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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

[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**

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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:*

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

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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

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

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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.

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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.

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

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

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:*

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