# **Results of Proficiency Test Ethanol (Food / Neutral grade)** December 2021

Organized by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

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iis21C17 Report:

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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 2021/2022 it was decided to continue the round robin for the analysis of Ethanol (Food / Neutral grade).

In this interlaboratory study 28 laboratories in 19 different 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 an ISO/IEC17025 accredited laboratory. It was decided to send two different samples of Ethanol (Food / Neutral grade): one bottle of 0.5L labelled #21265 for regular analyzes and one bottle of 250mL labelled #21266 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 48 amber glass bottles of 0.5L were filled and labelled #21265.

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 #21265-1	0.80574
sample #21265-2	0.80574
sample #21265-3	0.80574
sample #21265-4	0.80574
sample #21265-5	0.80574
sample #21265-6	0.80574
sample #21265-7	0.80574
sample #21265-8	0.80574

Table 1: homogeneity test results of subsamples #21265

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)	0.0000		
reference test method	ISO12185:96		
0.3 x R (reference test method)	0.00015		

Table 2: evaluation of the repeatability of subsamples #21265

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 a batch of approximately 22 liters Ethanol (Food / Neutral grade) was made available from the retain materials from earlier PTs. After homogenization 48 amber glass bottles of 250mL were filled and labelled #21266. The homogeneity of the subsamples was checked by determination of Isopropanol and Methanol in accordance with an in-house method on 8 stratified randomly selected subsamples.

	Isopropanol in mg/kg	Methanol in mg/kg
sample #21266-1	6.0	10.2
sample #21266-2	6.1	10.1
sample #21266-3	6.1	10.2
sample #21266-4	6.1	10.3
sample #21266-5	6.2	10.0
sample #21266-6	5.7	10.2
sample #21266-7	5.9	10.3
sample #21266-8	5.9	10.0

Table 3: homogeneity test results of subsamples #21266

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

	Isopropanol in mg/kg	Methanol in mg/kg
r (observed)	0.4	0.3
reference method	Horwitz	Horwitz
0.3 x R (reference method)	0.6	1.0

Table 4: evaluation of the repeatabilities of subsamples #21266

The calculated repeatabilities are in agreement with 0.3 times the estimated reproducibilities calculated with the Horwitz equation. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample Ethanol (Food / Neutral grade) labelled #21265 and one sample Ethanol (Food / Neutral grade) for GC only labelled #21266 was sent on November 10, 2021. 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 #21265: 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 #21266 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 appendix 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 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 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  $z_{(target)}$  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 the dispatch of the samples. Therefore, the reporting time on the data entry portal was extended with another two weeks. When considering the test results of the two samples together two participants reported test results after the final reporting date and one participant did not report any test result. Not all participants were able to perform all analyzes requested.

In total 27 participants reported 311 numerical test results. Observed were 8 outlying test results, which is 2.6%. 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). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D1363:06(2019)). In the test results tables of appendix 1 only the method number (sub) and year of adoption or revision (e.g. D1363:06) will be used.

<u>Appearance</u>: This determination was not problematic. All reporting participants agreed on a test result as Pass (Clear and Bright).

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

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

<u>Permanganate Time Test at 20°C</u>: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D1363:06(2019).

<u>pHe</u>: It is known that the pHe determined with a LiCl electrode will be lower than the pHe determined with a KCl electrode. 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>: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of EN15490:07.
- <u>pHe with KCl electrode</u>: This determination may be problematic (only three test results were reported). No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D6423:20a.
- <u>Strength (%M/M)</u>: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the reproducibility derived from the OIML table.
- Strength (%V/V): This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the reproducibility derived from the OIML table.

Water:

This determination was not problematic. 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 but is not in agreement with the strict requirements of ASTM E203:16.

Regretfully, no reference test method for this determination 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 preferable. In this PT nine laboratories used a 50 mm cuvette and eight laboratories used a 10 mm cuvette. Both groups were evaluated separately.

<u>UV - 50 mm cuvette</u>: In total over seven parameters (UV absorbance in nm) one statistical outlier was observed. Seven participants evaluated the sample as 'Pass' and one other evaluated as 'Not Smooth'.

<u>UV - 10 mm cuvette</u>: In total over seven parameters (UV absorbance in nm) two statistical outliers were observed. Five participants evaluated the sample as 'Pass' while two other evaluated as 'Fail'.

Purity of Ethanol on dry basis: This determination may not be problematic. One statistical outlier was 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 could be calculated.

<u>Methanol</u>: This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

<u>Benzene</u>: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the estimated reproducibility calculated with the Horwitz equation.

<u>Isopropanol</u>: This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

Other impurities: This determination may be problematic. No statistical outliers were observed. Due to a large variation in the test results it was decided not to calculate z-scores.

Acetone:

Total impurities: This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with the Horwitz equation for 7 components.

For impurities not listed above, but mentioned in paragraph 2.6, the participants agree on a concentration near or below the limit of detection. Therefore, these impurities were not further evaluated. 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 literature reference test methods (in casu ASTM, EN and ISO test methods) or the target reproducibility derived from the official test method or estimated using the Horwitz equation are presented in the next tables.

Parameter	unit	n	average	2.8 * sd	R(lit)
Appearance		19	Pass (C&B)	n.a.	n.a.
Density at 20°C	kg/L	23	0.8058	0.0002	0.0005
Nonvolatile matter	mg/100mL	14	<1	n.e.	n.e.
Permanganate Time Test 20°C	minutes	10	32.7	11.1	8.2
pHe with LiCl electrode		5	7.7	0.7	0.7
pHe with KCI electrode		3	8.1	1.3	1.0
Strength	%M/M	13	94.42	0.05	0.06
Strength	%V/V	19	96.40	0.03	0.06
Water	%M/M	17	5.55	0.20	0.48
UV – 50 mm cuvette:					
UV-absorbance 300 nm		8	0.012	0.008	n.a.
UV-absorbance 270 nm		9	0.038	0.007	n.a.
UV-absorbance 260 nm		9	0.062	0.015	n.a.
UV-absorbance 250 nm		9	0.118	0.014	n.a.
UV-absorbance 240 nm		9	0.246	0.027	n.a.
UV-absorbance 230 nm		8	0.559	0.068	n.a.
UV-absorbance 220 nm		8	1.181	0.207	n.a.
Conclusion UV-scan		7	Pass	n.a.	n.a.
UV – 10 mm cuvette:					
UV-absorbance 300 nm		7	0.002	0.005	n.a.
UV-absorbance 270 nm		8	0.010	0.013	n.a.
UV-absorbance 260 nm		8	0.015	0.014	n.a.
UV-absorbance 250 nm		8	0.027	0.016	n.a.
UV-absorbance 240 nm		8	0.055	0.020	n.a.
UV-absorbance 230 nm		8	0.120	0.041	n.a.

Parameter	unit	n	average	2.8 * sd	R(lit)
UV-absorbance 220 nm		7	0.238	0.037	n.a.
Conclusion UV-scan		5	Pass	n.a.	n.a.

Table 5: reproducibilities of tests on sample #21265

Parameter	unit	n	average	2.8 *sd	R(target)
Purity of Ethanol on dry basis	%M/M	17	99.99	0.006	n.e.
Methanol	mg/kg	17	11.4	8.2	3.5
Acetone	mg/kg	16	16.8	9.1	4.9
Benzene	mg/kg	7	3.1	1.3	1.2
Isopropanol	mg/kg	14	6.2	2.6	2.1
Other impurities	mg/kg	10	30.2	69.9	(14.0)
Total impurities	mg/kg	12	67.7	78.4	42.5

Table 6: reproducibilities of tests on sample #21266

Results between brackets no z-scores are calculated

Without further statistical calculations it can be concluded that for many tests there is not 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 2021 WITH PREVIOUS PTS

	December 2021	December 2020	December 2019	December 2018	December 2017
Number of reporting laboratories	27	24	25	25	29
Number of test results	311	315	337	303	301
Number of statistical outliers	8	19	19	20	22
Percentage of statistical outliers	2.6%	6.0%	5.6%	6.6%	7.3%

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 2021	December 2020	December 2019	December 2018	December 2017
Density at 20°C	++	++	++	++	++
Nonvolatile matter	n.e.	n.e.	-	++	++
Permanganate Time Test 20°C	-	-	+	-	-
pHe with LiCl electrode	+/-	-	-	++	-
pHe with KCI electrode	-	-			
Strength %M/M	+	-	+	++	++
Strength %V/V	++	+	+/-	+	++
Water	++	+	+	-	-

Parameter	December 2021	December 2020	December 2019	December 2018	December 2017
Purity Ethanol on dry basis	n.e.	(+/-)	(++)	(+)	(+)
Methanol		-	-		-
Acetal (1,1-diethoxyethane)	n.e.	n.e.	+	n.e.	n.e.
Acetaldehyde	n.e.	n.e.	n.a.	n.a.	n.a.
Acetone	-	-	+	+/-	
Benzene	+/-	-	-		n.e.
Isopropanol	-	-	-	+	+
Mono Ethylene glycol (MEG)	n.e.	()	n.e.	n.e.	n.e.
Other impurities	()	()	n.a.	n.a.	n.a.
Total impurities		+	-	-	-

Table 8: comparison determinations against the reference test methods

Results between brackets should be used with due care.

## 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

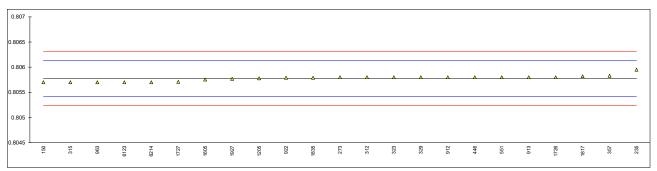
## **APPENDIX 1**

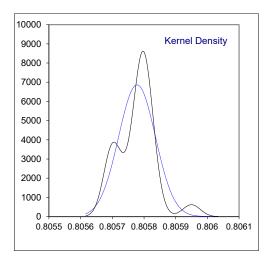
Determination of Appearance on sample #21265;

lab	method	value	mark	z(targ)	remarks
150	Visual	C&B			
235	Visual	C&B			
273	Visual	Pass			
312	Visual	CLCOL			
315	E2680	pass			
323	E2679	C&B			
329	Visual	clear liquid			
357		CFSM = Clear and free of suspended matter			
446	E2680	Pass			
551	Visual	Pass			
859					
912	Visual	Clear and bright			
913	E2680	Clear and Bright			
922	Visual	Clear & Bright			
963	Visual	Pass			
1205					
1438					
1574					
1605					
1726	Visual	Clear&Colorless			
1727	Visual	Clear&Colorless			
1817	Visual	Pass			
1835	Visual	C&C			
1927					
6123					
6214	Visual	clear & colourless			
6224					
6403					
	n	19			
	mean (n)	Clear and Bright / Pass			
	()				

## Determination of Density at 20°C on sample #21265; results in kg/L

lab	method	value	mark	z(targ)	remarks	
150	D4052	0.8057		-0.44		
235	D4052	0.80595		0.96		
273	D4052	0.8058		0.12		
312	ISO12185	0.8058		0.12		
315	D4052	0.8057		-0.44		
323	D4052	0.8058		0.12		
329	D4052	0.8058		0.12		
357	D4052	0.80583		0.29		
446	D4052	0.8058		0.12		
551	D4052	0.8058		0.12		
859						
912	ISO3675	0.8058		0.12		
913	D4052	0.8058		0.12		
922	D4052	0.80579		0.07		
963	ISO12185	0.8057		-0.44		
1205	In house	0.805779		0.00		
1438						
1574						
1605	D4052	0.805755		-0.13		
1726	D4052	0.80580		0.12		
1727	D4052	0.80571		-0.38		
1817	Table OIML	0.80582		0.23		
1835	ISO12185	0.80579		0.07		
1927	D4052	0.80577		-0.05		
6123	ISO3838	0.8057		-0.44		
6214	ISO12185	0.8057		-0.44		
6224						
6403						
	normality	not OK				
	n	23				
	outliers	0				
	mean (n)	0.805778				
	st.dev. (n)	0.0000581				
	R(calc.)	0.000163				
	st.dev.(ISO12185:96)	0.0001786				
	R(ISO12185:96)	0.0005				



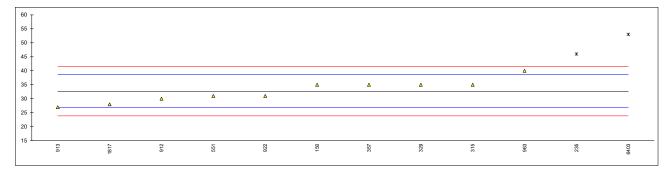


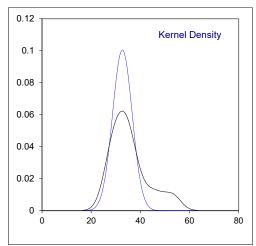
## Determination of Nonvolatile matter on sample #21265; results in mg/100mL

lab	method	value	mark	z(targ)	remarks
150	D1353	0.2			
235	D1353	0.5			
273					
312	D1353	<1			
315	D1353	< 1			
323	D1353	< 1			
329	D1353	0.8	С		first reported 8
357	D1353	< 1			
446	D1353	0			
551	D1353	<0.1			
859					
912	D1353	0.6			
913	D1353	<1			
922	D1353	<1.0			
963					
1205					
1438					
1574					
1605					
1726	EN15691	<10			
1727	EN15691	<1			
1817	In house	0			
1835	EN15691	<10			
1927					
6123					
6214					
6224					
6403					
	n	14			
	mean (n)	<1			

## Determination of Permanganate Time Test at 20°C on sample #21265; results in minutes

lab	method	value	mark	z(targ)	remarks
150	D1363	35		0.78	
235	D1363	46	DG(0.05)	4.52	
273					
312					
315	D1363	35		0.78	
323					
329	D1363	35		0.78	
357	D1363	35		0.78	
446	D1363	>20			
551	D1363	31		-0.58	
859					
912	D1363	30		-0.92	
913	D1363	27		-1.94	
922	D1363	31		-0.58	
963	D1363	40.0		2.48	
1205					
1438					
1574					
1605					
1726					
1727					
1817	In house	28		-1.60	
1835					
1927					
6123					
6214					
6224			DO(0.05)		
6403	In house	53	DG(0.05)	6.90	
	normality	OK			
	n	10			
	outliers	2			
	mean (n)	32.70			
	st.dev. (n)	3.974			
	R(calc.)	11.13			
	st.dev.(D1363:06)	2.943			
	R(D1363:06)	8.24			
	. ,				





## Determination of pHe with LiCl electrode on sample #21265;

lab	method	value	mark	z(targ)	remarks	
150						
235						
273						
312						
315						
323						
329						
357						
446	NDD40004	7.00		4.07		
551 859	NBR10891	7.33		-1.37		
912						
912						
922						
963	EN15490	7.55		-0.53		
1205	L. 110-000	7.00		-0.55		
1438						
1574						
1605						
1726	EN15490	7.96		1.02		
1727	EN15490	7.77		0.30		
1817						
1835	EN15490	7.84		0.57		
1927						
6123						
6214						
6224						
6403						
	normality	unknown				
	n	5				
	outliers	0				
	mean (n)	7.690				
	st.dev. (n)	0.2505				
	R(calc.)	0.701				
	st.dev.(EN15490:07)	0.2637				
	R(EN15490:07)	0.738				
	,					
9 T						
8.5 -						_
8 +	-					-
"					Δ	_
7.5 -		Δ				-
	Δ					_
7 +						_
6.5 +						
0.5						
6	Σ	e e			<u> </u>	<u> </u>
	55.	963			1727	17.26

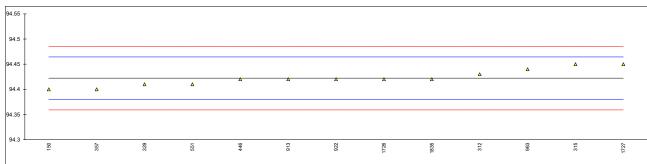
## Determination of pHe with KCI electrode on sample #21265;

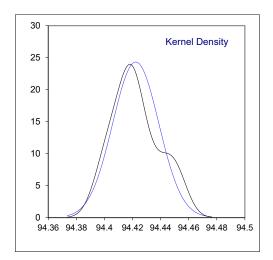
lab	method	value	mark	z(targ)	remarks
150	D6423	8.5		1.23	
235					
273					
312					
315					
323					
329 357					
446					
551	D6423	8.05		0.00	
859	D0423				
912					
913					
922	D6423	7.6		-1.23	
963					
1205					
1438					
1574					
1605					
1726					
1727 1817					
1835					
1927					
6123					
6214					
6224					
6403					
	normality	unknown			
	n outliers	3 0			
	mean (n)	8.050			
	st.dev. (n)	0.4500			
	R(calc.)	1.260			
	st.dev.(D6423:20a)	0.3668			
	R(D6423:20a)	1.027			
	•				
<sup>10</sup> T					
9.5 -					
9 +					
8.5 +					Δ
					<u>a</u>
8 +		Δ			
7.5 -	Δ				
7					



## Determination of Strength on sample #21265; results in %M/M

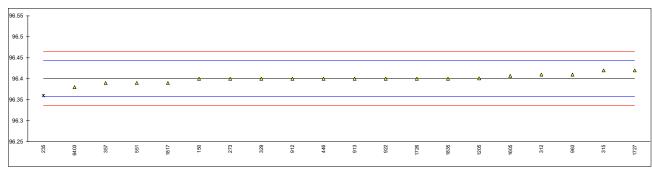
lab	method	value	mark z(targ)	remarks
150	Table OIML	94.4	-1.06	
235				
273				
312	Table OIML	94.43	0.37	
315	Table OIML	94.45	1.31	
323				
329	Table OIML	94.41	-0.58	
357	Table OIML	94.40	-1.06	
446	Table OIML	94.42	-0.11	
551	Table OIML	94.41	-0.58	
859				
912				
913	Table OIML	94.42	-0.11	
922	Table OIML	94.42	-0.11	
963	Table OIML	94.44	0.84	
1205				
1438				
1574				
1605				
1726	Table OIML	94.42	-0.11	
1727	Table OIML	94.45	1.31	
1817	T 11 01141			
1835	Table OIML	94.42	-0.11	
1927				
6123				
6214				
6224				
6403				
	normality	OK		
	n	13		
	outliers	0		
	mean (n)	94.422		
	st.dev. (n)	0.0164		
	R(calc.)	0.046		
	st.dev.(OIML table)	0.0211		
	R(OIML table)	0.059		OIML R022-e75
	•			

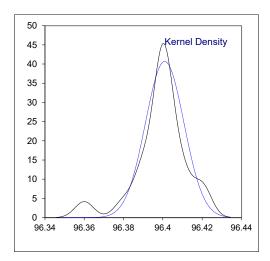




## Determination of Strength on sample #21265; results in %V/V

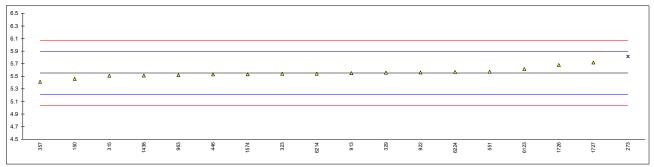
lab	method	value	mark	z(targ)	remarks
150	Table OIML	96.4		-0.04	
235	Table OIML	96.36	D(0.01)	-1.91	
273	Table OIML	96.40	, ,	-0.04	
312	Table OIML	96.41		0.42	
315	Table OIML	96.42		0.89	
323					
329	Table OIML	96.40		-0.04	
357	Table OIML	96.39		-0.51	
446	Table OIML	96.40		-0.04	
551	Table OIML	96.39		-0.51	
859					
912	Table OIML	96.40		-0.04	
913	Table OIML	96.40		-0.04	
922	Table OIML	96.40		-0.04	
963	Table OIML	96.41		0.42	
1205	Table OIML	96.401		0.00	
1438					
1574	<b>-</b>				
1605	Table OIML	96.407		0.28	
1726	Table OIML	96.40		-0.04	
1727	Table OIML	96.42		0.89	
1817	Table OIML	96.39		-0.51	
1835	Table OIML	96.40		-0.04	
1927					
6123					
6214 6224					
6403	Table OIML	96.38		-0.98	
0403	Table Olivic	90.30		-0.96	
	normality	OK			
	n	19			
	outliers	1			
	mean (n)	96.401			
	st.dev. (n)	0.0098			
	R(calc.)	0.027			
	st.dev.(OIML table)	0.0214			
	R(OIML table)	0.060			OIML R022-e75

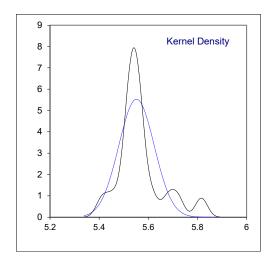




## Determination of Water on sample #21265; results in %M/M

lab	method	value	mark	z(targ)	remarks
150	E203	5.459		-0.54	
235					
273	E203	5.816	G(0.05)	1.54	
312					
315	E203	5.51		-0.25	
323	E203	5.540		-0.07	
329	E203	5.558		0.03	
357	E203	5.413		-0.81	
446 551	D1364 E203	5.53 5.573		-0.13 0.12	
859	E203	5.575		0.12	
912					
913	E203	5.551		-0.01	
922	E203	5.56		0.05	
963	E203	5.52		-0.19	
1205					
1438	E203	5.512		-0.23	
1574		5.5308		-0.12	
1605					
1726	EN15692	5.6803		0.75	
1727	D1364	5.72		0.98	
1817					
1835					
1927	1001000				
6123	ISO12937	5.617		0.38	
6214	la hacea	5.54164		-0.06	
6224 6403	In house	5.57		0.10	
0403					
	normality	suspect			
	n	17			
	outliers	1			
	mean (n)	5.5521			
	st.dev. (n)	0.07216			
	R(calc.)	0.2021			
	st.dev.(Horwitz)	0.17158			
	R(Horwitz)	0.4804			
	compare				
	R(E203:16)	0.078			

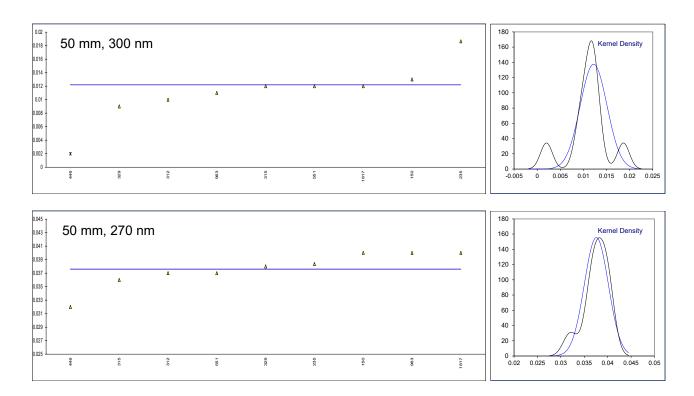


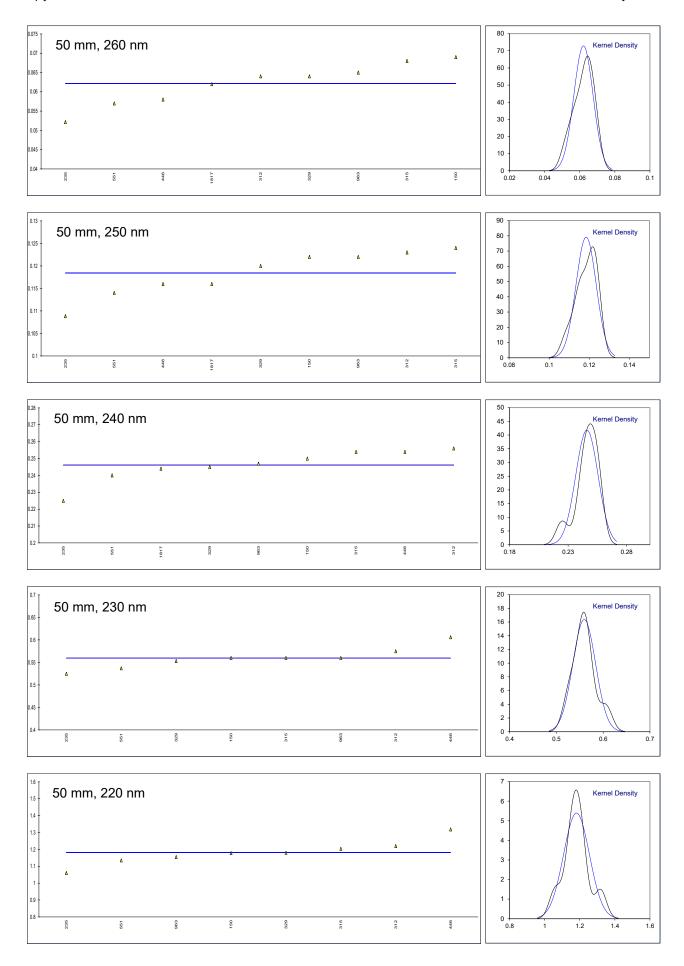


## Determination of UV absorbance (50 mm cuvette) on sample #21265;

lab	method	300 nm	270 nm	260 nm	250 nm	240 nm	230 nm	220 nm	Pass/Fail
150	IMPCA004	0.013	0.040	0.069	0.122	0.250	0.560	1.178	Pass
235	INH-13-001	0.01865 C	0.03835 C	0.05215 C	0.10885 C	0.22495 C	0.5247 C	1.06005 C	Pass
273									
312		0.010	0.037	0.064	0.123	0.256	0.575	1.220	
315	INH-013	0.012	0.036	0.068	0.124	0.254	0.560	1.204	Pass
323									Pass
329	INH-CM	0.009	0.038	0.064	0.120	0.245	0.553	1.180	Pass
357									
446	INH-13	0.002 D5	0.032	0.058	0.116	0.254	0.606	1.318	Pass
551	INH-3063	0.012	0.037	0.057	0.114	0.240	0.537	1.135	Not smooth
859									
912									
913									
922									
963	IMPCA004	0.011	0.040	0.065	0.122	0.247	0.560	1.155	Pass
1205									
1438									
1574									
1605									
1726									
1727									
1817	In house	0.012	0.040	0.062	0.116	0.244			
1835									
1927									
6123									
6214									
6224									
6403									
	normality	not OK	not OK	ОК	OK	not OK	unknown	unknown	
	n	8	9	9	9	9	8	8	7
	outliers	1	0	0	0	0	0	0	'
	mean (n)	0.0122	0.0376	0.0621	0.1184	0.2461	0.5595	1.1813	Pass
	st.dev. (n)	0.00290	0.00257	0.00548	0.00504	0.00955	0.02439	0.07392	1 433
	R(calc.)	0.00230	0.00237	0.0153	0.0141	0.0267	0.0683	0.2070	
	i t(Gaio.)	0.0001	0.0012	0.0100	0.0171	0.0201	0.0000	0.2010	

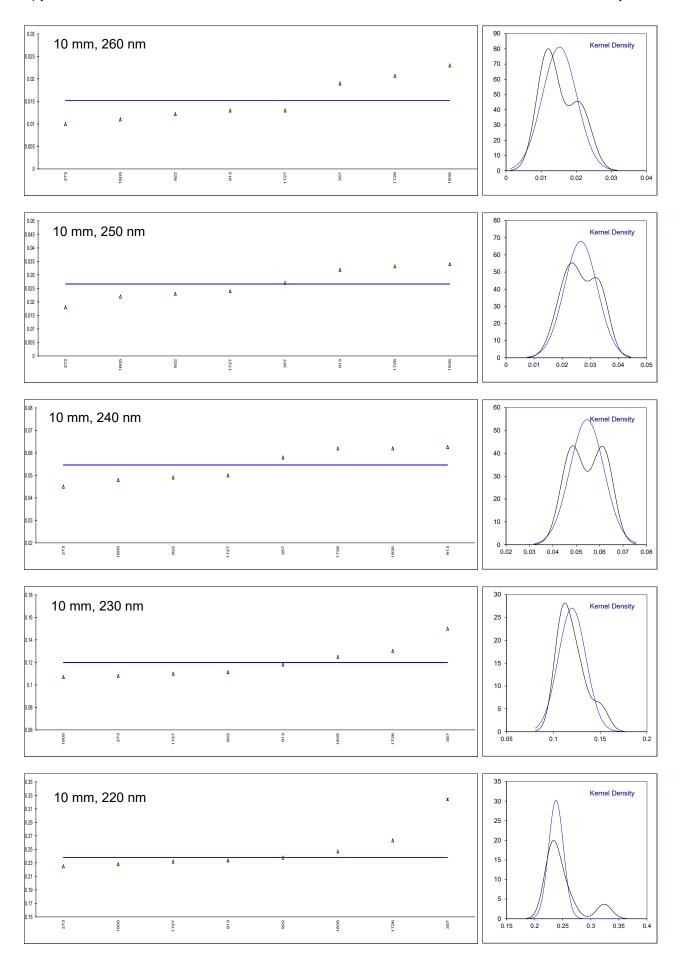
 $Lab\ 235\ first\ reported\ 0.0000,\ 0.0014,\ 0.03015,\ 0.0763,\ 0.19575,\ 0.5033,\ 1.1028\ respectively$ 





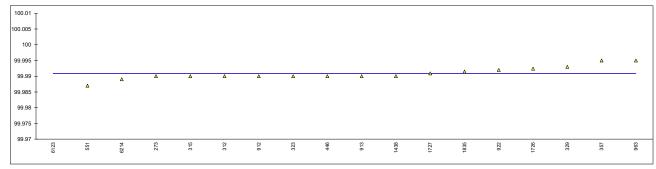
## Determination of UV absorbance (10 mm cuvette) on sample #21265;

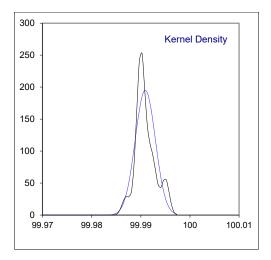
lab	method	300 nm	270 nm	260 nm	250 nm	240 nm	230 nm	220 nm	Pass/Fail
150 235									
273	IMPCA004	0.004	0.007	0.010	0.018	0.045	0.108	0.225	
312									
315									
323 329									
357	INH-13-001	0.002	0.013	0.019	0.027	0.058	0.150	0.324 D5	Pass
446									
551									
859 912									
913	IMPCA004	0.0003	0.0061	0.0130	0.0319	0.0626	0.1182	0.2337	Fail
922		0.0012	0.0070	0.0122	0.0230	0.0491	0.1113	0.2379	Fail
963									
1205 1438									
1574									
1605		0.000	0.006	0.011	0.022	0.048	0.107	0.228	Pass
1726 1727	IMPCA004 IMPCA004	0.0053 0.003	0.0151 0.008	0.0207 0.013	0.0332 0.024	0.0620 0.050	0.1303 0.110	0.2633 0.232	Pass Pass
1817	IIVII CA004				0.024	0.050		0.232	
1835		0.011 G5	0.018	0.023	0.034	0.062	0.125	0.247	Pass
1927									
6123 6214									
6224									
6403									
	normality	unknown	_						
	n outliers	7 1	8 0	8 0	8 0	8 0	8 0	7 1	5
	mean (n)	0.0023	0.0100	0.0152	0.0266	0.0546	0.1200	0.2381	Pass
	st.dev. (n)	0.00196	0.00466	0.00491	0.00587	0.00729	0.01475	0.01319	
	R(calc.)	0.0055	0.0131	0.0138	0.0164	0.0204	0.0413	0.0369	
0.012	0.000						250		
0.01	0 mm, 300 n	m				ж			ernel Density
							200 -	$\wedge$	
0.008							150 -	/ \	
0.006									
					Δ.	•	100 -		
0.004 +				Δ	Δ			/ \	
0.002 +		Δ	Δ				50 -		
0	913 A	922	367	727	273	88	-0.01 -0.	005 0 0.005	0.01 0.015 0.02
	£ 0	V		<del></del>		. *			
0.02 <sub>T</sub>							100		
	0 mm, 270 n	m				Δ	90 -	_ к	ernel Density
0.016					4		80 -	$/ \sim$	
0.014 +					Δ	•	70 -	//\ \	
0.012							60 -	// \ \	
0.01				Δ			50		
0.006	Δ Δ	Δ	Δ	•			30 -	//	
0.004							20 -	/	\\
1 1							11 1	/	\ \ \
0.002							10 -	1	



## Determination of Purity of Ethanol on dry basis on sample #21266; results in %M/M

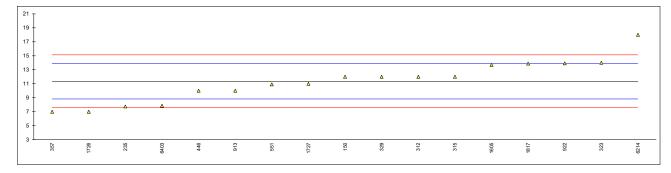
lab	method	value	mark	z(targ)	remarks
150					
235					
273	IMPCA004	99.99	С		first reported 99.96
312	INH-0001	99.99			
315	INH-933	99.99			
323	INH-001	99.99			
329	INH-0001	99.993			
357	INH-02	99.995	С		first reported 99.987
446	INH-043	99.99			
551	INH-1313	99.987			
859					
912	INH-02	99.99			
913	INH-0002	99.99			
922	INH-02	99.992			
963	D5501	99.995			
1205					
1438		99.99			
1574					
1605					
1726	In house	99.9924			
1727		99.9909			
1817					
1835	In house	99.9915			
1927			0.0000		
6123	In house	99.86	C,D(0.01)		first reported 94.1
6214		99.98905	С		first reported 95.0477
6224					
6403					
	normality	OK			
	n	17			
	outliers	1			
	mean (n)	99.9909			
	st.dev. (n)	0.00205			
	R(calc.)	0.0057			

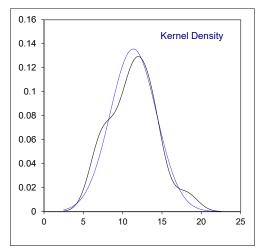




## Determination of Methanol on sample #21266; results in mg/kg

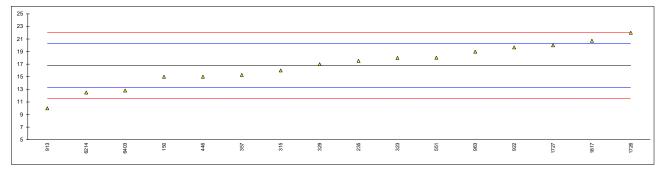
lab	method	value	mark z(targ	) remarks
150	INH-02	12	0.5	,
235	INH-01	7.75	-2.8	
273				
312	INH-0001	12	0.5	1
315	INH-933	12	0.5	
323	INH-001	14	2.1	
329	INH-0001	12	0.5	1
357	INH-02	7	-3.4	3
446	INH-043	10	-1.0	3
551	INH-1313	10.91	-0.3	5
859				-
912				
913	INH-0002	10	-1.0	3
922	INH-02	13.93	2.0	4
963	D5501	<10		-
1205				-
1438				-
1574				
1605		13.7	1.8	
1726	In house	7	-3.4	
1727		11	-0.2	
1817	In house	13.8860	2.0	1
1835	In house	<25		-
1927				-
6123				
6214		18	5.2	7
6224				
6403	In house	7.86	-2.7	7
	normality	OK		
	n	17		
	outliers	0		
	mean (n)	11.355		
	st.dev. (n)	2.9421		
	R(calc.)	8.238		
	st.dev.(Horwitz)	1.2603		
	R(Horwitz)	3.529		

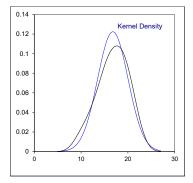




## Determination of Acetone on sample #21266; results in mg/kg

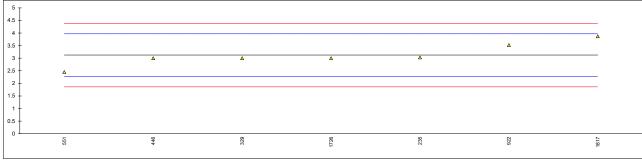
lab	method	value	mark	z(targ)	remarks
150	INH-02	15		-1.02	
235	INH-01	17.52		0.42	
273					
312	INH-0001	<5		<-6.71	possibly a false negative test result?
315	INH-933	16		-0.45	
323	INH-001	18		0.69	
329	INH-0001	17		0.12	
357	INH-02	15.3	С	-0.85	first reported 97
446	INH-043	15		-1.02	
551	INH-1313	18.04		0.71	
859					
912					
913	INH-0002	10		-3.86	
922	INH-02	19.67		1.64	
963	D5501	19		1.26	
1205					
1438					
1574					
1605					
1726	In house	22		2.97	
1727		20		1.83	
1817	In house	20.7455		2.25	
1835	In house	<25			
1927					
6123		40.5		0.44	
6214		12.5		-2.44	
6224	In the case of	40.00			
6403	In house	12.82		-2.26	
	normality	OK			
	n	16			
	outliers	0			
	mean (n)	16.787			
	st.dev. (n)	3.2598			
	R(calc.)	9.128			
	st.dev.(Horwitz)	1.7568			
	R(Horwitz)	4.919			

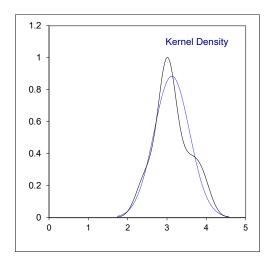




## Determination of Benzene on sample #21266; results in mg/kg

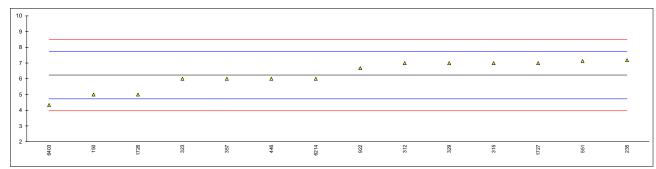
lab	method	value	mark	z(targ)	remarks
150	INH-02	<5			
235	INH-01	3.03		-0.22	
273					
312	INH-0001	<5			
315	INH-933	<5			
323	INH-001	< 5			
329	INH-0001	3		-0.30	
357	INH-02	< 5			
446	INH-043	3		-0.30	
551	INH-1299	2.45		-1.60	
859					
912					
913	INH-0002	<10			
922	INH-02	3.52		0.94	
963	D5501	<10			
1205					
1438					
1574					
1605					
1726	In house	3		-0.30	
1727		nd			
1817	In house	3.8697		1.77	
1835	In house	<10			
1927					
6123					
6214					
6224					
6403					
	normality	unknown			
	n	7			
	outliers	0			
	mean (n)	3.124			
	st.dev. (n)	0.4513			
	R(calc.)	1.264			
	st.dev.(Horwitz)	0.4211			
	R(Horwitz)	1.179			
	, ,				

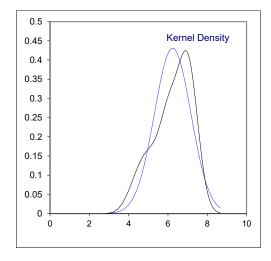




## Determination of Isopropanol on sample #21266; results in mg/kg

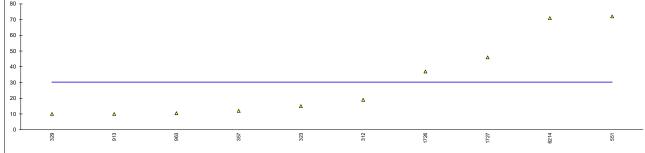
lab	moth od	value		va maulta
lab	method	value	mark z(targ)	remarks
150	INH-02	5	-1.63	
235	INH-01	7.18	1.24	
273				
312	INH-0001	7	1.01	
315	INH-933	7	1.01	
323	INH-001	6	-0.31	
329	INH-0001	7	1.01	
357	INH-02	6	-0.31	
446	INH-043	6	-0.31	
551	INH-1313	7.13	1.18	
859				
912				
913	INH-0002	<10		
922	INH-02	6.68	0.58	
963	D5501	<10		
1205				
1438				
1574				
1605				
1726	In house	5	-1.63	
1727		7	1.01	
1817				
1835	In house	<25		
1927				
6123				
6214		6	-0.31	
6224				
6403	In house	4.33	-2.52	
	normality	OK		
	n	14		
	outliers	0		
	mean (n)	6.237		
	st.dev. (n)	0.9254		
	R(calc.)	2.591		
	st.dev.(Horwitz)	0.7576		
	R(Horwitz)	2.121		
	,			

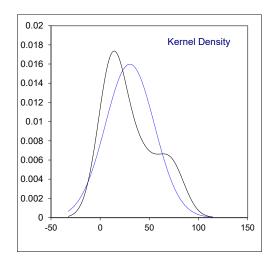




## Determination of Other impurities on sample #21266; results in mg/kg

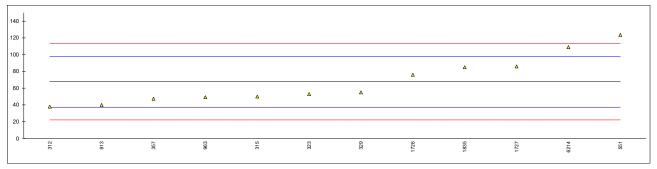
lab	method	value	mark	z(targ)	remarks	
150						
235						
273						
312	INH-0001	19				
315	INH-933	<50				
323	INH-001	15				
329	INH-0001	10				
357	INH-02	12				
446	INH-043	<20				
551	INH-1313	72.04				
859						
912						
913	INH-0002	10				
922						
963	D5501	10.42				
1205						
1438						
1574						
1605						
1726	In house	37				
1727		46				
1817	In house	< 300				
1835	In house	<50				
1927						
6123						
6214		71				
6224						
6403						
	normality	OK				
	n	10				
	outliers	0				
	mean (n)	30.246				
	st.dev. (n)	24.9580				
	R(calc.)	69.883				
	st.dev.(Horwitz, comp:3)	(5.0175)				
	R(Horwitz, comp:3)	(14.049)				
	, , , ,	7				
<sup>80</sup> T						
70					Δ Δ	Δ
60 +						
UU T						

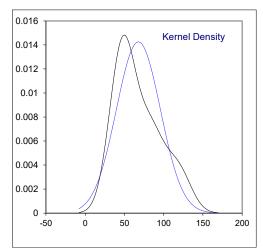




## Determination of Total impurities on sample #21266; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150					
235					
273					
312	INH-0001	38		-1.95	
315	INH-933	50		-1.16	
323	INH-001	53		-0.97	
329	INH-0001	55		-0.83	
357	INH-02	47.3	С	-1.34	first reported 129
446	INH-043	<50			
551	INH-1313	123.60		3.68	
859					
912					
913	INH-0002	40		-1.82	
922					
963	D5501	49.31		-1.21	
1205					
1438					
1574					
1605	In the case of	70			
1726 1727	In house	76 86		0.55	
1817				1.21	
1835	In house	85		1.14	
1927	III IIouse			1.14	
6123					
6214		109		2.72	
6224		100		2.72	
6403					
0400					
	normality	OK			
	n	12			
	outliers	0			
	mean (n)	67.684			
	st.dev. (n)	28.0064			
	R(calc.)	78.418			
	st.dev.(Horwitz, comp:7)	15.1929			
	R(Horwitz, comp:7)	42.540			
	•				





APPENDIX 2

Other reported impurities in sample #21266; results in mg/kg

lab	Acetal (1,1-diethoxyethane)	Acetaldehyde	Mono Ethylene glycol (MEG)
150	<5	<5	
235		0.183	
273			
312	<5	<5	<5
315	<5	<5	<5
323	< 5	< 5	
329	<5	<5	4
357	< 5	< 5	< 30
446	<5	<5	<5
551	1.80	3.22	8.01
859			
912			
913	<10	<10	<10
922	2.27	<2.0	7.2
963		<10	
1205			
1438			
1574			
1605			
1726	2		
1727	2 _	<1_	
1817	< 5	< 5	
1835	<25	<10	
1927			
6123			
6214	2	0	
6224			
6403			

#### **APPENDIX 3**

## Number of participants per country

- 3 labs in BELGIUM
- 1 lab in BRAZIL
- 1 lab in CHINA, People's Republic
- 1 lab in FINLAND
- 1 lab in HONG KONG
- 1 lab in HUNGARY
- 2 labs in INDIA
- 1 lab in ISRAEL
- 1 lab in MAURITIUS
- 4 labs in NETHERLANDS
- 1 lab in PAKISTAN
- 1 lab in ROMANIA
- 1 lab in SAUDI ARABIA
- 1 lab in SERBIA
- 1 lab in SOUTH AFRICA
- 3 labs in SPAIN
- 2 labs in THAILAND
- 1 lab in UNITED KINGDOM
- 1 lab 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 D(0.01) = outlier in Grubbs' outlier test D(0.05) = straggler in Grubbs' outlier test D(0.05) = outlier in Double Grubbs' outlier test D(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

- iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
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