation were used. Eight laboratories deducted ethanol, n-propanol and other impurities from 100%, one laboratory also deducted the water content. The other five laboratories used other ways of calculation.
- Ethanol: This determination was problematic for a number of participants. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the estimated requirements calculated using the Horwitz equation.
- n-Propanol: This determination was problematic for a number of participants. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the estimated requirements calculated using the Horwitz equation. One false negative test result was observed.
- n-Butanol: All reported test results were near or below the detection limit. Therefore, no significant conclusions were drawn.

Other Imp.: This determination may be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility calculated using the Horwitz equation (for 4 components). One should note that the actual amount of so called “other impurities” depends on a number of parameters, like: definition, GLC column, GLC conditions, Stop time, detection limits, etc. This may partly explain the variability.

Water: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D1364:02(2012).

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 #15240 are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acidity as Acetic Acid	mg/kg	17	8.3	6.4	14.0
Appearance		16	pass	n.e.	n.e.
Chloride, inorganic as Cl	mg/kg	9	<1	n.e.	n.e.
Colour Pt/Co		12	2.1	3.0	7.0
Density at 20°C	kg/L	17	0.7851	0.0003	0.0005
Specific Gravity at 20/20°C		15	0.7865	0.0002	0.0005
Initial Boiling Point	°C	17	82.1	0.3	1.3
50% evaporated	°C	16	82.3	0.2	0.6
Dry Point	°C	17	82.4	0.3	0.9
Nonvolatile Matter	mg/100mL	10	0.4	0.6	2.4
Purity on dry basis	%M/M	15	see pages 8 and 19		
Ethanol	mg/kg	11	1432	212	215
n-Propanol	mg/kg	11	433	66	78
n-Butanol	mg/kg	10	<20	n.e.	n.e.
Other Impurities	mg/kg	7	201	260	81
Water	%M/M	15	0.030	0.010	0.010

Table 3: Reproducibilities for sample #15240

Without further statistical calculations it can be concluded that for many tests there is 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 DECEMBER 2015 WITH THE PREVIOUS PTS.

	<i>December 2015</i>	<i>November 2013</i>	<i>November 2011</i>	<i>November 2009</i>
Number of reporting labs	17	16	13	15
Number of results reported	192	168	143	165
Statistical outliers	8	7	10	5
Percentage outliers	4.2%	4.2%	7.0%	3.0%

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>December 2015</i>	<i>November 2013</i>	<i>November 2011</i>	<i>November 2009</i>
Acidity as acetic acid	++	++	++	++
Chloride, inorganic as Cl	n.e.	n.e.	n.e.	-
Colour Pt/Co	++	++	++	++
Density at 20°C	++	+	++	++
Specific Gravity 20/20 °C	++	+	++	++
Initial Boiling Point	++	++	++	++
50% evaporated	++	++	++	++
Dry Point	++	++	++	++
Nonvolatile Matter	++	++	++	++
Purity "as received"	n.e.	(++)	(--)	(+)
Purity on dry basis	n.e.	(++)	(++)	(-)
Ethanol	+/-	n.e.	-	(--)
n-Propanol	+	-	+/-	+/-
n-Butanol	n.e.	n.e.	n.e.	--
Other impurities	--	--	+	n.e.
Water	+/-	+/-	--	++

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:

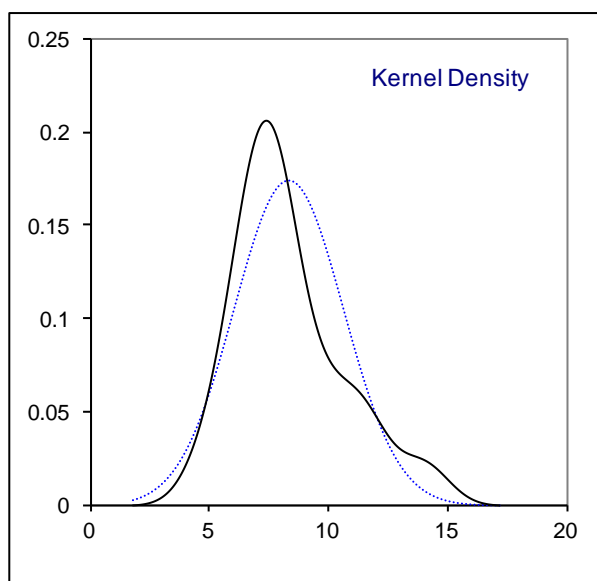
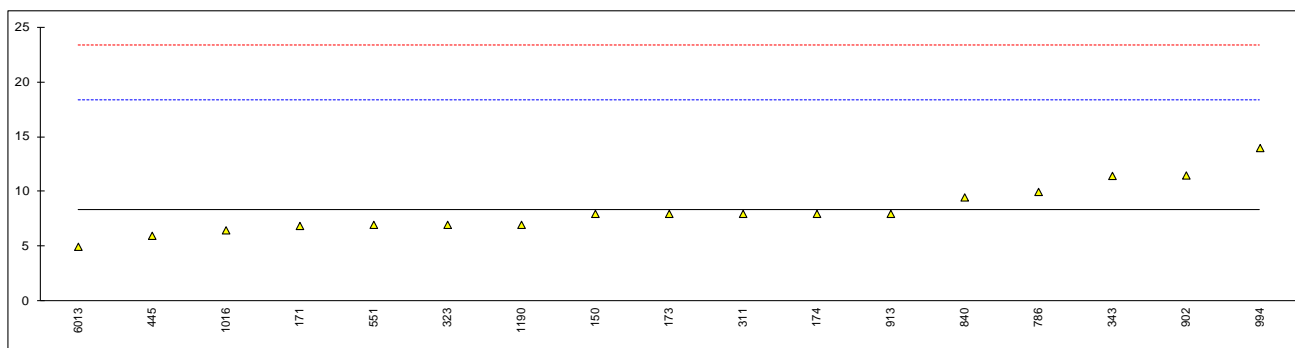
- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- : group performed worse than the standard
- : group performed much worse than the standard
- n.e.: not evaluated

APPENDIX 1

Determination of Acidity as Acetic Acid on sample #15240; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	D1613	8		-0.07	
171	D1613	6.9		-0.29	
173	D1613	8		-0.07	
174	D1613	8		-0.07	
311	D1613	8		-0.07	
323	D1613	7		-0.27	
343	D1613	11.46		0.62	
445	D1613	6	C	-0.47	First reported as 0.0006
551	D1613	7		-0.27	
786	D1613	10		0.33	
840	D1613	9.5		0.23	
902	D1613	11.5		0.63	
913	D1613	8.0		-0.07	
994	D1613	14		1.13	
1016	D1613	6.5		-0.37	
1190	D1613	7		-0.27	
6013	D1613	5		-0.67	

normality not OK
n 17
outliers 0
mean (n) 8.34
st.dev. (n) 2.290
R(calc.) 6.41
R(D1613:06) 14.00



Determination of Appearance on sample #15240;

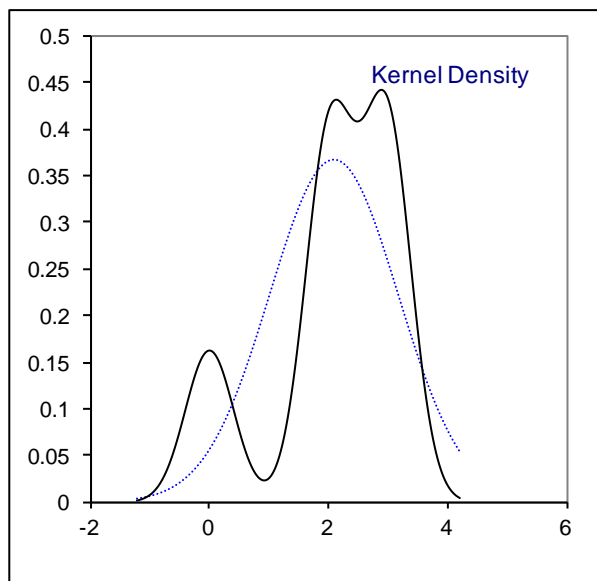
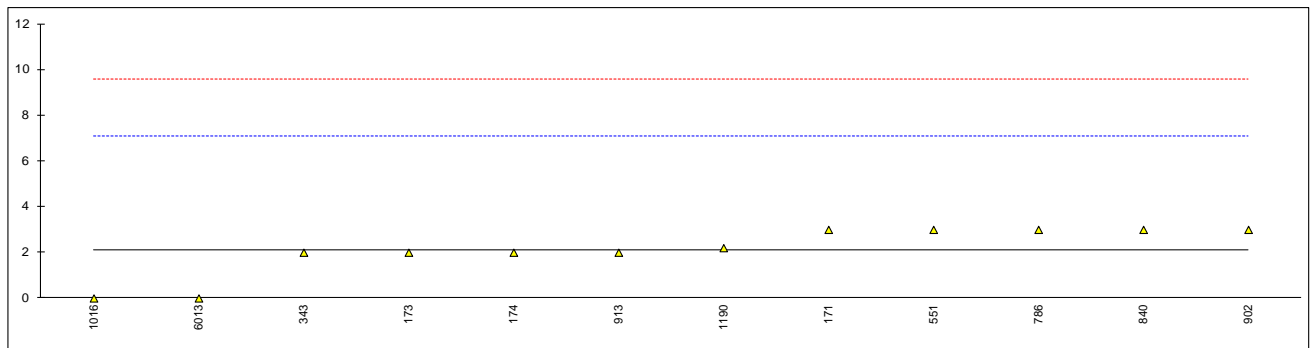
lab	method	value	mark	z(targ)	remarks
150	E2680	Pass		----	
171	E2680	Pass		----	
173	Visual	pass		----	
174	E2680	PASS		----	
311	E2680	pass		----	
323	E2680	clear & bright		----	
343	E2680	Pass		----	
445	E2680	PASS		----	
551	Visual	Pass		----	
786	E2680	Pass		----	
840	E2680	pass		----	
902	E2680	PASS		----	
913	E2680	Pass		----	
994	Visual	pass		----	
1016				----	
1190	Visual	CFSM		----	
6013	D4176	Clear Bright		----	
	normality	n.a.			
	n	16			
	outliers	n.a.			
	mean (n)	Pass			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(E2680:09)	n.a.			

Determination of Chloride, Inorganic as Cl on sample #15240; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	D7359	<1		----	
171	D7319	0.0229		----	
173		----		----	
174	E2469	0.039		----	
311	INH-158	<0.2		----	
323	INH-008	<1		----	
343		----		----	
445		----		----	
551		----		----	
786	IMPCA002	<0.25		----	
840	IMPCA002	0.056		----	
902		----		----	
913		----		----	
994		----		----	
1016		----		----	
1190	E2469	<1		----	
6013	INH-ST47	<0.2		----	
	normality	n.a.			
	n	9			
	outliers	n.a.			
	mean (n)	<1			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit.)	n.a.			

Determination of Colour Pt/Co scale on sample #15240;

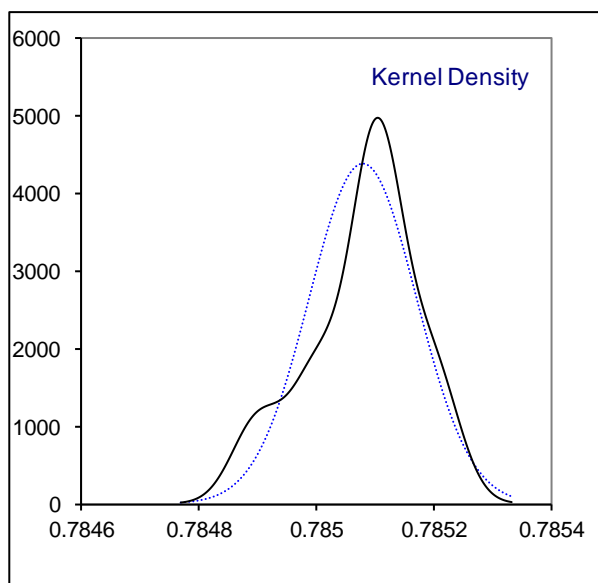
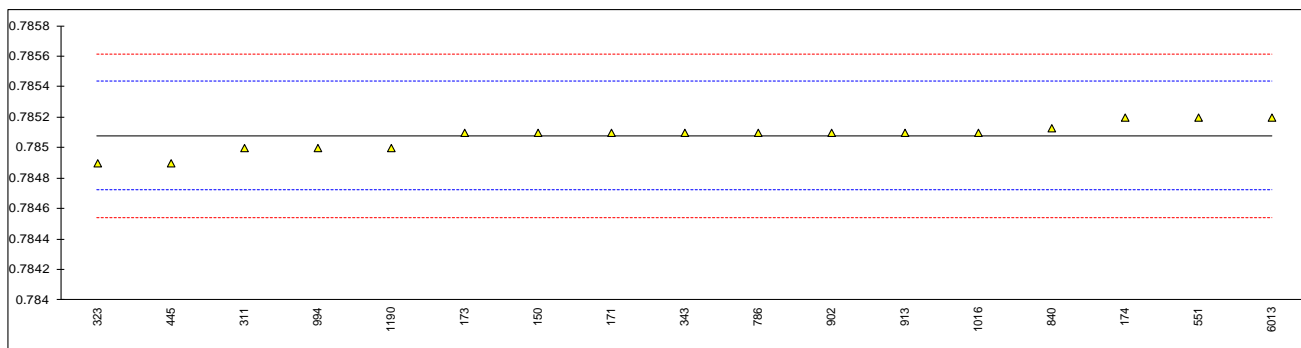
lab	method	value	mark	z(targ)	remarks
150	D5386	<5		-----	
171	D5386	3		0.36	
173	D1209	2		-0.04	
174	D1209	2		-0.04	
311	D1209	<5		-----	
323	D1209	<5		-----	
343	D5386	2		-0.04	
445		-----		-----	
551	D5386	3		0.36	
786	D1209	3		0.36	
840	D1209	3		0.36	
902	D5386	3		0.36	
913	D5386	2		-0.04	
994	D1209	L.5		-----	
1016	D1209	0		-0.84	
1190	D1209	2.2		0.04	
6013	D1209	0		-0.84	
normality		OK			
n		12			
outliers		0			
mean (n)		2.10			
st.dev. (n)		1.084			
R(calc.)		3.03			
R(D1209:05)		7.00			



Determination of Density at 20 °C on sample #15240; results in kg/L.

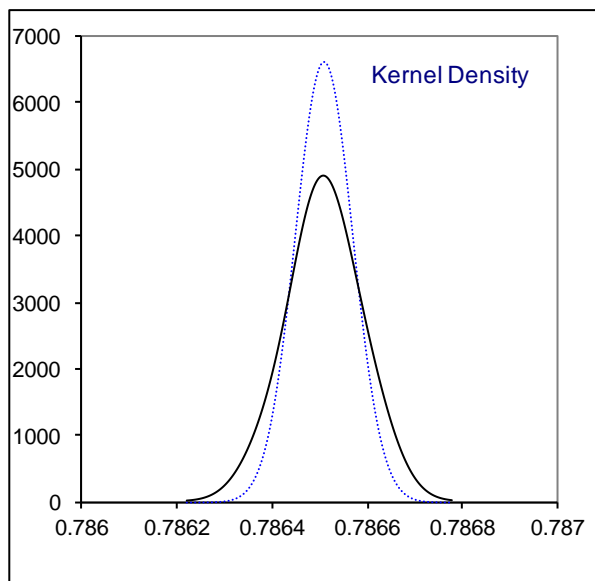
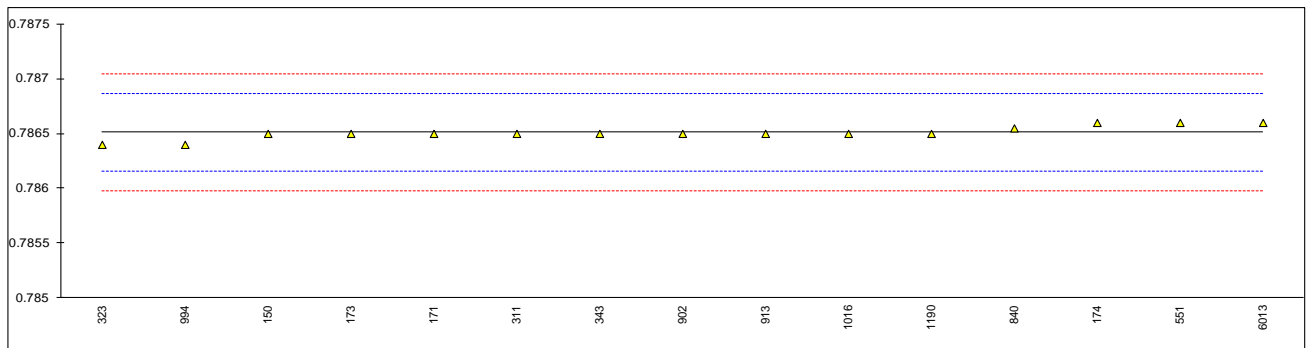
lab	method	value	mark	z(targ)	remarks
150	D4052	0.7851	C	0.12	First reported 785.1
171	D4052	0.7851		0.12	
173	D4052	0.7851		0.12	
174	D4052	0.7852		0.68	
311	D4052	0.7850		-0.44	
323	D4052	0.7849		-1.00	
343	D4052	0.7851		0.12	
445	ISO12185	0.7849		-1.00	
551	D4052	0.7852		0.68	
786	D4052	0.7851		0.12	
840	D4052	0.78513		0.29	
902	D4052	0.7851		0.12	
913	D4052	0.7851		0.12	
994	ISO12185	0.7850		-0.44	
1016	D4052	0.7851		0.12	
1190	D4052	0.7850	C	-0.44	First reported 785
6013	ISO12185	0.7852		0.68	

normality OK
n 17
outliers 0
mean (n) 0.78508
st.dev. (n) 0.000091
R(calc.) 0.00026
R(ISO12185:96) 0.00050



Determination of Specific Gravity at 20/20°C on sample #15240;

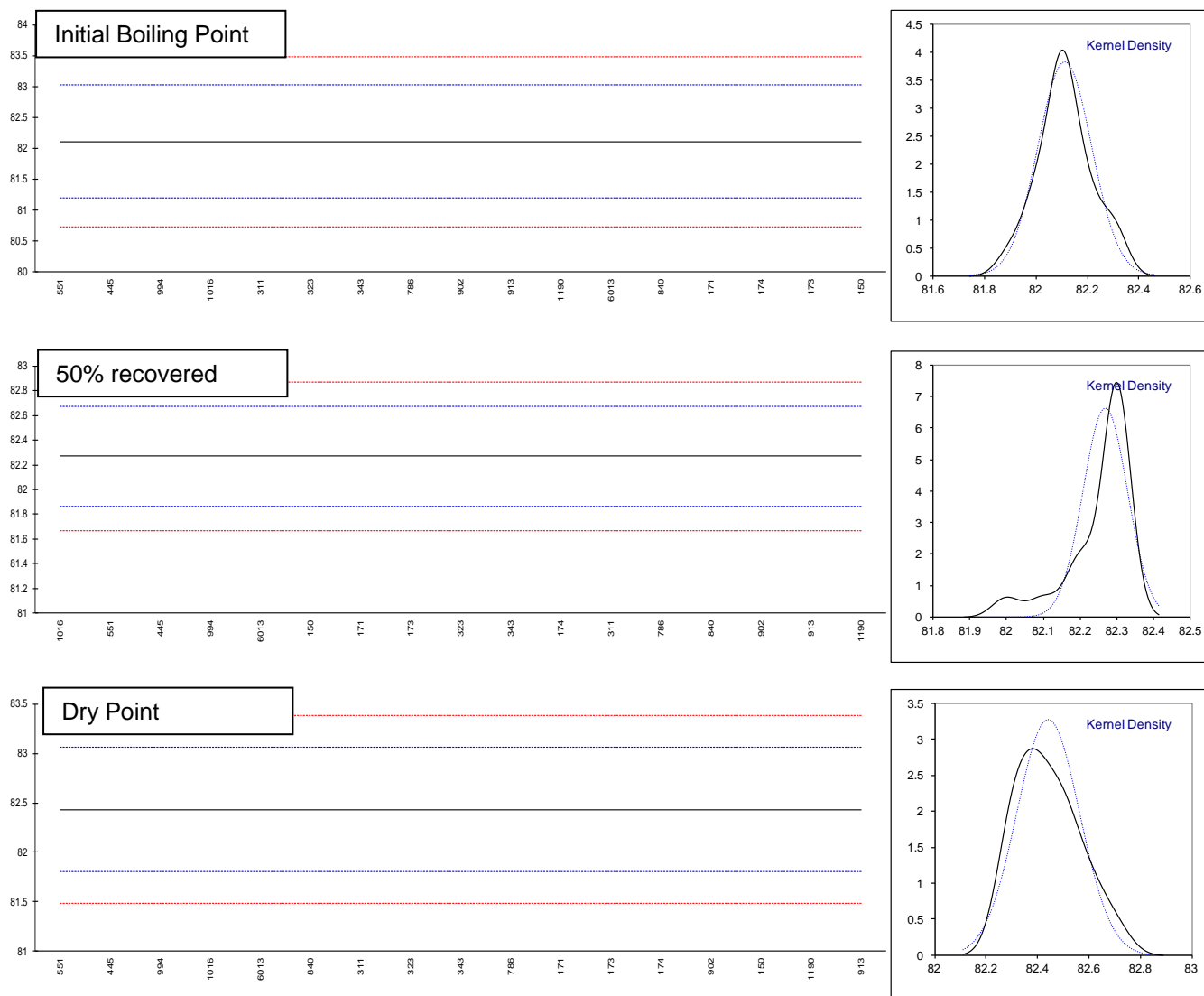
lab	method	value	mark	z(targ)	remarks
150	D4052	0.7865		-0.06	
171	D4052	0.7865		-0.06	
173	D4052	0.7865		-0.06	
174	D4052	0.7866		0.50	
311	D4052	0.7865		-0.06	
323	ISO12185	0.7864		-0.62	
343	D4052	0.7865		-0.06	
445		----		----	
551	D4052	0.7866		0.50	
786		----		----	
840	D4052	0.78655		0.22	
902	D4052	0.7865		-0.06	
913	D4052	0.7865		-0.06	
994	ISO12185	0.7864		-0.62	
1016	D4052	0.7865		-0.06	
1190	D4052	0.7865		-0.06	
6013	ISO12185	0.7866		0.50	
normality		OK			
n		15			
outliers		0			
mean (n)		0.78651			
st.dev. (n)		0.000060			
R(calc.)		0.00017			
R(ISO12185:96)		0.00050			



Determination of Distillation at 760 mmHg on sample #15240; results in °C.

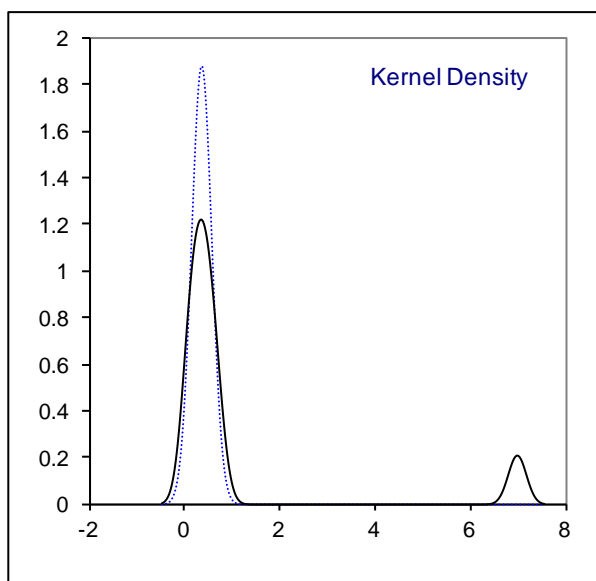
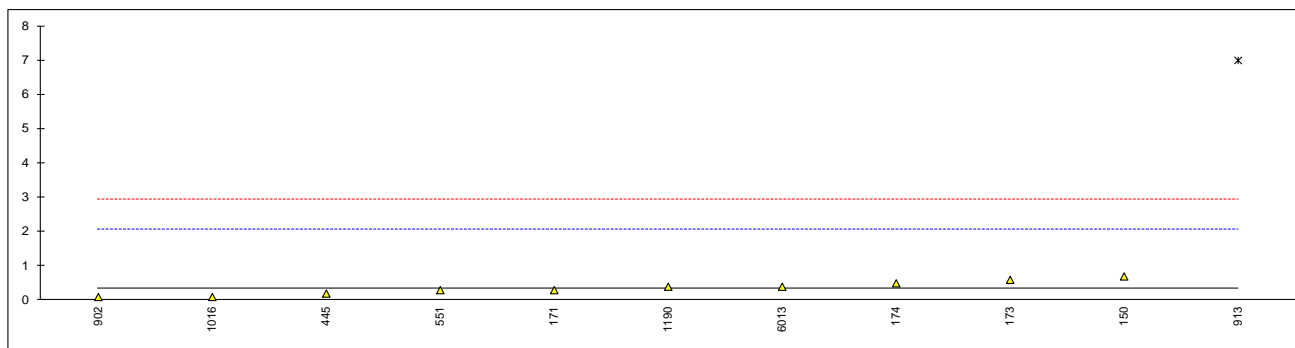
lab	method	mode	IBP	mark	z(targ)	50%rec	mark	z(targ)	DP	mark	z(targ)
150	D1078	Automated	82.3		0.41	82.3		0.16	82.6		0.53
171	D1078	Automated	82.2		0.20	82.3		0.16	82.5		0.21
173	D1078	Automated	82.3		0.41	82.3		0.16	82.5		0.21
174	D1078	Automated	82.2		0.20	82.3		0.16	82.5		0.21
311	D1078	Automated	82.1		-0.02	82.3		0.16	82.4		-0.11
323	D1078	Manual	82.1		-0.02	82.3		0.16	82.4		-0.11
343	D1078	Automated	82.1		-0.02	82.3		0.16	82.4		-0.11
445	D1078	Manual	82.0		-0.24	82.2		-0.34	82.3		-0.42
551	D1078	Automated	81.9		-0.46	82.1 *)		-0.84	82.3		-0.42
786	D1078	Manual	82.1		-0.02	82.3		0.16	82.4		-0.11
840	D1078	Automated	82.18		0.15	82.30		0.16	82.37		-0.20
902	D1078	Manual	82.1		-0.02	82.3		0.16	82.5		0.21
913	D1078	Manual	82.1		-0.02	82.3		0.16	82.7		0.85
994	D1078	Manual	82.0		-0.24	82.2		-0.34	82.3		-0.42
1016	D1078	Automated	82.0		-0.24	82.0 *)	G(0.01)	-1.34	82.3		-0.42
1190	D1078	Manual	82.1		-0.02	82.3		0.16	82.6		0.53
6013	D1078	Automated	82.1		-0.02	82.2		-0.34	82.3		-0.42
	normality		OK			not OK			OK		
	n		17			16			17		
	outliers		0			1			0		
	mean (n)		82.11			82.27			82.43		
	st.dev. (n)		0.104			0.060			0.123		
	R(calc.)		0.29			0.17			0.34		
	R(D1078:11-A)		1.28			0.56			0.88		

*) Results probably not corrected for barometric and thermometer corrections.



Determination of Nonvolatile Matter on sample #15240; results in mg/100 mL.

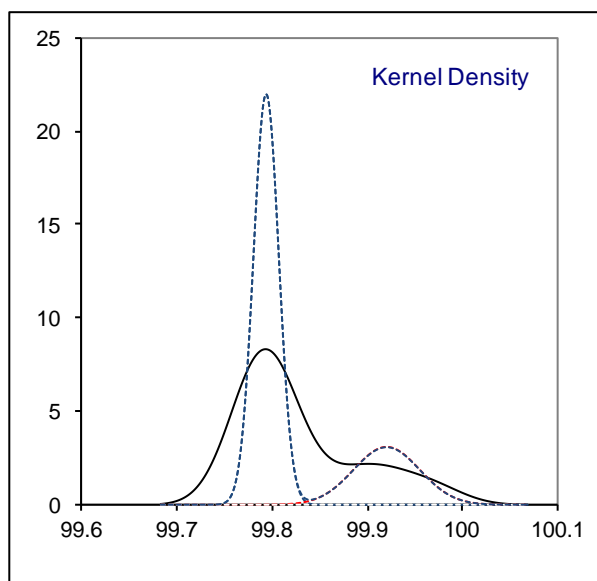
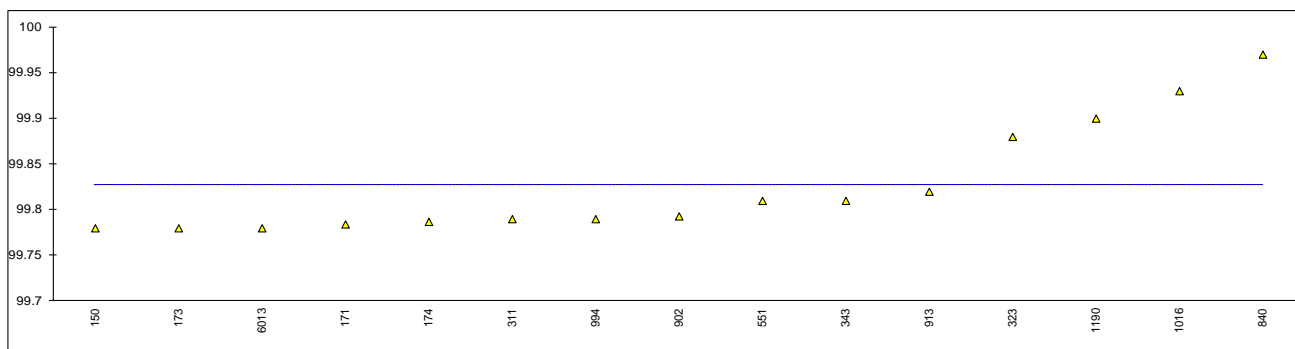
lab	method	value	mark	z(targ)	remarks
150	D1353	0.7		0.40	
171	D1353	0.3		-0.07	
173	D1353	0.6		0.28	
174	D1353	0.5		0.16	
311	D1353	<1		-----	
323	D1353	<1		-----	
343		-----		-----	
445	D1353	0.2		-0.19	
551	D1353	0.3		-0.07	
786		-----		-----	
840	D1353	<1		-----	
902	D1353	0.1		-0.30	
913	D1353	7.0	G(0.01)	7.75	Unit error?
994		-----		-----	
1016	D1353	0.1		-0.30	
1190	D1353	0.4		0.05	
6013	D1353	0.4		0.05	
normality		OK			
n		10			
outliers		1			
mean (n)		0.36			
st.dev. (n)		0.201			
R(calc.)		0.56			
R(D1353:13)		2.40			



Determination of Purity on dry basis on sample #15240, results in %M/M.

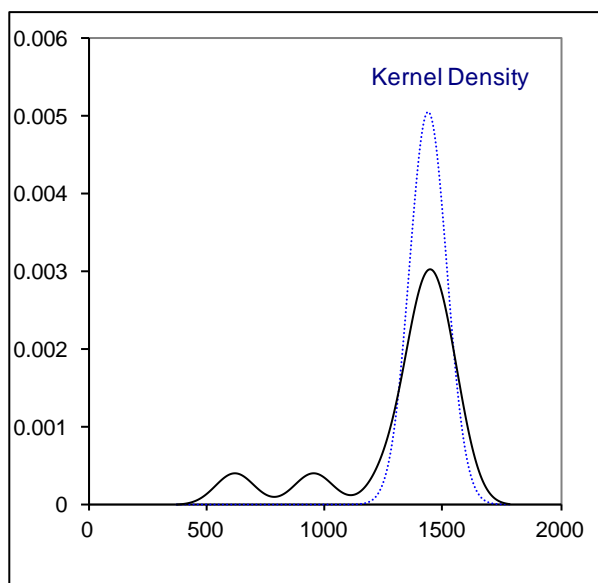
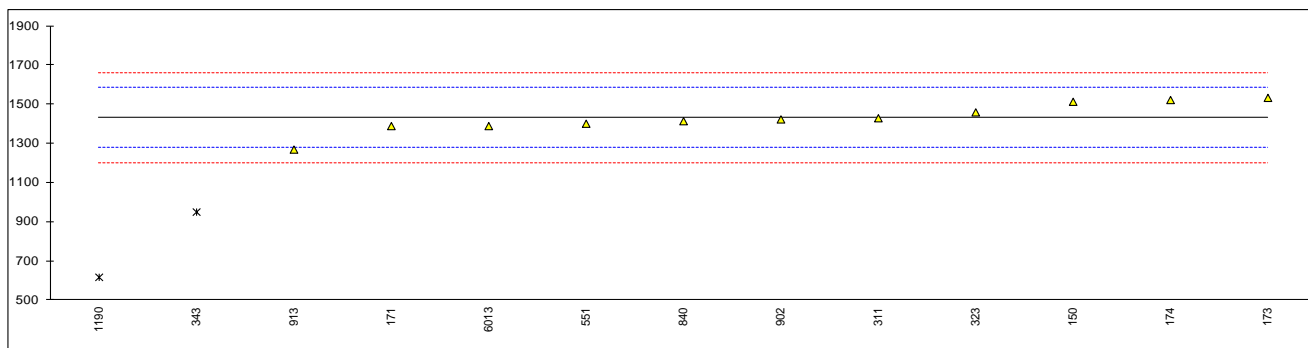
lab	method	value	mark	z(targ)	Remarks
150	INH-5290	99.78		-----	
171	ACS	99.7842		-----	
173	INH-6012	99.78		-----	
174	INH-6012	99.787		-----	
311	INH-082	99.79		-----	
323	INH-060	99.88		-----	
343	DIN55685	99.81		-----	
445		-----		-----	
551	INH-015	99.81		-----	
786		-----		-----	
840	INH-015	99.970		-----	
902	INH-129	99.793		-----	
913	D5501	99.82		-----	
994	In house	99.79		-----	
1016	DIN55685	99.93		-----	
1190	In house	99.9		-----	
6013	In house	99.78		-----	

	Group 1	Group 2
normality	OK	n.a.
n	11	4
outliers	0	0
mean (n)	99.7931	99.9200
st.dev. (n)	0.01395	0.03916
R(calc.)	0.0391	0.1096
R(lit.)	unknown	unknown



Determination of Ethanol content on sample #15240; results in mg/kg.

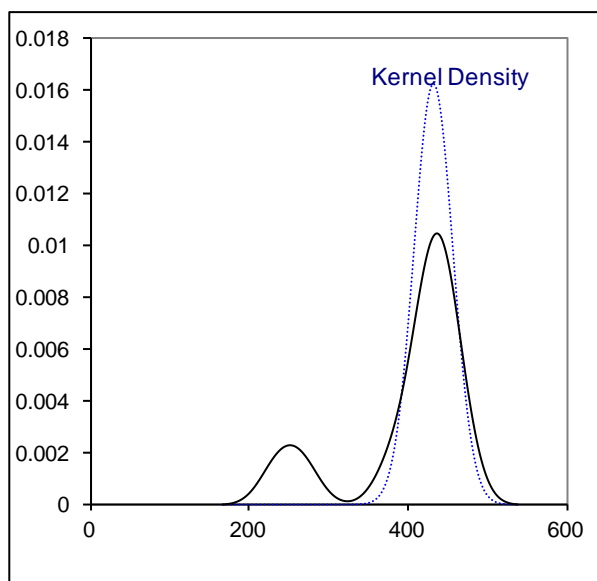
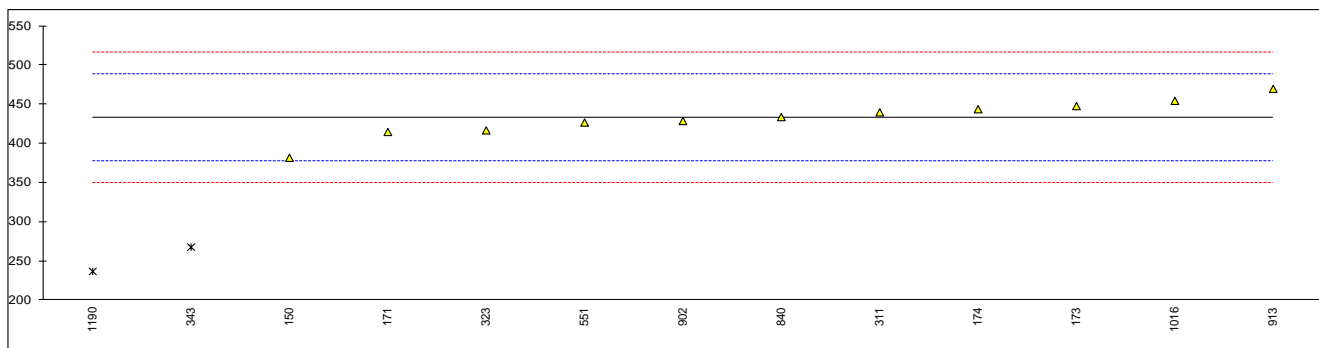
lab	method	value	mark	z(targ)	remarks
150	INH-5290	1514		1.07	
171	ACS	1390		-0.55	
173	INH-6012	1534		1.33	
174	INH-6012	1523		1.19	
311	INH-082	1430		-0.03	
323	INH-060	1460		0.36	
343	DIN55685	951	C,G(0.01)	-6.27	First reported <10
445		----		----	
551	INH-015	1402		-0.39	
786		----		----	
840	INH-015	1415		-0.22	
902	INH-129	1424		-0.10	
913	D5501	1270		-2.11	
994		----		----	
1016		----		----	
1190	In house	618	G(0.05)	-10.61	
6013	In house	1390		-0.55	
normality		OK			
n		11			
outliers		2			
mean (n)		1432.0			
st.dev. (n)		75.65			
R(calc.)		211.8			
R(Horwitz)		214.9			



Determination of n-Propanol on sample #15240; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	INH-5290	382		-1.83	
171	ACS	415		-0.64	
173	INH-6012	448		0.55	
174	INH-6012	444		0.40	
311	INH-082	440		0.26	
323	INH-060	417		-0.57	
343	DIN55685	268	C,DG(0.01)	-5.93	First reported <10
445		----		----	
551	INH-015	427		-0.21	
786		----		----	
840	INH-015	434		0.04	
902	INH-129	429		-0.14	
913	D5501	470		1.34	
994		----		----	
1016	DIN55685	454.7		0.79	
1190	In house	237	DG(0.01)	-7.05	
6013	In house	<1		<-15.54	False negative test result?

normality suspect
n 11
outliers 2
mean (n) 432.8
st.dev. (n) 23.38
R(calc.) 65.5
R(Horwitz) 77.8

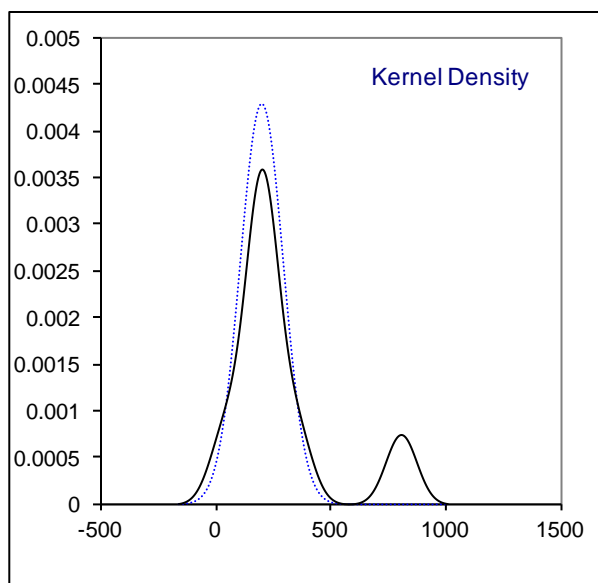
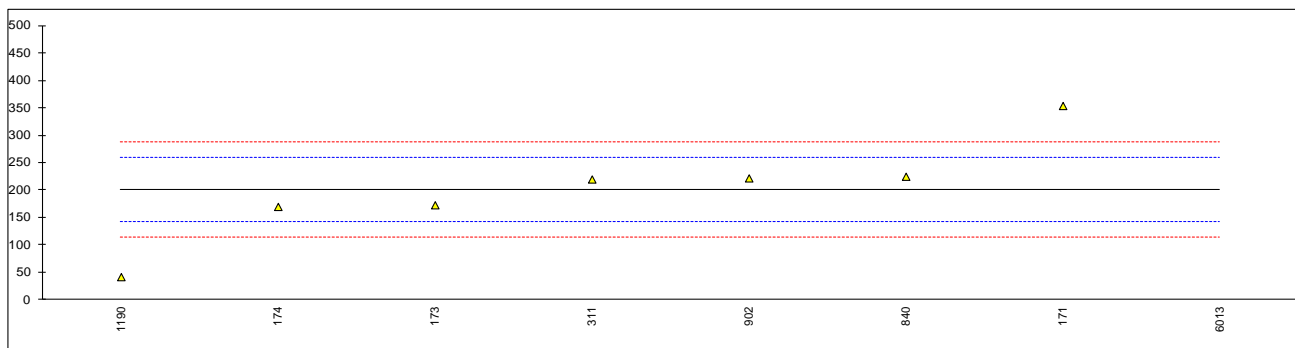


Determination of n-Butanol on sample #15240; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	INH-5290	<5		----	
171	ACS	<100		----	
173		----		----	
174	INH-6012	<10		----	
311	INH-082	<5		----	
323	INH-060	<20		----	
343	DIN55685	<10		----	
445		----		----	
551	INH-015	<5		----	
786		----		----	
840	INH-015	<5		----	
902		----		----	
913		----		----	
994		----		----	
1016	DIN55685	0		----	
1190	In house	Not Det.		----	
6013	In house	<1		----	
	normality	n.a.			
	n	10			
	outliers	n.a.			
	mean (n)	<20			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit.)	n.a.			

Determination of Other Impurities on sample #15240; results in mg/kg.

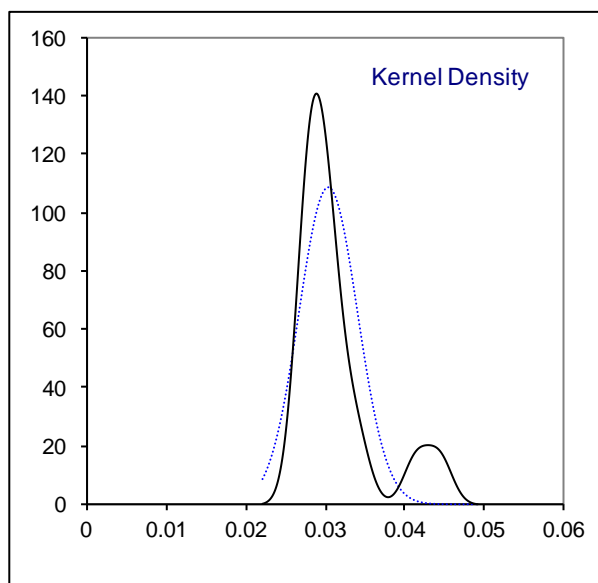
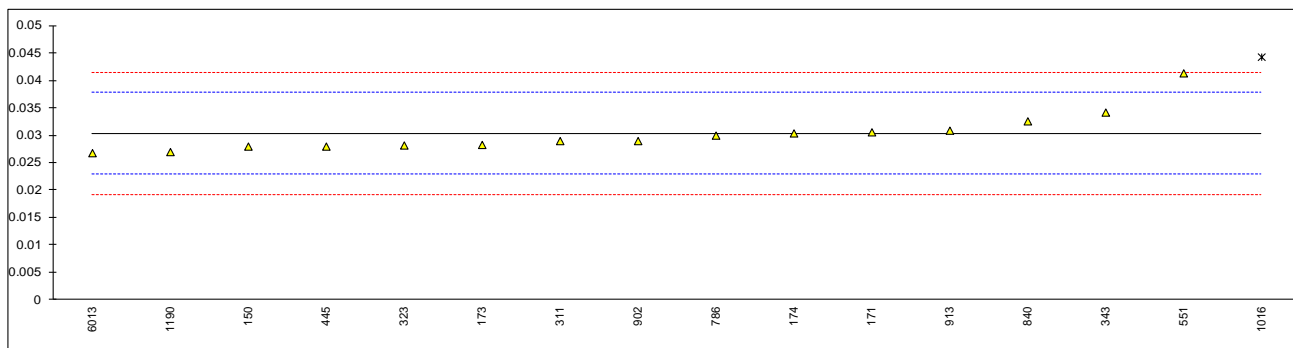
lab	method	value	mark	z(targ)	remarks
150		----		----	
171	ACS	354		5.29	
173	INH-6012	173		-0.96	
174	INH-6012	170		-1.07	
311	INH-082	220		0.66	
323		----		----	
343	DIN55685	<10		<-6.60	False negative test result?
445		----		----	
551		----		----	
786		----		----	
840	INH-015	225		0.83	
902	INH-129	222		0.73	
913		----		----	
994		----		----	
1016		----		----	
1190	In house	42		-5.49	
6013	In house	806	G(0.01)	20.91	
normality		unknown			
n		7			
outliers		1			
mean (n)		200.9			
st.dev. (n)		92.93			
R(calc.)		260.2			
R(Horwitz (4))		81.0			



Determination of Water content on sample #15240; results in %M/M.

lab	method	value	mark	z(targ)	remarks
150	E1064	0.028		-0.61	
171	D1364	0.0306		0.08	
173	E203	0.0283		-0.53	
174	D1364	0.030		0.03	
311	E203	0.029		-0.35	
323	D1364	0.0282		-0.56	
343	E1064	0.0342		1.05	
445	E1064	0.0280		-0.61	
551	E203	0.04135		2.97	
786	D1364	0.030		-0.08	
840	E1064	0.0326		0.62	
902	D1364	0.029		-0.35	
913	D1364	0.0309		0.16	
994		-----		-----	
1016	D1364	0.0443	G(0.01)	3.76	
1190	D1364	0.027		-0.88	
6013	D1364	0.0268		-0.94	

normality not OK
n 15
outliers 1
mean (n) 0.03029
st.dev. (n) 0.003671
R(calc.) 0.01028
R(D1364:02) 0.01044



APPENDIX 2

Number of participants per country

1 laboratory in AZERBAIJAN
1 laboratory in BELGIUM
1 laboratory in BRAZIL
1 laboratory in INDIA
1 laboratory in RUSSIAN FEDERATION
1 laboratory in SPAIN
2 laboratories in THE NETHERLANDS
2 laboratories in TURKEY
1 laboratory in UNITED KINGDOM
5 laboratories in UNITED STATES OF AMERICA
1 laboratory in VIETNAM

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
R(0.01)	= outlier in Rosner outlier test
R(0.05)	= straggler in Rosner outlier test
ex	= excluded from statistical calculations
E	= error in calculations
n.a.	= not applicable
W	= withdrawn on request participant
SDS	= Safety Data Sheet

Literature:

- 1 Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, April 2014
- 2 ASTM E178-02
- 3 ASTM E1301-03
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- 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.
- 14 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)