Designation: D6896 – 25

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, 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

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 For definitions of terms used in this test method, refer to Terminology D4175.

3.1.2 *apparent viscosity, n*—the determined viscosity obtained by use of this test method.

3.1.3 *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, 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 www.astm.org/contact. 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), 1180 Avenue of the Americas, 10th Floor, New York, NY 10036, http://www.ansi.org.

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

3.1.3.1 *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. This digital output goes to a digital display and/or recording device that may be internal or external to the device.

3.1.3.2 *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.

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

3.1.4 *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.

3.1.5 *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.

3.1.6 *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:

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

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

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,

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

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.

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

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

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

For the dimensions given in 6.1.1,

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

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

3.2.4 *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 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