



Standard Test Method for Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature¹

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

^{ε1} NOTE—Table X1.2 was corrected editorially in April 2000.

1. Scope

1.1 This test method covers the measurement of the yield stress and viscosity of engine oils after cooling at controlled rates over a period exceeding 45 h to a final test temperature between -10 and -40°C. The viscosity measurements are made at a shear stress of 525 Pa over a shear rate of 0.4 to 15 s⁻¹.

1.2 The applicability to petroleum products other than engine oils has not been determined.

1.3 This test method uses the millipascal second (mPa·s) as the unit of viscosity. For information, the equivalent centipoise unit is shown in parentheses.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Terminology

2.1 Definitions:

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

2.1.2 *Newtonian oil or fluid*—an oil or fluid that at a given temperature exhibits a constant viscosity at all shear rates or shear stresses.

2.1.3 *Non-Newtonian oil or fluid*—an oil or fluid that at a given temperature exhibits a viscosity that varies with changing shear stress or shear rate.

2.1.4 *shear rate*—the velocity gradient in fluid flow. For a Newtonian fluid in a concentric cylinder rotary viscometer in which the shear stress is measured at the inner cylinder surface (such as this apparatus, described in 5.1), and ignoring any end effects, the shear rate is given as follows:

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

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

where:

G_r = shear rate at the surface of the rotor in reciprocal seconds, s⁻¹,

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 5.1.1,

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

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

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

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

where:

T_r = torque applied to rotor, N·m,

M = applied mass, g,

R_o = radius of the shaft, mm,

R_t = radius of the string, mm,

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

h = height of the rotor, mm.

For the dimensions given in 5.1.1,

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

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

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

2.2 Definitions of Terms Specific to This Standard:

2.2.1 *calibration oils*—those oils that establish the instrument's reference framework of apparent viscosity versus speed, from which the apparent viscosities of test oils are determined. Calibration oils, which are essentially Newtonian fluids, are available commercially, and have an approximate

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viscosity of 30 Pa·s (30 000 cP) at -20°C .²

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

2.2.3 *yield stress*—the shear stress required to initiate flow. For all Newtonian fluids and some 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. 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.

3. Summary of Test Method

3.1 An engine oil sample is held at 80°C and then cooled at a programmed cooling rate to a final test temperature. A low torque is applied to the rotor shaft to measure the yield stress. A higher torque is then applied to determine the apparent viscosity of the sample.

4. Significance and Use

4.1 When an engine oil is cooled, the rate and duration of cooling can affect its yield stress and viscosity. In this laboratory test an 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 due to the lack of oil pumpability.³ These documented field failing oils have 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 excessive yield stress or viscosity of the engine oil, or both.

4.2 Cooling Profiles:

4.2.1 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)⁴ No significance has been determined for the -35 and -40°C temperature profiles because of the absence of appropriate reference oils.

4.2.2 For oils to be tested at -15 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 and -10°C .

² The sole source of supply of the apparatus known to the committee at this time is Cannon Instrument Co., P.O. Box 16, State College, PA 16804. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee¹, which you may attend.

³ Pumpability Reference Oils (PRO) 21 through 29.

⁴ The boldface numbers in parentheses refer to the references at the end of this standard.

5. Apparatus

5.1 *Mini-Rotary Viscometer*², an apparatus that consists of one or more viscometric cells in a temperature-controlled aluminum block. Each cell contains a calibrated rotor-stator set. Rotation of the rotor is achieved by an applied load acting through a string wound around the rotor shaft.

5.1.1 The mini-rotary viscometric cell has the following typical dimensions:

	millimetres
Diameter of rotor	17.0
Length of rotor	20.0
Inside diameter of cell	19.0
Radius of shaft	3.18
Radius of string	0.05

5.2 *Temperature Control System* that will operate the heater in the mini-rotary viscometer block and regulate the coolant flow to the block. This controller must regulate the temperature to within the temperature limits described in Table X1.1 or Table X1.2.

5.2.1 *Temperature Controller* is the most critical part of this procedure. The temperature control system can be a single-loop programmable controller to control the block temperature. A process controller that has a proportional band with integral reset and derivative rate control, sometimes referred to as a PID controller, is suitable for controlling the temperature. This programmable controller has one control loop and one temperature sensor that provides the appropriate information to the controller to hold the temperature at the programmed set temperature. It has an internal clock that controls the execution of the program. The controller must be connected so that only heat is supplied to the block during the first 2 h and 20 min of the temperature profile described in Table X1.1 or Table X1.2. The temperature control during the remaining portion of the temperature profile must be obtained by controlling the coolant flow. This control system must have a minimum temperature sensitivity of 0.1°C and be able to change the temperature at a prescribed rate. When the control system's proportional band, integral (reset), and derivative (rate) parameters are optimized the temperature excursions above and below the profile must be no greater than 0.2°C at a temperature below -5°C . The temperature sensor can be a platinum resistance thermal detector, a thermistor, or a thermocouple. A platinum resistance thermal detector or thermistor sensor is preferred. A $\frac{1}{8}$ -in. (3.2-mm) diameter temperature probe can be installed directly into the $\frac{1}{8}$ -in. diameter well located at the back of the block between cells Nos. 4 and 6. Alternatively, the temperature sensor can be inserted into one of the thermometer wells.

NOTE 1—The sensor is placed in the same unit that is being controlled. The sensor should be placed in the block if the supply of coolant is being controlled. Alternatively, the sensor would be placed in the bath if the bath temperature was being controlled. Do not try to control the block temperature by sensing the block temperature and controlling the refrigeration system.

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

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

5.3 *Thermometers*², for measuring the temperature of the block. Two are required, one graduated from at least -70 to

90°C in 1°C subdivisions, the other with a scale from at least -36 to +5°C or -45 to +5°C, in 0.2°C subdivisions. Other thermometric devices of equal accuracy and resolution may be used to calibrate the temperature sensor.

5.4 *Refrigeration Device*, consisting of a means of maintaining a coolant (such as methanol) to at least 10°C below the lowest test temperature.

5.5 *Circulating System*² that will circulate the liquid coolant to the block as needed. Methanol is a suitable coolant. One should observe toxicity and flammability precautions that apply to the use of methanol. The circulating system must be capable of maintaining test temperature during the test. If methanol is leaking from the system, discontinue the test and repair the leak. (**Warning**—Methanol is flammable.)

5.6 *Chart Recorder*—To verify that the correct cooling curve is being followed, it is recommended that a chart recorder be used to monitor the block temperature.

6. Reagents and Materials

6.1 *Newtonian Oil*², a low cloud-point of approximately 30 Pa·s (30 000 cP) viscosity at -20°C for calibration of the viscometric cells.

6.2 *Methanol*—Commercial or technical grade of dry methanol is suitable for the cooling bath.

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

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

7. Sampling

7.1 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.

8. Calibration and Standardization

8.1 Calibrate the temperature sensor while attached to the temperature controller. The sensed temperature shall be verified using either the -36 to +5°C reference thermometer or the -45 to +5°C reference thermometer noted in 5.3 at a minimum of three temperatures. Make these temperature measurements at least 5°C apart to establish a calibration curve for this combination of temperature sensor and controller. Recheck the temperature sensor periodically using the ice point of water as the reference point. If the corrected sensor temperature deviates from the ice point by more than 0.1°C, repeat the calibration in accordance with this paragraph.

NOTE 3—All temperatures in this test method refer to the actual temperature and *not* necessarily the indicated temperature.

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

8.2.1 Use steps 9.2-9.2.5.

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

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

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

8.2.5 Proceed to 9.3.1-9.3.3.

8.2.6 Perform step 9.4.1.

8.2.7 Repeat 8.2.5 and 8.2.6 for each of the remaining cells, taking the cells in order from left to right.

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

$$C = \eta_o/t \quad (8)$$

where:

η_o = viscosity of the standard oil, cP (mPa·s) at -20°C,

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

t = time in seconds for three complete revolutions.

8.3 It is essential that the ice point of the calibrated thermometer and the temperature sensor are initially measured and periodically measured thereafter. If any change in calibration is noted in the ice point the temperature controller/sensor combination, recalibrate in accordance with 8.1.

8.4 Check the cooling rate periodically to ensure that the programmed cooling follows the profile described in Table X1.1 or Table X1.2. This should be done at least monthly by recording the block temperature as measured by the temperature controller and a thermometer placed in the block at least three times during normal working hours. Continual monitoring of the temperature profile is strongly recommended during a cooling program below 0°C since deviations from the prescribed temperature profile, Table X1.1 or Table X1.2, can cause erroneous test results. This can be achieved through the use of a strip chart recorder or a data acquisition system that periodically (every 1 to 2 min) records the time and block temperature. The allowable temperature deviations are shown in Table X1.1 or Table X1.2, and are also outlined in 5.2.

9. Procedure

9.1 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 8.1. Table X1.3 lists the nominal times to reach a particular test temperature

9.2 *Test Sample and Viscometric Cell Preparation:*

9.2.1 Remove the nine rotors from the viscometric cells and ensure that both the cells and rotors are clean. See 9.6 for the cleaning procedure.

9.2.2 Place a 10 ± 1.0 mL oil sample in each cell.

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

9.2.4 Place the loop of the 700-mm long string over the crossarm 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 a (red or black) marked end of the crossarm at the top of the rotor shaft is pointing directly forward.