Results of Proficiency Test
Isopropanol
(Isopropyl alcohol)
December 2017

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 2017/2018 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, 19 laboratories in 15 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2017 proficiency test are presented and discussed. This report is also electronically available through the iis website 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 ISO/IEC 17025 accredited laboratory. It was decided to send one 0.5 L bottle with Isopropanol, labelled #17250 to the participants. The participants were requested to report rounded and unrounded test results. The unrounded test 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' of March 2017(iis-protocol, version 3.4). This protocol can be downloaded from the iis website 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 litres of Isopropanol were obtained from a local chemical supplier. After homogenisation, 39 amber glass bottles of 0.5 L with inner and outer caps were filled and labelled #17250. The homogeneity of the subsamples #17250 was checked by determination of the Density in accordance with ISO12185 and Water in accordance with ASTM D1364 on respectively 8 and 7 stratified randomly selected samples.

	Density at 20°C in kg/L	Water content in mg/kg
sample #17250-1	0.78503	270
sample #17250-2	0.78503	270
sample #17250-3	0.78503	270
sample #17250-4	0.78506	260
sample #17250-5	0.78504	270
sample #17250-6	0.78504	270
sample #17250-7	0.78503	250
sample #17250-8	0.78503	

Table 1: homogeneity test results of subsamples #17250

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities of the reference test 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 mg/kg
r (observed)	0.00003	22.0
reference test method	ISO12185:96	D1364:02(2012)
0.3 * R (ref. test method)	0.00015	29.3

Table 2: evaluation of the repeatabilities of subsamples #17250

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibilities of the target test methods. Therefore, homogeneity of the subsamples #17250 was assumed.

To each of the participating laboratories 1* 0.5 litre bottle, labelled #17250 was sent on November 8, 2017. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Isopropanol, packed in an 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 on sample #17250: Acidity as Acetic acid, Appearance, Inorganic Chloride, Colour Pt/Co, Density at 20°C, Specific Gravity at 20/20°C, Distillation (IBP, 50% recovered & DP), Nonvolatile Matter, Purity on dry basis, Ethanol, n-Propanol, n-Butanol and Other Impurities, Water.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results more, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical calculations.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results 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 test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test 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 reported test results (no reanalysis). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

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 Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4).
For the statistical evaluation, the *unrounded* (when available) figures were used instead of the rounded test results. Test 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. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO 5725 the original test results per determination were submitted to Dixon’s, 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. 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 them 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 test results are plotted. The corresponding laboratory numbers are on 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 reference test method. 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, EN or ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

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.

The z-scores were calculated according to:

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

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

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 dispatch of the samples. One participant did not receive the samples at all due to problems with custom clearance. From the total of 19 participants, two participants did not report any test results at all. Finally, 17 reporting laboratories submitted 157 numerical test results. Observed were 5 outlying test results, which is 3.2 %. In proficiency studies outlier percentages of 3 % - 7.5 % are quite normal.

4.1 EVALUATION PER TEST

In this section, the reported test results are discussed per test. The test methods, which are used by the various laboratories are taken into account for explaining the observed differences when possible and applicable. These methods are also in the table together with the original data. The abbreviations, used in these tables, are listed in appendix 3.

Unfortunately, a suitable reference test method providing precision data was not available for the determination of purity by GC. Therefore, the calculated reproducibility was compared to the reproducibility estimated from the Horwitz equation.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D1364) and an added designation for the year that the method was adopted or revised (e.g. D1364:02). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D1364:02(2012)). In the results tables of appendix 1 only the method number and year of adoption or revision e.g. D1364:02 are used.

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.

Acidity: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D1613:17.

Appearance: No analytical problems were observed. Almost all participants agreed about the appearance of sample #17250 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: Only four participants reported a numerical test result. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of IMPCA002:98. The average recovery of Inorganic Chloride (theoretical increment of 1.0 mg Chloride/kg) may be good (<107%), the actual Chloride content is unknown.

Colour Pt/Co: This determination was not problematic. No statistical outliers were observed. 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. 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. The calculated reproducibility is in good agreement with the requirements of ISO12185:96.

Distillation: This determination was not problematic. No statistical outliers were observed. All three calculated reproducibilities are in good agreement with the requirements of ASTM D1078:11 for the automated and the manual modes.

NVM: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D1353:13.

Purity on dry basis: Regretfully, the methods used do not provide any reproducibility limit. Therefore, no z-scores were calculated. Two statistical outliers were observed. In comparison with the previous proficiency test (iis15C17) of December 2015, the calculated reproducibility of the 2017 PT is small.

Ethanol: This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility using the Horwitz equation.
The average recovery of Ethanol (theoretical increment of 25.0 mg Ethanol/kg) may be marginal (<56%), the actual Ethanol content is unknown.

n-Propanol: 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 using the Horwitz equation.

n-Butanol: All reported test results were near or below the detection limit. Therefore, no significant conclusions were drawn.

Other Imp.: Only five participants reported a numerical test result. 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 for 4 components. Therefore, no z-scores were calculated. However, in comparison with the previous proficiency test (iis15C17) of December 2015, the calculated reproducibility of the 2017 PT is small.

Water: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in 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 reference test method and the reproducibility as found for the group of participating laboratories. The reproducibilities derived from reference test methods and the calculated reproducibilities of sample #17250 are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acidity as acetic acid	mg/kg	15	8.8	6.5	14
Appearance		14	pass	n.e.	n.e.
Chloride, inorganic as Cl	mg/kg	4	1.1	0.1	0.3
Colour Pt/Co		9	2.6	4.6	7
Density at 20°C	kg/L	16	0.7850	0.0003	0.0005
Specific Gravity at 20/20°C		13	0.7865	0.0002	0.0005
Initial Boiling Point	°C	13	82.2	0.3	1.3
50% recovered	°C	13	82.3	0.2	0.6
Dry Point	°C	13	82.4	0.5	0.9
Nonvolatile Matter	mg/100mL	10	0.5	1.3	2.1
Purity on dry basis	%M/M	12	99.962	0.019	n.a.
Ethanol	mg/kg	7	14.0	7.2	4.2
n-Propanol	mg/kg	10	243	56	48
n-Butanol	mg/kg	10	<20	n.e.	n.e.
Other Impurities	mg/kg	4	81	139	(38)
Water	mg/kg	13	251	121	95

Table 3: reproducibilities for sample #17250

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 2017 WITH THE PREVIOUS PTS.

	December 2017	*December 2015*	*November 2013*	*November 2011*
Number of reporting labs	17	17	16	13
Number of results reported	157	192	168	143
Statistical outliers	5	8	7	10
Percentage outliers	3.2%	4.2%	4.2%	7.0%

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	*December 2017*	*December 2015*	*November 2013*	*November 2011*
Acidity as acetic acid	++	++	++	++
Chloride, inorganic as Cl	++	n.e.	n.e.	n.e.
Colour Pt/Co	++	++	++	++
Density at 20°C	++	++	+	++
Specific Gravity at 20/20°C	++	++	+	++
Distillation	++	++	++	++
Nonvolatile Matter	++	++	++	++
Purity on dry basis	n.e.	n.e.	(++)	(++)
Ethanol	-	+/-	n.e.	-
n-Propanol	-	+	-	+/-
n-Butanol	n.e.	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 reference test methods is listed in the above table. The following performance categories were used:

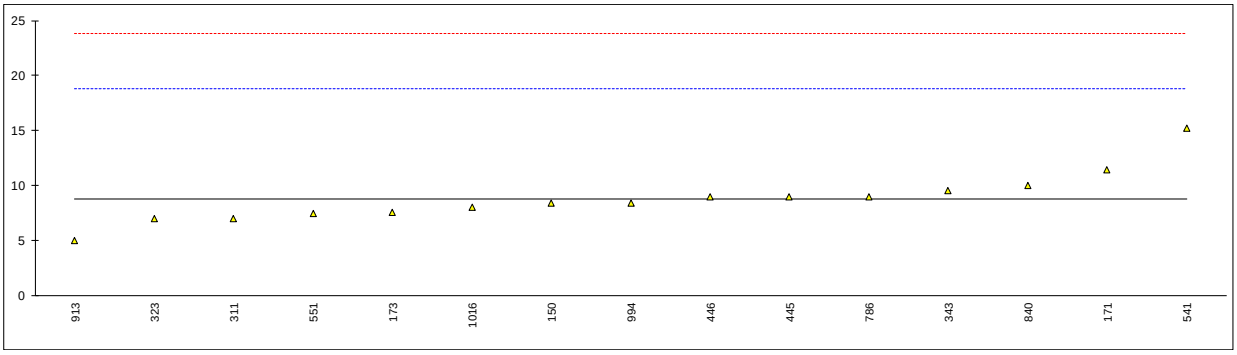
- ++: group performed much better than the reference test method
- + : group performed better than the reference test method
- +/-: group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test method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

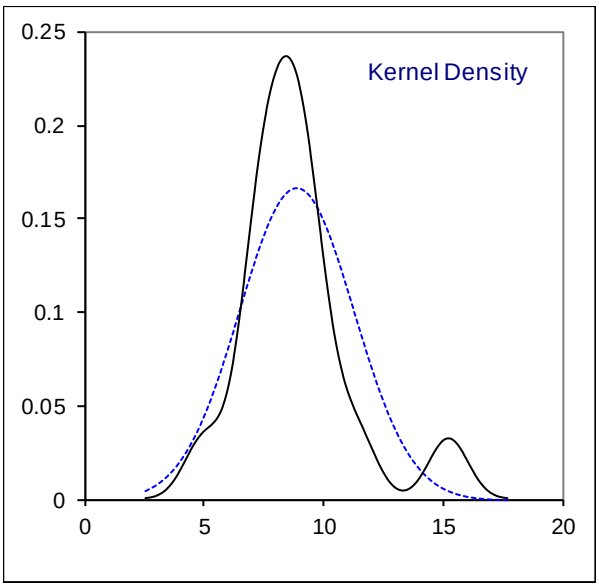
APPENDIX 1

Determination of Acidity as Acetic Acid on sample #17250; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	D1613	8.4		-0.08	
171	D1613	11.4		0.52	
173	D1613	7.6		-0.24	
311	D1613	7		-0.36	
323	D1613	7		-0.36	
343	D1613	9.6		0.16	
445	D1613	9	C	0.04	First reported as 0.0009 mg/kg
446	D1613	9		0.04	
522		-----		-----	
541	D1613	15.2		1.28	
551	D1613	7.5		-0.26	
786	D1613	9		0.04	
840	D1613	10.0		0.24	
902		-----		-----	
913	D1613	5.0		-0.76	
994	D1613	8.4		-0.08	
1016	D1613	8	C	-0.16	Reported 0.0008 mg/kg
1438		-----		-----	
6123		-----		-----	

normality	not OK
n	15
outliers	0
mean (n)	8.81
st.dev. (n)	2.313
R(calc.)	6.48
st.dev.(D1613:17)	5.000
R(D1613:17)	14





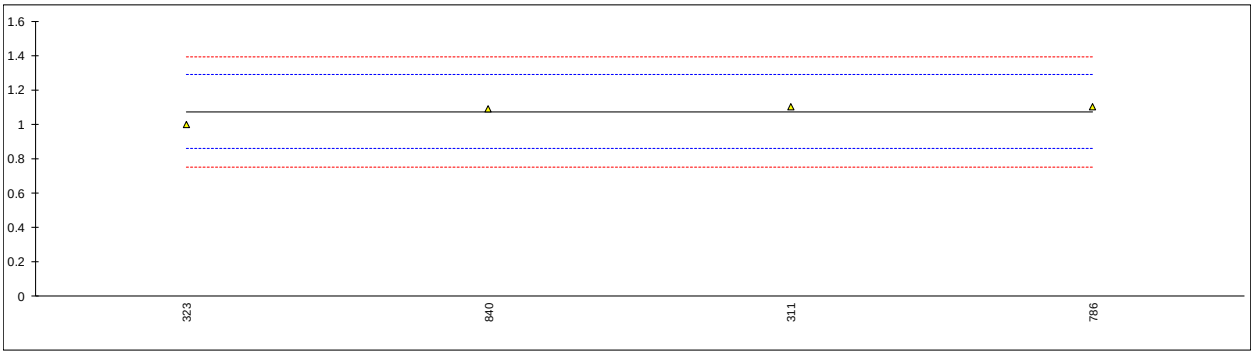
Determination of Appearance on sample #17250;

lab	method	value	mark	z(targ)	remarks
150	E2680	Pass		-----	
171	D4176	pass		-----	
173	Visual	clear & free		-----	
311	E2680	pass		-----	
323	E2680	clear & bright		-----	
343	E2680	PASS		-----	
445	E2680	Particulates		-----	
446	E2680	Pass		-----	
522		-----		-----	
541	E2680	Pass		-----	
551	NBR14954	Pass		-----	
786	E2680	Pass		-----	
840	E2680	Pass		-----	
902		-----		-----	
913	E2680	CFSM		-----	
994	Visual	pass		-----	
1016	In house	Pass		-----	
1438		-----		-----	
6123		-----		-----	

normality	n.a.
n	14
outliers	n.a.
mean (n)	Pass
st.dev. (n)	n.a.
R(calc.)	n.a.
st.dev.(E2680:16)	n.a.
R(E2680:16)	n.a.

Determination of Chloride, Inorganic as Cl on sample #17250; results in mg/kg.

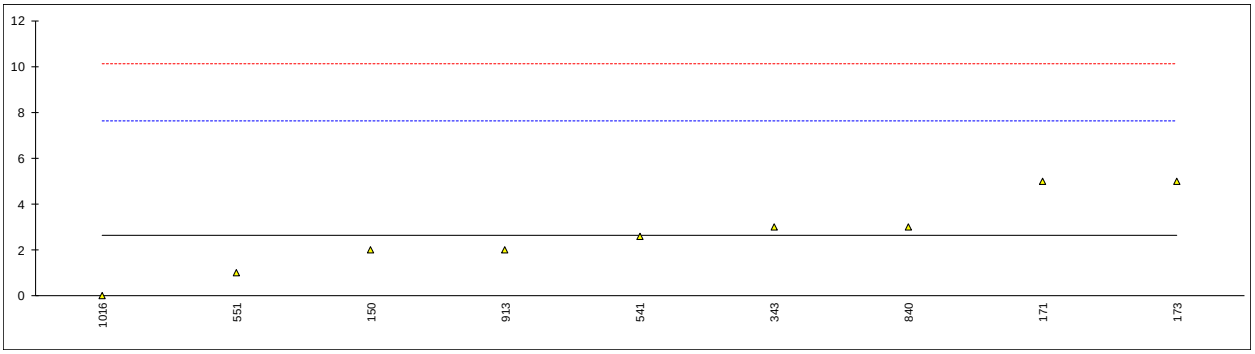
lab	method	value	mark	z(targ)	remarks
150		-----		-----	
171		-----		-----	
173		-----		-----	
311	INH-158	1.1		0.26	
323	INH-008	1		-0.68	
343		-----		-----	
445		-----		-----	
446		-----		-----	
522		-----		-----	
541		-----		-----	
551		-----		-----	
786	IMPCA002	1.1		0.26	
840	IMPCA002	1.09		0.16	
902		-----		-----	
913		-----		-----	
994		-----		-----	
1016		-----		-----	
1438		-----		-----	
6123		-----		-----	
	normality	unknown			
	n	4			
	outliers	0	Spike		
	mean (n)	1.07	1.00		Recovery < 107%
	st.dev. (n)	0.049			
	R(calc.)	0.14			
	st.dev.(IMPCA002:98)	0.107			
	R(IMPCA002:98)	0.3			

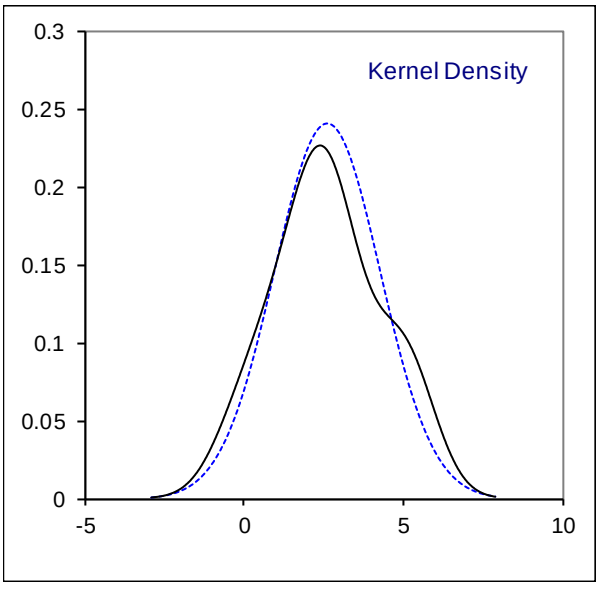


Determination of Colour Pt/Co scale on sample #17250;

lab	method	value	mark	z(targ)	remarks
150	D5386	2		-0.25	
171	D1209	5		0.95	
173	D1209	5		0.95	
311	D1209	<5		-----	
323	D1209	< 5		-----	
343	D5386	3		0.15	
445	D1209	<5		-----	
446	D1209	<5		-----	
522		-----		-----	
541	D5386	2.6		-0.01	
551	D1209	1		-0.65	
786	D1209	<5		-----	
840	D1209	3		0.15	
902		-----		-----	
913	D5386	2		-0.25	
994	D1209	<5		-----	
1016	D1209	0		-1.05	
1438		-----		-----	
6123		-----		-----	

normality	OK
n	9
outliers	0
mean (n)	2.62
st.dev. (n)	1.654
R(calc.)	4.63
st.dev.(D1209:05)	2.500
R(D1209:05)	7

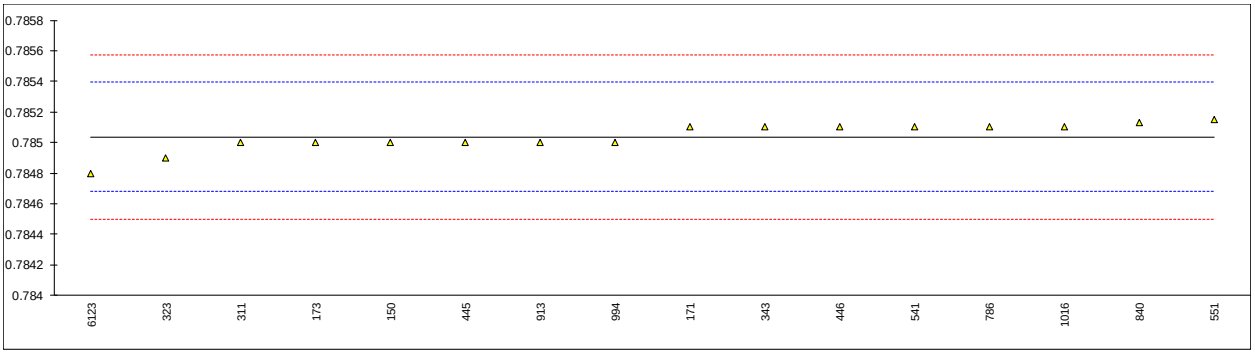


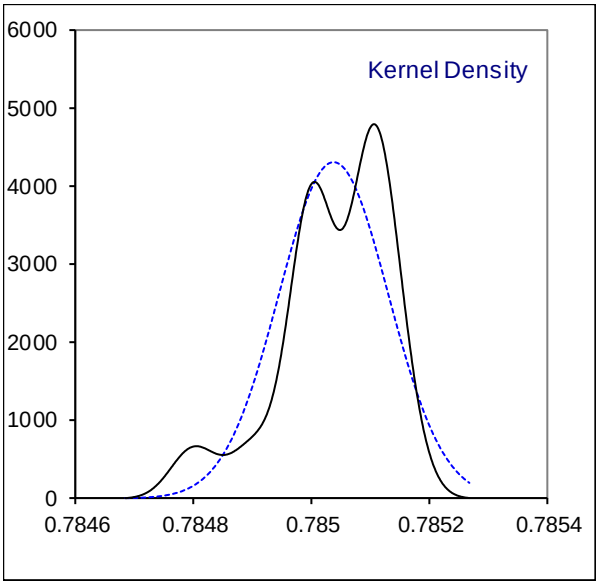


Determination of Density at 20°C on sample #17250; results in kg/L.

lab	method	value	mark	z(targ)	remarks
150	D4052	0.7850		-0.20	
171	D4052	0.7851		0.36	
173	D4052	0.7850		-0.20	
311	ISO12185	0.7850		-0.20	
323	D4052	0.7849		-0.76	
343	D4052	0.7851		0.36	
445	D4052	0.7850		-0.20	
446	D4052	0.7851		0.36	
522		-----		-----	
541	D4052	0.78510		0.36	
551	D4052	0.78515		0.64	
786	D4052	0.7851		0.36	
840	D4052	0.78513		0.52	
902		-----		-----	
913	D4052	0.7850		-0.20	
994	ISO12185	0.7850		-0.20	
1016	D4052	0.7851		0.36	
1438		-----		-----	
6123	ISO3838	0.7848	C	-1.32	First reported 0.7831

normality	suspect
n	16
outliers	0
mean (n)	0.78504
st.dev. (n)	0.000093
R(calc.)	0.00026
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

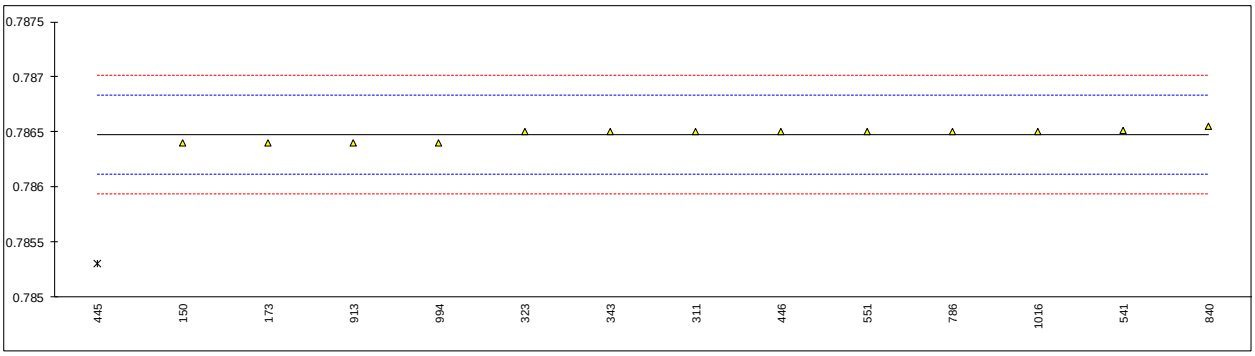


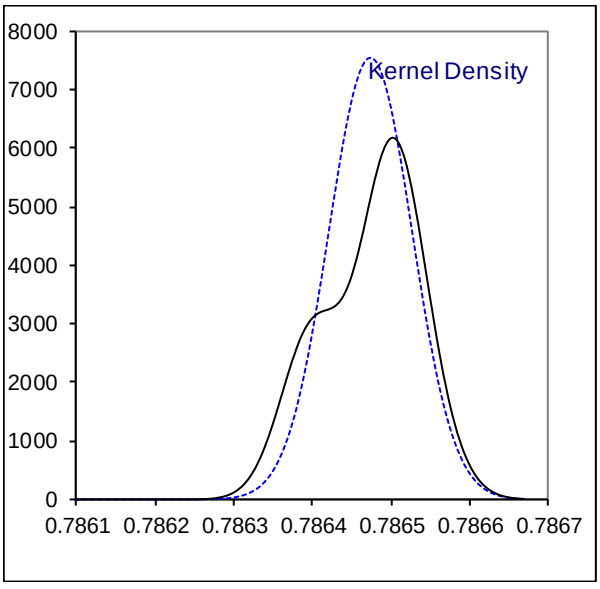


Determination of Specific Gravity at 20/20°C on sample #17250;

lab	method	value	mark	z(targ)	remarks
150	D4052	0.7864		-0.41	
171		-----		-----	
173	D4052	0.7864		-0.41	
311	ISO12185	0.7865		0.15	
323	D4052	0.7865		0.15	
343	D4052	0.7865		0.15	
445	D4052	0.7853	D(0.01)	-6.57	
446	D4052	0.7865		0.15	
522		-----		-----	
541	D4052	0.78651		0.20	
551	D4052	0.7865		0.15	
786	D4052	0.7865		0.15	
840	D4052	0.78655		0.43	
902		-----		-----	
913	D4052	0.7864		-0.41	
994	ISO12185	0.7864		-0.41	
1016	D4052	0.7865		0.15	
1438		-----		-----	
6123		-----		-----	

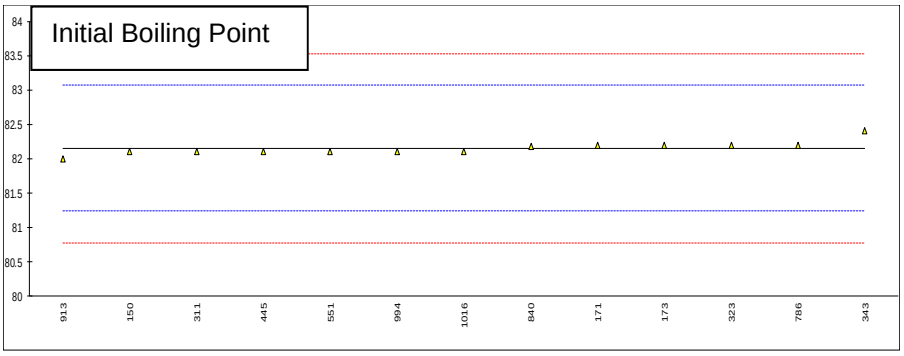
normality	OK
n	13
outliers	1
mean (n)	0.78647
st.dev. (n)	0.000053
R(calc.)	0.00015
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

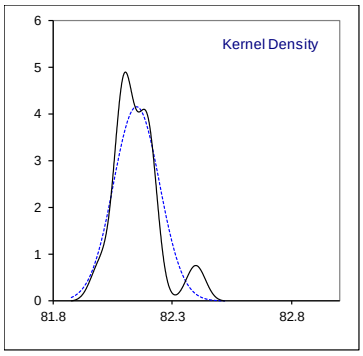


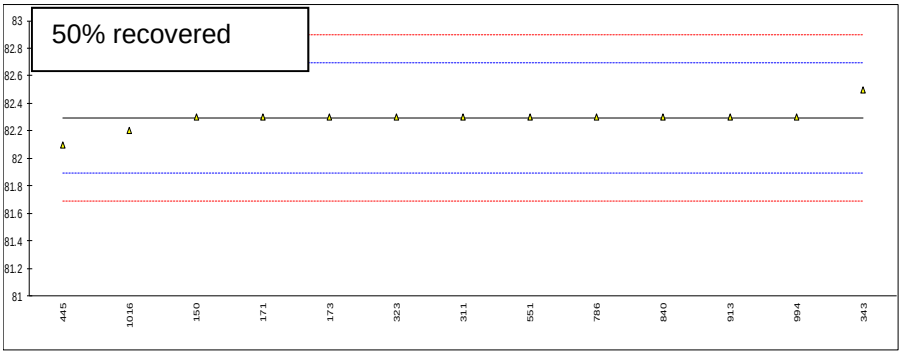


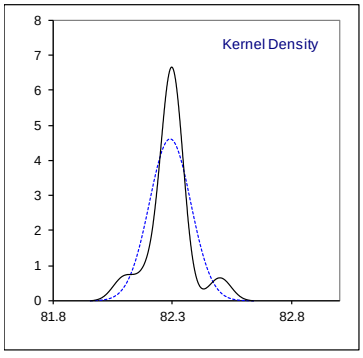
Determination of Distillation at 760 mmHg on sample #17250; results in °C.

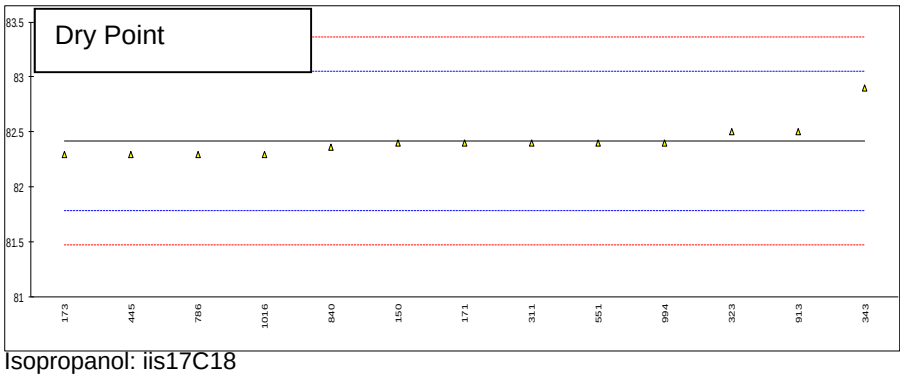
lab	method	IBP	mark	z(targ)	50%rec	mark	z(targ)	DP	mark	z(targ)
150	D1078-automated	82.1		-0.11	82.3		0.04	82.4		-0.06
171	D1078-automated	82.2		0.10	82.3		0.04	82.4		-0.06
173	D1078-automated	82.2		0.10	82.3		0.04	82.3		-0.38
311	D1078-automated	82.1		-0.11	82.3		0.04	82.4		-0.06
323	D1078-manual	82.2		0.10	82.3		0.04	82.5		0.25
343	D1078-automated	82.4		0.54	82.5		1.03	82.9		1.52
445	D1078-manual	82.1		-0.11	82.1		-0.96	82.3		-0.38
446		-----		-----	-----		-----	-----		-----
522		-----		-----	-----		-----	-----		-----
541		-----		-----	-----		-----	-----		-----
551	D1078-automated	82.1		-0.11	82.3		0.04	82.4		-0.06
786	D1078-manual	82.2		0.10	82.3		0.04	82.3		-0.38
840	D1078-automated	82.18		0.06	82.30		0.04	82.36		-0.19
902		-----		-----	-----		-----	-----		-----
913	D1078-manual	82.0		-0.33	82.30		0.04	82.50		0.25
994	D1078-manual	82.1		-0.11	82.3		0.04	82.4		-0.06
1016	D1078-automated	82.1		-0.11	82.2		-0.46	82.3		-0.38
1438		-----		-----	-----		-----	-----		-----
6123		-----		-----	-----		-----	-----		-----
	normality	not OK			not OK			not OK		
	n	13			13			13		
	outliers	0			0			0		
	mean (n)	82.15			82.29			82.42		
	st.dev. (n)	0.096			0.086			0.160		
	R(calc.)	0.27			0.24			0.45		
	st.dev.(D1078-A:11)	0.458			0.201			0.315		
	R(D1078-A:11)	1.28			0.56			0.88		
	Compare R(D1078-M:11)	0.88			0.53			1.07		

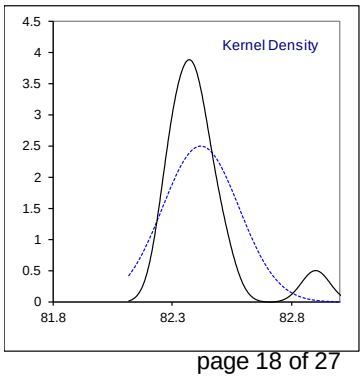








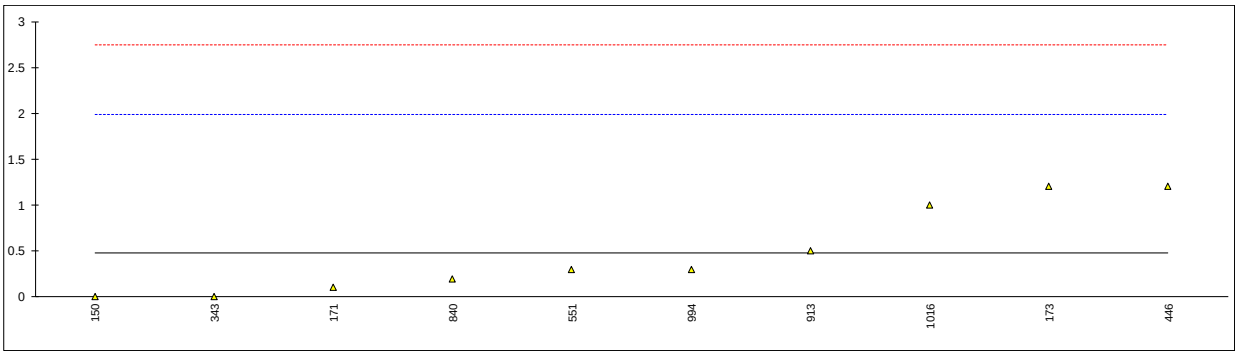


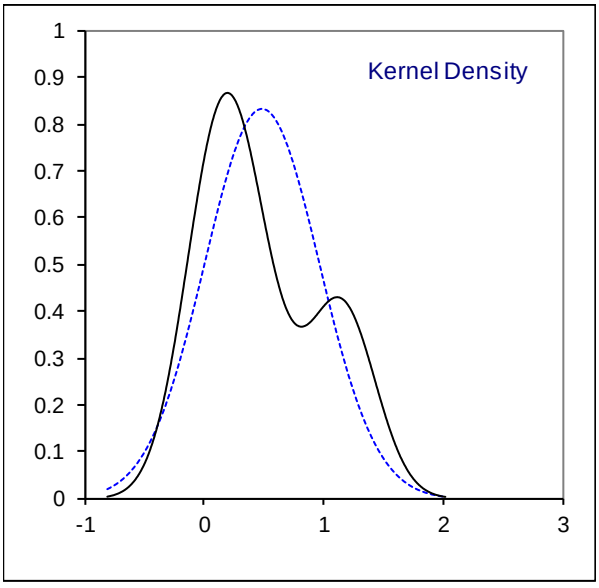


Determination of Nonvolatile Matter on sample #17250; results in mg/100 mL.

lab	method	value	mark	z(targ)	remarks
150	D1353	0.0		-0.64	
171	D1353	0.1		-0.50	
173	D1353	1.2		0.96	
311	D1353	<1		-----	
323	D1353	< 1		-----	
343	D1353	0.0000		-0.64	
445	D1353	<1		-----	
446	D1353	1.2		0.96	
522		-----		-----	
541	D1353	<0.1		-----	
551	D1353	0.3		-0.24	
786		-----		-----	
840	D1353	0.2		-0.37	
902		-----		-----	
913	D1353	0.5		0.03	
994	D1353	0.3		-0.24	
1016	D1353	1.0		0.69	
1438		-----		-----	
6123		-----		-----	

normality	OK
n	10
outliers	0
mean (n)	0.48
st.dev. (n)	0.478
R(calc.)	1.34
st.dev.(D1353:13)	0.754
R(D1353:13)	2.11

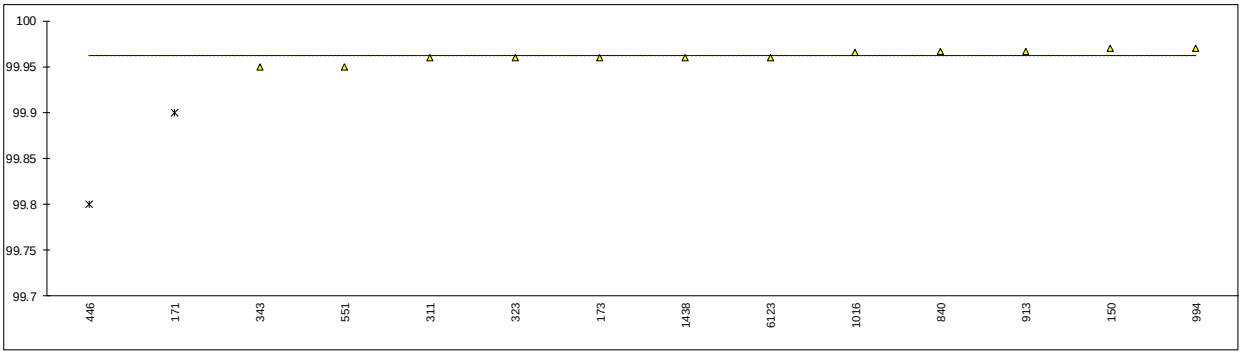


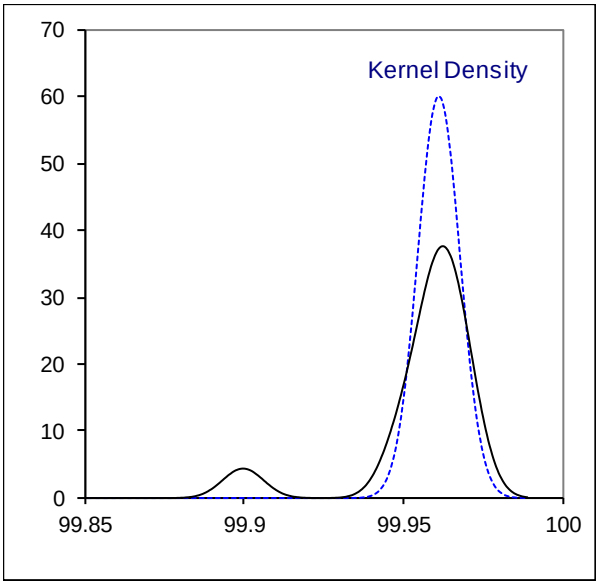


Determination of Purity on dry basis on sample #17250, results in %M/M.

lab	method	value	mark	z(targ)	remarks
150	INH-5290	99.97		-----	
171	INH-IPA	99.9	D(0.01)	-----	
173	INH-6012	99.96		-----	
311	INH-082	99.96		-----	
323	INH-060	99.96		-----	
343	DIN55685	99.95		-----	
445		-----		-----	
446	INH-595	99.80	D(0.01)	-----	
522		-----		-----	
541		-----		-----	
551	INH-6012	99.95		-----	
786		-----		-----	
840	DIN55685	99.966		-----	
902		-----		-----	
913	D5501	99.967		-----	
994		99.97		-----	
1016	DIN55685	99.965		-----	
1438	In house	99.96		-----	
6123	In house	99.96		-----	

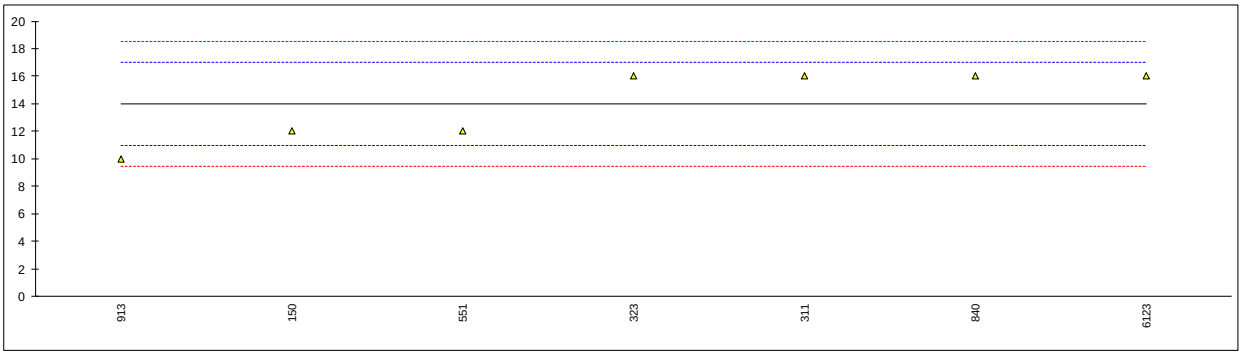
normality	OK
n	12
outliers	2
mean (n)	99.9615
st.dev. (n)	0.006628
R(calc.)	0.019
st.dev.(lit.)	n.a.
R(lit.)	n.a.
Compare R(iis15C17)	0.039

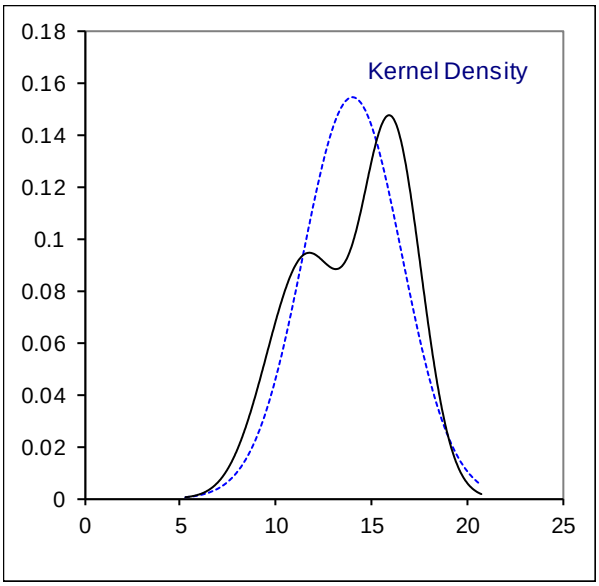




Determination of Ethanol content on sample #17250; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	INH-5290	12		-1.33	
171	INH-IPA	<100		-----	
173		-----		-----	
311	INH-082	16		1.33	
323	INH-060	16		1.33	
343	DIN55685	<10		-----	
445		-----		-----	
446		-----		-----	
522		-----		-----	
541		-----		-----	
551	INH-6012	12		-1.33	
786		-----		-----	
840	DIN55685	16		1.33	
902		-----		-----	
913	D5501	10		-2.66	
994		-----		-----	
1016		-----		-----	
1438		-----		-----	
6123	In house	16		1.33	
	normality	unknown			
	n	7			
	outliers	0	Spike		
	mean (n)	14.0	25.0		Recovery <56%
	st.dev. (n)	2.58			
	R(calc.)	7.2			
	st.dev.(Horwitz)	1.51			
	R(Horwitz)	4.2			

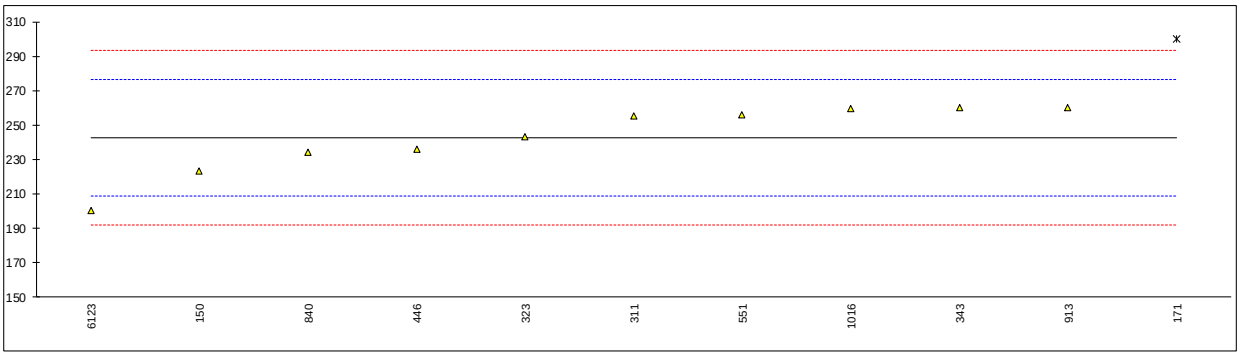


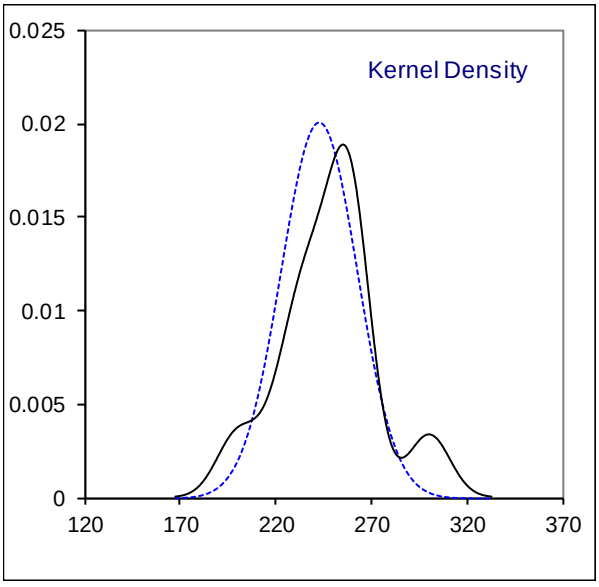


Determination of n-Propanol on sample #17250; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	INH-5290	223		-1.16	
171	INH-IPA	300	D(0.05)	3.38	
173		-----		-----	
311	INH-082	255		0.73	
323	INH-060	243		0.02	
343	DIN55685	260		1.02	
445		-----		-----	
446	INH-595	236		-0.39	
522		-----		-----	
541		-----		-----	
551	INH-6012	256		0.79	
786		-----		-----	
840	DIN55685	234		-0.51	
902		-----		-----	
913	D5501	260		1.02	
994		-----		-----	
1016	DIN55685	259.5		0.99	
1438		-----		-----	
6123	In house	200	C	-2.51	First reported 138

normality	OK
n	10
outliers	1
mean (n)	242.6
st.dev. (n)	19.84
R(calc.)	55.5
st.dev.(Horwitz)	16.99
R(Horwitz)	47.6



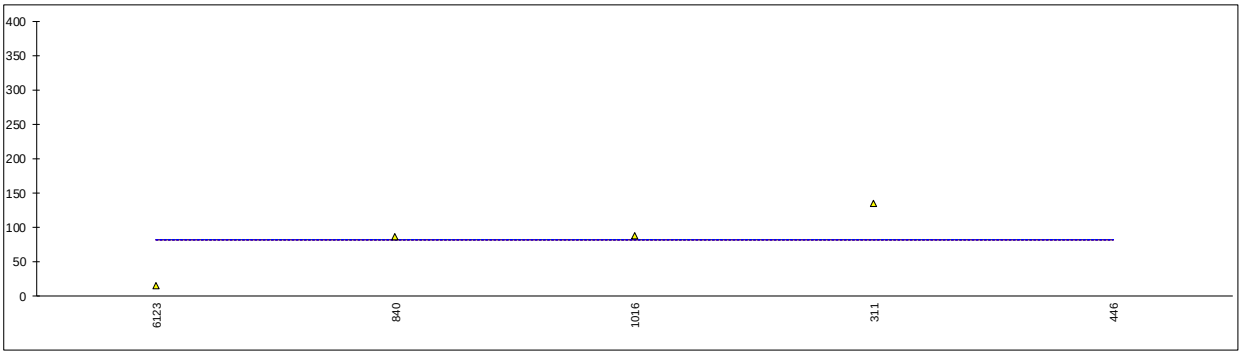


Determination of n-Butanol on sample #17250; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	INH-5290	<5		-----	
171	INH-IPA	<100		-----	
173		-----		-----	
311	INH-082	<5		-----	
323	INH-060	< 10		-----	
343	DIN55685	<10		-----	
445		-----		-----	
446	INH-595	<20		-----	
522		-----		-----	
541		-----		-----	
551	INH-6012	<5		-----	
786		-----		-----	
840	DIN55685	<5		-----	
902		-----		-----	
913	D5501	<5.0		-----	
994		-----		-----	
1016	DIN55685	<5		-----	
1438		-----		-----	
6123	In house	<10		-----	
	normality	n.a			
	n	10			
	outliers	n.a			
	mean (n)	<20			
	st.dev. (n)	n.a			
	R(calc.)	n.a.			
	st.dev.(Horwitz)	n.a.			
	R(Horwitz)	n.a.			

Determination of Other Impurities on sample #17250; results in mg/kg.

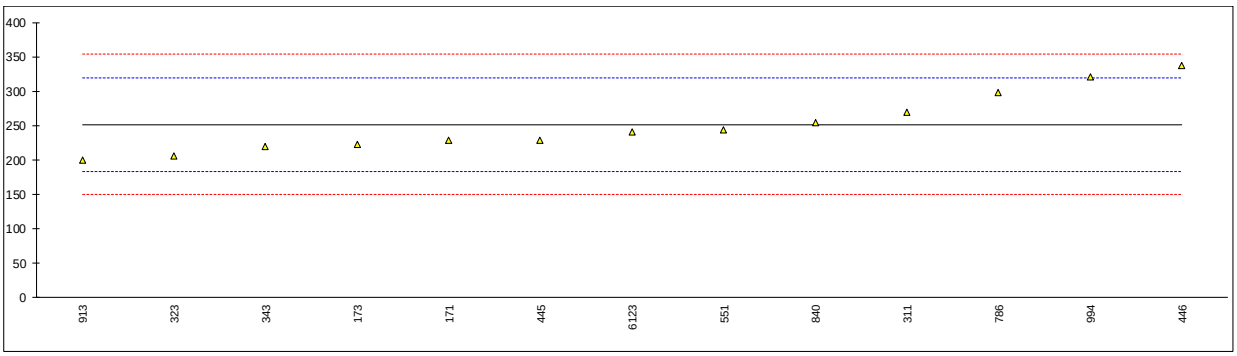
lab	method	value	mark	z(targ)	remarks
150		-----		-----	
171	INH-IPA	<100		-----	
173		-----		-----	
311	INH-082	135		-----	
323		-----		-----	
343	DIN55685	<10		-----	
445		-----		-----	
446	INH-595	1790	D(0.01)	-----	
522		-----		-----	
541		-----		-----	
551		-----		-----	
786		-----		-----	
840	DIN55685	87		-----	
902		-----		-----	
913		-----		-----	
994		-----		-----	
1016	DIN55685	88.7		-----	
1438		-----		-----	
6123	In house	15		-----	
	normality	unknown			
	n	4			
	outliers	1			
	mean (n)	81.4			
	st.dev. (n)	(49.55)			
	R(calc.)	138.7			
	st.dev.(Horwitz (4))	(13.44)			
	R(Horwitz (4))	(37.6)			
	Compare R(iis15C17)	269.2			

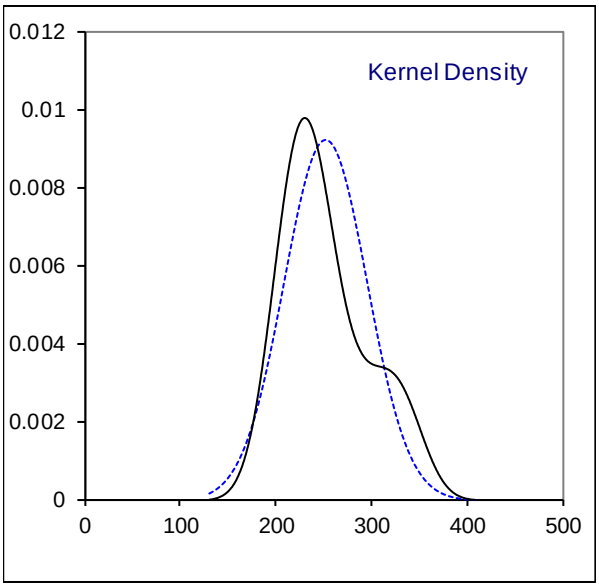


Determination of Water, titrimetric on sample #17250; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150		-----		-----	
171	D1364	228		-0.69	
173	E1064	222		-0.87	
311	D1364	270		0.54	
323	D1364	206		-1.34	
343	E1064	220		-0.93	
445	E203	229	C	-0.66	First reported 0.0229 mg/kg
446	D1364	338		2.54	
522		-----		-----	
541		-----		-----	
551	E203	244		-0.22	
786	D1364	298		1.37	
840	E1064	254		0.07	
902		-----		-----	
913	D1364	200		-1.52	
994	D1364	320.1		2.02	
1016		-----		-----	
1438		-----		-----	
6123	ISO12937	241.0475		-0.31	

normality	OK
n	13
outliers	0
mean (n)	251.5498
st.dev. (n)	43.30890
R(calc.)	121.2649
st.dev.(D1364:02)	33.98640
R(D1364:02)	95.1619





APPENDIX 2

Number of participants per country

1 lab in ARGENTINA
1 lab in AZERBAIJAN
1 lab in BELGIUM
1 lab in BRAZIL
1 lab in INDIA
1 lab in ISRAEL
1 lab in MEXICO
2 labs in NETHERLANDS
1 lab in ROMANIA
1 lab in RUSSIAN FEDERATION
1 lab in SPAIN
1 lab in TURKEY
2 labs in UNITED KINGDOM
3 labs in UNITED STATES OF AMERICA
1 lab in VIETNAM

APPENDIX 3

Abbreviations:

C	= final test result after checking of first reported suspect test 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
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= probably an error in calculations
U	= test result probably reported in a different unit
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= Safety Data Sheet

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, March 2017
2 ASTM E178:02
3 ASTM E1301:03
4 ISO 13528:05
5 ISO 5725:86
6 ISO 5725, parts 1-6, 1994
7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
9 IP 367:84
10 DIN 38402 T41/42
11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
12 J.N. Miller, Analyst, 118, 455, (1993)
13 Analytical Methods Committee Technical Brief, No 4 January 2001
14 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry 2002, Analyst, 127, 1359-1364 (2002)
15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)