Results of Proficiency Test
Ethanol (denaturated)
December 2017

Organised by: Institute for Interlaboratory Studies (iis)
Spijkenisse, the Netherlands

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1 INTRODUCTION

At the request of several participants, the Institute of Interlaboratory Studies decided to organise an interlaboratory study for Ethanol (Denaturated) in the 2017/2018 PT program.
In this interlaboratory study 11 laboratories in 9 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2017 interlaboratory study on Ethanol (Denaturated) are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET-UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. Sample analysis for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send two different samples of Ethanol (Denaturated) (each one in a 250 ml bottle, labelled resp. #17248 and #17249). 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 organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). 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

The necessary bulk material for sample #17248 was obtained from a local supplier. A batch of approximately 8.0 kg of this material was spiked with 239.4 grams Gasoline After homogenisation, 40 amber glass bottles of 250 mL were filled and labelled #17248. The homogeneity of subsamples #17248 was checked by determination of Density in accordance with ASTM D4052 and Gasoline in accordance with an inhouse test method on 8 stratified randomly selected samples.

	Density at 20°C in kg/L	Gasoline in %V/V
Sample #17248-1	0.78872	2.7
Sample #17248-2	0.78875	3.0
Sample #17248-3	0.78873	3.0
Sample #17248-4	0.78872	2.9
Sample #17248-5	0.78871	3.0
Sample #17248-6	0.78876	3.0
Sample #17248-7	0.78872	3.0
Sample #17248-8	0.78872	3.0

Table 1: homogeneity test results of subsamples #17248

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

	Density at 20°C in kg/L	Gasoline in %V/V
r (observed)	0.00005	0.3
reference method	ISO12185:16	Horwitz
0.3 x R (ref. method)	0.00015	0.3

Table 2: evaluation of the repeatabilities of subsamples #17248

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibility of the reference methods. Therefore, homogeneity of the subsamples #17248 was assumed.

The necessary bulk material for sample #17249 was obtained from a local supplier. To approximately 7.9 kg of this material, the components listed in table 3 were added:

Component	*Amount*
IPA (Isopropyl alcohol)	157.9 g
MEK (Methyl ethyl keton)	117.4 g
Methanol	236.7 g
TBA (tert-Butyl alcohol)	118.6 g

Table 3: components that were added to the bulk material for sample #17249

After homogenisation, 40 amber glass bottles of 250 mL were filled and labelled #17249. The homogeneity of subsamples #17249 was checked by determination of Density in accordance with ASTM D4052 and Isopropyl alcohol, Methyl ethyl keton, Methanol and ter-Butyl alcohol in accordance with In house test method on 8 stratified randomly selected samples.

Sample	Density at 20°C in kg/L	IPA in mg/kg	MEK in mg/kg	Methanol in mg/kg	TBA in mg/kg
Sample #17249-1	0.80508	9840	8333	13098	11056
Sample #17249-2	0.80507	9791	8323	13188	11008
Sample #17249-3	0.80507	9885	8413	13066	11115
Sample #17249-4	0.80507	10100	8622	12772	11344
Sample #17249-5	0.80507	9967	8485	13135	11204
Sample #17249-6	0.80508	10038	8535	13115	11276
Sample #17249-7	0.80507	9836	8348	12798	11056
Sample #17249-8	0.80507	9873	8406	13024	11108

Table 4: homogeneity test results of subsamples #17249

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

Sample	Density at 20°C in kg/L	IPA in mg/kg	MEK in mg/kg	Methanol in mg/kg	TBA in mg/kg
r (observed)	0.00001	303	298	436	330
reference method	ISO12185:16	Horwitz	Horwitz	Horwitz	Horwitz
0.3*R (ref. method)	0.00015	334	291	421	368

Table 5: evaluation of the repeatabilities of subsamples #17249

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibility of the reference methods. Therefore, homogeneity of the subsamples #17249 was assumed.

To each of the participating laboratories 1 x 250 mL bottle (labelled #17248) and 1 x 250 mL bottle (labelled #17249) were sent on November 8, 2017. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of the Ethanol 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 both samples: Density at 20°C, Water (titrimetric), Gasoline (in %V/V and in %M/M), Isopropyl alcohol (in %V/V and in %M/M), Methyl ethyl keton (in %V/V and in %M/M), Methanol (in %V/V and in %M/M) and tert-Butyl alcohol (in %V/V and in %M/M).
It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results more, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical calculations.

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

3 RESULTS

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

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

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

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

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

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.
Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

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

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

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM or ISO reproducibilities, 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. In some cases, a reproducibility based on former iis proficiency tests could be used.
When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

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

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

Absolute values for z<2 are very common and absolute values for z>3 are very rare.
The usual interpretation of z-scores is as follows:

	| 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 to one participant in Peru. Three participants (27% of the registered participants!) did not report any test results at all. Not all laboratories were able to report all analyses requested and for most of the determinations only a few test results were reported. Due to this it was not possible to draw significant conclusions for most of the determinations.

In total 8 laboratories reported 59 numerical test results. Observed were 2 outlying test results, which is 3.4% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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.

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, that are reported by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These methods are

also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 3.

Unfortunately, a suitable standard test method, providing the precision data, is not available for all determinations. For the test, that have no available precision data, the calculated reproducibility was compared against the reproducibility estimated from the Horwitz equation.

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

Sample #17248

Density: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ISO 12185:96.

Water, titrimetric: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTMD1364:02 (2012).

Gasoline (%V/V and %M/M): Only three laboratories reported in total 6 test results. Therefore, no significant conclusions were drawn. The average recovery of Gasoline (theoretical increment of 3.0 %M/M) may be acceptable (87%).

IPA, MEK, Methanol and TBA: Only a few test results were reported. Therefore, no significant conclusions were drawn. These components were not added to the Ethanol (purity >99%), used for the preparation of this sample.

Sample #17249

Density: This determination was not problematic. No statistical outlier was observed. The calculated reproducibility is in agreement with the requirements of ISO 12185:96.

Water, titrimetric: This determination may not be problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the estimated reproducibility using the Horwitz equation, but not in agreement with ASTM E203:16 and ASTM D1364:02 (application range 0.1-0.5 %M/M)

Gasoline (%V/V and %M/M): Only a few test results were reported. Therefore, no significant conclusions were drawn. Although two laboratories reported a Gasoline content of 3 %M/M (and 3 %V/V), Gasoline was not added to the Ethanol, used for this sample.

IPA (%V/V and %M/M): Only a few test results were reported. Therefore, no significant conclusions were drawn. The average recovery of IPA (theoretical increment of 2.0 %M/M) may be excellent (100%).

MEK (%V/V and %M/M): Only a few test results were reported. Therefore, no significant conclusions were drawn. The average recovery of MEK (theoretical increment of 1.5 %M/M) may be excellent (100%).

Methanol (%V/V and %M/M): Only a few test results were reported. Therefore, no significant conclusions were drawn. The average recovery of Methanol (theoretical increment of 3.0 %M/M) may be excellent (100%).

TBA (%V/V and %M/M): Only a few test results were reported. Therefore, no significant conclusions were drawn. The theoretical increment of TBA is 1.50 %M/M

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 and ISO test methods) are compared in the next tables:

Parameter *)	Unit	n	average	2.8 * sd	R (lit)
Density	kg/l	6	0.7888	0.0002	0.0005
Water, titrimetric	%M/M	6	0.240	0.032	0.078
Gasoline	%M/M	3	2.6	n.a.	n.a

Table 6: reproducibilities of tests on sample #17248
*) For the tests that are not listed, no significant conclusions were drawn

Parameter	Unit	n	average	2.8 * sd	R (lit)
Density	kg/L	7	0.8051	0.0002	0.0005
Water, titrimetric	%M/M	6	5.32	0.30	0.46
IPA	%M/M	2	2.0	n.a.	n.a
MEK	%M/M	2	1.5	n.a.	n.a
Methanol	%M/M	3	3.1	n.a.	n.a
TBA	%M/M	2	n.a.	n.a.	n.a

Table 7: reproducibilities of tests on sample #17249
*) For the tests that are not listed, no significant conclusions were drawn

Unfortunately, not all laboratories performed all tests, resulting in a low number of results for almost all tests. For these tests no comparison with the relevant reference test method can be made.

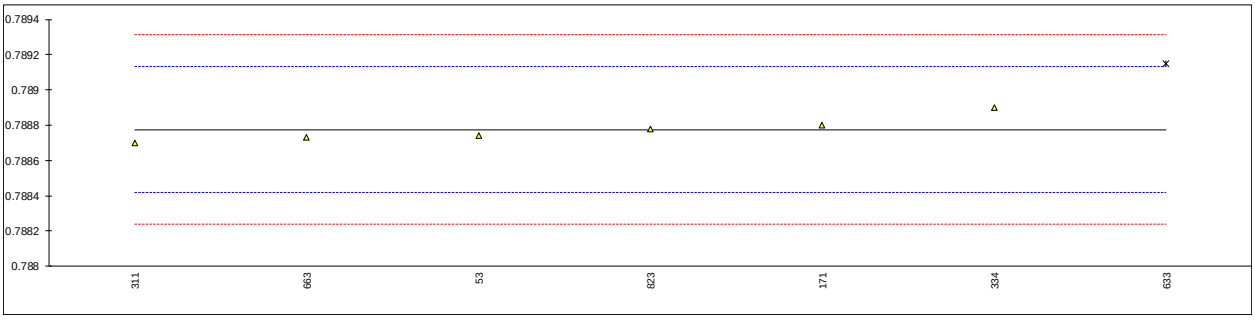
APPENDIX 1

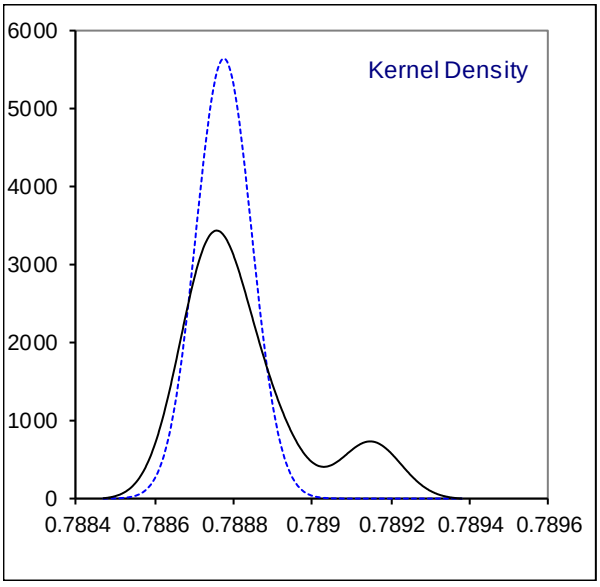
Determination of Density at 20°C on sample #17248; results in kg/L

lab	method	value	mark	z(targ)	remarks
52		-----		-----	
53	D4052	0.78874		-0.20	
62		-----		-----	
171	ISO12185	0.7888		0.14	
311	D4052	0.7887		-0.42	
334	ISO12185	0.7889		0.70	
511		-----		-----	
633	D4052	0.78915	G(0.05)	2.10	
663	D4052	0.78873		-0.25	
823	D4052	0.78878		0.03	
913		-----		-----	

	normality	unknown
	n	6
	outliers	1
	mean (n)	0.78877
	st.dev. (n)	0.000071
	R(calc.)	0.00020
	st.dev.(ISO12185:96)	0.000179
	R(ISO12185:96)	0.0005

compare R(D4052:16) = 0.00050

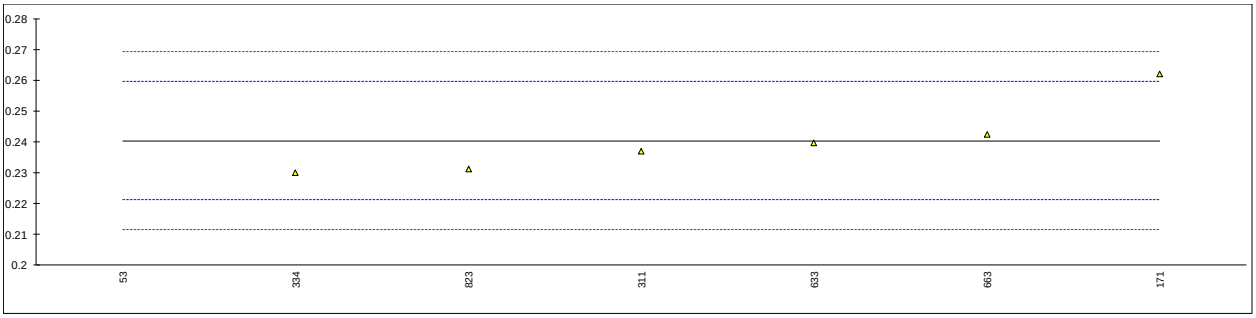


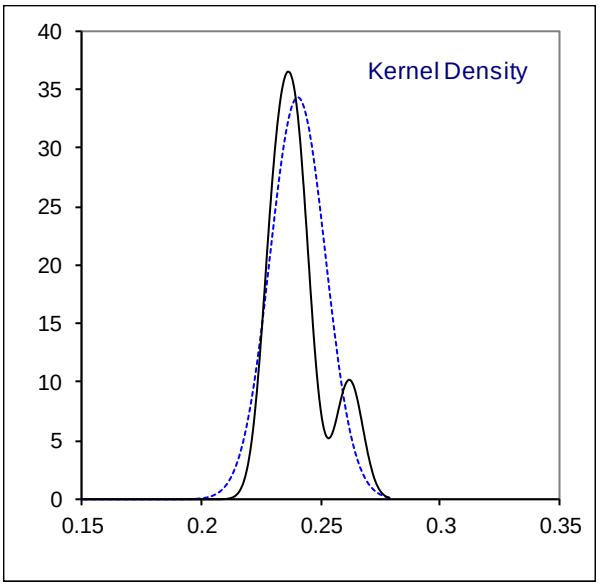


Determination of Water, titrimetric on sample #17248; results in %M/M

lab	method	value	mark	z(targ)	remarks
52		-----		-----	
53	E203	0.02334	D(0.01)	-22.51	
62		-----		-----	
171	D1364	0.262		2.24	
311	D1364	0.237		-0.35	
334	E203	0.230		-1.08	
511		-----		-----	
633	D6304	0.2397		-0.07	
663	E203	0.2423		0.20	
823	D1364	0.2313		-0.94	
913		-----		-----	

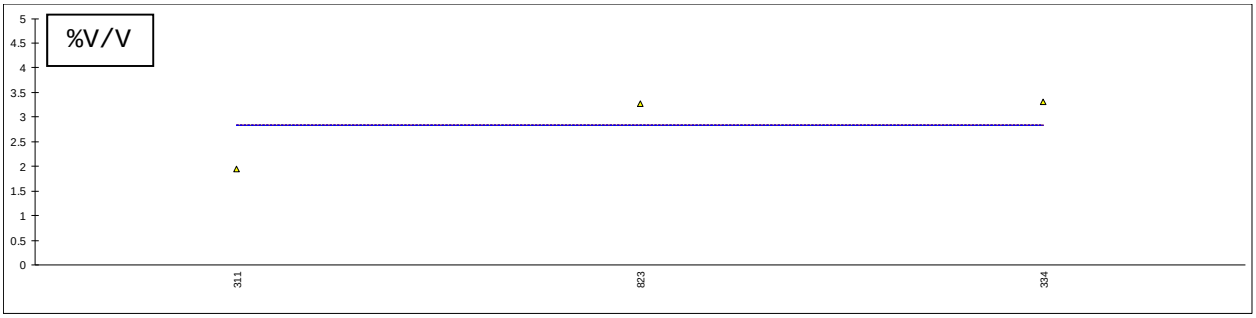
normality	unknown	
n	6	
outliers	1	
mean (n)	0.2404	
st.dev. (n)	0.01160	
R(calc.)	0.0325	compare R(E203:16) = 0.027, pyridine based reagents
st.dev.(D1364:02)	0.02786	compare R(E203:16) = 0.078, pyridine-free reagents
R(D1364:02)	0.0780	application range ASTM D1364 = 0.1 – 0.5 % M/M

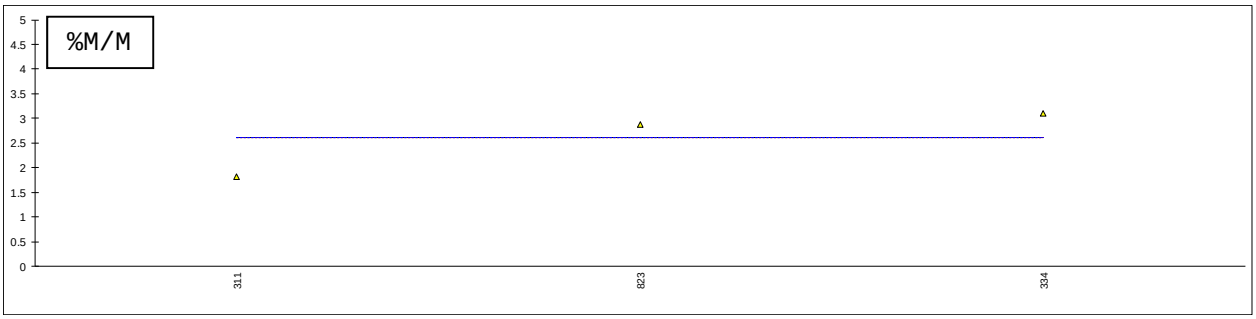




Determination of Gasoline on sample #17248; results in %V/V and %M/M.

lab	method	%V/V	mark	z(targ)	%M/M	mark	z(targ)	Remarks
52		-----		-----	-----		-----	
53		-----		-----	-----		-----	
62		-----		-----	-----		-----	
171		-----		-----	-----		-----	
311	INH-582	1.94		-----	1.82		-----	
334	In house	3.3		-----	3.1		-----	
511		-----		-----	-----		-----	
633		-----		-----	-----		-----	
663		-----		-----	-----		-----	
823	D5501	3.2771		-----	2.8841		-----	
913		-----		-----	-----		-----	
	normality	unknown			unknown			
	n	3			3			
	outliers	n.a.			n.a.	spike		
	mean (n)	2.8			2.6	3.0 %M/M		recovery = 87%
	st.dev. (n)	n.a.			n.a.			
	R(calc.)	n.a.			n.a.			
	st.dev. (lit)	n.a.			n.a.			
	R(lit)	n.a.			n.a.			





Determination of IPA (Isopropyl alcohol) on sample #17248; results in %V/V and %M/M.

lab	method	%V/V	mark	z(targ)	%M/M	mark	z(targ)	Remarks
52		-----		-----	-----		-----	
53		-----		-----	-----		-----	
62		-----		-----	-----		-----	
171		-----		-----	-----		-----	
311	INH-282	<1		-----	<1		-----	
334		-----		-----	-----		-----	
511		-----		-----	-----		-----	
633		-----		-----	-----		-----	
663	INH-002	-----		-----	0.001		-----	
823	INH-0002	0.0008		-----	0.0008		-----	
913		-----		-----	-----		-----	
	n	2			3			
	mean (n)	<1			<1			

Determination of MEK (Methyl ethyl keton) on sample #17248; results in %V/V and %M/M.

lab	method	%V/V	mark	z(targ)	%M/M	mark	z(targ)	Remarks
52		-----		-----	-----		-----	
53		-----		-----	-----		-----	
62		-----		-----	-----		-----	
171		-----		-----	-----		-----	
311	INH-282	<1		-----	<1		-----	
334		-----		-----	-----		-----	
511		-----		-----	-----		-----	
633		-----		-----	-----		-----	
663	INH-002	-----		-----	<0.0005		-----	
823	INH-0002	0		-----	0		-----	
913		-----		-----	-----		-----	
	n	2			3			
	mean (n)	<1			<1			

Determination of Methanol on sample #17248; results in %V/V and %M/M.

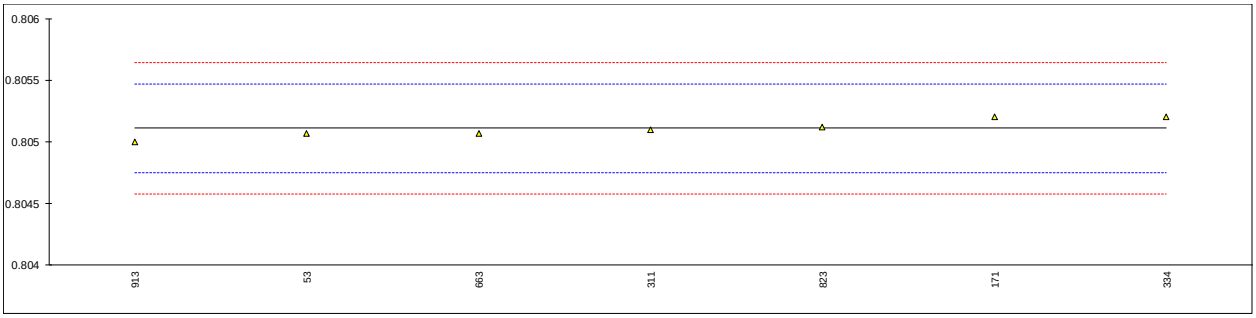
lab	method	%V/V	mark	z(targ)	%M/M	mark	z(targ)	Remarks
52		-----		-----	-----		-----	
53		-----		-----	-----		-----	
62		-----		-----	-----		-----	
171		-----		-----	-----		-----	
311	INH-582	<0.1		-----	<0.1		-----	
334		-----		-----	-----		-----	
511		-----		-----	-----		-----	
633		-----		-----	-----		-----	
663	INH-002	-----		-----	0.004		-----	
823	D5501	0.0060		-----	0.0060		-----	
913		-----		-----	-----		-----	
	n	2			3			
	mean (n)	<0.1			<0.1			

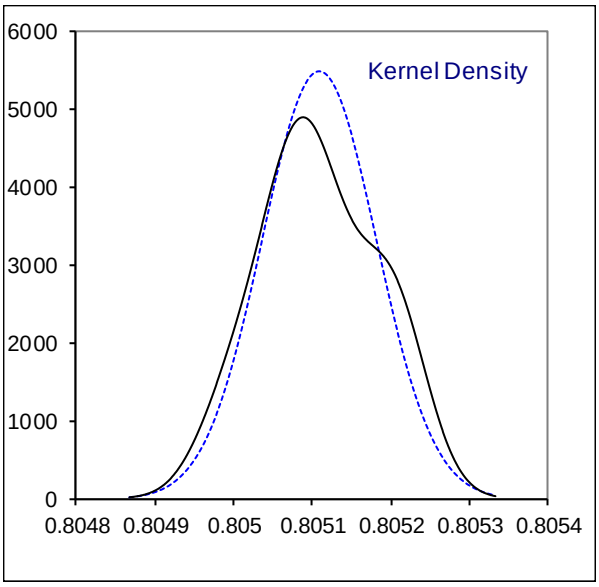
Determination of TBA (tert-Butyl alcohol) on sample #17248; results in %V/V and %M/M.

lab	method	%V/V	mark	z(targ)	%M/M	mark	z(targ)	Remarks
52		-----		-----	-----		-----	
53		-----		-----	-----		-----	
62		-----		-----	-----		-----	
171		-----		-----	-----		-----	
311	INH-529	<0.005		-----	<0.005		-----	
334		-----		-----	-----		-----	
511		-----		-----	-----		-----	
633		-----		-----	-----		-----	
663	INH-002	-----		-----	<0.0005		-----	
823	INH-0002	0		-----	0		-----	
913		-----		-----	-----		-----	
	n	2			3			
	mean (n)	<0.01			<0.01			

Determination of Density at 20°C on sample #17249; results in kg/L

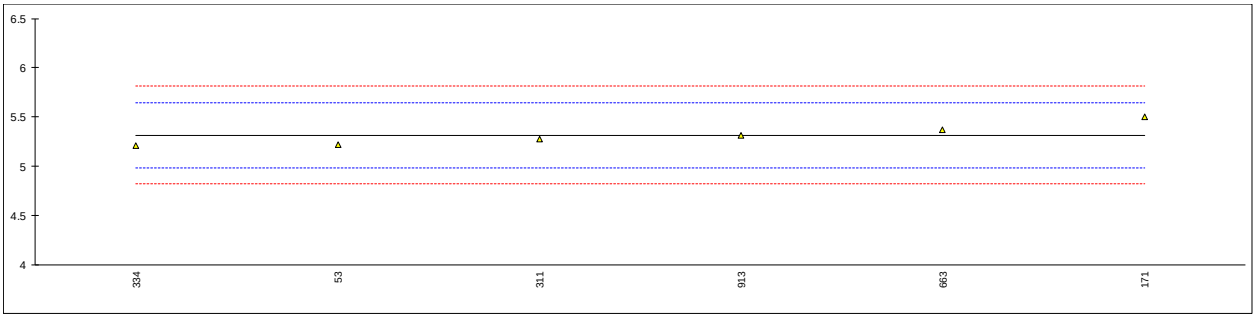
lab	method	value	mark	z(targ)	remarks
52		-----		-----	
53	D4052	0.80507		-0.22	
62		-----		-----	
171	ISO12185	0.8052		0.51	
311	D4052	0.8051		-0.05	
334	ISO12185	0.8052		0.51	
511		-----		-----	
633		-----		-----	
663	D4052	0.80507		-0.22	
823	D4052	0.80512		0.06	
913	D4052	0.8050		-0.61	
	normality	unknown			
	n	7			
	outliers	0			
	mean (n)	0.80511			
	st.dev. (n)	0.000073			
	R(calc.)	0.00020			
	st.dev.(ISO12185:96)	0.000179			
	R(ISO12185:96)	0.0005			compare R(D4052:16) = 0.00050





Determination of Water, titrimetric on sample #17249; results in %M/M

lab	method	value	mark	z(targ)	remarks
52		-----		-----	
53	E203	5.2221		-0.57	
62		-----		-----	
171	D1364	5.50		1.11	
311	D1364	5.276		-0.24	
334	E203	5.213		-0.62	
511		-----		-----	
633		-----		-----	
663	E203	5.3738		0.35	
823		-----		-----	
913	E203	5.31		-0.04	
	normality	unknown			
	n	6			
	outliers	0			
	mean (n)	5.316			
	st.dev. (n)	0.1079			
	R(calc.)	0.302			compare R(D1364:02) = 0.027, range 0.1-0.5 %M/M
	st.dev.(Horwitz)	0.1654			compare R(E203:16) = 0.027, pyridine based reagents
	R(Horwitz)	0.463			compare R(E203:16) = 0.078, pyridine-free reagents



Determination of Gasoline on sample #17249; results in %V/V and %M/M.

lab	method	%V/V	mark	z(targ)	%M/M	mark	z(targ)	Remarks
52		-----		-----	-----		-----	
53		-----		-----	-----		-----	
62		-----		-----	-----		-----	
171		-----		-----	-----		-----	
311	INH-582	<0.9		-----	<0.9		-----	
334		3.5		-----	3.2		-----	
511		-----		-----	-----		-----	
633		-----		-----	-----		-----	
663		-----		-----	-----		-----	
823	D5501	3.3781		-----	2.9441		-----	
913		-----		-----	-----		-----	
	n	2			2			
	mean (n)	3.4			3.1			

Determination of IPA (Isopropyl alcohol) on sample #17249; results in %V/V and %M/M.

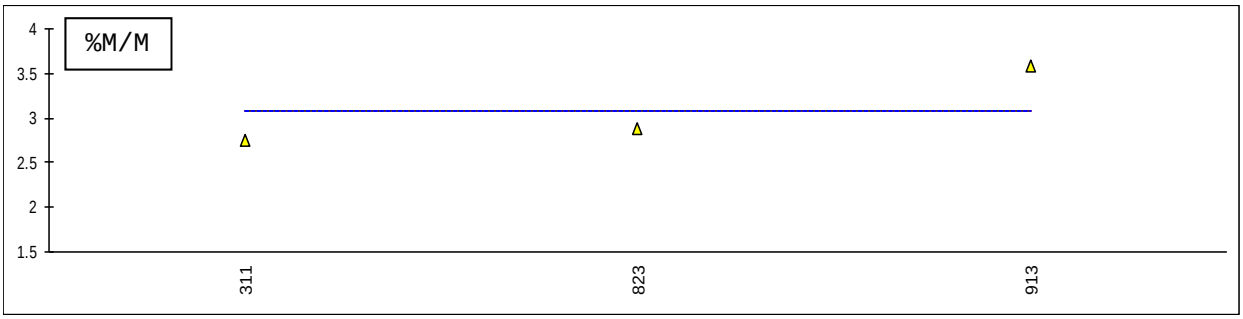
lab	method	%V/V	mark	z(targ)	%M/M	mark	z(targ)	Remarks
52		-----		-----	-----		-----	
53		-----		-----	-----		-----	
62		-----		-----	-----		-----	
171		-----		-----	-----		-----	
311	INH-282	2.0		-----	1.9		-----	
334		-----		-----	-----		-----	
511		-----		-----	-----		-----	
633		-----		-----	-----		-----	
663		-----		-----	-----		-----	
823		-----		-----	-----		-----	
913	INH-0001	-----		-----	2.13		-----	
	n	1			2	spike		
	mean (n)	n.a.			2.0	2.0 %M/M		recovery = 100%

Determination of MEK (Methyl ethyl keton) on sample #17249; results in %V/V and %M/M.

lab	method	%V/V	mark	z(targ)	%M/M	mark	z(targ)	Remarks
52		-----		-----	-----		-----	
53		-----		-----	-----		-----	
62		-----		-----	-----		-----	
171		-----		-----	-----		-----	
311	INH-282	1.5		-----	1.4		-----	
334		-----		-----	-----		-----	
511		-----		-----	-----		-----	
633		-----		-----	-----		-----	
663		-----		-----	-----		-----	
823		-----		-----	-----		-----	
913	INH-0001	-----		-----	1.57		-----	
	n	1			2	spike		
	mean (n)	n.a.			1.5	1.5 %M/M		recovery = 100%

Determination of Methanol on sample #17249; results in %V/V and %M/M.

lab	Method	%V/V	mark	z(targ)	%M/M	mark	z(targ)	Remarks
52		-----		-----	-----		-----	
53		-----		-----	-----		-----	
62		-----		-----	-----		-----	
171		-----		-----	-----		-----	
311	INH-582	2.81		-----	2.76		-----	
334		-----		-----	-----		-----	
511		-----		-----	-----		-----	
633		-----		-----	-----		-----	
663		-----		-----	-----		-----	
823	D5501	2.8875		-----	2.8766		-----	
913	INH-0001	-----		-----	3.60		-----	
	normality	unknown			unknown			
	n	2			3			
	outliers	n.a.			n.a.	spike		
	mean (n)	2.85			3.08	3.0 %M/M		recovery = 100%
	st.dev. (n)	n.a.			n.a.			
	R(calc.)	n.a.			n.a.			
	st.dev.(lit)	n.a.			n.a.			
	R(lit)	n.a.			n.a.			



Determination of TBA (tert-Butyl alcohol) on sample #17249; results in %V/V and %M/M.

lab	Method	%V/V	mark	z(targ)	%M/M	mark	z(targ)	Remarks
52		-----		-----	-----		-----	
53		-----		-----	-----		-----	
62		-----		-----	-----		-----	
171		-----		-----	-----		-----	
311	INH-529	>0.2		-----	>0.2		-----	
334		-----		-----	-----		-----	
511		-----		-----	-----		-----	
633		-----		-----	-----		-----	
663		-----		-----	-----		-----	
823		-----		-----	-----		-----	
913	INH-0001	-----		-----	1.70		-----	
	n	1			2	spike		
	mean (n)	n.a.			n.a.	1.5 %M/M		

APPENDIX 2

Number of participants per country

3 labs in CANADA
1 lab in FRANCE
1 lab in INDIA
1 lab in NETHERLANDS
1 lab in PERU
1 lab in PHILIPPINES
1 lab in SOUTH KOREA
1 lab in THAILAND
1 lab in UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations:

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= 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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14	Analytical Methods Committee Technical brief, No 4.January 2001
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