



# Standard Test Method for Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature<sup>1</sup>

This standard is issued under the fixed designation D4684; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

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

## 1. Scope\*

1.1 This test method covers the measurement of the yield stress and viscosity of engine oils after cooling at controlled rates over a period exceeding 45 h to a final test temperature between  $-10$  and  $-40^{\circ}\text{C}$ . The viscosity measurements are made at a shear stress of 525 Pa over a shear rate of 0.4 to  $15\text{ s}^{-1}$ . The viscosity as measured at this shear stress was found to produce the best correlation between the temperature at which the viscosity reached a critical value and borderline pumping failure temperature in engines.

1.2 This test method contains two procedures: Procedure A incorporates several equipment and procedural modifications from Test Method D4684–02 that have shown to improve the precision of the test, while Procedure B is unchanged from Test Method D4684–02. Additionally, Procedure A applies to those instruments that utilize thermoelectric cooling technology or direct refrigeration technology of recent manufacture for instrument temperature control. Procedure B can use the same instruments used in Procedure A or those cooled by circulating methanol.

1.3 Procedure A of this test method has precision stated for a yield range from less than 35 Pa to 210 Pa and apparent viscosity range from 4300 to 270 000 mPa·s. The test procedure can determine higher yield stress and viscosity levels.

1.4 This test method is applicable for unused oils, sometimes referred to as fresh oils, designed for both light duty and heavy duty engine applications. It also has been shown to be suitable for used diesel and gasoline engine oils. The applicability to petroleum products other than engine oils has not been determined.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5.1 *Exception*—This test method uses the milliPascal second (mPa·s) as the unit of viscosity. For information, the equivalent unit, centiPoise (cP), 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*:<sup>2</sup>

**D3829** Test Method for Predicting the Borderline Pumping Temperature of Engine Oil

2.2 *ISO Standard*:<sup>3</sup>

**ISO 17025** General Requirements for the Competence of Testing and Calibration Laboratories

**ISO Guide 34** General Requirements for the Competence of Reference Material Producers

**ISO Guide 35** Certification of Reference Materials

## 3. Terminology

3.1 *Definitions*:

3.1.1 *apparent viscosity*—the determined viscosity obtained by use of this test method.

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 at a given temperature exhibits a viscosity that varies with changing shear stress or shear rate.

3.1.4 *shear rate*—the velocity gradient in fluid flow. For a Newtonian fluid in a concentric cylinder rotary viscometer in which the shear stress is measured at the inner cylinder surface

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

Current edition approved Dec. 1, 2008. Published January 2009. Originally approved in 1987. Last previous edition approved in 2007 as D4684–07<sup>e1</sup>. DOI: 10.1520/D4684-08.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> Available from International Organization for Standardization (ISO), 1 rue de Varembe, Case postale 56, CH-1211, Geneva 20, Switzerland, <http://www.iso.ch>.

(such as this apparatus, described in 6.1), and ignoring any end effects, the shear rate is given as follows:

$$G_r = \frac{2(\Omega)R_s^2}{R_s^2 - R_r^2} \quad (1)$$

$$= \frac{4(\pi) R_s^2}{t (R_s^2 - R_r^2)} \quad (2)$$

where:

$G_r$  = shear rate at the surface of the rotor in reciprocal seconds,  $s^{-1}$ ,

$\Omega$  = angular velocity, rad/s,

$R_s$  = stator radius, mm,

$R_r$  = rotor radius, mm, and

$t$  = time in seconds for one revolution of the rotor.

For the specific apparatus being described in 6.1.1,

$$G_r = 63/t \quad (3)$$

3.1.5 *shear stress*—the motivating force per unit area for fluid flow. For the rotary viscometer being described, the rotor surface is the area under shear or the shear area.

$$T_r = 9.81 M (R_o + R_r) \times 10^{-6} \quad (4)$$

$$S_r = \frac{T_r}{2 (\pi) R_r^2 h} \times 10^9 \quad (5)$$

where:

$T_r$  = torque applied to rotor, N·m,

$M$  = applied mass, g,

$R_o$  = radius of the shaft, mm,

$R_r$  = radius of the string, mm,

$S_r$  = shear stress at the rotor surface, Pa, and

$h$  = height of the rotor, mm.

For the dimensions given in 6.1.1,

$$T_r = 31.7 M \times 10^{-6} \quad (6)$$

$$S_r = 3.5 M \quad (7)$$

3.1.6 *viscosity*—the ratio between the applied shear stress and rate of shear, sometimes called the coefficient of dynamic viscosity. This value is thus a measure of the resistance to flow of the liquid. The SI unit of viscosity is the Pascal second [Pa·s]. A centipoise (cP) is one milliPascal second [mPa·s].

### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *calibration oils*—those oils that establish the instrument's reference framework of apparent viscosity versus speed, from which the apparent viscosities of test oils are determined. Calibration oils, which are essentially Newtonian fluids, shall be obtained from suppliers complying with **ISO Guide 34**, **ISO Guide 35**, and **ISO 17025** with traceability to a national metrology institute (NMI). These calibration oils will have an approximate viscosity of 30 Pa·s (30 000 cP) at  $-20^\circ\text{C}$  or 60 Pa·s (60 000 cP) at  $-25^\circ\text{C}$ .

3.2.2 *cell constant*—the ratio of the calibration fluid viscosity to the time required to complete the first three measured revolutions of the rotor.

3.2.3 *test oil*—any oil for which the apparent viscosity and yield stress are to be determined by this test method.

3.2.4 *unused oil*—an oil which has not been used in an operating engine.

3.2.5 *used oil*—an oil which has been used in an operating engine.

3.2.6 *yield stress*—the shear stress required to initiate flow. For all Newtonian fluids and many non-Newtonian fluids, the yield stress is zero. An engine oil can have a yield stress that is a function of its low-temperature cooling rate, soak time, and temperature.

## 4. Summary of Test Method

4.1 An engine oil sample is held at  $80^\circ\text{C}$  and then cooled at a programmed cooling rate to a final test temperature and held for a specified time period. At the end of this period, a series of increasing low torques are applied to the rotor shaft until rotation occurs to determine the yield stress, if any is exhibited. A higher torque is then applied to determine the apparent viscosity of the sample.

## 5. Significance and Use

5.1 When an engine oil is cooled, the rate and duration of cooling can affect its yield stress and viscosity. In this laboratory test, a fresh engine oil is slowly cooled through a temperature range where wax crystallization is known to occur, followed by relatively rapid cooling to the final test temperature. These laboratory test results have predicted as failures the known engine oils that have failed in the field because of lack of oil pumpability.<sup>4</sup> These documented field failing oils have all consisted of oils normally tested at  $-25^\circ\text{C}$ . These field failures are believed to be the result of the oil forming a gel structure that results in either excessive yield stress or viscosity of the engine oil, or both.

### 5.2 Cooling Profiles:

5.2.1 For oils to be tested at  $-20^\circ\text{C}$  or colder, **Table X1.1** applies. The cooling profile described in **Table X1.1** is based on the viscosity properties of the ASTM Pumpability Reference Oils (PRO). This series of oils includes oils with normal low-temperature flow properties and oils that have been associated with low-temperature pumpability problems (**1-5**).<sup>5</sup> Significance for the  $-35$  and  $-40^\circ\text{C}$  temperature profiles is based on the data collected from the “Cold Starting and Pumpability Studies in Modern Engines” conducted by ASTM (**6,7**).

5.2.2 For oils to be tested at  $-15$  or  $-10^\circ\text{C}$ , **Table X1.2** applies. No significance has been determined for this temperature profile because of the absence of appropriate reference oils. Similarly, precision of the test method using this profile for the  $-10^\circ\text{C}$  test temperature is unknown. The temperature profile of **Table X1.2** is derived from the one in **Table X1.1** and has been moved up in temperature, relative to **Table X1.1**, in consideration of the expected higher cloud points of the viscous oils tested at  $-15$  and  $-10^\circ\text{C}$ .

## 6. Apparatus

6.1 *Mini-Rotary Viscometer*—An apparatus that consists of one or more viscometric cells in a temperature-controlled block made of a metallic material with high thermal conductivity. Each cell contains a calibrated rotor-stator set. The rotor shall

<sup>4</sup> Pumpability Reference Oils (PRO) 21 through 29.

<sup>5</sup> The boldface numbers in parentheses refer to the references at the end of this standard.

have a crossbar near the top of the shaft extending in both directions far enough to allow the locking pin (6.6) to stop rotation at successive half turns. Rotation of the rotor is achieved by an applied force acting through a string wound around the rotor shaft.

6.1.1 The mini-rotary viscometric cell has the following dimensions:

Diameter of rotor	17.06 ± 0.08 mm
Length of rotor	20.0 ± 0.14 mm
Inside diameter of cell	19.07 ± 0.08 mm
Radius of shaft	3.18 ± 0.13 mm
Radius of string	0.1 mm

6.1.2 *Cell Cap*—A cover inserted into the top of the viscometer cell to minimize room air circulation into the cells is required for thermometrically cooled instruments. The cell cap is a stepped cylinder 38 ± 1 mm (1.5 ± 0.05 in.) in length made of a low thermal conductivity material, for example, thermoplastic such as acetyl copolymers that have known solvent resistivity and are suitable for use between the temperature ranges of this test method. The top half is 28 ± 1 mm (1.10 ± 0.05 in.) in diameter and the bottom half is 19 mm (0.745 in.) in diameter with a tolerance consistent with the cell diameter. The tolerance on the bottom half is such that it will easily fit into cell but not allow cap to contact rotor shaft. The piece has a center bore of 11 ± 1 mm (0.438 ± 0.05 in.). The cap is made in two halves to facilitate placement in the top of the cell.

6.1.2.1 Cell caps shall not be used in the direct refrigeration instruments, since such use would block the flow of cold, dry air into the stators to keep them frost-free.

## 6.2 Weights:

6.2.1 *Yield Stress Measurement*—A set of ten weights, each with a mass of 10 ± 0.1 g. One of the weights is a holder for the other weights.

6.2.2 *Viscosity Measurement*—Weight with mass of 150 ± 1.0 g.

6.3 *Temperature Control System*—Regulates the mini-rotary viscometer block temperature in accordance with the temperature requirements described in Table X1.1 or Table X1.2.

6.3.1 *Temperature Controller*—As a very critical part of this procedure, a description of the requirements that the controller shall meet are included in Appendix X2.

6.3.2 *Temperature Profile*—The temperature profile is fully described in Table X1.1 and Table X1.2.

6.4 *Thermometers*—For measuring the temperature of the block. Two are required, one graduated from at least +70 to 90°C in 1°C subdivisions, the other with a scale from above +5°C down to at least -41°C or lower, in 0.2°C subdivisions. Other thermometric devices of equal accuracy and resolution may be used to measure the temperature, such as digital meters using a resistance temperature detector (RTD) or a thermistor sensor.

6.4.1 When using metal encased thermometric devices, care should be taken that the metal case does not create a biased temperature reading. It has been observed that some metal sheathed devices indicate a higher than actual temperature of the sample. This is typically caused by heat conduction through the metal sheath but there can be other causes.

6.5 *Supply of Dry Gas*—A supply of dry filtered dry gas to minimize moisture condensation on the upper portions of the instrument.

6.5.1 For thermoelectric cooled instruments, which use cell caps, the dry gas supply is connected to the housing cover. The supply of dry gas is discontinued when the cover is removed for the measurement phase of the test.

6.6 *Locking Pin*—A device to keep the rotor from turning prematurely and able to stop the rotor at the nearest half revolution by interaction with the rotor crossbar.

## 7. Reagents and Materials

7.1 *Newtonian Oil*—Low cloud-point of approximately 30 Pa·s (30 000 cP) viscosity at -20°C for Procedure B or 60 Pa·s (60 000 cP) at -25°C for Procedure A for calibration of the viscometric cells.

7.2 *Methanol*—Commercial or technical grade of dry methanol is suitable for the refrigerated cooling bath required for some units. (**Warning**—Flammable.)

7.3 *Oil Solvent*—Commercial heptanes or similar solvent that evaporates without leaving a residue is suitable. (**Warning**—Flammable.)

7.4 *Acetone*—A technical grade of acetone is suitable provided it does not leave a residue upon evaporation. (**Warning**—Flammable.)

## Procedure A

## 8. Sampling

8.1 A representative sample of test oil free from suspended solid material and water is necessary to obtain valid viscosity measurements. If the sample in its container is received below the dew-point temperature of the room, allow the sample to warm to room temperature before opening the container.

## 9. Calibration and Standardization

9.1 *Calibration Procedure*—For those instruments in which the temperature sensor is not permanently attached to the temperature controller, calibrate the temperature sensor in the MRV block while the sensor is attached to the temperature controller.

9.1.1 The sensed temperature calibration shall be verified using a reference thermometer noted in 6.4 at a minimum of three temperatures.

9.1.2 During the calibration all the cells are to contain 10 mL of a typical fluid with the rotor and, if required, cell caps in place. Cell caps shall not be used for direct refrigeration instruments (see 6.1.2).

9.1.3 Make these temperature measurements at least 5°C apart and include both -5°C and the lowest test temperature used to establish a calibration curve for this combination of temperature sensor and controller. Make at least two temperature measurements at every calibration temperature with at least 10 min between observations. For instruments using an independent temperature controller, see X2.1 for calibration guidance.

NOTE 1—All temperatures in this test method refer to the actual temperature and not necessarily the indicated temperature.

9.2 The calibration constant of each rotor/stator combination is determined by conducting two tests at  $-25^{\circ}\text{C}$  using a viscometric standard as a test sample.

9.2.1 Each cell shall be calibrated twice and the resulting calibration constant is to be calculated from the average of the two determinations of the time for three revolutions of the rotor. When the two cell calibrations are consecutive, the second test shall be on a new sample of standard with cleaning between the steps.

NOTE 2—Once a set of rotors have been calibrated in an instrument, subsequent calibration checks can be single determinations if the criteria of 9.11 are met.

9.2.2 The same 150 g weight is to be used for both calibration and viscosity measurements. However, different weights may be used for calibration and viscosity measurements if the masses of the two weights are certified to be  $150 \pm 0.1$  g.

9.3 Using steps in 10.1, prepare the cells for the calibration test cycle.

NOTE 3—Before inserting the rotors in the cells, inspect each rotor to be sure that the shaft is straight, that the rotor surface is smooth and free from dents, scratches and other imperfections. For rotors with a bearing point at the bottom of the shaft, ensure that the point is sharp and centered on the rotor shaft. If these conditions are not met, repair or replace the rotor.

9.4 Using either the calibration temperature profile given for the instrument (or, alternatively, the cooling profile given in Test Method D3829) for the test temperature of the reference fluid, follow the owner's manual instructions for the instrument to initiate the cooling profile program.

NOTE 4—The use of the calibration temperature profile makes it possible to complete two cell constant determinations in one day.

9.5 Place the thermometer in the thermowell. The same thermowell location is to be used for all measurements.

9.5.1 The thermometer must be placed into the thermowell at least one hour prior to completion of the test.

9.6 At the completion of the temperature profile, check that the final test temperature is at the desired calibration temperature within  $\pm 0.1^{\circ}\text{C}$ . Final test temperature is to be verified independently of the temperature controller using the thermometer in the thermowell.

9.7 Beginning with the cell farthest to the left facing the instrument, follow this procedure for each cell in turn.

9.7.1 Align the pulley wheel with the rotor shaft for the cell to be tested.

9.7.2 Hang the string over the timing wheel.

9.7.3 Suspend the weight holder plus a 10-g weight (total mass 20 g) from string.

9.7.4 Disengage the locking pin.

9.7.5 As soon as the crossbar is clear of the locking pin, reengage the locking pin. This will stop the rotation at approximately one half revolution.

9.7.6 Remove weight holder and 10-g weight from the string.

9.7.7 Suspend the 150-g weight from the string.

9.7.8 Disengage the locking pin and simultaneously start timing as soon as the rotor is released.

9.7.9 Determine the time for exactly three revolutions of the rotor.

NOTE 5—For some instruments, the timing may be done automatically.

9.7.10 After three revolutions, reengage the locking pin and remove the weight from the string.

9.7.11 Record the time for three revolutions and the cell number.

9.8 Repeat 9.7.1-9.7.11 for each of the remaining cells in numerical order.

9.9 Repeat 9.3-9.8 for a second set of calibration data.

9.10 For each cell (rotor/stator combination) calculate of the calibration constant using Eq 8 and 9.

$$t = (t_1 + t_2)/2 \quad (8)$$

$$C = \eta/t \quad (9)$$

where:

$\eta$  = viscosity of the standard oil, mPa·s (cP) at test temperature,

$C$  = cell constant,

$t_1$  = time of three rotor revolutions for first calibration,

$t_2$  = time of three rotor revolutions for second calibration, and

$t$  = average time of three rotor revolutions.

9.11 After the calibration constants have been determined, check to see if any cell has a calibration constant differing by more than 4% from the average of all cells or if the difference between  $t_1$  and  $t_2$  for any cell is greater than 4% of the average of  $t_1$  and  $t_2$ . If so, then one or both of the results should be considered suspect. If these criteria are not met, examine the indicated rotor for damage, repair or replacement as necessary, and repeat the cell calibrations.

9.12 If corrected values for the controller temperature and thermometer deviate by more than the tolerance ( $\pm 0.1^{\circ}\text{C}$ ), use the procedure in X2.2 to assist in determining the cause and correction.

## 10. Yield Stress and Viscosity Measurement Procedure

10.1 *Test Sample and Viscometric Cell Preparation:*

10.1.1 If the cells are not clean, see 10.7 for the cleaning procedure.

10.1.2 Place  $10 \pm 0.2$  mL of test oil samples into the clean cells.

10.1.2.1 All cells should contain a fluid and rotor; if there are less than a full set of samples to run, fill each of the unused cells with a typical test sample.

10.1.3 *Loading Cells with Test Oils*—Place each rotor and test oil in its cell, and place upper pivot pin in position, including any unused cells.

10.1.4 When use is required, install a cell cap on all cells, including any unused cells.

10.1.5 For each cell, except any unused ones, place a loop of the nominal 700-mm long string over the crossbar. Hang the string over the timing wheel with a small weight attached such as a large paper clip. Wind the string around the shaft until the end is about 100 mm below the wheel. Do not overlap windings.

NOTE 6—The strings can be pre-wound around the shafts before they are installed in 10.1.3.



10.1.5.1 Engage the locking pin to prevent the rotor from turning.

10.1.5.2 Lay the remaining string over the top of the bearing plate letting it hang over the back of the plate.

10.1.5.3 Repeat 10.1.5-10.1.5.2 until all cells with samples to be measured are prepared.

10.1.6 Place the housing cover over the viscometric cells.

10.1.7 Connect the dry gas supply to the housing cover, as noted in 6.5. Set the dry gas flow to approximately 1 L/h. Increase or decrease the flow as necessary to minimize frost or moisture condensation around the cells.

10.2 Select the cooling profile for the desired test temperature and follow the instrument instructions to initiate the program. Table X1.3 lists the nominal times to reach a particular test temperature.

10.3 Place the thermometer in the thermowell at least one hour prior to completion of the test. The same thermowell location is to be used for all measurements and must be the same one as was used in the calibration.

10.4 At the completion of the cooling profile, check the time-temperature plot for the run to ensure that the time-temperature profile is within tolerance and that the test temperature as measured in the thermowell is within  $\pm 0.2^\circ\text{C}$  of the final test temperature. Both of these checks may be done automatically by the control software incorporated in some instruments. Final test temperature is to be verified independently from the temperature controller. If the final test temperature is more than  $0.1^\circ\text{C}$  from the set point on two consecutive runs, the temperature sensor must be recalibrated according to 9.1.

10.5 If the temperature profile is within tolerance, proceed with measurements. If not, then abort the test and recalibrate temperature controller as in 9.1.

#### 10.6 *Measurement of the Yield Stress and Viscosity:*

10.6.1 Immediately prior to starting measurements, take the cell housing cover off the instrument.

10.6.2 *Yield Stress Determination*—Starting with the cell farthest to the left while facing the instrument, use the following procedure for each cell in turn, bypassing the unused cells.

10.6.2.1 Align the pulley wheel with the rotor shaft of the cell to be tested.

10.6.2.2 Hang the string over the timing wheel.

10.6.2.3 Suspend the 10-g weight holder from the string.

10.6.2.4 For instruments with automatic timing, start timing and then release the locking pin. For manual timing, start timing immediately after the locking pin is disengaged.

10.6.2.5 Observe whether the end of the crossbar moves more than 3 mm in 15 s. (This 3 mm is approximately twice the diameter of the crossbar.) An alternative procedure is the use of a marked rotation of the timing wheel equivalent to a rotor shaft rotation of 3 mm.

10.6.2.6 Electronic or timing wheel motion-sensing devices, which are available on some instruments, are suitable alternatives to direct observation.

10.6.2.7 If rotor movement of more than 3 mm, or alternative, in 15 s is observed in 10.6.2.5, remove all of the 10-g weights from the end of the string, and proceed to 10.6.3.

10.6.2.8 If a rotor movement of less than 3 mm in 15 s is observed in 10.6.2.5, stop timing and lift the weight holder so it is not supported by the string. Then add an additional 10-g weight to weight holder.

NOTE 7—As additional weights are added to the weight holder it is necessary to suspend the holder with the additional weights from the string and restart timing without the use of the locking pin for the remainder of the yield stress assessment. When using software available for some instruments, ensure that the mass applied is the mass requested by the program.

10.6.2.9 Carefully and gently, suspend the weight holder with the additional weights from the string and start timing.

10.6.2.10 Repeat steps in 10.6.2.8 and 10.6.2.9 until the accumulated weights causes rotation of the rotor. At this point, remove all the weights from the string.

10.6.2.11 If no rotation is observed with a total of 100 g, record that the yield stress is  $>350$  Pa, and proceed with 10.6.3.

#### 10.6.3 *Viscosity Determination:*

10.6.3.1 Gently suspend the 150-g mass from the string.

10.6.3.2 If the applied mass of 150 g will move the rotor, as soon as the cross-arm is clear of the locking pin, reengage the locking pin. Allow rotation to continue until the cross-arm contacts the locking pin causing rotation to stop. If no appreciable rotation occurs, terminate the test and proceed to 10.6.3.7.

NOTE 8—Yield stresses exceeding the stress exerted by 150 g have been encountered.

10.6.3.3 When using instruments capable of timing rotation automatically, initiate viscosity measurement by starting timing, then release the locking pin. When timing manually, start timing immediately after the locking pin is disengaged.

10.6.3.4 Stop the timer after three revolutions of the rotor from point of release. When the time for one revolution is greater than 60 s, time only one revolution.

NOTE 9—The timing of three revolutions may be done automatically.

10.6.3.5 After completing three revolutions (one revolution if the time for it is greater than 60 s), remove mass from string.

10.6.3.6 Record the time for three revolutions (one revolution) and the number of revolutions for calculation of the viscosity in Section 11.

10.6.3.7 If no rotation occurs with the application of the 150-g weight, record the result for that sample as being “Too Viscous To Measure” (TVTM).

10.6.3.8 Repeat 10.6.2-10.6.3.7 for the remaining cells to be measured.

#### 10.7 *Cleaning:*

10.7.1 When all measurements have been completed, set the instrument to warm to room temperature or somewhat above. Cleaning cells above a temperature of  $55^\circ\text{C}$  is not recommended.

10.7.2 When the desired cleaning temperature is reached:

10.7.2.1 For instruments with non-removable cells, remove strings, rotors, and cell caps, when used, then proceed with 10.7.3.

10.7.2.2 For instruments with removable cells, either follow instructions for non-removable or remove cells from instrument. The removable cells are to be cleaned by generally following the instructions in 10.7.3.

**TABLE 1 Precision**

Yield Stress Precision		
	Repeatability	Reproducibility
Unused Oils	35 Pa	70 Pa
Used Gasoline Engine Oils		
Yield Stress ≤ 35 Pa	35 Pa	35 Pa
Yield Stress > 35 Pa	70 Pa	70 Pa
Apparent Viscosity Precision		
	Repeatability	Reproducibility
Unused Oils		
Viscosity: 4300 to 20 000 mPa · s	6.3% of mean	8.2% of mean
Viscosity: >20 000 mPa · s	7.5% of mean	14.6% of mean
Used Gasoline Engine Oils		
Yield stress ≤ 35 Pa	11% of mean	15% of mean
Yield stress > 35 Pa	25% of mean	34% of mean

### 10.7.3 Cleaning Cells:

10.7.3.1 Remove oil samples from cells by using a vacuum hose.

10.7.3.2 Using an appropriate solvent, rinse, the cells at least three times with approximately 15 mL of an appropriate solvent for each rinse. Then rinse once with acetone.

10.7.3.3 Remove traces of residual solvent by flushing cell with dry air or preferably with a vacuum hose to prevent contamination with house air. (**Warning**—When flushing cells with air, be sure that the air is clean and free from oil, water and other contaminants as these could be left in the cell. House air is frequently contaminated.)

10.7.4 Clean rotors with appropriate sample solvent, and dry.

## 11. Calculation of Yield Stress and Apparent Viscosity

11.1 Yield stress is given by the following equation:

$$Y_s = 3.5 M \quad (10)$$

where:

$Y_s$  = yield stress, Pa, and

$M$  = applied mass, g, at which rotation was observed.

11.2 The viscosity is given by the following equation when using the cell constant (C) obtained in Eq 11:

$$\eta_a = C \cdot t \cdot 3/R \quad (11)$$

where:

$\eta_a$  = apparent viscosity in mPa·s, (cP),

$C$  = cell constant,

$t$  = time for number ( $R$ ) of complete revolutions of the rotor, and

$r$  = number of revolutions timed; either one or three revolutions.

## 12. Report

12.1 *Apparent Viscosity and Yield Stress*—Report the final test temperature, apparent viscosity, and yield stress by Test Method D4684, Procedure A.

12.2 *Yield Stress*—Report as less than the value at which rotation was observed; that is, if rotation was observed with a mass of 20 g, report the yield stress as <70 Pa (20 g × 3.5). If the rotor did not move with the application of 100 g of total weight, report the yield stress as >350 Pa.

NOTE 10—If the rotation was observed with a mass of 10 g, the minimum weight, report the yield stress as <35 Pa rather than as 0 Pa.

12.3 *Apparent Viscosity*—Report as follows:

12.3.1 If the apparent viscosity is less than 5000 mPa·s (cP), then report the apparent viscosity as less than 5000 mPa·s (cP).

12.3.2 If the apparent viscosity is between 5000 and 100 000 mPa·s (cP), then report the apparent viscosity to the nearest 100 mPa·s (cP).

12.3.3 If the apparent viscosity is between 100 000 and 400 000 mPa·s (cP), then report the apparent viscosity to the nearest 1000 mPa·s (cP).

12.3.4 If the apparent viscosity is greater than 400 000 mPa·s (cP), then the apparent viscosity should be reported as greater than 400 000 mPa·s (cP).

12.3.5 If the rotor did not move with the application of the 150 g weight, report that the sample was “Too Viscous To Measure” (or “TVTM”).

## 13. Precision and Bias <sup>6</sup>

13.1 *Precision*—The precision of this test method for unused oils was determined by the statistical examination of results from an interlaboratory program. For the thermoelectrically cooled MRV's, the program included 10 to 11 samples tested in 7 to 9 laboratories at −25, −30, −35 and −40°C. For direct refrigeration cooled MRV's, the program included 20 samples tested in 6 laboratories at −25, −30, −35 and −40°C. These samples consisted of multigrade engine oils and base stocks that had a yield stress range of less than 35 to 210 Pa and a apparent viscosity range from 4300 to 270 000 mPa·s.

The precision of this test method for used gasoline engine oils was determined by the statistical examination of interlaboratory test results at −25°C and −30°C.

13.1.1 *Repeatability*—In Table 1, the difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, in the normal and correct operation of the test method, exceed the following values only in 1 case in 20.

<sup>6</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Reports D02-1404, D02-1612, D02-1613, and D02-1654.

13.1.2 *Reproducibility*—In **Table 1**, the difference between two single and independent results obtained by different operators working in different laboratories on identical test materials would, in the long run, exceed the following values only in 1 case in 20.

#### 13.2 *Bias*:

13.2.1 *Bias (Unused Gasoline Engine Oils)*—No statement on bias is being made for this test method, because there is no accepted reference material suitable for determining absolute bias.

#### 13.2.2 *Relative Biases*:

13.2.2.1 *Yield Stress*—There was no significant relative bias between yield stresses measured by Procedure A and those measured by Procedure B for oils with a yield stresses less than 105 Pa. No statement can be made with those with yield stress greater than 105 Pa.

13.2.2.2 *Viscosity*—There is no statistically significant relative bias between the viscosities as measured by either Procedure A or Procedure B.

13.2.3 *Bias (Used Gasoline Engine Oils)*—Since there is no accepted reference material suitable for determining the bias for this test method, no statement on bias is being made.

### Procedure B

## 14. Calibration and Standardization

14.1 Calibrate the temperature sensor while attached to the temperature controller. The sensed temperature shall be verified using a reference thermometer noted in **6.3** at a minimum of three temperatures. Make these temperature measurements at least 5°C apart to establish a calibration curve for this combination of temperature sensor and controller.

NOTE 11—All temperatures in this test method refer to the actual temperature and not necessarily the indicated temperature.

14.2 The calibration of each viscometric cell (viscometer constants) can be determined with the viscosity standard and the following procedure at –20°C:

14.2.1 Use steps in **15.2-15.2.5**.

14.2.2 Program the temperature controller to cool the mini-rotary viscometer block to –20°C within 1 h or less, then start the program.

14.2.3 Allow the oil in the cells to soak at  $-20 \pm 0.2^\circ\text{C}$  for at least 1 h, making small temperature adjustments, if necessary, to maintain the test temperature.

14.2.4 At the end of the soak period, record the temperature reading (test temperature) and remove the cover of the viscometer cell.

14.2.5 Proceed to **15.3.1-15.3.3**.

14.2.6 Perform step in **15.4.1**.

14.2.7 Repeat **14.2.5** and **14.2.6** for each of the remaining cells, taking the cells in order from left to right.

14.2.8 Calculate the viscometer constant for each cell (rotor/stator combination) with the following equation:

$$C = \eta_o/T \quad (12)$$

$\eta_o$  = Viscosity of the standard oil, cP (mPa·s) at –20°C,

$C$  = cell constant with 150 g mass, Pa, and

$T$  = time in seconds for three complete revolutions.

14.2.9 If any cell has a calibration constant more than 10 % higher or lower than the average for the other cells, the fault may be a problem with rotor operation. Examine rotor for damage, and recalibrate the instrument.

14.3 If corrected values for controller temperature and thermometer deviate by more than the tolerance, use **X2.2** to assist in determining the fault.

## 15. Procedure

15.1 Program the temperature controller to control the mini-rotary viscometer block temperature as outlined in **Table X1.1** or **Table X1.2**. The programmed temperature is the temperature in **Table X1.1** or **Table X1.2** plus the appropriate temperature correction factor determined in **14.1**. **Table X1.3** lists the nominal times to reach a particular test temperature.

### 15.2 *Test Sample and Viscometric Cell Preparation*:

15.2.1 Remove the nine rotors from the viscometric cells and ensure that both the cells and rotors are clean. See **15.6** for the cleaning procedure.

15.2.2 Place a  $10 \pm 1.0$  mL oil sample in each cell.

15.2.3 Install the rotors in the proper stators and install the upper pivots.

15.2.4 Place the loop of the 700-mm long string over the cross-arm at the top of the rotor shaft and wind all but 200 mm of the length of the string around the shaft. Do not overlap strings. Loop the remaining end of the string over the top bearing cover. Orient the rotor such that an end of the cross-arm at the top of the rotor shaft is pointing directly forward. If available, secure cross-arm with locking pin. If the rotations are manually timed, it is helpful to color one end of the cross-arm.

15.2.4.1 The string may be prewound around the shaft before installation of the rotor in **15.2.3**.

15.2.5 Place the housing cover over the viscometric cells to minimize the formation of frost on the cold metal parts exposed to air. In some climates, it may be necessary to flush the cover with a dry gas (for example, dry air or nitrogen) to minimize the frost formation.

15.2.6 Start the programmed temperature profile. The program will warm the oil samples to  $80 \pm 1^\circ\text{C}$  and maintain at  $80 \pm 1^\circ\text{C}$  for 2 h to allow solution of any material not in true solution at room temperature.

15.2.7 At the end of the 2-h soak at 80°C, the cooling cycle starts to cool the samples in accordance with the programmed cooling sequence as programmed in **15.1**.

15.2.8 At the completion of the temperature profile, the temperature of the block should be within 0.2°C of the desired test temperature when measured by a thermometer other than the temperature controller in the same thermometer well used during calibration. If the block temperature is within this range, proceed with the yield stress and viscosity measurements within 30 min of the completion of the temperature profile (see **15.3**).

15.2.8.1 If the final temperature of the block is 0.2 to 0.5°C warmer than the desired temperature, proceed as follows. Set the temperature controller to bring the block temperature to the correct test temperature and then hold at the correct test

temperature for 30 min before proceeding. This entire temperature correction should not take longer than 1 h. The data obtained in this way are considered valid test results, otherwise the test is invalid.

15.2.8.2 If the final test temperature is more than 0.2°C cooler or more than 0.5°C warmer than the preselected test temperature, then the test is NOT VALID for the preselected temperature. For Information Only, the yield stress and viscosity may be measured without further temperature adjustment. These results are characteristic of the actual temperature, not the preselected one.

15.2.9 If the final temperature as noted in 15.2.8 is in error in either direction by more than 0.2°C, see X2.2 before starting another test.

15.2.10 With models CMRV-4 and higher, if the program reports cooling profile out of tolerance, the operation of the instrument shall be thoroughly reviewed for correct operation. With models earlier than CMRV-4, check the logged data for excessive temperature deviation. See X2.2-X2.4.

### 15.3 *Measurement of the Yield Stress:*

15.3.1 Beginning with the cell farthest to the left of the instrument, follow this procedure for each cell in turn.

15.3.2 Align the pulley wheel with the rotor shaft for the cell to be tested, such that the string hangs past the front of the housing. Make sure that the weights clear the edge of the bench during testing.

15.3.3 Remove the string from the upper bearing support and carefully place it over the pulley wheel so as not to disturb the test oil. (Do not allow the rotor shaft to turn.)

15.3.4 Follow the instrument model specific instructions:

NOTE 12—For users with CMRV-4 or newer instruments wishing to manually time yield stress and viscosity, follow the instructions in 15.3.4.1 and 15.4.1.1, respectively.

### **Model CMRV-3 or Earlier**

15.3.4.1 Visually observe the rotor for movement of the cross-arm. (Do not measure yield stress by way of the electronic optics.)

15.3.4.2 For instruments not equipped with locking pins: carefully, so as not to disturb the gel structure, attach a 10-g mass to the string and gently suspend the weight on the string. Proceed to 15.3.4.4.

15.3.4.3 For instruments equipped with locking pins: suspend the 10-g mass on the string, then raise the locking pin.

15.3.4.4 If the end of the cross-arm does not move at least 3 mm in 15 s (approximately twice the diameter of the cross-arm or 13° of rotation) then record that the sample has yield stress. Proceed to 15.3.4.5. If movement is detected, record weight and proceed to 15.4.

15.3.4.5 If no movement is detected, for instruments without locking pins, hold weight assembly and add 10 g, then proceed with 15.3.4.4. If equipped with locking pins, lower the locking pin to re-engage cross-arm. Add 10 g to the weight assembly, raise the locking pin, and proceed with 15.3.4.4.

### **Model CMRV-4 or Later Model MRV**

15.3.4.6 The operator shall follow the on-screen instructions for the addition of weight increments.

15.3.4.7 For instruments with locking pins: suspend 10-g weight cage on string, press the flashing start button, then immediately raise the locking pin and follow the on-screen instructions.

15.3.4.8 If additional weight is requested, capture cross-arm in locking pin, then add one additional 10 g weight, and follow the on-screen instructions. Press the flashing start button, then immediately raise the locking pin. Repeat procedure until no additional weight is requested. Proceed to 15.4.

15.3.4.9 For instruments without locking pins: carefully suspend and hold the 10-g weight cage on the string without jerking rotor, and follow the on-screen instructions. Press the flashing start button, and immediately release the weight cage.

15.3.4.10 If no movement is detected, then carefully weight the cage. Add next 10-g weight increment as indicated on the computer screen, without pulling on the string, and follow the on-screen instructions. Press the flashing start button, and immediately release weight cage. Repeat procedure until no additional weight is requested. Proceed with 15.4.

NOTE 13—When the 10-g load is first applied, some oils may show momentary movement of the cross-arm. If there is no further movement of the cross-arm for 15 s, disregard the initial movement.

### 15.4 *Measurement of Apparent Viscosity:*

15.4.1 Follow the instrument model specific instructions.

#### **For CMRV-3 or Earlier**

15.4.1.1 Attach a 150-g mass to the string and slowly suspend the weight on the string. Start the timer when the cross-arm of the rotor shaft points directly forward and continue timing in accordance with the following constraints.

15.4.1.2 If the first half-revolution requires less than 10 s, measure and record the time for the first three revolutions.

15.4.1.3 If the first half-revolution requires 10 s or greater, measure and record the time for the first revolution and identify it as the time for one revolution.

15.4.1.4 If the first revolution has not been completed in 60 s, end the measurement. Record the time as greater than 60 s for one revolution. Report that the viscosity is greater than the value calculated in 16.2.

15.4.1.5 If the time for the first three revolutions is less than 4 s, record the time as less than 4 s. Report that the viscosity is less than the value calculated in 16.2.

#### **For CMRV-4 and Later**

15.4.1.6 Follow the on-screen instructions, press the start button, and slowly suspend the weight on the string. Timing will automatically begin with the first movement. Do not remove weight while viscosity LED on instrument is flashing. The time and viscosity will be displayed. Proceed to 15.5.

15.5 Repeat 15.3–15.4 for each of the remaining cells in order from left to right.

### 15.6 *Cleaning:*

15.6.1 After all of the cells have been completed, exit the cooling program and turn on the heater to warm the viscometric cells to room temperature or somewhat higher. The temperature shall not exceed 50°C.

15.6.2 Remove the upper rotor pivots and the rotors.



15.6.3 With a vacuum, remove the oil samples and rinse the cells with an oil solvent several times, followed by two washings with acetone. Use a vacuum to remove the solvent from the cells after each rinse and allow the acetone to evaporate to dryness after the final rinse.

15.6.4 Clean the rotors in a similar manner.

## 16. Calculation of Yield Stress and Apparent Viscosity

16.1 Yield stress is given by the following equation:

$$Y_s = 3.5 M \quad (13)$$

where:

$Y_s$  = yield stress, Pa, and

$M$  = applied mass, g.

16.2 The viscosity is given by the following equation when using the cell constant obtained in Eq 11:

$$\eta_a = C t 3/r \quad (14)$$

where:

$\eta_a$  = apparent viscosity in mPa·s, (cP)

$C$  = cell constant obtained in Eq 11,

$t$  = time for number ( $r$ ) of complete revolutions of the rotor, and

$r$  = number of revolutions timed.

## 17. Report

17.1 *Apparent Viscosity and Yield Stress*—For unused oils, report the final test temperature and either the apparent viscosity or the existence of yield stress, but not both, by Test Method D4684, Procedure B. For used oils, report both apparent viscosity and yield stress by Test Method D4684, Procedure B.

17.2 *Yield Stress*—Report as less than the value at which rotation was observed.

17.3 *Apparent Viscosity*—Report as follows:

17.3.1 If the apparent viscosity is less than 5000 mPa·s (cP), then report the apparent viscosity as less than 5000 mPa·s (cP).

17.3.2 If the apparent viscosity is between 5000 and 100 000 mPa·s (cP), then report the apparent viscosity to the nearest 100 mPa·s (cP).

17.3.3 If the apparent viscosity is between 100 000 and 400 000 mPa·s (cP), then report the apparent viscosity to the nearest 1000 mPa·s (cP).

17.3.4 If the apparent viscosity is greater than 400 000 mPa·s (cP), then the apparent viscosity should be reported as greater than 400 000 mPa·s (cP).

17.3.5 When employing software that provides three viscosity values, the first value shall be reported as the apparent viscosity by Test Method D4684. If desired, report all three

values, taking care to also report the sequence of the values. Never report a value that is the average of the three measured values.

## 18. Precision and Bias<sup>7</sup>

18.1 *Precision (Unused Oils)*—The precision of this test method as determined by the statistical examination of interlaboratory test results is as follows:

18.1.1 *Yield Stress*—In the case of pass-fail data, no generally accepted method for determining precision is currently available.

18.1.2 *Apparent Viscosity*:

18.1.2.1 *Repeatability*—The difference between successive 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 1 case in 20. The repeatability as a percent of the mean apparent viscosity is shown as follows:

Test Temperature, °C	Repeatability, Percent of Mean
−15	4.2
−20	7.3
−25	11.7
−30	9.3
−35	13.2
−40	19.8

18.1.2.2 *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, exceed the following values only in 1 case in 20. The reproducibility as a percent of the mean apparent viscosity is shown as follows:

Test Temperature, °C	Reproducibility, Percent of Mean
−15	8.4
−20	12.1
−25	17.5
−30	18.4
−35	35.8
−40	34.1

18.1.3 The interlaboratory program included nine test oils at the −15°C test temperature with eleven laboratories participating. Nine test oils were included at the −20°C test temperature with eleven laboratories participating. The −25°C test temperature included 18 test oils with 14 laboratories participating. Nine test oils were evaluated at −30°C in 13 laboratories. At the −35 and −40°C test temperatures, six test oils were evaluated in twelve laboratories.

18.2 *Precision (Used Diesel Engine Oils)*—The precision of this test method as determined by the statistical examination of interlaboratory test results is as follows:

18.2.1 *Yield Stress*:

18.2.1.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus

<sup>7</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Reports D02-1212, D02-1249, D02-1277, and D02-1517.

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 1 case in 20:

Test Temperature, °C	Repeatability, Pa
–20	$1.735 \cdot (X + 1)$
–25	$1.014 \cdot (X + 1)$

where:

X = mean value in Pa

NOTE 14—When no yield stress is detected (movement with 10-g weight), X = 0.

**18.2.1.2 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, exceed the following values only in 1 case in 20.

Test Temperature, °C	Reproducibility, Pa
–20	$2.993 \cdot (X + 1)$
–25	$2.976 \cdot (X + 1)$

where:

X = mean value in Pa

### 18.2.2 Apparent Viscosity:

**18.2.2.1 Repeatability**—The difference between successive 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 1 case in 20. The repeatability as a percent of the mean apparent viscosity is shown as follows:

Test Temperature, °C	Repeatability, Percent of Mean
–20	14.3
–25	10.3

**18.2.2.2 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, exceed the following values only in 1 case in 20. The reproducibility as a percent of the mean apparent viscosity is shown as follows:

Test Temperature, °C	Reproducibility, Percent of Mean
–20	21.1
–25	20.8

**18.2.2.3** The interlaboratory program included nine laboratories and nine test oils at the –20 and –25°C test temperatures. The used oils included end-of-test drain samples from Mack T8, Mack T8E, Cummins M11-EGR and Mack T10 engine tests, with soot loadings (as measured by thermogravimetric analysis) ranging from approximately 5 to 9 % (see D02-1517).<sup>7</sup>

**18.3 Bias**—Since there is no accepted reference material suitable for determining the bias for this test method, no statement on bias is being made.

## 19. Keywords

19.1 low temperature flow properties; low temperature viscosity; mini-rotary viscometer; pumping viscosity; used diesel engine oil; viscosity; yield stress

## APPENDIXES

### (Nonmandatory Information)

## X1. TEMPERATURE PROFILES FOR TEST TEMPERATURES

X1.1 See [Tables X1.1-X1.3](#).

**TABLE X1.1 Temperature Profile for Test Temperatures –20 to –40°C**

Segment Time h:min	Segment Temperature <sup>A</sup>			Allowable Temperature Change <sup>B</sup> °C
	Beginning °C	Final °C	Rate of Change °C/h	
nominally 0:20	above 20	to 80		
2:00	80	to 80		±1.0
nominally 0:20	80	to 0		
nominally 0:03	0	to –3.0		
nominally 0:07	–3.0	to –4.0	8.5	±0.5
nominally 0:10	–4.0	to –5.0	6.0	±0.2
6:00	–5.0	to –8.0	0.5	±0.2
36:00	–8.0	to –20.0	0.33	±0.2
Hold at this point for –20°C test temperature. <sup>C</sup>				
2:00	–20.0	to –25.0	2.5	±0.2
Hold at this point for –25°C test temperature. <sup>C</sup>				
2:00	–25.0	to –30.0	2.5	±0.2
Hold at this point for –30°C test temperature. <sup>C</sup>				
2:00	–30.0	to –35.0	2.5	±0.2
Hold at this point for –35°C test temperature. <sup>C</sup>				
2:00	–35.0	to –40.0	2.5	±0.2
Hold at this point for –40°C test temperature. <sup>C</sup>				

<sup>A</sup> If the dual control loop concept is used, the bath set point temperatures should be 5°C below the corresponding block temperature desired. The maximum bath temperature shall not exceed –5°C.

<sup>B</sup> Holding the temperature variation to less than ±0.1°C improves the precision and reproducibility of your viscosity measurements.

<sup>C</sup> The measurement of yield stress and apparent viscosity are to be made within 30 min of reaching the test temperature.

**TABLE X1.2 Temperature Profile for Test Temperatures –10 and –15°C**

Segment Time h:min	Segment Temperature <sup>A</sup>			Allowable Temperature Change <sup>B</sup> °C
	Beginning °C	Final °C	Rate of Change °C/h	
nominally 0:20	above 20	to 80		
2:00	80	to 80		±1.0
nominally 0:20	80	to 10		
nominally 0:03	10	to 7.0		
nominally 0:07	7.0	to 6.0	8.5	±0.5
nominally 0:10	6.0	to 5.0	6.0	±0.2
6:00	5.0	to 2.0	0.5	±0.2
36:00	2.0	to –10.0	0.33	±0.2
Hold at this point for –10°C test temperature. <sup>C</sup>				
2:00	–10.0	to –15.0	2.5	±0.2
Hold at this point for –15°C test temperature. <sup>C</sup>				

<sup>A</sup> If the dual control loop concept is used, the bath set point temperatures should be 5°C below the corresponding block temperature desired. The maximum bath temperature shall not exceed –5°C.

<sup>B</sup> Holding the temperature variation to less than ±0.1°C improves the precision and reproducibility of your viscosity measurements.

<sup>C</sup> The measurement of yield stress and apparent viscosity are to be made within 30 min of reaching the test temperature.

**TABLE X1.3 Nominal Elapsed Time to Test Temperature**

Test Temperature, °C	Nominal Elapsed Time, h
–10	45
–15	47
–20	45
–25	47
–30	49
–35	51
–40	53

## X2. SUPPORTING OPERATIONAL INFORMATION

**X2.1 Temperature Controller** is the most critical part of this procedure. For systems using a liquid media to control cell temperature, the temperature control system can be a single-loop programmable controller to control the block temperature. A process controller that has a proportional band with integral reset and derivative rate control, sometimes referred to as a

PID controller, is suitable for controlling the temperature. This programmable controller has one control loop and one temperature sensor that provides the appropriate information to the controller to hold the temperature at the programmed set temperature. It has an internal clock that controls the execution of the program. The controller shall be connected so that only

heat is supplied to the block during the first 2 h and 20 min of the temperature profile described in [Table X1.1](#) or [Table X1.2](#) or the heat shall be applied uniformly across all viscometer cells.

For systems using a liquid media to control cell temperature, the temperature control during the remaining portion of the temperature profile shall be obtained by controlling the coolant flow. This control system shall have a minimum temperature sensitivity of 0.1°C and be able to change the temperature at a prescribed rate. When the control system's proportional band, integral (reset), and derivative (rate) parameters are optimized, the temperature excursions above and below the profile shall be no greater than 0.2°C at a temperature below -5°C. The temperature sensor can be a platinum resistance thermal detector, a thermistor, or a thermocouple. A platinum resistance thermal detector or thermistor sensor is preferred. A 1/8-in. (3.2-mm) diameter temperature probe can be installed directly into the 1/8-in. diameter well located at the back of the block between cells Nos. 4 and 6. Alternatively, the temperature sensor can be inserted into one of the thermometer wells.

**NOTE X2.1**—The sensor is placed in the same unit that is being controlled. The sensor should be placed in the block if the supply of coolant is being controlled. Alternatively, the sensor would be placed in the bath if the bath temperature was being controlled. Do not try to control the block temperature by sensing the block temperature and controlling the refrigeration system. For systems using direct refrigeration (no external liquid circulating systems), temperature control shall be obtained by controlling the degree of heating applied to the refrigerant gas cooled block. This control system shall have a minimum temperature sensitivity of 0.1°C and be able to change the temperature of the block at the prescribed rate required for this test. Use of a resistance temperature detector (RTD) is both sensitive and rapid in response and, for this reason, is made integral, together with the temperature controlling module, for these instruments.

**NOTE X2.2**—An internally delayed start for the controller is a desirable feature since this will allow starting the temperature profile unattended.

**X2.2** If the final temperature is in error in either direction by more than 0.2°C, do the following before starting another analysis:

**X2.2.1** Check the thermometer calibration. For liquid in glass thermometers, check the ice point. An error in the ice point usually indicates separation of the liquid at some point in the thermometer.

**X2.2.2** Check temperature sensor of the temperature controller for accuracy, in accordance with [9.1](#).

**X2.2.3** For those instrument that require external liquid circulation:

**X2.2.3.1** Check whether the coolant is flowing or whether there is adequate coolant in the reservoir.

**X2.2.3.2** For cold sources operating below -20°C, replace methanol if wet, as indicated by ice crystals in the top of the cold source reservoir. Cold methanol absorbs water, and as it absorbs water, its cooling capacity decreases. In high humidity areas, it may be necessary to change the methanol once a month. Other heat transfer can be used, but it should be similar to methanol in viscosity and heat capacity at the bath temperature.

**X2.2.4** Check to see that the refrigeration system is working properly. The instrument manual in conjunction with the bath manufacturer will be sources of appropriate information.

**X2.2.5** If manually programmed or using a custom profile, examine the temperature profile program for an error and make the appropriate corrections.

**X2.3** The simplest way to check a liquid in glass thermometer calibration is to check its ice point. Other calibration sources are available for both liquid in glass and electronic temperature sensor and are appropriate if they are sufficiently accurate.

**X2.4** For some instruments, the software controlling temperature creates a temperature log during the test. For other instruments a sensor connected to a strip chart recorder will provide the information to determine if temperature deviations are greater than those permitted in [Table X1.1](#) or [Table X1.2](#) and correct accordingly.

**X2.5** Verify that the preheat program for 80°C lasts for a minimum of 2 h. If not, correct accordingly to the owner's manual or through the instrument manufacturer.

## REFERENCES

- (1) Stambaugh, R. L., and O'Mara, J. H., "Low Temperature Flow Properties of Engine Oils," SAE Paper No. 821247 or 820509.
- (2) Shaub, H., Smith, M. F., Jr., and Murphy, C. K., "Predicting Low Temperature Engine Oil Pumpability with the Mini-Rotary Viscometer," SAE Paper No. 790732, published in SAE SP-460 and ASTM STP-621-S4.
- (3) Stewart, R. M., Shaub, H., Smith, M. F., Jr., and Selby, T. W., "Summary of ASTM Activities on Low Temperature Engine Oil Pumpability," SAE Paper No. 821206.
- (4) Smith, M. F., Jr., "Better Prediction of Engine Oil Pumpability Through a More Effective MRV Cooling Cycle," SAE Paper No. 831714.
- (5) Henderson, K. O., Manning, R. E., May, C. J., and Rhodes, R. B., "New Mini-Rotary Viscometer Temperature Profiles That Predict Engine Oil Pumpability," SAE Paper No. 850443.
- (6) ASTM Research Report D02-1442, "Cold Starting and Pumpability Studies in Modern Engines," ASTM International, W. Conshohocken, PA, 1999 (order #COLDSTART).
- (7) Shaub, Harold, Editor, "Oil Flow Studies at Low Temperature in Modern Engines," ASTM STP 1388, ASTM International, W. Conshohocken, PA, 2000.



## SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D4684–07<sup>e1</sup>) that may impact the use of this standard. (Approved Dec. 1, 2008.)

(I) Incorporated direct refrigeration instruments.

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D4684–02a) that may impact the use of this standard. (Approved Jan. 15, 2007.)

- (1) Added new terms to Section 3.
- (2) Revised wording for block composition in 6.1.
- (3) Revised cell cap in 6.1.2.
- (4) Revised 6.2 to restate weight tolerance.
- (5) Reworded 6.4.
- (6) Added new Procedure A in Sections 8-13. This procedure, which was developed to improve the precision of the test method, includes these significant modifications to the equipment and procedure compared with Procedure B:

Calibration at –25°C using oil with approximate viscosity of 60 000 mPa.s (3.2.1, 7.1, 9.2)

Calibration constants based on two determinations (9.9 through Section 10)

Limits on deviation of cell constants (9.11)

Filling of unused cells (10.1.2)

Making measurements on cell from left to right during calibration and during measurements (9.7 and 10.6.2)

Standardization of method of turning rotor after measurement of yield stress (10.6.3.2)

- (7) Added Procedure B in Sections 14-18, extracted from D4684-02 as published with one minor editorial change.
- (8) Added 13.2, precision and bias for used gasoline engine oils.

*ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.*

*This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.*

*This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).*