Designation: D6896 – 17

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*

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 °C or –25 °C. The precision is stated for test temperatures –20 °C and –25 °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 and health 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.*

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

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

E644 Test Methods for Testing Industrial Resistance Thermometers

E1137 Specification for Industrial Platinum Resistance Thermometers

E2877 Guide for Digital Contact Thermometers

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,

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[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**

computes the temperature from the measured quantity, and provides a digital output. This digital output goes to a digital display and/or recording device that may be internal or external to the device. These devices are sometimes referred to as "digital thermometers."

3.1.2.2 *Discussion*—PET is an acronym for portable electronic thermometers, a subset of digital contact thermometers (DCT).

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

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

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

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

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

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,

$$\dot{\gamma} = 63/t \tag{3}$$

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

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

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

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

For the dimensions given in 6.1.1,

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

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

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

3.2 *Definitions 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 *test 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, n*—an oil which has been used in an operating engine.

3.2.4 *yield 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)**.[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 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)**.

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

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.

[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.

6.4.1 Digital contact thermometer requirements:

Criteria	Minimum Requirements
DCT	E2877 Class B
Temperature range	–45 °C to 100 °C
Display resolution	0.1 °C minimum, preferably 0.01 °C
Sensor type	RTD, such as a PRT or thermistor
Sensor, metal sheathed	3 mm O.D. with an sensing element less than 30 mm in length to be used with a thermowell sleeve, 6 mm O.D. × 58 mm long with a ~3 mm hole in center.
Sensor, glass sheathed	6 mm O.D. with a sensing element less than 12 mm in length
Display accuracy	±50 mK (±0.05 °C) for combined probe and sensor
Response time	less than or equal to 25 s as defined in Specification E1137
Drift	less than 50 mK (0.05 °C) per year
Calibration Error	less than 50 mK (0.05 °C) over the range of intended use.
Calibration Range	–40 °C to 85 °C
Calibration Data	4 data points evenly distributed over the range of –40 °C to –1 °C and included in calibration report.
Calibration Report	From a calibration laboratory with demonstrated competency in temperature calibration which is traceable to a national calibration laboratory or metrology standards body

NOTE 2—With respect to DCT probe immersion depth, a procedure to determine minimum depth can be found in Guide E2877, Section 5.3, or Test Methods E644, Section 7.

6.4.1.1 The DCT calibration drift shall be checked at least annually by either measuring the ice point or against a reference thermometer in a constant temperature bath at the prescribed immersion depth to ensure compliance with 6.4.1. With respect to an ice bath, Practice E563 provides guidance on the preparation and use of an ice bath. However, for this use, variance from the specific steps, such as water source, is permitted provided preparation is consistent. The basis for the variance is due to the reference being used to track change in calibration not verification.

NOTE 3—When a DCT's calibration drifts in one direction over several calibration checks, that is, ice point, it may be an indication of deterioration of the DCT.

6.4.2 For liquid-in-glass thermometers, LiG, two are required. One LiG shall be a calibrated 76 mm partial immersion thermometer with a scale from +5 °C to 1 degree less than the lowest test temperature in 0.2 °C subdivisions. This low temperature LiG thermometer shall have a report of calibration showing the temperature deviation at each calibrated test temperature. The second LiG thermometer shall be a 76 mm partial immersion thermometer graduated from at least +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 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 *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 °C ± 1 °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 °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 maybe used if available for the instrument. Place the cover on instrument.

9.1.2 Place the thermometer in the thermowell. See Note 4. This thermowell is to be used for all temperature measurements below 25 °C.

NOTE 4—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 5—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.

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 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 °C then holds for 2 h or, alternatively, the cooling profile given in Test Method D3829 for a –20 °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 4. 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 ±0.1 °C of the desired calibration temperature with a thermometer. If the temperature meets the criteria remove the cell cover and proceed.

9.2.5 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:

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

where:

η_o = viscosity of the standard oil, mPa·s at –20 °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 °C ± 1 °C.

10. Procedure

10.1 *Test Sample Preparation:*

10.1.1 Using suitable closed container, preheat the samples in an oven to 80 °C ± 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.

10.1.2 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 ±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 6—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 7—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 8—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 4). 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 4. 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 9—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.

10.7.7.2 If an additional disk is requested, capture cross-arm in locking pin, add one additional disk, and follow the on-screen instructions. Press the flashing start button, and immediately raise the locking pin. Repeat procedure until no additional disks are requested. Proceed to 10.8.

10.7.7.3 For instruments without locking pins, carefully suspend and hold the disk holder on the string without jerking rotor and follow on-screen instructions. Press the flashing start button, and immediately release the disk holder.

10.7.7.4 If no movement is detected, carefully lift the disk holder to relieve load and add a disk as indicated on computer screen without pulling on string and follow on-screen instructions. Press the flashing start button and immediately release disk holder. Repeat procedure until no additional disks are requested. Proceed with 10.8.

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

10.8 *Measurement of Apparent Viscosity:*

10.8.1 For CMRV-3 and earlier models follow the instructions in 10.8.2. For CMRV-4 or later models, if using the automatic timing devices, follow the instructions in 10.8.3. If manual timing measurements are used, follow the instructions in 10.8.2.

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

10.8.2.1 If the first half-revolution requires less than 10 s, measure and record the time for the first three revolutions, and proceed to 10.9.

10.8.2.2 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 10.9.

10.8.2.3 If the first revolution has not been completed in 60 s, end the measurement. Record the time as greater than 60 s for one revolution, then proceed to 10.9, reporting that the viscosity is greater than the value calculated in 11.2.

10.8.2.4 If the time for the first three revolutions is less than 4 s, record the time as less than 4 s, then proceed to 10.9, reporting that the viscosity is less than the value calculated in 11.2.

10.8.3 Follow on-screen instructions, press start button and slowly suspend the weight on the string. Timing will automatically begin with first movement. Do not remove the mass while the viscosity LED on the instrument is flashing. Once the time and viscosity are displayed or the viscosity LED stops flashing, proceed to 10.9.

10.9 Repeat 10.7 and 10.8 for each of the remaining cells in order from left to right.

10.10 *Cleaning:*

10.10.1 After all of the cells have been completed, exit the cooling program and turn on the heater to warm the viscometric cells to room temperature or somewhat higher. The temperature shall not exceed 50 °C.

10.10.2 Remove the upper rotor pivots and the rotors.

10.10.3 With a vacuum, remove the oil samples and rinse the cells with an oil solvent several times, followed by two washings with acetone. Use a vacuum to remove the solvent from the cells after each rinse and allow the acetone to evaporate to dryness after the final rinse.

10.10.4 Clean the rotors in a similar manner.

11. Calculation of Yield Stress and Apparent Viscosity

11.1 Yield stress is given by the following equation:

$$Ys = 3.5\,M \tag{9}$$

where:

Ys = yield stress, Pa, and
M = applied mass, g.

11.2 The apparent viscosity is given by the following equation when using the cell constant obtained in Eq 8:

$$\eta_a = C\,t\,3/r \tag{10}$$

where:

η_a = apparent viscosity, mPa·s,
C = cell constant obtained in Eq 8,
t = time for number (r) of complete revolutions of the rotor, and
r = number of revolutions timed.

12. Report

12.1 *Apparent Viscosity and Yield Stress*—For used oils, report the final test temperature and both apparent viscosity and yield stress.

12.2 *Yield Stress*—Report as less than the value at which rotation was observed.

12.3 *Apparent Viscosity*—Report as follows:

12.3.1 If the apparent viscosity is less than 5000 mPa·s, then report the apparent viscosity as less than 5000 mPa·s.

12.3.2 If the apparent viscosity is between 5000 mPa·s and 100 000 mPa·s, then report the apparent viscosity to the nearest 100 mPa·s.

12.3.3 If the apparent viscosity is between 100 000 mPa·s and 400 000 mPa·s, then report the apparent viscosity to the nearest 1000 mPa·s.

12.3.4 If the apparent viscosity is greater than 400 000 mPa·s, then the apparent viscosity should be reported as greater than 400 000 mPa·s.

12.3.5 When employing software that provides three viscosity values, the first value shall be reported as the apparent viscosity by this test method, D6896. If desired, report all three values, exercising care to also report the sequence of the values. Never report a value that is the average of the three measured values.

13. Precision and Bias[6]

13.1 *Precision (Used Diesel Engine Oils)*—The precision of this test method as determined by the statistical examination of interlaboratory test results is as follows:

13.1.1 *Yield Stress:*

13.1.1.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

Test Temperature, °C	Repeatability, Pa
–20	$0.543{\cdot}(X{+}1)$
–25	$0.504{\cdot}(X{+}1)$

where:

X = mean value, Pa.

13.1.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, exceed the following values only in one case in twenty.

Test Temperature, °C	Repeatability, Pa
–20	$0.926{\cdot}(X{+}1)$
–25	$0.773{\cdot}(X{+}1)$

where:

X = mean value, Pa.

NOTE 11—When no yield stress is detected (movement with 10 g weight), $X = 0$.

13.1.2 *Apparent Viscosity:*

13.1.2.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty. The repeatability as a percent of the mean apparent viscosity is shown as follows:

Test Temperature, °C	Repeatability, % of Mean
–20	$0.0879{\cdot}X$
–25	$0.0616{\cdot}X$

13.1.2.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, exceed the following values only in one case in twenty. The reproducibility as a percent of the mean apparent viscosity is shown as follows:

Test Temperature, °C	Repeatability, % of Mean
–20	$0.186{\cdot}X$
–25	$0.209{\cdot}X$

13.1.3 The interlaboratory program included six laboratories and seven test oils at the –20 °C and –25 °C test temperatures. The used oils included end-of-test drain samples from Mack T8, Mack T8E, Cummins M11-EGR and Mack T10 engine tests, with soot loadings (as measured by thermogravimetric analysis) ranging from approximately 5 to 9 %.[7]

13.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for this test method, no statement on bias is being made.

14. Keywords

14.1 low temperature flow properties; low temperature viscosity; mini-rotary viscometer; pumping viscosity; used diesel engine oil; viscosity; yield stress

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1442.

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1517.

APPENDIXES

(Nonmandatory Information)

X1. TEMPERATURE PROFILES FOR TEST TEMPERATURES

X1.1 See Tables X1.1-X1.3.

TABLE X1.1 Temperature Profile for Test Temperatures –20 °C to –25 °C

Segment Time, h:min	Segment Temperature[A] Beginning, °C		Final, °C	Rate of Change, °C/h	Allowable Temperature Change,[B] °C
nominally 0:02	above 20	to	25.0		
nominally 0:21	25.0	to	1.5		
nominally 0:17	1.5	to	0.0		
nominally 0:03	0.0	to	–3.0		
nominally 0:07	-3.0	to	–4.0	8.5	±0.5
nominally 0:10	-4.0	to	–5.0	6.0	±0.2
6:00	-5.0	to	–8.0	0.5	±0.2
36:00	-8.0	to	–20.0	0.33	±0.2
Hold at this point for –20 °C test temperature[C]					
2:00	-20.0	to	–25.0	2.5	±0.2
Hold at this point for –25 °C test temperature[C]					

[A] If the dual control loop concept is used, the bath set point temperatures should be 5 °C below the corresponding block temperature desired. The maximum bath temperature shall not exceed –5 °C.
[B] Holding the temperature variation to less than ±0.1 °C improves the precision and reproducibility of your viscosity measurements.
[C] The measurement of yield stress and apparent viscosity are to be made within 30 min of reaching the test temperature.

TABLE X1.2 Nominal Elapsed Time to Test Temperature

Test Temperature, °C	Nominal Elapsed Time, h
–20	43
–25	45

TABLE X1.3 Custom Profile Values

Elapsed Time, hh:mm	Test Temperature, of –20.0 °C	Test Temperature, of –25.0 °C	Temperature Tolerance, °C
00:02	25.0	25.0	. . .
00:23	1.5	1.5	. . .
00:40	0.0	0.0	. . .
00:43	–3.0	–3.0	. . .
00:50	–4.0	–4.0	0.5
01:00	–5.0	–5.0	0.2
07:00	–8.0	–8.0	0.2
43:00	–20.0	–20.0	0.2
45:00		–25.0	0.2

X2. SUPPORTING OPERATIONAL INFORMATION

X2.1 If the final temperature is in error in either direction by more than 0.2 °C, do the following before starting another analysis.

X2.1.1 Check the thermometer calibration. For liquid in glass thermometers, check the ice point. An error in the ice point usually indicates air in the thermometer bulb or in the column of liquid.

X2.1.2 Check temperature sensor of the temperature controller for accuracy, in accordance with 8.1.

X2.1.3 Determine if the coolant is flowing and if there is adequate coolant in the reservoir.

X2.1.4 For cold sources operating below –20 °C, replace methanol if wet, as indicated by ice crystals in the top of the cold source reservoir. Cold methanol absorbs water, and as it absorbs water, its cooling capacity decreases. In high humidity areas it may be necessary to change the methanol once a month. Other heat transfer can be used but should be similar to methanol in viscosity and heat capacity at the bath temperature.

X2.1.5 Is the bath refrigeration working properly?

X2.1.6 If manually programmed or using a custom profile, examine the temperature profile program for an error and make the appropriate corrections.

X2.2 The simplest way to check a liquid in glass thermometer calibration is to check its ice point. Other calibration sources are available for both liquid in glass and electronic temperature sensor and are appropriate if they are sufficiently accurate.

X2.3 The software for controlling temperature creates a temperature log during the test. Inspect the recorded cooling profile temperature data for temperature deviations greater than those permitted in Table X1.1. Verify that the cooling rates during the test are in conformance with those in Table X1.1 for temperatures below –4 °C.

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SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D6896 – 14) that may impact the use of this standard. (Approved May 1, 2017.)

(1) Revised 3.1.2.