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

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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 (1.4×10^{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] used for engine oil specifications. Viscosities are determined directly from calibrations that have been established with Newtonian oils with viscosities from 2 to 5 mPa-s at 150°C.

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

3. Terminology

3.1 *Definitions:*

3.1.1 *apparent shear rate at the wall*—shear rate at the wall of the capillary calculated for a Newtonian fluid, as follows:

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

where:

S_a = apparent shear rate at the wall, s^{-1},
V = volume, mm^3,
R = capillary radius, mm, and
t = measured flow time, s.

3.1.1.1 *Discussion*—The actual shear rate at the wall will differ for a non-Newtonian fluid.

3.1.2 *apparent viscosity*—the determined viscosity obtained by this test method.

3.1.3 *density*—mass per unit volume.

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

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[2] Manning, R. E., and Lloyd, W. A., "Multicell High Temperature High-Shear Capillary Viscometer," SAE Paper 861562. Available from Society of Automotive Engineers (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, www.sae.org.

[3] Girshick, F., "Non-Newtonian Fluid Dynamics in High Temperature High Shear Capillary Viscometers," SAE Paper 922288. Available from Society of Automotive Engineers (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, www.sae.org.

[4] 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.

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

3.1.3.1 *Discussion*—In the SI, the unit of density is the kilogram per metre cubed (kg/m^3); the gram per cubic centimetre (g/cm^3) is often used. One kg/m^3 is 10^{-3} g/cm^3.

3.1.4 *kinematic viscosity*—the ratio of the viscosity to the density of the fluid.

3.1.4.1 *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.

3.1.5 *Newtonian oil or fluid*—an oil or fluid that exhibits a constant viscosity at all shear rates or shear stresses.

3.1.6 *non-Newtonian oil or fluid*—an oil or fluid that exhibits a viscosity that varies with changing shear rate or shear stress.

3.1.7 *shear rate*—the spatial gradient of velocity in laminar flow; the derivative of velocity with respect to distance in a direction perpendicular to the direction of flow.

3.1.7.1 *Discussion*—The derived unit of shear rate is velocity divided by length. With the time in seconds and with consistent units of length, shear rate becomes reciprocal seconds, or s^{-1}.

3.1.8 *shear stress*—force per area of fluid in the direction of flow.

3.1.8.1 *Discussion*—In a capillary viscometer, the significant shear stress is the shear stress at the wall, that is, the total force acting on the cross section of the capillary divided by the area of the inside surface of the capillary. The shear stress at the wall does not depend on the fluid properties (that is, Newtonian or non-Newtonian). The SI unit for shear stress is the pascal (Pa). Mathematically, the shear stress at the wall of a capillary viscometer is as follows:

$$Z = PR/2L \tag{2}$$

where:

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

3.1.9 *viscosity*—the ratio between shear stress and shear rate at the same location.

3.1.9.1 *Discussion*—Viscosity is sometimes called the coefficient of viscosity, or the dynamic viscosity. It is a measure of a fluid's resistance to flow. In the SI, the unit of viscosity is a pascal second (Pa·s); for practical use a submultiple (millipascal second, mPa-s) is more convenient.

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 1.4×10^6 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 *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

[5] The sole source of supply 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] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1378.

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 HTHS viscometer has the following typical dimensions and specifications:

Diameter of capillary	0.15 mm
Length of capillary	15 to 18 mm
Temperature control	150 ± 0.1°C
Pressure range	350 to 3500 kPa (~50 to 500 psi)
Pressure control	±1 %
Sample volume	7 ± 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 certified viscosities of 2 to 7 mPa-s at 150°C. See Table 1.

7.2 *Non-Newtonian Reference Sample,*[5,7] 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.

9. Calibration and Standardization

9.1 *Calibration:*

9.1.1 The volume and capillary diameter of each viscometric cell is provided by the manufacturer, and the 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:

$$t_o = 4V/1.4*10^6 \pi R^3 \tag{3}$$

where symbols are defined as in 3.1.1.

9.1.2 Using a minimum of four Newtonian calibration oils covering the viscosity range from 2 to 5 mPa-s at 150°C, determine the relationship between pressure and flow rate. The pressure should be adjusted for each calibration oil such that the flow time is within ±20 % of the nominal flow time, t_o. Make three determinations for each oil in each cell.

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

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

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 viscometer 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 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.

9.3 *Stability of Temperature Calibration*—Check the calibration of the temperature sensor at least once a year using a standardized thermometer inserted in the thermowell in the aluminum block.

10. Procedure

10.1 Bring the viscometer to the test temperature and allow test temperature to stabilize for at least 30 min. Because the viscometer uses only a small amount of electrical power, it may be desirable to leave the viscometer at test temperature unless use is not anticipated for an extended period of time.

10.2 Flush the previous sample with 4 to 6 mL of the new test sample. Open the plug valve. (**Warning—**Always keep the plug valve closed except when charging or adjusting the volume of sample; NEVER turn on the pressure with the plug valve open.) Insert a 4 to 6-mL test sample, and *close* the plug valve. Turn on the pressure (it is not necessary to adjust the pressure from the previous run.) until the flush sample has passed through the capillary to waste. It is not necessary to

TABLE 1 Calibration Oils

Calibration Oil	Approximate Viscosity[A]	Approximate Pressure for Test Method	
	(mPa-s)	psi	kPa
HT39	2.0	225	1500
HT75	2.7	290	2000
HT150	3.7	375	2500
HT240	5.0	480	3300
HT390	7.0	645	4500

[A] Consult the supplier for specific values.