Designation: ~~D4684 – 17a~~ D4684 – 18

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, health, and environmental 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

[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.

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

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

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

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 *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:*~~*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 ~~*apparent viscosity,*~~*cell constant, n*—the ~~determined viscosity obtained by use of this test method.~~ratio of the calibration fluid viscosity to the time required to complete the first three measured revolutions of the rotor.

~~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—*~~

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3.2.3 *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,

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

3.2.3.1 *Discussion*—

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,

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

3.2.4 *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,

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

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

3.2.4.1 *Discussion*—

For the rotary viscometer being described in 6.1, the rotor surface is the area under shear or the shear area. For this test method, end effects are not considered.

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

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

where:

T_r = torque applied to rotor, N·m,
M = applied mass, g,
R_a = 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,

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

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

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

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

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

3.2.8 ~~*viscosity,* *yield stress, 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].~~shear stress required to initiate flow.

3.2.8.1 *Discussion—*

For all Newtonian fluids and many non-Newtonian fluids, the yield stress is zero. An oil can have a yield stress that is a function of its low-temperature cooling rate, soak time, and temperature. Yield stress measurement by this test method determines only whether the test oil has a yield stress of at least 35 Pa; a yield stress below 35 Pa is considered to be insignificant for engine oils.

~~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.

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 a minimum of 25 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

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

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 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).