Designation: ~~D6616 – 20a~~ D6616 – 21

Standard Test Method for Measuring Viscosity at High Shear Rate by Tapered Bearing Simulator Viscometer at 100 °C[1]

This standard is issued under the fixed designation D6616; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

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

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

1.2 The Newtonian calibration oils used to establish this test method range from approximately 5 mPa·s (cP) to 12 mPa·s (cP) at 100 °C and either the manual or automated protocol was used by each participant in developing the precision statement. The viscosity range of the test method at this temperature is from 1 mPa·s (cP) to above 25 mPa·s (cP), depending on the model of TBS.

1.3 The non-Newtonian reference oil used to establish the shear rate of $1 \cdot 10^6 s^{-1}$ for this test method has a viscosity of approximately 10 mPa·s at 100 °C.

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

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard. This test method uses the milliPascal second (mPa·s) as the unit of viscosity. This unit is equivalent to the centiPoise (cP), which is shown in parentheses.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and to determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

[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.

Current edition approved ~~Sept. 1, 2020~~Jan. 1, 2021. Published ~~October 2020~~January 2021. Originally approved in 2001. Last previous edition approved in 2020 as ~~D6616 – 20.~~D6616 – 20a. DOI: ~~10.1520/D6616-20A.~~10.1520/D6616-21.

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

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

2. Referenced Documents

2.1 *ASTM Standards:*[3]

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

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

2.2 *Coordinating European Council (CEC) Standard:*[4,5]

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

2.3 *Energy Institute Standard:*[6,5]

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

3. Terminology

3.1 *Definitions:*

3.1.1 *density, n*—mass per unit ~~volume at a specified temperature.~~volume.

~~3.1.1.1 *Discussion*—~~

~~For common fuel and lubricant applications, density at atmospheric pressure is assumed. However, high pressure can impact density.~~

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

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

3.1.4 *shear rate, n*—velocity gradient perpendicular to the direction of flow.

3.1.5 *shear stress, n*—the force per unit area in the direction of the flow.

3.1.6 *viscosity, n*—the ratio between the applied shear stress and rate of shear which is sometimes called the coefficient of dynamic viscosity and is a measure of the resistance to flow of the liquid.

3.1.6.1 *apparent viscosity, n*—the viscosity of a non-Newtonian fluid at a given shear rate or shear stress determined by this test method.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *idling oil*[2], *n*—an oxidatively stable Newtonian oil injected into the operating viscometer stator when the instrument is likely to be held for periods of time greater than 30 min and up to two weeks at 100 °C. Use of this oil prevents stator deposits from additives, which may decompose after longer exposure times in the operating viscometer and permits continuous operation of the viscometer without need to shut the instrument off.

3.2.2 *Newtonian Reference Oil*[2], *n*—a specially blended Newtonian oil that has the same viscosity at 100 °C as the non-Newtonian reference oil of 3.2.3.

3.2.3 *non-Newtonian reference oil*[2], *n*—a specially formulated non-Newtonian oil, identified as NNR-10, having a selected apparent viscosity at $1 \cdot 10^6 s^{-1}$ shear rate. The oil is used to establish an operating gap between the rotor and stator which will produce $1 \cdot 10^6 s^{-1}$ shear rate when the rotor height is adjusted to give a torque output equivalent to that of the special reference oil described in 3.2.2.

3.2.4 *reciprocal torque intersection, $1/T_i$, n*—the rotor position on the micrometer defined by the intersection of two straight lines

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

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

generated by the reciprocal torque method using the Newtonian reference oil of 3.2.2 and non-Newtonian reference oil of 3.2.3. Reciprocal torque versus rotor height measurements on both oils gives straight lines whose intersection, $1/T_i$, establishes the desired rotor position for operation at $1{\cdot}10^6 s^{-1}$ shear rate.

3.2.5 *reference Newtonian calibration oils*[2], *n*—specially chosen Newtonian oils used to determine the viscosity-torque relationship of the TBS viscometer at 100 °C from which the viscosity of an unknown oil is calculated.

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

3.2.6.1 *stored rotor height (rotor position), n*—the rotor position with the rotor 0.50 mm above the rubbing contact position (see 3.2.7) when the instrument is shut down.

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

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

4. Summary of Test Method

4.1 A motor drives a tapered rotor closely fitted inside a matched tapered stator. Appropriate technique establishes operation of the viscometer to yield $1{\cdot}10^6 s^{-1}$ at a temperature of 100 °C at which point test oils are introduced into the gap between the spinning rotor and stationary stator. The rotor exhibits a reactive torque to the viscous resistance of each test oil and the value of this torque response is used to determine the apparent viscosity of the test oil at 100 °C.

5. Significance and Use

5.1 Viscosity at the shear rate and temperature of this test method is thought to be particularly representative of bearing conditions in large medium speed reciprocating engines as well as automotive and heavy duty engines operating in this temperature regime.

5.2 The importance of viscosity under these conditions has been stressed in railroad specifications.

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

6. Apparatus

6.1 *Tapered Bearing Simulator Viscometers*[2] (TBS)—A viscometer consisting of a motor connected to a slightly tapered rotor that fits into a matched stator. Several models of the TBS are in use. All of these are capable of analyzing test oils at 100 °C but earlier models are more limited in their upper viscosity range.

6.2 Different models of the tapered bearing simulator (TBS) have the following upper levels of operating viscosities at $1{\cdot}10^6 s^{-1}$ shear rate:

6.2.1 Model Series 400 (similar to Fig. 1)—~14 mPa·s (cP), dual speed.

6.2.2 Model Series 500 (Fig. 1)—~16 mPa·s (cP) single speed.

6.2.3 Model Series 600 (Fig. 2)—~100 mPa·s (cP) (usually liquid cooled), dual speed.

6.2.4 Model Series SS (SuperShear) (similar to Fig. 1)—~20 mPa·s (cP), multi-speed.

6.2.5 Model Series 2100 E (Fig. 3)—~20 mPa·s (cP) (see Note 2), multi-speed.

NOTE 2—TBS Models 500, 600, and SS use a so-called *bouncer* to automate unloading and reloading the load cell just before taking a torque reading. (All automated units apply the bouncer at the appropriate point of operation as part of their program.) If a bouncer is not on the TBS model used (Model

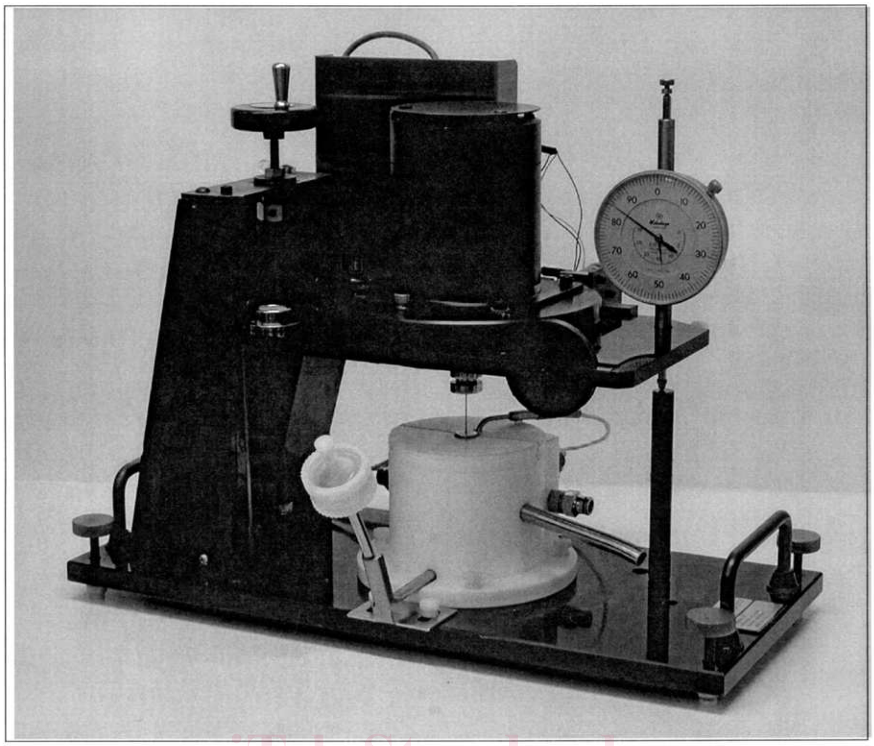

FIG. 1 Tapered Bearing Simulator Viscometer Model 500

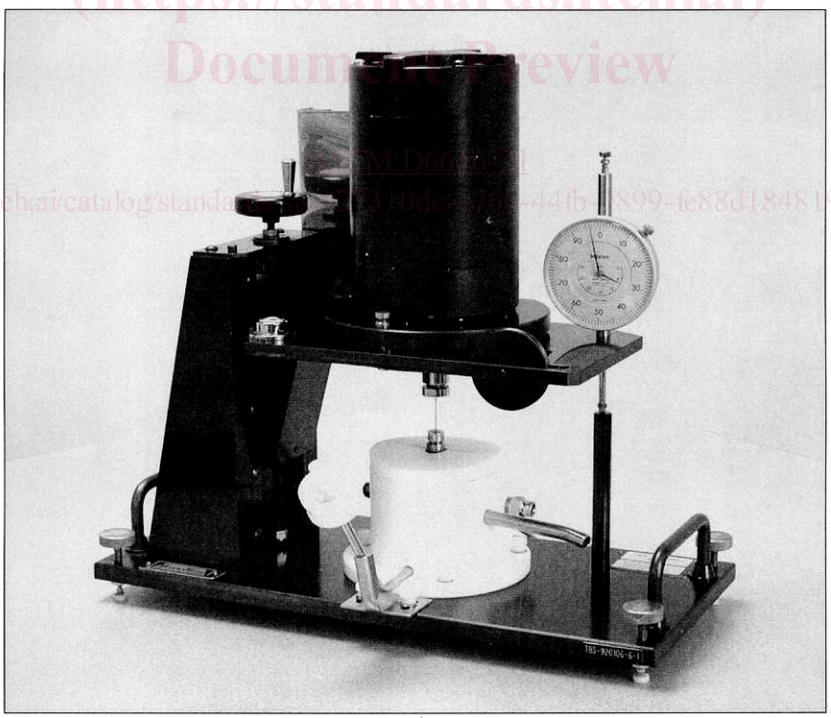

FIG. 2 High Torque Tapered Bearing Simulator Viscometer Model 600

400), the effect is generated by placing the thumb on the brass weight pin and turning the turntable slightly in a clockwise direction and quickly releasing the turntable. The bearingless Models 2100 E do not require unloading the cell since there is no turntable bearing.

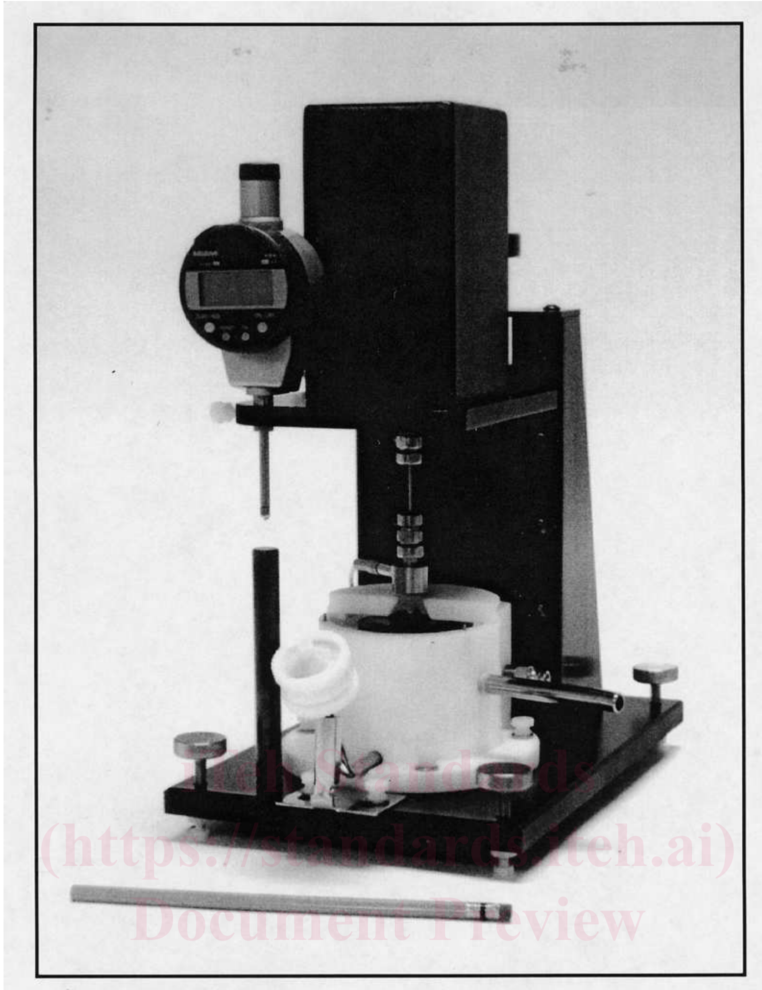

FIG. 3 Multi-Speed Tapered Bearing Simulator Viscometer Model 2100E

6.3 *Automated System for Calibration, Injection, and Data Analysis Programs*—An automated program for the Tapered Bearing Simulator, simulating the manual method has been used.

6.4 *Console*—The console shown in Fig. 4 is similar in Models 400, 500, and 600. Consoles for Series SS and 2100 E have provisions for changing motor speed. All consoles contain the power source for the load cell, thermoregulator circuit, stator-heating element, and motor. They also contain the circuitry for regulating and monitoring the temperature of the oil in the stator as well as the amplifier and digital readout of the load cell.

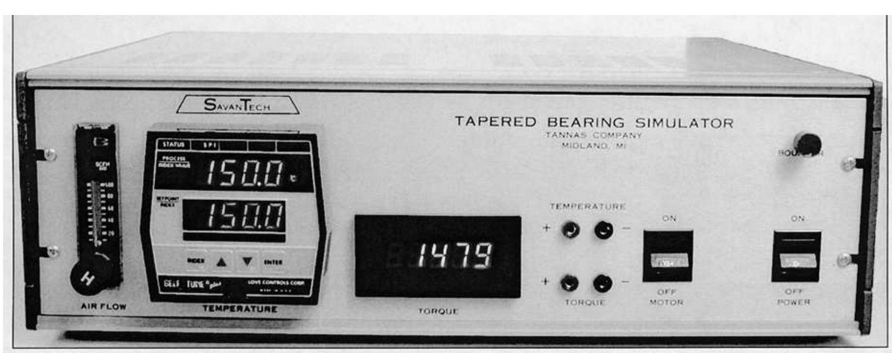

FIG. 4 Control Console for Tapered Bearing Simulator Viscometer Models 400, 500, and 600

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

6.5 *Cooling Systems*—Two cooling systems are available for TBS viscometer work at 100 °C: forced air cooling and liquid bath cooling. The stator housing is prepared for the former but must be modified for the latter according to directions from the manufacturer.

6.6 *Glass Syringe*—A 50 mL glass syringe equipped with a Luer needle lock fits the tip of the filling tube for injection of test oil into the test cell. Smaller glass and plastic syringes can be used if any air bubble in the fill tube caused by the exchange of syringes is first pulled up into the next syringe to be used.

6.7 *Filter Assembly*—A unit made of a filter holder[2] and nominal 10 µ filter[2] is interposed between the syringe and the filling tube to remove particles capable of damaging the rotor/stator cell.

6.8 *Data Recording Equipment*—Some form of recording the torque and temperature data produced by the tapered bearing simulator is desired in order to (*1*) determine torque/temperature equilibrium and (*2*) determine the torque with sufficient precision to calculate viscosity to the second decimal place. Early in the use of the TBS viscometer, a strip-chart recorder was used, later an automated, computer-based recording system was developed with both a computer-simulated strip chart and with data digitally recorded.

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

6.8.1 *Strip-chart Recorder:*

6.8.1.1 If a strip-chart recorder is used to record the torque and temperature output signals, use the manufacturer's directions for calibrating and setting up the strip chart for recording torque/temperature data (see Note 5). The torque reading must be in milliVolts and the temperature in °C with a full-scale chart range of 20 °C to 120 °C.

6.8.1.2 Use a chart speed of 1 cm/min for recording.

6.8.1.3 Set and, when necessary, reset, the strip chart torque voltage to that which will permit recording the torque as much as possible on the upper two-thirds of the chart paper for maximum sensitivity.

6.8.1.4 Factor the resulting voltage values to calculate the correct values of torque.

NOTE 5—Although the digital information from the torque output meter on the viscometer console can be, and is, used for recording additional test information, it is desirable to use a two-pen, strip-chart recorder or its computer equivalent since this provides a continuous torque/temperature record of torque/temperature equilibrium necessary for precision in calibration and in calculating viscosity.

6.8.2 *Computer Accumulation of Torque and Temperature Data*—Computer recording of digital data can also be used for the test method. Such programs should show data for both torque and stator temperature. Torque information should be capable of permitting the calculation of viscosity to the second decimal place.

7. Materials

7.1 *Reference Newtonian Calibration Oils,* [2] Newtonian oils of known dynamic viscosity at 100 °C. Table 1 shows the dynamic viscosity values of five Newtonian oils used in developing the information for this test method.

7.2 *Idling Oil*—See 3.2.1 for information and use.

7.3 *Non-Newtonian Reference Oil,* [2] essential in setting the rotor/stator gap to $1 \cdot 10^6 s^{-1}$ shear rate. The nominal level of apparent viscosity of non-Newtonian reference oil, NNR-10 used in applying this test method is given in Table 1.

7.4 *Polar Solvent,* such as dimethyl sulfoxide is used to dissolve any deposits on the rotor/stator surfaces after extended use.

TABLE 1 Reference Oil Viscosities at ~~100.0°C~~100.0 °C

Reference Oil	Characteristic	Nominal Viscosities[A]
		mPa·s at $1 \cdot 10^6 s^{-1}$
R-2200	Newtonian	~3
R-2300	Newtonian	~5
R-2350	Newtonian	~7
R-2400	Newtonian	~10
R-2450	Newtonian	~12
NNR-10[B]	Non-Newtonian	~10

[A] Contact supplier for certified value of Reference Oil.
[B] Special reference oil closely equivalent to R-2400 at a value of $1 \cdot 10^6 s^{-1}$ shear rate.

7.5 Source of moderate pressure (<100 PSI) *dry* air or nitrogen.

NOTE 6—Depending on room temperatures, higher torque levels at 100 °C and $1 \cdot 10^6 s^{-1}$ may require air or other gas cooling. Use of *dry* gas is required (to keep moisture from entering the stator housing).

8. Sampling

8.1 Fifty millilitres of a representative sample of fresh or used test oil is placed in a 50 mL syringe equipped with attached filter holder and 10 µ filter disk[2] in preparation for injection into the TBS viscometer.

NOTE 7—It is important to always use a filter and filter disk to prevent larger particles from entering the rotor-stator gap. However, it is also important to note that the TBS viscometer will work with heavily particle laden used oils as long as they are passed through the 10 µ filter.

9. Preparation of Apparatus

9.1 Set up stator cooling method, air or liquid, according to the manufacturer's directions.

NOTE 8—When analyzing relatively viscous oils, stator cooling is necessary. This is particularly the case at lower operating temperatures such as 100 °C where simple radiation from the stator through the stator housing is not sufficient to carry away the heat generated by viscous resistance to shear.

9.1.1 *Air Cooling*—Connect cooling air tubing to the ports on the stator housing and the back of the console following directions given by the manufacturer in the Owner's Manual. This will permit use of the flow meter on the left side of the console to adjust the cooling-air flow rate.

9.1.1.1 Set the airflow rate at 100 SCFH.

NOTE 9—Once airflow rate has been set, it is important that this level be maintained throughout calibration and operation. If desired, the air may be passed through a copper coil in a chilling bath containing water, ice, or dry ice, as necessary, to obtain desired stator temperature. The cooling level must be kept constant.

9.1.2 *Liquid Cooling*—Connect liquid cooling bath tubing from bath pump to the stator housing and the back of the console using insulated tubing according to the manufacturer's directions.

9.2 If some days or weeks have elapsed since last use of the TBS viscometer, follow the manufacturer's instructions regarding set-up and alignment of the rotor in the stator, checking the accuracy of the RTD and, if necessary, adjusting to 100.0 °C. Shut the power off and go to 9.3.

NOTE 10—Directions for preparation of the tapered bearing simulator viscometer and console are supplied with the equipment. One of the most important directions to be followed is the alignment of the rotor and stator before initial use of the viscometer. For those TBS Models (other than Model 2100E) requiring bearing inspection, low bearing hysteresis and bearing cleanliness are also important to obtaining reliable data.

NOTE 11—Bearing hysteresis should be checked every few months and if the values of increasing and decreasing torque are significantly different, the bearing should be cleaned and re-checked by hysteresis measurements.

9.3 If the TBS viscometer has been turned off for some period of time (>1 h), make sure the motor is off and slowly (~2 min) inject 50 mL of R-2400 into the stator while turning the rotor using the upper Siamese collet connecting the motor shaft and the drive wire slowly between the thumb and forefinger.

9.4 If the TBS Viscometer has been operating at 100 °C, proceed to Section 11 unless recalibration is desired.

9.4.1 If recalibration is desired, proceed to 10.2.

10. Calibration

10.1 If the operating position of the rotor in the stator has already been established in previous work at 100 °C, proceed to Section 12.

10.2 If the operating position of the rotor in the stator must be established from a cold start for operation at 100 °C, follow the manufacturer's instructions to find the rubbing contact position of the rotor with the stator by rotating the upper Siamese collet between the thumb and forefinger as the rotor is slowly lowered by using the elevator wheel (see Note 12).

NOTE 12—The indicator dial reading *decreases* when the rotor and platform are raised and vice-versa. Exercise care in using the TBS elevator wheel to move the rotor in the direction intended by the indicating arrow on top of the wheel (clockwise to simultaneously raise both the platform and rotor, counter clockwise to lower both).

10.2.1 After finding the rubbing contact position, turn the elevator wheel clockwise (see Note 12) to *raise* the platform and the rotor by decreasing the depth indicator dial reading by 0.5 mm.

10.2.2 After making sure that the MOTOR switch is in the *off* position, turn on the POWER switch and permit the electronic components of the console to warm up for a suitable length of time according to the manufacturer's instructions.

10.2.3 Set the console's temperature controller to 100.0 °C and permit the stator to begin warming up to that temperature. Do *not* turn on the motor.

NOTE 13—The viscosity of the oil decreases exponentially with temperature and it is important that the motor *not* be started at higher viscosities than 20 mPa·s (cP) to 30 mPa·s (cP) to protect the wire drive shaft.

10.2.4 When the stator temperature has reached 70 °C, turn on the motor while pushing the red bouncer button on the console or gently holding the platform ball away from the load-cell platen (see Note 2) by lightly pushing clockwise on the brass pulley post set into the motor turntable. (This is not required or possible with Model 2100E.)

NOTE 14—For TBS Models 500, 600, and SS, it is good practice to always press on and hold the bouncer button, or otherwise push the contact ball away from the load-cell platen before turning the motor on or off. This use of the bouncer button prevents the initial start-up or shutdown surge of the motor from causing the contact ball to hammer on the platen of the sensitive load-cell.

NOTE 15—Sufficient warm-up time is important for consistency and precision. Operating with R-2400 adds the effect of viscous heating to the electrical heating of the stator and speeds the process.

10.2.5 When the temperature stabilizes at 100.0 °C ± 0.2 °C, go to Section 11.

10.3 If the TBS viscometer is already operating but at temperatures higher than 100 °C, set the temperature control to 100.0 °C and slowly (~1 min) inject 50 mL of idling oil into the stator through the fill tube. If using air cooling, turn the dry gas flow-meter knob on the console to maximum flow rate (100 SCFH). If using liquid cooling, start circulation.

10.3.1 When the temperature reaches 100.0 °C ± 0.2 °C and while the motor is turning, slowly (~1 min) inject 50 mL of Newtonian calibration oil, R-2400.

10.4 Maintain appropriate air or liquid cooling rate.

11. Setting the Rotor Position at $1 \cdot 10^6 s^{-1}$ Shear Rate – the Reciprocal Torque Intercept

NOTE 16—In all data collected for the determination of viscosity, torque and temperature, equilibrium is necessary. Normally, this is reached within 5 min after injection of oil having a viscosity less than about 12 mPa·s (cP) to 15 mPa·s (cP). However, with oils that are more viscous, the time to establish equilibrium may extend to 7 min or 8 min at 100 °C. Under all circumstances, equilibrium is assumed within 10 min of injection, at which point values should be taken and the next step in the method begun.

11.1 *R-2400 Newtonian Oil*—Establish the reciprocal torque information for R-2400 Newtonian oil by turning on the strip-chart recorder and slowly (~1 min) injecting 50 mL of R-2400 calibration oil.

11.1.1 Press and hold the bouncer button, then *turn off the motor* (see Note 14). Proceed to find rubbing contact of the rotor with the stator as described in 10.2.

11.1.2 After finding the rubbing contact position, use the elevator to *raise* the platform and the rotor by *~0.15 mm* to the nearest exact whole tenth (0.100) millimetre value on the depth indicator dial (see Table 2, Example).

NOTE 17—When the rotor is raised and lowered, the indicator dial needle should be read as exactly as possible (to the third decimal place by estimating between dial markings) at each position. Parallax in reading the dial indicator should be avoided by placing the eye at the same level as the dial. A magnifying glass can be further helpful.

11.1.3 While pushing the red bouncer button on the console *turn motor on* (see Note 14).

11.1.4 Hold the red bouncer button in the depressed position and adjust the torque readout to zero (*0000* on the dial) according to the manufacturer's instructions.

11.1.5 When torque/temperature equilibrium has been reached (see Note 16) at this initial position, press the bouncer button briefly (see Note 2) and release. As soon as the computer-simulated or actual strip-chart recorder again shows constant torque/temperature traces (see Note 16), record the torque value given by the data recording equipment as well as the rotor (platform) position shown by the depth indicator dial.

11.1.6 Continue with, and complete 11.1.7 through 11.1.9.4 without using bouncer button again.

11.1.7 Use the elevator to move the rotor (platform) *up* exactly 0.100 mm (see Note 17 and Table 2). Again, record the rotor (platform) position and the indicated torque after torque/temperature equilibrium is achieved (see Note 16).

11.1.8 Again raise the rotor (platform) progressively and exactly 0.100 mm (see Note 17) three more times for a total of five sets of decreasing values of indicated torque with increasing rotor (platform) height as in Table 2 (see Example) and record the torque and rotor height values.

NOTE 18—The full sequence required is shown in Table 2 and provides five sets of torque/height data for each of R-2400 and NNR-10.

11.1.9 Calculate the reciprocals of the torque values collected in 11.1 through 11.1.8 (see Table 2) for the Newtonian reference oil R-2400.

11.1.9.1 Linearly regress the rotor height (platform) position versus the *reciprocal* torque values found for these rotor heights. Record the slope, intercept, and correlation coefficient, *R*, of this best line.

11.1.9.2 The value of *R* should be equal to or greater than (≥) 0.999.

TABLE 2 Example of Torque/Height Data

Rotor (Platform) Position	Example	Torque, T		Reciprocal Torque, 1/T	
		R-2400	NNR-10	R-2400	NNR-10
Rubbing Contact	14.176 mm[A]	–	–	–	–
Up ~0.15 mm to nearest 0.1 mm	14.000 mm[A]	value	value	1/value	1/value
Up to 0.100 mm	13.900 mm[A]	value	value	1/value	1/value
Up to 0.100 mm	13.800 mm[A]	value	value	1/value	1/value
Up to 0.100 mm	13.700 mm[A]	value	value	1/value	1/value
Up to 0.100 mm	13.600 mm[A]	value	value	1/value	1/value

[A] See Note 12 for relationship between rotor (platform) position and micrometer reading

11.1.9.3 If R is less than (<) 0.999, slowly (~1 min) re-inject 50 mL of R-2400 and repeat 11.1.2 through 11.1.9.2 using special care to set and record the exact values of height to the third decimal place on the depth indicator dial (see Note 17). Recalculate the value of R.

11.1.9.4 If the value of R is now acceptable, proceed to 11.2. If the value of R is still not high enough, contact the manufacturer of the TBS.

11.2 *Non-Newtonian Reference Oil, NNR-10*—Establish the reciprocal torque information for the non-Newtonian reference oil, NNR-10 by slowly (~1 min) injecting 50 mL of non-Newtonian reference oil NNR-10 and setting the rotor (platform) height to the exact initial position previously used for the Newtonian Reference oil, R-2400 in 11.1.2 (see Note 17).

11.2.1 Establish temperature/torque equilibrium, then record the rotor (platform) position indicated by the depth indicator dial and use the bouncer button once only to initiate the series of reciprocal torque determinations. Do not use the bouncer button again while establishing the reciprocal torque values.

11.2.2 Repeat the rotor (platform) adjusting, torque-collecting sequence in 11.1.2 through 11.1.9 using identical rotor (platform) positions. Record all values of rotor (platform) position, resultant torques, and reciprocal torque values (see Notes 17 and 18).

11.2.2.1 As in 11.1.9.1, linearly regress the rotor (platform) position versus reciprocal torque values collected in 11.2 for the non-Newtonian reference oil, NNR-10. Record the slope, intercept, and correlation coefficient.

11.2.2.2 The value of R should be equal to or greater than (≥) 0.999. If the value of R is acceptable, record and proceed to 11.3.

11.2.2.3 If the value of R is less than 0.999, repeat 11.2.1 through 11.2.2.2 with particular attention to setting, reading, and recording the values of platform position and torque. If the value of R is still less than 0.999, contact the manufacturer.

11.3 *Determination of Reciprocal Torque Intersection Position for* $1{\cdot}10^6 s^{-1}$ *Shear Rate:*

11.3.1 Calculate and record the reciprocal torque value, $1/T_i$, for the intersection point of the linear equations of 11.1 and 11.2 (see Note 19 and Fig. 5). This provides the experimentally determined rotor operating position for $1{\cdot}10^6 s^{-1}$ shear rate.

NOTE 19—The linear equation obtained from 11.1 using Newtonian reference oil R-2400 is $H = m/T + b$ in which m is the slope and b the intercept and H and $1/T$ are rotor height and reciprocal torque. The linear equation obtained from 11.2 using reference oil NNR-10 is $H' = n/T' + c$ in which n is the slope and c the intercept, and H' and $1/T'$ are rotor height and reciprocal torque at the intersection point $H_i = H_i'$ as well as $1/T_i = 1/T_i'$. Setting equation

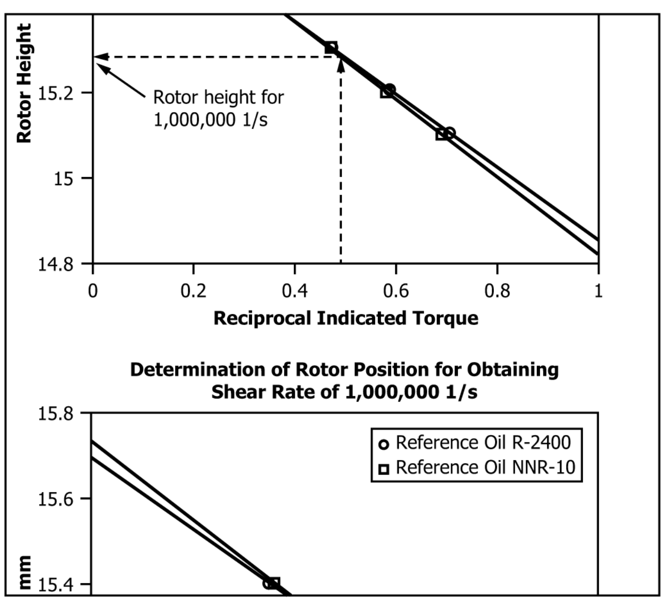

FIG. 5 Reciprocal Torque Intercept of R-2400 and NNR-10 for Setting Appropriate Rotor Position for $1{\cdot}10^6 s^{-1}$ Shear Rate

$H = m/T + b = H' = n/T' + c$: $m/T_i + b = n/T_i + c$ and $1/T_i = (c\text{-}b)/(m\text{-}n)$ substituting $1/T = (c\text{-}b)/\text{m-n})$ into $H = m/T + b$ $H_i = [\text{m(c-b)/(m-n)}] + \text{b}$; the intersection height for $1 \cdot 10^6 s^{-1}$.

12. Calibration of TBS Rotor and Stator

12.1 Set rotor position exactly to that indicated by 11.3.1 (see Note 17).

12.2 *Check Rotor Position at* $1 \cdot 10^6 s^{-1}$ *Shear Rate:*

NOTE 20—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 $1 \cdot 10^6 s^{-1}$ shear rate and it is prudent to recheck the rotor position and to make slight adjustments, if necessary.

12.2.1 Slowly (~1 min) inject 50 mL of Newtonian reference oil, R-2400, and allow for torque/temperature equilibration (see Note 16). Apply the bouncer button briefly, allow the torque value to stabilize, and record torque.

NOTE 21—In determinations of viscosity using all TBS viscometer models except Model 2100 E, it is necessary to use the bouncer button once after temperature/torque equilibrium has been established. See Note 2.

12.2.2 Slowly (~1 min) inject 50 mL of non-Newtonian reference oil, NNR-10, again allow for torque/temperature equilibration (see Note 16). Apply the bouncer button briefly, allow the torque value to stabilize, and record torque.

12.2.3 Calculate the NNR-10/R-2400 viscosity ratio from the values on the container.

12.2.4 If the torque ratio NNR-10/R-2400 is within ±0.015 of the viscosity ratio of 12.2.3, proceed to 12.3.

12.2.5 If the torque ratio NNR-10/R-2400 is *greater than* 1.015, *lower* the rotor (platform) 0.010 mm for each 0.010 unit of ratio greater than 1.000 (see Notes 12 and 17); repeat 12.2.1 and 12.2.2, and recalculate the NNR-10/R-2400 torque ratio.

12.2.5.1 If the NNR-10/R-2400 torque ratio is still greater than 1.015, repeat 12.2.1 and 12.2.2 until the proper value is obtained.

12.2.6 If the NNR-10/R-2400 torque ratio is *less than* 0.985, raise the rotor (platform) 0.010 mm for each 0.010 unit of ratio less than 1.000 (see Notes 12 and 17), repeat 12.2.1 and 12.2.2, and recalculate the NNR-10/R-2400 torque ratio.

12.2.6.1 If the NNR-10/R-2400 torque ratio is still less than 0.985, repeat 12.2.1 and 12.2.2 until the proper value is obtained.

NOTE 22—The test method given in 12.2 is a quick and precise method of readjusting rotor (platform) position to the appropriate shear rate and can be used at any time.

12.3 Simultaneously initiate the calibration and recheck the operationally correct rotor (platform) position by slowly injecting Newtonian reference oil R-2200 and waiting until torque/temperature equilibrium is obtained (see Section 15). Use the bouncer button after torque/temperature equilibrium, allow the torque value to stabilize, and record torque.

12.3.1 Repeat 12.3 for Newtonian reference oil R-2450 and record resulting torque.

12.3.2 Use the known viscosities of Newtonian reference oils R-2200 and R-2450 and the torque values from 12.3 and 12.3.1 to calculate the slope, m_v, and intercept, b_v, of the line connecting these two pairs of values with indicated torque as variable T_i, and viscosity as variable V, in Eq 1.

$$V = m_V \cdot T_i + b_V \quad (1)$$

12.3.3 Use the value of torque for non-Newtonian reference oil NNR-10 from 12.2.3 and substitute it into Eq 1. Calculate the value of the viscosity of NNR-10 and compare to the viscosity value of NNR-10 on the container. If the viscosity value is within ±2 % of the value on the container, proceed to 12.4.

12.3.3.1 If the value of NNR-10 is not within ±2 % of the container value, first check the NNR-10/R-2400 torque ratio by slowly (~1 min) injecting 50 mL of R-2400 (see 12.2.1), obtaining the torque value. Then use the last determined value of NNR-10 from 12.2.2 to re-calculate the NNR-10/R-2400 torque ratio.

(1) If the NNR-10/R-2400 torque ratio is within 1.000 ± 0.015, return to 12.3 and re-run 12.3 to 12.3.3.