Designation: D 6895 – 03

An American National Standard

Standard Test Method for
Rotational Viscosity of Heavy Duty Diesel Drain Oils at 100°C[1]

This standard is issued under the fixed designation D 6895; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the rotational viscosity and the shear thinning properties of heavy duty diesel engine drain oils at 100°C, in the shear rate range of approximately 10 to 300 s^{-1}, in the shear stress range of approximately 0.1 to 10 Pa and the viscosity range of approximately 12 to 35 mPa·s. Rotational viscosity values can be compared at a shear rate of 100 s^{-1} by this test method.[2,3]

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[4]

D 5967 Test Method for Evaluation of Diesel Engine Oils in T-8 Diesel Engine[5]

D 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance[5]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *rate index*—the exponent, *c*, in these expressions relating shear rate and shear stress:

$$\text{shear stress} = b(\text{shear rate})^c \qquad (1)$$

$$\ln(\text{shear stress}) = \ln b + c \ln(\text{shear rate}) \qquad (2)$$

3.1.1.1 *Discussion*—A rate index of $c = 1$ signifies Newtonian fluid behavior. Values less than one indicate increasing non-Newtonian, shear thinning behavior.[3]

3.1.2 *rotational viscosity*—the viscosity obtained by use of this test method.

3.1.3 *VIS100 DEC*—rotational viscosity at shear rate of 100 s^{-1}, decreasing shear stress or shear rate sweep.

3.1.4 *VIS100 INC*—rotational viscosity at shear rate of 100 s^{-1}, increasing shear stress or shear rate sweep.

4. Summary of Test Method

4.1 The sample is placed in a controlled stress or controlled shear rate rheometer/viscometer at 100°C. The sample is presheared at 10 s^{-1} for 30 s followed by heating at 100°C for 10 min. An increasing shear rate (approximately 10 to 300 s^{-1}) or shear stress (0.1 to 10 Pa) sweep is run followed by a decreasing sweep. The rotational viscosity for each step (increasing and decreasing) at 100 s^{-1} shear rate is interpolated from the viscosity versus shear rate data table. The rate index, as a measure of shear thinning, is calculated from a plot of ln (shear stress) versus ln (shear rate).

5. Significance and Use

5.1 Rotational viscosity measurements allow the determination of the non-Newtonian, shear thinning property of drain oil. Rotational viscosity values can be compared at a shear rate of 100 s^{-1} by this test method.[2,3]

6. Apparatus

6.1 This test method uses rheometers/viscometers of the controlled stress or controlled rate mode of operation. The test method requires the use of cone and plate or concentric cylinder measuring geometries capable of operating in the range of approximately 0.1 to 10 Pa for shear stress and 10 to 300 s^{-1} for shear rate. Instrument data logging or software shall be capable of delivering shear stress versus shear rate data and viscosity versus shear rate data in tabular form. Temperature shall be controlled to 100 ± 0.2°C at equilibrium. Some

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

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[2] Selby, K., "Rheology of Soot–thickened Diesel Engine Oils," *SAE 981369*, May 1998.

[3] George, H. F., Bardasz, E. A., and Soukup, B., "Understanding SMOT through Designed Experimentation Part 3: An Improved approach to Drain Oil Viscosity Measurements—Rotational Rheology," *SAE 97692*, May 1997.

[4] *Annual Book of ASTM Standards*, Vol 05.02.

[5] *Annual Book of ASTM Standards*, Vol 05.03.

rheometers have a 99.9°C set point limit and would be acceptable for this test method.

7. Reagents and Materials

7.1 *Standard Newtonian Reference Oil*, calibrated in viscosity in the range of 12 to 35 mPa·s at 100°C.

8. Sampling, Test Specimens, and Test Units

8.1 Ensure the test specimen is homogeneous. Engine sampling is generally specified in the test method, for example, Test Method D 5967. Manual sampling from the container can be done in accordance with Practice D 4057.

9. Preparation of Apparatus

9.1 Prepare the apparatus in accordance with manufacturers directions. The apparatus shall be capable of viscosity measurement to within 5 % of the standard Newtonian reference oil viscosity and a rate index value of 0.98 to 1.02 indicating a Newtonian fluid.

10. Calibration and Standardization

10.1 A Newtonian viscosity standard in the range 12 to 35 mPa·s at 100°C shall be used to verify instrument calibration. Run the procedure as in Section 12. A plot of shear stress (Pa) versus shear rate (s^{-1}) shall be linearly regressed to yield a slope and intercept. Results shall be:

Intercept, < 0.1 Pa
Slope = viscosity value within 5 % of certified value, mPa·s
Correlation coefficient, r^2 > 0.9998
This calibration procedure should be repeated if any
   criteria are not met
The instrument manufacturer should be contacted if the
   criteria cannot be met
The operator shall not proceed with this procedure if the
   calibration criteria are not met

10.2 New SAE 15W-40 oil shall be used as a daily control chart standard. Run the procedure (see Section 12) and perform an analysis (see Section 13). Results shall be a rate index between 0.98 to 1.02 and a viscosity value at 100 s^{-1}, mPa·s, VIS100. Control chart the values of VIS100 and rate index. The procedure shall be checked and the instrument calibration rechecked if the reference oil does not fall within control limits. Practice D 6299 shall be used as a guide in this area.

10.3 Some instruments and geometries will exhibit significant instrument/electronic noise at low shear stress or low shear rate levels, or both. This may be determined by plotting viscosity versus shear rate or viscosity versus shear stress for measurements of the standard oil. A horizontal line is obtained in regions far from noise. Annex A1 shows two examples of this type of plot. The minimum shear rate or shear stress to use in the analysis of data can then be determined for the particular instrument and geometry.

11. Conditioning

11.1 Shake all new and used oil samples using the following procedure. Do not prepare more than two samples at one time for one instrument.

11.2 Ensure cap is tight on container.

11.3 Shake vigorously by hand for 30 s. Wait 60 s for air bubbles to dissipate.

11.4 A specimen of the sample shall be taken for analysis promptly following the shaking and dissipation procedure of 11.3.

12. Procedure

12.1 Run the procedure in accordance with the instrument geometry requirements and the manufacturers' recommendations to obtain shear stress versus shear rate data in the ranges of 0.1 to 10 Pa and 10 to 300 s^{-1}. The order of steps is as follows:

12.1.1 Load sample.

12.1.2 Equilibrate at 100°C (maximum 10 min).

12.1.3 Preshear sample at 10 s^{-1} for 30 s.

12.1.4 Stop preshear.

12.1.5 Preheat sample at 100°C for 10 min.

12.1.6 Run increasing stress or rate sweep for duration of approximately 10 min to generate data of shear stress, shear rate and viscosity followed immediately by the next step. The run time will vary somewhat among different instruments and procedures. Times as low as 2 min and as high as 20 min have been utilized successfully to run this test method.

12.1.7 Run decreasing stress or rate sweep for duration of approximately 10 min to generate data of shear stress, shear rate, and viscosity.

12.1.8 Clean sample from instrument in accordance with the manufacturers' instructions. Cone and plate systems may be cleaned by wiping with a rag followed by an appropriate solvent.

13. Calculation or Interpretation of Results

13.1 Analyze the increasing and decreasing sweeps separately.

13.2 Import the shear stress (Pa), shear rate (s^{-1}) and viscosity (mPa·s) into a spreadsheet program. This calculation may be done with a calculator. Delete data below the noise limit as determined in 10.3.

13.3 Calculate two additional columns for ln (shear rate) and ln (shear stress). See Appendix X1 for sample calculation.

13.4 Plot the ln stress versus ln rate columns as a scatter plot with ln rate on the *x*-axis.

13.5 Fit a least squares linear regression to the data plot. Obtain the equation of the line. This line follows Eq 2. There is no criterion for correlation coefficient of candidate oils. See Appendix X1 for sample calculation.

13.6 Obtain the slope of the line, *c* which is the rate index, to three decimal places and the intercept as ln *b* to four decimal places.

13.7 Calculate the viscosity at 100 s^{-1} by interpolation between two data points spanning 100 s^{-1} as follows:

$$VIS100 = VIS1 + D(100 - T1) \qquad (3)$$

where:
D = ((VIS2 − VIS1)/(T2 − T1)),
VIS2 and *T2* = viscosity and shear rate respectively at the first data point above 100 s^{-1}, and
VIS1 and T1 = viscosity and shear rate respectively at the first data point below 100 s^{-1}.