Results of Proficiency Test Turbine Oil (used) May 2016

Organised by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2013, the Institute for Interlaboratory Studies (iis) organizes a proficiency test for used Turbine Oil. During the annual proficiency testing program 2015/2016, it was decided to continue the round robin for the analysis of used Turbine Oil. In this interlaboratory study 40 laboratories in 28 different countries have registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2016 used Turbine Oil proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organizer of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one bottle of 1L (labelled #16076) of used Turbine Oil.

The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization 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 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.

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2.4 SAMPLES

The necessary bulk material was obtained from a third party. The 50 litre bulk material was homogenized and transferred into 49 brown glass bottles of 1 litre (labelled #16076). The homogeneity of the subsamples #16076 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 #16076-1	939.42	405
Sample #16076-2	939.43	420
Sample #16076-3	939.43	420
Sample #16076-4	939.42	430
Sample #16076-5	939.42	425
Sample #16076-6	939.42	410
Sample #16076-7	939.41	405
Sample #16076-8	939.43	420

Table 1: homogeneity test results of subsamples #16076

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/m³	Water in mg/kg
r (sample #16076)	0.02	26
reference test method	ASTM D1298:12b	ASTM D6304:16
0.3 x R(reference test method)	0.45	189

Table 2: evaluation of the repeatabilities of the subsamples #16076

Both calculated repeatabilities are less than 0.3 times the corresponding reproducibilities of the reference test method. Therefore, homogeneity of the subsamples #16076 was assumed.

To each of the participating laboratories, one sample of 1.0 L brown glass bottle (labelled #16076) was sent on April 27, 2016.

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.

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2.6 ANALYSES

The participants were requested to determine on sample #16076: Acid Number, Base Number, Color ASTM, Density at 15°C, Flash Point (C.O.C. & PMcc), Insoluble Color Bodies, Kinematic Viscosity at 40°C and at 100°C, Viscosity Index, Oxidation Stability RPVOT, Water by KF, Water separability and Level of contamination (parts per ml and ISO Class).

To get comparable test results a detailed report form, on which the units were prescribed as well as the reference test methods and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/. A SDS and a form to confirm receipt of the samples were added to the sample package.

3 RESULTS

During five weeks after sample dispatch, the test results of the participants 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 reanalyses). 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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the test results 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 visualise 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 reproducibilities, the z-scores 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:

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

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The $z_{\text{(target)}}$ scores are listed in the test result tables in appendix 1. Absolute values for z<2 are very common and absolute values for z>3 are very rare. 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, no problems were encountered with the dispatch of the samples to laboratories. Only two participants were not able to report any test results due to several problems. Not all laboratories were able to report all analyses requested. In total 38 participants reported 474 test results. Observed were 15 outlying results, which is 3.2% 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.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D2270) and an added designation for the year that the method was adopted or revised (e.g. D2270:10). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D2270:10(2016). In the results tables of Appendix 1 only the method number and year of adoption or revision e.g. D2270:10 will be used.

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. One statistical outlier was observed.

The calculated reproducibility after rejection of the statistical outlier is not in

agreement with the requirements of ASTM D664:11a.

Base Number: This determination may be problematic at the very low level of 0.76 mg

KOH/g. One statistical outlier was observed. Regretfully, ASTM D2896:11 does provide only precision data on base numbers ranging from 5-27 mg

KOH/g. Therefore no significant conclusions were drawn.

Color ASTM: This determination was not problematic. No statistical outliers were

observed. The calculated reproducibility is in agreement with ASTM

D1500:12.

Density at 15°C:

This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D1298:12b.

Flash Point C.O.C.: This determination was very problematic. A wide range of test results was reported (from 150-246°C). No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of ASTM D92:16. Therefore no z-scores were calculated. Probably ASTM D92 is not applicable for this sample. Looking at the average result found for Flash Point PMcc, it appears that part of the sample has a lower flash point, which might not have been detected to the same extent by all laboratories using the Cleveland Open Cup.

Flash Point PMcc: This determination may not be problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D93:16, procedure B. When evaluated separately for procedure A (14 participants) and procedure B (8 participants), the calculated reproducibilities for both procedures are in agreement with the requirements of the respective procedure of ASTM D93:16.

Insoluble Color Bodies: This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is almost in agreement with the requirements of ASTM D7843:12 (table 1, sample 1).

Kin.Visco.at 40°C: 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.

Kin.Visco.at 100°C: This determination was not problematic. One statistical outlier was 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 outlier, is in full agreement with this literature reproducibility requirement.

Viscosity Index

This determination was very problematic. No statistical outliers were observed, but four test results were excluded as the test results were a statistical outlier in the Kinematic Viscosity determinations and one test result was excluded, because it was calculated based on test results from method D7279 (which is not mentioned in paragraph 1.3 of ASTM D 2270). The calculated reproducibility after rejection of the suspect data is not at all in agreement with ASTM D2270:10(2016).

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 three 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. 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. The calculated reproducibility after rejection of the suspect data is surprisingly larger than for the reported VI test results and again not in agreement with ASTM D2270:10(2016).

Oxidation Stability RPVOT: This determination is not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier, is in good agreement with the requirements of ASTM D2272:14a.

Water:

This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D6304:16.

Water separability: Twelve laboratories reported various results for this sample. All laboratories agreed that a high amount of emulsion was present after 30 minutes or even 60 minutes. Only six laboratories reported all information that is required for reporting ASTM D1401. From these six, four aborted the test after 30 minutes and two after 60 minutes. The test results varied between 32-38 ml oil, 4-20 ml water and 22-43 ml emulsion. Surprisingly, the other six laboratories did not report according to ASTM D1401 by not reporting any (abort) time or by reporting volumes that did not match the reported time.

Level of Cont:

This determination was problematic for the test results reported in parts per ml. In total two statistical outliers were observed and nine test results were excluded as the reported tests results for parts/ml did not match the reported ISO-class. The calculated reproducibilities for number of particles $\geq 4\mu m$, $\geq 6\mu m$ and $\geq 14\mu m$, after rejection of the suspect data, are not in agreement with the requirements of ASTM D7647:10.

This determination was also problematic for the test results reported in ISO-classes. No statistical outliers were observed, but nine test results were excluded as the reported tests results ISO-class for did not match the reported parts/ml. The calculated reproducibilities for number of particles $\geq 4\mu m$, $\geq 6\mu m$ and $\geq 14\mu m$, 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	34	0.27	0.18	0.12
Base Number, total	mg KOH/g	16	0.76	1.46	(0.05)
Color ASTM		16	4.1	0.9	1.0
Density at 15°C	kg/m ³	31	939.3	1.0	1.5
Flash Point C.O.C.	°C	21	209	75	(18)
Flash Point PMcc	°C	26	117	11	10
Insoluble Color Bodies		8	10.9	7.9	6.3
Kinematic Viscosity at 40°C	mm²/s	32	25.58	0.21	0.46
Kinematic Viscosity at 100°C	mm²/s	35	5.002	0.120	0.110
Viscosity Index		24	124	7	2
Oxidation Stability RPVOT	minutes	8	513	53	116
Water by KF	mg/kg	31	429	97	642
Water Separability at 54°C					
- Time to reach 3 ml or less emulsion	minutes	12	n.a.	n.a.	n.a.
- Time to reach 37 of water	minutes	12	n.a.	n.a.	n.a.
Level of Contamination ≥4µm	parts/ml	18	6165	10140	6967
Level of Contamination ≥6µm	parts/ml	18	857	1126	652
Level of Contamination ≥14µm	parts/ml	19	61	95	82
Level of Contamination ≥4µm	ISO class	19	20	3	2
Level of Contamination ≥6µm	ISO class	21	17	3	1
Level of Contamination ≥14µm	ISO class	22	13	4	2

Table 3: reproducibilities of tests of sample #16076

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.

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4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2016 WITH PREVIOUS PTs

	May 2016	May 2015	May 2014	May 2013
Number of reporting labs	38	39	29	27
Number of results reported	474	398	301	336
Statistical outliers	15	36	10	14
Percentage outliers	3.2%	9.0%	3.3%	4.2%

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 2016	May 2015	May 2014	May 2013
Acid Number, total	-	+		-
Base Number, total	()	()		
Color ASTM	+/-	+	++	-
Density at 15°C	++	++	+/-	
Flash Point C.O.C.	()	n.e.	n.e.	n.e.
Flash Point PMcc	+/-	+/-	-	-
Insoluble Color Bodies	+/-	n.e.	n.e.	n.e.
Kinematic Viscosity at 40°C	++	++	++	+
Kinematic Viscosity at 100°C	+/-	+	+/-	+
Viscosity Index				
Oxidation Stability RPVOT	++	n.e.	n.e.	n.e.
Water by KF	++	++	++	++
Water Separability	n.e.	+	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

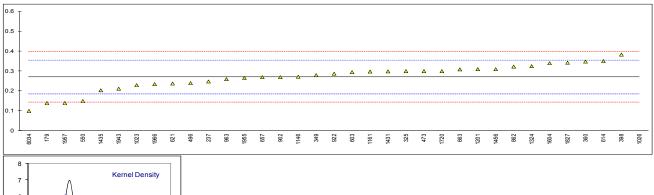
- : group performed worse than the standard

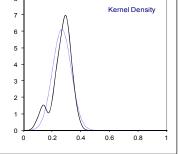
-- : group performed much worse than the standard

n.e.: not evaluated

APPENDIX 1Determination of Acid Number (total) on sample #16076; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
178			man		
179	D664	0.14		-3.06	
214					
237	D974	0.248		-0.51	
325	D664	0.30		0.71	
331					
349	D664	0.28	С	0.24	First reported 0.028
360	ISO6618	0.348		1.84	
398	D664	0.383		2.67	
473	D664	0.3		0.71	
496	D664	0.24		-0.70	
541					
550	D664	0.15		-2.83	
603	D664	0.295		0.59	
614	D664	0.35		1.89	
621	D664	0.238		-0.75	
657	D664	0.27		0.00	
663	D664	0.309		0.92	
862	D664	0.323		1.25	
902	D664	0.27		0.00	
922 963	D664 D664	0.287 0.26		0.41	
1023	In house	0.26		-0.23 -0.94	
1023	D664	1.4	R(0.01)	26.65	
1146	D664	0.272	K(0.01)	0.05	
1161	D664	0.272		0.66	
1201	D664	0.31		0.95	
1324	D664	0.325		1.30	
1431	D664	0.2988		0.68	
1435	D664	0.204		-1.55	
1456	D974	0.31		0.95	
1604	D664	0.34		1.65	
1720	D664	0.30		0.71	
1827	D664	0.342		1.70	
1943	ISO6618	0.212		-1.36	
1955	D664	0.266		-0.09	
1957	D664	0.14		-3.06	
1966	ISO6618	0.2353		-0.81	
6016					
6034		0.1		-4.01	
					Only ASTM D664
	normality	OK			OK
	n	34			28
	outliers	1			1
	mean (n)	0.2698			0.2711
	st.dev. (n)	0.06545			0.06858
	R(calc.)	0.1833			0.1920
	R(D664:11a)	0.1187			0.1193





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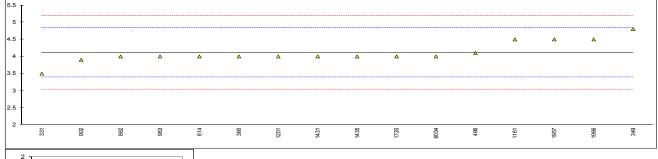
Determination of Base Number on sample #16076; results in mg KOH/g

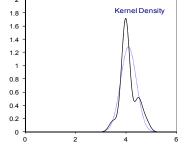
lab	method	•	value		mark	7	z(targ)	remark	S							
178																<u>.</u>
179	D2896 - B		0.69													
214 237	D2896 - B		1.13													
325	D2896 - B		1.00													
331	22000 2															
349																
360	D0000 4															
398	D2896 - A		0.04													
473 496	D2896 - B		0.24													
541	D2896 - B		<0.1													
550	D2896 - A		0.07													
603																
614	D2896 - B		1.06													
621 657																
663																
862	D2896 - B		1.05													
902																
922	D2896 - B		< 0.5													
963 1023	D2896 - A		1.31													
1026	D2896 - B		1.7													
1146	D2896 - A		0.58													
1161	D2896 - B		0													
1201	D2896 - A		0.16													
1324 1431	D2896 - A		1.074													
1435	D2896 - A		<1													
1456	D2896 - A		0.97													
1604																
1720 1827	D2896 - B		8.8		G(0.01)			False n	ositive t	est resul	t2					
1943	D2000 B				O(0.01)			i dioc p	Ooitive t	cot resur						
1955																
1957 1966	D2896 - A		1.10													
6016																
6034																
	normality		OK													
	n outliers		16 1													
	mean (n)		0.7609)												
	st.dev. (n)		0.5197													
	R(calc.) R(D2896:15 (A))	1.4554					Applica	tion rand	ne ASTN	1 D2896	i = 5 -27	mg KO	H/a		
		-//	(*****													
1.8															Δ	
1.6 +															_	
1.2														Δ		
1 +							Δ	Δ	Δ	Δ	Δ	Δ	^			
0.8						Δ										
0.6 -					Δ											
0.2 -			Δ	Δ												
0 7		<u>∆</u>	1201	96	1146	179	1456	325	862	614	1324	1957	237	963	1026	1827
=	: ° '	n	12	4	7	-	4	e e			5	19	- 7	6	6	82
0.9		Kerne	el Density													
0.8 - 0.7 -	\wedge	1101110	. Donony													
0.6 -																
0.5 - 0.4 -		١														
0.3 -																
0.2 -																
0 -3			3	5												
	·															

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Determination of Color ASTM on sample #16076

lab	method	value	mark z(targ)	remarks
178				
179	D1500	L4.5		
214	2.000			
237	D1500	L4.5		
325	D6045	L4.0		
331	D1500	3.5	-1.72	
349	D6045	4.8	1.92	
360	D1500	4.0	-0.32	
398	D1500	L 4.0		
473	B 1000			
496	D6045	4.1	-0.04	
541	D1500	L4.0		
550	D 1000			
603	D1500	L5.0		
614	D1500	4	-0.32	
621	D1500	L 4.0		
657	D 1000			
663	D1500	L4.0		
862	D1500	4.0	-0.32	
902	D1500	3.9	-0.60	
922	D1500	L4.5	-0.00	
963	D1500	4.0	-0.32	
1023	D 1000		-0.02	
1026	D1500	L 4.5		
1146	D 1000			
1161	D1500	4.5	1.08	
1201	D1500	4.0	-0.32	
1324	D1000			
1431	D1500	4.0	-0.32	
1435	D1500	4.0	-0.32	
1456	D1500	L4.0		
1604	B 1000			
1720	D1500	4.0	-0.32	
1827	B 1000			
1943				
1955				
1957	D1500	4.5	1.08	
1966	D1500	4.5	1.08	
6016	2.000			
6034	D1500	4	-0.32	
		OI		
	normality	OK		
	n	16		
	outliers	0		
	mean (n)	4.11		
	st.dev. (n)	0.312		
	R(calc.)	0.87		
	R(D1500:12)	1.00		
5.5 T				

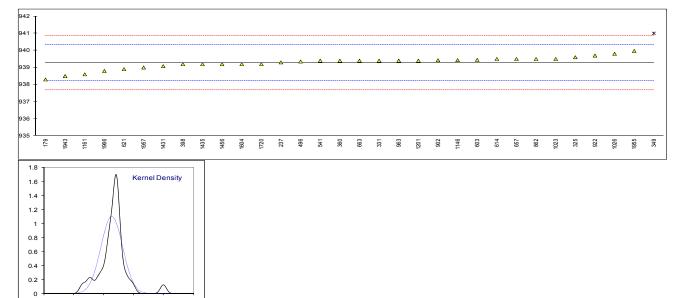




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Determination of Density at 15°C on sample #16076; results in kg/m³

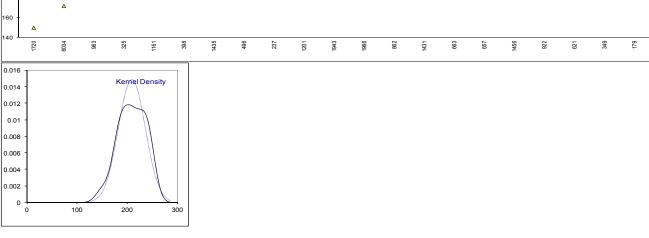
lab	method	value	mark	z(targ)	remarks
178					
179	D1298	938.3		-1.82	
214					
237	D4052	939.3		0.04	
325	D4052	939.6		0.60	
331	ISO12185	939.4		0.23	
349	D4052	941.0	R(0.01)	3.22	
360	D4052	939.4	(5.5.)	0.23	
398	D1298	939.2		-0.14	
473	2.200				
496	D4052	939.35		0.14	
541	D4052	939.4		0.23	
550	D-1002				
603	D4052	939.45		0.32	
614	D4052	939.5	С	0.42	First reported 0.9395 kg/m ³
621	D4052	938.9	Ü	-0.70	That reported 0.0000 kg/m
657	D4052	939.5		0.42	
663	D4052	939.40		0.23	
862	D4052 D4052	939.5		0.42	
902	D4052	939.425		0.42	
922	D4052 D4052	939.7		0.20	
963	D4052 D4052	939.4		0.73	
1023	D4052 D4052	939.5		0.23	
1025	D4052 D4052	939.8		0.42	
1146	ISO12185	939.43		0.98	
1161	ISO3675	938.6		-1.26	
1201	ISO12185	939.4		0.23	
1324	130 12 103	939.4			
1431	D4052	939.08		-0.37	
1435	D4052 D4052	939.00	С	-0.37 -0.14	First reported 0.937
1456	D4052 D4052	939.2	C	-0.14	i list reported 0.957
1604	D1298	939.2		-0.14	
1720	D4052	939.2		-0.14	
1827	D4032	939.2		-0.14	
1943	ISO3675	938.5		-1.45	
1955	D7042	939.97		1.29	
1955	D4052	939.97		-0.52	
1966	D1298	938.8		-0.32	
6016	D1290	930.0		-0.09	
6034					
0034					
	normality	suspect			
	n	31			
	outliers	1			
		939.28			
	mean (n) st.dev. (n)	0.362			
	R(calc.)	1.01			
	R(D1298:12b)	1.50			
	14(01200.120)	1.50			



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Determination of Flash Point C.O.C. on sample #16076; results in °C

lab	method	k	-	value		marl	(z(targ)		emarks	3									
178 179 214	D92			246 																
237	D92			202																
325	D92			184.0																
331	D00																			
349 360	D92			246																
398	D92			186																
473																				
496 541	D92			200.0																
550																				
603																				
614	Doo			0440																
621 657	D92 D92			244.0 231.6																
663	D92			231																
862	D92			216																
902 922	D92			240																
963	D92 D92			182																
1023																				
1026																				
1146 1161	ISO259	2		 185.0																
1201	D92	_		204.0																
1324																				
1431 1435	D92 D92			220.4 193																
1456	D92			232																
1604																				
1720	D92			150.0																
1827 1943	ISO259	2		210					·											
1955	100200	_																		
1957	D00																			
1966 6016	D92			214																
6034	D92			172																
	normali	ty		OK																
	n outliers			21 0																
	mean (ı			209.00																
	st.dev.	(n)		26.807																
	R(calc.)) 16\		75.06																
	R(D92:	10)		(18.00)	'															
²⁶⁰ T																				
240 -																	Δ	Δ	Δ	Δ
220 -													Δ	Δ	Δ	Δ				
-									Δ	Δ	Δ	Δ								
200 +						Δ	Δ	Δ	-											
180 -	Δ	Δ	Δ	Δ	Δ															
160 -																				
140		e e	10		e-	10	· · ·	~			· ·	61		m	~	sc.	61	_		
1720	6034	963	325	1161	398	1435	496	237	1201	1943	1966	862	1431	963	657	1456	922	621	349	179



Used Turbine Oil: iis16L03 page 16 of 31

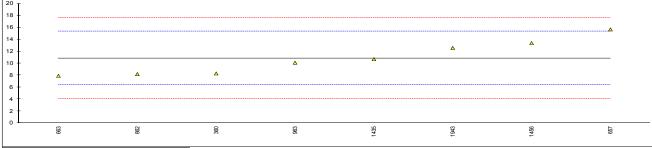
Determination of Flash Point PMcc on sample #16076; results in °C

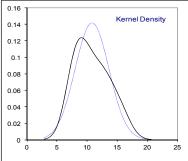
lab	metho	t		value)	n	nark		z(targ)	re	marks	;											
178																								
179	D93-A			122.0						1.28														
214																								
237	D93-A			114.0						-0.96														
325 331	D93-A D93-B			116.0 123.3						-0.40 1.65														
349	D93-B			111						-1.80														
360	D30-D																							
398	D93-A			120						0.72														
473	D93-B			116.5						-0.26														
496	D93-B			114.3						-0.87														
541	D93-A			113.5						-1.10														
550	D3828			127						2.68														
603	D3828			119.0						0.44														
614 621	D93-A			122.0						1.28														
657																								
663	D93-B			116.5						-0.26														
862	D93-B			111.0						-1.80														
902	D93-A			113.5						-1.10														
922	D93-A			119						0.44														
963																								
1023	D93-A	o 4		116						-0.40														
1026 1146	ISO271 In hous	9-A		118 115.2						0.16 -0.62														
1161	III IIOus	C								-0.02														
1201	D93-A			116.0						-0.40														
1324	20071																							
1431	D93-A			115.7						-0.48														
1435	D93-A			121.5						1.14														
1456	D93-B			117.3	25					-0.03														
1604																								
1720	D93-B			115.5						-0.54														
1827 1943																								
1955																								
1957																								
1966	D93-A			117						-0.12														
6016																								
6034				122						1.28														
													05	DO	3 me	thad	^	Only	. רוחס	math	od D			
	normal	tv		OK									OK		<u>s me</u>	lilou	A	susp		meu	od B			
	n	Ly		26									14					8	COL					
	outliers			0									0					0						
	mean (117.4										′.44				115.						
	st.dev.			3.858									3.0					3.92						
	R(calc. R(D93:) 16 D\		10.80		_	omn	oro D)/D03	3:16-A	.) – 0	24	8.5					10.9						
	К(D95.	10-6)		10.00		•	Julip	ale in	(Dac). 10-7-	() – 0	4	8.3	+				10.0	U					
130 T																								
																								Δ
125																			Δ	Δ	Δ	Δ	Δ	
120 -															Δ	Δ	Δ	Δ						
115 -			Δ		Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ										
	Δ 4	. 4		_																				
110 + 4																								
105 -																								
100																								
88	862	905	237	496	1146	1720	1431	325	1023	1201	473	983	1986	1456	1026	903	922	88	1435	179	614	6034	331	220
0.12						1																		
0.12			Kern	el Densi	ty																			
0.1 -		M																						
0.08 -		$/\!/ \setminus$																						
0.08	1	/ \\																						
0.06 -			\																					
			V																					
0.04 -																								
0.02 -																								
0.02 -	110	120		130	140																			

Used Turbine Oil: iis16L03 page 17 of 31

Determination of Insoluble Color Bodies, membrane patch colorimetry on sample #16076;

lab	method	value	mark	z(targ)	remarks
178	mounou		HIMIN	2(tary)	Tomano
179					
214					
237					
325					
331					
349					
360	D7843	8.3		-1.14	
398	27010				
473					
496					
541					
550					
603					
614					
621					
657	D7843	15.679		2.14	
663	D7843	7.9		-1.31	
862	D7843	8.2		-1.18	
902					
922					
963	D7843	10.1		-0.34	
1023					
1026					
1146					
1161					
1201					
1324					
1431					
1435	D7843	10.7		-0.07	
1456	D7843	13.389		1.13	
1604					
1720					
1827	D7040	40.570		0.77	
1943	D7843	12.579		0.77	
1955					
1957					
1966					
6016 6034					
0034					
	normality	unknown			
	n	8			
	outliers	0			
	mean (n)	10.86			
	st.dev. (n)	2.817			
	R(calc.)	7.89			
	R(D7843:12)	6.30			
	Table 1 sample 1	2.00			
	. ac.o . odnipio i				
²⁰ T					

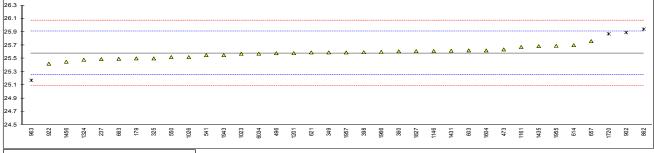


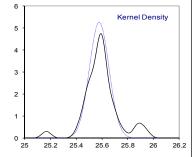


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Determination of Kinematic Viscosity at 40°C on sample #16076; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178					
179	D445	25.5		-0.49	
214					
237	D445	25.49		-0.55	
325	D445	25.50		-0.49	
331					
349	D445	25.59		0.06	
360	D445	25.607		0.16	
398	D445	25.593		0.08	
473	D7042	25.635		0.33	
496	D445	25.578		-0.01	
541	D7042	25.550		-0.18	
550	D7279	25.52		-0.36	
603	D7042	25.62		0.24	
614	D445	25.7		0.73	
621	D445	25.59		0.06	
657	D7279	25.76		1.10	
663	D445	25.494		-0.52	
862	D445	25.94	R(0.05)		
902	D7279	25.89	R(0.05)		
922	D445	25.42		-0.97	
963	D445	25.17	R(0.05)		
1023	D445	25.569		-0.07	
1026	D445	25.52		-0.36	
1146	D445	25.612		0.20	
1161	ISO3104	25.672		0.56	
1201	D445	25.58		0.00	
1324	ISO3104	25.480		-0.61	
1431	D7042	25.616		0.22	
1435	D7042	25.684		0.63	
1456	D7042	25.45		-0.79	
1604	D445	25.62	- (a a -)	0.24	
1720	D445	25.87	R(0.05)		
1827	D445	25.61		0.18	
1943	ISO3104	25.55		-0.18	
1955	D7042	25.6897		0.67	
1957	D7042	25.59		0.06	
1966	D445	25.598		0.11	
6016	D445				
6034	D445	25.57		-0.06	
	normality	OK			
	n	32			
	outliers	4			
	mean (n)	25.580			
	st.dev. (n)	0.0760			
	R(calc.)	0.213			
	R(lit. ref 16)	0.460		R calculated	d from iis PTs on used oils for ASTM D445 (see ref 16.): 1.8% of mean
20.2					· · ·

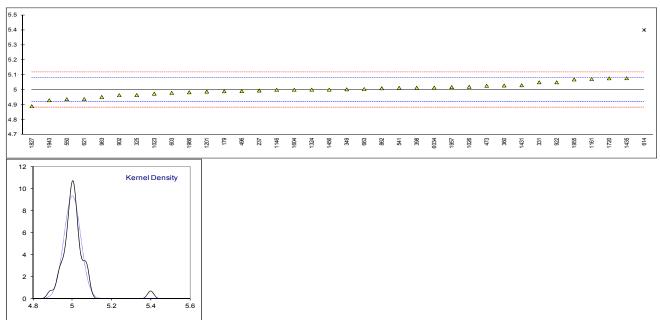




Used Turbine Oil: iis16L03 page 19 of 31

Determination of Kinematic Viscosity at 100°C on sample #16076; results in mm²/s

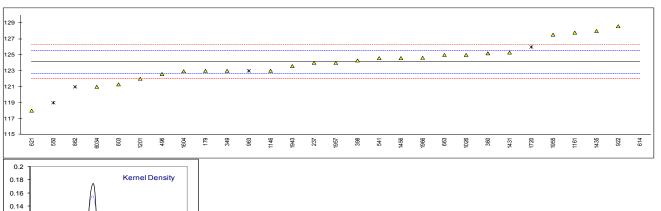
lab	method	value	mark	z(targ)	remarks
178					
179	D445	4.99		-0.29	
214					
237	D445	4.995		-0.17	
325	D445	4.965		-0.93	
331	D7279	5.05		1.23	
349	D445	5.003		0.04	
360	D445	5.0286		0.69	
398	D445	5.0132		0.30	
473	D7042	5.0271		0.65	
496	D445	4.9910		-0.27	
541	D7042	5.0129		0.29	
550	D7279	4.937		-1.64	
603	D7042	4.979		-0.57	
614	D445	5.40	R(0.01)	10.14	
621	D445	4.938	11(0.01)	-1.62	
657	D770			-1.02	
663	D445	5.0048		0.08	
862	D445	5.010		0.21	
902	D7279	4.964		-0.96	
922	D445	5.050		1.23	
963	D445	4.952		-1.26	
1023	D445	4.974		-0.70	
1026	D445	5.02		0.47	
1146	D445	4.9994		-0.06	
1161	ISO3104	5.072	С	1.79	First reported 5.161
1201	D445	4.988	O	-0.35	That reported 5.101
1324	ISO3104	5.0010		-0.01	
1431	D7042	5.0302		0.73	
1435	D7042	5.0774		1.93	
1456	D7042	5.001		-0.01	
1604	D445	5.000		-0.04	
1720	D7042	5.0765		1.91	
1827	D445	4.89		-2.84	
1943	ISO3104	4.93		-1.82	
1955	D7042	5.0691		1.72	
1957	D7042	5.018		0.42	
1966	D445	4.983		-0.47	
6016	D443	4.903 		-0.47	
6034	D445	5.015		0.34	
0034	D443	3.013		0.54	
	normality	OK			
	n	35			
	outliers	1			
	mean (n)	5.0016			
	st.dev. (n)	0.04270			
	R(calc.)	0.1196			
	R(lit. ref 16)	0.1190	D	calculated	d from iis PTs on used oils for ASTM D445 (see ref 16.): 2.2% of mean
	1 (iit. 161 10)	0.1100	IX.	Calculatet	a month list 13 on used ons for Activi D770 (see 161 10.). 2.2 /0 of filedit

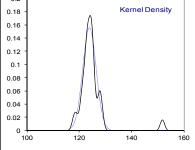


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Determination of Viscosity Index on sample #16076

178	lab	method	value	mark	z(targ)	Calc. by iis	mark	remarks
214 237 D2270 124 -0.18 123.83 325 121.46 331 349 D2270 123 -1.58 123.52 360 D2270 125.2 1.50 125.29 398 D2270 124.3 0.24 124.92 473 124.92 496 D2270 122.6 -2.14 122.72 541 D2270 124.6 0.66 124.63 550 D2270 119 ex -7.18 No calculation, not applicable for D7279 603 D2270 121.3 -3.96 121.43 Outlier in kinematic Viscosity at 100°C 621 D2270 118 -8.58 118.59 657 663 D2270 125 1.22 124.54	178							
214 237 D2270 124 -0.18 123.83 325 121.46 331 349 D2270 123 -1.58 123.52 360 D2270 125.2 1.50 125.29 398 D2270 124.3 0.24 124.92 473 124.92 496 D2270 122.6 -2.14 122.72 541 D2270 124.6 0.66 124.63 550 D2270 119 ex -7.18 No calculation, not applicable for D7279 603 D2270 121.3 -3.96 121.43 Outlier in kinematic Viscosity at 100°C 621 D2270 118 -8.58 118.59 657 663 D2270 125 1.22 124.54	179	D2270	123		-1.58	123.36		
325 121.46 331 125.2 123.52 360 D2270 125.2 1.50 125.29 398 D2270 124.3 0.24 124.26 473 124.92 496 D2270 122.6 -2.14 122.72 541 D2270 124.6 0.66 124.63 550 D2270 119 ex -7.18 No calculation, not applicable for D7279 603 D2270 151.794 ex 38.74 151.57 ex Outlier in kinematic Viscosity at 100°C 621 D2270 118 -8.58 118.59 657 663 D2270 125 1.22 124.54	214							
331	237	D2270	124		-0.18	123.83		
331	325					121.46		
349 D2270 123 -1.58 123.52 360 D2270 125.2 1.50 125.29 398 D2270 124.3 0.24 124.26 473 124.92 496 D2270 122.6 -2.14 122.72 541 D2270 124.6 0.66 124.63 550 D2270 119 ex -7.18 No calculation, not applicable for D7279 603 D2270 121.3 -3.96 121.43 Outlier in kinematic Viscosity at 100°C 621 D2270 118 -8.58 118.59 Outlier in kinematic Viscosity at 100°C 657 663 D2270 125 1.22 124.54								
360 D2270 125.2 1.50 125.29 398 D2270 124.3 0.24 124.26 473 124.92 496 D2270 122.6 -2.14 122.72 541 D2270 124.6 0.66 124.63 550 D2270 119 ex -7.18 No calculation, not applicable for D7279 603 D2270 121.3 -3.96 121.43 614 D2270 151.794 ex 38.74 151.57 ex Outlier in kinematic Viscosity at 100°C 621 D2270 118 -8.58 118.59 657 663 D2270 125 1.22 124.54		D2270	123		-1.58	123.52		
398 D2270								
473 124.92 496 D2270 122.6 -2.14 122.72 541 D2270 124.6 0.66 124.63 550 D2270 119 ex -7.18 No calculation, not applicable for D7279 603 D2270 121.3 -3.96 121.43 Outlier in kinematic Viscosity at 100°C 621 D2270 118 -8.58 118.59 Outlier in kinematic Viscosity at 100°C 657 663 D2270 125 1.22 124.54								
496 D2270 122.6 -2.14 122.72 541 D2270 124.6 0.66 124.63 550 D2270 119 ex -7.18 No calculation, not applicable for D7279 603 D2270 121.3 -3.96 121.43 121.43 Outlier in kinematic Viscosity at 100°C 621 D2270 118 -8.58 118.59		2-2.0						
541 D2270 124.6 0.66 124.63 550 D2270 119 ex -7.18 No calculation, not applicable for D7279 603 D2270 121.3 -3.96 121.43 Outlier in kinematic Viscosity at 100°C 621 D2270 118 -8.58 118.59 663 D2270 125 1.22 124.54 124.54		D2270						
550 D2270 119 ex -7.18 No calculation, not applicable for D7279 603 D2270 121.3 -3.96 121.43 614 D2270 151.794 ex 38.74 151.57 ex Outlier in kinematic Viscosity at 100°C 621 D2270 118 -8.58 118.59 657 663 D2270 125 1.22 124.54								
603 D2270 121.3 -3.96 121.43 614 D2270 151.794 ex 38.74 151.57 ex Outlier in kinematic Viscosity at 100°C 621 D2270 118 -8.58 118.59 657				ex				No calculation, not applicable for D7279
614 D2270 151.794 ex 38.74 151.57 ex Outlier in kinematic Viscosity at 100°C 621 D2270 118 -8.58 118.59 657 663 D2270 125 1.22 124.54				O.A.				The salediation, flot applicable for B7276
621 D2270				ex			ex	Outlier in kinematic Viscosity at 100°C
657 663 D2270 125 1.22 124.54				CX			OX.	Cutilet in Milematic viscosity at 100 C
663 D2270 125 1.22 124.54		DZZIO			-0.50			
		D2270			1 22			
862 D2270 121 ex -4.38 120.90 ex Outlier in kinematic Viscosity at 40°C	862	D2270 D2270	121	AV	-4.38	120.90	AV	Outlier in kinematic Viscosity at 40°C
902 No calculation, not applicable for D7279		DZZIO		CX	-4.50		CX	
922 D2270 128.6 6.26 128.65		D2270			6.26			No calculation, not applicable for D1219
963 D2270 123 ex -1.58 123.50 ex Outlier in kinematic Viscosity at 40°C				AV			ΩV	Outlier in kinematic Viscosity at 40°C
1023 121.51		D2210		CX			CX	Outlier in kinematic viscosity at 40 C
1026 D2270 125 1.22 125.45		D2270						
1146 D2270 123.0 -1.58 123.05								
				C				First reported 124 F
1161 D2270 127.8 C 5.14 127.94 First reported 134.5 1201 D2270 122 -2.98 122.47				C				First reported 134.5
		D2270						
1324 124.38 1431 D2270 125.3 1.64 125.33		D2270						
1435 D2270 128 5.42 128.24								
1456 D2270 124.6 0.66 124.65								
1604 D2270 122.945 -1.65 123.02 1720 D2270 126 ex 2.62 126.46 ex Outlier in kinematic Viscosity at 40°C				٥٧,			0.4	Outlier in kinematic Vincesity at 40°C
•		D2270		ex			ex	Outlier in kinematic viscosity at 40 C
1827 114.75		1000000		о г				First non-orted 447.0
1943 ISO2909 123.6 C, E -0.74 118.33 First reported 117.3				C, E				First reported 117.3
1955 D2270 127.538 4.78 127.56								
1957 D2270 124 -0.18 124.65				_				
1966 D2270 124.63 E 0.71 121.93		D2270		E	0.71			
6016		D0070		_	4.00			
6034 D2270 121 E -4.38 124.61	6034	D2270	121	E	-4.38	124.61		
OK.			OK					
normality OK suspect		•						
n 24 29								
outliers 0 (+5 excl) 0 (+4 excl)								
mean (n) 124.13 123.62		` '						
st.dev. (n) 2.401 2.984		, ,						
R(calc.) 6.72 8.35		` ,						
R(D2270:10) 2.00 2.00		K(D22/0:10)	∠.00			∠.00		





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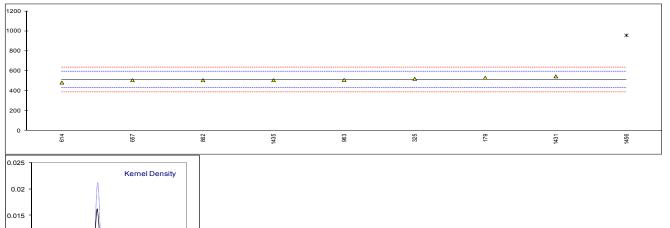
0.01

0.005

200 400

Determination of Oxidation Stability RPVOT on sample #16076; results in minutes

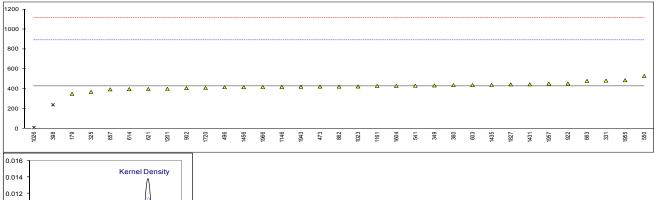
lab	method	value	mark	z(targ)	remarks
178					
179	D2272	532		0.45	
214					
237	D2272	>480			
325	D2272	520		0.16	
331					
349					
360					
398					
473					
496					
541					
550					
603					
614	D2272	483		-0.73	
621	DZZIZ			-0.73	
657	D2272			-0.18	
	DZZIZ	506 		-0.16	
663	D0070				
862	D2272	506		-0.18	
902					
922	D0070				
963	D2272	509		-0.11	
1023					
1026					
1146					
1161					
1201					
1324	D				
1431	D2272	545		0.76	
1435	D2272	506		-0.18	
1456	D2272	957	C,G(0.01)	10.68	First reported 900
1604					
1720					
1827					
1943					
1955					
1957					
1966					
6016					
6034					
	normality	OK			
	n	8			
	outliers	1			
	mean (n)	513.4			
	st.dev. (n)	18.88			
	R(calc.)	52.9			
	R(D2272:14a)	116.3			
	11(02212.14a)	1 10.0			

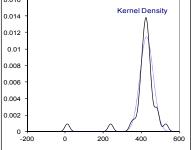


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Determination of Water by KF on sample #16076; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178					
179	D6304-C	350		-0.35	
214					
237					
325	D6304-C	369		-0.26	
331	In house	482.8		0.23	
349	D6304-A	434		0.02	
360	ISO12937	437		0.03	
398	ISO12937	240	G(0.01)	-0.83	
473	D6304-C	423		-0.03	
496	D6304-C	417		-0.05	
541	D6304-A	431.5		0.01	
550	E203	530		0.44	
603	D6304-C	438		0.04	
614	D6304-C	397.9		-0.14	
621	D6304-B	399		-0.13	
657	D6304-C	395		-0.15	
663	D6304-C	480		0.22	
862	D6304-A	423		-0.03	
902	D6304-A	407.0897		-0.10	
922	D6304-A	454		0.11	
963					
1023	D6304-A	424		-0.02	
1026	D6304-C	12	G(0.01)	-1.82	
1146	D6304-A	420		-0.04	
1161	D6304-C	429.370		0.00	
1201	D6304-C	400		-0.13	
1324	D00044				
1431	D6304-A	445.5		0.07	
1435	EN60814	439.6		0.05	
1456	D6304-C	417	•	-0.05	-
1604	D6304-A	430	С	0.00	First reported 0.043 mg/kg
1720	D6304-B	408.9		-0.09	
1827	D6304-A	445.4		0.07	
1943	EN60814	421.48		-0.03	
1955	D6304-A	486.3		0.25	
1957	D6304-B	453		0.10	
1966	EN60814	418.52		-0.05	
6016 6034					
6034					
	normality	cuencet			
	normality	suspect 31			
	n outliers	2			
	mean (n)	429.27			
	st.dev. (n)	34.752			
	R(calc.)	97.31			
	R(D6304:16)	641.67			
	. 1(20007.10)	011.01			





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Determination of Water Separability at 54°C on sample #16076; results in minutes

		Aluan An unna la				Aires As useels		
		time to reach		41		time to reach	44	41 44
lab		3 ml or less	-(4a)	time to reach	-/4	complete break	test	time test
lab	method	emulsion	z(targ)	37 ml of water	z(targ)	(40-40-0)	aborted	aborted
178	D4404							
179 214	D1401					30		
							YES	
237	D1401	 >60		 >60		>60	YES	60
325	D1401	>60					_	
331								
349	D4404							20
360	D1401						YES	30
398								
473								
496								
541								
550								
603	54464							
614	D1401	30		30		30	YES	
621								
657	D1401	>30		>30		>30	YES	
663								
862	D1401						YES	30
902								
922								
963								
1023								
1026								
1146	D1401	>30		>30		>30	YES	30 min
1161								
1201								
1324								
1431	D1401	20.0		20.0			NO	
1435	D1401						YES	>60min
1456								
1604								
1720								
1827								
1943								
1955								
1957								
1966								
6016								
6034								
0004			Į.	1		I	ı	I .

Determination of Water Separability at 54°C on sample #16076; results in ml

lab	method	volume oil phase	volume water phase	volume emulsion phase	test OK/NOK	remark
178 179	D1401	36	6	38	NOK*	reported 30 min. to complete break, but did not report 40-40-0
214 237 325 331	D1401 D1401	0 36	0 16	80 28	NOK* OK	did not report any (abort) time reported to abort after 60 min., also reported 36-16-28 (60)
349 360 398 473 496	D1401 D1401	34 31 	12 0 	34 49 	OK NOK*	reported to abort after 30 min. did not report any (abort) time
541 550 603						
614 621	D1401	34	0	46 	NOK*	see below**
657 663	D1401	35	4	41	OK	
862 902 922 963	D1401	35 	11 	34	OK	reported to abort after 30 min., also reported 35-11-34 (30 min)
1023 1026 1146 1161 1201	D1401 D1401	40 32 	24 5 	16 43 	NOK* OK	did not report any (abort) time reported to abort after 30 min.
1324 1431 1435 1456 1604 1720	D1401	38	20	22 	NOK* OK	only reported time to reach 3 ml emulsion and 37 ml water reported to abort after 60 min
1827 1943 1955 1957						
1966 6016 6034						

^{*} according to ASTM D1401 the amount of ml oil, ml water and ml emulsion should be reported as well as either the time the different volume requirements are met or when these are not reached, the time when the test is aborted (usually 30 minutes for tests at 54°C and 60 minutes for tests at 82°C).

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^{**} lab 614: reported 30 min. to reach 3 ml emulsion, 30 min. to reach 37 ml water and 30 min. to reach complete break 40-40-0, but reported also 34 ml oil, 0 ml water and 46 ml emulsion

Determination of Level of contamination on sample #16076; results in parts per ml

lab	method	≥ 4 µm	mark	z(targ)	≥ 6 µm	mark	z(targ)	≥ 14 µm	mark	z(targ)	remarks
178	ISO4406	4007		-0.87	442		-1.78	24		-1.26	
179	D7647	5938		-0.09	613		-1.05	42		-0.64	
214											
237	ISO4406	4629.4		-0.62	454.1		-1.73	6.9		-1.84	
325	ISO4406	6585.60		0.17	876.67		0.08	49.13		-0.40	
331	ISO4406	9012.4	ex	1.14	1120.0	ex	1.13	83.1	ex	0.76	
349	ISO4406	13356	GX	2.89	1733	CX	3.76	80	CA	0.76	
					1321.2				C D(0.01)		
360	ISO4406	5339.2		-0.33			1.99	222.3	C,R(0.01)	5.50	
398	ISO4406	6378		0.09	650		-0.89	17		-1.49	
473											
496											
541											
550	ISO4406	21984	ex	6.36	9581	ex	37.49	1326	ex	43.11	
603											
614	ISO4406	13757		3.05	2591	R(0.01)	7.45	116		1.88	
621											
657	ISO4406	1363		-1.93	491		-1.57	62		0.04	
663	D7647	2175		-1.60	326		-2.28	19		-1.43	
862	ISO4406	5650		-0.21	871		0.06	34		-0.92	
902	ISO4406	2572.8		-1.44	870.4		0.06	116.3		1.89	
922	ISO4406	6884.7		0.29	759.8		-0.42	114		1.81	
963	ISO4406	1131	0.4	-2.02	542	OV	-1.35	67		0.21	
1023	ISO4406	8624	ex	0.99	1298	ex	1.89	74		0.45	
	1304400		0			0			0		
1026	10044500		С	4.50		С	4.70		С		
1146	ISO11500	2274.7		-1.56	442.1		-1.78	41.2		-0.67	
1161											
1201											
1324											
1431	ISO4406	9231	С	1.23	1363	С	2.17	50	С	-0.37	
1435	ISO4406	9449.77		1.32	1361.93		2.17	88.97		0.96	
1456	ISO4406	2759		-1.37	859		0.01	74		0.45	
1604	ISO4406	2209	ex	-1.59	699		-0.68	81		0.69	
1720											
1827											
1943											
1955						W			W		
1957						**			**		
1966											
6016											
6034											
		014			014			014			
	normality	OK			OK			OK			
	n	18			18			19			
	outliers	0	(+4 excl)		1	(+3 excl)		1	(2 excl)		
	mean (n)	6165.2			857.29			60.868			
	st.dev. (n)	3621.49			402.090			33.7884			
	R(calc.)	10140.2			1125.85			94.608			
	R(D7647:10)	6966.7			651.54			82.172			

Lab 1026 reported the ISO class results instead of the number of particles

Lab 1431: first reported resp. 923112, 136295 and 4978

Lab 1955: results withdrawn, used an old version of ISO4406 (results reported: 17532 for >5 μ m and 588 for >15 μ m)

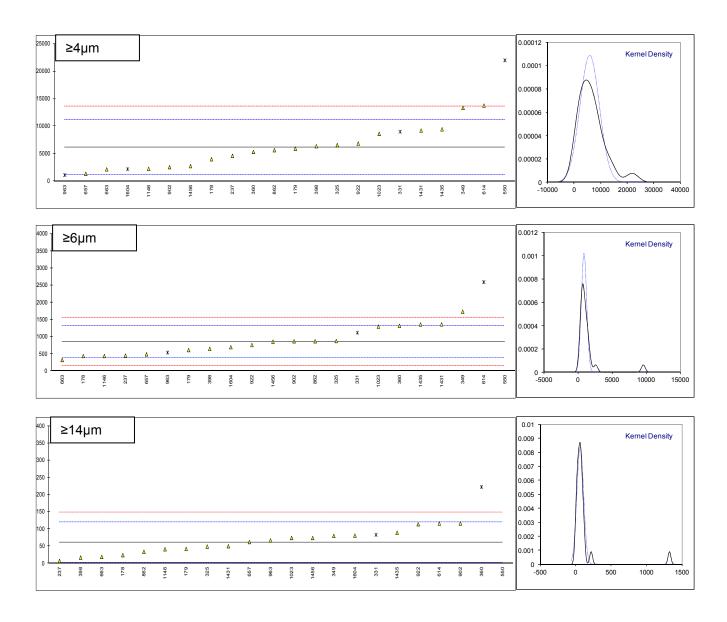
Lab 331: results excluded as parts/ml do not match ISO-classes

Lab 550: results excluded as parts/ml do not match ISO-classes

Lab 963: results excluded as parts/ml do not match ISO-classes

Lab 1604: result excluded as parts/ml do not match ISO-class

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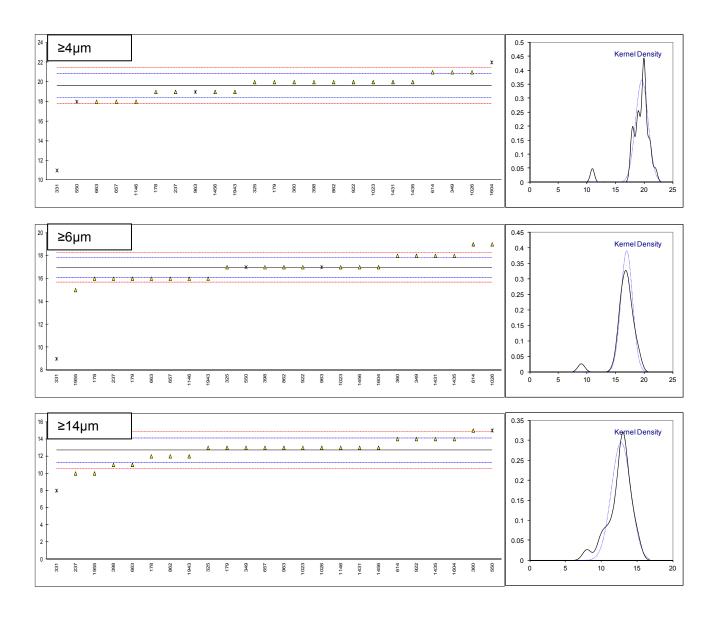
Determination of Level of contamination on sample #16076; results in ISO class

lab	method	≥ 4 µm	mark	z(targ)	≥ 6 µm	mark	z(targ)	≥ 14 µm	mark	z(targ)	remarks
178	ISO4406	19		-1.04	16		-2.22	12		-0.95	
179	D7647	20		0.61	16		-2.22	13		0.45	
214											
237	ISO4406	19		-1.04	16		-2.22	10		-3.75	
325	ISO4406	20		0.61	17		0.11	13		0.45	
331	ISO4406	11	E,ex	-14.22	9	E,ex	-18.56	8	E,ex	-6.55	
349	ISO4406	21	_,0//	2.25	18	_,0/.	2.44	13	_,0//	0.45	
360	ISO4406	20		0.61	18		2.44	15	С	3.25	
398	ISO4406	20		0.61	17		0.11	11	Ü	-2.35	
473	1001100										
496											
541											
550	ISO4406	18	E,ex	-2.69	17	E,ex	0.11	15	E,ex	3.25	
603	1304400		L,GX	-2.03		L,GX			L,GX		
614	ISO4406	21		2.25	19		4.78	14		1.85	
	1504406										
621	1004400	40			40			40		0.45	
657	ISO4406	18		-2.69	16		-2.22	13		0.45	
663	ISO4406	18		-2.69	16		-2.22	11		-2.35	
862	ISO4406	20		0.61	17		0.11	12		-0.95	
902	1004400				4					4.05	
922	ISO4406	20		0.61	17		0.11	14		1.85	
963	ISO4406	19	ex	-1.04	17	ex	0.11	13		0.45	
1023	ISO4406	20	_	0.61	17		0.11	13		0.45	
1026	ISO4406	21	С	2.25	19		4.78	13		0.45	
1146	ISO11500	18		-2.69	16		-2.22	13		0.45	
1161											
1201											
1324											
1431	ISO4406	20		0.61	18		2.44	13		0.45	
1435	ISO4406	20		0.61	18		2.44	14		1.85	
1456	ISO4406	19		-1.04	17		0.11	13		0.45	
1604	ISO4407	22	E,ex	3.90	17		0.11	14		1.85	
1720											
1827											
1943	ISO4406	19		-1.04	16		-2.22	12		-0.95	
1955					15		-4.56	10		-3.75	
1957											
1966											
6016											
6034											
	normality	OK			OK			OK			
	n	19			21			22			
	outliers	0	(+4 excl)		0	(+3 excl)		0	(+2 excl)		
	mean (n)	19.6	(10/10)		17.0	(.0 000)		12.7	(· Z CAOI)		
	st.dev. (n)	0.96			1.07			1.29			
	R(calc.)	2.7			3.0			3.6			
	R(D7647:10)	1.7			1.2			2.0			
	. ((5, 57, 10)	1.7			1.4			2.0			

Lab 360: first reported 16

Lab 331: results excluded as parts/ml do not match ISO-classes Lab 550: results excluded as parts/ml do not match ISO-classes Lab 963: results excluded as parts/ml do not match ISO-classes Lab 1604: result excluded as parts/ml do not match ISO-class

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APPENDIX 2

Number of participants per country

- 1 lab in ALGERIA
- 1 lab in ARGENTINA
- 2 labs in AUSTRALIA
- 2 labs in BELGIUM
- 1 lab in BRAZIL
- 1 lab in BULGARIA
- 2 labs in CHINA, People's Republic of
- 1 lab in FRANCE
- 1 lab in GEORGIA
- 1 lab in GERMANY
- 1 lab in GREECE
- 1 lab in INDONESIA
- 1 lab in ISRAEL
- 1 lab in ITALY
- 1 lab in KAZAKHSTAN
- 1 lab in LATVIA
- 3 labs in MALAYSIA
- 3 labs in NETHERLANDS
- 1 lab in NIGERIA
- 2 labs in NORWAY
- 1 lab in PAKISTAN
- 1 lab in SAUDI ARABIA
- 1 lab in SINGAPORE
- 2 labs in SPAIN
- 1 lab in SUDAN
- 1 lab in THAILAND
- 3 labs in TURKEY
- 2 labs in UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations:

C = final result after checking of first reported suspect result

 $\begin{array}{ll} D(0.01) &= \text{outlier in Dixon's outlier test} \\ D(0.05) &= \text{straggler in Dixon's outlier test} \\ G(0.01) &= \text{outlier in Grubbs' outlier test} \\ G(0.05) &= \text{straggler in Grubbs' outlier test} \\ DG(0.01) &= \text{outlier in Double Grubbs' outlier test} \\ \end{array}$

R(0.01) = outlier in Rosner's outlier test
R(0.05) = straggler in Rosner's outlier test
ex = excluded from calculations
U = reported in different unit

W = result withdrawn on request of the participants

= straggler in Double Grubbs' outlier test

fr. = first reported n.a. = not applicable n.e. = not evaluated

SDS = Material Safety Data Sheet

Literature:

DG(0.05)

1 iis Interlaboratory Studies, Protocol for the Organization, Statistics and Evaluation, April 2014

2 ASTM E178-89

- 3 ASTM E1301-89
- 4 ISO 5725-86
- 5 ISO 5725, parts 1-6, 1994
- 6 ISO13528-05
- 7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 9 IP 367/84
- 10 DIN 38402 T41/42
- 11 P.L. Davies, First reported Z. Anal. Chem, 331, 513, (1988)
- 12 J.N. Miller, Analyst, <u>118</u>, 455, (1993)
- 13 Analytical Methods Committee Technical brief, No4 January 2001.
- The Royal Society of Chemistry 2002, Analyst 2002, 127 pages 1359-1364, P.J. Lowthian and M. Thompson.
- Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)
- 16 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

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