



Designation: D6896 – 20a

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

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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 h or 45 h to a final test temperature of $-20\text{ }^{\circ}\text{C}$ or $-25\text{ }^{\circ}\text{C}$. The precision is stated for test temperatures $-20\text{ }^{\circ}\text{C}$ and $-25\text{ }^{\circ}\text{C}$. 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} . 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 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *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.*

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. 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

D8278 Specification for Digital Contact Thermometers for Test Methods Measuring Flow Properties of Fuels and Lubricants

E563 Practice for Preparation and Use of an Ice-Point Bath as a Reference Temperature

2.2 ISO Standards: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.

3.1.2.1 *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

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 American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

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

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.

3.1.2.2 *Discussion*—The devices are often referred to as a “digital thermometers,” however the term includes devices that sense temperature by means other than being in physical contact with the media.

3.1.2.3 *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 which is sometimes called the coefficient of dynamic viscosity and is a measure of the resistance to flow of the liquid.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *calibration 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.

3.2.2 *shear rate, n*—the velocity gradient in fluid flow.

3.2.2.1 *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 the 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 for one revolution of the rotor, s.

For the specific apparatus described in 6.1,

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

3.2.3 *shear stress, n*—the motivating force per unit area for fluid flow.

3.2.3.1 *Discussion*—For the rotary viscometer 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.

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

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

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

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.

3.2.6.1 *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 agglomeration phenomenon when heated under quiescent conditions. The current method uses a separate preheat and agitation step to break up any soot agglomeration 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).⁴

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

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

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*⁵, an apparatus that consists of one or more viscometric cells in a temperature-controlled aluminum block. 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 load acting through a string wound around the rotor shaft.

6.1.1 The mini-rotary viscometric cell has the following typical 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	0.10 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*, that will regulate the mini-rotary viscometer block temperature in accordance with the temperature limits described in [Table X1.1](#).

6.3.1 *Temperature Profile*—The temperature profile is fully described in [Table X1.1](#).

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 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 *Digital Contact Thermometer*—Use D02-DCT14 listed in Specification [D8278](#). As an alternative to the metal sheathed probe noted in Specification [D8278](#), a glass sheathed DCT probe with a 6 mm O.D. is acceptable provided it meets the other requirements shown for D02-DCT14 in Specification [D8278](#). A DCT display resolution of 0.01 C is preferable. If thermowell ID is larger than the probe OD, then a metallic sleeve must be used to fill the gap between the probe OD and thermowell ID with a length of 58 mm.

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 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 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 +20 °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 in 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.

⁵ 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.

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.

6.7 *Sample Pre-treatment Oven*, an oven capable of maintaining a temperature of $80\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ for a minimum of 2 h.

7. Reagents and Materials

7.1 *Low Cloud-point Newtonian Oil*, a calibration oil of approximately 30 Pa·s viscosity at $-20\text{ }^{\circ}\text{C}$ 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 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 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. Cell caps may be 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 is to be used for all temperature measurements below $25\text{ }^{\circ}\text{C}$.

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 $5\text{ }^{\circ}\text{C}$ apart between $-5\text{ }^{\circ}\text{C}$ 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 manufacturer's 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.

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 $-20\text{ }^{\circ}\text{C}$.

9.2.1 Following the steps in 10.2 to prepare the cells for calibration using the calibration oil as the sample.

9.2.2 Use either the calibration temperature profile for the instrument which cools to $-20\text{ }^{\circ}\text{C}$ then holds for 2 h or, alternatively, the cooling profile given in Test Method **D3829** for a $-20\text{ }^{\circ}\text{C}$ test temperature and follow the owner's manual instructions for the instrument to initiate the cooling profile program.

9.2.3 Place the thermometer in the thermometer well at least 30 min prior to executing 9.2.5. See **Note 3**. This thermowell location is to be used for calibration and temperature monitoring during the test procedure.

9.2.4 At the completion of the temperature profile and soak period, check that the test temperature is within $\pm 0.1\text{ }^{\circ}\text{C}$ of the desired calibration temperature with a thermometer. If the temperature meets the criteria remove the cell cover and proceed.

9.2.5 Beginning with the cell farthest to the left, perform step 10.8.

9.2.6 Repeat 9.2.5 for each of the remaining cells in numerical order.

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, mPa·s at $-20\text{ }^{\circ}\text{C}$,
 C = cell constant with 150 g mass, mPa, 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 **X2.1** 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 $80\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$.

10. Procedure

10.1 *Test Sample Preparation*:

10.1.1 Using suitable closed container, preheat the samples in an oven to $80\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ 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.

10.1.2 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 *Viscometric Cell Preparation:*

10.2.1 If the cells are not clean, clean according to 10.10.

10.2.2 Place 10 mL \pm 1 mL of a test oil sample into a clean cell.

10.2.3 Repeat 10.2.2 until all test samples are in their cells.

NOTE 5—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.

10.2.4 Place each rotor in its cell then place upper pivot pin in position, including those for any unused cells.

NOTE 6—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.

10.2.5 *Optional*—Install a cell cap on all cells, including any unused cells.

10.2.6 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 7—The strings can be pre-wound around the shafts before they are installed in 10.2.4.

10.2.6.1 Engage the locking pin to prevent the rotor from turning.

10.2.6.2 Lay the remaining string over the top of the bearing plate letting it hang over the back of the plate.

10.2.6.3 Repeat 10.2.6 – 10.2.6.2 until all cells with samples to be measured are prepared.

10.2.7 Place the housing cover over the viscometric cells.

10.2.8 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.

10.3 Select the cooling profile for the desired test temperature and follow the instrument instructions to initiate the program. Table X1.2 lists the nominal times to reach a particular test temperature.

10.3.1 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.

10.4 Place the thermometer in the thermowell at least 30 min prior to completion of the cooling profile (see Note 3). The same thermowell location is to be used for all measurements and must be the same one as was used in the calibration.

10.5 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 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.

10.6 If the temperature profile is within tolerance, proceed with measurements. If not, then abort the test and recalibrate temperature controller as in 9.1. See X2.1 – X2.3 for assistance in determining cause of temperature error.

10.7 *Measurement of the Yield Stress:*

10.7.1 Immediately prior to starting measurements, take the cell housing cover off the instrument.

10.7.2 *Yield Stress Determination*—Starting with the cell farthest to the left while facing the instrument, use the following procedure for each cell in turn, bypassing the unused cells.

10.7.3 Align the pulley wheel with the rotor shaft for the cell to be tested.

10.7.4 Hang the string over the timing wheel such that the string hangs past the front of the housing. Make sure that the disk holder clears the edge of the bench during testing. (Do not allow the rotor shaft to turn.)

10.7.5 For CMRV-3 and earlier models, follow the instructions in 10.7.6. For CMRV-4 or later models, if using the automatic timing devices, follow the instructions in 10.7.7. If manual timing measurements are used, follow the instructions in 10.7.6.

10.7.6 Visually observe the rotor for movement of the cross-arm. (Do not measure yield stress by way of the electronic optics.)

10.7.6.1 For instruments not equipped with locking pins, carefully, so as not to disturb the gel structure, attach a disk holder to the string and gently suspend the weight on the string. Proceed to 10.7.6.3.

10.7.6.2 For instruments equipped with locking pins, suspend the disk holder on the string, then raise the locking pin.

10.7.6.3 If the end of the cross-arm does not move at least 3 mm in 15 s (approximately twice the diameter of the cross-arm or 13° of rotation) then record that the sample has yield stress. Proceed to 10.7.6.4. If movement is detected, record weight and proceed to 10.8.

10.7.6.4 If no movement is detected, for instruments without locking pins, hold disk holder assembly and add a disk to it, then proceed with 10.7.6.3. If equipped with locking pins, lower the locking pin to re-engage cross-arm. Add a disk to the disk holder, raise the locking pin, and proceed with 10.7.6.3.

NOTE 8—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.

10.7.7 The operator shall follow the on-screen instructions adding additional disks to disk holder.

10.7.7.1 For instruments with locking pins, suspend disk holder on string, press the flashing start button then immediately raise the locking pin and follow on-screen instructions.