Results of Proficiency Test
Isopropanol
(Isopropyl alcohol)
December 2021

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

1 INTRODUCTION

Since 2003 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Isopropanol according to the latest version of specification ASTM D770 once every two years. During the annual program 2021/2022 it was decided to continue the round robin for the analysis of Isopropanol.

In this interlaboratory study 14 laboratories in 9 different countries registered for participation. See appendix 2 for the number of participants per country. In this report the results of this Isopropanol 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 organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send one sample Isopropanol in a 0.5L glass bottle labelled #21270 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/IEC17043:2010. 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 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 June 2018 (iis-protocol, version 3.5). This protocol is electronically available through 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

A batch of approximately 15 liters of Isopropanol was obtained from a local chemical supplier. After homogenization 30 amber glass bottles of 0.5L were filled and labelled #21270.
The homogeneity of the subsamples was checked by determination of Density at 20°C in accordance with ISO12185 on 8 stratified randomly selected subsamples.

	Density at 20°C in kg/L
sample #21270-1	0.78499
sample #21270-2	0.78500
sample #21270-3	0.78501
sample #21270-4	0.78501
sample #21270-5	0.78501
sample #21270-6	0.78501
sample #21270-7	0.78501
sample #21270-8	0.78501

Table 1: homogeneity test results of subsamples #21270

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Density at 20°C in kg/L
r (observed)	0.00002
reference test method	ISO12185:96
0.3 x R (reference test method)	0.00015

Table 2: evaluation of the repeatability of subsamples #21270

The calculated repeatability is in agreement with 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample Isopropanol labelled #21270 was sent on November 10, 2021. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYZES

The participants were requested to determine: Acidity as Acetic acid, Appearance, Inorganic Chloride as Cl, Color Pt/Co, Density at 20°C, Specific Gravity 20/20°C, Distillation (IBP, 50% recovered & DP), Nonvolatile matter, Purity by GC on dry basis, Ethanol, n-Propanol, n-Butanol, other Impurities and Water.

It was explicitly requested to treat the sample as if it was a routine sample and to report the test results using the indicated units on the report form and not to round the test results 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 evaluations.

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 (when applicable) 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 analyzes and original test results are placed under 'Remarks' in the 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 June 2018 (iis-protocol, version 3.5).
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.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner’s outlier test can be used. 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. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

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 (derived from e.g. ISO or ASTM test methods), 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, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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. Therefore, 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 dispatch of the samples. One participant reported the test results after the finals reporting date and one other participant did not report any test results.

In total 13 participants submitted 133 numerical test results. Observed were 2 outlying test results, which is 1.5%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Not all 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.

4.1 EVALUATION PER TEST

In this section the reported test results are discussed per test. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 3.

In the iis PT reports ASTM test methods are referred to with a number and an added designation for the year that the test method was adopted or revised (e.g. D1209:05). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D1209:05(2019)). In the test results tables of appendix 1 only the method number and year of adoption or revision (e.g. D1209:05) will be used.

Unfortunately, a suitable reference test method providing the precision data is not available for all determinations. For these tests the calculated reproducibility was compared against the estimated reproducibility calculated with the Horwitz equation.

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

Appearance: This determination was not problematic. All reporting participants agreed on the appearance as Pass or Clear and Bright.

Inorganic Chloride as Cl: Only four participants reported a test result. They agree on a level of <0.3 mg/kg. Therefore, no z-scores were calculated.

Color Pt/Co: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D1209:05(2019).

Density at 20°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in 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 agreement with the requirements of ISO12185:96.

Distillation at 760 mmHg: This determination was not problematic. No statistical outliers were observed. All three calculated reproducibilities are in agreement with the requirements of ASTM D1078:11(2019) for the automated and the manual modes.

Nonvolatile matter: This determination was not problematic. All reporting participants agreed on a level of <1 mg/100mL. Therefore, no z-scores were calculated.

Purity by GC on dry basis: Regretfully, the methods used do not provide any reproducibility limit. Therefore, no z-scores were calculated. One statistical outlier was observed. In comparison with the previous proficiency test (iis19C18) of December 2019, the calculated reproducibility of the 2021 PT is somewhat larger.

Ethanol: This determination was very problematic. No statistical outliers were observed. Due to the large variation in test results, no z-scores were calculated.

n-Propanol: This determination was very problematic. No statistical outliers were observed. Due to the large variation in test results, no z-scores were calculated.

n-Butanol: This determination was very problematic. No statistical outliers were observed. Due to the large variation in test results, no z-scores were calculated.

Other impurities: Only two participants reported a test result. Therefore, no z-scores were calculated.

Water: This determination was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of ASTM E1064:16.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the reproducibility derived from reference test methods (in casu ASTM and ISO test methods) are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Acidity as Acetic Acid	mg/kg	13	8.3	4.9	14
Appearance		13	Pass	n.a.	n.a.
Inorganic Chloride as Cl	mg/kg	4	<0.3	n.e.	n.e.
Color Pt/Co		8	1.4	2.9	7
Density at 20°C	kg/L	13	0.7850	0.0002	0.0005
Specific Gravity at 20/20°C		12	0.7865	0.0002	0.0005
Initial Boiling Point	°C	11	82.1	0.3	1.3
50% recovered	°C	11	82.3	0.2	0.6
Dry Point	°C	11	82.4	0.3	0.9
Nonvolatile matter	mg/100mL	10	<1	n.e.	n.e.
Purity by GC on dry basis	%M/M	11	99.970	0.019	n.e.
Ethanol	mg/kg	6	86	49	(20)

Parameter	unit	n	average	2.8 * sd	R(lit)
n-Propanol	mg/kg	7	93	70	(21)
n-Butanol	mg/kg	7	26	23	(7)
Other impurities	mg/kg	2	n.e.	n.e.	n.e.
Water	mg/kg	12	146	47	23

Table 3: performance evaluation sample #21270

For values in brackets no z-scores are calculated.

Without further statistical calculations it can be concluded that for many tests there is a good compliance of the group of participants with the reference test methods. The tests that are problematic have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2021 WITH THE PREVIOUS PTS

	December 2021	December 2019	December 2017	December 2015	November 2013
Number of reporting laboratories	13	15	17	17	16
Number of test results	133	148	157	192	168
Number of statistical outliers	2	3	5	8	7
Percentage of statistical outliers	1.5%	2.0%	3.2%	4.2%	4.2%

Table 4: comparison with previous proficiency tests

In proficiency tests outlier percentages of 3 % - 7.5 % are quite normal.

The performance of the determinations of the proficiency tests was compared against the requirements of the reference test methods. The conclusions are given the following table.

Parameter	December 2021	December 2019	December 2017	December 2015	November 2013
Acidity as Acetic Acid	++	+	++	++	++
Inorganic Chloride as Cl	n.e.	--	++	n.e.	n.e.
Color Pt/Co	++	+	++	++	++
Density at 20°C	++	++	++	++	+
Specific Gravity at 20/20°C	++	++	++	++	+
Distillation at 760 mmHg	++	++	++	++	++
Nonvolatile matter	n.e.	++	++	++	++
Purity by GC on dry basis	n.e.	n.e.	n.e.	n.e.	n.e.
Ethanol	(--)	+	-	+/-	n.e.
n-Propanol	(--)	--	-	+	-
n-Butanol	(--)	n.e.	n.e.	n.e.	n.e.
Other impurities	n.e.	-	n.e.	--	--
Water	--	-	-	+/-	+/-

Table 5: comparison determinations against the reference test methods

For values in brackets no z-scores are calculated.

The following performance categories were used:

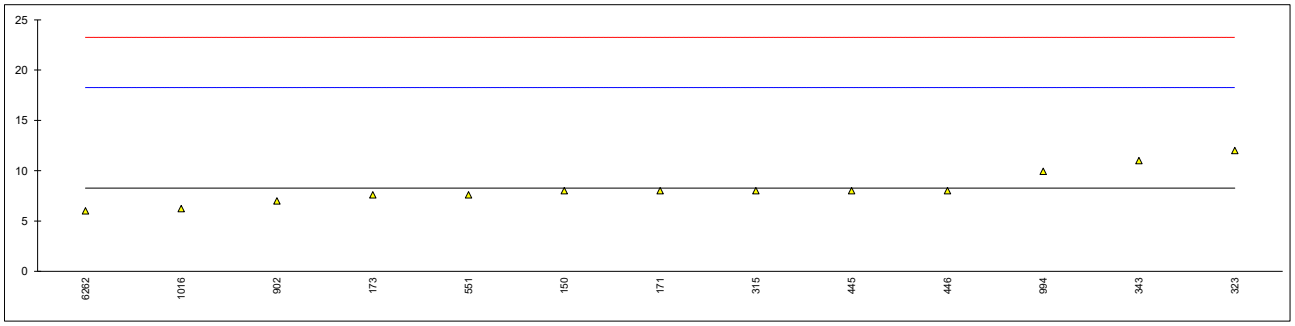
++ : group performed much better than the reference test method
+ : group performed better than the reference test method
+/- : group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.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

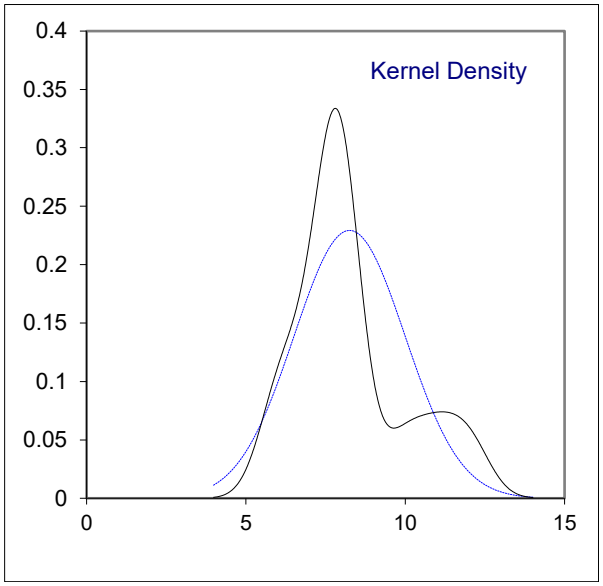
APPENDIX 1

Determination of Acidity as Acetic Acid on sample #21270; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	D1613	8		-0.05	
171	D1613	8		-0.05	
173	D1613	7.6		-0.13	
315	D1613	8		-0.05	
323	D1613	12		0.75	
343	D1613	11		0.55	
445	D1613	8		-0.05	
446	D1613	8		-0.05	
551	D1613	7.6		-0.13	
902	D1613	7		-0.25	
913		-----		-----	
994	D1613	9.93		0.33	
1016	D1613	6.22		-0.41	
6262	D1613	6		-0.45	

normality	suspect
n	13
outliers	0
mean (n)	8.26
st.dev. (n)	1.740
R(calc.)	4.87
st.dev.(D1613:17)	5
R(D1613:17)	14





Determination of Appearance on sample #21270;

lab	method	value	mark	z(targ)	remarks
150	E2680	Pass		-----	
171	E2680	Pass		-----	
173	E2680	Pass		-----	
315	E2680	pass		-----	
323	D2679	C&B		-----	
343	E2680	PASS		-----	
445	E2680	C&B		-----	
446	E2680	Pass		-----	
551	E2680	Pass		-----	
902	E2680	PASS		-----	
913		-----		-----	
994	Visual	c&b		-----	
1016	Visual	Pass		-----	
6262	Visual	Cl. & Br.		-----	
	n	13			
	mean (n)	Pass (Clear and Bright)			

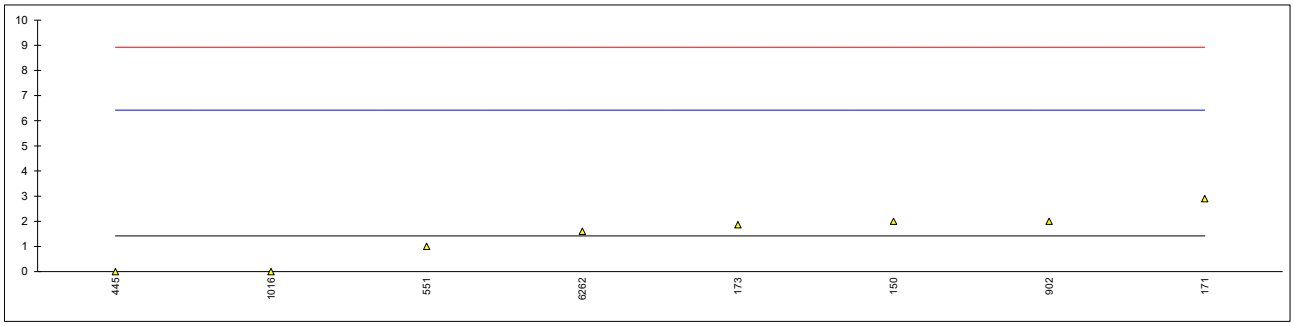
Determination of Inorganic Chloride as Cl on sample #21270; results in mg/kg.

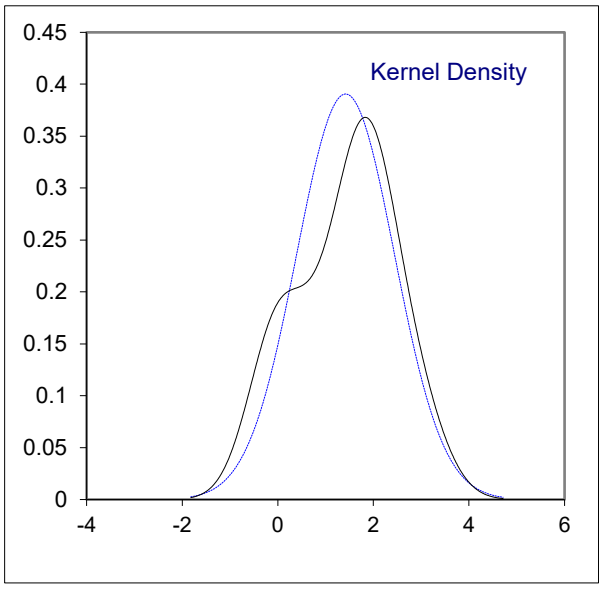
lab	method	value	mark	z(targ)	remarks
150	IMPCA002	<0.25		-----	
171	IMPCA002	0.1		-----	
173		-----		-----	
315	INH-158	<0.2		-----	
323	IMPCA002	< 0.3		-----	
343		-----		-----	
445		-----		-----	
446		-----		-----	
551		-----		-----	
902		-----		-----	
913		-----		-----	
994		-----		-----	
1016		-----		-----	
6262		-----		-----	
	n	4			
	mean (n)	<0.3			

Determination of Color Pt/Co on sample #21270;

lab	method	value	mark	z(targ)	remarks
150	D5386	2		0.23	
171	D5386	2.9		0.59	
173	D5386	1.86		0.18	
315	D1209	<5		-----	
323	D1209	< 5		-----	
343		-----		-----	
445	D1209	0.0		-0.57	
446	D1209	<5		-----	
551	D1209	1		-0.17	
902	D1209	2		0.23	
913		-----		-----	
994	D1209	<5		-----	
1016	D1209	0		-0.57	
6262	D5386	1.6		0.07	

normality	unknown
n	8
outliers	0
mean (n)	1.42
st.dev. (n)	1.021
R(calc.)	2.86
st.dev.(D1209:05)	2.5
R(D1209:05)	7

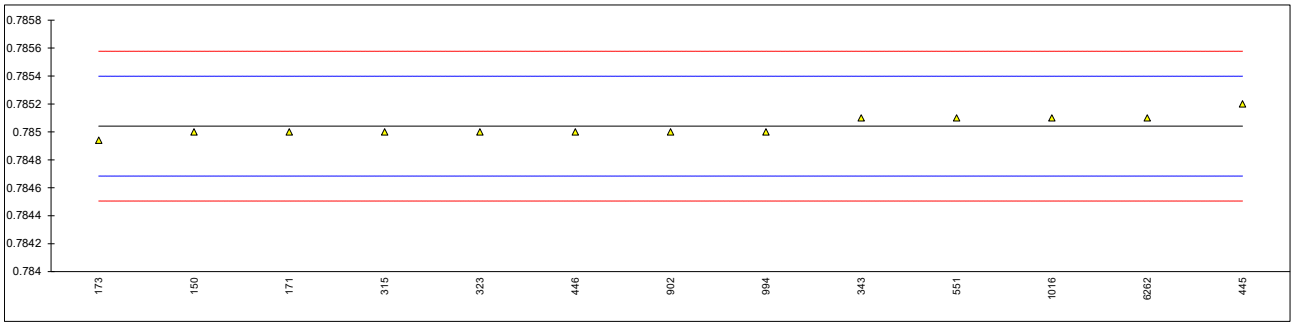


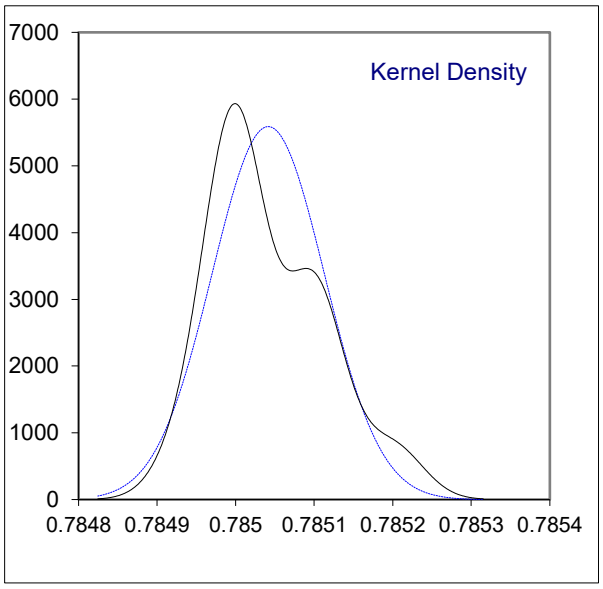


Determination of Density at 20°C on sample #21270; results in kg/L.

lab	method	value	mark	z(targ)	remarks
150	D4052	0.7850		-0.23	
171	D4052	0.7850		-0.23	
173	D4052	0.78494		-0.57	
315	D4052	0.7850		-0.23	
323	D4052	0.7850		-0.23	
343	D4052	0.7851		0.33	
445	ISO12185	0.7852		0.89	
446	D4052	0.7850		-0.23	
551	D4052	0.7851		0.33	
902	ISO12185	0.7850		-0.23	
913		-----		-----	
994	ISO12185	0.7850		-0.23	
1016	D4052	0.7851		0.33	
6262	ISO12185	0.7851		0.33	

normality	OK
n	13
outliers	0
mean (n)	0.78504
st.dev. (n)	0.000071
R(calc.)	0.00020
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

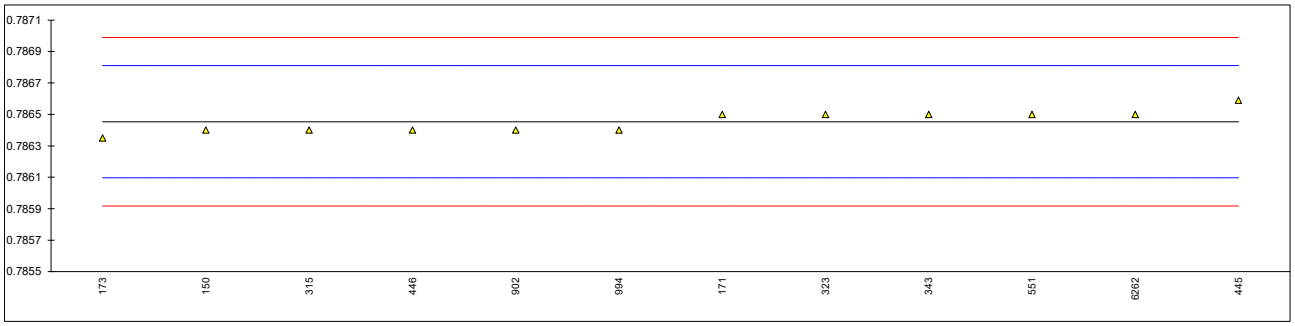


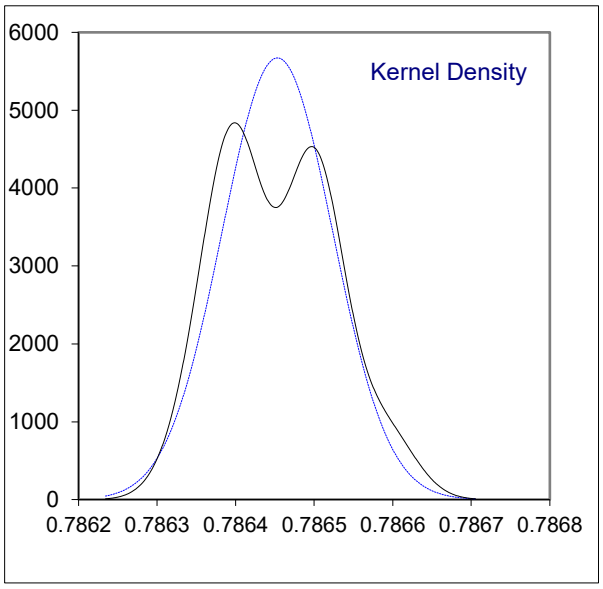


Determination of Specific Gravity at 20/20°C on sample #21270;

lab	method	value	mark	z(targ)	remarks
150	D4052	0.7864		-0.30	
171	D4052	0.7865		0.26	
173	D4052	0.78635		-0.58	
315	D4052	0.7864		-0.30	
323	D4052	0.7865		0.26	
343	D4052	0.7865		0.26	
445	ISO12185	0.78659		0.77	
446	D4052	0.7864		-0.30	
551	D4052	0.7865		0.26	
902	ISO12185	0.7864		-0.30	
913		-----		-----	
994	ISO12185	0.7864		-0.30	
1016		-----		-----	
6262	ISO12185	0.7865		0.26	

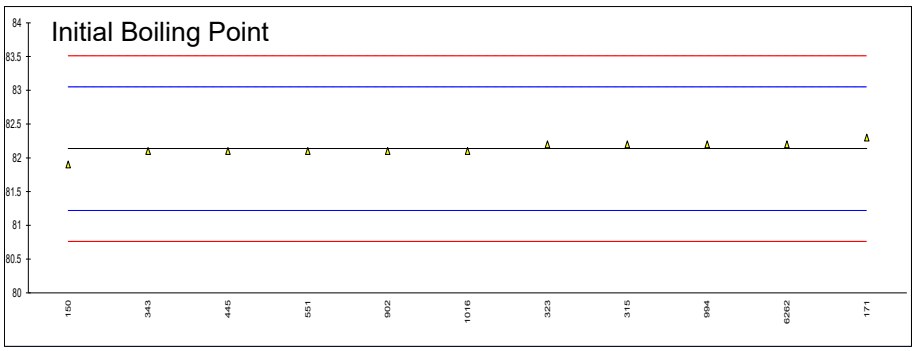
normality	OK
n	12
outliers	0
mean (n)	0.78645
st.dev. (n)	0.000070
R(calc.)	0.00020
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

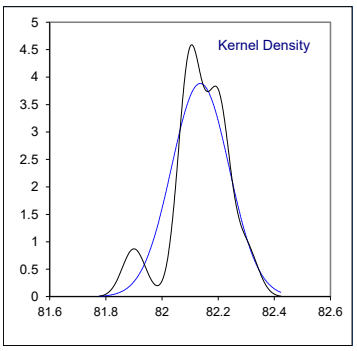


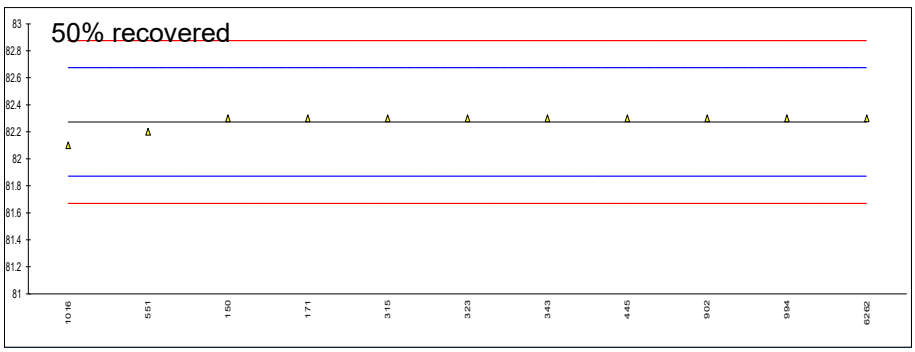


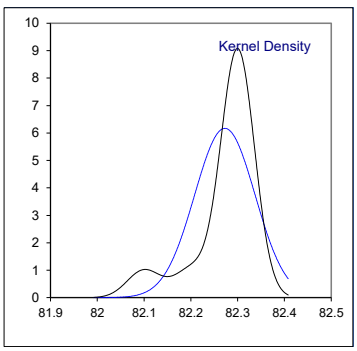
Determination of Distillation at 760 mmHg on sample #21270; results in °C.

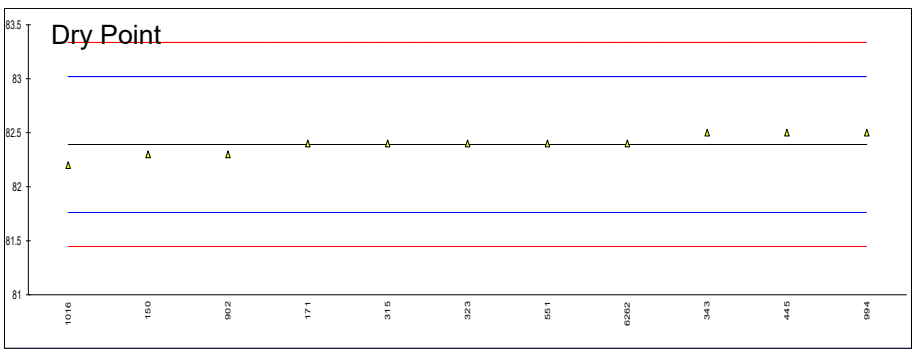
lab	method	IBP	mark	z(targ)	50%rec	mark	z(targ)	DP	mark	z(targ)
150	D1078-automated	81.9		-0.52	82.3		0.14	82.3		-0.29
171	D1078-automated	82.3		0.36	82.3		0.14	82.4		0.03
173		-----		-----	-----		-----	-----		-----
315	D1078-automated	82.2		0.14	82.3		0.14	82.4		0.03
323	D1078-automated	82.2		0.14	82.3		0.14	82.4		0.03
343	D1078-automated	82.1		-0.08	82.3		0.14	82.5		0.35
445	D1078-automated	82.1		-0.08	82.3		0.14	82.5		0.35
446		-----		-----	-----		-----	-----		-----
551	D1078	82.1		-0.08	82.2		-0.36	82.4		0.03
902	D1078-automated	82.1		-0.08	82.3		0.14	82.3		-0.29
913	D1078-manual	-----		-----	-----		-----	-----		-----
994	D1078-manual	82.2		0.14	82.3		0.14	82.5		0.35
1016	D1078-automated	82.1		-0.08	82.1		-0.86	82.2		-0.61
6262	D1078-automated	82.2		0.14	82.3		0.14	82.4		0.03
	normality	not OK			not OK			OK		
	n	11			11			11		
	outliers	0			0			0		
	mean (n)	82.14			82.27			82.39		
	st.dev. (n)	0.103			0.065			0.094		
	R(calc.)	0.29			0.18			0.26		
	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		

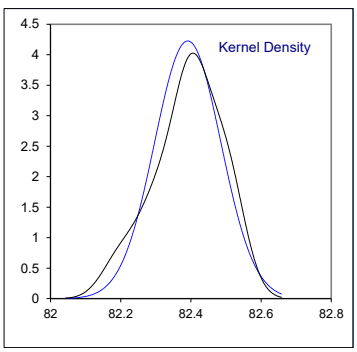










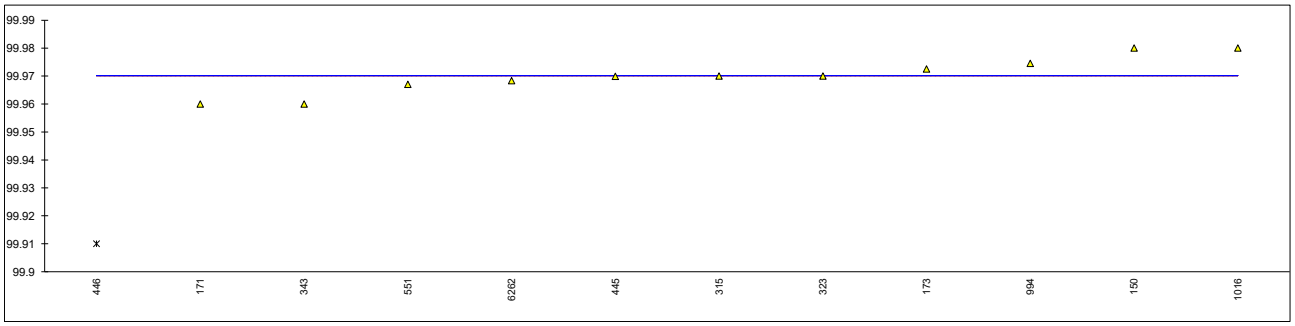


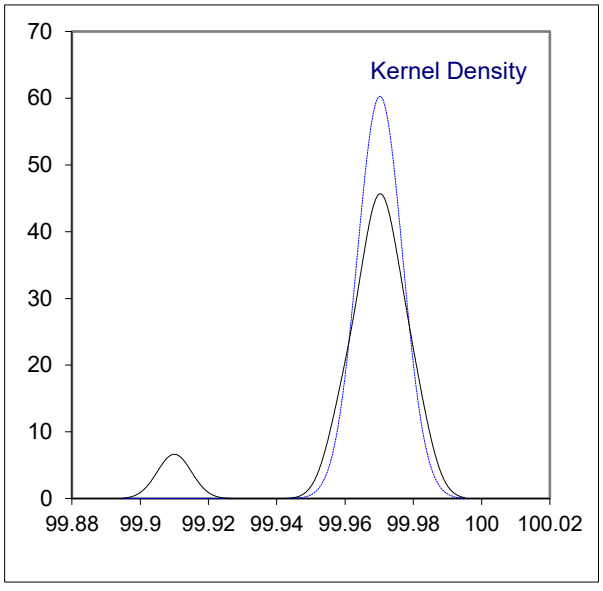
Determination of Nonvolatile matter on sample #21270; results in mg/100mL.

lab	method	value	mark	z(targ)	remarks
150	D1353	0.0		-----	
171	D1353	<1.0		-----	
173		-----		-----	
315	D1353	<1		-----	
323	D1353	< 1		-----	
343	D1353	1		-----	
445	D1353	0.5		-----	
446	D1353	0		-----	
551	D1353	0.2		-----	
902	D1353	<1		-----	
913		-----		-----	
994		-----		-----	
1016		-----		-----	
6262	D1353	0		-----	
	n	10			
	mean (n)	<1			

Determination of Purity by GC on dry basis on sample #21270, results in %M/M.

lab	method	value	mark	z(targ)	remarks
150	INH-5290	99.98		-----	
171	INH-ACS	99.96	C	-----	first reported: 99.97
173	INH-6012	99.9725		-----	
315	INH-082	99.97		-----	
323	INH-060	99.97		-----	
343	DIN55685	99.96	C	-----	first reported: 99.95
445	INH-70	99.9699		-----	
446	INH-595	99.91	G(0.01)	-----	
551	INH-3064	99.967		-----	
902		-----		-----	
913		-----		-----	
994	INH-15-12	99.9745		-----	
1016	DIN55685	99.98		-----	
6262	ISO6353-2	99.9683		-----	
	normality	OK			
	n	11			
	outliers	1			
	mean (n)	99.9702			
	st.dev. (n)	0.00662			
	R(calc.)	0.0185			
	st.dev.(lit.)	n.a.			
	R(lit.)	n.a.			
compare					
	R(iis19C18)	0.0142			

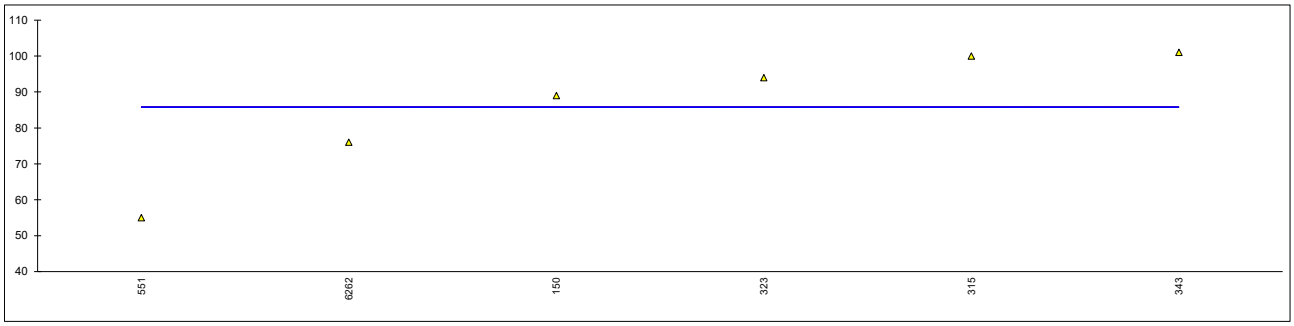




Determination of Ethanol on sample #21270; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	INH-5290	89		-----	
171		-----		-----	
173		-----		-----	
315	INH-082	100		-----	
323	INH-060	94		-----	
343	DIN55685	101		-----	
445		-----		-----	
446		-----		-----	
551	INH-3064	55		-----	
902		-----		-----	
913		-----		-----	
994		-----		-----	
1016		-----		-----	
6262	ISO6353-2	76		-----	

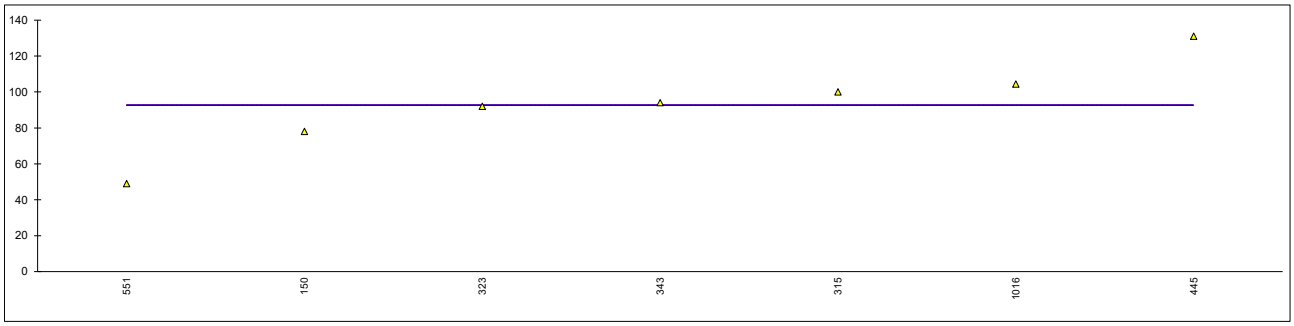
normality	unknown
n	6
outliers	0
mean (n)	85.83
st.dev. (n)	17.634
R(calc.)	49.38
st.dev.(Horwitz)	(7.026)
R(Horwitz)	(19.67)



Determination of n-Propanol on sample #21270; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	INH-5290	78		-----	
171		-----		-----	
173		-----		-----	
315	INH-082	100		-----	
323	INH-060	92		-----	
343	DIN55685	94		-----	
445	INH-70	131		-----	
446	INH-595	<20		-----	possible false negative test result?
551	INH-3064	49		-----	
902		-----		-----	
913		-----		-----	
994		-----		-----	
1016	DIN55685	104.4		-----	
6262		-----		-----	

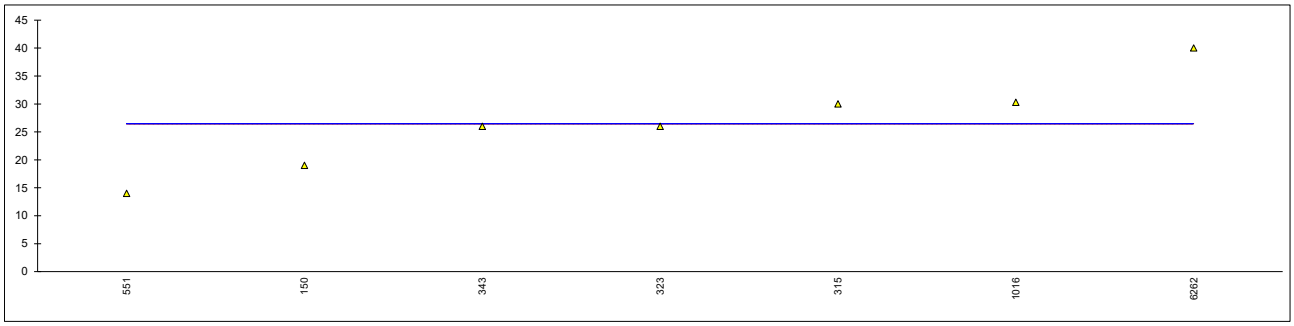
normality	unknown
n	7
outliers	0
mean (n)	92.63
st.dev. (n)	25.116
R(calc.)	70.33
st.dev.(Horwitz)	(7.496)
R(Horwitz)	(20.99)



Determination of n-Butanol on sample #21270; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	INH-5290	19		-----	
171		-----		-----	
173		-----		-----	
315	INH-082	30		-----	
323	INH-060	26		-----	
343	DIN55685	26	C	-----	first reported: 197
445		-----		-----	
446	INH-595	<20		-----	
551	INH-3064	14		-----	
902		-----		-----	
913		-----		-----	
994		-----		-----	
1016	DIN55685	30.3		-----	
6262	ISO6353-2	40		-----	

normality	unknown
n	7
outliers	0
mean (n)	26.47
st.dev. (n)	8.386
R(calc.)	23.48
st.dev.(Horwitz)	(2.587)
R(Horwitz)	(7.24)



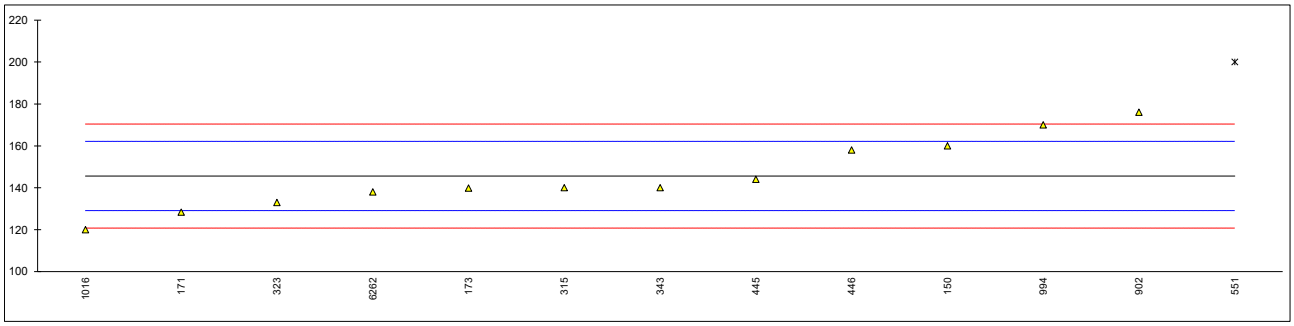
Determination of Other impurities on sample #21270; results in mg/kg.

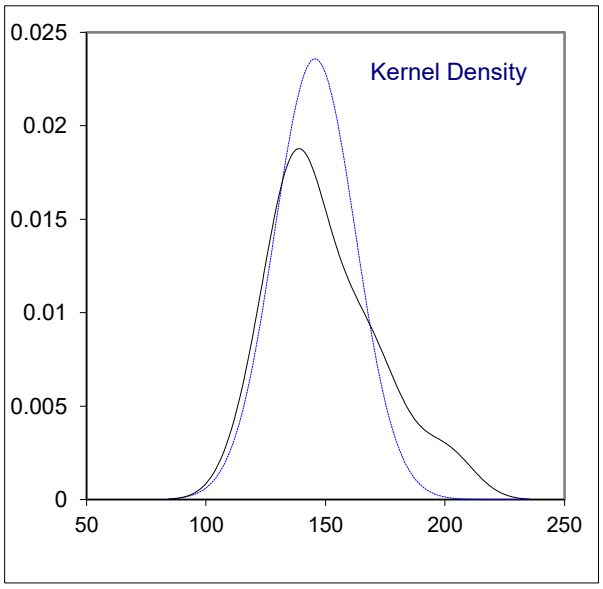
lab	method	value	mark	z(targ)	remarks
150		-----		-----	
171		-----		-----	
173		-----		-----	
315		-----		-----	
323	INH-060	105		-----	
343		-----		-----	
445		-----		-----	
446		-----		-----	
551		-----		-----	
902		-----		-----	
913		-----		-----	
994		-----		-----	
1016	DIN55685	56		-----	
6262		-----		-----	
	n	2			

Determination of Water on sample #21270; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	E1064	160		1.74	
171	E1064	128.37		-2.08	
173	E1064	139.823		-0.70	
315	E1064	140		-0.68	
323	E1064	133		-1.52	
343	E1064	140		-0.68	
445	E1064	144		-0.19	
446	D1364	158		1.50	
551	E1064	200	G(0.05)	6.58	
902	E1064	176		3.68	
913		-----		-----	
994	E1064	170		2.95	
1016	D1364	120		-3.10	
6262	E1064	138		-0.92	

normality	OK
n	12
outliers	1
mean (n)	145.60
st.dev. (n)	16.917
R(calc.)	47.37
st.dev.(E1064:16)	8.268
R(E1064:16)	23.15





APPENDIX 2

Number of participants per country

1 lab in AZERBAIJAN
2 labs in BELGIUM
1 lab in BRAZIL
1 lab in INDIA
2 labs in NETHERLANDS
1 lab in SPAIN
1 lab in TURKEY
2 labs in UNITED KINGDOM
3 labs in UNITED STATES OF AMERICA

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	= calculation difference between reported test result and result calculated by iis
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
SDS	= Safety Data Sheet

Literature

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12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)