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Standard Test Method for
Measuring Apparent Viscosity at High-Temperature and High-Shear Rate by Multicell Capillary Viscometer[1]

This standard is issued under the fixed designation D5481; 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.

INTRODUCTION

Several different configurations of capillary viscometers have been successfully used for measuring the viscosity of engine oils at the high shear rates and high temperatures that occur in engines. This test method covers the use of a single apparatus[2] at a single temperature and single shear rate to achieve greater uniformity and improved precision.

1. Scope*

1.1 This test method covers the laboratory determination of high-temperature high-shear (HTHS) viscosity of engine oils at a temperature of 150 °C using a multicell capillary viscometer containing pressure, temperature, and timing instrumentation. The shear rate for this test method corresponds to an apparent shear rate at the wall of 1.4 million reciprocal seconds $(1.4 \times 10^{6}\ s^{-1})$.[3] This shear rate has been found to decrease the discrepancy between this test method and other high-temperature high-shear test methods[3] (Test Methods D4683 and D4741) used for engine oil specifications. Viscosities are determined directly from calibrations that have been established with Newtonian oils with nominal viscosities from 1.4 mPa·s to 5.0 mPa·s at 150 °C. The precision has only been determined for the viscosity range 1.45 mPa·s and 5.05 mPa·s at 150 °C for the materials listed in the precision section.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.2.1 The centiPoise (cP) is a non-SI metric unit of viscosity that is numerically equal to the milliPascal-second (mPa·s).

1.2.2 Pounds per square inch (psi) is a non-SI unit of pressure that is approximately equal to 6.895 kPa. These units are provided for information only in 6.1.1, 7.3, 9.1.2.1, and the tables.

1.3 *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:*[4]

D4683 Test Method for Measuring Viscosity of New and Used Engine Oils at High Shear Rate and High Temperature by Tapered Bearing Simulator Viscometer at 150 °C

D4741 Test Method for Measuring Viscosity at High Temperature and High Shear Rate by Tapered-Plug Viscometer

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants

D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material

3. Terminology

3.1 *Definitions:*

3.1.1 *apparent viscosity, n*—viscosity of a non-Newtonian liquid determined by this test method at a particular shear rate and shear stress.

3.1.2 *density, n*—mass per unit volume of the test liquid at a given temperature.

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

3.1.2.1 *Discussion*—In SI notation, the unit of density is the kilogram per cubic metre. However, for practical use, gram per cubic centimetre is customarily used and is equivalent to 10^3kg/m^3.

3.1.3 *kinematic viscosity, n*—the ratio of the viscosity to the density of the fluid.

3.1.3.1 *Discussion*—Kinematic viscosity is a measure of a fluid's resistance to flow under the force of gravity. In the SI, the unit of kinematic viscosity is the metre squared per second (m^2/s); for practical use, a submultiple (millimetre squared per second, mm^2/s) is more convenient. The centistoke (cSt) is 1 mm^2/s and is often used.

3.1.4 *Newtonian oil or liquid, n*—an oil or liquid 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 liquid that exhibits a viscosity that varies with changing shear rate or shear stress.

3.1.6 *shear rate*—velocity gradient in liquid flow in millimetres per second per millimetre (mm/s per mm) resulting from applied shear stress; the System International (SI) unit for shear rate is reciprocal seconds, s^{-1}.

3.1.6.1 *Discussion*—The velocity gradient in the Multi-Cell Capillary Viscometer varies across the capillary annulus from a maximum at the wall of the capillary to zero at the center of the capillary annulus. Assuming a parabolic flow profile across the capillary, the apparent shear rate at the capillary wall can be calculated as follows:

$$S_a = 4V/\pi R^3 t \qquad (1)$$

where:

S_a = apparent shear rate (at the wall, s^{-1}),
V = volume of fluid (mm^3) passed through the capillary in time t (s), and
R = capillary radius (mm).

S_a is precise for Newtonian liquids which generate a parabolic flow profile but may be approximate for non-Newtonian liquids that do not necessarily generate a parabolic flow profile.

3.1.7 *shear stress, n*—force per unit area causing liquid flow over the area where viscous shear is being caused; in SI, the unit of shear stress is the Pascal (Pa).

3.1.7.1 *Discussion*—In a capillary viscometer, the significant shear stress is at the wall of the capillary. That is, the total force acting on the area of the capillary annulus divided by the inside area of the capillary through which the liquid flows. The shear stress at the wall does not depend on the nature of the liquid (that is, whether the liquid is Newtonian or non-Newtonian). The shear stress at the capillary wall may be calculated as follows:

$$Z = \text{PR}/2\text{L} \qquad (2)$$

where:

Z = shear stress (Pa),
P = pressure drop (in Pa),
R = capillary radius, and
L = capillary length (in units consistent with R).

3.1.8 *viscosity, n*—ratio of applied shear stress and the resulting rate of shear.

3.1.8.1 *Discussion*—It is sometimes called dynamic or absolute viscosity. Viscosity is a measure of the resistance to flow of the liquid at a given temperature. In SI the unit of viscosity is the Pascal second (Pa·s), often conveniently expressed as milliPascal second (mPa·s), which has the English system equivalent of the centiPoise (cP).

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *calibration oils*—those oils used for establishing the instrument's reference framework of apparent viscosity versus pressure drop from which the apparent viscosities of the test oils are determined.

3.2.1.1 *Discussion*—Calibration oils, which are Newtonian fluids, are available commercially[5] or can be blended by the user.

3.2.2 *test oil*—any oil for which the apparent viscosity is to be determined by the test method.

3.2.3 *viscometric cell*—that part of the viscometer comprising all parts which may be wet by the test sample, including exit tube, working capillary, fill tube, pressure/exhaust connection, plug valve, and fill reservoir.

4. Summary of Test Method

4.1 The viscosity of the test oil in any of the viscometric cells is obtained by determining the pressure required to achieve a flow rate corresponding to an apparent shear rate at the wall of $1.4 \times 10^6 \text{ s}^{-1}$. The calibration of each cell is used to determine the viscosity corresponding to the measured pressure.

4.2 Each viscometric cell is calibrated by establishing the relationship between pressure and flow rate for a series of Newtonian oils of known viscosity.

5. Significance and Use

5.1 Viscosity is an important property of fluid lubricants. The viscosity of all fluids varies with temperature. Many common petroleum lubricants are non-Newtonian: their viscosity also varies with shear rate. The usefulness of the viscosity of lubricants is greatest when the viscosity is measured at or near the conditions of shear rate and temperature that the lubricants will experience in service.

5.2 The conditions of shear rate and temperature of this test method are thought to be representative of those in the bearing of automotive engines in severe service.

5.3 Many equipment manufacturers and lubricant specifications require a minimum high-temperature high-shear viscosity at 150 °C and 10^6 s^{-1}. The shear rate in capillary viscometers varies across the radius of the capillary. The apparent shear rate at the wall for this test method is increased to compensate for the variable shear rate.[3]

5.4 This test was evaluated in an ASTM cooperative program.[6]

6. Apparatus

6.1 *Multi-Cell Capillary High-Temperature High-Shear (HTHS) Viscometer,*[5] consisting of several viscometer cells in a temperature-controlled block and including means for controlling and measuring temperature and applied pressure and for timing the flow of a predetermined volume of test oil. Each viscometric cell contains a precision glass capillary and means for adjusting the test oil volume to the predetermined value.

6.1.1 The Multi-Cell Capillary HTHS viscometer has the following typical dimensions and specifications:

Diameter of capillary	0.15 mm
Length of capillary	15 mm to 18 mm
Temperature control	150 °C ± 0.1 °C
Pressure range	350 kPa to 3500 kPa (~50 to 500 psi)
Pressure control	±1 %
Sample volume	7 mL ± 1 mL

6.1.2 The thermometer for measuring the temperature of the block is a preset digital resistance thermometer. The accuracy of this thermometer may be checked by means of a special thermowell and calibrated thermometer[5] whose accuracy is ±0.1 °C or better. See manufacturer's recommendations for procedure.

7. Reagents and Materials

7.1 *Newtonian Oils,*[5] having nominal certified viscosities of 1.5 mPa·s to 7.0 mPa·s at 150 °C. See Table 1.

7.2 *Non-Newtonian Reference Sample,*[5] having a certified viscosity at 150 °C and $10^6 s^{-1}$.

7.3 *Carbon Dioxide or Nitrogen Cylinder,* with reducer valve having a maximum pressure of at least 3500 kPa (~500 psi).

8. Sampling

8.1 A representative sample of test oil, free from suspended solid material and water, is necessary to obtain valid results. When the sample is suspected to contain suspended material, filter with about 10-µm filter paper.

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1767.

TABLE 1 Calibration Oils

Calibration Oil	Approximate Viscosity at 150 °C[A]	Approximate Pressure for Test Method	
	(mPa·s)	psi	kPa
HT22	1.5	190	1310
HT39	2.0	225	1550
HT75	2.7	290	2000
HT150	3.7	375	2590
HT240	5.0	480	3310
HT390[B]	7.0	645	4450

[A] Consult the supplier for specific values.
[B] Consult the supplier for use in instruments with pressure limiters of 525 psi.

9. Calibration and Standardization

9.1 *Calibration:*

9.1.1 The volume and capillary diameter of each viscometric cell in the instrument is provided by the manufacturer, and the nominal flow time, t_o, corresponding to an apparent shear rate at the wall of 1.4×10^6 s^{-1} is calculated by the manufacturer using the following equation:

$$t_o = 4V/1.4*10^6\pi R^3 \qquad (3)$$

where symbols are defined as in 3.1.6.

9.1.2 Using a minimum of four Newtonian calibration oils covering the viscosity range from 1.5 mPa·s to 5 mPa·s at 150 °C, determine the relationship between pressure and flow rate for each viscometric cell. The pressure should be adjusted for each calibration oil such that the measured flow time is within ±2 % of the nominal flow time, t_o. Make three determinations for each oil in each cell. Follow the instrument supplier's documentation for using the software "High Shear Viscosity Calculator" to record these results.

9.1.2.1 The following relationship can be used to express the data:

$$\eta_i = \left[C_1 \cdot t \cdot P - \frac{C_2}{t} \right] \cdot \left[1 + C_3 \cdot \left(1 - \frac{t}{t_o} \right) \right] \qquad (4)$$

where:

η_i = intermediate viscosity, mPa·s,
t = flow time, s,
P = pressure, kPa, and
C_1, C_2, C_3 = coefficients specific to each viscometric cell.

9.1.2.2 Coefficient C_1 is specific to the units in which pressure is expressed, as well as to each cell. Coefficient C_2 will be essentially constant over the relatively narrow range of shear rates and viscosities of interest in measurement of the high-temperature viscosity of automotive engine oil. In more general applications, C_2 may not be constant for all values of Reynolds Number.

9.1.2.3 Intermediate viscosity equals viscosity for the calibration oils.

9.1.2.4 Use the suppliers "High Shear Viscosity Calculator" software to determine the calibration coefficients for each viscometric cell.

9.1.2.5 Alternatively, Annex A1 describes the procedure for determining coefficients C_1, C_2, and C_3.

9.2 *Stability of Viscosity Calibration*—Check the stability of the calibration by running a calibration oil in the same manner as a test oil would be run. This shall be done no less frequently than before each new series of runs and every twentieth run. The non-Newtonian calibration oil should be run at least monthly. The calibration oil viscosity determined in this way must not differ from the standard value by more than the repeatability of the test (see 12.1). If it is out of limits, and if the result is confirmed by a repeat run, look for the source of the trouble, rectify it, and repeat the entire calibration procedure, if necessary. Some possible steps to find the source of the trouble are to check the system thoroughly for faults, including foreign material in the capillary, verify the fidelity of the operating procedure, and accuracy of temperature control, and readout.