Designation: D 6896 – 03ε1

An American National Standard

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

This standard is issued under the fixed designation D 6896; 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.

ε[1] NOTE—Table X1.3 was corrected editorially in August 2004.

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 of 43 or 45 h to a final test temperature of -20 or -25°C. The viscosity measurements are made at a shear stress of 525 Pa over a shear rate of 0.4 to 15 s^{-1}. This test method is suitable for measurement of viscosities ranging from 4000 mPa·s to >400 000 mPa·s, and is suitable for yield stress measurements of 7 Pa to >350 Pa.

1.2 This test method is applicable for used diesel oils. The applicability and precision to other used or unused engine oils or 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. Referenced Documents

2.1 *ASTM Standards:*

D 3829 Test Method for Predicting the Borderline Pumping Temperature of Engine Oil[2]

D 4684 Test Method for Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature[2]

D 5133 Test Method for Low Temperature, Low Shear Rate, Viscosity/Temperature Dependence of Lubricating Oils Using a Temperature-Scanning Technique[2]

3. Terminology

3.1 *Definitions:*

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

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

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

3.1.4 *shear rate*—the velocity gradient in fluid flow. For a Newtonian fluid in a concentric cylinder rotary viscometer in which the shear stress is measured at the inner cylinder surface (such as the apparatus described in 6.1), and ignoring any end effects, the shear rate is given as follows:

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

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

where:

G_r = shear rate at the surface of the rotor in reciprocal seconds, s^{-1},
Ω = angular velocity, rad/s,
R_s = stator radius, mm,
R_r = rotor radius, mm, and
t = time for one revolution of the rotor, s.

For the specific apparatus described in 6.1,

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

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

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

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

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,

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

Current edition approved March 10, 2003. Published May 2003.

[2] *Annual Book of ASTM Standards*, Vol 05.02.

S_r = shear stress at the rotor surface, Pa, and
h = height of the rotor, mm.

For the dimensions given in 6.1.1,

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

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

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *calibration oils*—those oils that establish the instrument's reference framework of apparent viscosity versus speed, from which the apparent viscosities of test oils are determined. Calibration oils, which are essentially Newtonian fluids, are available commercially and have an approximate viscosity of 30 Pa·s (30 000 cP) at -20°C.

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

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

3.2.4 *yield stress*—the shear stress required to initiate flow.

3.2.4.1 *Discussion*—For all Newtonian fluids and some 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.

4. Summary of Test Method

4.1 A used engine oil sample is heated at 80°C and then vigorously agitated. The sample is 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.

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, used 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. As in other low temperature rheological tests such as Test Methods D 3829, D 4684, and D 5133, a preheating condition is required to ensure that all residual waxes are solubilized in the oil prior to the cooldown (that is, remove thermal memory). However, it is also known that highly sooted used diesel engine oils can experience a soot agglomerization phenomenon when heated under quiescent conditions. The current method uses a separate preheat and agitation step to break up any soot agglomerization that may have occurred prior to cooldown. The viscosity of highly sooted diesel engine oils as measured in this test method have been correlated to pressurization times in a motored engine test **(1)**.[3]

5.2 *Cooling Profiles*:

5.2.1 For oils to be tested at -20°C and -25°C, 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 **(2-7)**.

6. Apparatus

6.1 *Mini-Rotary Viscometer*[4], 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.

6.1.1 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	0.10

6.2 *Weights*:

6.2.1 *Yield Stress*, weight set consists of ten 10 g units with a tolerance of 1 % for each unit.

6.2.2 *Viscosity*, 150 g weight with a 1 % tolerance.

6.3 *Temperature Control System*, that will regulate the mini-rotary viscometer block temperature in accordance with the temperature limits described in Table X1.1.

6.3.1 *Temperature Controller* is the most critical part of this procedure. A description of the requirements that the controller shall meet are included in Appendix X2.

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

6.4 *Thermometers*, for measuring the temperature of the block. Two ranges are required, one graduated from at least +70 to 90°C in 1°C subdivisions, the other with a range 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.

6.5 *Refrigeration Device*, consisting of a means of removing heat from the instrument such that the cell temperature is controlled in accordance with the program described in Table X1.1.

6.6 *Circulating System*, that will circulate the liquid coolant to the instrument as needed. Methanol is a suitable coolant if the circulating coolant is below -10°C. One should observe toxicity and flammability precautions that apply to the use of methanol. The circulating system shall be capable of maintaining test temperature during the test. If methanol is leaking from

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

[4] 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 International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee [1], which you may attend.

the system, discontinue the test and repair the leak. (**Warning**—Methanol is flammable.)

6.7 *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.

6.8 *Sample Pre-treatment Oven*, an oven capable of maintaining a temperature of 80 ± 1°C for a minimum of 2 h.

7. Reagents and Materials

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

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

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 granular material and water is necessary to obtain valid viscosity measurements. If the sample in its container is received below the dew-point temperature of the room, allow the sample to warm to room temperature before opening the container.

9. Calibration and Standardization

9.1 Calibrate the temperature sensor in place while attached to the temperature controller. The sensed temperature shall be verified using a reference thermometer specified in 6.4 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. For instruments using an independent temperature controller, see X2.1 for calibration guidance.

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

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

9.2.1 Use steps 10.2.3-10.6.

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

9.2.3 Allow the oil in the cells to soak at -20 ± 0.2°C for at least 1 h, making small temperature control adjustments, if necessary, to maintain the test temperature.

9.2.4 At the end of the soak period, record the temperature reading of the measuring device in the left thermowell (test temperature) and remove the cover of the viscometer cell.

9.2.5 Perform step 10.4.1.

9.2.6 Repeat 9.2.5 for each of the remaining cells, taking the cells in order from left to right.

9.2.7 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 for three complete revolutions, s.

9.2.8 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 instrument.

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

9.4 *Oven*—Check the calibration of the temperature sensing device by appropriate methods. The temperature should be constant at 80 ± 1°C.

10. Procedure

10.1 Select the cooling profile for the desired test temperature. Table X1.2 lists the nominal times to reach a particular test temperature.

10.1.1 Choose the preprogrammed temperature profile. If the profile is not available, enter it using the custom profile part of the software program. The instrument manual provides instructions on adding custom profiles. The entries for a custom program will be found in Table X1.3.

10.1.2 If the instrument temperature is controlled by an external controller, it will need to be programmed to follow the cooling program in Table X1.1 with adjustment for the temperature difference found in 9.1, if any.

10.2 *Test Sample and Viscometric Cell Preparation*:

10.2.1 Using suitable closed container, preheat the samples in an oven to 80 ± 1°C for 2.25 h. At the end of this time, remove the samples from the oven and allow to cool for 15 min at room temperature.

10.2.2 Agitate each sample using vigorous mechanical or manual shaking for 60 s. Allow the samples to stand for a minimum of 10 min to allow for settling.

10.2.3 Remove the nine rotors from the viscometric cells and ensure that both the cells and rotors are clean. See 10.6 for the cleaning procedure.

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

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

10.2.6 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 an end of the crossarm at the top of the rotor shaft is pointing directly forward. If available, secure crossarm with locking pin. If the rotations are manually timed, it is helpful to color one end of the crossarm.

10.2.6.1 The string may be prewound around the shaft before installation of the rotor in 10.2.5.

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

10.2.8 Start the programmed temperature profile.

10.2.9 The cooling cycle starts to cool the samples in accordance with the programmed cooling sequence as programmed in 10.1.

10.2.10 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 10.3).

10.2.10.1 If the final temperature of the block is 0.2 to 0.5°C warmer than the desired temperature, proceed as follows. Set the temperature controller to bring the block temperature to the correct test temperature and then hold at the correct test temperature for 30 min before proceeding. This entire temperature correction should not take longer than 1 h. The data obtained in this way are considered valid test results, otherwise the test is invalid.

10.2.10.2 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.

10.2.11 If the final temperature as noted in 10.2.10 is in error in either direction by more than 0.2°C, see X2.2 before starting another test.

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

10.3 *Measurement of the Yield Stress*:

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

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

10.3.3 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.)

10.3.4 For CMRV-3 and earlier models, follow the instructions in 10.3.5. For CMRV-4 or later models, if using the automatic timing devices, follow the instructions in 10.3.6. If manual timing measurements are used, follow the instructions in 10.3.5.

10.3.5 Visually observe the rotor for movement of the crossarm. (Do not measure yield stress by way of the electronic optics.)

10.3.5.1 For instruments not equipped with locking pins, carefully, so as not to disturb the gel structure, attach a 10-g mass to the string and gently suspend the weight on the string. Proceed to 10.3.5.3.

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

10.3.5.3 If the end of the crossarm does not move at least 3 mm in 15 s (approximately twice the diameter of the crossarm or 13° of rotation) then record that the sample has yield stress. Proceed to 10.3.5.4. If movement is detected, record weight and proceed to 10.4.

10.3.5.4 If no movement is detected, for instruments without locking pins, hold weight assembly and add 10 g, then proceed with 10.3.5.3. If equipped with locking pins, lower the locking pin to re-engage crossarm. Add 10 g to the weight assembly, raise the locking pin and proceed with 10.3.5.3.

NOTE 2—The total amount of weight available for measurement of yield stress is normally 100 g; if no movement is detected with this weight, yield stress would be recorded as >350 Pa.

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

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

10.3.6.2 If additional weight is requested, capture crossarm in locking pin, add one additional 10 g weight, and follow the on-screen instructions. Press the flashing start button, and immediately raise the locking pin. Repeat procedure until no additional weight is requested. Proceed to 10.4.

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

10.3.6.4 If no movement is detected, carefully weight the cage. Add next 10 g weight increment as indicated on computer screen without pulling on string and follow on-screen instructions. Press the flashing start button and immediately release weight cage. Repeat procedure until no additional weight is requested. Proceed with 10.4.

NOTE 3—When the 10-g load is first applied, some oils may show momentary movement of the crossarm. If there is no further movement of the crossarm for 15 s, disregard the initial movement.

10.4 *Measurement of Apparent Viscosity*:

10.4.1 For CMRV-3 and earlier models follow the instructions in 10.4.2. For CMRV-4 or later models, if using the automatic timing devices, follow the instructions in 10.4.3. If manual timing measurements are used, follow the instructions in 10.4.2.

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

10.4.2.1 If the first half-revolution requires less than 10 s, measure and record the time for the first three revolutions, and proceed to 10.5.

10.4.2.2 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; then proceed to 10.5.

10.4.2.3 If the first revolution has not been completed in 60 s, end the measurement. Record the time as greater than 60 s