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Standard Test Method for Measuring Viscosity at High Shear Rate by Tapered Bearing Simulator Viscometer at 100°C100 °C1

This standard is issued under the fixed designation D6616; 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-Scope*

1.1 This test method covers the laboratory determination of the viscosity of engine oils at $\frac{100^{\circ}\text{C}}{100}$ °C and $1 \cdot 10^{6}\text{s}^{-1}$ using the Tapered Bearing Simulator (TBS) viscometer.²

Note 1—This test method is similar to Test Method D4683 which uses the same TBS viscometer to measure high shear viscosity at 150°C.150°C.

- 1.2 The Newtonian calibration oils used to establish this test method range from approximately 5 to 12 mPa·s 5 mPa·s (cP) to 12 mPa·s (cP) at 100°C100 °C and either the manual or automated protocol was used by each participant in developing the precision statement. The viscosity range of the test method at this temperature is from 1 mPa·s (cP) to above 25 mPa·s 25 mPa·s (cP), depending on the model of TBS.
- 1.3 The non-Newtonian reference oil used to establish the shear rate of 1.10^6 s⁻¹ for this test method has a viscosity of approximately 10 mPa·s 10 mPa·s at 100°C.100 °C.
- 1.4 Application to petroleum products other than engine oil has not been determined in preparing the viscometric information for this test method.
- 1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard. This test method uses the milliPascal second (mPa·s) as the unit of viscosity. This unit is equivalent to the centiPoise (cP), which is shown in parentheses.
- 1.6 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 to determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:³

D4683 Test Method for Measuring Viscosity of New and Used Engine Oils at High Shear Rate and High Temperature by Tapered Bearing Simulator Viscometer at 150 °C

D4741 Test Method for Measuring Viscosity at High Temperature and High Shear Rate by Tapered-Plug Viscometer 2.2 Coordinating European Council (CEC) Standard: 4,5

CEC L-36-90 The Measurement of Lubricant Dynamic Viscosity under Conditions of High Shear

2.3 Energy Institute Standard:^{6,5}

IP 370 Test Method for the Measurement of Lubricant Dynamic Viscosity Under Conditions of High Shear Using the Ravenfield Viscometer

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

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² Available from Tannas Co., 4800 James Savage Rd., Midland, MI 48642. This viscometer and associated equipment as listed in the research report was used to develop the precision statement. To date, no other equipment has demonstrated, through ASTM International interlaboratory testing, the ability to meet the precision of this test. This is not an endorsement or certification by ASTM International.

³ 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.

Available from Coordinating European Council (CEC), Services provided by Kellen Europe, Avenue Jules Bordet 142 - 1140, Brussels, Belgium, http://www.cectests.org.

⁵ This test equipment is identical to that described in CEC L-36-90 (under the jurisdiction of the CEC Engine Lubricants Technical Committee) and IP 370 references CEC

⁶ Available from Energy Institute, 61 New Cavendish St., London, W1G 7AR, U.K., http://www.energyinst.org.

3. Terminology

- 3.1 Definitions:
- 3.1.1 *density, n*—mass per unit volume. In the SI, the unit of density is the kilogram per cubic metre. For practical use, the submultiple, gram per cubic centimetre, is more convenient. The density in gram per cubic centimetre is equal to 1/1000 the density in kg/m³.
- 3.1.2 *Newtonian oil or fluid*, *n*—an oil or fluid that at a given temperature exhibits a constant viscosity at all shear rates or shear stresses.
 - 3.1.3 non-Newtonian oil or fluid, n—an oil or fluid that exhibits a viscosity that varies with changing shear stress or shear rate.
 - 3.1.4 shear rate, n—the velocity gradient in fluid flow. The SI unit for shear rate is s^{-1} .
- 3.1.5 *shear stress*, *n*—the motivating force per unit area for fluid flow. The *area* is the area under shear. The SI unit for shear stress is the Pa.
- 3.1.6 *viscosity*, *n*—the ratio between the applied shear stress and the rate of shear. It is sometimes called the coefficient of dynamic viscosity. This coefficient is a measure of the resistance to flow of the liquid. In the SI, the unit of viscosity is the Pascal-second; often the milliPascal-second or its equivalent the centiPoise is found more convenient.
- 3.1.6.1 apparent viscosity, n—the viscosity of a non-Newtonian fluid at a given shear rate or shear stress determined by this test method.
 - 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 *idling oil*², n— an oxidatively stable Newtonian oil injected into the operating viscometer stator when the instrument is likely to be held for periods of time greater than 30 min 30 min and up to two weeks at 100°C.100°C. Use of this oil prevents stator deposits from additives, which may decompose after longer exposure times in the operating viscometer and permits continuous operation of the viscometer without need to shut the instrument off.
- 3.2.2 Newtonian Reference Oil^2 , n—a specially blended Newtonian oil that has the same viscosity at $\frac{100^{\circ}\text{C}}{100^{\circ}\text{C}}$ as the non-Newtonian reference oil of 3.2.3.
- 3.2.3 non-Newtonian reference oil², n—a specially formulated non-Newtonian oil, identified as NNR-10, having a selected apparent viscosity at $1 \cdot 10^6 \text{s}^{-1}$ shear rate. The oil is used to establish an operating gap between the rotor and stator which will produce $1 \cdot 10^6 \text{s}^{-1}$ shear rate when the rotor height is adjusted to give a torque output equivalent to that of the special reference oil described in 3.2.2.
- 3.2.4 reciprocal torque intersection, $1/T_i$, n—the rotor position on the micrometer defined by the intersection of two straight lines generated by the reciprocal torque method using the Newtonian reference oil of 3.2.2 and non-Newtonian reference oil of 3.2.3. Reciprocal torque versus rotor height measurements on both oils gives straight lines whose intersection, $1/T_i$, establishes the desired rotor position for operation at $1 \cdot 10^6 \text{ s}^{-1}$ shear rate.
- 3.2.5 reference Newtonian calibration oils², n—specially chosen Newtonian oils used to determine the viscosity-torque relationship of the TBS viscometer at 100°C100 °C from which the viscosity of an unknown oil is calculated.
- 3.2.6 rotor height (rotor position), n— the vertical position of the rotor relative to the stator and measured by the platform micrometer.
- 3.2.6.1 *stored rotor height (rotor position), n*—the rotor position with the rotor $0.50 \text{ mm} \ 0.50 \text{ mm}$ above the rubbing contact position (see 3.2.7) when the instrument is shut down.
- 3.2.7 rubbing contact position, n—the rotor height determined when the tapered rotor is lightly brought into contact with the similarly tapered stator.
 - 3.2.8 test oil, n—any oil for which the apparent viscosity is to be determined by this test method.

4. Summary of Test Method

4.1 A motor drives a tapered rotor closely fitted inside a matched tapered stator. Appropriate technique establishes operation of the viscometer to yield $1\cdot10^6 \text{s}^{-1}$ at a temperature of 100°C at which point test oils are introduced into the gap between the spinning rotor and stationary stator. The rotor exhibits a reactive torque to the viscous resistance of each test oil and the value of this torque response is used to determine the apparent viscosity of the test oil at 100°C .

5. Significance and Use

- 5.1 Viscosity at the shear rate and temperature of this test method is thought to be particularly representative of bearing conditions in large medium speed reciprocating engines as well as automotive and heavy duty engines operating in this temperature regime.
 - 5.2 The importance of viscosity under these conditions has been stressed in railroad specifications.



5.3 For other industry needs this method may also be run at 80 °C by using different crossover calibration oils available from the manufacturer. No precision has been determined at this temperature. The equipment is also used at higher temperatures as shown in Test Method D4683 and CEC L-36-90 (also referenced from IP 370).

6. Apparatus

- 6.1 Tapered Bearing Simulator Viscometers² (TBS)—a viscometer consisting of a motor connected to a slightly tapered rotor that fits into a matched stator. Several models of the TBS are in use. All of these are capable of analyzing test oils at 100°C but earlier models are more limited in their upper viscosity range.
- 6.2 Different models of the tapered bearing simulator (TBS) have the following upper levels of operating viscosities at $1 \cdot 10^6 \text{s}^{-1}$ shear rate:
 - 6.2.1 Model Series 400 (similar to Fig. 1)—~14 mPa·s (cP), dual speed.
 - 6.2.2 Model Series 500 (Fig. 1)— \sim 16 mPa·s (cP) single speed.
 - 6.2.3 Model Series 600 (Fig. 2)—~100 mPa·s (cP) (usually liquid cooled), dual speed.
 - 6.2.4 Model Series SS (SuperShear) (similar to Fig. 1)—~20 mPa·s (cP), multi-speed.
 - 6.2.5 Model Series 2100 E (Fig. 3)—~20 mPa·s (cP) (see Note 2), multi-speed.

Note 2—TBS Models 500, 600, and SS use a so-called *bouncer* to automate unloading and reloading the load cell just before taking a torque reading. (All automated units apply the bouncer at the appropriate point of operation as part of their program.) If a bouncer is not on the TBS model used (Model 400), the effect is generated by placing the thumb on the brass weight pin and turning the turntable slightly in a clockwise direction and quickly releasing the turntable. The bearingless Models 2100 E do not require unloading the cell since there is no turntable bearing.

- 6.3 Automated System for Calibration, Injection, and Data Analysis Programs—An automated program for the Tapered Bearing Simulator, simulating the manual method has been used.
- 6.4 *Console*—The console shown in Fig. 4 is similar in Models 400, 500, and 600. Consoles for Series SS and 2100 E have provisions for changing motor speed. All consoles contain the power source for the load cell, thermoregulator circuit, stator-heating element, and motor. They also contain the circuitry for regulating and monitoring the temperature of the oil in the stator as well as the amplifier and digital readout of the load cell.

Note 3—The thermoregulator circuit of the TBS viscometers has evolved as improvements have been made in the solid-state temperature controller and heater. To achieve the 5 min 5 min analysis time specified in this test method requires a late model solid-state controller with automatic reset coupled to a thermo-foil stator heater with small heat inertia or a fast-responding thermoregulated liquid bath.²

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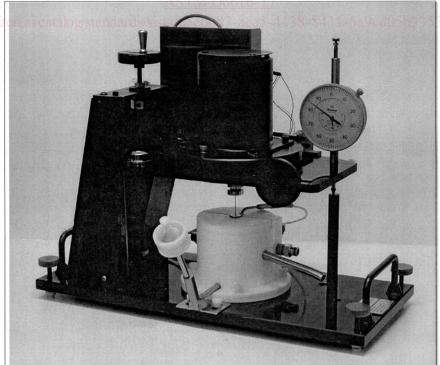


FIG. 1 Tapered Bearing Simulator Viscometer Model 500

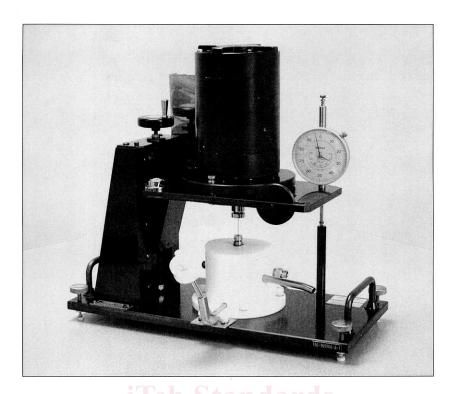


FIG. 2 High Torque Tapered Bearing Simulator Viscometer Model 600

- 6.5 Cooling Systems— Two cooling systems are available for TBS viscometer work at 100°C: forced air cooling and liquid bath cooling. The stator housing is prepared for the former but must be modified for the latter according to directions from the manufacturer.
- 6.6 Glass Syringe— A 50-mL 50 mL glass syringe equipped with a Luer needle lock fits the tip of the filling tube for injection of test oil into the test cell. Smaller glass and plastic syringes can be used if any air bubble in the fill tube caused by the exchange of syringes is first pulled up into the next syringe to be used.
- 6.7 Filter Assembly— A unit made of a filter holder² and nominal $\frac{10-\mu 10 \mu}{10 \mu}$ filter² is interposed between the syringe and the filling tube to remove particles capable of damaging the rotor/stator cell.
- 6.8 Data Recording Equipment—Some form of recording the torque and temperature data produced by the tapered bearing simulator is desired in order to (1) determine torque/temperature equilibrium and (2) determine the torque with sufficient precision to calculate viscosity to the second decimal place. Early in the use of the TBS viscometer, a strip-chart recorder was used, later an automated, computer-based recording system was developed with both a computer-simulated strip chart and with data digitally recorded.

Note 4—Although the console has a torque indicator that can be used for determining viscosity, it has been found that the small oscillatory variation of torque with time makes desirable the recording and analysis of the torque output more precise, particularly when determining torque equilibrium.

- 6.8.1 Strip-chart Recorder:
- 6.8.1.1 If a strip-chart recorder is used to record the torque and temperature output signals, use the manufacturer's directions for calibrating and setting up the strip chart for recording torque/temperature data (see Note 5). The torque reading must be in milliVolts and the temperature in °C with a full-scale chart range of 20°20 °C to 120°C:120 °C.
 - 6.8.1.2 Use a chart speed of 11 cm -cm/min/min for recording.
- 6.8.1.3 Set and, when necessary, reset, the strip chart torque voltage to that which will permit recording the torque as much as possible on the upper two-thirds of the chart paper for maximum sensitivity.
 - 6.8.1.4 Factor the resulting voltage values to calculate the correct values of torque.
- Note 5—Although the digital information from the torque output meter on the viscometer console can be, and is, used for recording additional test information, it is desirable to use a two-pen, strip-chart recorder or its computer equivalent since this provides a continuous torque/temperature record of torque/temperature equilibrium necessary for precision in calibration and in calculating viscosity.
- 6.8.2 Computer Accumulation of Torque and Temperature Data—Computer recording of digital data can also be used for the test method. Such programs should show data for both torque and stator temperature. Torque information should be capable of permitting the calculation of viscosity to the second decimal place.

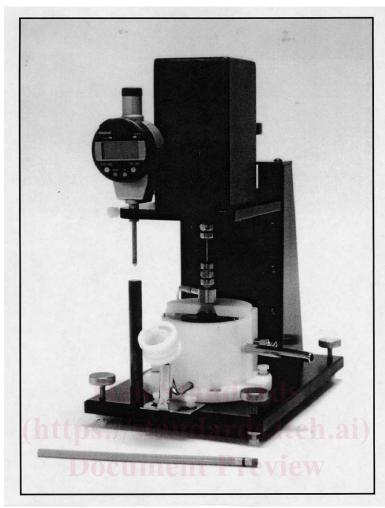


FIG. 3 Multi-Speed Tapered Bearing Simulator Viscometer Model 2100E

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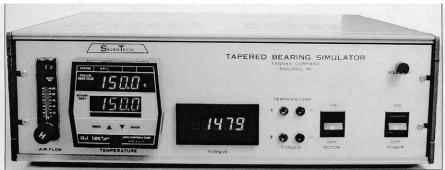


FIG. 4 Control Console for Tapered Bearing Simulator Viscometer Models 400, 500, and 600

7. Materials

- 7.1 Reference Newtonian Calibration Oils, ² Newtonian oils of known dynamic viscosity at 100°C. 100 °C. Table 1 shows the dynamic viscosity values of five Newtonian oils used in developing the information for this test method.
 - 7.2 *Idling Oil*—See 3.2.1 for information and use.
- 7.3 Non-Newtonian Reference Oil, 2 essential in setting the rotor/stator gap to $1\cdot10^6 \mathrm{s}^{-1}$ shear rate. The nominal level of apparent viscosity of non-Newtonian reference oil, NNR-10 used in applying this test method is given in Table 1.
 - 7.4 Polar Solvent, such as dimethyl sulfoxide is used to dissolve any deposits on the rotor/stator surfaces after extended use.
 - 7.5 Source of moderate pressure (<100 PSI) dry air or nitrogen.

TABLE 1 Reference Oil Viscosities at 100.0°C

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	Reference Oil	Characteristic	Nominal Viscosities ^A
			mPa⋅s at 1⋅10 ⁶ s ⁻¹
	R-2200	Newtonian	~3
	R-2300	Newtonian	~5
	R-2350	Newtonian	~7
	R-2400	Newtonian	~10
	R-2450	Newtonian	~12
	NNR-10 ^B	Non-Newtonian	~10

^A Contact supplier for certified value of Reference Oil.

Note 6—Depending on room temperatures, higher torque levels at $\frac{100^{\circ}\text{C}}{100^{\circ}\text{C}}$ and $1\cdot10^{6}\text{s}^{-1}$ may require air or other gas cooling. Use of dry gas is required (to keep moisture from entering the stator housing).

8. Sampling

- 8.1 Fifty millilitres of a representative sample of fresh or used test oil is placed in a 50 mL syringe equipped with attached filter holder and $\frac{10-\mu 10 \mu}{\mu}$ filter disk² in preparation for injection into the TBS viscometer.
- Note 7—It is important to always use a filter and filter disk to prevent larger particles from entering the rotor-stator gap. However, it is also important to note that the TBS viscometer will work with heavily particle laden used oils as long as they are passed through the $\frac{10-\mu 10 \ \mu}{10 \ \mu}$ filter.

9. Preparation of Apparatus

- 9.1 Set up stator cooling method, air or liquid, according to the manufacturer's directions.
- Note 8—When analyzing relatively viscous oils, stator cooling is necessary. This is particularly the case at lower operating temperatures such as $\frac{100^{\circ}\text{C}}{100^{\circ}\text{C}}$ where simple radiation from the stator through the stator housing is not sufficient to carry away the heat generated by viscous resistance to shear.
- 9.1.1 *Air Cooling* Connect cooling air tubing to the ports on the stator housing and the back of the console following directions given by the manufacturer in the Owner's Manual. This will permit use of the flow meter on the left side of the console to adjust the cooling-air flow rate.
 - 9.1.1.1 Set the airflow rate at 100 SCFH.
- Note 9—Once airflow rate has been set, it is important that this level be maintained throughout calibration and operation. If desired, the air may be passed through a copper coil in a chilling bath containing water, ice, or dry ice, as necessary, to obtain desired stator temperature. The cooling level must be kept constant.
- 9.1.2 *Liquid Cooling* Connect liquid cooling bath tubing from bath pump to the stator housing and the back of the console using insulated tubing according to the manufacturer's directions.
- 9.2 If some days or weeks have elapsed since last use of the TBS viscometer, follow the manufacturer's instructions regarding set-up and alignment of the rotor in the stator, checking the accuracy of the RTD and, if necessary, adjusting to 100.0°C. 100.0°C. Shut the power off and go to 9.3.
- Note 10—Directions for preparation of the tapered bearing simulator viscometer and console are supplied with the equipment. One of the most important directions to be followed is the alignment of the rotor and stator before initial use of the viscometer. For those TBS Models (other than Model 2100E) requiring bearing inspection, low bearing hysteresis and bearing cleanliness are also important to obtaining reliable data.
- Note 11—Bearing hysteresis should be checked every few months and if the values of increasing and decreasing torque are significantly different, the bearing should be cleaned and re-checked by hysteresis measurements.
- 9.3 If the TBS viscometer has been turned off for some period of time (>1 h), make sure the motor is off and slowly $(\sim 2 \text{ min})$ inject 50 mL $(\sim 2 \text{ min})$ inject 50 mL of R-2400 into the stator while turning the rotor using the upper Siamese collet connecting the motor shaft and the drive wire slowly between the thumb and forefinger.
 - 9.4 If the TBS Viscometer has been operating at 100°C, 100 °C, proceed to Section 11 unless recalibration is desired.
 - 9.4.1 If recalibration is desired, proceed to 10.2.

10. Calibration

- 10.1 If the operating position of the rotor in the stator has already been established in previous work at 100°C, <u>100°C</u>, proceed to Section 12.
- 10.2 If the operating position of the rotor in the stator must be established from a cold start for operation at \(\frac{100 \circ C}{100 \circ C}\), follow the manufacturer's instructions to find the rubbing contact position of the rotor with the stator by rotating the upper Siamese collet between the thumb and forefinger as the rotor is slowly lowered by using the elevator wheel (see Note 12).
 - Note 12—The indicator dial reading decreases when the rotor and platform are raised and vice-versa. Exercise care in using the TBS elevator wheel

^B Special reference oil closely equivalent to R-2400 at a value of 1·10⁶s⁻¹ shear rate