Results of Proficiency Test Ethanol/Gasoline mix December 2016

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1 INTRODUCTION

Since 2013, the Institute for Interlaboratory Studies organises a proficiency test for the analysis of Ethanol/Gasoline mixtures every year. It was decided to continue this interlaboratory study in the 2016/2017 program. In this interlaboratory study, 16 laboratories in 8 different countries did register for participation. See appendix 2 for a list of number of participants per country. In this report the results of the 2016 proficiency test are presented and discussed. This report is electronically available through the iis website www.iisnl.com.

2 SET-UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. Sample analysis for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send three different samples of Ethanol/Gasoline mixtures (each one in a 30 ml bottle, labelled resp. #16265, #16266 and #16267). Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for the statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC 17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on a regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). The protocol can be downloaded from 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

The necessary bulk materials Fuel Ethanol (99%M/M) and Gasoline (EN228) were obtained from a local supplier. The following three different mixtures were prepared:

Sample id	Mixture	approx. composition
#16265	Ethanol/Gasoline	80 / 20 %V/V
#16266	Ethanol/Gasoline	60 / 40 %V/V
#16267	Ethanol/Gasoline	20 / 80 %V/V

Table 1: Composition of #16265, #16266 and #16267

Of each mixture a bulk amount of 2 litre was prepared. Out of each mixture were after homogenisation, 50 amber glass bottles of 30 ml filled and labelled. The homogeneity of the sub samples was checked by determination of Density in accordance with ASTM D4052 on 7 stratified randomly selected samples.

Sample	Density at 15°C in kg/L (sample #16265)	Density at 15°C in kg/L (sample #16266)	Density at 15°C in kg/L (sample #16267)
Sample 1	0.78210	0.77004	0.74616
Sample 2	0.78227	0.77026	0.74629
Sample 3	0.78211	0.77012	0.74618
Sample 4	0.78230	0.77005	0.74630
Sample 5	0.78227	0.77004	0.74614
Sample 6	0.78205	0.77012	0.74628
Sample 7	0.78210	0.77018	0.74629

Table 2: Homogeneity test results of sub samples #16265, #16266, #16267

From the test results of table 2 the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility of the reference test method in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density at 15°C in kg/L (sample #16265)	Density at 15°C in kg/L (sample #16266)	Density at 15°C in kg/L (sample #16267)
r (observed)	0.00029	0.00023	0.00020
reference test method	ASTM D4052:16	ASTM D4052:16	ASTM D4052:16
0.3 * R (ref. test method)	0.00028	0.00039	0.00062

Table 3: Repeatability of sub samples #16265, #16266 and #16267

The repeatabilities of the test results from the homogeneity test for sample #16265, #16266 and #16267 were in agreement with the requirements of the reference test method. Therefore, homogeneity of the sub samples of #16265, #16266 and #16267 was assumed.

To each of the participating laboratories were sent on November 9, 2016; 1 x 30 ml sample #16265, 1 x 30 ml sample #16266 and 1 x 30 ml sample #16267. A SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of the Ethanol/Gasoline mixtures, packed in brown glass bottles, was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were asked to determine on the samples: Water content (in %M/M) and Ethanol content (in %M/M and in %V/V), calculated by using the given densities, see paragraph 4.

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

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

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalysis). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<...' or '>...' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the results of the statistical evaluation should be used with due care.

According to ISO 5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM, EN or ISO reproducibilities, the zscores were calculated using a target standard deviation. This results in an evaluation independent of the variation of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use. The z-scores were calculated according to:

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

The $z_{(target)}$ scores are listed in the test result tables in appendix 1. Absolute values for z<2 are very common and absolute values for z>3 are very rare. The usual interpretation of z-scores is as follows:

	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 dispatch of the samples. Three participants did not report any test result at all. Finally, the 13 reporting laboratories did send in 93 test results. Nine outlying test results were observed, which is 9.7%. In proficiency studies, outlier percentages of 3% - 7.5% are normal.

It was decided to share the averaged Density of the three different ethanol/gasoline mixtures, as given in table 2 in paragraph 2 of this report, at the start of the proficiency test. The participants were instructed to use this averaged Density per sample for the conversion of %M/M to %V/V. This was requested to eliminate the possible variation caused by the Density determination by each laboratory in this conversion. The densities of the three different ethanol/gasoline mixtures for the conversion of %M/M to %V/V during this round robin are listed in the table below:

Sample id	Ratio Ethanol/Gasoline	Density in kg/L
#16265	80 / 20 % V/V	0.78217
#16266	60 / 40 % V/V	0.77012
#16267	20 / 80 % V/V	0.74625

Table 4: Densities given by iis of sub samples #16265, #16266 and #16267

4.1 EVALUATION PER SAMPLE AND PER TEST

In this section, the results are discussed per sample and per test. The test methods used are listed in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 3.

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

Sample #16265

- <u>Water:</u> This determination may be problematic dependent on the test method used. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers was in not agreement with the requirements of ASTM D1364:02(2012), but in agreement with the requirements of ASTM E203:16.
- Ethanol %M/M: This determination was very problematic. No statistical outliers were observed, but two test results were excluded. After contact with the participants it appeared that the determination of Ethanol with IP466 and ISO22854 is too different compared to ASTM D5501. The calculated reproducibility after rejection of the suspect data was not at all in agreement with the requirements of ASTM D5501:12(2016). The Kernel Density plot shows clearly a bimodal distribution. In this PT the water content was reported but this data could not explain this bimodal distribution. Therefore an extra email was sent to ask whether the

reported Ethanol content was determined on dry basis (no water) or as received (wet basis). The participants who responded indicated all to correct for the water content (wet basis).

Ethanol %V/V: The reported test results for this determination are converted from %M/M to %V/V by iis using the Density of Ethanol at 20°C given in ASTM D5501 and the Density of sample #16265 at 20°C (see table 4 above). Initially 5 out of 10 reporting participants (EtOH in %M/M) did not calculate the %V/V test results according to ASTM D5501 equation 8. The participants were contacted to explain how the conversion was done. It appeared that 3 participants had used the Relative Density of Ethanol at 15.56°C instead of the Density at 20°C, both listed in table 3 of ASTM D5501. Two of those three participants corrected these test results. After the correction of the two test results, the determination was still very problematic. No statistical outliers were observed, but three test results were excluded. After contact with the participants it appeared that the conversion to %V/V of Ethanol as per IP466 and ISO22854 is too deviating from ASTM D5501. Therefore these two test results were excluded. The other test result is excluded because the reported test values of the other two mixtures are quite deviating. In email correspondence the laboratory had indicated to analyse only samples with very high EtOH content (>95%).

The calculated reproducibility after rejection of the suspect data was still not at all in agreement with the requirements of ASTM D5501:12(2016).

Sample #16266

- <u>Water:</u> This determination was problematic for a number of participants. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers was in agreement with the requirements of ASTM D1364:02(2012) and with the requirements of ASTM E203:16.
- Ethanol %M/M: This determination was very problematic. No statistical outliers were observed, but three test results were excluded. One test result due to an outlier in the water determination, the other two test results because of the use of a different test method as explained above at sample #16265. The calculated reproducibility after rejection of the suspect data was not at all in agreement with the requirements of ASTM D5501:12(2016). The Kernel Density plot shows again a bimodal distribution.
- Ethanol %V/V: After the correction of two test results, this determination was still very problematic. One statistical outlier was observed and three test results excluded for the same reasons as mentioned at EtOH in %M/M of this sample. The calculated reproducibility after rejection of the suspect data was not at all in agreement with the requirements of ASTM D5501:12(2016).

Sample #16267

- Water:This determination was not problematic. Two statistical outliers were
observed. However, the calculated reproducibility after rejection of the
statistical outliers was in agreement with the requirements of ASTM
D1364:02(2012) and with the requirements of ASTM E203:16.
- Ethanol %M/M: This determination was problematic. No statistical outliers were observed, but two test results were excluded because of the use of a different test method as explained above at sample #16265. The calculated reproducibility after rejection of the suspect data was not in agreement with the requirements of ASTM D5501:12(2016). The Kernel Density plot shows again a bimodal distribution.
- Ethanol %V/V: After the correction of two test results, this determination was still problematic. One statistical outlier was observed and two test results were excluded for the same reasons as mentioned at EtOH in %M/M of this sample. The calculated reproducibility after rejection of the suspect data was not in agreement with the requirements of ASTM D5501:12(2016).

4.2 **PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES**

A comparison has been made between the reproducibility as declared by the relevant reference test method and the reproducibility as found for the group of participating laboratories. The average results per sample, calculated reproducibilities and reproducibilities derived from reference test methods (in casu ASTM test methods).

Parameter	Unit	n	average	2.8 * sd	R (lit)
Water	%M/M	10	0.16	0.04	0.03
Ethanol	%M/M	8	81.7	2.8	1.1
Ethanol	%V/V	8	80.9	3.0	1.1

Table 5: Reproducibilities of sample #16265

Parameter	Unit	n	average	2.8 * sd	R (lit)
Water	%M/M	8	0.14	0.01	0.03
Ethanol	%M/M	6	62.7	2.7	1.3
Ethanol	%V/V	6	61.2	2.7	1.3

Table 6: Reproducibilities of sample #16266

Parameter	Unit	n	average	2.8 * sd	R (lit)
Water	%M/M	9	0.072	0.015	0.016
Ethanol	%M/M	7	24.3	2.8	2.3
Ethanol	%V/V	7	23.0	2.6	2.4

Table 7: Reproducibilities of sample #16267

Without further statistical calculations, it can be concluded that there is not a good compliance of the group of participating laboratories with the target reproducibility.

4.3 EVALUATION OF THE PROFICIENCY TEST OF DECEMBER 2016 WITH PREVIOUS PTs

	December 2016	December 2015	December 2014	December 2013
Number of reporting labs	13	15	10	12
Number of test results reported	93	83	52	63
Number of statistical outliers	9	15	0	13
Percentage statistical outliers	9.7%	18%	0%	21%

Table 8: Evaluation with previous proficiency tests

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

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

	December 2016	December 2015	December 2014	December 2013
Water (20:80)	-	n.e.	n.e.	n.e.
Gasoline/Ethanol (20:80)		+/-		
Water (40:60)	++	n.e.	n.e.	n.e.
Gasoline/Ethanol (40:60)		++		
Water (80:20)	+/-	n.e.	n.e.	n.e.
Gasoline/Ethanol (80:20)	-	-		

Table 9: Comparison determinations against the reference test methods

The performance of the determinations against the requirements of the respective reference test methods is listed in the above table. The following performance categories were used:

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

APPENDIX 1

lab	method	value	mark	z(targ)	remarks
120	D1364	0.1297		-3.62	
132	E1064	0.180		1.59	
150	E203	0.1666		0.21	
171	E203	0.2304	DG(0.05)	6.82	
174					
175	E203	0.164		-0.06	
194					
311	D1364	0.163		-0.17	
323	D1364	0.162		-0.27	
334	D1364	0.2423	DG(0.05)	8.06	
447					
511	E1064	0.1843		2.04	
631					
633	E203	0.1536		-1.14	
663	E203	0.1790		1.49	
1201	D1364	0.164		-0.06	
	normality	not OK			
	n	10			
	outliers	2			
	mean (n)	0 1646			
	st dev (n)	0.01558			
	R(calc)	0.0436			
	R(D1364:02)	0.0270			Compare R(E203:16)=0.0780

Determination of Water on sample #16265; results in %M/M





Determination of Ethanol acc ASTM D5501 on sample #16265; results in %M/M and %V/V.

Lab	method	%M/M	mark	z(targ)	%V/V	mark	z(targ)	Remarks
120	D5501	81.06		-1.53	80.36	С	-1.36	first reported: 79.85
132	D5501	81.37		-0.76	80.67	С	-0.59	first reported: 80.20
150								
171	D5501	80.67		-2.51	79.5	E	-3.50	iis calc: 80.0
174								
175	D5501	83.02		3.37	82.30		3.47	
194								
311	D5501	82.73		2.65	82.01		2.75	
323								
334	ISO22854	82.47	ex	2.00	81.2	E, ex	0.73	iis calc: 81.8, ex see § 4.1
447	IP466	83.0	ex	3.32	78.2	E, ex	-6.73	iis calc: 82.3; ex see § 4.1
511	D5501	81.302		-0.93	80.598		-0.77	
631								
633	D5501	82.7363		2.66	82.0201		2.77	
663	D5501	80.487		-2.96	79.790		-2.78	
1201	D5501mod.				77.71	ex	-7.95	ex see § 4.1
	normality	OK			OK			
	n	8			8			
	outliers	0+2ex			0+3ex			
	mean (n)	81.6719			80.9060			
	st.dev. (n)	1.00570			1.07400			
	R(calc.)	2.8160			3.0072			
	R(D5501:12)	1.1191			1.1255			

iis calculation is based on the given density of Ethanol at 20°C in test method ASTM D5501 (0.789 g/ml) and the given density of sample at 20°C #16265 (0.78217 g/ml) by iis.



Determination of Water on sample #16266; results in %M/M

lah	method	value	mark	z(tara)	romarks	
100	D1004	0.1057		2(tary)	ICIIIaino	
120	D1364	0.1057	DG(0.01)	-3.47		
132	E1064	0.144		0.50		
150	E203	0.13465		-0.47		
171	E203	0.1957	G(0.05)	5.86		
174						
175						
194						
311	D1364	0.136		-0.33		
323	D1364	0.133		-0.64		
334	D1364	0.1444		0.54		
447						
511	F1064	0.147		0.81		
631	2.00.					
633	E203	0 1049	DG(0.01)	-3 55		
663	E200	0.1303	DO(0.01)	0.00		
1201	D1264	0.1355		0.01		
1201	D1304	0.155		-0.43		
	normality	OK				
	n	8				
	outliore	2				
		0 1 2 0 2				
		0.1392				
	si.uev. (n)	0.00532				
	R(calc.)	0.0149				
	R(D1364:02)	0.0270			Compare R(E203:16)=0.0780	





Determination of Ethanol acc ASTM D5501 on sample #16266; results in %M/M and %V/V.

lab	method	%M/M	mark	z(targ)	%V/V	Mark	z(targ)	Remarks
120	D5501	62.42		-0.68	60.93	С	-0.53	first reported: 60.54
132	D5501	62.96		0.47	61.45	С	0.57	first reported: 61.12
150								
171	D5501	62.18		-1.19	60.36	E	-1.73	iis calc: 60.69
174								
175								
194								
311	D5501	64.56		3.89	63.01		3.85	
323								
334	ISO22854	64.77	ex	4.34	62.8	E, ex	3.41	iis calc: 63.22; ex see § 4.1
447	IP466	65.4	ex	5.69	61.6	E, ex	0.88	iis calc: 63.8; ex see § 4.1
511	D5501	62.336		-0.86	60.844		-0.71	
631								
633	D5501	61.3990	ex	-2.86	59.9298	ex	-2.63	ex see § 4.1
663	D5501	61.972		-1.64	60.490		-1.45	
1201	D5501mod.				55.46	D(0.05)	-12.04	
	normality	not OK			not OK			
	normality	6			6			
	outliors	0			1,207			
	moon (n)	62 7280			61 1907			
	at day (n)	02.7300			01.1007			
	P(colc)	2 6651			2 7270			
	D(DEE01-12)	2.0001			2.1219			
	R(D5501:12)	1.3110			1.3309			

iis calculation is based on the given density of Ethanol at 20°C in test method ASTM D5501 (0.789 g/ml) and the given density of sample at 20°C #16266 (0.77012 g/ml) by iis.





Determination of Water on sample #16267; results in %M/M

lab	method	value	mark	z(targ)	remarks
120	D1364	0.0679		-0.75	
132	E1064	0.076		0.66	
150	E203	0.0711		-0.19	
171	E203	1.055	G(0.01)	170.69	
174					
175					
194					
311	D1364	0.067		-0.90	
323	D1364	0.070		-0.38	
334	D1364	0.0826		1.81	
447					
511	E1064	0.076		0.66	
631					
633	E203	0.0508	G(0.05)	-3.72	
663	E203	0.0722		0.00	
1201	D1364	0.067		-0.90	
	normality	OK			
	n	a			
	outliers	2			
	mean (n)	0 0722			
	st dev (n)	0.0722			
	R(calc.)	0.0145			
	R(D1364-02)	0.0161			Compare R(E203:16)=0.0780
	(21004.02)	0.0101			





Determination of Ethanol acc ASTM D5501:12 on sample #16267; results in %M/M and %V/V.

lab	method	%M/M	mark	z(targ)	%V/V	mark	z(targ)	Remarks
120	D5501	23.87		-0.58	22.58	С	-0.51	first reported: 22.44
132	D5501	25.05		0.85	23.69	С	0.79	first reported: 23.56
150								
171	D5501	24.15		-0.24	22.74	E	-0.32	iis calc: 22.84
174								
175								
194								
311	D5501	25.72		1.66	24.33		1.54	
323								
334	ISO22854	26.03	ex	2.03	24.4	E, ex	1.62	iis calc: 24.6; ex see § 4.1
447	IP466	26.0	ex	2.00	24.4	E, ex	1.62	iis calc: 24.6; ex see § 4.1
511	D5501	23.177		-1.42	21.921		-1.28	
631								
633	D5501	23.2637		-1.31	22.0032		-1.18	
663	D5501	25.212		1.04	23.846		0.97	
1201	D5501mod.				10.73	G(0.01)	-14.38	
	normality	OK			OK			
	normality	7			7			
	II outliers	1			1,201			
		24 2400			22 0157			
	st dov. (p)	24.3490			23.0137			
	B(aala)	0.99400			0.94024			
	R(Calc.)	2.1001			2.0407			
	R(D0001.12)	2.3133			2.3920			

iis calculation is based on given the density of Ethanol at 20°C in test method ASTM D5501 (0.789 g/ml) and the given density of sample at 20°C #16267 (0.74625 g/ml) by iis.



APPENDIX 2

Number of participants per country

- 1 lab in BELGIUM
- 1 lab in FRANCE
- 2 labs in NETHERLANDS
- 1 lab in PERU
- 2 labs in PHILIPPINES
- 1 lab in THAILAND
- 1 lab in UNITED KINGDOM
- 7 labs in UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations:

С	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
Е	= probably an error in calculations
U	= test result probably reported in a different unit
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= safety data sheet

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