Designation: D4683 – 17

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 U.S. 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. The precision has only been determined for the viscosity range 1.47 mPa·s to 5.09 mPa·s at 150 °C for the materials listed in the precision section.

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 by using the absolute viscometry of the TBS.

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

1.5 Application to petroleum products such as base oils and formulated engine oils was 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

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants

D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material

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

3. Terminology

3.1 *Definitions:*

[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] 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.

[4] 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 method is technically identical to that described in CEC L-36 (under the jurisdiction of the CEC Engine Lubricants Technical Committee) and IP 370 references CEC L-036.

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

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

3.1.1 *apparent viscosity, n*—viscosity of a non-Newtonian liquid determined by this test method at a particular shear rate and shear stress.

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

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

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

3.1.4.1 *Discussion*—The velocity gradient in the tapered bearing simulator viscometer is constant at any chosen rotor-stator gap and rotor speed.

3.1.5 *shear stress, n*—force per unit area causing liquid flow over the area where viscous shear is being caused; in SI, the unit of shear stress is the Pascal (Pa).

3.1.6 *viscosity, n*—ratio of applied shear stress and the resulting rate of shear. It is sometimes called dynamic or absolute viscosity. Viscosity is a measure of the resistance to flow of the liquid at a given temperature. In SI, the unit of viscosity is the Pascal·second (Pa·s), often conveniently expressed as milliPascal·second (mPa·s), which has the English system equivalent of the centipoise (cP).

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *calibration and reference oils*[2], *n*—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], *n*—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], *n*—NNR-03—An oil specially formulated for, and critical to, this test method. The reference oil NNR-03 produces a selected value of apparent viscosity at a desired temperature and shear rate (see Note 1).

3.2.1.3 *Newtonian reference oil*[2], *n*—R-400—A specially formulated Newtonian oil that has the same viscosity at 150 °C as the non-Newtonian reference oil NNR-03 of 3.2.1.2.

3.2.2 *filter*[2], *n*—special filter for removing particles potentially damaging to the rotor-stator interface from the test oil being injected.

3.2.3 *idling oil*[2], *n*—oxidatively stable Newtonian oil injected into the operating viscometer cell when the instrument is likely to be waiting for use and held at operating temperature for more than 20 min and up to two weeks without need for replacing the idling oil.

3.2.3.1 *Discussion*—Use of this idling oil prevents formation of stator and rotor deposits of a test oil, which if left for more than 20 min at 150 °C in the instrument may begin to decompose. The idling oil this permits continuous operation of the TBS viscometer without the need to shut the instrument off when not being used for extended periods, such as overnight or over several days, if desired.

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

3.2.4.1 *Discussion*—Mechanical micrometers increase readings with rotor depth. The digital micrometer interacts with the TBS viscometer's program and permits the program to maintain the rotor height at the desired shear rate using the non-Newton reference oil of 3.2.1.2.

3.2.5 *reciprocal torque, 1/T, n*—determined 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 (programmed) mode.

3.2.6 *reciprocal torque intersection,* $1/T_i$, *n*—rotor position on the micrometer defined by the intersection of two lines generated by the reciprocal indicated torque versus rotor height for both 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.

3.2.6.1 *Discussion*—This technique of accurately establishing the shear rate is called the Reciprocal Torque Intercept Method and requires the absolute viscometry of the TBS (see 10.1.4 and Annex A2) as well as the use of both the Newtonian reference oil of 3.2.1.3 and the non-Newtonian reference oil of 3.2.1.2.

3.2.7 *rotor height (rotor position), n*—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 of the TBS.

3.2.7.1 *Discussion*—For all TBS viscometers, the rotor decreases in position and approaches contact with the stator with indicated increasing values on the mechanical or electronic micrometers.

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

3.2.9 *stored position of rotor height, n*—rotor position with the rotor 0.50 mm above the *rubbing contact* position (see 3.2.8) when the instrument is shut down.

3.2.10 *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 turns a tapered rotor closely fitted inside a matched tapered stator at a rotor-stator gap found by the Reciprocal Torque Intersection Method (see Annex A2) to provide $1.0 \cdot 10^6$ s^{-1} at 150 °C, which are the test conditions of this particular 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.[7]

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.[7]

6. Apparatus

6.1 *Tapered Bearing Simulator-Viscometer (TBS)*[2]—A patented viscometer consisting of a motor directly connected to a 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. This is the same apparatus as used in the CEC L-36-90 test method listed under "Tannas Equipment".

NOTE 1—Regarding the physics of simple flow, fluids are commonly divided into two major classes, Newtonian and non-Newtonian. Newtonian fluids follow Newton's equation of flow in which shear rate is directly proportional to shear stress and viscosity does not change with either shear rate or shear stress at constant temperature. In contrast, the shear rate of a non-Newtonian fluid is not directly proportional to shear stress and the viscosity of such a fluid is not constant with shear rate at a given temperature.

Since the shear rate and shear stress of a fluid can be directly measured if desired with the Tapered Bearing Simulator (TBS) Viscometer with no calibration with reference fluids, the TBS is an absolute viscometer and the shear rate at which it is operating can be determined during operation and adjusted to the desired value as shown in Annex A2. As such, the TBS provides non-Newtonian reference oils having known viscosities at whatever shear rate and temperature is desired such as the non-Newtonian Reference Oil, NNR-03, mentioned in 7.2 to, in this case, assure precise operation at operation at $1.0 \cdot 10^6$ s^{-1} and 150 °C. Other reference oils are available for other temperatures and shear rates.

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

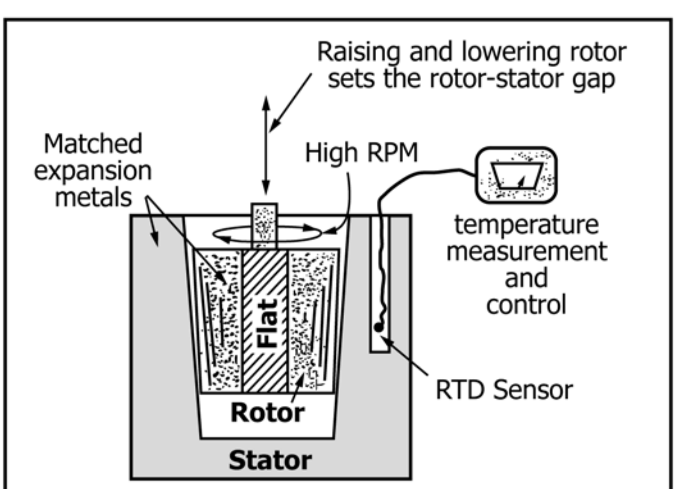


FIG. 1 Matched Tapered Stator

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 2—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 3—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.

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 µ porosity 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 4—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 <689 kPa (<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 5—It is recommended that even fresh sample be mixed by gentle stirring or inverting the closed container several times.

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 porosity filter disc on the viscometer's filling tube (see 6.6).

9. Preparation of Apparatus

9.1 *Choose and Set Up Stator Cooling Mode*—The modes are 1. none, 2. gas, 3. cooled gas, or 4. liquid mantle and are set up 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 using boiling pure water and atmospheric pressure correction, 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 *When 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 at the appropriate rotor position 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* before establishing the appropriate rotor position to provide $1.0{\cdot}10^6\ s^{-1}$ shear rate.

9.3.2 Shut power off and go to 9.4.1.

NOTE 6—Directions for preparation of the TBS viscometer and console are supplied with the equipment in the Owner's Manual. 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 Models 2100E and 2100E-F), which require bearing inspection, bearing cleanliness and low levels of bearing hysteresis are also important to obtaining reliable data.

NOTE 7—For those TBS viscometer models using ball bearings to support the motor platform (all but Models 2100E and 2100E-F 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 (by approximately 2 % or more), the bearing should be cleaned and then re-checked by the same hysteresis measurement method.

9.4 *When 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 8—Turning the motor switch off before turning the main switch on protects 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 and 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.

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

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

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

NOTE 11—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 $1.0·10^6\ s^{-1}$ 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 $1.0·10^6\ s^{-1}$ 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 16). 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.

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

NOTE 12—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 3), 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 13—The calculation of the linear regression equation from the data gathered in 11.1.3.2 is:

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

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

Then slope is:

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

and intercept is:

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

N = number of data pairs of X,Y.

The Correlation Coefficient is:

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

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:

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

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 16), 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 14—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 15—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 3). 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 16—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 17—-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 18—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[8]

15.1 *Precision*—The precision of this test method (both manual and automated protocols), which was determined by the statistical evaluation of interlaboratory results using Practice D6300, is as follows:

15.1.1 *Repeatability*—The difference between repetitive test results, obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time, 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:

$$0.01322{\cdot}X + 0.0160\ \text{mPa·s} \quad (7)$$

where:

X = the average of two successive test results, X_1 and X_2.

NOTE 19—Repeatability can be interpreted as the maximum difference between two results obtained under repeatability conditions that is accepted as plausible due to random causes under normal and correct operation of the test method.

15.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators applying the same test method in different laboratories using different apparatus 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:

$$0.03207{\cdot}X + 0.0389\ \text{mPa·s} \quad (8)$$

where:

X = the average of two single and independent test results, X_1 and X_2.

NOTE 20—Reproducibility can be interpreted as the maximum difference between two results obtained under reproducibility conditions that is accepted as plausible due to random causes under normal and correct operation of the test method.

15.1.3 *Laboratories and Oils Used in Generating Data*—Twelve laboratories submitted data on 16 fresh oils measured in duplicate (384 observations in total) used in the round robin program to obtain the precision statement. Some of these test oils were commercial engine oils. Statistical analysis using Practice D6300 was obtained on blind coded samples covering a range of viscosities from 1.47 mPa·s to 5.09 mPa·s at 150 °C at a shear rate of $1.0{\cdot}10^6\ s^{-1}$ consisting of four commercial engine oils, seven experimental (lower viscosity) engine oils, four Newtonian calibration oils, and one non-Newtonian calibration oil.

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 using Practice D6708 by comparing two other high shear rate viscometers to this test method which was chosen as the referee method.[8] These two instruments were the Tapered Plug Viscometer, Test Method D4741 and the MultiCell Capillary Viscometer, Test Method D5481. Both of these latter instruments were also tested on the same 16 oils mentioned in 15.1.3. Cross-comparing results from the original ASTM Multi-Instrument Round Robin (MIRR) and data produced in the

TABLE 2 Precision Over a Range of Mean TBS Results

Mean Result (X)	Repeatability	Reproducibility
	Difference between two results	
mPa·s	mPa·s	mPa·s
1.4	0.035	0.084
1.7	0.039	0.093
2.0	0.042	0.103
2.3	0.046	0.113
2.6	0.050	0.122
2.9	0.054	0.132
3.5	0.062	0.151
3.7	0.065	0.158
4.0	0.069	0.167
4.5	0.076	0.183
5.0	0.082	0.199

[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: RR:D02-1767.

ongoing ASTM Interlaboratory Cross Check program[9] 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 RR:D02-1698 the results from 15 ASTM InterLaboratory Cross Check programs (ILCP) showed no systematic difference (bias) between the two methods.

15.2.1.1 *Relative Bias to the Tapered Plug Viscometer (Test Method D4741)*—Cross-comparing results of the Tapered Plug Viscometer to the referee Tapered Bearing Simulator Viscometer showed a small bias for the Tapered Plug Viscometer of –0.0147 mPa·s. This correction was determined by the ASTM D02.07 Subcommittee to be inconsequential and therefore no correction to the reported result when using D4741 is required.

[9] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1698.

15.2.1.2 *Relative Bias to the MultiCell Capillary Viscometer (Test Method D5481)*—Cross-comparing results of the MultiCell Capillary Viscometer to the referee Tapered Bearing Simulator Viscometer showed no bias for the MultiCell Capillary Viscometer.

NOTE 21—A broader range of non-Newtonian behavior than in the polymer-containing oils of this study might show greater disparity between results from the MCC and the TBS viscometers. This would be more likely at lower temperatures where shear rate effects on polymer-containing, non-Newtonian oils would be expected to be greater.

16. Keywords

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

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

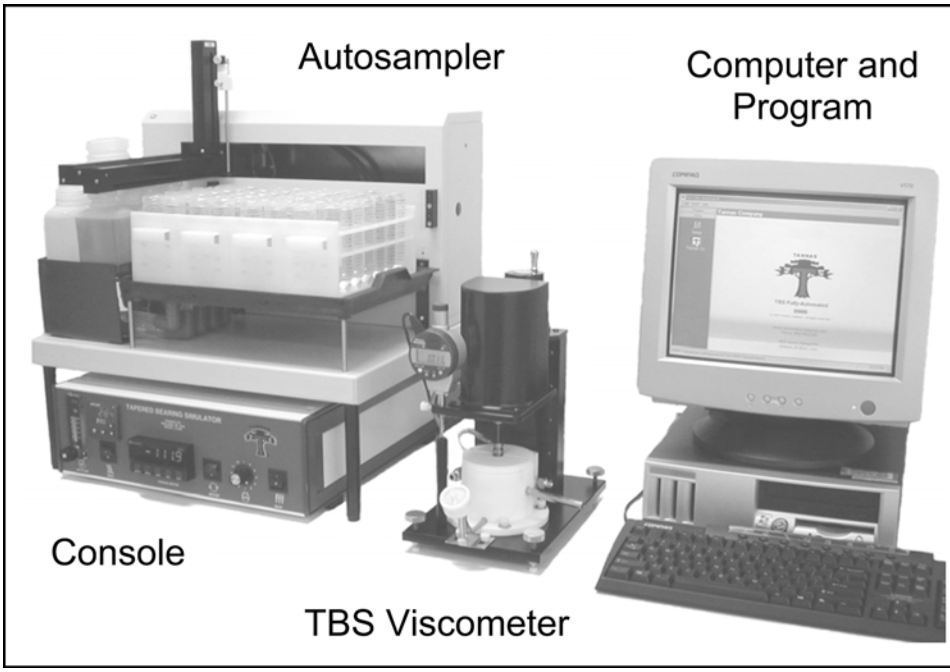


FIG. A1.1 Fully Automated TBS Viscometer Model 2100 EF Showing Control Console, Autosampler, TBS Viscometer, and Computer

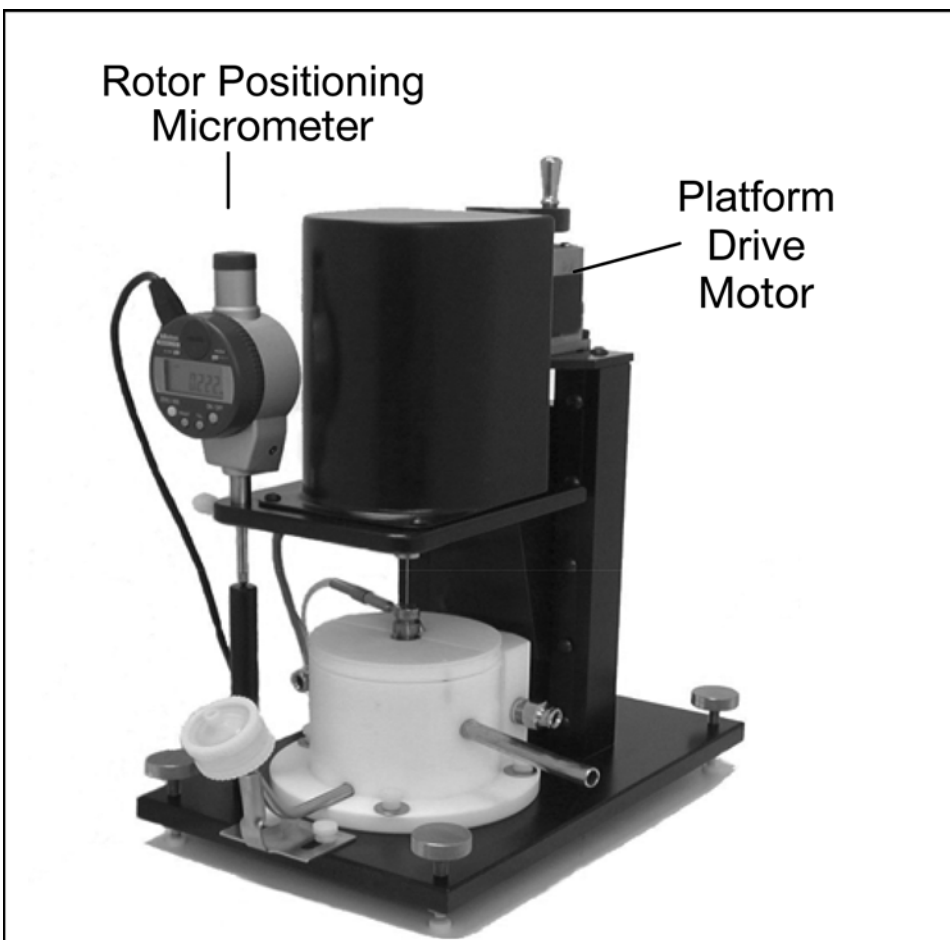
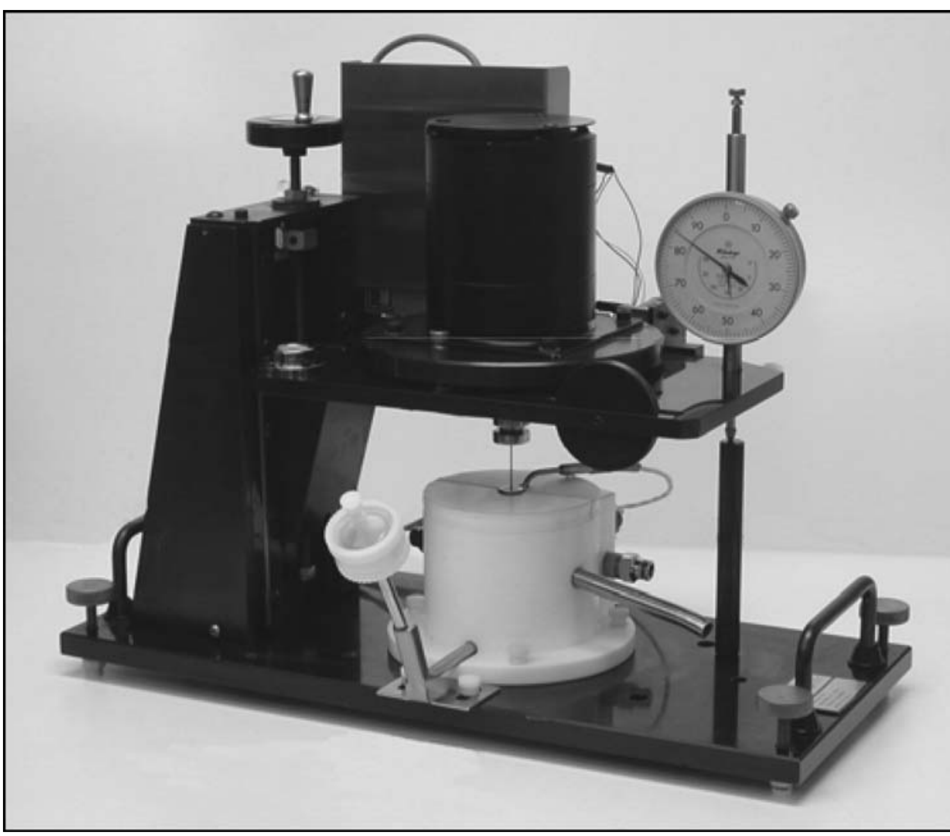


FIG. A1.2 TBS Viscometer Model 2100 EF Showing Interacting Rotor Positioning Micrometer and Platform Drive Motor, with Both Devices Controlled by Computer Program

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



NOTE 1—Earlier model has motor with less torque than later models but permitting analysis of fluids with viscosities up to 17 mPa·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.[10] 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:

$$\eta = \tau/\gamma \tag{A2.1}$$

where:

η = dynamic viscosity,
τ = shear stress, and
γ = shear rate.

As shown in Eq A2.1, at a given temperature, the ratio of shear stress to shear rate—and thus, viscosity—is always constant. This is the definition of a Newtonian fluid (see 3.1.2).

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

$$\gamma \alpha \tau \tag{A2.2}$$

Lowering and raising the rotor in the stator changes the fluid-filled gap between them in which gap the fluid's viscosity is measured.

NOTE A2.1—Since contact between the rotor and the stator walls can occur below a certain rotor depth, care is taken to always operate the viscometer above this maximum rotor depth.

A2.1.2.1 *Shear Rate and Rotor-Stator Gap*—Shear rate is defined as:

$$\gamma = V/D \tag{A2.3}$$

where:

V = the difference in velocity of one surface moving parallel to another surface d units of distance apart.

Shear rate is accordingly expressed in reciprocal seconds, s^{-1} (see 3.1.4).

From Eq A2.3, at constant rotor velocity and rotor height above contact with the stator, shear rate is constant. This shear rate can be readily changed in operation by precisely raising and lowering the rotor in the stator thus opening and closing the rotor-stator gap. Moreover, raising and lowering the slightly tapered rotor changes the shear rate, γ, inversely proportionately to the change in rotor height, H, above stator contact:

$$\gamma \alpha (1/H) \text{ and thus } H \alpha 1/\gamma \tag{A2.4}$$

Substituting from Eq A2.2 gives a relationship between rotor height (above stator contact) and the indicated torque required to turn the rotor at that height:

$$H - H_c = k_1(1/\tau) = k_2(1/T_V) \tag{A2.5}$$

where:

H_c = rotor height at stator contact,
k_1 and k_2 = constants, and
τ = shear stress expressed as the indicated torque T_V, experienced by the rotor spinning at surface velocity V as it responds to the viscous friction of a Newtonian or non-Newtonian fluid in the rotor-stator gap.

Accordingly, the latter term, $1/T_V$, is called *reciprocal torque*. This relationship among (*a*) rotor height, (*b*) rotor distance from the stator's parallel wall and (*c*) the shear rate, permits determination of several important variables.

A2.1.2.2 *Direct Determination of Operating Shear Rate*—Eq A2.5 can be stated as:

$$H = k_2(1/T_V) + H_c \tag{A2.6}$$

To set any desired shear rate, the viscometer must be operating at the desired temperature. It is necessary to determine the value of H_c from which to determine the value of H for the desired shear rate. The constant H_c is simply determined for a given rotor and stator by obtaining several values of T_V at different rotor heights H_x above rotor-stator contact, H_c. These values of H_x are used to empirically establish the two constants k_2 and H_c of Eq A2.6.

H_c is the rotor height at stator contact (where $1/T_V = 0$). From value of H_c and the known taper of the rotor-stator cell the value of H required to give the desired shear rate is:

$$\text{Shear rate} = \gamma = (S_r - R_r) \cdot V_r = (t_p \cdot \Delta H) \cdot V_R \tag{A2.7}$$

where:

S_r = stator radius,
R_r = rotor radius,
t_p = rotor-stator taper, and
ΔH = $H - H_c$, the difference in rotor height required to give the desired rotor/stator gap, $(S_r - R_r)$ at a rotor-surface velocity of V_R.

A2.1.3 *Non-Newtonian Reference Oils*—In contrast to a Newtonian fluid where viscosity is constant at all shear rates at a given temperature (see Note 1), the viscosity of a non-Newtonian fluid is dependent on shear rate at a given temperature (see Note 1). Since, as shown in A2.1.2, shear rate in the TBS can be directly established by the viscometer from Newtonian principles, the instrument does not require calibration fluids and is thus considered absolute.[10]

[10] 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.

Knowing the rotor-stator gap, rotor speed and taper, the viscosity or a non-Newtonian fluid can be established at a desired shear rate and temperature.

A2.1.4 *Use of NNR-03 and R-400 to set Rotor Height*—At the known shear rate of $1.0 \cdot 10^6\ s^{-1}$ and 150 °C, the non-Newtonian Reference Oil NNR-03 has been blended to have the same viscosity as the Newtonian Reference Oil R-400. Thus, the equivalence of the two reference oils can be used to show with certainty that the TBS viscometer is operating at the appropriate shear rate.

A2.1.4.1 Once the appropriate shear rate has been established at the rotor height at which NNR-03 gives a torque value exactly matching R-400 the viscometer is functioning at $1.0 \cdot 10^6\ s^{-1}$ at 150 °C. In practice, this torque value of NNR-03 is periodically used during operation of the TBS viscometer to check that the rotor-stator gap is still producing the desired shear rate (see Section 12 of the method).

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D4683 – 13) that may impact the use of this standard. (Approved Jan. 1, 2017.)

(1) Revised subsection 6.1.

(2) Added new Referenced Documents in 2.2 and 2.3.