Results of Proficiency Test Turbine Oil (used) May 2015

Organised by: Institute for Interlaboratory Studies Spijkenisse, the Netherlands

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CONTENTS

1	INTRODUCTION	3
2	SET UP	3
2.1	ACCREDITATION	3
2.2	PROTOCOL	3
2.3	CONFIDENTIALITY STATEMENT	3
2.4	SAMPLES	4
2.5	STABILITY OF THE SAMPLES	4
2.6	ANALYSES	5
3	RESULTS	5
3.1	STATISTICS	5
3.2	GRAPHICS	6
3.3	Z-SCORES	6
4	EVALUATION	7
4.1	EVALUATION PER TEST	7
4.2	PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES	10
4.3	COMPARISON OF THE PROFICIENCY TEST OF MAY 2015 WITH PREVIOUS PTS	11

Appendices:

1.	Data and statistical results	12
2.	Number of participants per country	26
3.	Abbreviations and literature	27

1 INTRODUCTION

Since 2013, the Institute for Interlaboratory Studies organises every year a proficiency test for used Turbine Oil. In the annual proficiency testing program 2014/2015, it was decided to continue the proficiency test for the analyses of used Turbine Oil. In this interlaboratory study 40 laboratories in 28 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the 2015 used Turbine Oil proficiency test are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organizer of this proficiency test. It was decided to send one bottle of 1L (labelled #15056) of used Turbine Oil that was provided by a third party. The analyses for fit-for-use and homogeneity were subcontracted. Participants were requested to report rounded and unrounded results. The unrounded 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/IEC 17043:2010, since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially 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 Organization, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol is electronically available through the iis internet site 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 was provided by a third party. The 30 litre bulk material was transferred after homogenizing into 58 brown glass bottles of 0.5 litre (labelled #15056). The homogeneity of the subsamples #15056 was checked by determination of Density at 15°C in accordance with ASTM D4052 and Water in accordance with ASTM D6304 on 8 stratified randomly selected samples.

	Density at 15 °C in kg/m³	Water in mg/kg
Sample #15056-1	874.03	110
Sample #15056-2	873.98	110
Sample #15056-3	874.03	110
Sample #15056-4	873.99	110
Sample #15056-5	874.05	110
Sample #15056-6	874.04	110
Sample #15056-7	874.05	100
Sample #15056-8	874.05	100

Table 1: homogeneity test results of subsamples #15056

From the above test results, the repeatability was calculated and compared with 0.3 times the corresponding reproducibilities in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density at 15 °C in kg/m ³	Water in mg/kg
r (sample #15056)	0.04	13
reference test	ASTM 1298:12b	ASTM D6304:07
0.3 x R(reference test)	0.45	84

Table 2: evaluation of the repeatability of the subsamples #15056

The calculated repeatability is less than 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the subsamples #15056 was assumed.

To each of the participating laboratories, one sample of 0.5 L brown glass bottle (labelled #15056) was sent on April 15, 2015.

2.5 STABILITY OF THE SAMPLES

The stability of the Turbine Oil (used), packed in the brown glass bottles, was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were requested to determine on sample #15056: Acid Number, Base Number, Color, Density at 15°C, Flash Point PMcc, Kinematic Viscosity at 40°C and at 100°C, Viscosity Index, Level of contamination, Water by KF and Water separability.

To get comparable results a detailed report form, on which the units were prescribed as well as the required standards and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The detailed report form was also made available for download on the iis website www.iisnl.com. A SDS and a form to confirm receipt of the samples were added to the sample package.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original data are tabulated per determination in Appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder fax was sent to those laboratories that had not reported results at that moment.

Shortly after the deadline, the available results were screened for suspect data. A 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 results. Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (iis-protocol, version 3.3) of April 2014. For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. 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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

According to ISO 5725 the original results per determination were submitted to Dixon's and/or 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 (ref. 15). Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(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 analysis results are plotted. The corresponding laboratory numbers are under 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 standard. Outliers and other data, which were excluded from the calculations, are represented as a "x". Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. The Kernel Density is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nos.13 and 14). 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 reproducibilities, the z-scores were calculated using a target standard deviation. This 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:

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

The $z_{(target)}$ scores are listed in the 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 interlaboratory study, some problems were encountered with the dispatch of the samples to laboratories in Iran and Sudan. Seven participants reported after the final reporting date and one participant did not report any test results at all. Not all laboratories were able to report all analyses requested. In total 39 participants reported 398 test results. Observed were 36 outlying results, which is 9.0% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

In this section, the results are discussed per test. The methods, which are used by the various laboratories, are 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.

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.

- <u>Acid Number:</u> This determination was problematic for a number of laboratories. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D664:11a.
- <u>Base Number:</u> This determination may be very problematic at the level of 0.3 mg KOH/g. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the estimated reproducibility requirements of ASTM D2896:11. It should be noted that the precision data of ASTM D2896:11 used as target requirements, are based on acid numbers ranging from 5 – 27 mg KOH/g.
- Color:This determination was not problematic. Two statistical outliers were
observed. The calculated reproducibility after rejection of the statistical
outliers is in agreement with ASTM D1500:12.
iis also calculated a numerical color value for all results that were reported
as 'less than' for example L4.5 or L5.0. This test method uses color
standards with values that are 0.5 points apart, ranging from 0.5 8.0.
Since the color is determined by comparing the color of the sample to these
standards, it is assumed when for example L4.5 is reported, the actual

colour lies between 4.0 and 4.5. iis calculated this value as 4.25 (4.50 minus 0.25).

After conversion of the reported results, again a statistical evaluation was done on this larger group of data (n=32). This resulted in a similar consensus value and a slightly smaller spread as compared to the original group with 21 numerical data.

- <u>Density at 15°C:</u> This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D1298:12b.
- <u>Flash Point PMcc</u>: This determination was problematic for a number of laboratories. Three statistical outliers were observed and one laboratory was excluded from the statistical evaluation for reporting a test result from an <u>Open</u> Cup Flash Point method. The calculated reproducibility after rejection of the suspect data is in full agreement with the requirements of ASTM D93:15, procedure B (and A).
- <u>Kin.Visco.at 40°C</u>: This determination was not problematic. Four statistical outliers were observed. In ASTM D445:15 there is no reproducibility for used oils. Therefore iis did publish an article with the estimated reproducibility based on the reproducibilities as observed in a large number of iis PTs on used oils (ref. 16). The calculated reproducibility, after rejection of the statistical outliers, is in

good agreement with this literature reproducibility requirement.

<u>Kin.Visco.at 100°C:</u> This determination was problematic for a number of laboratories. Five statistical outliers were observed. In ASTM D445:15 there is no reproducibility for used oils. Therefore iis did publish an article with the estimated reproducibility based on the reproducibilities as observed in a large number of iis PTs on used oils (ref. 16). The calculated reproducibility, after rejection of the statistical outliers, is in good agreement with this literature reproducibility requirement.

Viscosity IndexThis determination was very problematic. Four statistical outliers were
observed. The calculated reproducibility after rejection of the statistical
outliers is not at all in agreement with ASTM D2270:10e1.
From the test results reported for kinematic viscosity at 40°C and at 100°C,
iis calculated the Viscosity Indices and compared the results with the
reported Viscosity Indices. It appeared that eight participants may not have
calculated the viscosity index correctly; one participant corrected the value
for the kinematic viscosity at 40°C without revising also the viscosity index.
Five participants used Houillon viscosity and five participants used
Stabinger viscosity to calculate the viscosity index.

A separate statistical evaluation was done on the group of (iis) calculated viscosity indices without the values of the laboratories that had outlying test

results in the Viscosity D445 tests or did not correct the viscosity index, while a corrected result for Viscosity was submitted. The calculated reproducibility after rejection of the suspect data is surprisingly larger than before and again not in agreement with ASTM D2270:10e1.

<u>Water</u>: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D6304:07.

Water separability: This determination for this oil may be problematic for a number of laboratories. Various test results were reported, ranging from 5 to 51 min to reach 3 ml or less emulsion, 19.5 to 51 min to reach 37 ml of water and 15 to 60 min to reach complete break. In ASTM D1401 is stated that the test may be aborted after 30 minutes. It appears that eight participants aborted the test and 9 participants did not abort the test. In total five statistical outliers were observed. The calculated reproducibility for "time to reach 37 ml of water" after rejection of the statistical outliers are both in agreement with the requirements of ASTM D1401:12.

 Level of Cont:
 This determination was problematic. In total 5 statistical outliers were observed. One test result from lab 1599 was excluded from the statistical evaluation as the two other reported test results from lab 1599 were designated as statistical outliers and the three test results are not independent from each other.

 The calculated reproducibilities for number of particles ≥4µm, ≥6µm and

 \geq 14µm, after rejection of the suspect data, are not in agreement with the requirements of ASTM D7647:10.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories that participated. The average results, calculated reproducibilities and reproducibilities derived from literature standards (in casu ASTM and IP standards), are compared in the next table.

Parameter	unit	n	Average	2.8 * sd	R(lit)
Acid Number, Total	mg KOH/g	26	0.43	0.12	0.19
Base Number, Total	mg KOH/g	13	0.33	0.52	(0.02)
Color		21	4.1	0.8	1.0
Density at 15 °C	kg/m ³	32	874.0	0.8	1.5
Flash Point PMcc	°C	25	220.5	9.4	10.0
Kinematic Viscosity at 40 °C	mm²/s	34	40.17	0.38	0.72
Kinematic Viscosity at 100 °C	mm²/s	32	6.506	0.111	0.143
Viscosity Index		29	113	3	2
Water by KF	mg/kg	27	89.0	84.1	249.7
Water Separability					
- Time to reach ≤3 ml emulsion	min	8	27.4	16.3	20.0
- Time to reach 37 of water	min	10	26.5	16.7	20.0
Level of Contamination ≥4µm Parts/r		18	25200	41700	28500
Level of Contamination ≥6µm Parts/m		18	2320	4950	1760
Level of Contamination ≥14µm	Parts/ml	18	45	137	61

Table 3: reproducibilities of results of sample #15056

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

	May 2015	May 2014	May 2013
Number of reporting labs	39	29	27
Number of results reported	398	301	336
Statistical outliers	36	10	14
Percentage outliers	9.0%	3.3%	4.2%

4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2015 WITH PREVIOUS PTS

Table 4: comparison with previous proficiency tests

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

The performance of the determinations of the proficiency tests was compared to the requirements of the respective standards. The conclusions are given in the following table:

Determination	May 2015	May 2014	May 2013
Acid Number, Total	+		-
Base Number, Total	()		
Color	+	++	-
Density at 15 °C	++	+/-	
Flash Point PMcc	+/-	-	-
Kinematic Viscosity at 40 °C	++	++	+
Kinematic Viscosity at 100 °C	+	+/-	+
Viscosity Index			
Water by KF	++	++	++
Water Separability	+	n.e.	+
Level of Contamination ≥4µm		n.e.	n.e.
Level of Contamination ≥6µm		n.e.	n.e.
Level of Contamination ≥14µm		n.e.	n.e.

Table 5: comparison determinations against the standard

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

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

APPENDIX 1

0.1

0

1161 963 179 331

Turbine Oil: iis15L03

Determination of Acid Number on sample #15056; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
178					
179	D664	0.37		-0.88	
228					
237	D664	0.41		-0.29	
325	D664	0.455		0.37	
331	D664	0.38		-0.74	
340	D664	0.414		-0.23	
349	D664	0.41		-0.29	
360	D664	0.50		1.04	
398	D664	0.551		1.80	
432					
445	5001				
496	D664	0.42		-0.14	
511	D074				
541	D974	0.64	R(0.01)	3.11	
614				0.00	
662	D664	0.41		-0.29	
003	D664	0.424		-0.09	
002	D004	0.41		-0.29	
902	D664	0.47	P(0.01)	5.04	
063	D664	0.77	1((0.01)	_1 18	
903	D664	0.33		0.88	
1023	D664	0.400		-0.14	
1026	D664	0.43		0.00	
1059	ISO6619	0.49		0.89	
1134	D664	0.45		0.30	
1146	D664	0.434		0.06	
1161	D664	0.079	R(0.01)	-5.19	
1417	IP177	0.43	()	0.00	
1431	D664	0.3929		-0.55	
1435	D664	0.39		-0.59	
1461					
1599					
1720	D664	0.40		-0.44	
1854	D664	0.43		0.00	
1966	ISO6618	0.444		0.21	
1981					
7002	D664	0.6865	R(0.01)	3.80	
7015					
	normality	0.105+			
	normality	suspect			
	11 outlions	20			
	outilers	4			
	st dev (n)	0.430			
	R(calc.)	0.123			
	R(D664·11a)	0.120			
		01100			
0.9					10
0.8 -					x 9 - Kemer Density
0.7					x 8 1
0.6 -					
0.5					
				<u> </u>	
Δ					4 -
0.3					3 -
0.2					

2 1

0

0

0.2

0.4

0.6

0.8

7002 922

Determination of Base Number on sample #15056; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
178					
179	D2896 - B	0.08			
228					
237					
325	D2896 - B	0.50			
331	D2896 - B	0.03			
340	D2896 - A	0.48			
349					
360					
398	D2896 - A	0.43			
432	D2896 - B	0.37			
445					
496	D2896 - A	0.19			
511					
541	D2896 - B	<0.5	С		first reported:10.1
614					
657					
663					
862	D2896 - B	0.50			
902	D2896 - B	0.35			
922	D2896 - B	2.2	G(0.01)		false positive result?
963					
994					
1023					
1026	D2896 - B	0.45			
1059					
1134					
1146	D2896 - A	0.372			
1161					
1417	in house	0.0			
1431					
1435	D2896	0.48			
1461					
1599					
1/20	D				
1854	D2896 - A	missing E.P.			
1966					
1981					
7002					
7015					
	normality	OK			
	normality	12			
	outliers	10			
	moon (n)	0 3255			
	st dev (n)	0.3200			
	P(calc)	0.10020			
	R(D2896.11 (Δ))	(0.023)			Application range Δ STM D2896 = 5 - 27 mg KOH/g
	$(D_2000.11(\Lambda))$	(0.020)			





Determination of Color on sample #15056

lab	method	value	mark	z(targ)	value calc. *)	remarks
178						
179	D1500	L4.5			4.25	
228	D1500	4		-0.24	4	
237	D1500	L4.5			4.25	
325	D6045	L5.0			4.75	
331	D1500	3.5		-1 64	3.5	
340	D1500	4.0		-0.24	4	
349	D6045	5.4	R(0.01)	3.68	5.4	
360	D1500	4 0		-0.24	4	
398	D1500	4 0		-0.24	4	
432	D1500	145			4 25	
445	2.000					
496	D1500	4.0		-0.24	4	
511	D6045	4.5		1 16	4.5	
541	D1500	L4.5			4.25	
614	D1500	14.5			4 25	
657	D1500	4 0		-0 24	4	
663	D1500	4.0		-0.24	4	
862	D1500	14.5			4 25	
902	D1500	43		0.60	4.3	
922	D1500	145			4.25	
963	D1500	4 2		0.32	4.20	
994	D1500	145		0.52	4 25	
1023	D1500	4.0		-0 24	4.20	
1020	D1500	145		-0.24	4 25	
1020	D1500	4.0		-0 24	4.20	
1134	D1500	35		-1.64	35	
1146	BTOOD					
1161	D6045	43		0.60	43	
1417	D6045	4.5		1 16	4.5	
1431	D1500	4.0		-0.24	4.0	
1435	D1500	4.50		1 16	4 5	
1461	1502049	4.0		-0.24	4.5	
1500	1002040			0.24		
1720	D1500	40		-0 24	4	
1854	D1500	14	R(0.01)	-7.52	14	
1966	D1500	4.5	1(0.01)	1 16	4.5	
1981	D1500	145			4 25	
7002	D1000	L4.5			4.20	
7002						
1015						
	normality	ОК			suspect	
	n	21			32	
	outliers	2			2	
	mean (n)	4 09			4 16 (145)	
	st dev (n)	0.280			0.261	
	R(calc.)	0.78			0.73	
	R(D1500:12)	1.00			1.00	

*) In the calculation of the mean, standard deviation and the reproducibility of this column, a reported value of 'L y' is changed into y-0.25 (for example L4.5 into 4.25)





Determination of Density at 15°C on sample #15056; results in kg/m³

lab	method	value	mark	z(targ)	remarks
178					
179	D1298	874 0	С	-0.05	first reported 878 4
228	D1298	874 1	•	0.14	
237	D4052	874.3		0.51	
325	D4052	874.1		0.01	
321	04002	074.1		0.14	
340	D4052	874 37		0.64	
240	D4052	074.37		1.04	
349	D4052	0/4./		1.20	
300	D4052	073.9		-0.24	
398	D4052	873.0		-0.80	
432	15012185	874.15		0.23	
445	D / 0 - 0				
496	D4052	873.8		-0.42	
511	D1298	873.95		-0.14	
541	D4052	874.0		-0.05	
614					
657	D4052	874.1		0.14	
663	D4052	873.96		-0.13	
862	D4052	874.0		-0.05	
902	D4052	874.07		0.08	
922	D4052	873.9		-0.24	
963	D4052	874.0		-0.05	
994	D4052	873.9		-0.24	
1023	D4052	874	С	-0.05	first reported:0.8740
1026	D4052	874.6	-	1.07	
1059	ISO12185	874.0		-0.05	
1134	D4052	873.9		-0.24	
1146	D4052	874.05		0.24	
1161	1903675	872 5	P(0.01)	-2.85	
1/17	D4052	974.3	11(0.01)	0.51	
1/121	D4052	973 71		0.51	
1405	D4032	073.71		-0.59	probably unit error reported: 0.972
1433	D1290	013	C,R(0.05)	-1.92	probably unit error, reported. 0.875
1401	1503075	074.0		0.00	
1599	D4052	874		-0.05	
1720	D4052	873.9		-0.24	
1854	D4052	873.3		-1.36	
1966	D1298	873.7		-0.61	
1981	D4052	874.01	С	-0.03	probably unit error, reported: 0.87401
7002	D1298	876.6	C,R(0.01)	4.80	probably unit error, reported: 0.8766
7015					
	normality	suspect			
	n	32			
	outliers	3			
	mean (n)	874.03			
	st.dev. (n)	0.277			
	R(calc.)	0.77			
	R(D1298:12b)	1.50			





Determination of Flash Point PMcc on sample #15056; results in °C

lab	method	value	mark	z(targ)	remarks
178					
179	D93-A	198	C,R(0.01)	-6.30	first reported:140.0
228	D93-A	218.0		-0.70	
237					
325	D93-A	220.0		-0.14	
331					
340	D93-A	216.0		-1.26	
349	D93-A	220		-0.14	
360					
398	D93-A	225.0		1.26	
432	D93-A	223.0		0.70	
445	20071				
496	D93-B	213.0		-2 10	
511	D93	223.5		0.84	
541	D93-A	222.0		0.42	
614	D93-A	217.6		-0.81	
657	DOON				
663	D93-B	218 5		-0.56	
862	D93-B	2210.0		0.00	
902	D00 D	219.0		-0.42	
922	D93-A	220.0		-0.42	
963	D03-A	218.0		-0.14	
001	D93-A	220.0		-0.70	
1023	D93-A	220.0		-0.14	
1025	1902710	210.0		_0.42	
1020	1502719	213.0		0.42	
1124	1302/19	223.5		0.04	
11/6	D03 V	220.03		0.12	
1140	1902502	220.95	07	1 34	The reported test method is not equivalent to D03
1417	1302392	230.0	ex	4.54	The reported test method is not equivalent to Das
1417		220 7		0.06	
1431	D93-A	220.7		1 1 2	
1455	D93-A	224.30		1.12	
1401	002		D(0.01)	4 00	
1720	D03 B	133.0	P(0.01)	24.50	
1954	D93-D	228	K(0.01)	-24.30	
1066	D93-D	220		2.10	
1001	D93-A	215		-1.04	
7002	D03	224		0.08	
7002	D93	224		0.90	
7015					
	normality	OK			
	normanty	25			
	outliers	20 3 (+1 ovol)			
	mean (n)	220 /0			
	st dev (n)	220.78			
	P(calc)	0/1			
	R(D03.15 R)	10.00			Compare R (D93:15 $-$ A) = 15.65
	N(D35.15-D)	10.00			O(mpare (1000, 10 - A) - 10.00)



Determination of Kinematic Viscosity at 40°C on sample #15056; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178	D445	40.2		0.11	
179	D445	40.2		0.11	
228	D445	40.11		-0.24	
237	D445	40.14		-0.12	
325	D445	40.15		-0.08	
331	D7279	36.81	R(0.01)	-13.02	
340	D445	39.859	()	-1.21	
349	D445	40.115		-0.22	
360	ISO3104	40.115		-0.22	
398	D445	40.231		0.23	
432	ISO3104	40.21		0.15	
445					
496	D445	40.160		-0.04	
511	D445	40.197		0.10	
541	D7042	40.33	С	0.62	first reported:38.40
614	D445	39.89		-1.09	
657	D7279	40.23		0.23	
663	D7279	40.190		0.07	
862	D445	40.00		-0.66	
902	D7279	40.30		0.50	
922	D7042	40.34		0.65	
963	D445	40.21		0.15	
994	D7042	40.65	R(0.05)	1.85	
1023	D445	40.1600	. ,	-0.04	
1026	D445	40.20		0.11	
1059	D7042	40.48		1.20	
1134	D445	43.9513	R(0.01)	14.64	
1146	D445	40.144		-0.10	
1161	ISO3104	40.08		-0.35	
1417	D7279	40.20		0.11	
1431	D7042	40.189		0.07	
1435		40.17		0.00	
1461	ISO3104	40.5245	С	1.37	first reported:4.5245
1599		39.95		-0.86	
1720		39.596	R(0.05)	-2.23	
1854	D445	40.14		-0.12	
1966	D445	40.19		0.07	
1981	D445	40.13		-0.16	
7002		40.0808		-0.35	
7015					
		a 1			
	normality	Suspect			
	n	34			
	outliers	4			
	mean (n)	40.1710			
	st.dev. (n)	0.13393			
	R(calc.)	0.3/50			ated from its DTs on wood sile for ACTM DAAF (see set 40); 4.00/ of second
	rt(III. 161 16)	0.7231		K calcula	ated from its PTS on used oils for ASTM D445 (see ref. 16): 1.8% of mean



Determination of Kinematic Viscosity at 100°C on sample #15056; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178	D445	6.49		-0.32	
179	D445	6.47		-0.71	
228	D445	6.519		0.25	
237	D445	6 4 4 1		-1 27	
325	D445	6 5 1 0		0.08	
331	D7279	6.08	R(0.01)	-8.34	
340	D445	6 5236	1((0.01)	0.34	
349	D445	6 486		-0.39	
360	ISO3104	6 5040		-0.04	
398	D445	6 4772		-0.57	
432	ISO3104	6.502		-0.08	
445	1000101				
496	D445	6 5045		-0.03	
511	D445	6.552		0.90	
541	D7042	6.366	R(0.05)	-2 74	
614	DIVIL		1((0.00)		
657	D7279	6 800	R(0.01)	5 75	
663	D7279	6 5035	1((0.01)	-0.05	
862	D445	6 479		-0.53	
902	D7279	6 532		0.00	
922	D7042	6 538		0.01	
963	D445	6 517		0.02	
900 904	D7042	6 552		0.21	
1023	D445	6 4729		-0.65	
1026	D445	6 50		-0.12	
1020	D7042	6 547		0.80	
1134	D445	7 5728	R(0.01)	20.87	
1146	D445	6 5154	14(0.01)	0.18	
1161	ISO3104	6.365	R(0.01)	-2 76	
1417	D7279	6 531	1((0.01)	0.49	
1431	D7042	6.5017		-0.09	
1435	DIGE	6.47		-0.71	
1461	ISO3104	6 5360		0.58	
1599	1000104	6.62		2 23	
1720		6 4 1 3 5		-1.81	
1854	D445	6 507		0.02	
1966	D445	6 4 2 9		-1 51	
1981	D445	6.506		0.00	
7002	D-1-10	6 5456		0.00	
7015		0.0400			
1010					
	normality	suspect			
	n	32			
	outliers	5			
	mean (n)	6.5061			
	st.dev. (n)	0.03980			
	R(calc.)	0.1114			
	R(lit.ref.16)	0.1431		R calcul	ated from iis PTs on used oils for ASTM D445 (see ref.16): 2.2% of mean



Determination of Viscosity Index on sample #15056

lab	method	value	mark	z(targ)	Calc iis	mark		
178	D2270	112		-1.41	112.42			
179	D2270	112		-1.41	111.53			
228	D2270	114		1.39	114.14			
237	D2270	111		-2.81	110.52			
325	D2270	114		1.39	113 54			
331	INH-2270	111		-2.81	110.04	AV	F	Outlier in Kin Visco at 40 & 100 °C
340	D2270	115		2.01	115.54	CA	Ē	
340	D2270	113		2.75	112.64		L	
360	1502000	113 /		-0.01	112.04			
200	1302909 D2270	113.4		1 02	113.44			
390	1502000	112.0		-1.03	112.00			
432	1502909	112.9		-0.15	112.90			
445	D0070			0.01	440.05			
496	D2270	113		-0.01	113.25		-	
511	D2270	115.1		2.93	115.21		E	
541	D2270	112		-1.41	106.35	R(0.01)	E	Corr in D445 at 40°C, no correction in VI
614								
657	D2270	126	R(0.01)	18.19	125.98	R(0.01)		Outlier in Kin.Visco. 100°C
663	D2270	113		-0.01	113.06			
862	D2270	113		-0.01	112.87			
902	D2270	114		1.39	113.83			
922	D2270	114		1.39	113.91			
963	D2270	114		1.39	113.58			
994					113.09			
1023					111.85			
1026	D2270	113		-0.01	112.86			
1059	ISO2909	114		1.39	113.66			
1134	IP226	130	R(0.01)	23.79	138,96	R(0.01)	Е	Outlier in Kin. Visco at 40 & 100 °C
1146	D2270	113 82	()	1 14	113 82	(/		
1161	D2270	108	R(0.01)	-7 01	107 45	R(0.01)		
1417	D2270	113		-0.01	114 25		F	
1431	D2270	113.0		-0.01	112.99		-	
1435	02210				111.67			
1461	1502909	113		-0.01	112.96			
1500	D2270	119.6	R(0.01)	9.23	110.43	R(0.01)	F	
1720	D2270	111.0	13(0.01)	-1.60	111.40	1((0.01)	-	
1954	D2270	112.45		0.62	112.46			
1004	1502000	110.45		4.21	100.76			
1001	D22303	114		-4.21	113.70		E	
7000	D2270	114		1.59	115.40			
7002					115.47			
7015								
	normality	OK			OK			
	n	29			31			
	outliers	4			5 (+1 excl)			
	mean (n)	113.01			113.07			
	st.dev. (n)	1.189			1.284			
	R(calc.)	3.33			3.60			
	R(D2270:10e1)	2.00			2.00			
	(



Determination of Water by KF on sample #15056; results in mg/kg

lak	un a file a al			-(1)	
lab	method	value	mark	z(targ)	remarks
178	D6304-C	65		-0.27	
179	D6304-C	127		0.43	
228					
237					
325	D6304-C	34		-0.62	
331	D6304-C	102.4		0.15	
340	D6304-A	100.9		0.13	
349	D6304-C	57		-0.36	
360	D6304-A	97.0		0.09	
398	D6304-C	23		-0.74	
432	20001.0				
445					
106	D6304_C	85		0.05	
430 511	D0304-C	05		-0.05	
511					
041	D0004 0	100			
614	D6304-C	126		0.41	
657	D6304-C	98		0.10	
663	D6304-A	110		0.23	
862	D6304-C	118.6		0.33	
902	D6304-A	101.57		0.14	
922	D6304	24.2		-0.73	
963	D6304-C	110		0.23	
994					
1023	D6304	97		0.09	
1026	D6304-C	40		-0.55	
1059	D6304-A	90		0.01	
1134	IP438	91.53		0.03	
1146	D6304-C	92		0.03	
1161					
1417	D6304-A	115		0 29	
1431	D6304-A	101 87		0.14	
1435	D6304	122		0.14	
1461	D0304	122		0.57	
1500	D6204	101 00		0.15	
1099	D0304	101.99		0.15	
1720				0.00	
1854	D6304-A	71		-0.20	
1966	D 0004				
1981	D6304	102.1		0.15	
7002					
7015					
	normality	OK			
	n	27			
	outliers	0			
	mean (n)	89.043			
	st.dev. (n)	30.0434			
	R(calc.)	84.121			
	R(D6304:07)	249.708			





Determination of Water Separability at 54°C on sample #15056; results in minutes

								time to		
		time to			time to			reach		
		reach 3			reach 37			complete		
		ml or less			ml of			break		time test
lab	method	emulsion	mark	z(targ)	water	mark	z(targ)	(40-40-0)	z(targ)	aborted
178										
179								30		
228	D1404			0.47						
237	D1401	24.05		-0.47	23.25		-0.46	20.55		 Voc. at 20 min
320										1es, at 50 mm
340	D1401	50	DG(0.05)	3 17	50	DG(0.05)	3 29	60		
349			_ = (= = = ;)			_ = (0.000)				
360										Yes, 30 min
398	D1401	33		0.79	35		1.19	40		Yes
432	D1401	25		-0.33	25		-0.21	>30		Yes
445	D1401	19.5		-1.10	19.5		-0.98	22.1		No
496										
511										
04 I 614										
657	D1401	33 58		0.87	33.60		0.99	50		No
663	DIHOI									
862	D1401	27		-0.05	27		0.07			Yes
902										
922										
963	D1401	>30			>30			>30		Yes, 30
994										
1023										
1026								15		
1009	D1401	 Fail			Fail			Fail		Ves
1146	ISO6614	22		-0.75	22		-0.63	26		
1161	1000011									
1417	D1401	35		1.07	35		1.19	37		No
1431					25.0		-0.21			Yes
1435	D1401	51	DG(0.05)	3.31	51	DG(0.05)	3.43			No
1461										
1599										
1720										
1004										
1981	D1401	5	G(0, 05)	-3 13	20		-0.91	25		
7002	Briot		0(0.00)							
7015										
									I	
	normality	OK			OK					
	n	8			10					
	outliers	3			2					
	mean (n)	21.39			20.53 5.070					
	SLUEV. (II) R(calc.)	0.007 16.26			0.979 16 74					
	R(D1401:12)	20.00			20.00					



Determination of Water Separability at 54°C on sample #15056; results in ml

lab	method	volume oil phase	volume water phase	volume emulsion phase	reported
178					-
179	D1401	39	39	2	
228					
237					
325	D1401	35	24	21	
331					
340	D1401	40	37	3	
349					
360	D1401	41	34	5	
398	D1401	40	33	7	
432	D1401	1	39	41	
445	D1401	40	40	40	
496	21.01				
511					
541					
614					
657	D1401	40	40	0	
663	DITOT				
862	D1401	41	39	0	
902	DITOT				
922					
963	D1401	40	30	10	
903	01401				
1023					
1025					
1020					
113/	D1401	40	28	12	
11/6	D1401	40	20	12	
1161					
1/17					
1417	D1401	34.0	37.0	0.0	
1431	D1401	34.0	37.0	9.0	
1455					
1401					
1599					
1057					
1004					
1966	D1404		40		
1981	D1401	40	40	U	
7002					
7015					

Determination of Level of contamination on sample #15056; results in ml⁻¹

lab	method	≥ 4 µm	mark	z(targ)	≥ 6 µm	mark	z(targ)	≥ 14 µm	mark	z(targ)	remarks
178	ISO4406	26405		0.12	2489		0.27	91		2.10	
179	ISO4406	4602		-2.03	652		-2 65	23		-1 02	
228	1001100										
237											
325	1504406	41807 80		1.64	5/11 03		/ 01	32.60		0.58	
321	1004406	46442		2.00	5830		5.59	64		0.00	
340	1504406	24205.0		2.03	1026.8		2.05	66		1 77	
240	1304400	24303.9		-0.09	1020.0		-2.00	165		-1.77	
260	1304400	30924 7672 1		1.33	4049		4.02	100		1 56	22/10/15
200	1304400	20200		-1.72	1502		-2.07	11.5		-1.00	22/19/15
122	1304400	32300		1.04	1303		-1.30	9		-1.00	
432	1504406	44901		1.94	3700		2.33	140		4.35	
445											
496											
511	10.0 ((0.0										
541	1804406	23550		-0.16	993		-2.11	11.0		-1.57	
614	10.0 ((0.0										
657	ISO4406	1430		-2.34	223		-3.33	/		-1.75	
663	ISO4406	34368		0.90	3220		1.43	52		0.31	22/19/13
862	ISO4406	27446		0.22	1026		-2.05	8		-1./1	22/17/10
902	ISO4406										23/21/15
922	ISO4406	16699		-0.83	2265		-0.09	20		-1.16	
963											
994						_			-		
1023	ISO4406	32873.43	С	0.76	2565.37	С	0.39	24.27	С	-0.96	
1026											
1059	D7647	2300.2		-2.25	534.4		-2.84	21.9		-1.07	
1134											
1146											20/18/12
1161											
1417	ISO4406	37184.90		1.18	1174.47		-1.82	18.70		-1.22	
1431	ISO4406	9987.0		-1.50	3552.0		1.96	108.0		2.88	
1435	ISO4406										23/21/14
1461											
1599	ISO4406	48110	ex	2.26	28446	R(0.01)	41.51	955	R(0.01)	41.76	
1720											
1854											
1966											
1981	ISO4406	179906	R(0.01)	15.22	8295	R(0.05)	9.49	545	R(0.01)	22.94	
7002											
7015											
	normality	OK			OK			not OK			
	normality	10			10			101 UK			
	outliors				10			10 2			
	outilers				∠ 2210.0			۲ ۲5 10			
	niedn (n)	20102.2			2310.9 1767 74			40.19			
	D(colo)	14904.09			1/0/./1			40.931			
	R(Calc.)	41/31.4			4949.0			61 00			
	R(D/04/.10)	20400.9			1/02.3			01.00			

Lab 1023 first reported results; ≥4 µm 3287343, ≥6 µm 256537, ≥21 µm 24.27



APPENDIX 2

Number of participants per country

1 lab in ARGENTINA 1 lab in AUSTRALIA 1 lab in AUSTRIA 1 lab in AZERBAIJAN 2 labs in BELGIUM 2 labs in BULGARIA 1 lab in CHINA, People's Republic of 2 labs in FRANCE 1 lab in GEORGIA 1 lab in GERMANY 2 labs in GREECE 2 labs in IRAN, Islamic Republic of 1 lab in ITALY 2 labs in NETHERLANDS 1 lab in NIGERIA 1 lab in NORWAY 1 lab in PAKISTAN 1 lab in PERU 1 lab in SAUDI ARABIA 1 lab in SINGAPORE 1 lab in SLOVENIA 2 labs in SPAIN 1 lab in SUDAN 1 lab in THAILAND 1 lab in TOGO 3 labs in TURKEY 3 labs in UNITED KINGDOM 2 labs in UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations:

С	= final result after checking of first reported suspect 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 outlier test
R(0.05)	= straggler in Rosner outlier test
ex	= excluded from calculations
U	= reported in different unit
W	= result withdrawn on request of the participants
fr.	= first reported
n.a.	= not applicable
n.e.	= not evaluated
SDS	= Material Safety Data Sheet

Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organization, Statistics and Evaluation, April 2014
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- 12 J.N. Miller, Analyst, <u>118</u>, 455, (1993)
- 13 Analytical Methods Committee Technical brief, No4 January 2001.
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- 15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)
- R.G. Visser and C. Nijssen-Wester, Estimation of reproducibility and measurement uncertainty of a viscosity test method from proficiency test data, Accred Qual Assur (2015) 20:125-129, DOI 10.1007/s00769-015-1110-y