



Designation: D6895 – 21

Standard Test Method for Rotational Viscosity of Heavy Duty Diesel Drain Oils at 100 °C¹

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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

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.

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:

*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