This document is not an ASTM standard and is intended only to provide the user of an ASTM standard an indication of what changes have been made to the previous version. Because it may not be technically possible to adequately depict all changes accurately, ASTM recommends that users consult prior editions as appropriate. In all cases only the current version of the standard as published by ASTM is to be considered the official document.



Designation: D4683 - 10 D4683 - 13

# 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<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 ( $\varepsilon$ ) 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  $1.0 \cdot 10^6 \text{ s}^{-1}$  using a viscometer having a slightly tapered rotor and stator called the Tapered Bearing Simulator (TBS) Viscometer.<sup>2</sup>

1.2 The Newtonian calibration oils used to establish this test method range from approximately 1.2 mPa·s <u>1.2 mPa·s</u> 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  $1.0 \cdot 10^6 \text{ s}^{-1}$  for this test method has a viscosity closely held to 3.55 mPa·s at  $\frac{150 \text{ °C}}{150 \text{ °C}}$  by using the absolute viscometry of the TBS.

1.4 Manual, semi-automated, and fully automated <u>TBS</u> 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 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. ASTM D4683-13

2. Referenced Documents ai/catalog/standards/sist/407b715a-bf37-4955-a0db-ab5a5e6db35f/astm-d4683-13

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

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

#### 3. Terminology

3.1 Definitions:

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

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959. United States

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

Current edition approved Oct. 1, 2010Oct. 1, 2013. Published November 2010November 2013. Originally approved in 1987. Last previous edition approved in 20092010 as D4683D4683 - 10.-09. DOI: 10.1520/D4683-10.1520/D4683-13.

<sup>&</sup>lt;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,<sup>1</sup> which you may attend.

<sup>&</sup>lt;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.



3.1.1 *density*—*apparent viscosity, n*—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 viscosity of a non-Newtonian liquid determined by this test method at a particular shear rate and shear stress.<sup>3</sup>-kg/m<sup>3</sup>-

3.1.2 *Newtonian oil or <u>fluid</u>\_liquid, n*\_oil or liquid that at a given temperature exhibits a constant viscosity at all shear rates or<u>and</u> shear stresses.

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

3.1.4 *shear rate—<u>rate, n</u>*velocity gradient in liquid flow in millimetres per second per millimetre (mm/s per mm). The SI 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—<u>stress, n</u>force per unit area causing liquid flow. The flow over unit area noted is the area over which where viscous shear is being caused; in SI, the unit of shear stress is the Pascal (Pa).

3.1.6 *viscosity*—*viscosity*, *n*—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\_viscosity. Viscosity is a measure of the resistance to flow of the liquid. In the SI 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), or aswhich has the English system equivalent, equivalent of the centipoise (cP).

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*<sup>2</sup>—, <u>n</u>—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>—, <u>n</u>—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  $oil^2$ —, <u>n</u>—NNR-03—An oil specially formulated <u>for</u>, and critical to, this test method which method. The reference oil NNR-03 produces a selected value of apparent viscosity at a desired <u>temperature and</u> shear rate <u>or(see</u> Note <u>1</u>shear stress.).

3.2.1.3 Newtonian reference  $oil^2$ —, <u>n</u>—R-400—A specially formulated Newtonian oil that has the same viscosity at 150 °C as the non-Newtonian reference oil <u>NNR-03</u> of <u>3.2.1.2</u><del>3.2.1.3.</del>

3.2.2 *filter<sup>2</sup>—, <u>n</u>*-special filter for removing particles <u>potentially damaging to the rotor-stator interface</u> from the <del>injected</del>-test oil that might damage the rotor-stator interface.being injected</del>.

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

#### 3.2.3.1 Discussion-

Use of this <u>idling</u> oil prevents formation of stator <del>deposits</del> from the liquid, which may begin to decompose after exposure times greater than 20 min and rotor deposits of a test oil, which if left for more than 20 min at 150 °C in the <del>operating viscometer and</del> <u>instrument may begin to decompose. The idling oil this permits continuous operation of the TBS viscometer without the need to shut the instrument <del>off.off</del> when not being used for extended periods, such as overnight or over several days, if desired.</u>

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



Mechanical micrometers increase readings with rotor depth. <del>Digital micrometers interact</del> <u>The digital micrometer interacts</u> with the TBS viscometer programs.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,  $\frac{1}{T}$  <u> $\frac{1}{T}$ , n</u>—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,  $1/T_i$ , <u>n</u>-rotor position on the micrometer defined by the intersection of two straight lines. These are 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<sup>-1</sup> 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 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<sup>-1</sup>.

3.2.7 rotor height (rotor <u>position)</u>—<u>position)</u>, <u>n</u>—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 <u>of the TBS</u>.

3.2.7.1 Discussion-

For those TBS viscometers equipped with a mechanical micrometer (Models 400, 450, 500, 600 and SS) the rotor height decreases all TBS viscometers, the rotor decreases in position and approaches contact with the stator with increasing indicated increasing 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.mechanical or electronic micrometers.

3.2.8 *rubbing contact <u>position</u>\_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 <u>height</u><u>height</u>, <u>n</u>rotor position with the rotor 0.50 mm above the *rubbing contact* position (see 3.2.93.2.8) when the instrument is shut down.

3.2.10 test oil\_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  $1.0 \cdot 10^6 \text{ s}^{-1}$  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.<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>

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

#### 6. Apparatus

6.1 *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). 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.

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  $1.0 \cdot 10^6 \text{ s}^{-1}$  and 150 °C. Other reference oils are available for other temperatures and shear rates.

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.<sup>2</sup>

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. Indards/sist/407b715a-b137-4955-a0db-ab5a566db35fastm-d4683-13

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-mL50 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>

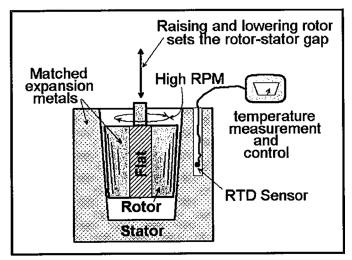


FIG. 1 Matched Tapered Stator



6.6 *Filter Assembly*—A filter holder, able to be disassembled, containing five nominal 10 micron  $\mu$  porosity 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 4-Refer to the Owner's Owner's Manual for frequency of changing filter cartridges, particularly with used engine oil.

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

### 7. Materials

7.1 *Calibration Oils*<sup>2</sup>—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 <u>Owner's Owner's</u> Manual.

7.2 Non-Newtonian Reference Oil—This reference oil is essential in setting the rotor/stator gap to  $1.0 \cdot 10^6 \text{ s}^{-1}$  shear rate (see <u>3.2.1.33.2.1.3).</u>). 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 <u>3.2.1.23.2.1.2).</u>).

7.3 Idling Oil—See 3.2.3 and the Owner's 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 manufacturer's instructions in the Owner's 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  $\leq 689 \text{ kPa}$  (<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 <u>console'sconsole's</u> 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 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 50 mL syringe or into the sampling tubes of the auto-sampling apparatus.

8.3 The <u>50-mL50 mL</u> sample is injected either by hand or by the auto-sampling apparatus through the special  $\frac{10-\mu}{10 \mu}$  porosity filter disc on the viscometer's filling tube (see 6.6).

# 9. Preparation of Apparatus catalog/standards/sist/407b715a-bf37-4955-a0db-ab5a5e6db35f/astm-d4683-13

9.1 *Choose and Set Up Stator Cooling Mode*—None, gas, <u>The modes are 1. none, 2. gas, 3.</u> cooled gas, or <del>liquid mantle, 4. liquid mantle and are set up in accordance with the manufacturer's manufacturer's</del> directions in the <del>Owner's</del> <u>Owner's</u> Manual.

9.2 Check the accuracy of the RTD (Resistance Thermometric Device) as directed in the Owner's Manual and, if necessary, 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 <u>*HWhen 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  $1.0 \cdot 10^6 \text{ s}^{-1}$  shear rate.</u>

9.3.1 Follow the manufacturer's manufacturer's instructions in the Owner's 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*. *contact* before establishing the appropriate rotor position to provide  $1.0 \cdot 10^6 \text{ s}^{-1}$  shear rate.

9.3.2 Shut power off and go to 9.4.1.

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

#### TABLE 1 Nominal Reference Oil Viscosities at 150.0 °C

Reference Oil R-	-100 F	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 <sup>A</sup>

<sup>A</sup> Value at 1.0.10<sup>6</sup> s<sup>-1</sup>-1 shear rate



NOTE 7—For those TBS viscometer models using ball bearings to support the motor platform (all but Models  $\frac{2100 \text{ Er}}{2100 \text{ Er}}$  which have no bearings), bearing hysteresis should be checked every few months according to the <del>Owner's</del><u>Owner's</u> Manual and if the values of increasing and decreasing torque by this hysteresis analysis are significantly different (-2%), (by approximately 2 % or more), the bearing should be cleaned and then re-eneckedre-checked by the same measurement method for hysteresis. hysteresis measurement method.

9.4 <u>*HfWhen*</u> 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 <u>offOFF</u> position then turn on the main switch.

Note 8—Turning the motor switch off before turning the main switch on prevents breakage of 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 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  $\pm$  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.

## 10. Establishing Operating Position of the Rotor for 1.0.10<sup>6</sup> s<sup>-1</sup> 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  $\frac{\text{Owner's}Owner's}{\text{Owner's}}$  Manual for setting the rotor-stator gap for  $1.0 \cdot 10^6 \text{ s}^{-1}$  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  $1.0 \cdot 10^6$  s<sup>-1</sup> shear rate according to the <del>Owner's</del>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  $1.0 \cdot 10^6$  s<sup>-1</sup> 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  $1.0 \cdot 10^6 \text{ s}^{-1}$  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 1516). 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 11.1.2.1 with 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  $\tau_c$ , and viscosity as variable  $\eta$ , in Eq. 1.

$$=m\cdot\tau_c+b\tag{1}$$

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  $\pm 1.5\%$  of the value on its container, proceed to 11.1.3.

11.1.2.7 If the value of NNR-03 is not within  $\pm 1.5\%$  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.