Designation: ~~D5481 – 10~~ D5481 – 13

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~~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~~33~~ (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 ~~2~~1.4 mPa·s to ~~5 mPa·s at 150°C.~~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).~~(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

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum ~~Products~~ Products, Liquid Fuels, 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. Terminology

3.1 *Definitions:*

3.1.1 *apparent ~~shear~~viscosity, rate n—~~at the wall—shear rate at the wall of the capillary calculated for a Newtonian fluid, as follows:~~*viscosity of a non-Newtonian liquid determined by this test method at a particular shear rate

$$S_a = 4V/\pi R^3 t \tag{1}$$

~~and~~

~~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.~~
shear stress.

~~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.~~

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

3.1.2.1 *Discussion*—

In ~~the SI,~~SI notation, the unit of density is the kilogram per ~~metre cubed (kg/m~~cubic metre. However,[3]~~);~~ the ~~for practical use,~~ gram per cubic centimetre ~~(g/cm^3)~~ is often used. ~~One kg/m~~is customarily used and is~~^{3}~~ is ~~10~~equivalent to~~$^{-3}$~~ ~~g/cm~~10^3kg/m^3.

3.1.3 *kinematic ~~viscosity—~~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 ~~fluid—~~liquid, n—*an oil or ~~fluid that~~ 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—~~fluid, n—*an oil or ~~fluid~~liquid that exhibits a viscosity that varies with changing shear rate or shear stress.

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

3.1.6.1 *Discussion*—

The ~~derived unit of shear rate is velocity divided by length. With the time in seconds and with consistent units~~ 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:

$$S_a = 4V/\pi R^3 t \tag{1}$$

~~of~~

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).
~~length, shear rate becomes reciprocal seconds, or s^{-1}.~~

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—~~ stress, n*—force per ~~area of fluid in the direction of flow.~~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 ~~the shear stress at the wall, that~~at the wall of the capillary. That is, the total force acting on the ~~cross section~~ area of the capillary annulus divided by the inside area of the ~~inside surface of the capillary.~~capillary through which the liquid flows. The shear stress at the wall does not depend on the ~~fluid properties (that is,~~ nature of the liquid (that is, whether the liquid 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~~ shear stress at the capillary wall may be calculated as follows:

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

where:

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

3.1.8 *~~viscosity—~~ viscosity, n*—~~the~~ ratio ~~between~~ of applied shear stress and ~~shear rate at the same location.~~the resulting rate of shear.

3.1.8.1 *Discussion*—

~~Viscosity~~It is sometimes called ~~the coefficient of viscosity, or the dynamic viscosity. It~~dynamic or absolute viscosity. Viscosity is a measure of ~~a fluid's~~ the resistance to ~~flow. In the SI,~~ flow of the liquid at a given temperature. In SI the unit of viscosity is ~~a pascal second (Pa·s); for practical use a submultiple (millipascal second, mPa·s) is more convenient.~~ 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 1.4×10^6 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.

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[5] The sole source of supply known to the committee at this time is Cannon Instrument Co., ~~P.O. Box 16,~~ 2139 High Tech Rd., State College, PA ~~16804.~~16803. 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.

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~~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 to 18 mm~~
Length of capillary	15 mm to 18 mm
~~Temperature control~~	~~150 ± 0.1°C~~
Temperature control	150 °C ± 0.1 °C
Pressure range	350 kPa to 3500 kPa (~50 to 500 psi)
Pressure control	±1 %
~~Sample volume~~	~~7 ± 1 mL~~
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~~±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 ~~2~~1.5 mPa·s to ~~7 mPa·s~~7.0 mPa·s at ~~150°C.~~150 °C. See Table 1.

7.2 *Non-Newtonian Reference Sample,*[5][7]having a certified viscosity at ~~150°C~~150 °C and 10^6s^{-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 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:

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report ~~RR:D02-1378~~RR:D02-1767.

TABLE 1 Calibration Oils

Calibration Oil	Approximate Viscosity at 150 °C[A]	Approximate Pressure for Test Method	
	~~(mPa·s)~~(mPa·s)	psi	kPa
HT22	1.5	190	1310
~~HT39~~	~~2.0~~	~~225~~	~~1500~~
HT39	2.0	225	1550
HT75	2.7	290	2000
~~HT150~~	~~3.7~~	~~375~~	~~2500~~
HT150	3.7	375	2590
~~HT240~~	~~5.0~~	~~480~~	~~3300~~
HT240	5.0	480	3310
~~HT390~~	~~7.0~~	~~645~~	~~4500~~
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.