INTERNATIONAL

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

This standard is issued under the fixed designation D6895; 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 s^{-1} to 300 s^{-1}, in the shear stress range of approximately 0.1 Pa to 10 Pa and the viscosity range of approximately 12 mPa·s 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.3 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[4]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D5967 Test Method for Evaluation of Diesel Engine Oils in T-8 Diesel Engine

D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

3. Terminology

3.1 *Definitions:*

3.1.1 *shear rate, n*—the velocity gradient perpendicular to the direction of flow.

3.1.1.1 *Discussion*—For a Newtonian fluid in a concentric cylinder rotary viscometer in which the shear stress is measured at the inner or outer cylinder surface and ignoring any end effects, the shear rate is given as follows:

$$\dot{\gamma} = \frac{2\Omega R_o^2}{R_o^2 - R_i^2} \quad (1)$$

$$= \frac{4\pi R_o^2}{t(R_o^2 - R_i^2)}$$

where:

$\dot{\gamma}$ = shear rate at the surface of the rotor in reciprocal seconds, s^{-1},
Ω = angular velocity, rad/s,
R_o = outer radius, mm,
R_i = inner radius, mm, and
t = time for one revolution of the rotor, s.

3.1.1.2 *Discussion*—For a fluid in a cone and plate viscometer in which the shear stress is measured in a controlled-stress or controlled strain mode of operation, the shear rate is given as follows:

$$\dot{\gamma} = \frac{\Omega}{B} \quad (2)$$

where:

$\dot{\gamma}$ = shear rate at the surface of the rotor or stator in reciprocal seconds, s^{-1},
Ω = angular velocity, rad/s,
B = cone angle, rad.

3.1.2 *shear stress, n*—the force per unit area in the direction of the flow.

3.1.2.1 *Discussion*—For a Newtonian fluid in a concentric cylinder rotary viscometer in which the shear stress is measured at the inner or outer cylinder surface and ignoring any end effects, the shear stress is given as follows:

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, 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] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

*A Summary of Changes section appears at the end of this standard

$$\sigma = \frac{T_r}{2\,\pi\,R_i^2\,h} \quad (3)$$

where:

σ = shear stress at the surface of the rotor or stator, Pa,
T_r = torque applied to the moving fixture, N·m,
R_i = inner radius, m, and
h = height of the rotor, m

3.1.2.2 *Discussion*—For a fluid in a cone and plate viscometer in which the shear stress is measured in a controlled-stress or controlled-strain mode of operation, the shear stress is given as follows:

$$\sigma = \frac{3T_r}{2\pi R^3} \quad (4)$$

where:

σ = shear stress at the surface of the rotor or stator, Pa,
T_r = torque applied to the moving fixture, N·m, and
R = radius of the cone.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *maximum point time, n*—instrument setting that limits the amount of time the instrument will maintain a constant shear stress or shear rate before accepting the value as the equilibrium value.

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

$$\sigma = b\dot{\gamma}^c \quad (5)$$

$$\ln(\sigma) = \ln(b) + c\ \ln(\dot{\gamma}) \quad (6)$$

where:

c = rate index, and
b = viscosity coefficient, mPa·s.

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

3.2.3 *rotational viscosity, n*—the viscosity obtained by use of this test method.

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

3.2.5 *VIS100 INC, n*—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 s^{-1} to 300 s^{-1}) or shear stress (0.1 Pa 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 concentric cylinder measuring geometry or cone and plate measuring geometries, with a minimum cone diameter of 50 mm, capable of operating in the range of approximately 0.1 Pa to 10 Pa for shear stress and 10 s^{-1} to 300 s^{-1} for shear rate.

6.2 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. During the experiment, a minimum of 20 points must be taken. The method for data logging shall be an equilibrium method where the controlled stress or controlled rate value is held constant until the data point equilibrium is reached. The use of a maximum point time is acceptable, but it must be set to at least 30 s.

6.3 Temperature shall be controlled to 100 °C ± 0.2 °C at equilibrium. Some 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 mPa·s 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 D5967. Manual sampling from the container can be done in accordance with Practice D4057.

9. Preparation of Apparatus

9.1 Prepare the apparatus in accordance with manufacturer's 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 mPa·s 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

NOTE 1—It has been determined that use of a specific reference oil in the aforementioned viscosity range did not improve the precision. For laboratory to laboratory consistency, it is suggested to use Cannon S200 as the standard calibration fluid.[5]

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 D6299 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 Pa to 10 Pa and 10 s^{-1} to 300 s^{-1}. The order of steps is as follows:

12.1.1 Load sample.

12.1.2 Equilibrate at 100 °C (minimum 5 min, 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 shall be rinsed with a suitable solvent followed by wiping with a rag or towel.

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 6. 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:

$$\mathrm{VIS100} = \mathrm{VIS1} + D\,(100 - \mathrm{T1}) \tag{7}$$

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}.

14. Report

14.1 Report the rate index value as *c*, the viscosity as VIS100 and intercept as ln *b* . Label increasing and decreasing sweep data using INC and DEC.

14.2 Rate index *c* = dimensionless, three decimal places, x.xxx.

14.3 VIS100 = mPa·s, two decimal places xx.xx.

14.4 ln *b* = Pa, four decimal places, x.xxxx.

15. Precision[6,7]

15.1 Precision was found to be dependent on the mean value of the measured property. The data in this section was derived

[5] The sole source of supply of the calibration fluid known to the committee at this time is Cannon Instrument Co., P.O. Box 16, State College, PA 16804. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1594. Contact ASTM Customer Service at service@astm.org.

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1607. Contact ASTM Customer Service at service@astm.org.

from a six laboratory/six sample interlaboratory study and a three laboratory/seven sample interlaboratory study. All data was pooled to generate the repeatability and reproducibility data. VIS100 range was 14.99 mPa·s to 36.28 mPa·s and rate index range was 0.623 to 1.002.

	r	R
Rate index, INC	0.140 – 0.128x	0.592 – 0.552x
Rate index, DEC	0.050 – 0.048x	0.490 – 0.480x
VIS100, INC	0.12 + 0.0089y	-2.08 + 0.168y
VIS100, DEC	0.33 + 0.0066y	-0.54 + 0.073y

where:

x = mean value of rate index, and
y = mean value of VIS100 in mPa·s.

15.2 *Sample Precision Calculations:*

15.2.1 The maximum value for rate index is 1.000. This occurs for fluids which exhibit Newtonian behavior as observed for fresh diesel oils within the shear rate range of measurement in this test method. The rate index, DEC reproducibility for fresh oils would be:

$$0.490 - 0.480\,(1.000) = 0.010$$

and for a drain with a value of 0.900:

$$0.490 - 0.480\,(0.900) = 0.058$$

15.2.2 The VIS100 precision values are valid for viscosities within the scope of this test method, 12 mPa·s to 35 mPa·s. A drain with a VIS100, DEC value of 15.20 mPa·s would have a repeatability of:

$$0.33 + 0.0066\,(15.2) = 0.430$$

and a reproducibility of:

$$-0.54 + 0.073\,(15.2) = 0.570$$

16. Keywords

16.1 drain oils; heavy duty diesel; rotational viscosities; shear thinning

ANNEX

(Mandatory Information)

A1. TYPICAL EXAMPLE TO DETERMINE NOISE LEVEL AND MINIMUM SHEAR RATE VALUE

A1.1 The data are plotted as shown and the minimum noise level determined in the low shear region. Data below this level are deleted for analysis. The examples in Fig. A1.1 and Fig. A1.2 are from two different cone and plate systems.

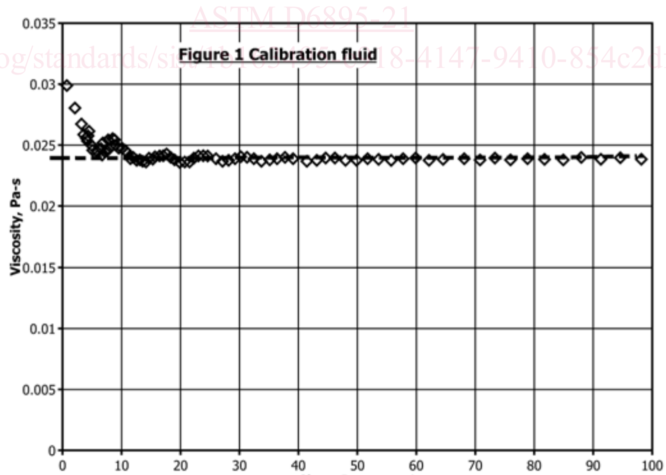

FIG. A1.1 Example 1