Designation: ~~D6896 – 03 (Reapproved 2007)~~ D6896 – 12

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 D6896; 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.

1. ~~Scope~~ 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 precision is stated for test temperatures -20 and -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.~~The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3.1 *Exception*—This test method uses the SI based unit of milliPascal second (mPa·s) for viscosity which is equivalent to centiPoise (cP).

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:*[2]

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

D4684 Test Method for Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature

D5133 Test Method for Low Temperature, Low Shear Rate, Viscosity/Temperature Dependence of Lubricating Oils Using a Temperature-Scanning Technique

E1137 Specification for Industrial Platinum Resistance Thermometers

3. Terminology

3.1 *Definitions:*

3.1.1 *apparent ~~viscosity—~~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.

[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 ~~Nov. 1, 2007~~Nov. 1, 2012. Published ~~January 2008~~April 2013. Originally approved in 2003. Last previous edition approved in ~~2003~~2007 as ~~D6896~~D6896–03(2007).~~–03~~ ε1. DOI: ~~10.1520/D6896-03R07.~~10.1520/D6896-12.

[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.

3.1.2.1 *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 or display

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

of the temperature, or both. The temperature sensing probe is in contact with the material whose temperature is being measured. This device is sometimes referred to as a *digital thermometer*.

3.1.3 *Newtonian oil or ~~fluid~~ 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~~ 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~~ 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 the apparatus described in 6.1), and ignoring any end effects, the shear rate is given as follows:

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

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

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,

$$G_r = 63/t \tag{3}$$

3.1.6 *shear ~~stress~~ 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.

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

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

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 6.1.1,

$$T_r = 31.7\,M \times 10^{-6} \tag{6}$$

$$S_r = 3.5\,M \tag{7}$$

3.1.7 *~~viscosity~~ 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. A centipoise (cP) is one millipascal second mPa·s.~~(Pa·s).

3.2 *Definitions of Terms Specific to This Standard:*

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

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

3.2.4 *yield ~~stress~~ stress, n*—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 D3829, D4684, and D5133, 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,*~~ *Temperature Measuring Device*~~—for measuring the temperature of the block. Two~~Use either a calibrated DCT described in 6.4.1 ~~ranges are required, one graduated~~or liquid-in-glass thermometers described in 6.4.2~~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.~~. A DCT or a calibrated low temperature liquid-in-glass thermometer shall be used as the thermometer for temperature measurement 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 A DCT shall meet the following:

(1) A range from -45 to 100°C with a display resolution to at least 0.01°C.

(2) The only acceptable sensors are a resistance temperature device (RTD), such as a platinum resistance thermometer (PRT) or a thermistor that are either glass or metal sheathed.

[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.

(3) For metal sheathed probe, use a 3 mm diameter probe with a sensing element that is less than 30 mm in length; a metal sheathed probe requires a thermowell sleeve measuring 6 mm outside diameter by 58 mm long with a hole in the center to accommodate the probe. For a glass sheathed probe, use a probe 6 mm in diameter containing a sensing element less than 12 mm in length.

(4) A combined (display and probe) minimum accuracy of ±50 mK (0.05°C).

(5) A response time of less than or equal to 25 s as defined in Specification E1137.

(6) A drift of less than 50 mK (0.05°C) per year.

(7) Error of less than 50 mK (0.05°C) over the range of intended use.

(8) The DCT shall have a report of temperature calibration from a calibration laboratory with demonstrated competency in temperature calibration which is traceable to a national calibration laboratory or metrology standards body.

(9) The DCT shall be calibrated over a range from -40 to 85°C with at least 4 data points evenly distributed over the range of -40 to -1°C. The test report shall include the calibration data.

6.4.1.1 The DCT calibration 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 (6 and 7).

NOTE 2—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 Two liquid in glass thermometers are required. One 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. The low temperature thermometer shall have a report of calibration showing the temperature deviation at each calibrated test temperature. The second shall be a 76 mm partial immersion thermometer graduated from at least +20 to 90°C in 1°C subdivisions.

6.4.2.1 *Calibration Check*—Verify the low temperature thermometer at least annually against a reference thermometer in a constant temperature bath or in an ice bath. The thermometer is to be insert 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 *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 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 *Temperature Control Calibration Procedure*—Calibrate the ~~temperature sensor in place while attached to the temperature controller. The sensed temperature shall be verified using a reference thermometer specified~~ 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 ~~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.~~

9.1.1 Place 10 mL of a typical test fluid and rotor in each cell. Cell caps maybe used if available for the instrument. Place the cover on instrument.

9.1.2 Place the thermometer in the thermowell. See Note 3. This thermowell should be used for all measurements.

NOTE 3—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 at least four temperature measurements that are at least 5°C apart between –5°C and the lowest test temperature used to establish a calibration curve between the 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 4—All temperatures in this test method refer to the actual temperature as measured in the left thermowell 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 the thermometer and the instrument's measured temperature then adjust each temperature of the cooling program by the offset determined with the correction equation.

~~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 *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.

9.2.1 Use steps ~~10.2.3-10.6~~10.2.3~~,~~–10.2.7 to prepare the cells for calibration using the reference oil as the sample.

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.~~h.

9.2.4 Place the thermometer in the thermometer well at least 30 min prior to executing 9.2.6. See Note 3. This thermowell location is to be used for calibration and temperature monitoring during the test procedure.

9.2.5 At the ~~end~~completion of the temperature profile and soak period, ~~record~~ check that the ~~temperature reading of the measuring device in the left thermowell (test temperature) and remove the cover of the viscometer cell.~~test temperature is within ±0.1°C of the desired calibration temperature with a thermometer. If the temperature meets the criteria remove the cell cover and proceed.

9.2.6 Perform step 10.4.1.

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

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

$$C = \eta_o / t \tag{8}$$

where:

~~η_o = viscosity of the standard oil, cP (mPa·s) at -20°C,~~
η_o = viscosity of the standard oil, mPa·s at -20°C,
C = cell constant with 150 g mass, Pa, and
T = time for three complete revolutions, s.

9.2.9 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 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. Cell caps maybe used if available for the instrument. 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 Place the thermometer in the thermowell at least 30 min prior to completion of the cooling profile. The thermowell used should be the same one that was used during calibration (see Note 3).

10.2.11 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.~~ 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 3. 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). 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.

10.2.11.1 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.

10.2.11.2 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.

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

10.2.13 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.