



# Standard Test Method for Measuring Apparent Viscosity at High-Temperature and High-Shear Rate by Multicell Capillary Viscometer<sup>1</sup>

This standard is issued under the fixed designation D 5481; 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 ( $\epsilon$ ) 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. See Test Method D 4624. This test method covers the use of a single apparatus<sup>2</sup> 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 ( $1.4 \times 10^6 \text{ s}^{-1}$ ).<sup>3</sup> This shear rate has been found to decrease the discrepancy between this test method and other high-temperature high-shear test methods<sup>3</sup> 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 *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:

D 4624 Test Method for Measuring Apparent Viscosity by Capillary Viscometer at High Shear Rate and High Temperature<sup>4</sup>

D 4683 Test Method for Measuring Viscosity at High Shear

Rate and High Temperature by Tapered Bearing Simulator<sup>5</sup>

D 4741 Test Method for Measuring Viscosity at High Temperature and High Shear Rate by Tapered Plug Viscometer<sup>5</sup>

### 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,  $\text{s}^{-1}$ ,

$V$  = volume,  $\text{mm}^3$ ,

$R$  = 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.

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

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 ( $\text{m}^2/\text{s}$ ); for practical use, a submultiple (millimetre squared per second,  $\text{mm}^2/\text{s}$ ) is more convenient. The centistoke (cSt) is  $1 \text{ mm}^2/\text{s}$  and is often used.

3.1.5 *Newtonian oil or fluid*—an oil or fluid that exhibits a

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<sup>2</sup> Manning, R. E., and Lloyd, W. A., "Multicell High Temperature High-Shear Capillary Viscometer," SAE Paper 861562. Available from Society of Automotive Engineers, 400 Commonwealth Dr., Warrendale, PA 15096.

<sup>3</sup> Girshick, F., "Non-Newtonian Fluid Dynamics in High Temperature High Shear Capillary Viscometers," SAE Paper 922288. Available from Society of Automotive Engineers, 400 Commonwealth Dr., Warrendale, PA 15096.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 05.02.

<sup>5</sup> *Annual Book of ASTM Standards*, Vol 05.03.

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:

$$Z = PR/2L \quad (2)$$

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. The centipoise (cP) is 1 mPa·s and is often used.

### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *calibrations 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<sup>6</sup> 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  $1.4 \times 10^6 s^{-1}$ . 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.<sup>3</sup>

5.4 This test was evaluated in an ASTM cooperative program.<sup>7</sup>

## 6. Apparatus

6.1 *High-Temperature High-Shear (HTHS) Viscomete*,<sup>8</sup> 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 HTHS viscometer has the following typical dimensions and specifications:

Diameter of capillary	0.15 mm
Length of capillary	15 to 18 mm
Temperature control	$150 \pm 0.1^\circ\text{C}$
Pressure range	350 to 3500 KPa (50 to 500 psi)
Pressure control	$\pm 1\%$
Sample volume	$7 \pm 1$ mL

6.1.2 The thermometer for measuring the temperature of the block is a pre-set digital resistance thermometer. The accuracy of this thermometer may be checked by means of a special thermowell and calibrated thermometer<sup>7</sup> whose accuracy is  $\pm 0.1^\circ\text{C}$  or better. See manufacturer's recommendations for procedure.

## 7. Reagents and Materials

7.1 *Newtonian Oils*,<sup>7</sup> having certified viscosities of 2 to 7 mPa·s at 150°C. See Table 1.

7.2 *Non-Newtonian Reference Sample*,<sup>7</sup> having a certified viscosity at 150°C and  $10^6 s^{-1}$ .

<sup>7</sup> Report is available from ASTM International Headquarters. Request RR:D02-1378.

<sup>8</sup> A source of supply is Cannon Instrument Co., P.O. Box 16, State College, PA 16804.

<sup>6</sup> Calibration oils, suitable for this purpose, are available from Cannon Instrument Co., P. O. Box 16, State College, PA 16804.

**TABLE 1 Calibration Oils**

Calibration Oil	Approximate Viscosity <sup>A</sup>	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

<sup>A</sup> Consult the supplier for specific values.

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

## 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- $\mu$ m 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  $1.4 \times 10^6 \text{ s}^{-1}$  is calculated by the manufacturer using the following equation:

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

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 (cP) 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  $\pm 20\%$  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:

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

where:

- $\eta$  = viscosity, mPa·s,
- $t$  = flow time, s,
- $P$  = pressure, kPa or psi, 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 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 achieve temperature equilibrium since no time measurement is being made. Turn off the pressure.

10.3 Charge a 9 to 11-mL test sample into the viscometric cell by opening the plug valve, inserting the test sample, and then closing the plug valve.

10.4 Repeat 10.2 and 10.3 for each of the viscometric cells.

10.5 Allow 15 min for the test sample to attain  $150 \pm 0.1^\circ\text{C}$ .

10.6 After temperature equilibrium has been established, ensure that the plug valve is closed on each cell and make measurement of efflux time and pressure as follows:

10.6.1 From the calibration of the viscometric cell and the expected viscosity of the sample (if known), estimate the required pressure to achieve the nominal flow time,  $t_o$  (see 9.1.1). Table 2 provides a guide for setting pressure if the SAE viscosity grade is known. Adjust the pressure in the ballast tank to this value within  $\pm 1\%$ ; allow approximately 10 s for this pressure to stabilize.

10.6.2 Reset the timer to zero.

10.6.3 Open the plug valve and withdraw excess sample by vacuum through the filling tube until no more liquid is being withdrawn. Immediately *close* plug valve and immediately proceed to 10.6.4.

**TABLE 2 Approximate Pressure for Test Method**

SAE Grade	Pressure	
	psi	kPa
20	225	1500
30	250	1750
40	300	2100
50	350	2450

10.6.4 Turn on the run switch for the viscometric cell. Read and record the pressure approximately 10 s after turning on the pressure switch.

10.6.5 When the timer has stopped (indicating that the measurement has been completed), turn off the pressure switch, and record the flow time. It may be necessary to adjust the indicator meter of the timer during the course of a test to ensure clear “start” and “end” signals.

10.6.6 Repeat 10.6.1 through 10.6.5 for each cell.

## 11. Calculation of Results

11.1 Using the calibration equation, calculate the viscosity in mPa·s and the apparent shear rate at the wall in  $s^{-1}$  for each cell. If the apparent shear rate at the wall is not within  $\pm 5\%$  of  $1.4 \times 10^6 s^{-1}$ , recalculate the pressure required and make a second determination in the same or a different cell.

11.2 In some instances, it can only be necessary to determine that the viscosity of the test sample exceeds a specified viscosity. When the exact viscosity of the test sample is not required, the pressure for the cell can be calculated from the calibration using the specified viscosity and flow time,  $t_o$ . If the measured flow time for the test sample exceeds  $t_o$ , then the viscosity must exceed the specified viscosity.

## 12. Report

12.1 Report the viscosity in millipascal seconds (mPa·s) to two decimal places and the test temperature ( $^{\circ}C$ ).

## 13. Precision and Bias<sup>9</sup>

13.1 The precision of the test method was determined by the statistical examination of interlaboratory test results. The precision calculated from the test results is as follows:

13.1.1 The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, in the normal and correct operation of the test method, exceed the following value in only one case in twenty:

$$\text{Repeatability} = 1.6\% \text{ of the mean} \quad (5)$$

13.1.2 The difference between successive results obtained by different operators working in different laboratories on identical test materials would, in the long run, in the normal and correct operation of the test method, exceed the following value in only one case in twenty:

$$\text{Reproducibility} = 5.4\% \text{ of the mean} \quad (6)$$

13.2 *Bias*—This test method uses standards of known viscosity to establish a calibration curve which is then used to determine the viscosities of test oils by interpolation. The standards are thereby forced to their correct values. As a result of this relative method of determining viscosities, this test method has no bias.

13.3 *Relative Bias*—Results from this test method were found, in an interlaboratory study, to agree with those from Test Methods D 4683 and D 4741. They can be expected to give, on average in the long run, the same results for the same oil.

## 14. Keywords

14.1 capillary method; shear testing—petroleum products; viscosity; viscosity—apparent

<sup>9</sup> Supporting data is available from ASTM International Headquarters. Request RR: D02-1378.

## ANNEXES

### (Mandatory Information)

#### A1. CALCULATION OF EQUATION COEFFICIENTS

A1.1 The equation:

$$\eta = \left[ C_1 \cdot t \cdot P - \frac{C_2}{t} \right] \cdot \left[ 1 + C_3 \cdot \left( 1 - \frac{t}{t_o} \right) \right] \quad (A1.1)$$

requires determination of the coefficients  $C_1$ ,  $C_2$ , and  $C_3$ . The value of the nominal flow time,  $t_o$ , corresponding to an apparent shear rate at the wall of  $1.5 \times 10^6 s^{-1}$  must also be determined.

A1.2 To determine  $t_o$ , run the tube as described in Section 10 using a viscosity standard of known density and viscosity between 2.5 and 4.0 cP.

A1.2.1 After making the final adjustment of the volume to be timed (10.6.3), and before turning on the run switch (10.6.4), insert a preweighed vial under the exit from the tube to collect the efflux and measure the weight  $M$  of the efflux.

A1.2.2 Run in accordance with 10.6.4 and 10.6.5. Calculate the timed volume as:

$$V = 1000 M / \rho \quad (A1.2)$$

where:

$V$  = net volume of the efflux,  $mm^3$ ,  
 $M$  = net weight, g, and  
 $\rho$  = density,  $g/cm^3$ .

A1.2.3 Calculate  $t_o$  as described in 9.1.1. Assume  $R = 0.075$  mm, unless otherwise specified for the capillary.

A1.3 The constant  $C_3$  is a small correction factor possibly corresponding to slight changes in efflux volume over the range of efflux time of  $t_o \pm 5$  s. It can be determined with viscosity

standards over the flow times of  $t_o \pm 5$  s. Normally a value of 0.075 will suffice.

A1.4 Using a minimum of four viscosity standards at pressures such that the measured efflux time is within one second of  $t_o$ , determine the flow time and determine by regression the best fit of the data using the equation:

$$\frac{\eta}{(1 + C_3(1 - t/t_o))tP} = C_1 - C_2 \frac{1}{t^2P} \quad (\text{A1.3})$$

## A2. DETERMINATION OF TUBE VOLUME

A2.1 Clean the test cells while maintaining temperature at 100°C. Inject 10 mL of toluene (or other highly aromatic solvent) into each test cell, then flush out. Repeat twice, then flush with *iso*-octane or suitable alternative solvent.

A2.2 Remove solvent by purging tubes with air, nitrogen, or carbon dioxide.

A2.3 Select a reference fluid (Cannon Standard HT 150 or equivalent alternative). Place reference fluid in each cell, and let equilibrate 15 min at a temperature of 150°C. If tests are to be made at a temperature other than 150°C, use this temperature instead of 150°C. Open the plug valves and withdraw excess sample. Close the plug valves, and by way of gas pressure, force the reference fluid out the exhaust tube at the bottom of each cell.

A2.4 Close the lower end of each metal tube with a plastic syringe cap or small rubber stopper. This creates an air-lock between the oil that will be stored in the cell above the glass capillary tube and the end of the exhaust tube.

A2.5 Obtain and weigh a small sample vial and place under each exhaust tube so that the mouth of each vial is slightly higher than the end of each tube.

A1.4.1 Using the values of  $C_1$ ,  $C_2$ , and  $C_3$ , calculate the viscosity using the determined data of  $P$  and  $t$ , determine the viscosity, and calculate the deviations from the certified values in percent of the certified viscosity. Deviations greater than 1.5 % may indicate questionable data items.

A1.4.2 The calculator supplied with the high-temperature high-shear viscometer performs the regression and comparisons described in A1.4 and A1.4.1.

A2.6 Fill each cell with the reference fluid and let equilibrate 15 min at the test temperature. Open the plug valves and withdraw excess sample. Close the plug valves. Remove the caps or stoppers from the exhaust tube, and by way of gas pressure, force the reference fluid out of the exhaust tube.

A2.7 Turn off the gas pressure switch.

A2.8 Weigh each vial. Subtract the mass of the filled vial from the empty vial and determine the volume ( $V$ ) of the oil as:

$$V = W/d \quad (\text{A2.1})$$

where:

$V$  = volume, mL,

$W$  = measured mass, g, and

$d$  = fluid density, g/mL, as provided by the fluid supplier.

A2.9 Repeat A2.4 through A2.8.

A2.10 Repeat A2.4 through A2.8 again.

A2.11 Determine the average volume,  $V$ , and the standard deviation for each cell from the three determinations of volume. If the standard deviation is greater than 0.05 mL, repeat the entire procedure for volume determination.

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