VISCOL-10AS
AUTOMATIC KINEMATIC VISCOMETER
REPEATABILITY STUDY

1. Introduction

Viscosity measurements are important in many fields of both industry and research. Many manufacturers now regard viscometers as crucial part of their research, development, and quality control programs.

Repeatability, intermediate precision and reproducibilty are important characteristics to determine the performance of any analytical instrument.

Two sets of measurements presented in this study which were both performed in Biolab Application Laboratory with the same instrument by different operators in 2 seperate days to test the repeatability and intermediate precision performance of Viscol-10 Series Full Automatic Kinematic Viscometer.

This study, tests the repeatability and intermediate precision of Viscol-10 Series Full Automatic Kinematic Viscometer.

2. What is Viscosity?

Viscosity is defined as the rate of a fluid's internal resistance to the force that is required to flow. Intermolecular force, molecular mass and temperature of a fluid is considered as the three main factors effecting the viscosity. Fluids such as water, air, oil etc. that have directly proportional flow rate with friction resistance are called as Newtonian fluids.

"Kinematic viscosity is a measure of the resistive flow of a fluid under the influence of gravity."

Globally accepted and the most precise method to measure viscosity of Newtonian fluids is by using capillary viscometers. With capillary viscometers, viscosity is determined based on the flow time of a fluid, which is kept at a specific temperature inside a capillary with known diameter and length.

3. What is Repeatability?

Repeatability is the measurement variation with a single operator and single instrument on the same sample, over a short amount of time with all other variables held constant. Intermediate precision is a measure of variation with multiple operators under a defined set of conditions as same procedure, same instrument, same location over an extended period of time.

Repeatability according to ASTM D445 reference standard is defined as "The difference between successive results obtained by the same operator in the same laboratory with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of this test method, exceed the values indicated only in one case in twenty". Table 1 shows the repeatability values for different types of oils.

Base oils at 40°C[10]	0.0101 x	(1.01%)
Base oils at 100°C[10]	0.0085 x	(0.85%)
Formulated oils at 40°C[10]	0.0074 x	(0.74%)
Formulated oils at 100°C[10]	0.0084 x	(0.84%)
Formulated oils at 150°C[11]	0.0056 x	(0.56%)
Petroleum wax at 100°C[12]	0.0141 $x^{1.2}$	
Residual fuel oils at 80°C[13]	0.013 (x+8)	
Residual fuel oils at 100°C[13]	0.08088 x	(8.08%)
Residual oils at 50°C[13]	0.07885 x	(7.88%)
Additives at 100°C[14]	0.00192 $x^{1.1}$	
Gas oils at 40°C[15]	0.0043 (x+1)	
Jet fuels at -20°C[16]	0.007 x	(0.7%)
Kerosine, diesel fuels, biodiesel fuels and biodiesel fuel blends at 40°C[17]	0.0056 x	(0.56%)
Used (in-service) formulated oils at 40°C[18]	0.000233 x1.722	
Used (in-service) formulated oils at 100°C[18]	0.001005 x1.4633	

Table 1 - ASTM D445 Repeatability Table

4. Repeatability Study

The repeatability study was performed with 2 sets of 20 samples which were collected from the same bottle of Shell Helix HX5 15W-40 Engine Oil. Each set of 20 measurements were conducted by different operaters in two separate days with the analysis temperature of 40°C and the same viscometer tube. Samples were measured with same automatic cleaning cycles between the measurements with the same amount of solvent usage. Viscometer cleaning cycles include solvent insertion and drying of the capillary viscometer for around 300 seconds in total.

As described in ASTM D445/446, each sample was measured 2 times and the average of this 2 measurements stated as the sample's viscosity.

5. Instrument Details

Instrument: VISCOL-10AS Automatic Kinematic Viscometer
Autosampler: 23 samples
Methodology: ASTM D445/D446
Analysis Temperature: 40°C
Temperature Stability: 0.01°C
Temperature Sensitivity: 0.001°C
Viscometer Type: Ubbelohde
Viscometer Tube Coefficient: 0.28720
Sample Volume: 12 ml
Cleaning Solvent: Hexane
Cleaning Duration: 300 seconds

Picture 1 - Viscol-10AS Automatic Kinematic Viscometer

6. Results

6. 1. Repeatability Study 1 - Day 1

20 samples were prepared to the glass sample cups each around 12 ml. The viscometer with a coefficient of 0.2872 is selected to keep flow time more than 200 seconds to avoid the necessity of applying a kinetic energy correction as noted in ASTM D445/D446 specifications. Analysis of 20 samples including cleaning and drying cycles in between was concluded within 6 hours.

#	Flow Time 1	Viscosity 1	Flow Time 2	Viscosity 2	Viscosity Average
1	383.30	110.08	384.17	110.33	110.21
2	384.22	110.35	383.95	110.27	110.31
3	384.20	110.34	383.82	110.23	110.29
4	383.70	110.20	383.15	110.04	110.12
5	383.95	110.27	382.85	109.95	110.11
6	384.49	110.43	385.15	110.61	110.52
7	384.34	110.38	383.89	110.25	110.32
8	384.65	110.47	384.47	110.42	110.45
9	383.97	110.28	383.89	110.25	110.27
10	383.47	110.13	382.70	109.91	110.02
11	383.47	110.13	382.77	109.93	110.03
12	383.91	110.26	383.80	110.23	110.24
13	384.67	110.48	384.22	110.35	110.41
14	384.37	110.39	384.12	110.32	110.36
15	383.75	110.21	383.25	110.07	110.14
16	384.42	110.41	383.92	110.26	110.33
17	383.95	110.27	383.40	110.11	110.19
18	383.30	110.08	383.17	110.05	110.07
19	383.47	110.13	383.57	110.16	110.15
20	384.50	110.43	383.57	110.16	110.30
* Flow times are in seconds (s)				**AVERAGE (cSt)**	**110.24**
				STD DEV	**0.138**
				%RSD	**0.13**

Table 2 - Repeatability Study 1 Results

Repeatability Study 1 - Summary

To study the performance of the instrument each sample was measured two times and the average of these two measurements are stated as the kinematic viscosity.

Table 2 shows the gathered flow times and kinematic viscosity values from each measurement and the final (average) viscosity value for each sample.

The result of first 20 measurements was an average of 110.24 cSt. The standard deviation value is 0.138 and the relative standard deviation (%RSD) is %0.13.

6.2. Repeatability Study 2 - Day 2

20 samples were measured applying the same procedure of Repeatability Study 1 with same instrument, same capillary and the same sample by different operator in the same laboratory.

#	Flow Time 1	Viscosity 1	Flow Time 2	Viscosity 2	Viscosity Average
1	382.90	109.97	383.52	110.15	110.06
2	383.99	110.28	383.99	110.28	110.28
3	384.27	110.36	383.92	110.26	110.31
4	384.10	110.31	383.92	110.26	110.29
5	384.27	110.36	384.07	110.30	110.33
6	384.55	110.44	384.05	110.30	110.37
7	384.47	110.42	384.07	110.30	110.36
8	383.92	110.26	383.92	110.26	110.26
9	384.82	110.52	385.42	110.69	110.61
10	384.02	110.29	383.60	110.17	110.23
11	384.05	110.30	383.70	110.20	110.25
12	384.15	110.33	383.80	110.23	110.28
13	384.65	110.47	383.89	110.25	110.36
14	384.34	110.38	384.15	110.33	110.36
15	383.99	110.28	384.07	110.30	110.29
16	383.89	110.25	383.60	110.17	110.21
17	384.52	110.43	383.52	110.15	110.29
18	384.34	110.38	384.07	110.30	110.34
19	384.60	110.46	383.97	110.28	110.37
20	384.55	110.44	384.40	110.40	110.42
* Flow times are in seconds (s)				**AVERAGE (cSt)**	**110.31**
				STD DEV	**0.10**
				%RSD	**0.09**

Table 3 - Repeatability Study 2 Results

Repeatability Study 2 – Summary

To study the performance of the instrument each sample was measured two times and the average of these two measurements are stated as the kinematic viscosity.

Table 2 shows the gathered flow times and kinematic viscosity values from each measurement and the final (average) viscosity value for each sample.

The result of first 20 measurements was an average of 110.31 cSt. The standard deviation value is 0.10 and the relative standard deviation (%RSD) is %0.09.

6.3. Intermediate Precision Calculation

Intermediate precision as part of precision in general gives us an indication of the degree of scatter of the results due to underlying random errors. Therefore, intermediate precision can be expressed by the relative standard deviation. Considering more variable conditions than applied for repeatability experiments, it is easily understandable that the relative standard deviation calculated for intermediate precision experiments is usually larger than the one observed for the repeatability experiments.

From the table it can be seen that the mean values for 2 sets of 20 measurements are quite similar and the variation of the results is slightly higher for analyst 1, which might be due to his operation.

	MEAN	SD	%RSD
Day 1 - Analyst 1	110.24	0.138	0.13
Day 2 - Analyst 2	110.31	0.10	0.09
Intermediate Precision	**110.275**	**0.049**	**0.045**

Table 4 - Intermediate Precision Calculation

Conclusions

This study mimics the real-world conditions for kinematic viscosity measurements according to ASTM D445/D446 reference standard. This data is crucial for Viscol-10 Series Viscometer users to get a better insight of their measurements precision.

As shown in Table 1, ASTM D445 mentiones different repeatability values for different type of oils. The lowest deviation is stated for kerosine samples which is %0.56. The studies with Viscol-10 Series Viscometer on 2 sets of 20 samples of formulated oil evaluated %RSD values are 0.13% and 0.09% and the intermediate precision deviation is %0.045. These deviation values exceeds even the lowest requirements of ASTM D445 reference standard.

As a result, this study proves that Viscol- 10AS Automatic Kinematic Viscometer exceeds the repeatability requirements of ASTM D445 standard which the repeatability of formulated oils at 40°C is defined as 0.74%.

References

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