Standard Test Method for Measuring Viscosity at High Shear Rate and High Temperature by Tapered Bearing Simulator[1]

This standard is issued under the fixed designation D 4683; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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

1. Scope

1.1 This test method covers the laboratory determination of the viscosity of engine oils at 150°C and 1×10^{6} s^{-1} shear rate using a tapered bearing simulator-viscometer (TBS Viscometer) [2]equipped with a refined thermoregulator system. Older TBS units not so equipped must use Test Method D 4683 – 87.[3]

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.

1.3 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 1×10^{6} s^{-1}.

1.4 Applicability to petroleum products other than engine oils has not been determined in preparing 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.

1.6 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 4741 Test Method for Measuring Viscosity at High Temperature and High Shear Rate by Tapered-Plug Viscometer[3]

D 5481 Test Method for Measuring Apparent Viscosity at High-Temperature and High-Shear Rate by Multicell Capillary Viscometer[3]

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} kg/m^{3} and is customarily used.

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

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

3.1.5 *shear stress*—the motivating force per unit area for fluid flow. The *area* is the area under shear.

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.

3.1.6.1 *apparent viscosity*—the determined viscosity obtained by this test method.

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^{2}/s and is customarily used.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *calibration oils*[2]—Newtonian oils used to establish the reference framework of viscosity versus torque from which is determined the test oil viscosity.

3.2.2 *contact position*—the rotor height when in rubbing contact with the stator.

3.2.3 *idling oil*[2]—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*[2]—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 1×10^{6} s^{-1}.

[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricantsand is the direct responsibility of Subcommittee D02.07.0Bon High Temperature Rheology of Non-Newtonian Fluids.

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[2] Available from Tannas Co., P.O. Box 327, Midland, MI 48640.

[3] *1990 Annual Book of ASTM Standards*, Vol 05.03.

3.2.5 *reciprocal torque intersection, Rti*—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 1×10^{6} 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.

3.2.8 *test oil*—any oil for which apparent viscosity is to be determined.

4. Summary of Test Method

4.1 A 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 1×10^{6} 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°C.

5. Significance and Use

5.1 Viscosity 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.

5.2 The importance of viscosity at these conditions to engine lubrication has been addressed in many publications.[4]

6. Apparatus

6.1 *Tapered Bearing Simulator-Viscometer* [2] (Fig. 1)—a viscometer consisting of a synchronous two-speed motor that drives a slightly tapered bearing in a matched stator (Fig. 2).

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.

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

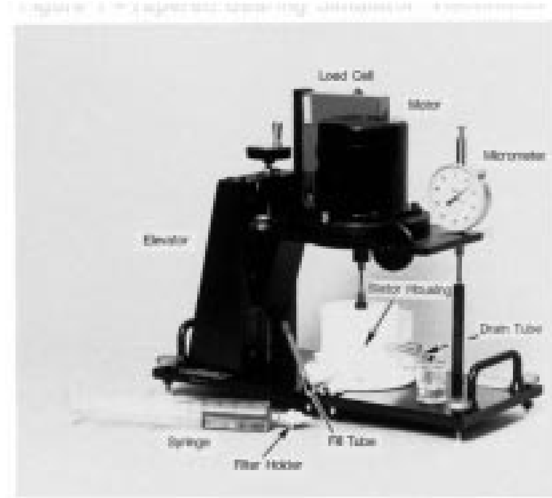

FIG. 1 Tapered Bearing Simulator-Viscometer

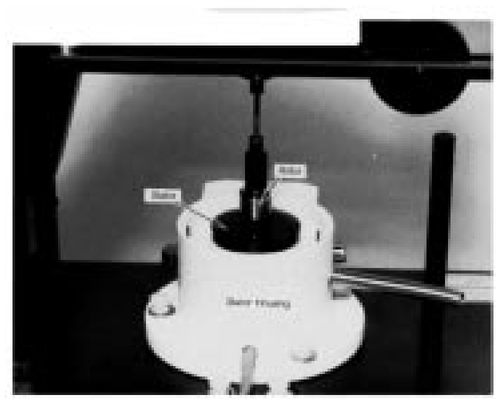

FIG. 2 Rotor, Stator, and Stator Housing

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.

NOTE 1—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.[2]

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.

6.4 *Glass Syringe*, 50-mL, equipped with Luer needle lock fits the tip of the filling tube for injection of test oil into the test cell.

6.5 *Filter*—A filter is used on the syringe to remove particles capable of damaging the rotor/stator cell.[2]

7. Materials

7.1 *Calibration Oils* [2] are Newtonian oils of known kinematic viscosity and density at 150°C. The defined viscosities in

FIG. 3 Console

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. Certified viscosities are supplied with each oil.

7.2 *Idling Oil*—See 3.2.3.

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

8. Sampling

8.1 A representative sample of test oil, free from evident suspended solid material, is necessary to obtain valid results and to avoid lock-up and marring of the rotor/stator mating surfaces. *Do not* draw test oil into the syringe from the bottom of any container. When *visible* particulates are present in the oil, it is mandatory to remove them by filtration before the oil is injected into the test cell (see 6.5). When used oils are evaluated, in some instances it may be desirable to filter the oil prior to injection. Care must be taken that air or other gas is not inadvertently injected into the operating cell.

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.

TABLE 1 Calibration and Reference Oils

Code No.	Viscometric Characteristics	Nominal Viscosity^A cP (mPa·s) at 150°C
R-200	Newtonian	1.9
R-300	Newtonian	2.8
R-400	Newtonian	3.5^B
R-500	Newtonian	5.3
NNR-03	non-Newtonian	3.5^C

^{A}Nominal viscosity values. Consult supplier for certified values.
^{B}Matched to NNR-03.
^{C}At 10^8 s^{-1}(matched to R-400).

9.2 With continuous use, a weekly room-temperature flush of the rotor/stator cell is recommended following directions in 11.4.

10. Calibration

10.1 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 stand-by 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.

10.4 *Determining the Stored Position*:

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 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.*

10.4.3 Raise the rotor to a position 0.50 mm (500 micrometres) above the contact position. Record this reading as the stored position.

10.4.3.1 It is important to observe whether the micrometer reading is 0.50 mm *smaller* or 0.50 mm *larger* than the reading at the contact position. For units that have mechanical micrometers, the reading for the stored position will be 0.50 mm smaller than that recorded for the contact position. If an electronic micrometer is used, the reading for the stored position will be 0.50 mm larger than that recorded for the contact position. See 3.2.6.1.

10.5 *Determination of the Reciprocal Torque Intercept (Rti) to Determine Rotor Position for* $1 \times 10^{6}\ s^{-1}$ *Shear Rate*:

10.5.1 With the rotor in the stored position, gently move the motor turntable clockwise a few degrees to disengage the ball from contact with the platen, hold the motor housing firmly in this position, set the speed switch to HI and flip the motor switch to the ON position. Release the grip on the motor and let the reaction torque of the spinning motor bring the ball into contact with the load cell platen (See Fig. 4).

10.5.2 Let the motor run at least 1 h before proceeding to 10.5.3 .

10.5.3 Fill the test cell with NNR-03 non-Newtonian reference oil by either of the methods described in 10.5.3.1 or 10.5.3.2. See Section 8 for sampling precautions. After the test cell has been filled, all subsequent injections should be made with the motor running. (If this is a first time operation or after the viscometer has been idled for more than an hour, make a second injection after waiting the requisite 5 min 30 s.)

10.5.3.1 Fill the syringe with 50 ± 3 mL of oil. Slowly inject the oil into the inlet tube of the test cell at a rate of approximately 2 mL/s. Fluids with viscosities greater than 4 cP at 150°C can be preheated to about 40°C to make the injection rate easier to meet.

10.5.3.2 A different injection procedure may be used when the amount of test fluid is limited. Fill the syringe with 30 mL of fluid and make three 10-mL injections, waiting 10 s between each injection.

10.5.3.3 Note the time when the injection is completed.

10.5.4 Stabilize the temperature at 150 ± 0.2°C by regulation of the temperature controller on the console. Air circulation to the stator housing may be required for supplementary cooling of fluids with viscosities greater than 9 cP. Five minutes after injection proceed to 10.5.5.

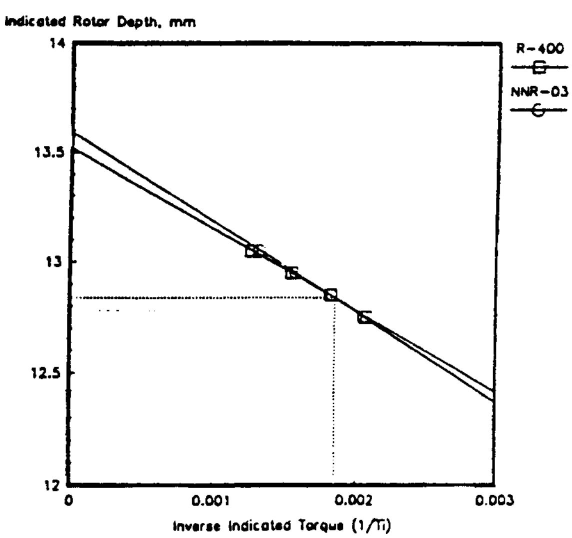

FIG. 4 Reciprocal Torque Intercept Method for Setting Appropriate Rotor Position

NOTE 2—A temperature controller with automatic reset and a thermofoil heater are required to achieve 150 ± 0.2°C within the specified 5 min.

10.5.5 Separate the load-cell/motor-turntable contact as in starting (see 10.5.1), then shut off the motor. Reconfirm the rotor/stator contact position determined in 10.4.2. Now, raise the rotor to a position 0.10 mm above the contact position just established.

10.5.6 Restart the motor using the technique described in 10.5.1.

10.5.7 Disengage the turntable contact ball from the platen of the load cell and at the same time set the torque indicator reading to zero if it has drifted more than ±2 display units from zero. See Fig. 3 for the location of the zero adjustment port or knob.

10.5.8 Record the torque indicator reading and the rotor position when the temperature is stabilized at 150 ± 0.2°C.

10.5.9 Raise the rotor to a position that is 0.20 mm above the contact position and repeat 10.5.8.

10.5.10 Repeat 10.5.8 at rotor heights 0.30 and 0.40 mm above the contact position which now gives four values of rotor height and torque readout for analysis in 10.5.12.

10.5.11 Return the rotor to the stored position obtained in 10.4.3, fill the test cell with R-400 calibration oil, and repeat 10.5.6 through 10.5.10.

10.5.12 Calculate the reciprocals of the four values of indicated torque. Use these and the associated rotor height values to generate linear regression analyses for the NNR-03 and R-400. A correlation coefficient of 0.999 or higher (close to unity) is required for both NNR-03 and R-400 data.

10.5.13 Determine the intersection of these two straight lines to obtain the rotor height position at which $1 \times 10^{6}\ s^{-1}$ shear rate will be generated by the viscometer.

NOTE 3—A number of pocket calculators have linear regression analysis programs built into them and can be programmed to determine the intersection point of NNR-03 and R-400 as well. A preprogrammed calculator can be obtained commercially.[2] Alternatively, but not as accurately, the *Rti* can be determined by graphical analysis on linear coordinate paper using the *Y*-axis for rotor height and the *X*-axis for reciprocal indicated torque.

NOTE 4—If the lines do not intersect or the calculated intersection position would require the rotor to be inserted into the stator near (within 0.01 mm) or beyond the contact position, repeat 10.5.3 through 10.5.13, starting at a point 0.5 mm above the contact position. This algebraic/graphical analysis is based on the torque response of R-400 and NNR-03 fluids and assumes that these fluids have equivalent viscosities at $1 \times 10^{6}\ s^{-1}$ shear rate and 150°C. In cases where the rotor/stator cell is contaminated with heavy oxidation deposits or the surfaces of the cell have been damaged, it may not be possible to reach the gap necessary for a shear rate of $1 \times 10^{6}\ s^{-1}$ operation. If the intersection still is not obtained after the second attempt, it will be necessary to sonically clean the rotor/stator cell or in cases of surface damage, to re-lap those rotor and stator surfaces exposed to high shear.

10.5.14 Move the rotor to the reciprocal torque intercept position, *Rti*, and proceed to 11.2.

11. Procedure

11.1 *Equipment Activation*:

11.1.1 If the tapered bearing simulator-viscometer has been turned off, activate console in accordance with 10.2.

11.1.2 If there is no oil in the test cell, inject fluid in accordance with 10.3.1.

11.1.3 Bring the operating temperature to 150°C.

11.1.4 Turn on the motor in accordance with instructions in 10.5.1. Let the motor run for at least 1 h before proceeding to 10.5.3 where the reciprocal torque intersection (*Rti*) will be determined.

11.2 *Viscosity/Torque Response of Calibration and Test Oils*:

11.2.1 With the rotor being driven by the motor at *Rti*, inject the R-200 calibration oil in accordance with 10.5.3 and wait until the temperature stabilizes at 150 ± 0.2°C. Five and one-half min after completing the injection, check the no-load torque reading according to the technique in 10.5.7. Record the torque indicator reading for R-200. Follow this procedure to obtain torque indicator readings for R-500 and NNR-03.

11.2.2 Using the torque indicator readings for R-200 and R-500, and the certified viscosities for these oils, make a preliminary appraisal of the calibration constants c and d using the following equation:

$$V = c + d(Tr) \tag{1}$$

where:

V = certified viscosity of the oil, cP (mPa·s),
Tr = torque indicator reading,
c = Y-axis intercept, cP (mPa·s), and
d = slope of the best line, cP/Tr.

11.2.3 Solve Eq 1 as recommended in 11.1 using the torque indicator reading determined for the NNR-03. The resulting viscosity should be within 1 % of the certified viscosity value. If it is not, and the determined viscosity value is too high, lower the rotor by 0.01 mm for each 0.01 cP higher than the certified viscosity value. The opposite adjustment is used if the value for NNR-03 is too low (that is, raise the rotor 0.01 mm for each 0.01 cP lower than certified). After such adjustment, repeat 11.2.1 through 11.2.3. If still outside the 2 % range, determine the *Rti* again following the instructions in 10.5.

11.2.4 Choose a first test oil and fill the TBS Viscometer according to 10.5.3.1 or, if a limited quantity of the test oil is available, use procedure 10.5.3.2.

11.2.5 Wait 5½min, then check the no-load torque response in accordance with 10.5.7 and record the torque indicator reading as soon as the temperature is stabilized at 150 ± 0.2°C.

11.2.6 If desired, continue testing with up to three (additional) unknown oils.

11.2.7 Proceed to collect torque readings for the calibration oils R-400 and R-300 respectively.

11.2.8 Gathering the torque readings and certified viscosities on all four reference oils, R-200 and R-500 from 11.2.2, and R-300 and R-400 from 11.2.7, obtain a linear regression analysis of this data. This will give final values for c and d in Eq 1 as well as the correlation coefficient (R).

11.2.9 A correlation coefficient of at least 0.999 is expected for the linear regression analysis of 11.2.8. If a correlation coefficient less than this minimum is obtained, then the data generated from the calibration must be plotted to see if one of the data points falls significantly off the best line through the four points. If so, then the oil that provided the apparently spurious result must be tested again. If there are no data out of obvious agreement, then the analyst must recheck the setup of the TBS Viscometer in accordance with the owner's manual and the foregoing steps of calibration.

11.2.10 Calculate the viscosity for each of the test oils as directed in Section 12 for each of the test oils and the NNR-03.

11.2.11 When more than four test oils are to be evaluated, inject the cell with NNR-03, wait the requisite 5½ min, and collect the torque reading for the fluid. Use this to calculate the viscosity of NNR-03 according to Eq 1 using the constants found in 11.2.8.

11.2.12 When the calculated viscosity for NNR-03 is still within 1 % of the certified viscosity, proceed to 11.2.14.

11.2.13 If the calculated viscosity for NNR-03 is not within the 1 % range, re-inject NNR-03 into the test cell and repeat the viscosity determination. If the viscosity is again outside the 1 % range, return to 11.2.3.

11.2.14 Evaluate (additional) test oils. Follow each six evaluations with a check evaluation of NNR-03 to ensure staying within the 1 % range of the established value at 10^6 s^{-1} shear rate.

11.2.15 If the TBS-Viscometer is unattended (or anticipated to be unattended) for more than ½h, inject 25 mL of idling oil into the cell. Before resuming operation, flush with an additional 25 mL of idling oil and then evaluate the viscosity of NNR-03 to check that it is within 1 % of its certified value.

11.3 *Shutdown Procedure*—When testing is complete, inject the idling oil and return the rotor to the stored position. Follow directions in 10.5.1 to separate the load-cell/motor-turntable contact when shutting off the motor. Turn the temperature control to ambient. It is prudent to also uncouple the heater cord at the housing or at the back of the console.

NOTE 5—If another series of tests is to be run the next day, and the rotor/stator cell is protected by overheating and power-failure safety switches, inject 50 mL of idling oil and leave the viscometer at temperature and operating gap. Start the next day with 10.5.3 to determine *Rti*.

11.4 *Test Cell Cleanup*:

NOTE 6—The continuous operation of the viscometer without an occasional flush with a polar organic solvent will eventually lead to a test cell whose operating surfaces will be coated with oxidation debris from the decomposition of the oils. While adjustment of the vertical rotor position can offset the change in operating gap caused by the deposit, and occasional (weekly) flush of the test cell is recommended. As a further precaution, anytime the viscometer is idle at operating gap and temperature, the test cell should be filled with idling oil.

11.4.1 After a routine shutdown in accordance with 11.3 and reaching ambient temperature, fill the test cell with a polar organic solvent.

11.4.2 Following 10.5.1, start the viscometer motor and lower the rotor 0.2 mm below the redetermined stored position.

11.4.3 After 10 min, purge the solvent from the cell with idling fluid using a 60-mL stepwise total injection of one 30-mL injection followed by two 15-mL injections all spaced about 5 min apart.

11.4.4 Shut down the TBS Viscometer as in 11.3 unless further testing is desired. Otherwise testing should be resumed with 10.3.2.

NOTE 7—If a dirty test cell is suspected and there is a desire to renew operation as soon as possible, shut the motor off, raise the motor platform and remove the rotor (**Caution**—The rotor will initially be at operating temperature). The rotor can then be sonically cleaned in a water/soap solution. After 15 min of cleaning, dry the rotor, reinstall for operation, and restart operations at 10.3.2.

12. Calculation

12.1 Using the final calibration constants *c* and *d* determined in 11.2.8, calculate the dynamic viscosity of each test oil in accordance with Eq 1.

13. Report

13.1 Report the apparent viscosity to the nearest 0.01 cP (mPa·s) at 150°C and 1×10^6 s^{-1} shear rate.

14. Precision and Bias [5]

14.1 *Precision*—The following criteria should be used for judging the acceptability of results:

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

2.3 % of the mean.

[5] Supporting data are available from ASTM Headquarters. Request RR: D02-1211 and RR: D02–1378.

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

3.6 % of the mean.

14.1.3 Eight laboratories were used in a program to obtain the precision statement. Statistical analysis was obtained using ASTM engine oils in the range from 2.4 to 4.8 mPa-s. The test series included SAE 0W-30, 5W-20, 5W-30 (4) 10W-30, 10W-40 (2), 15W-40, 20W-50, and 30 grade oils.

14.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for this test method, bias cannot be determined.

14.3 *Relative Bias*— Results from this test method were found, in an interlaboratory study, to agree with those from Test Methods D 4741 and D 5481. They can be expected to give, on average in the long run, the same results for the same oil.

15. Keywords

15.1 dynamic viscosity; high temperature viscosity; high shear viscosity; rotational viscometer