Designation: D4683 – 09

Standard 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[1]

This standard is issued under the fixed designation D4683; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 This test method covers the laboratory determination of the viscosity of engine oils at 150 °C and $1.0{\cdot}10^6$ s^{-1} using a viscometer having a slightly tapered rotor and stator called the Tapered Bearing Simulator (TBS) Viscometer.[2]

1.2 The Newtonian calibration oils used to establish this test method range from approximately 1.2 mPa·s to 7.7 mPa·s at 150 °C.

1.3 The non-Newtonian reference oil used to establish the shear rate of $1.0{\cdot}10^6$ s^{-1} for this test method has a viscosity closely held to 3.55 mPa·s at 150 °C.

1.4 Manual, semi-automated, and fully automated viscometers were used in developing the precision statement for this test method.

1.5 Application to petroleum products other than engine oils has not been determined in preparing the viscometric information for this test method.

1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6.1 This test method uses the milliPascal·second (mPa·s) as the unit of viscosity. This unit is equivalent to the centipoise (cP).

1.7 *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:*[3]

D4741 Test Method for Measuring Viscosity at High Temperature and High Shear Rate by Tapered-Plug Viscometer

D5481 Test Method for Measuring Apparent Viscosity at High-Temperature and High-Shear Rate by Multicell Capillary Viscometer

3. Terminology

3.1 *Definitions:*

3.1.1 *density*—mass per unit volume of the test liquid. In SI, the unit of density is the kilogram per cubic metre, but, for practical use, a submultiple is more convenient. Thus, gram per cubic centimetre is customarily used and is equivalent to 10^3 kg/m^3.

3.1.2 *Newtonian oil or fluid*—oil or liquid that at a given temperature exhibits a constant viscosity at all shear rates or shear stresses.

3.1.3 *non-Newtonian oil or fluid*—oil or liquid that exhibits a viscosity that varies with changing shear stress and shear rate.

3.1.4 *shear rate*—velocity gradient in liquid flow in millimetres per second per millimetre (mm/s per mm). The SI unit for shear rate is reciprocal seconds, s^{-1}.

3.1.5 *shear stress*—force per unit area causing liquid flow. The *unit area* noted is the area over which viscous shear is being caused.

3.1.6 *viscosity*—ratio of applied shear stress and the resulting rate of shear. It is sometimes called the coefficient of dynamic or absolute viscosity (in contrast to kinematic 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 (Pa·s), often conveniently expressed as milliPascal·second (mPa·s), or as the English system equivalent, the centipoise (cP).

[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] The sole source of supply of the apparatus known to the committee at this time is Tannas Co., 4800 James Savage Rd., Midland, MI 48642. 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.

[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.

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

3.1.6.1 *apparent viscosity*—viscosity of a non-Newtonian liquid determined by this test method at a particular shear rate or shear stress.

3.1.6.2 *kinematic viscosity*—ratio of the viscosity (dynamic, absolute) to the density of the liquid. It is a measure of the resistance to flow of a liquid where the shear stress (force causing flow) is applied by gravity. Kinematic viscosity values are thus affected by both the dynamic viscosity (absolute viscosity) of the liquid and its density. In SI, the unit of kinematic viscosity is the metre squared per second, often conveniently expressed as millimetre squared per second and termed the centiStoke.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *calibration and reference oils*[2]—oils used to establish the viscosity-torque relationship of the TBS Viscometer at 150 °C from which both appropriate rotor/stator gap and the viscosity of an unknown oil is calculated.

3.2.1.1 *Newtonian calibration oils*[2]—Newtonian oils formulated to span a viscosity range suitable for generating the torque-viscosity relationship necessary to calculate the viscosity of unknown liquids from their indicated torque values.

3.2.1.2 *non-Newtonian reference oil*[2]—oil specially formulated and critical to this test method which produces a selected value of apparent viscosity at a desired shear rate or shear stress.

3.2.1.3 *Newtonian reference oil*[2]—specially formulated Newtonian oil that has the same viscosity at 150 °C as the non-Newtonian reference oil of 3.2.1.3.

3.2.2 *filter*[2]—special filter for removing particles from the injected test oil that might damage the rotor-stator interface.

3.2.3 *idling oil*[2]—oxidatively stable Newtonian oil injected into the operating viscometer cell when the instrument is likely to be operating for more than 20 min and up to two weeks without further replacement.

3.2.3.1 *Discussion*—Use of this oil prevents formation of stator deposits from the liquid, which may begin to decompose after exposure times greater than 20 min at 150 °C in the operating viscometer and permits continuous operation of the TBS viscometer without the need to shut the instrument off.

3.2.4 *mechanical or digital micrometer*—mechanical or electronic device to measure the position of the TBS viscometer rotor in the stator.

3.2.4.1 *Discussion*—Mechanical micrometers increase readings with rotor depth. Digital micrometers interact with the TBS viscometer programs.

3.2.5 *reciprocal torque, 1/T*—value of the inverse of the torque generated by the TBS viscometer which torque is indicated on the console or computer depending on whether the viscometer is being used in the manual or automated mode.

3.2.6 *reciprocal torque intersection, $1/T_i$*—rotor position on the micrometer defined by the intersection of two straight lines. These are generated by the reciprocal indicated torque versus rotor height for the non-Newtonian NNR-03 and the Newtonian R-400. The intersection indicates the rotor height at which the rotor/stator cell will generate $1.0 \cdot 10^6$ s^{-1} shear rate.

3.2.7 *reciprocal torque intersection, 1/Tj*—desired shear rate rotor position indicated by the micrometer at the intersection of two straight lines generated by the Reciprocal Torque Intercept Method (see 10.1.4 and Annex A2) using both the Newtonian reference oil of 3.2.1.2 and the non-Newtonian reference oil of 3.2.1.3.

3.2.7.1 *Discussion*—A series of reciprocal torque values obtained at several rotor heights on both oils give linear equations whose intersection establishes the desired rotor height position for operation at a chosen shear rate. For this test method the shear rate is $1.0 \cdot 10^6$ s^{-1}.

3.2.8 *rotor height (rotor position)*—vertical position of the rotor relative to the stator and measured by a mechanical or electronic micrometer (see 3.2.4) depending on the Model TBS.

3.2.8.1 *Discussion*—For those TBS viscometers equipped with a mechanical micrometer (Models 400, 450, 500, 600 and SS) the rotor height decreases and approaches contact with the stator with increasing indicated values on the micrometer. For those TBS viscometers equipped with electronic micrometers (Models 2100 E and 2100 EF) the rotor height increases with increasing indicated values.

3.2.9 *rubbing contact position*—rotor height determined when the tapered rotor is brought into slipping contact with the similarly tapered stator.

3.2.10 *stored position of rotor height*—rotor position with the rotor 0.50 mm above the *rubbing contact* position (see 3.2.9) when the instrument is shut down.

3.2.11 *test oil*—any oil for which the apparent viscosity is to be determined by this test method.

4. Summary of Test Method

4.1 A motor turns a tapered rotor closely fitted inside a matched tapered stator at a rotor-stator gap found by the Reciprocal Torque Intersection Method to provide $1.0 \cdot 10^6$ s^{-1} at 150 °C, which are the test conditions of this test method. When this operating condition is established, test oils are introduced into the gap between the spinning rotor and stationary stator either directly by the operator or indirectly by automated injection. When a test liquid is injected, the rotor experiences a reactive torque to the liquid's resistance to flow (viscous friction) and this torque response level is used to determine the apparent viscosity.

5. Significance and Use

5.1 Viscosity values at the shear rate and temperature of this test method have been indicated to be related to the viscosity providing hydrodynamic lubrication in automotive and heavy duty engines in severe service.[4]

5.2 The viscosities of engine oils under such high temperatures and shear rates are also related to their effects on fuel efficiency and the importance of high shear rate, high temperature viscosity has been addressed in a number of publications and presentations.[4]

6. Apparatus

6.1 *Tapered Bearing Simulator-Viscometer (TBS)*[2]—A patented viscometer consisting of a motor directly connected to a

[4] For a comprehensive review, see "*The Relationship Between High-Temperature Oil Rheology and Engine Operations*," ASTM Data Series Publication 62.

slightly tapered rotor that fits into a matched tapered stator (see Fig. 1). The reaction torque of the rotor to the liquid in the cell is measured and used to calculated viscosity. Several models of the TBS Viscometer are in use (see Annex A1 for information and pictures of later models). All TBS models are capable of analyzing test oils at temperatures from 40 °C to 200 °C, but earlier models were more limited in their upper viscosity range.

6.1.1 The stator enclosed within its insulated housing is held immobile while the motor and the connected rotor are set above and within the stator, respectively, on a cantilevered platform attached to a mechanical elevator that can be moved vertically either manually or by a computer program using a stepper motor to change the platform height (see Annex A1).

6.1.2 The resistive force of the test oil is transferred to a load cell that measures the torque required to turn the rotor at the speed selected. Earlier models of the TBS viscometer operated at 3500 or 3600 r/min depending on the frequency of the supplied voltage. Later models (2100 E, and 2100 EF) have been equipped to operate at multiple speeds which allow the operator to produce a series of shear rates variable by choice of the combination of initial rotor-stator gap and rotor speed.

NOTE 1—This technique applies to all TBS viscometer models, manual, semi-automated, and fully automated.

6.2 Three models of the TBS Viscometer (Models 500, 2100 E, and EF) are shown in Annex A1 and have the operating viscosities, cooling modes, and temperatures given in Table A1.1.

NOTE 2—TBS Models 400, 450, 500, 600, and SS use a so-called *bouncer* to prepare the load cell for taking a torque reading except when determining the Reciprocal Torque Intercept. (The semi-automated version of Model 500 automatically applies the bouncer at the appropriate point of operation automatically as part of its program.) Models 2100 E and 2100 EF do not require the bouncer technique, since neither has turntable bearings.[2]

6.3 *Automated and Semi-automated Systems for Calibration, Injection, and Data Analysis Programs*—Automated programs for the TBS Viscometer simulate the manual method. Programmed as well as manually operated TBS Viscometers were used in producing the data supporting this test method.

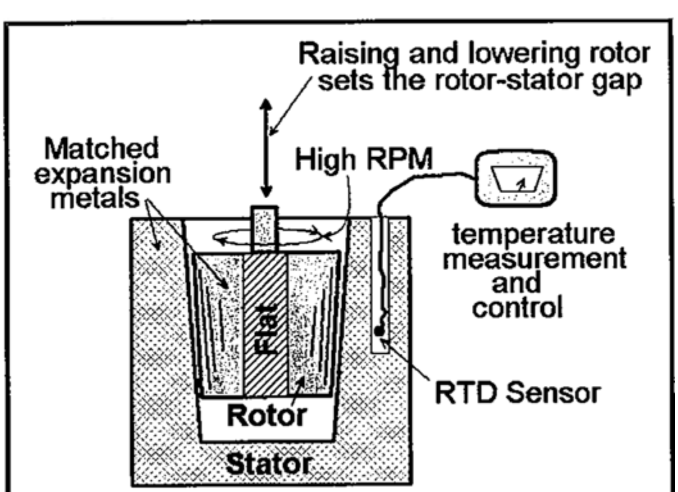


FIG. 1 Matched Tapered Stator

6.4 *Cooling Systems*—As shown in Table A1.1, in addition to natural radiation and convection of heat from the stator, two stator cooling systems are available for TBS Viscometers depending on the viscosity of test liquid to be analyzed. A stator housing is designed for each type of cooling system.

6.5 *Sample Injection*—Sample injection depends on the manner in which the TBS viscometer is operated. In manual mode, sample injection is with either re-usable 50-mL glass or disposable plastic syringes equipped with Luer lock connections fitting the tip of the filling tube. In semi-and fully-automated mode, the filling line from the autosampler is connected by a Luer lock fitting to the filling tube.[2]

6.6 *Filter Assembly*—A filter holder, able to be disassembled, containing five nominal 10 micron filter discs or a one piece discardable filter cartridge[2] is interposed between the syringe (or autosampler line) injecting the test oil and the stator filling tube to remove particles capable of damaging the rotor/stator cell.

NOTE 3— Refer to the Owner's Manual for frequency of changing filter cartridges, particularly with used engine oil.

6.7 *Data Recording Equipment*—Refer to the Owner's Manual for the viscometer model.

7. Materials

7.1 *Calibration Oils*[2]—These are Newtonian oils of known dynamic viscosity at 150 °C (see 3.2.1). Table 1 shows the dynamic viscosity values of eight Newtonian oils, R-100 to R-600, which are available from the manufacturer of the TBS Viscometer and described in the Owner's Manual.

7.2 *Non-Newtonian Reference Oil*—This reference oil is essential in setting the rotor/stator gap to $1.0 \cdot 10^6$ s^{-1} shear rate (see 3.2.1.3). The nominal apparent viscosity of non-Newtonian Reference Oil, NNR-03 used in applying this test method at 150 °C is given in Table 1 and is matched to the viscosity of R-400 both held closely to 3.55 mPa·s (see 3.2.1.2).

7.3 *Idling Oil*—See 3.2.3 and the Owner's Manual for information and use.

7.4 *Solvent*—Such as VarClean,[2] used to remove any varnish and deposits on the rotor/stator surfaces after extended use. Follow manufacturer's instructions in the Owner's Manual for use in the TBS viscometer.

7.5 *Cooling Gas for Temperature Control*—If gas is chosen to cool the stator, a source of moderate pressure (<100 psi) clean, dry air or nitrogen is required. Use of a dry gas is required to keep moisture from entering the stator housing. Flow rate to the stator is controlled by a flowmeter on the left side of the console's front panel (see Annex A1, Fig. A1.1, and Fig. A1.3.

8. Sampling

8.1 A representative, homogeneous sample of the oil is required, particularly with used engine oil in which particles may have settled to the bottom of the container. Such homogeneous samples are obtained by vigorous agitation and mixing techniques (see Owner's Manual).

NOTE 4—It is recommended that even fresh sample be mixed by gentle stirring or inverting the closed container several times.

TABLE 1 Nominal Reference Oil Viscosities at 150.0 °C

Reference Oil	R-100	R-200	R-300	R-350	R-400	R-450	R-500	R-600	NNR-03
Viscosity, mPa·s	~1.2	~1.5	~1.8	~2.7	~3.55	~4.1	~5.0	~7.7	~3.55[A]

[A] Value at $1.0 \cdot 10^6$ s^{-1} shear rate

8.2 Fifty millilitres of a representative sample of the homogenized fresh or used engine test oil is drawn into a 50-mL syringe or into the sampling tubes of the auto-sampling apparatus.

8.3 The 50-mL sample is injected either by hand or by the auto-sampling apparatus through the special 10-μ filter disc on the viscometer's filling tube (see 6.6).

9. Preparation of Apparatus

9.1 *Choose and Set Up Stator Cooling Mode*—None, gas, cooled gas, or liquid mantle, in accordance with the manufacturer's directions in the Owner's Manual.

9.2 Check the accuracy of the RTD (Resistance Thermometric Device) as directed in the Owner's Manual and, if necessary, make whatever slight temperature offset is needed for the temperature controller to bring the readout to 100.0 °C (the latter alignment of temperature should be checked at least once per year).

9.3 *If the TBS Viscometer has been Turned Off for a Week or More*—It is necessary to ensure that the viscometer rotor and stator are still operating to provide $1.0 \cdot 10^6$ s^{-1} shear rate.

9.3.1 Follow the manufacturer's instructions in the Owner's Manual regarding set-up and alignment of the rotor in the stator and the determination of the *stored position* of the rotor by determining *rubbing contact* followed by raising the rotor to the indicated height from *rubbing contact*.

9.3.2 Shut power off and go to 9.4.1.

NOTE 5—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 2100 EJ requiring bearing inspection, bearing cleanliness and low levels of bearing hysteresis are also important to obtaining reliable data.

NOTE 6—For those TBS viscometer models using ball bearings to support the motor platform (all but Models 2100 E and 2100 EF which have no bearings), bearing hysteresis should be checked every few months according to the Owner's Manual and if the values of increasing and decreasing torque by this hysteresis analysis are significantly different (~2%), the bearing should be cleaned and then re-cnecked by the same measurement method for hysteresis.

9.4 *If the TBS Viscometer has been Turned Off for a Relatively Short Time*—(More than 1 h, but less than a week):

9.4.1 Make sure the motor switch is in *off* position then turn on the main switch.

NOTE 7—Turning the motor switch off before turning the main switch on prevents breakage of the flexible shaft connecting the motor and rotor.

9.4.2 Slowly (~2 mL/s) inject 50 mL of R-400 into the stator while also slowly turning the rotor between the thumb and forefinger using the upper portion of the Siamese collet connecting the motor shaft and the drive wire.

9.4.3 Place the rotor in the *stored position* (see 9.3.1 and Owner's Manual).

9.4.4 Set the desired temperature to 150.0 °C when the rotor/stator cell temperature reaches about 140 °C, turn on the motor and wait until the cell temperature settles at 150.0 ± 0.1 °C for 1 h before proceeding with analysis.

9.5 *If the TBS Viscometer has been Operating at 150 °C*—Proceed to Section 12, unless recalibration is desired.

9.5.1 If recalibration is desired, proceed to Section 11.

10. Establishing Operating Position of the Rotor for $1.0 \cdot 10^6$ s^{-1} Shear Rate

NOTE 8— If the rotor position has already been established, proceed to 11.1.

10.1 *Manual TBS Viscometer Method*:

10.1.1 *Activating the Console*—Confirm that the motor switch on the console is in the *off* position. Then turn the main Power switch to the *on* position for 1 h to permit the electronic circuits to come to equilibrium in this stand-by condition before proceeding to calibrate the TBS viscometer.

10.1.2 *Test Cell Filling*—If there is no oil in the test cell, slowly inject (~2 mL/s) 50 mL of Reference Oil R-400 in the test cell and proceed with the determination of the so-called *stored position* of the rotor as described in 9.3.1.

10.1.3 Bring the cell temperature to 150 °C and allow the rotor-stator cell to stabilize.

10.1.4 *Determination of Operating Position*—Use the Reciprocal Torque Intersection Method in the Owner's Manual for setting the rotor-stator gap for $1.0 \cdot 10^6$ s^{-1} shear rate. For more understanding of the basis of the equations used by the program to obtain semi- and full-automated operation, see Annex A2.

11. Viscosity Calibration of TBS Viscometer

11.1 *Manual Method*:

11.1.1 Set rotor position exactly to that determined in 10.1.4 and make sure the unit is warmed up for at least 1 h.

NOTE 9—If desired, recheck or readjust rotor position at $1.0 \cdot 10^6$ s^{-1} shear rate according to the Owner's Manual.

NOTE 10—Slow expansion of the rotor and stator after start up of the TBS Viscometer may slightly change the originally determined position of the rotor at $1.0 \cdot 10^6$ s^{-1} shear rate and it is, thus, prudent to recheck the rotor position and to make slight adjustments if necessary.

11.1.2 Calibration of the TBS viscometer cell with confirming recheck of the operationally correct rotor position to generate $1.0 \cdot 10^6$ s^{-1} shear rate:

11.1.2.1 Inject Newtonian Reference Oil R-200 slowly (~2 mL/s) and wait until torque/temperature equilibrium is obtained (see Note 15). Use the Bouncer button briefly (only for older Models 400 through 600 and SS) after torque/temperature equilibrium, allow the torque value to stabilize, and record the torque value.

11.1.2.2 Repeat 11.1.2.1with Newtonian Reference Oil R-450.

11.1.2.3 Repeat 11.1.2.1 with Non-Newtonian Reference Oil NNR-03.

11.1.2.4 Use the known viscosities of Newtonian Reference Oils R-200 and R-450 and the torque values from 11.1.2.2 and

11.1.2.3 to algebraically calculate the slope, *m*, and intercept, *b*, of equation given by these two pairs of values with the torque read from the console as variable τ_c, and viscosity as variable η, in Eq 1.

$$\eta = m \cdot \tau_c + b \quad (1)$$

NOTE 11—The algebraic formulas for *m* (slope) and *b* (intercept) are:

$$m = (\eta_{R-450} - \eta_{R-200})/(\tau_{cR-450} - \tau_{cR-200})$$

and

$$b_v = (\eta_{R-450} - \eta_{R-200}) - [(\eta_{R-450} - \eta_{R-200})/(\tau_{cR-450} - \tau_{cR-200})]$$

11.1.2.5 Insert the indicated torque value obtained on Reference Oil NNR-03 into Eq 1, calculate its viscosity and compare this to the viscosity on the NNR-03 container.

11.1.2.6 If the viscosity value calculated for NNR-03 is within ±1.5% of the value on its container, proceed to 11.1.3.

11.1.2.7 If the value of NNR-03 is not within ±1.5% of its container value, slowly (~2 mL/s) re-inject 50 mL of R-400 (first used in 10.1.4) and redetermine the R-400 torque value.

11.1.2.8 Substitute this new torque value of R-400 for its previously determined value in 10.1.4, and determine the NNR-03/R-400 torque ratio using NNR-03 data from 11.1.2.3.

11.1.2.9 If the NNR-03/R-400 torque ratio is within 1.000 ± 0.015, go to 11.1.3 and continue calibration of the rotor/stator cell.

11.1.2.10 If the NNR-03/R-400 torque ratio is again outside of 1.000 ± 0.015, return to 10.1.4, re-establish the correct rotor position, and recalibrate the rotor/stator cell by following 11.1.2.1 to 11.1.2.9.

11.1.2.11 If repeated efforts do not produce a value of NNR-03 within 1.000 ± 0.015 of the container value, contact the instrument manufacturer.

11.1.2.12 When the NNR-03/R-400 torque ratio is within 1.000 ± 0.015, and the value of NNR-03 is within 1.5% of the container value, record the new setting of the rotor and reinitiate 11.1.2 from 11.1.2.1 to 11.1.2.9.

11.1.3 Continue the calibration of 11.1.2:

11.1.3.1 Sequentially and slowly (~2 mL/s) inject 50 mL of Newtonian Reference Oils R-350 and R-400. For each reference oil, if the particular Model of TBS viscometer requires this technique (see Note 2), use the Bouncer technique immediately after the torque/temperature equilibrium is attained, and record the torque values for each reference oil.

11.1.3.2 Using the viscosities calculated from the torque values at 150.0 °C for R-200, R-450, (11.1.2.4) and R-350, and R-400 from 11.1.3.1, linearly regress the viscosities and their related torque values to determine the slope, intercept, and Correlation Coefficient, R, related to their linear relationship.

NOTE 12—The calculation of the linear regression equation from the data gathered in 11.1.3.2 is:

$$Y = m \cdot x + b \quad (2)$$

Y = known viscosity of the given calibration oil, and
X = indicated torque obtained by using that calibration oil.

Then slope is:

$$m = [\Sigma XY - (\Sigma X \cdot \Sigma Y/N)]/[\Sigma X^2 - (\Sigma X)^2/N] \quad (3)$$

and intercept is:

$$b = (\Sigma Y - m \cdot \Sigma X)/N \quad (4)$$

N = number of data pairs of X,Y.

The Correlation Coefficient is:

$$R = m \cdot \sigma_x/\sigma_y \quad (5)$$

σ_x = Standard Deviation of the X values, and
σ_y = Standard Deviation of the Y values.

11.1.3.3 This yields Eq 6, which is used for subsequent calculations of the viscosity of unknown oil, η_U, from the torque value, τ_c, obtained from the console:

$$\eta_U = m \cdot \tau_c + b \quad (6)$$

11.1.3.4 The Correlation Coefficient, *R*, should result in a value of ≥0.999. If so, proceed to 11.1.4.

11.1.3.5 If the value of R is less than 0.999, repeat 11.1.2 through 11.1.3.4.

11.1.3.6 If there is still a problem in obtaining a Correlation Coefficient ≥0.999, contact the instrument manufacturer.

11.1.4 Following calibration of the rotor/stator cell, confirm the rotor position using Non-Newtonian Reference Oil NNR-03

11.1.4.1 Slowly (~2 mL/s) inject 50 mL of Non-Newtonian Reference Oil NNR-03, obtain the torque value, τ_c, from the console after temperature and torque equilibrium have been established (see 10.1.4 and Note 15), substitute the value of τ_c in Eq 6, and with the previously obtained values of *m* and *b* in 11.1.3.3, calculate the indicated viscosity of NNR-03.

11.1.4.2 The viscosity value determined for NNR-03 should be within ±1.5% of the value on the container. If so, proceed to Section 12.

11.1.4.3 If the viscosity value determined for NNR-03 is not within 1.5% of the value on the container, recalculate from the torques of R-400 in 11.1.3.2 and NNR-03 obtained in 11.1.2.3 if the torque ratio of NNR-03/R-400 is 1.000 ± 0.015.

11.1.4.4 If the NNR-03/R-400 torque ratio is within 1.000 ± 0.015, repeat the calibration steps of 11.1.2.1 through 11.1.4.2.

11.1.4.5 If the NNR-03/R-400 torque ratio is outside of 1.000 ± 0.015, adjust the rotor height according to directions in the Owner's Manual until the correct NNR-03/R-400 torque ratio value is obtained and then repeat the calibration steps of 11.1.2.1 through 11.1.4.2.

11.1.4.6 If the viscosity of NNR-03 is still not within 1.5% agreement with the value on the container, contact the instrument manufacturer.

11.2 *Semi-Manual Method*:

11.2.1 Set the rotor to the value of H_i given by the program (see Owner's Manual).

11.2.2 Initiate the calibration program to run the analysis of R-200, R-450, and NNR-03 to compare the latter value to the value entered into the program data from the NNR-03 container.

NOTE 13—The program automatically injects R-200, R-450, and NNR-03 and then automatically calculates the value of NNR-03.

11.2.3 If the value of NNR-03 determined by the programmed analysis of R-200 and R-450 is greater or lesser than

0.05 mPa·s of the value on the NNR-03 container, the program will wait for operator reaction for response.

NOTE 14—The Owner's Manual recommends first re-running the programmed analysis of 11.2.1.

11.2.3.1 If the calculated viscosity value of NNR-03 is greater than that on the container, lower the rotor into the stator by 0.02 mm for each 0.05 mPa·s greater viscosity value and repeat 11.2.2.

11.2.3.2 If the calculated viscosity value of NNR-03 is less than that on the container, raise the rotor out of the stator by 0.02 mm for each 0.05 mPa·s viscosity value less than that on the container and repeat 11.2.2.

11.2.4 If the viscosity value is within 0.05 mPa·s of the value on the NNR-03 container previously entered by the operator into the required program information, the program will automatically proceed with calibration using Reference Oils R-300, R-400 and recalculate the value of NNR-03 using all four values of Reference Oils R-200, R-450, R-300 and R-400.

11.2.5 If the recalculation of the viscosity of NNR-03 is within ±0.05 mPa·s of the value on the container, the program will move on to test sample analysis. Proceed to 12.2.

11.2.5.1 If the recalculation of the viscosity of NNR-03 is outside of ±0.05 mPa·s, adjust the rotor position as directed in 11.2.3 or 11.2.3.2 and repeat 11.2.1 to 11.2.5.

11.2.5.2 If redetermination of the viscosity of NNR-03 is still not within ±0.05 mPa·s(cP), contact the viscometer manufacturer.

11.3 *Fully-Automated Method*—The fully-automated program automatically calibrates the rotor-stator cell after automatically determining the proper rotor position for $1.0{\cdot}10^6\ s^{-1}$ shear rate. If correction of rotor position is required, the program makes this change and recalibrates. Proceed to 12.3.

12. Viscometric Analysis of Sample Oils

12.1 *Manual Method*:

12.1.1 Inject 50 mL of a test oils slowly (~2 mL/s) into the fill tube, wait for temperature/torque equilibrium, (apply the Bouncer button if the Model viscometer requires this action, see Note 2). Record the torque value after it again stabilizes.

12.1.2 Use the torque reading, τ_c, obtained in Eq 6 to calculate the viscosity of the unknown oil.

12.1.3 Progressively analyze five more test oils.

12.1.4 After six unknown oils have been analyzed, re-check rotor position by slowly injecting Non-Newtonian Reference Oil NNR-03 and determine its viscosity using Eq 6.

12.1.5 The viscosity value determined for NNR-03 should be within ±1.5% of the value on the container. If so, proceed to the next six unknown oils.

12.1.5.1 If the viscosity value determined for NNR-03 is not within 1.5% of the value on the container, see 11.1.4.2. (It may be necessary to re-establish the rotor position giving $1.0{\cdot}10^6\ s^{-1}$ shear rate by directions given in 11.1.2.8 or in the Owner's Manual and reestablish the correct values of *m* and *b* in Eq 6 by recalibration.

12.1.6 Continue with viscometric analysis and injecting of NNR-03 after ever six unknown test samples to maintain certainty that the proper shear rate is being applied.

12.1.7 Manual analysis of smaller available volumes of oil

12.1.7.1 If 30 mL of test oil are available, fill the syringe with this amount of oil and slowly (20 s per injection) make three injections of 10 mL each. Wait 10 s between injections to allow the new oil to incorporate and remove remnants of the previous injection.

NOTE 15—If the amount of test oil is severely limited, contact the manufacturer regarding a technique requiring no more than 10 to 15 mL of test oil.

NOTE 16—-After several hundred samples of fresh oil (and more frequently if engine oils are being analyzed) it is good practice to clean the rotor/stator cell with a solvent for any oxidized residue on the wall of the stator or surface of the rotor (see 7.4).

12.2 *Semi-Automated Method*:

12.2.1 After automated calibration (11.2.2), the program continues by automatically initiating the analyses of the unknown test samples.

12.2.2 After every six samples are run, the program automatically reanalyzes the Non-Newtonian Reference Oil NNR-03 to assure continued application of the correct shear rate.

12.2.2.1 If the redetermined value of NNR-03 is in range (within ±0.05 mPa·s), the program continues test oil analysis.

12.2.2.2 If the redetermined value of NNR-03 is outside of its allowed range (within ±0.05 mPa·s), the program will pause and wait for operator response which is to return to 11.2.3 to readjust the rotor position and recalibrate the rotor-stator cell.

12.2.3 The program automatically calculates the viscosity of the unknown test oils and reports these values on the computer screen as well as by the attached printer.

12.3 *Fully-Automated Method*—

12.3.1 After calibration, the operator can choose whether or not to allow the program to move to the analysis of test oils. Instruct the program to continue to analyzing test oils.

NOTE 17—The program will analyze the test oils. Every, seventh analysis is of NNR-03. If this value falls out of range, the program automatically readjusts the rotor position and recalibrates.

13. Calculation

13.1 *Manual Method*—For each test oil, insert the torque reading, τ_c, produced by the analysis into Eq 6 and using the predetermined values of slope, *m*, and intercept, *b*, calculate the apparent viscosity of the test oil at $1.0{\cdot}10^6\ s^{-1}$ shear rate in mPa·s (cP).

13.2 *Semi-Automated Method*—The program calculates the viscosity of the unknown test oil in mPa·s (cP).

13.3 *Fully-Automated Method*—The program calculates the viscosity of the unknown test oil in mPa·s (cP).

14. Report

14.1 *All Methods*—Report the apparent viscosity to the nearest 0.01 mPa·s at 150 °C and $1.0{\cdot}10^6\ s^{-1}$ shear rate for each test oil for either the manual or automated protocols.

15. Precision and Bias [5]

15.1 *Precision*—The following criteria should be used for judging the acceptability of results from either the manual protocol or the automated protocols:

15.1.1 *Repeatability*—The difference between two successive test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

$$1.04\% \text{ of the mean} \tag{7}$$

15.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

$$3.58\% \text{ of the mean} \tag{8}$$

15.2 *Bias*—There is no accepted reference material suitable for determining the bias of this test method.

15.2.1 *Relative Bias*—Relative Bias was determined by comparing the Tapered Bearing Simulator Viscometer of this test method with another rotational viscometer called the Tapered Plug Viscometer, Test Method D4741. Both of these instruments operate at constant shear rate. Cross-comparing results from the original ASTM Multi-Instrument Round Robin (MIRR) and data produced in the ongoing ASTM Cross Check program showed little or no Relative Bias and essentially no change in their correlation from the MIRR in 1996 to present day. As shown in Research Report D02-1671 for this test method, the combination of values from the MIRR and the ASTM Cross Check program gave a value for the Coefficient of Determination, R^2, of 0.997 (a value of unity being perfect), a slope of the best line through the collected data of 0.98 (unity again being perfect non-bias), and an intercept of 0.04 mPa·s (0.00 being best).

15.2.2 Correlation and Relative Bias was also generated with another high shear rate viscometer using a capillary technology and thus having non-constant shear rate across the capillary. This instrument, called the Multi-Capillary Viscometer, Test Method D5481, was found to correlate with the TBS Viscometer, and gave further support to very little Relative Bias with no change in this relationship with time. Research Report D02-1671 provides further information.

15.3 *Laboratories and Oils Used in Generating Data*—Nine laboratories submitted data on 10 engine oils used in the round robin program to obtain the precision statement. Statistical analysis was obtained on blind coded engine oils covering an approximate range of viscosities from approximately 2.5 to 4.5 mPa·s at 150 °C at a shear rate of $1.0 \cdot 10^6$ s^{-1}. The test series was composed of six fresh and four used engine oils.

16. Keywords

16.1 dynamic viscosity; engine oils; high shear rate viscosity; high shear viscosity; high temperature viscosity; high shear rate viscosity at 150 °C; rotational viscometer; Tapered Bearing Simulator Viscometer; TBS; used engine oils

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1671.

ANNEXES

(Mandatory Information)

A1. MODELS OF THE TAPERED BEARING SIMULATOR VISCOMETER

A1.1 See Table A1.1 for present TBS models. See Fig. A1.1 for Model 2100 EF: Fully automated system. See Fig. A1.2 for TBS Viscometer Model 2100 EF components. See Fig. A1.3 for TBS Viscometer Model 2100E. See Fig. A1.4 for TBS Viscometer Model 500.

TABLE A1.1 Present TBS Models, Properties, and Ranges

TBS Model	Speeds, r/min	Viscosity Range, mPa·s	Temperature Range, °C	Program Application	Cooling Mode Application
2100 EF	800 to 8000	0.2 to 26	40 to 200	Manual or Fully-Auto	Gas circulation or water mantle
2100 E	800 to 8000	0.2 to 26	40 to 200	Manual or Semi-Auto	Gas circulation or water mantle
500	3600	0.2 to 17	100 to 200	Manual or Semi-Auto	Gas circulation or water mantle

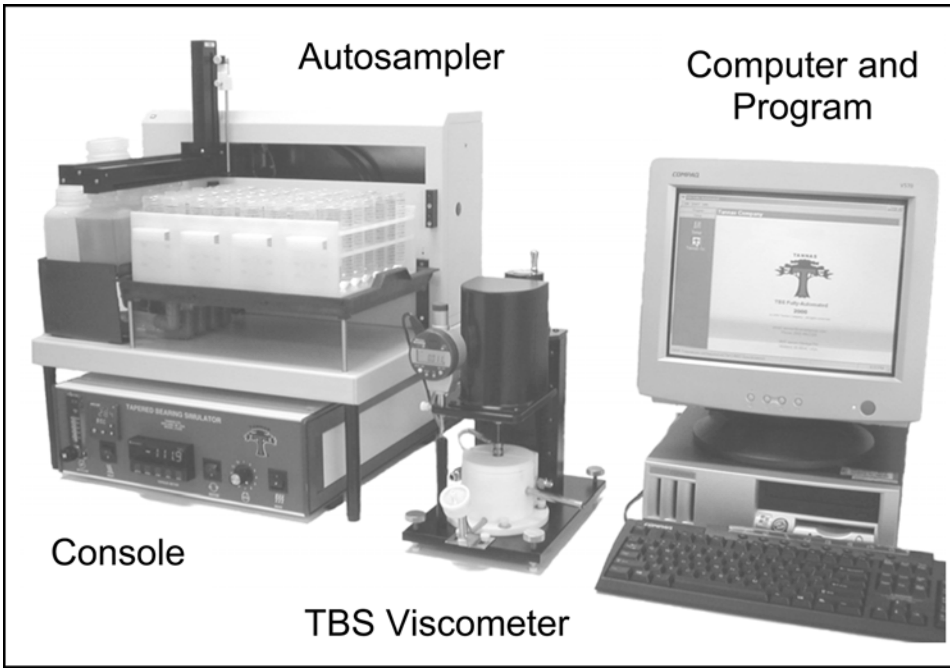


FIG. A1.1 Fully Automated TBS Viscometer Model 2100 EF Showing Control Console, Autosampler, TBS Viscometer, and Computer

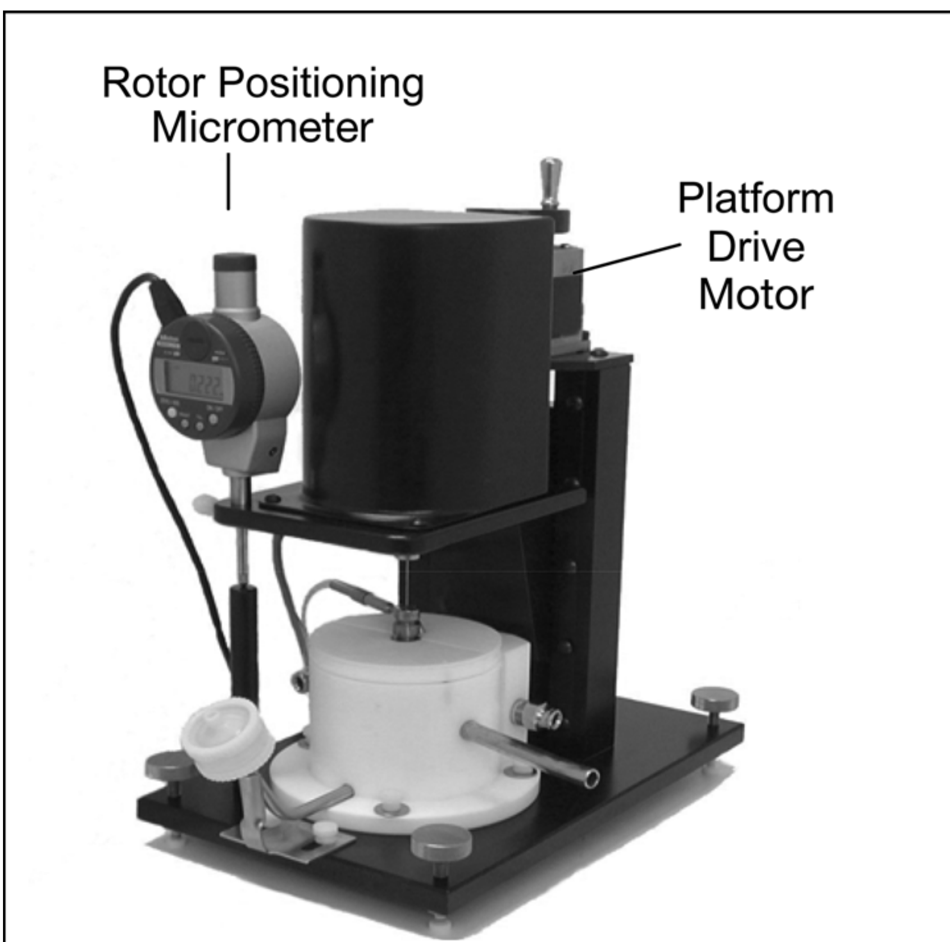
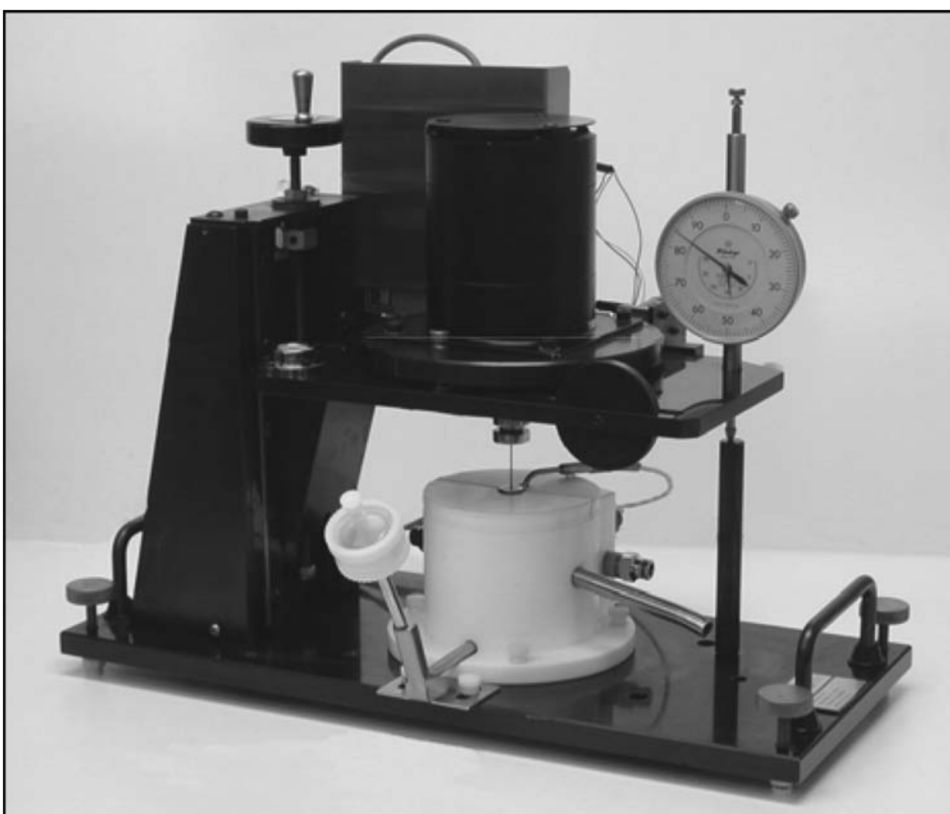


FIG. A1.2 TBS Viscometer Model 2100 EF Showing Interacting Rotor Positioning Micrometer and Platform Drive Motor, with Both Devices Controlled by Computer Program

FIG. A1.3 TBS Viscometer Model 2100 E Equipped for Manual Operation



NOTE—Earlier model has motor with less torque than later models but permitting analysis of fluids with viscosities up to 17mPa·s.

FIG. A1.4 TBS Viscometer Model 500

A2. SOME EQUATIONS USED WITH ABSOLUTE TAPERED BEARING SIMULATOR VISCOMETRY

A2.1 *Background*—The Tapered Bearing Simulator (TBS) Viscometer has been shown to be an absolute viscometer.[6] That is, the TBS is a viscometer that in operation provides values for both shear rate and shear stress on a liquid from which the viscosity of the liquid can be calculated directly. That is, without need for calibration of the viscometer with reference oils.

A2.1.1 Although this absolute characteristic is a property of the high shear rate TBS Viscometer and absolute viscometry can be applied at any time, it is more cumbersome than simple calibration. However, the principles by which shear rate can be determined through absolute viscometry also permitted establishing equations helpful in setting up programs utilizing these relationships.

A2.1.2 *Reciprocal Torque Relationship*—The mathematical expression for the viscosity of a Newtonian fluid is that viscosity is equal to shear stress divided by shear rate:

$$\eta = \tau / R \quad \text{(A2.1)}$$

η = viscosity,
τ = shear stress,
R = shear rate.

At constant viscosity, shear rate is proportional to shear stress:

$$R \propto \tau \quad \text{(A2.2)}$$

However, in the TBS Viscometer, the slight and matching tapers of the rotor and stator permits this equation of proportionality to take another form. The shear rate is defined as:

$$R = V/D \quad \text{(A2.3)}$$

where:
V = velocity of one surface moving parallel to another D units distance apart.

Thus, shear rate is metres/second per metre and has the dimension of reciprocal seconds, S^{-1}.

Shear rate is thus inversely proportional to the distance between the parallel surfaces of the rapidly spinning rotor and the stationary stator. Raising and lowering the tapered rotor changes the shear rate inversely proportionately to the change in rotor height:

$$R \propto 1/H \quad \text{(A2.4)}$$

H = rotor height.

Substituting and transposing from Eq A2.2 gives:

$$H \propto 1/\tau \propto 1/T \quad \text{(A2.5)}$$

where:
T = shear stress expressed as torque experienced by the spinning rotor when it encounters the viscous friction of a fluid.

Accordingly, the latter term is called *reciprocal torque*. This relationship among rotor height, its surface's distance from the stator wall and the consequent variation of shear rate, permits determination of several important variables.

A2.1.3 *Infinite Torque*—When the inverse relationship between rotor height and torque is analyzed, the linear relationship of Eq A2.5 can be extrapolated to a value of zero reciprocal torque. Such extrapolation of the data to zero reciprocal torque produces a value in rotor height at which torque itself is infinite and can be viewed as being equivalent to contact between the rotor and stator.

A2.1.4 *Shear Rate Choice*—Thus, the analysis of data from Eq A2.5establishes the value of rotor height from which, at a known rotor/stator taper, a chosen shear rate may be selected by the rotor height above this contact point shown by the reciprocal torque intercept using a Newtonian fluid.

$$H_R = (1/T)_R \cdot m_R + b_R \quad \text{(A2.6)}$$

m = slope,
b = intercept of zero reciprocal torque, and
R = use of a Newtonian fluid.

With the foregoing information, shear rate can be chosen on the basis of a rotor position determined by a distance from rotor/stator contact — the intercept, b, indicating infinite torque, the interfacial rotational velocity of the rotor past the stator and the rotor/stator taper.

The gap, G, between the rotor and stator surfaces is:

$$G = t/H_{b+h} \quad \text{(A2.7)}$$

t = rotor-stator taper, and
H_{b+h} = rotor height above the intercept, b, in Eq A2.6.

Thus, the shear rate may be calculated from the value of G, in millitres, and the rotor surface velocity, N, in millimetres per second.

$$R = N \cdot G \quad \text{(A2.8)}$$

A2.1.5 *Use of NNR-03 and R-400 to set Rotor Height*—For reasons that lie beyond the need of this annex (but are related to the high shear rate viscometric behavior of chose non-Newtonian oils), the relationship between the reciprocal torque value of the non-Newtonian Reference Oil NNR-03 and the rotor height is also linear. Thus,

$$H_n = (1/T_n) \cdot m_n + b_n \quad \text{(A2.9)}$$

where:
n = respective values obtained using NNR-03.

In order to relatively easily set and check the best rotor position to yield $1.0 \cdot 10^6$ s^{-1}, Reference Oil NNR-03 is formulated to have an apparent viscosity of ~3.55 mPa·s at this shear rate. To easily and precisely establish the appropriate rotor position, a Newtonian oil, R-400, is formulated to have a

[6] Selby, T. W., and Piasecki, D. A., "The Tapered Bearing Simulator—An Absolute Viscometer," SAE Paper 830031, SAE International Congress and Exposition, Detroit, MI, Feb. 28 – March 4, 1983.

closely identical viscosity of ~3.55 mPa·s (see 3.2.1.2 and 3.2.1.3). Where the two linear reciprocal lines intersect:

$$H_R = H_n = H_{IS} \tag{A2.10}$$

where:

H_{IS} = intersection of the Newtonian and non-Newtonian lines

and

$$(1/T)_R \cdot m_R + b_R = H_{IS} = (1/T_n) \cdot m_n + b_n \tag{A2.11}$$

The intersection value of H_{IS} gives the rotor operating position for $1.0 \cdot 10^6$ s^{-1} shear rate and:

$$T_{R-400} = T_{NNR-03} \tag{A2.12}$$

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D4683–04) that may impact the use of this standard.

(*1*) The test method has been expanded to include both used and unused engine oils as well as manual, semi-automated and full-automated protocols of measuring high shear rate viscosity. Many small changes of sections, descriptions, equipment, notes, etc. have been made to encompass the expanded application of the test method.
(*2*) Included both fresh and used engine oils in the round-robin study and data supporting this present version of Test Method D4683.
(*3*) Computer programmed collection of data from semi-automated and fully-automated Tapered Bearing Simulator instruments, all of which utilize the basic approach of the previous version, Test Method D4683–04.
(a) Use of the theoretically correct intersection of the linear rotor height versus reciprocal torque curves of a Newtonian and non-Newtonian reference oil at high shear rate ($1.0 \cdot 10^6$ s^{-1}) to determine the appropriate operating position of the rotor, followed by
(b) Calibration of the viscometer using four Newtonian reference oils of known viscosities with a Correlation Coefficient of at least 0.999.
(c) Determination of the viscosity of each unknown oil after acceptable temperature stabilization at 150 °C.
(d) A check of proper rotor operating position after every sixth unknown sample.
(e) In semi-automatic mode the computer calls for re-adjustment of rotor position if the operating position is greater than ±0.05 mPa·s. In fully automatic mode, the computer itself readjusts the rotor position and continues the analyses without operator intervention.
(f) Termination of an analytical run by an injection of Idling Oil to prevent accumulation of varnish on the rotor and stator.
(*4*) Programmed printout of collected data.
(*5*) Added the various model types of the TBS Viscometer currently being used and applied in the round robin.
(*6*) Annex A1 is added to show the different models and configurations of the more recent models of Tapered Bearing Simulator Viscometer that are employed in making high temperature, high shear rate determinations according to this test method.
(*7*) Annex A2 is added to give understanding of the equations used in establishing the correct shear rate and calibration.