Designation: D4684 – 17

Standard Test Method for Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature[1]

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 (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. 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 °C and –40 °C. The precision is stated for test temperatures from –40 °C to –15 °C. The viscosity measurements are made at a shear stress of 525 Pa over a shear rate of 0.4 s^{-1} to 15 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 contain 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 mPa·s 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 SI based unit of milliPascal second (mPa·s) for viscosity which is equivalent to, centiPoise (cP).

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.*

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

E563 Practice for Preparation and Use of an Ice-Point Bath as a Reference Temperature

E644 Test Methods for Testing Industrial Resistance Thermometers

E1137 Specification for Industrial Platinum Resistance Thermometers

E2877 Guide for Digital Contact Thermometers

2.2 *ISO Standard:*[3]

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

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

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[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.

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

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

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

3. Terminology

3.1 *Definitions:*

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

3.1.2 *digital contact thermometer (DCT), n*—an electronic device consisting of a digital display and associated temperature sensing probe.

3.1.2.1 *Discussion*—This device consists of a temperature sensor connected to a measuring instrument; this instrument measures the temperature-dependent quantity of the sensor, computes the temperature from the measured quantity, and provides a digital output. This digital output goes to a digital display and/or recording device that may be internal or external to the device. These devices are sometimes referred to as "digital thermometers."

3.1.2.2 *Discussion*—PET is an acronym for portable electronic thermometers, a subset of digital contact thermometers (DCT).

3.1.3 *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.

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

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

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

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

where:

$\dot{\gamma}$ = shear rate at the surface of the rotor in reciprocal seconds, s^{-1},
Ω = 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,

$$\dot{\gamma} = 63/t \quad (3)$$

3.1.6 *shear stress, n*—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.

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

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

where:

T_r = torque applied to rotor, N·m,
M = applied mass, g,
R_o = radius of the shaft, mm,
R_t = radius of the string, mm,
τ = shear stress at the rotor surface, Pa, and
h = height of the rotor, mm.

For the dimensions given in 6.1.1,

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

$$\tau = 3.5\,M \quad (7)$$

3.1.7 *viscosity, n*—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].

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *calibration oils, n*—those oils that establish an instrument's reference framework of apparent viscosity versus speed, from which the apparent viscosities of test oils are determined.

3.2.2 *cell constant, n*—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, n*—any oil for which the apparent viscosity and yield stress are to be determined by this test method.

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

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

3.2.6 *yield stress, n*—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 °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.[4] These documented field failing oils all consisted of oils normally tested at –25 °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:*

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[4] Pumpability Reference Oils (PRO) 21 through 29.

5.2.1 For oils to be tested at −20 °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**).[5] Significance for the −35 °C and −40 °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 °C or −10 °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 °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 °C and −10 °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 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 mm ± 0.08 mm
Length of rotor	20.00 mm ± 0.14 mm
Inside diameter of cell	19.07 mm ± 0.08 mm
Radius of shaft	3.18 mm ± 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 mm ± 1 mm 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 mm ± 1 mm in diameter and the bottom half is 19 mm 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 mm ± 1 mm. 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 nine disks and a disk holder, each with a mass of 10 g ± 0.1 g.

6.2.2 *Viscosity Measurement*—A mass of 150 g ± 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 Profile*—The temperature profile is fully described in Table X1.1 and Table X1.2.

6.4 *Temperature Measuring Device*—Use either a DCT meeting the requirements described in 6.4.1 or liquid-in-glass thermometers described in 6.4.2. A calibrated DCT or calibrated low temperature liquid-in-glass thermometer shall be used as the thermometer for temperature measurement below 25 °C independent of the instrument's temperature control, and shall be located in the thermowell.

NOTE 1—The display device and sensor must be correctly paired. Incorrect pairing will result in temperature measurement errors and possibly irreversible damage to the electronics of the display.

6.4.1 Digital contact thermometer requirements:

Criteria	Minimum Requirements
DCT	E2877 Class B
Temperature range	–45 °C to 100 °C
Display resolution	0.1 °C minimum, preferably 0.01 °C
Sensor type	RTD, such as a PRT or thermistor
Sensor, metal sheathed	3 mm O.D. with an sensing element less than 30 mm in length to be used with a thermowell sleeve, 6 mm O.D. × 58 mm long with a ~3 mm hole in center.
Sensor, glass sheathed	6 mm O.D. with a sensing element less than 12 mm in length
Display accuracy	±50 mK (±0.05 °C) for combined probe and sensor
Response time	less than or equal to 25 s as defined in Specification E1137
Drift	less than 50 mK (0.05 °C) per year
Calibration Error	less than 50 mK (0.05 °C) over the range of intended use.
Calibration Range	–40 °C to 85 °C
Calibration Data	4 data points evenly distributed over the range of –40 °C to –1 °C and included in calibration report.
Calibration Report	From a calibration laboratory with demonstrated competency in temperature calibration which is traceable to a national calibration laboratory or metrology standards body

NOTE 2—With respect to DCT probe immersion depth, a procedure to determine minimum immersion depth can be found in Guide E2877, Section 5.3, or Test Methods E644, Section 7.

6.4.1.1 The DCT calibration drift shall be checked at least annually by either measuring the ice point or against a reference thermometer in a constant temperature bath at the prescribed immersion depth to ensure compliance with 6.4.1.With respect to an ice bath, Practice E563 provides guidance on the preparation and use of an ice bath. However for this use variance from the specific steps, such as water source, is permitted provided preparation is consistent. The basis for the variance is due to the reference being used to track change in calibration not verification.

NOTE 3—When a DCT's calibration drifts in one direction over several calibration checks, that is, ice point, it may be an indication of deterioration of the DCT.

6.4.2 For liquid-in-glass thermometers, LiG, two are required. One LiG shall be a calibrated 76 mm partial immersion

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[5] The boldface numbers in parentheses refer to the references at the end of this standard.

thermometer with a scale from +5 °C to 1 degree less than the lowest test temperature in 0.2 °C subdivisions. This low temperature LiG thermometer shall have a report of calibration showing the temperature deviation at each calibrated test temperature. The second LiG thermometer shall be a 76 mm partial immersion thermometer graduated from at least +70 °C to 90 °C in 1 °C subdivisions, which is used to verify the preheat temperature.

6.4.2.1 *Calibration Check*—Verify the low temperature thermometer at least annually against a reference thermometer in a constant temperature bath or an ice bath. The thermometer is to be inserted to its immersion depth. If using an ice bath, the ice point reading is to be taken within 60 min after the thermometer has been at test temperature for at least 3 min. If the corrected temperature reading deviates from the reference thermometer or the ice point then repeat this calibration check. If the thermometer deviates from the reference value on two successive checks then a full thermometer recalibration is needed.

6.4.2.2 *Recalibration*—A complete recalibration of the liquid-in-glass thermometer, while permitted, is not necessary in order to meet the accuracy ascribed to liquid-in-glass thermometer's design until the thermometers corrected measured temperature deviates from the reference thermometer or ice point by one scale division, or until five years has elapsed since the last full calibration.

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 *Low Cloud-point Newtonian Oil*—Calibration oil of approximately 30 Pa·s viscosity at –20 °C for Procedure B or 60 Pa·s at –25 °C for Procedure A for calibration of the viscometric cells. The calibration oil shall be obtained from suppliers complying with ISO Guide 34 and ISO 17025 with traceability to a national metrology institute (NMI).

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.)

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.

Procedure A (TE Cooled and Direct Refrigeration Instruments)

9. Calibration and Standardization

9.1 *Temperature Control Calibration Procedure*—Calibrate the MRV temperature control by comparing the instrument's displayed temperature against a thermometer in the thermowell. The thermometer used shall meet the requirements in 6.4.

9.1.1 Place 10 mL of a typical test fluid and rotor in each cell. If required, place cell caps over each cell then place cover on instrument. Cell caps shall not be used on direct refrigeration instruments (see 6.1.2).

9.1.2 Place the thermometer in the thermowell. See Note 4. This thermowell is to be used for all temperature measurements below 25 °C.

NOTE 4—Prior to inserting the thermometer or DCT probe in the thermowell, place several drops (~3) of a heat transfer fluid such as 50/50 water/ethylene glycol mix, CCS reference oil CL100 or a dewaxed low viscosity mineral oil in the thermowell.

9.1.3 Make these temperature measurements at 80 °C then at least three measurements that are 5 °C apart from –5 °C to the lowest test temperature used, including both end points to establish a calibration curve for this combination of thermometer and the instrument's temperature control. Make at least two temperature measurements at every calibration temperature with at least 10 min between observations.

NOTE 5—All temperatures in this test method refer to the actual temperature and not necessarily the indicated temperature.

9.1.4 Follow the instrument manufacturers instructions for correcting the instrument's measured temperature. Alternatively establish a correction equation between thermometer and the instruments's measured temperature then adjust each temperature of the cooling program by the offset determined with the correction equation.

9.2 *Viscometer Cell Calibration*—The calibration constant of each rotor/stator combination is determined by conducting two tests at –25 °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 6—Once a set of rotors has been calibrated in an instrument, subsequent calibration checks can be single determinations if the criteria of 9.11 are met.

9.2.2 Use the same 150 g mass for both calibration and viscosity measurements. However, different units may be used for calibration and viscosity measurements provided they are certified to be 150 g ± 0.1 g.

9.3 Following steps in 10.1, prepare the cells for calibration using the calibration oil as the sample.

9.4 Use either the calibration temperature profile for the instrument or, alternatively, the cooling profile given in Test Method D3829 for a –25 °C test temperature, and follow the owner's manual instructions for the instrument to initiate the cooling profile program.

NOTE 7—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 thermometer well at least 30 min prior to executing 9.7. See Note 4. This thermowell location is to be used for calibration and temperature monitoring during the test procedure.

9.6 At the completion of the temperature profile for cell calibration, check that the final test temperature is within 0.1 °C of the desired calibration temperature. Final test temperature is to be verified independently of the instrument's temperature control with a thermometer that has been in the thermometer well for at least the time prescribed in 9.5. See Note 4.

9.7 Beginning with the cell farthest to the left facing the instrument, follow 10.6.3 and record the rotation time for three revolutions.

9.8 Repeat 9.7 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 the calibration constant using Eq 8 and 9.

$$t = (t_1 + t_2)/2 \tag{8}$$

$$C = \eta/t \tag{9}$$

where:

η = viscosity of the standard oil, mPa·s at test temperature,
C = cell constant with a 150 g mass, mPa,
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 (±0.1 °C), use the procedure in X2.1 to assist in determining the cause and correction.

10. Yield Stress and Viscosity Measurement Procedure

10.1 *Viscometric Cell Preparation:*

10.1.1 If the cells are not clean, clean according to 10.7.

10.1.2 Place 10 mL ± 0.2 mL of a test oil sample into a clean cell.

NOTE 8—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 Repeat 10.1.2 until all test samples are in their cells.

NOTE 9—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.

10.1.4 Place each rotor in its cell, and place upper pivot pin in position, including those for any unused cells.

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

10.1.6 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 10—The strings can be pre-wound around the shafts before they are installed in 10.1.4.

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

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

10.1.6.3 Repeat 10.1.6 – 10.1.6.2 until all cells with samples to be measured are prepared.

10.1.7 Place the housing cover over the viscometric cells.

10.1.8 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 30 min prior to completion of the cooling profile (see Note 4). 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 ±0.2 °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 instrument's temperature control using a thermometer that has been in the thermowell for at least 30 min prior to reaching the test temperature. See Note 4. If the final test temperature is more than 0.1 °C from the set point on two consecutive runs, the instrument's temperature control 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:*

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 such that the string hangs past the front of the housing. Make sure that the disk holder clears the edge of the bench during testing.

10.6.2.3 Suspend the disk 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 the disk holder 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 disk holder so it is not supported by the string. Then add an additional 10 g disk to disk holder.

NOTE 11—As additional disks are added to the disk holder, it is necessary to suspend the holder with the additional disks 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 disk holder 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 mass causes rotation of the rotor. At this point, remove the disk holder 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 *Measurement of Apparent Viscosity:*

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 12—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 13—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 both the time and the number of revolutions timed.

10.6.3.7 If no rotation occurs with the application of the 150 g mass, 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 °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.

10.7.3 *Cleaning Cells:*

10.7.3.1 After all of the measurements 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.

10.7.3.2 Remove the upper rotor pivots and the rotors.

10.7.3.3 With vacuum, remove samples, then 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.

10.7.3.4 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:

$$Ys = 3.5\ M \tag{10}$$

where:

Ys = 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 9.10:

$$\eta_a = C \cdot t \cdot 3/r \tag{11}$$

TABLE 1 Precision—Procedure A

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

where:

η_a = apparent viscosity in mPa·s,
C = cell constant obtained in Eq 9,
t = time in seconds for number (r) of complete revolutions of the rotor in 10.6.3.6, 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 14—If the rotation was observed with a mass of 10 g, the minimum mass, 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, then report the apparent viscosity as less than 5000 mPa·s.

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

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

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

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[6]

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 °C, –30 °C, –35 °C, and –40 °C. For direct refrigeration cooled MRV's, the program included 20 samples tested in 6 laboratories at –25 °C, –30 °C, –35 °C, and –40 °C. These samples consisted of multigrade engine oils and base stocks that had a yield stress range of less than 35 Pa to 210 Pa and a apparent viscosity range from 4300 mPa·s 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.

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 in being made.

Procedure B (Methanol cooled MRV Instruments and those used for Procedure A)

14. Calibration and Standardization

14.1 *Temperature Control Calibration Procedure*—Calibrate the MRV temperature control by comparing the

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Reports RR:D02-1404, D02–1612, D02–1613, and D02–1654.

instrument's displayed temperature against a thermometer in the thermowell. The thermometer used shall meet the requirements in 6.4.

14.1.1 Place 10 mL of a typical test fluid and rotor in each cell. If required, place cell caps over each cell then place cover on instrument. Cell caps shall not be used on direct refrigeration instruments (see 6.1.2).

14.1.2 Place the thermometer in the thermowell. See Note 4. This thermowell is to be used for all measurements below 25 °C.

14.1.3 Make these temperature measurements at 80 °C then at least three measurements that are 5 °C apart from –5 °C to the lowest test temperature used, including both end points to establish a calibration curve for this combination of thermometer and the instrument's temperature control. Make at least two temperature measurements at every calibration temperature with at least 10 min between observations.

NOTE 15—All temperatures in this test method refer to the actual temperature and not necessarily the indicated temperature.

14.1.4 Follow the instrument manufacturers instructions for correcting the instrument's measured temperature. Alternatively, establish a correction equation between thermometer and the instruments's measured temperature then adjust each temperature of the cooling program by the offset determined with the correction equation.

14.2 *Viscometer Cell Calibration*—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 Use either the calibration temperature profile for the instrument or, alternatively, the cooling profile given in Test Method D3829 for a –20 °C test temperature and follow the owner's manual instructions for the instrument to initiate the cooling profile program.

14.2.3 Place the thermometer in the thermometer well at least 30 min prior to continuing. See Note 4. This thermowell location is to be used for calibration and temperature monitoring during the test procedure. Allow the oil in the cells to soak at –20 °C ± 0.2 °C for at least 1 h, making small temperature adjustments, if necessary, to maintain the test temperature.

14.2.4 At the completion of the temperature profile for cell calibration, check that the final test temperature is within 0.1 °C of the desired calibration temperature. Final test temperature is to be verified independently of the instrument's temperature control with a thermometer that has been in the thermometer well for at least the time prescribed in 14.2.3. See Note 4.

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:

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

η_o = Viscosity of the standard oil, mPa·s at –20 °C,
C = cell constant with 150 g mass, mPa, and
T = time in seconds for three complete revolutions obtained in 15.4.1.

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 temperature values shown by the instrument's temperature control and thermometer deviate by more than ±0.1 °C, use X2.1 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 mL ± 1.0 mL oil sample in each cell.

NOTE 16—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.

NOTE 17—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.

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.

NOTE 18—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. This is 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 Select the cooling profile for the desired test temperature and follow the instrument instructions to initiate the program. Table Table X1.3 lists the nominal times to reach a particular test temperature.

15.2.7 Place the thermometer in the thermowell at least 30 min prior to completion of the temperature profile. The thermowell used, should be the same one that was used during calibration. (See Note 4.)

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 °C 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 X2.1 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 X2.1 and X2.2.

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 disk holder clears 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 19—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 disk holder to the string and gently suspend it on the string. Proceed to 15.3.4.4.

15.3.4.3 For instruments equipped with locking pins: suspend the disk holder 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 the mass and proceed to 15.4.

15.3.4.5 If no movement is detected, for instruments without locking pins, hold weight assembly and add a disk, then proceed with 15.3.4.4. If equipped with locking pins, lower the locking pin to re-engage cross-arm. Add a disk to the disk holder, 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 disk holder 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 mass is requested, capture cross-arm in locking pin, then add one additional disk, and follow the on-screen instructions. Press the flashing start button, then immediately raise the locking pin. Repeat procedure until no additional mass is requested. Proceed to 15.4.

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

15.3.4.10 If no movement is detected, then carefully support the disk holder. Add a disk 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 disk holder. Repeat procedure until no additional disks are requested. Proceed with 15.4.

NOTE 20—When the 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 it 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 150 g mass on the string. Timing will automatically begin with the first movement. Do not remove mass 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 measurements 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:

$$Ys = 3.5\,M \tag{13}$$

where:

Ys = yield stress, Pa, and
M = applied mass, g, recorded in 15.3.

16.2 The viscosity is given by the following equation when using the cell constant obtained in 14.2.8:

$$\eta_a = C\,t\,3/r \tag{14}$$

where:

η_a = apparent viscosity in mPa·s,
C = cell constant obtained for a 150 g mass in Eq 12,
t = time for number (r) of complete revolutions of the rotor recorded in 15.4, and
r = number of revolutions timed in 15.4.

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, then report the apparent viscosity as less than 5000 mPa·s.

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

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

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

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[7]

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 °C 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:*

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[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Reports RR:D02-1212, D02-1249, D02-1277, and D02-1517.

18.2.1.1 *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:

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

where:
X = mean value in Pa

NOTE 21—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 · (X + 1)
–25	2.976 · (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 °C 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 RR:D02-1517).[7]

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 °C to –40 °C

Segment Time h:min	Segment Temperature[A]				Allowable Temperature Change[B] °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.[C]					
2:00	–20.0	to	–25.0	2.5	±0.2
Hold at this point for –25 °C test temperature.[C]					
2:00	–25.0	to	–30.0	2.5	±0.2
Hold at this point for –30 °C test temperature.[C]					
2:00	–30.0	to	–35.0	2.5	±0.2
Hold at this point for –35 °C test temperature.[C]					
2:00	–35.0	to	–40.0	2.5	±0.2
Hold at this point for –40 °C test temperature.[C]					

[A] 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.

[B] Holding the temperature variation to less than ±0.1 °C improves the precision and reproducibility of your viscosity measurements.

[C] 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 °C and –15 °C

Segment Time h:min	Segment Temperature[A]				Allowable Temperature Change[B] °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.[C]					
2:00	–10.0		–15.0	2.5	±0.2
Hold at this point for – 15 °C test temperature.[C]					

[A] 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.

[B] Holding the temperature variation to less than ±0.1 °C improves the precision and reproducibility of your viscosity measurements.

[C] 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 If the final temperature is in error in either direction by more than 0.2 °C, do the following before starting another analysis:

X2.1.1 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.

X2.1.2 Check temperature sensor of the temperature controller for accuracy, in accordance with 9.1.

X2.1.3 For those instrument that require external liquid circulation:

X2.1.3.1 Check whether the coolant is flowing or whether there is adequate coolant in the reservoir.

X2.1.3.2 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.

X2.1.4 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.

X2.1.5 If manually programmed or using a custom profile, examine the temperature profile program for an error and make the appropriate corrections.

X2.2 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.

X2.3 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.

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SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D4684 – 14) that may impact the use of this standard. (Approved May 1, 2017.)

(1) Revised 3.1.2.

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