

Results of Proficiency Test Ethanol (Food / Neutral grade) December 2023

Organized by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

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1 Introduction

Since 2007 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Ethanol (Food / Neutral grade) every year. During the annual proficiency testing program of 2023 it was decided to continue the round robin for the analysis of Ethanol (Food / Neutral grade).

In this interlaboratory study 28 laboratories in 16 countries registered for participation, see appendix 3 for the number of participants per country. In this report the results of the Ethanol (Food / Neutral grade) 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 a laboratory that has performed the tests in accordance with for ISO/IEC17043 relevant requirements of ISO/IEC17025.

It was decided to send two different samples of Ethanol (Food / Neutral grade); one 0.5 L bottle labelled #23265 for regular analyzes and one 250 mL bottle labelled #23266 for GC determination only. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. 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

For the preparation of the sample for the regular analyzes a batch of approximately 30 liters of Ethanol (Food / Neutral grade) was obtained from a local supplier. After homogenization 60 amber glass bottles of 0.5 L were filled and labelled #23265.

The homogeneity of the subsamples was checked by determination of Density at 20 °C in accordance with ASTM D4052 on 8 stratified randomly selected subsamples.

	Density at 20 °C in kg/L
sample #23265-1	0.80568
sample #23265-2	0.80568
sample #23265-3	0.80568
sample #23265-4	0.80569
sample #23265-5	0.80568
sample #23265-6	0.80568
sample #23265-7	0.80568
sample #23265-8	0.80568

Table 1: homogeneity test results of subsamples #23265

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)	0.00001
reference test method	ISO12185:96
0.3 x R (reference test method)	0.00015

Table 2: evaluation of the repeatability of subsamples #23265

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

For the preparation of the GC sample two different batches of in total approximately 20 liters Ethanol (Food / Neutral grade) were mixed together and spiked with Methanol, Acetal (1,1-diethoxyethane), Acetone, Benzene, Isopropanol, and Mono Ethylene glycol (MEG). After homogenization 50 amber glass bottles of 250 mL were filled and labelled #23266. The homogeneity of the subsamples was checked by determination of Isopropanol in accordance with an in house method on 8 stratified randomly selected subsamples.

	lsopropanol in mg/kg
sample #23266-1	26.5
sample #23266-2	25.5
sample #23266-3	26.7
sample #23266-4	26.5
sample #23266-5	26.0
sample #23266-6	25.9
sample #23266-7	27.3
sample #23266-8	25.5

Table 3: homogeneity test results of subsamples #23266

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

	lsopropanol in mg/kg
r (observed)	1.8
reference method	Horwitz
0.3 x R (reference method)	2.2

Table 4: evaluation of the repeatability of subsamples #23266

The calculated repeatability is in agreement with 0.3 times the estimated reproducibility calculated with the Horwitz equation. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one 0.5 L sample of Ethanol (Food / Neutral grade) labelled #23265 and one 250 mL sample of Ethanol (Food / Neutral grade) labelled #23266 was sent on November 15, 2023. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Ethanol 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 on sample #23265: Appearance,

Density at 20 °C, Nonvolatile matter, Permanganate Time Test at 20 °C, pHe (with LiCl and KCl electrode), Strength (in %M/M and %V/V), Water and UV absorbance at 300, 270, 260, 250, 240, 230 and 220 nm with an evaluation of the UV-scan.

On sample #23266 it was requested to determine: Purity of Ethanol on dry basis, Methanol, Acetal (1,1-diethoxyethane), Acetaldehyde, Acetone, Benzene, Isopropanol, Mono Ethylene glycol (MEG), Other impurities and Total impurities.

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, 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 appendices 1 and 2 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 reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendices 1 and 2. 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 in 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_{\text{(target)}} = \text{(test result - average of PT)} / \text{target standard deviation}
```

The $z_{\text{(target)}}$ 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 no problems were encountered with the dispatch of the samples. When considering the test results of the two samples together four participants reported test results after the final reporting date and two participants did not report any test results. Not all participants were able to report all tests requested.

In total 26 participants reported 360 numerical test results. Observed were 16 outlying test results, which is 4.4%. In proficiency tests 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 SAMPLE AND PER TEST

In this section the reported test results are discussed per sample and 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 4.

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.

In the iis PT reports ASTM test methods are referred to with a number (e.g. D1363) and an added designation for the year that the test method was adopted or revised (e.g. D1363:06). When a method has been reapproved an "R" will be added and the year of approval (e.g. D1363:06R19).

sample #23265

Appearance: All reporting participants agreed on a test result of Pass (Clear & Bright).

<u>Density at 20 °C</u>: The group of participants met the target requirements. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO12185:96.

Nonvolatile matter: Almost all reporting participants agreed on a test result of <1 mg/100 mL. Therefore, no z-scores are calculated.

Permanganate Time Test at 20 °C: The group of participants had difficulty to meet the target requirements. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D1363:06R19.

It is known that the pHe determined with a LiCl electrode generally gives a lower test result than the pHe determined with a KCl electrode. Therefore, the test results are requested separately. Test method EN15490 describes the use of a LiCl electrode and test method ASTM D6423 describes the use of a KCl electrode.

- <u>pHe with LiCl electrode</u>: The group of participants had difficulty to meet the target requirements. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of EN15490:07.
- <u>pHe with KCl electrode</u>: The group of participants met the target requirements. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D6423:20a.
- <u>Strength (%M/M)</u>: The group of participants met the target requirements. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the reproducibility derived from the OIML table.
- Strength (%V/V): The group of participants met the target requirements. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the reproducibility derived from the OIML table.
- Water: The group of participants met the target requirements. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation but is not in agreement with the strict requirements of ASTM E203:24.

Regretfully, no reference test method for the determination of UV absorbance exists. Some participants reported test results obtained with a 50 mm cuvette, others with a 10 mm cuvette. In order to determine a Pass or Fail based on the sample UV-graph it is important that even the smallest deviation is detected. Therefore, the use of a 50 mm cuvette is advised. In this PT ten laboratories used a 50 mm cuvette and eleven laboratories used a 10 mm cuvette. Both groups were evaluated separately.

<u>UV - 50 mm cuvette</u>: One statistical outlier was observed over all seven parameters. Nine participants evaluated the sample as 'Pass'.

<u>UV - 10 mm cuvette</u>: Three statistical outliers were observed over all seven parameters. Ten participants evaluated the sample as 'Pass'.

sample #23266

Purity of Ethanol on dry basis: Two statistical outliers were observed. Regretfully, no reference test method is available that provides precision data for the determination of purity in Ethanol (Food / Neutral grade). Therefore, no z-scores are calculated.

Methanol: The group of participants may have had difficulty to meet the target requirements. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

<u>Acetal (1,1-diethoxyethane)</u>: The group of participants met the target requirements. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility calculated with the Horwitz equation.

Acetone: The group of participants may have had difficulty to meet the target requirements. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

Benzene: The group of participants may have had difficulty to meet the target requirements. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

Isopropanol: The group of participants met the target requirements. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility calculated with the Horwitz equation.

<u>Total impurities</u>: The group of participants met the target requirements. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility calculated with the Horwitz equation for 7 components.

The participants agreed on a concentration near or below the limit of detection for all other elements mentioned in paragraph 2.6. Therefore, no z-scores are calculated for these elements. The reported test results are given in appendix 2.

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 target reproducibility derived from reference methods are presented in the next tables.

Parameter	unit	n	average	2.8 * sd	R(lit)
Appearance		21	Pass (C&B)	n.a.	n.a.
Density at 20 °C	kg/L	21	0.8057	0.0001	0.0005
Nonvolatile matter	mg/100 mL	13	<1	n.e.	n.e.
Permanganate Time Test 20 °C	minutes	11	32.5	16.7	8.2
pHe with LiCl electrode		5	7.6	1.1	0.7
pHe with KCI electrode		7	8.1	0.9	1.0
Strength	%M/M	14	94.45	0.04	0.06
Strength	%V/V	18	96.42	0.02	0.06
Water	%M/M	17	5.52	0.20	0.48
UV – 50 mm cuvette:					
UV-absorbance 300 nm		10	0.005	0.012	n.a.
UV-absorbance 270 nm		9	0.026	0.020	n.a.
UV-absorbance 260 nm		9	0.048	0.023	n.a.
UV-absorbance 250 nm		9	0.108	0.026	n.a.
UV-absorbance 240 nm		10	0.247	0.043	n.a.
UV-absorbance 230 nm		9	0.564	0.059	n.a.
UV-absorbance 220 nm		9	1.132	0.174	n.a.
Conclusion UV-scan		9	Pass	n.a.	n.a.
UV – 10 mm cuvette:					
UV-absorbance 300 nm		9	0.0006	0.0037	n.a.
UV-absorbance 270 nm		10	0.005	0.009	n.a.
UV-absorbance 260 nm		11	0.011	0.016	n.a.
UV-absorbance 250 nm		11	0.022	0.016	n.a.
UV-absorbance 240 nm		11	0.051	0.021	n.a.
UV-absorbance 230 nm		11	0.115	0.034	n.a.
UV-absorbance 220 nm		11	0.225	0.057	n.a.
Conclusion UV-scan		10	Pass	n.a.	n.a.

Table 5: reproducibilities of tests on sample #23265

Parameter	unit	n	average	2.8 *sd	R(target)
Purity of Ethanol on dry basis	%M/M	15	99.99	0.010	n.a.
Methanol	mg/kg	18	45.5	25.0	11.5
Acetal (1,1-diethoxyethane)	mg/kg	13	15.6	4.1	4.6
Acetone	mg/kg	16	26.5	13.9	7.2
Benzene	mg/kg	15	22.6	9.1	6.3
Isopropanol	mg/kg	15	19.6	4.0	5.6
Total impurities	mg/kg	10	152.3	60.7	84.7

Table 6: reproducibilities of tests on sample #23266

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 problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2023 WITH PREVIOUS PTS

	December 2023	December 2022	December 2021	December 2020	December 2019
Number of reporting laboratories	26	24	27	24	25
Number of test results	360	276	311	315	337
Number of statistical outliers	16	18	8	19	19
Percentage of statistical outliers	4.4%	6.5%	2.6%	6.0%	5.6%

Table 7: 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 to the requirements of the reference test methods. The conclusions are given in the following table.

Parameter	December 2023	December 2022	December 2021	December 2020	December 2019
Density at 20 °C	++	++	++	++	++
Nonvolatile matter	n.e.	n.e.	n.e.	n.e.	-
Permanganate Time Test 20 °C		-	-	-	+
pHe with LiCl electrode	-	-	+/-	-	-
pHe with KCI electrode	+	++	-	-	n.e.
Strength %M/M	+	-	+	-	+
Strength %V/V	++	++	++	+	+/-
Water	++	++	++	+	+
Methanol		-		-	-
Acetal (1,1-diethoxyethane)	+	n.e.	n.e.	n.e.	+
Acetaldehyde	n.e.	n.e.	n.e.	n.e.	n.a.
Acetone	-	-	-	-	+
Benzene	-	+	+/-	-	-
Isopropanol	+	-	-	-	-

Parameter	December 2023	December 2022	December 2021	December 2020	December 2019
Mono Ethylene glycol (MEG)	n.e.	n.e.	n.e.	()	n.e.
Other impurities	n.e.	n.e.	()	()	n.e.
Total impurities	+	+		+	-

Table 8: comparison of determinations to the reference test methods

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

For results between brackets no z-scores are calculated.

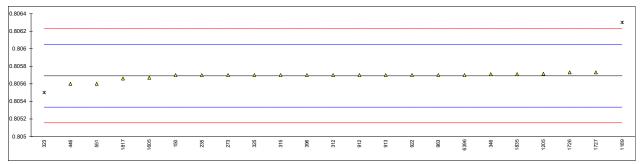
APPENDIX 1

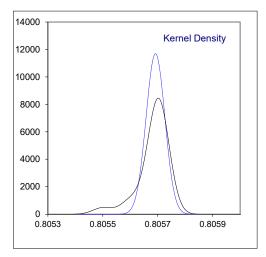
Determination of Appearance on sample #23265;

lab	method	value	mark	z(targ)	remarks
150	Visual	clear & bright			
235	Visual	C & B			
273	Visual	PASS [C&B]			
312	Visual	CLCOL			
315	E2680	pass			
323	Visual	PASS			
325	Visual	clear & bright			
346	Visual	clear &free			
396	Visual	Clear bright			
446	Visual	Pass			
551	Visual	Pass			
912	E2680	Pass			
913	E2680	Clear and Bright			
922	Visual	Clear & Bright			
963	Visual	Pass			
1189	Visual	Br&Cl			
1205					
1438					
1574					
1605					
1726	Visual	Clear&Colorless			
1727	Visual	Clear&colorless			
1817	Visual	Pass			
1835	Visual	c&C			
6214					
6224					
6396		Clear + Bright			
6546					
	n	21			
	mean (n)	Pass (Clear & Bright)			

Determination of Density at 20 °C on sample #23265; results in kg/L

lab	method	value	mark	z(targ)	remarks
	D4052	0.8057		0.05	
	ISO12185	0.80570		0.05	
273		0.8057		0.05	
312	ISO12185	0.8057		0.05	
315	D4052	0.8057		0.05	
323	D4052	0.8055	R(0.01)	-1.07	
325	D4052	0.8057		0.05	
346	D4052	0.80571		0.10	
396	D4052	0.8057		0.05	
446	D4052	0.8056	С	-0.51	reported 805.6 kg/L
551	D4052	0.8056		-0.51	
912	D4052	0.8057		0.05	
913	D4052	0.8057		0.05	
922	D4052	0.8057		0.05	
963	D4052	0.8057		0.05	
1189	ISO12185	0.8063	R(0.01)	3.41	
1205	In house	0.805714	, ,	0.13	
1438					
1574					
1605	D4052	0.805669		-0.13	
1726	D4052	0.80573		0.21	
1727	D4052	0.80573		0.21	
1817	Table OIML	0.80566		-0.18	
1835	ISO12185	0.80571		0.10	
6214					
6224					
6396		0.8057		0.05	
6546					
	normality	not OK			
	•				
	(2.0000			
	normality n outliers mean (n) st.dev. (n) R(calc.) st.dev.(ISO12185:96) R(ISO12185:96)	not OK 21 2 0.805692 0.0000341 0.000096 0.0001786 0.0005			



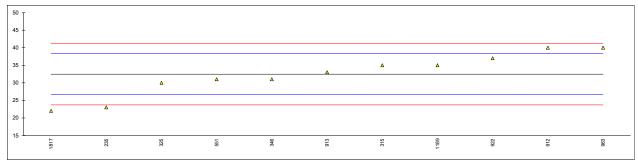


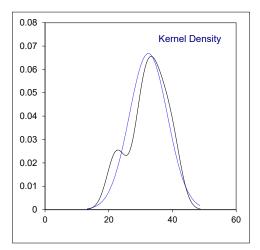
Determination of Nonvolatile matter on sample #23265; results in mg/100 mL

lab	method	value	mark	z(targ)	remarks
150	D1353	0.2			
235	EN15691	1.0			
273	D1353	0.0044			
312					
	D1353	<1			
	D1353	2			
	D1353	0.3			
346					
396					
	D1353	0			
	D1353	0.2			
	D1353	0.4			
	D1353	0.6			
	D1353	< 1.0			
	D1353	<1			
	D1353	<1			
1205					
1438					
1574					
1605	EN45004				
	EN15691	<10			
	EN15691	not detected			
	In house	1			
1835	EN15691	<10			
6214					
6224					
6396					
6546					
	n	13			
	mean (n)	<1			
	` '				

Determination of Permanganate Time Test at 20 °C on sample #23265; results in minutes

	method	value	mark	z(targ)	
	D1363	>60		>9.43	possibly a false positive test result?
	D1363	23		-3.24	•
273					
312					
315	D1363	35		0.87	
	D1363	>30			
325	D1363	30		-0.84	
346	D1363	31		-0.50	
396					
446	D1363	>20			
551	D1363	31		-0.50	
	D1363	40		2.58	
913	D1363	33		0.19	
	D1363	37		1.56	
	D1363	40		2.58	
1189	D1363	35		0.87	
1205					
1438					
1574					
1605					
1726					
1727					
1817	In house	22		-3.58	
1835					
6214					
6224					
6396					
6546					
	normality	OK			
	normality	0K 11			
	n outliers	0			
	mean (n) st.dev. (n)	32.45 5.973			
		5.973 16.72			
	R(calc.) st.dev.(D1363:06R19)	2.921			
	R(D1363:06R19)	8.18			



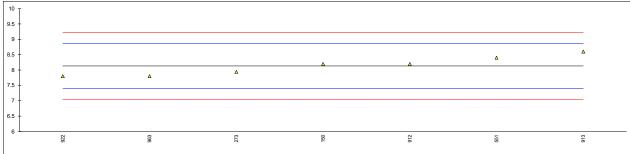


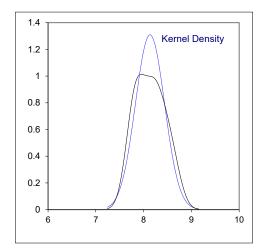
Determination of pHe with LiCl electrode on sample #23265;

lab	method	value	mark	z(tarq)	remarks	
150						
235						
273						
312						
315						
323						
325						
346						
396						
446						
551	NBR10891	7.5		-0.41		
912						
913						
922						
963						
1189	EN15490	7.0		-2.32		
1205						
1438						
1574						
1605						
	EN15490	7.79		0.71		
1727	EN15490	7.77		0.63		
1817						
	EN15490	7.97		1.40		
6214						
6224						
6396						
6546						
	normality	unknown				
	n	5				
	outliers	0				
	mean (n)	7.606				
	st.dev. (n)	0.3781				
	R(calc.)	1.059				
	st.dev.(EN15490:07)	0.2608				
	R(EN15490:07)	0.730				
	. ((=)	000				
9 _T						
8.5						
8 +				4	Δ	
7.5 +		Δ			-	
		_				
7 +	Δ					
6.5 +				-		
0.5						
6	1189				52 93	
	1	25		7071	1726	

Determination of pHe with KCl electrode on sample #23265;

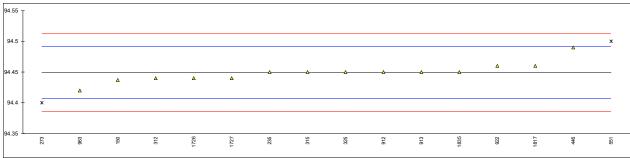
	method	value	mark z(targ)	remarks
150	D6423	8.2	0.18	
235				
	D6423	7.94	-0.53	
312				
315				
323				
325				
346				
396				
446				
	D6423	8.4	0.73	
	D6423	8.2	0.18	
	D6423	8.6	1.28	
	D6423	7.8	-0.92	
	D6423	7.8	-0.92	
1189				
1205				
1438				
1574				
1605				
1726				
1727				
1817				
1835				
6214				
6224				
6396				
6546				
	normality	unknown		
	n	7		
	outliers	0		
	mean (n)	8.134		
	st.dev. (n)	0.3048		
	R(calc.)	0.853		
	st.dev.(D6423:20a)	0.833		
	R(D6423:20a)	1.021		

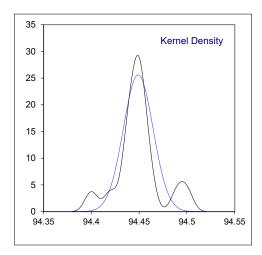




Determination of Strength on sample #23265; results in %M/M

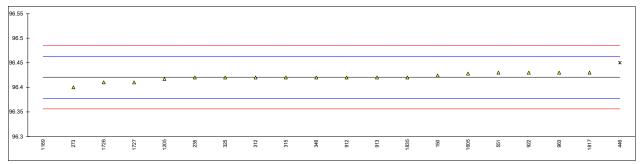
lab	method	value	mark	z(targ)	remarks
150	INH-982.10	94.437	С	-0.57	first reported 192.83
235	Table OIML	94.45	С	0.04	first reported 96.42
273	Table OIML	94.40	D(0.05)	-2.33	
312		94.44		-0.43	
315	Table OIML	94.45		0.04	
323					
325	Table OIML	94.45		0.04	
346					
396	Table OIM			4.04	
446	Table OIML	94.49	D(0.04)	1.94	
551	NBR15639	94.5	D(0.01)	2.42	
912	Table OIML	94.45		0.04	
913	Table OIML	94.45		0.04	
922 963	Table OIML	94.46		0.52	
1189	Table OIML	94.42		-1.38	
1205					
1438					
1574					
1605					
1726	Table OIML	94.44		-0.43	
1727	Table OIML	94.44		-0.43	
1817	Table OIML	94.46		0.52	
1835	Table OIML	94.45		0.04	
6214					
6224					
6396					
6546					
	normality	not OK			
	n	14			
	outliers	2			
	mean (n)	94.449			
	st.dev. (n)	0.0156			
	R(calc.)	0.044			
	st.dev.(OIML table)	0.0211			
	R(OIML table)	0.059			OIML R022-e75

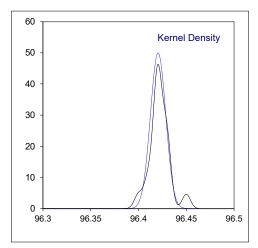




Determination of Strength on sample #23265; results in %V/V

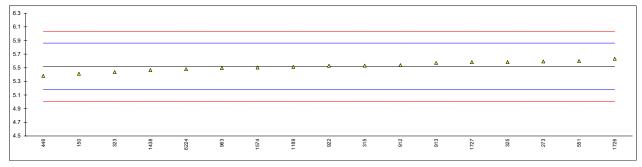
lab	method	value	mark	z(targ)	remarks
150	INH-982.10	96.424	С	0.16	first reported 96.12
235	Table OIML	96.42	С	-0.02	first reported 94.45
273	Table OIML	96.40		-0.96	
312	Table OIML	96.42		-0.02	
315	Table OIML	96.42		-0.02	
323					
325	Table OIML	96.42		-0.02	
346	Table OIML	96.42		-0.02	
396					
446	Table OIML	96.45	G(0.05)	1.38	
551	NBR15639	96.43	С	0.44	first reported 96.5
912	Table OIML	96.42		-0.02	
913	Table OIML	96.42		-0.02	
	Table OIML	96.43		0.44	
963	Table OIML	96.43		0.44	
1189	Table OIML	96.2	G(0.01)	-10.29	
1205	Table OIML	96.417		-0.16	
1438					
1574					
1605	Table OIML	96.428		0.35	
1726	Table OIML	96.41		-0.49	
1727	Table OIML	96.41		-0.49	
1817		96.43		0.44	
1835	Table OIML	96.42		-0.02	
6214					
6224					
6396					
6546					
	normality	suspect			
	n	18			
	outliers	2			
	mean (n)	96.421			
	st.dev. (n)	0.0080			
	R(calc.)	0.022			
	st.dev.(OIML table)	0.0214			
	R(OIML table)	0.060			OIML R022-e75
	, ,				

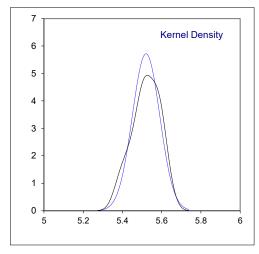




Determination of Water on sample #23265; results in %M/M

lab	method	value	mark z(ta	g) remarks
150	E203	5.412	-0.	
235				
	E203	5.592		42
312	E203	5.531		 06
	E203	5.438	-0.	
	E203	5.584		37
346				
396				
	D1364	5.381	-0.	
551	E203	5.599		46
	E203	5.5371		10
	E203 E203	5.57 5.529		29 05
	E203 E203	5.529 5.497	-0.	
	D1364	5.5128	-0. -0.	
1205	2.00.			
	E203	5.467	-0.	31
1574	In house	5.5035	-0.	10
1605				
	EN15692	5.6324		66
1727 1817	EN15692	5.5821		36
1835			 	
6214				
	In house	5.48	-0.	24
6396				
6546			-	
	normality	OK		
	n	17		
	outliers	0		
	mean (n)	5.5205		
	st.dev. (n)	0.06981		
	R(calc.)	0.1955		
	st.dev.(Horwitz)	0.17075		
Compa	R(Horwitz)	0.4781		
Compa	R(E203:24)	0.078		
	(==00.2 1)	5.070		

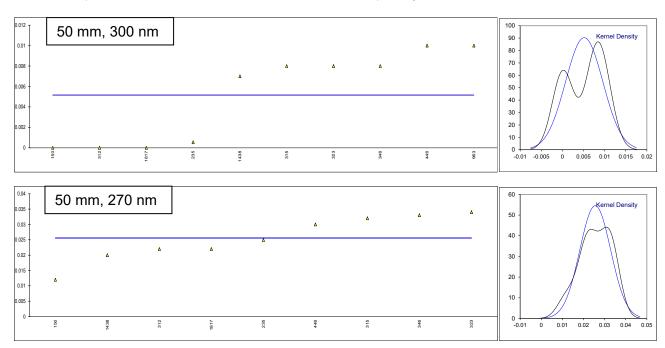


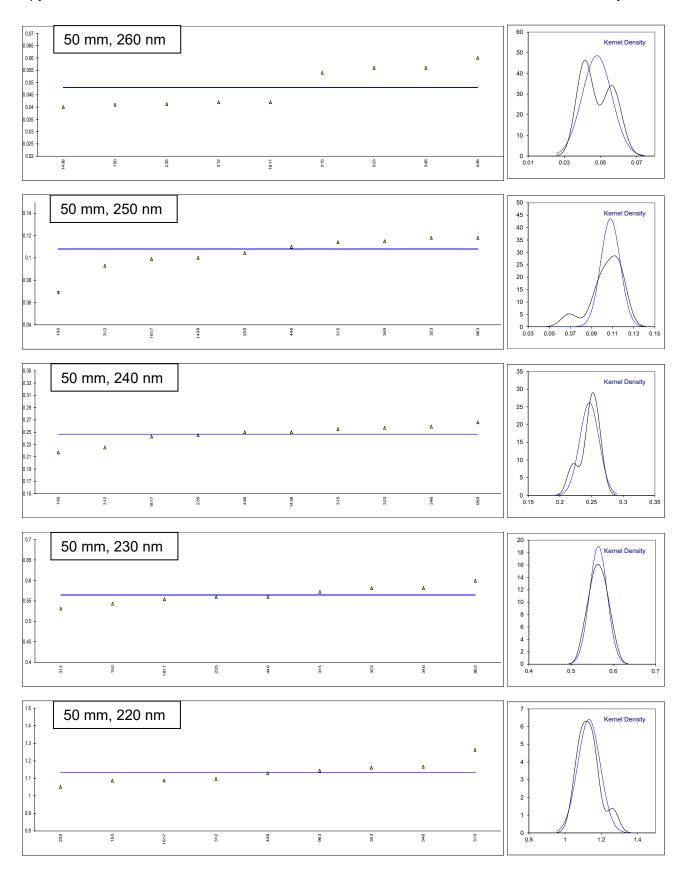


Determination of UV absorbance (50 mm cuvette) on sample #23265;

lab	method	300 nm	270 nm	260 nm	250 nm	240 nm	230 nm	220 nm	Pass/Fail
150	IMPCA004	0.000	0.012	0.041	0.069 G(5)	0.217	0.543	1.087	Pass
235	IMPCA004	0.00055	0.02495	0.0412	0.10435	0.2453	0.5595	1.05145	Pass
273								4.007	 D
312	INII I 004	0.000	0.022	0.042	0.093	0.225	0.531	1.097	Pass
315 323	INH-094 IMPCA004	0.008 0.008	0.032 0.034	0.054 0.056	0.114 0.118	0.255 0.257	0.572 0.581	1.263 1.161	 Pass
325	IIVIF CAUU4	0.006	0.034	0.050	0.116	0.237	0.561	1.101	
346	IMPCA004	0.008 C	0.033 C	0.056 C	0.115 C	0.259 C	0.581 C	1.166 C	Pass
396	0, 100 1								
446	INH-1318	0.01	0.03	0.06	0.11	0.25	0.56	1.13	Pass
551									
912									
913									
922	11.450.400.4				0.440		0.500	4 4 4 4	 D
963 1189	IMPCA004	0.01			0.118	0.266	0.599	1.144	Pass
1205									
1438		0.007	0.02	0.04	0.10	0.25			Pass
1574									
1605									
1726									
1727									
1817	In house	0	0.022	0.042	0.099	0.243	0.554	1.088	Pass
1835									
6214									
6224 6396									
6546									
0040									
	normality	OK	OK	OK	OK	OK	OK	not OK	
	n	10	9	9	9	10	9	9	9
	outliers	0	0	0	1	0	0	0	
	mean (n)	0.0052	0.0256	0.0480	0.1079	0.2467	0.5645	1.1319	Pass
	st.dev. (n)	0.00442	0.00732	0.00821	0.00917	0.01524	0.02101	0.06223	
	R(calc.)	0.0124	0.0205	0.0230	0.0257	0.0427	0.0588	0.1742	
	st.dev.(lit.)	unknown							
	R(lit.)	unknown							

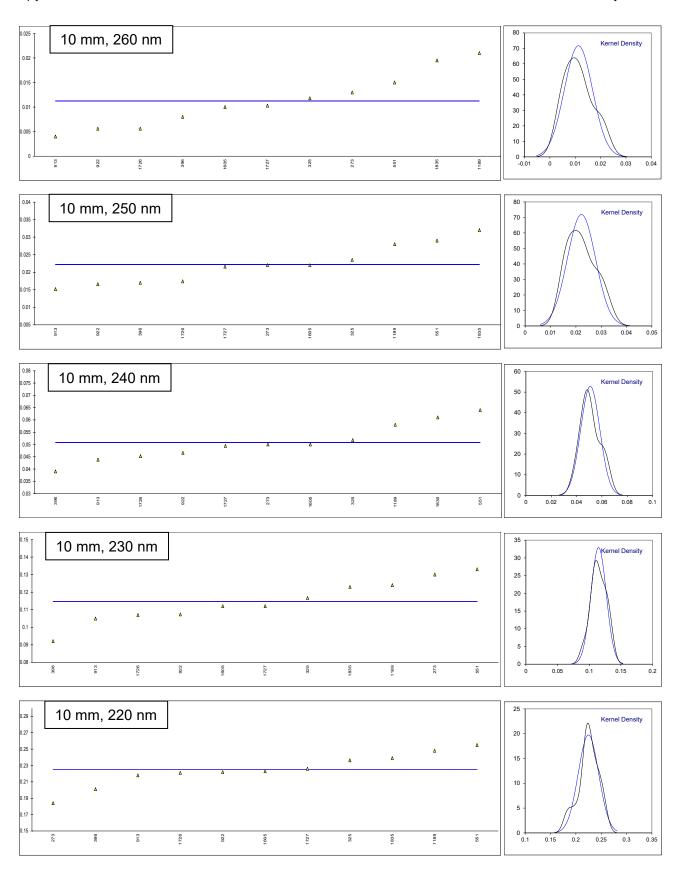
Lab 346 first reported 0.024, 0.055, 0.081, 0.142, 0.292, 0.617 and 1.206 respectively





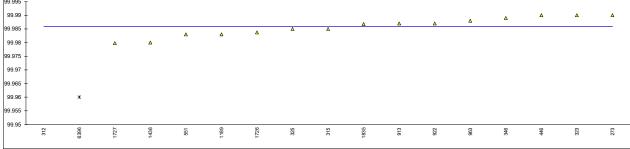
Determination of UV absorbance (10 mm cuvette) on sample #23265;

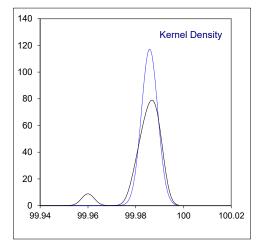
lab	method	300 nm	270 nm	260 nm	250 nm	240 nm	230 nm	220 nm	Pass/Fail
150 235									
273	IMPCA004	0.002	0.008	0.013	0.022	0.050	0.130	0.184	Pass
312									
315 323									
325	INH-CM	0.0017	0.0074	0.0118	0.0235	0.0518	0.1167	0.2363	Pass
346 396		0.000	0.004	0.008	0.017	0.039	0.092	0.201	Pass
446									
551 912	INH-2047	0.002	0.010	0.015	0.029	0.064	0.133	0.255	Pass
913	IMPCA004	0.0000	0.0005	0.0040	0.01520	0.0438	0.1049	0.2180	Pass
922 963	INH-13	-0.002 	0.0015 	0.0056	0.0166	0.0466	0.1073	0.2220	Pass
1189	IMPCA004	0.005 DG(5)	0.018 G(5)	0.021	0.028	0.058	0.124	0.248	Pass
1205									
1438 1574									
1605		0.000	0.006	0.010	0.022	0.050	0.112	0.223	
1726 1727	In house IMPCA004	0.000 0.0015	0.00133 0.005	0.00561 0.0103	0.0174 0.0216	0.0452 0.0494	0.107 0.112	0.221 0.226	Pass Pass
1817	67 100 1								
1835 6214		0.0075 DG(5)	0.0014	0.0195	0.032	0.061	0.123	0.239	Pass
6224									
6396 6546									
0340									
	normality	OK	OK	OK	OK	OK	OK	OK	40
	n outliers	9 2	10 1	11 0	11 0	11 0	11 0	11 0	10
	mean (n)	0.0006	0.0045	0.0113	0.0222	0.0508	0.1147	0.2248	Pass
	st.dev. (n) R(calc.)	0.00133 0.0037	0.00331 0.0093	0.00557 0.0156	0.00555 0.0156	0.00756 0.0212	0.01212 0.0339	0.02022 0.0566	
	st.dev.(lit.)	unknown	unknown	unknown	unknown	unknown	unknown	unknown	
	R(lit.)	unknown	unknown	unknown	unknown	unknown	unknown	unknown	
0.01 _T	40 004	0					350		
0.008 +	10 mm, 30	0 nm					x 300 -	٥	Kernel Density
0.006 +							250 -	\bigwedge	
0.004 -						×	200 -		
0.002 -			Δ	Δ	Δ Δ		150 -	/ \	
0 +	Δ	Δ Δ	Δ				100 -	/	
-0.002 -	Δ						50 -	/ \	
-0.004									
	913	396	1726	325	2773	1189	-0.01	-0.005 0 0.0	05 0.01 0.015
[
0.02 T 0.018 -	10 mm, 270	0 nm					x 140		Kernel Density
0.016 -							120 -	\bigwedge	
0.014 +							100 -		
0.01 -						Δ	80 -		
0.008 -					Δ		60 -	/ \	
0.006 +			Δ	Δ			40 -	// \	
0.002	Δ	Δ					20 -		\sim
	17.26	1835	398	1605	325	551	0 - 0.01	0 0.01	0.02 0.03



Determination of Purity of Ethanol on dry basis on sample #23266; results in %M/M

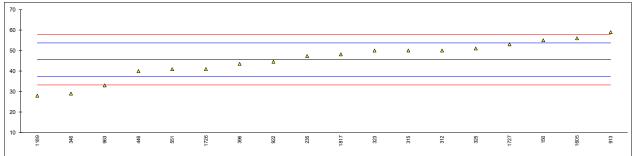
lab	method	value	mark	z(targ)	remarks
150	metilou		IIIQIK	Z(tary)	Tolliano
235					
273	IMPCA001	99.99			
	INH-0001	99.9	G(0.01)		
315		99.985	,		
323	INH-001	99.99			
	INH-0001	99.985			
346	ETOH-02	99.989			
396					
	INH-043	99.99			
551	INH-1313	99.983			
912					
	ETOH-002	99.987			
	INH-02	99.987			
963	D5501	99.988			
1189 1205		99.983			
1438		99.98			
1574		99.90			
1605					
1726		99.9837			
1727	In house	99.97982			
1817	iii iiodoo				
	In house	99.9868			
6214					
6224					
6396		99.96	G(0.01)		
6546			. ,		
		011			
	normality	OK			
	n di:	15			
	outliers	2			
	mean (n)	99.9858			
	st.dev. (n) R(calc.)	0.00341 0.0095			
	st.dev.(Horwitz)	unknown			
	R(Horwitz)	unknown			
	r((101Witz)	UTIKITOWIT			
20.005					
99.995 T					
99.99 -					Δ Δ Δ Δ Δ Δ Δ Δ Δ Δ Δ Δ Δ Δ Δ Δ Δ Δ Δ
99.985 -		Δ	Δ Δ	Δ	Δ
99.98 +	Δ Δ				
99.975					
99.97					
99.965					
99.96	*				
99.955					
00.05					

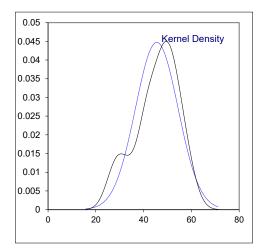




Determination of Methanol on sample #23266; results in mg/kg

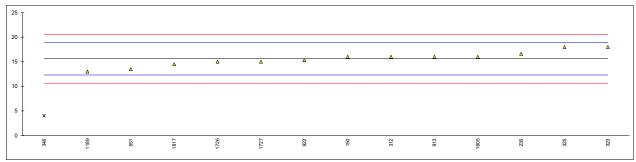
lab	method	value	mark	z(targ)	remarks
150	ETOH-02	55		2.31	
	INH-0001	47.31	С	0.44	first reported 0.00
273					
	INH-0001	50		1.09	
315		50		1.09	
	INH-001	50		1.09	
	INH-0001	51	_	1.34	
		29	С	-4.03	first reported 0
	ANS-008/043	43.5		-0.49	
	INH-043	40		-1.35	
551	INH-1313	40.9		-1.13	
912	ETOU 000			2.00	
	ETOH-002	59		3.29	
	INH-02 D5501	44.56		-0.23 -3.05	
1189	D330 I	33 28		-3.03 -4.27	
1205		20		-4.21	
1438					
1574					
1605		56		2.56	
1726		41		-1.10	
	In house	53		1.82	
1817	In house	48.1471		0.64	
1835	In house	<50			
6214					
6224					
6396					
6546					
	normality	OK			
	n	18			
	outliers	0			
	mean (n)	45.523			
	st.dev. (n)	8.9165			
	R(calc.)	24.966			
	st.dev.(Horwitz)	4.0998			
	R(Horwitz)	11.479			

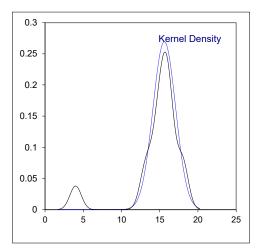




Determination of Acetal (1,1-diethoxyethane) on sample #23266; results in mg/kg

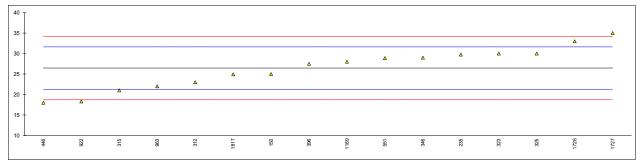
lab	method	value	mark	z(targ)	remarks
150	ETOH-02	16		0.24	
235	INH-0001	16.60	С	0.60	first reported 0.00
273					
312	INH-0001	16		0.24	
315					
323	INH-001	18		1.45	
325		18		1.45	
	ETOH-02	4	C,G(0.01)	-7.03	first reported 9.8
396					
	INH-043	<5			possibly a false negative test result?
551	INH-1313	13.5		-1.28	
912					
	ETOH-002	16		0.24	
922	INH-02	15.32		-0.18	
963					
1189		13		-1.58	
1205					
1438					
1574					
1605		16		0.24	
1726	la la cua a	15		-0.37	
	In house	15		-0.37	
1817		14.5026		-0.67	
1835 6214	In house	<25			
6224					
6396 6546					
0340					
	normality	OK			
	n	13			
	outliers	1			
	mean (n)	15.609			
	st.dev. (n)	1.4813			
	R(calc.)	4.148			
	st.dev.(Horwitz)	1.6515			
	R(Horwitz)	4.624			

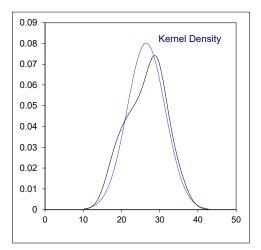




Determination of Acetone on sample #23266; results in mg/kg

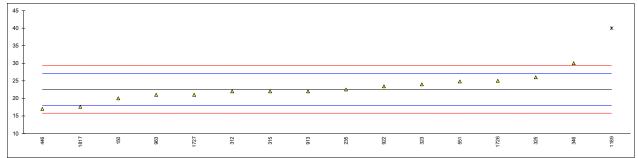
lab	method	value	mark	z(targ)	remarks
150	ETOH-02	25		-0.57	
235	INH-0001	29.77		1.28	
273					
312	INH-0001	23	С	-1.34	first reported 47
315	ETOH-0002	21		-2.11	
323	INH-001	30		1.37	
	INH-0001	30		1.37	
	ETOH-02	29		0.98	
	ANS-008/043	27.5		0.40	
446	INH-043	18		-3.27	
551	INH-1313	28.9		0.94	
912					
	ETOH-002	<5		<-8.30	possibly a false negative test result?
	INH-02	18.30		-3.16	
	D5501	22		-1.73	
1189		28		0.59	
1205					
1438					
1574					
1605				0.50	
1726	la harra	33		2.53	
1817	In house In house	35 24.9240		3.30 -0.59	
1835	In house	<50			
	in nouse	<50			
6214 6224					
6396					
6546					
0340					
	normality	OK			
	n	16			
	outliers	0			
	mean (n)	26.462			
	st.dev. (n)	4.9793			
	R(calc.)	13.942			
	st.dev.(Horwitz)	2.5859			
	R(Horwitz)	7.241			

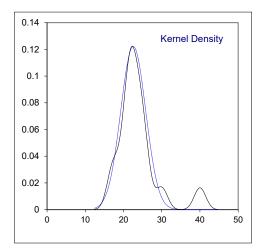




Determination of Benzene on sample #23266; results in mg/kg

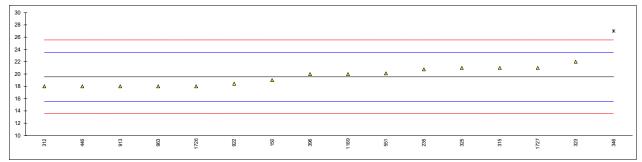
lab	method	value	mark	z(targ)	remarks
150	ETOH-02	20		-1.13	
235	INH-0001	22.53	С	-0.01	first reported 56.00
273					
	INH-0001	22		-0.25	
315		22		-0.25	
	INH-001	24		0.64	
325	INH-0001	26	_	1.52	
346	ETOH-02	30	С	3.30	first reported 37
396					
	INH-043	17		-2.46	
551	INH-1313	24.8		0.99	
912	ETOU 000				
913	ETOH-002	22		-0.25	
	INH-02	23.43		0.39	
963	D5501	21	C(0.04)	-0.69	
1189		40	G(0.01)	7.73	
1205 1438					
1574					
1605					
1726		25		1.08	
	In house	21		-0.69	
1817	In house	17.5914		-2.20	
	In house	<25			
6214					
6224					
6396					
6546					
	normality	OK			
	n	15			
	outliers	1			
	mean (n)	22.557			
	st.dev. (n)	3.2618			
	R(calc.)	9.133			
	st.dev.(Horwitz)	2.2579			
	R(Horwitz)	6.322			
	,				

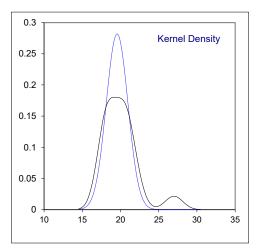




Determination of Isopropanol on sample #23266; results in mg/kg

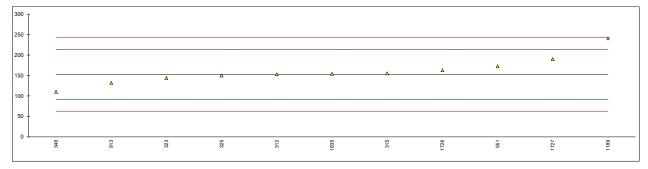
lab	method	value	mark	z(targ)	remarks
	ETOH-02	19		-0.28	
235		20.76	С	0.60	first reported 53.90
273					·
312	INH-0001	18		-0.78	
315	ETOH-0002	21		0.72	
323	INH-001	22		1.22	
325		21		0.72	
	ETOH-02	27	G(0.01)	3.72	
	ANS-008/043	20		0.22	
446		18		-0.78	
551	INH-1313	20.1		0.27	
912					
	ETOH-002	18		-0.78	
	INH-02	18.43		-0.56	
963	D5501	18		-0.78	
1189		20		0.22	
1205					
1438					
1574 1605					
1726		18		-0.78	
	In house	21		0.72	
1817	III IIOuse	Z I		0.72	
	In house	<25			
6214	III IIOGOC				
6224					
6396					
6546					
	normality	OK			
	n	15			
	outliers	1			
	mean (n)	19.553			
	st.dev. (n)	1.4155			
	R(calc.)	3.963			
	st.dev.(Horwitz)	1.9998			
	R(Horwitz)	5.599			

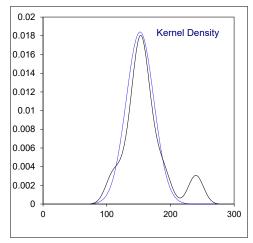




Determination of Total impurities on sample #23266; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150					
235					
273					
312	INH-0001	153	С	0.02	first reported 177
315	ETOH-0002	155		0.09	
323	INH-001	144		-0.28	
325	INH-0001	150		-0.08	
346	ETOH-02	110		-1.40	
396					
446	INH-043	<100			
551	INH-1313	172.4		0.66	
912					
913	ETOH-002	132		-0.67	
922					
963					
1189		240	G(0.05)	2.90	
1205					
1438					
1574					
1605					
1726		163		0.35	
1727	In house	190		1.24	
1817	In house	< 300			
1835	In house	154		0.05	
6214					
6224					
6396					
6546					
	normality	suspect			
	n	10			
	outliers	1			
	mean (n)	152.340			
	st.dev. (n)	21.6692			
	R(calc.)	60.674			
	st.dev.(Horwitz, 7 comp.)	30.2647			
	R(Horwitz, 7 comp.)	84.741			





APPENDIX 2

Other reported impurities in sample #23266; results in mg/kg

lab Acetaldehyde Mono Ethylene glycol (MEG) Other impurities	
150 <5	
235 0.00	
273	
312 < 0.5 15 9	
315	
323 <5 <5	
325 <5 <5 <5	
346 7	
396 <5	
446 <5 <5 <20	
551 3.2 31.74 C	
912	
913 <5 6 10	
922 2.33 < 10	
963 <10	
1189 5 9 100	
1205	
1438	
1574	
1605	
1726 31	
1727 <1 45	
1817 ND < 300	
1835 <10 <50	
6214	
6224	
6396	
6546	

Lab 551 first reported 39.7

APPENDIX 3

Number of participants per country

- 3 labs in BELGIUM
- 1 lab in BRAZIL
- 1 lab in HONG KONG
- 1 lab in HUNGARY
- 2 labs in INDIA
- 1 lab in ISRAEL
- 1 lab in ITALY
- 1 lab in MAURITIUS
- 5 labs in NETHERLANDS
- 2 labs in PAKISTAN
- 1 lab in SAUDI ARABIA
- 1 lab in SOUTH AFRICA
- 4 labs in SPAIN
- 1 lab in THAILAND
- 1 lab in UNITED KINGDOM
- 2 labs in UNITED STATES OF AMERICA

APPENDIX 4

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

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
fr. = first reported

f+? = possibly a false positive test result?f-? = possibly a false negative test result?

SDS = Safety Data Sheet

Literature

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