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# 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.*

## 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, computes the temperature from the measured quantity, and provides a digital output or display 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*.

NOTE 1—Portable electronic thermometers (PET) is an acronym sometimes used to refer to a subset of the devices covered by this definition.

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.

[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] 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

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:

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

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

where:

$\dot{\gamma}$ = shear rate at the surface of the rotor in reciprocal seconds, $s^{-1}$,
$\Omega$ = 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,

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

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.

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

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

where:

$T_r$ = torque applied to rotor, N·m,
$M$ = applied mass, g,
$R_o$ = radius of the shaft, mm,
$R_t$ = radius of the string, mm,
$\tau$ = shear stress at the rotor surface, Pa, and
$h$ = height of the rotor, mm.

For the dimensions given in 6.1.1,

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

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

3.1.7 *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**).

## 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:

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

[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.

| | |
|---|---|
| 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 2—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 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 3—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 4—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 5. This thermowell is to be used for all temperature measurements below 25 °C.

NOTE 5—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 6—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 5. 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:

$\eta_o$ = 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.