



Designation: ~~D4683-04~~ Designation: D4683 - 09

# ~~Standard Test Method for Measuring Viscosity at High Shear Rate and High Temperature by Tapered Bearing Simulator~~ Measuring Viscosity of New and Used Engine Oils at High Shear Rate and High Temperature by Tapered Bearing Simulator Viscometer at 150 °C<sup>1</sup>

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 ( $\epsilon$ ) 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  $1 \times 10^6$  s<sup>-1</sup> shear rate using a tapered bearing simulator viscometer (TBS Viscometer) using a viscometer having a slightly tapered rotor and stator called the Tapered Bearing Simulator (TBS) Viscometer.<sup>2</sup> equipped with a refined thermoregulator system. Older TBS units not so equipped must use Test Method ~~D4683-87~~.~~  
1.1 This test method covers the laboratory determination of the viscosity of engine oils at 150 °C and  $1 \times 10^6$  s<sup>-1</sup> shear rate using a viscometer having a slightly tapered rotor and stator called the Tapered Bearing Simulator (TBS) Viscometer.<sup>2</sup> equipped with a refined thermoregulator system. Older TBS units not so equipped must use Test Method ~~D4683-87~~.

1.2 ~~The Newtonian calibration oils used to establish this test method cover the range from approximately 1.5 to 5.6 cP (mPa·s) at 150°C.~~  
1.2 The Newtonian calibration oils used to establish this test method cover the 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 this test method has a viscosity of approximately 3.5 cP (mPa·s) at 150°C and a shear rate of  $1 \times 10^6$  s<sup>-1</sup>.~~  
1.3 The non-Newtonian reference oil used to establish this test method has a viscosity of approximately 3.5 cP (mPa·s) at 150°C and a shear rate of  $1 \times 10^6$  s<sup>-1</sup>.

1.4 ~~Applicability to petroleum products other than engine oils has not been determined in preparing this test method.~~  
1.4 Manual, semi-automated, and fully automated viscometers were used in developing the precision statement for this test method.

1.5 ~~This test method uses the centipoise (cP) as the unit of viscosity. For information on the equivalent SI unit, the millipascal second (mPa·s) is shown in parentheses.~~  
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 ~~for this test method has a viscosity closely held to 3.55 mPa·s at 150 °C.~~  
1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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

1.8 ~~Application to petroleum products other than engine oils has not been determined in preparing the viscometric information for this test method.~~  
1.8 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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

1.10 ~~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:<sup>3</sup>

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

<sup>1</sup> 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.

Current edition approved Feb. 1, 2004. Published March 2004. Originally approved in 1987. Last previous edition approved in 1996 as ~~D4683-96~~ D4683-04. DOI: 10.1520/D4683-04.

<sup>2</sup> 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, which you may attend.

<sup>3</sup> 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. Terminology

#### 3.1 Definitions:

3.1.1 *density*—the mass per unit volume. In the SI, the unit of density is the kilogram per cubic metre, but for practical use a submultiple is more convenient. The gram per cubic centimetre is  $10^{-3}$  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  $10^3 \text{ kg/m}^3$  and is customarily used.

3.1.2 *Newtonian oil or fluid*—an oil or fluid 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*—an oil or fluid liquid that exhibits a viscosity that varies with changing shear stress and shear rate.

3.1.4 *shear rate*—the velocity gradient in fluid flow. The SI unit for shear rate is the reciprocal second (s<sup>-1</sup>).—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<sup>-1</sup>.

3.1.5 *shear stress*—the motivating force per unit area for fluid flow. The *area* is the area under shear.—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*—the ratio between the applied shear stress and rate of shear. It is sometimes called the coefficient of dynamic viscosity. This coefficient is thus a measure of the resistance to flow of the liquid. In the SI the unit of viscosity is the pascal-second; for practical use, a submultiple, millipascal second, is more convenient. The centipoise is 1 mPa·s and is customarily used.—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).

3.1.6.1 *apparent viscosity*—the determined viscosity obtained by this test method.—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*—the ratio of the viscosity to the density of the liquid. It is a measure of the resistance to flow of a liquid under gravity. In the SI the unit of kinematic viscosity is the metre squared per second; for practical use, a submultiple (millimetre squared per second) is more convenient. The centistoke (cSt) is 1 mm<sup>2</sup>/s and is customarily used.—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 oils*<sup>2</sup>—Newtonian oils used to establish the reference framework of viscosity versus torque from which is determined the test oil viscosity.

3.2.1 *calibration and reference oils*<sup>2</sup>—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*<sup>2</sup>—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*<sup>2</sup>—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*<sup>2</sup>—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 *contact position*—the rotor height when in rubbing contact with the stator. *filter*<sup>2</sup>—special filter for removing particles from the injected test oil that might damage the rotor-stator interface.

3.2.3 *idling oil*<sup>2</sup>—an oxidatively stable Newtonian oil used to minimize deposits on the rotor/stator operating surfaces when the instrument is held for long periods of time at operating temperatures of 150 °C at which other oils may in reasonably short time decompose and leave residues.

3.2.4 *non-Newtonian reference oil*<sup>2</sup>—a specially selected non-Newtonian reference oil required to establish the proper gap between the rotor and stator to produce an operating shear rate of  $1 \times 10^6 \text{ s}^{-1}$ .—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 intersection, R<sub>ti</sub>*—the 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  $1 \times 10^6$  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,  $1/T$ —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  $1.0 \cdot 10^6 \text{ s}^{-1}$  shear rate.

3.2.6 rotor height (rotor position)—the vertical position of the rotor relative to the stator and measured by the platform micrometer.

3.2.6.1 Discussion—For most instruments, a mechanical micrometer is used; the micrometer reading *increases* as the rotor is lowered and approaches the stator. However, if an electronic micrometer is used, the micrometer reading *decreases* when the rotor is lowered.

3.2.7 stored position—the rotor position with the rotor 0.50 mm above the contact position. reciprocal torque intersection,  $1/T_j$ —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  $1.0 \cdot 10^6 \text{ s}^{-1}$ .

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

## 4. Summary of Test Method

4.1A motor drives a tapered rotor that is closely fitted inside a matched stator. The rotor exhibits a reactive torque response when it encounters a viscous resistance from an oil that fills the gap between the rotor and stator. Two oils, a calibration oil and a non-Newtonian reference oil, are used to determine the gap distance between the rotor and stator so that a shear rate of  $1 \times 10^6$

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  $1.0 \cdot 10^6 \text{ s}^{-1}$  is maintained. Additional calibration oils are used to establish the viscosity/torque relationship which is required for the determination of the apparent viscosity of test oils at  $150^\circ\text{C}$ . at  $150^\circ\text{C}$ , 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

5.1Viscosity at the shear rate and temperature of this test method is thought to be representative of the condition encountered in the bearings of automotive engines in severe service.

5.2The importance of viscosity at these conditions to engine lubrication has been addressed in many publications.

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.<sup>4</sup>

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.<sup>4</sup>

## 6. Apparatus

6.1 *Tapered Bearing Simulator-Viscometer*<sup>2</sup> (Tapered Bearing Simulator-Viscometer (TBS)<sup>2</sup>—A patented viscometer consisting of a motor directly connected to a slightly tapered rotor that fits into a matched tapered stator (see Fig. 1)—a viscometer

<sup>4</sup> For a comprehensive review, see “*The Relationship Between High-Temperature Oil Rheology and Engine Operations*,” ASTM Data Series Publication 62.

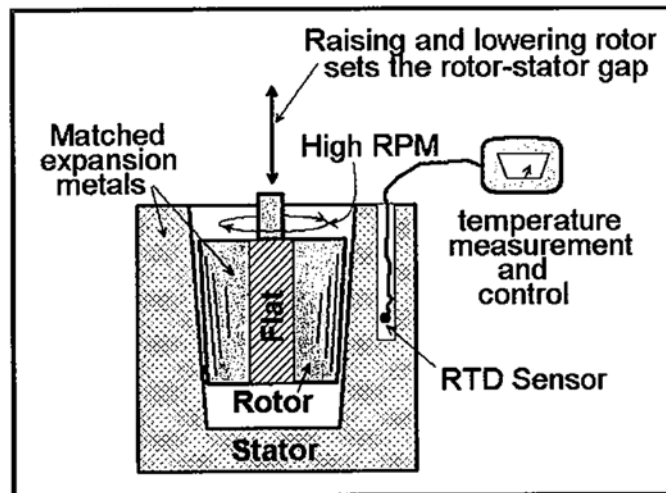


FIG. 1 Matched Tapered Stator

consisting of a synchronous two-speed motor that drives a slightly tapered bearing in a matched stator (Fig. 2). The reaction torque of the rotor to the liquid in the cell is measured and used to calculate 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).

6.1.1 The motor and rotor are raised and lowered by means of a platform, which, in turn, is cantilevered from an elevator device. The gap between the rotor and stator is controlled by adjustment of the platform height.

6.1.2 The resistive force of the test oil is transferred to the load cell by the turntable on which the motor sits. This turntable has a projecting arm on which is mounted a contact ball. The rotor is spun by the motor at a constant speed of 50 or 60 r/s depending on the frequency of the alternating current. When the rotor encounters viscous resistance, the reactive force presses the ball against the platen of the load cell to register the resistance given by the viscosity of the oil.

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.

NOTE1—An automated system for the TBS Viscometer has been developed employing all the steps in the procedure, and it was used for some of the round robin data generated for this test method.

6.2 Console—The console shown in Fig. 3 contains the power source for the load cell, thermoregulator circuit, heating coil, and motor. It also contains the circuitry for regulating and monitoring the temperature of the oil in the test cell, as well as the amplifier and digital readout of the load cell response. 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.

NOTE2—The thermoregulator circuit of the TBS Viscometer has evolved as improvements have been made in the solid-state temperature controller and heater. To achieve the five-minute analysis time specified in this test method requires a late model solid-state controller with automatic reset coupled to a thermofoil heater with small heat inertia or a fast-responding thermoregulated oil bath.<sup>2</sup> 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.<sup>2</sup>

6.3 Air Circulation System—A flow of dry compressed air is passed around the stator to provide supplementary cooling when testing fluids of higher viscosity (greater than approximately 9 cP). Ports are provided in the stator housing for the circulation of compressed air. 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 Syringe, glass or polypropylene (in the latter case, use a non-lubricated plunger), equipped with Luer needle lock to fit the tip of the filling tube for injection of test oil into the annulus between the rotor and the stator. 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 *Filter*—A filter is used on the syringe to remove particles capable of damaging the rotor/stator cell.<sup>2</sup> *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.<sup>2</sup>

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<sup>2</sup> 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*<sup>2</sup> are Newtonian oils of known kinematic viscosity and density at 150°C. The defined viscosities in centipoise (mPa·s) are calculated by multiplying the kinematic viscosity in centistokes by the density in grams per cubic centimetre. Approximate viscosities for the calibration oils are listed in Table 1. These are Newtonian oils of known dynamic viscosity at 150 °C (see 3.2.1). Table 1 : Certified viscosities are supplied with each oil. 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 1.0·10<sup>6</sup> s<sup>-1</sup> 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,<sup>2</sup> 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).

7.3 *Non-Newtonian Reference Oil*<sup>2</sup> is essential in setting the rotor/stator gap to 1×10<sup>6</sup> s<sup>-1</sup> shear rate. An approximate viscosity of a suitable non-Newtonian reference oil is given in Table 1. The certified viscosity at 1×10<sup>6</sup> s<sup>-1</sup> and 150°C is supplied with the oil and is matched to the viscosity of reference fluid R-400 (see Table 1).

8. Sampling

8.1A representative sample of test oil is obtained. When used oils are evaluated, it is desirable to change the filter attached to the syringe periodically to reduce injection pressure caused by particle buildup on the filter surface. 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 3— Precision for used oils has not been determined. 4—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.

TABLE 1 Calibration and Reference Oil Viscosities at 150.0 °C

Code No.	Viscometric Characteristics	Nominal Viscosity <sup>A</sup> cP (mPa·s) at 150°C								
Reference Oil	R-100	Nominal Viscosity <sup>A</sup> cP (mPa·s) at 150°C								
	R-200	Newtonian	1.9							
	R-300	Newtonian	2.8							
	R-300	R-350	2.8							
	R-400	Newtonian	3.5 <sup>B</sup>							
	R-400	R-450	3.5 <sup>B</sup>							
	R-500	Newtonian	5.3							
	R-500	R-600	NNR-03							
	NNR-03	non-Newtonian	~1.5	~1.8	~2.7	~3.55	~4.1	~5.0	~7.7	~3.55 <sup>C</sup>
Viscosity, mPa·s	~1.2	~1.5	~1.8	~2.7	~3.55	~4.1	~5.0	~7.7	~3.55 <sup>A</sup>	

<sup>A</sup> Nominal viscosity values. Consult supplier for certified values. <sup>B</sup> Matched to NNR-03. <sup>C</sup> At 10<sup>6</sup> s<sup>-1</sup> (matched rate R-400) e

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

## 9. Preparation of the Apparatus

9.1 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/stator before initial use of the viscometer.

9.2 With continuous use, a weekly room-temperature flush of the rotor/stator cell is recommended following directions in 11.4 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  $1.0 \cdot 10^6 \text{ s}^{-1}$  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*.

## 10. Calibration

10.1 Proceed to Section

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-checked 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  $150.0 \pm 0.1$  °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 if the operating position has already been established.

10.2 *Activating the Console*—Be sure the MOTOR switch on the console is in the OFF position. Then, turn on the POWER switch. Leave the console in this standby condition for at least 1 h before using the tapered bearing simulator viscometer.

10.3 *Oil in Test Cell:*

10.3.1 If there is no oil in the test cell, slowly inject 50 mL of the idling oil or other suitable oxidation-resistant fluid.

10.3.2 When there is oil in the test cell, proceed with the determination of the stored position as described in 10.4. If this position has been determined, proceed to 10.5.

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

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

10.4 *Determining the Stored Position*

10.1 *Manual TBS Viscometer Method:*

10.4.1 Bring the operating temperature to 150 °C by setting the thermostat on the console.

10.4.2 Be careful not to touch the hot upper stator surface when the following operation is performed. Slowly lower the rotor into the stator by means of the height adjustment wheel on the elevator assembly while turning the flexible shaft connecting the motor and the rotor with the fingers until slight rubbing contact is felt between the rotor and the stator. Then slowly continue to lower the rotor in small increments (approximately  $\frac{1}{10}$  of the smallest division or 0.001 mm until further turning is prevented (without forcing rotation)). This is the point of rubbing contact. Record the micrometer reading to the third decimal place (that is, estimate the last place from the needle position between the minor division marks). *All subsequent readings of the micrometer dial will be to the nearest 0.001 mm.*