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Standard Test Method for Measuring Apparent Viscosity at High-Temperature and High-Shear Rate by Multicell Capillary Viscometer¹

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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 apparatus² 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 ($1.4 \times 10^6 \text{ s}^{-1}$).³ This shear rate has been found to decrease the discrepancy between this test method and other high-temperature high-shear test methods³ (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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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.*

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 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.

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

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, Liquid Fuels, 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 at a specified temperature. volume.

3.1.2.1 Discussion—

For common fuel and lubricant applications, density at atmospheric pressure is assumed. However, high pressure can impact density.

3.1.3 *kinematic viscosity, n*—the ratio of the dynamic viscosity (η) to the density (ρ) of a liquid at a given temperature.

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, 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 liquid that exhibits a viscosity that varies with changing shear rate or shear stress.

3.1.6 *shear rate, n*—velocity gradient perpendicular to the direction of flow.

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:

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

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*—the force per unit area in the direction of the flow.

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:

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.

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 commercially⁵ 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

⁵ The sole source of supply known to the committee at this time is Cannon Instrument Co., 2139 High Tech Rd., State College, PA 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.

⁶ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1767. Contact ASTM Customer Service at service@astm.org.

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 psi 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 thermometer⁵ 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](#).

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.

7.2 *Non-Newtonian Reference Sample*,⁵ having a certified viscosity at 150 °C and 10⁶s⁻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:

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

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 $\pm 2\%$ 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:

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

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.

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 mL 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 mL 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 mL 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 $150\text{ }^{\circ}\text{C} \pm 0.1\text{ }^{\circ}\text{C}$.

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.

10.6.1.1 The supplier’s software “High Shear Viscosity Calculator” will calculate the approximate pressure for the expected viscosity of the sample for the calibrated viscometric cell being used.

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 bubbles appear and no more liquid is being withdrawn for a duration of approximately 1 s. Immediately *close* plug valve and immediately proceed to 10.6.4.

10.6.4 Turn on the run (start/stop) switch for the viscometric cell. Read and record the pressure approximately 10 s after turning on the run switch.

10.6.5 When the timer has stopped (indicating that the measurement has been completed), turn off the run 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.

10.6.5.1 On instruments which automatically stop the pressure when the measurement is completed, simply record the flow time.

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 apparent 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 $1.4 \times 10^6\text{ s}^{-1}$, recalculate the pressure required and make a second determination in the same or a different cell. The supplier’s software “High Shear Viscosity Calculator” can be used to calculate the apparent viscosity and shear rate using the measured flow time and cell pressure.

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

13. Precision and Bias

13.1 *Precision*—The precision of this test method, which was determined by statistical examination of interlaboratory test results using Practice D6300, is as follows:⁶

TABLE 2 Approximate Pressures for Test Method

SAE Grade Engine Oil	Pressure	
	psi	kPa
16	200	1380
20	225	1500
30	250	1750
40	300	2100
50	350	2450

13.1.1 *Repeatability*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test materials within short intervals of time 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:

2.48 % of the mean at 150 °C

NOTE 1—Repeatability can be interpreted as the maximum difference between two results obtained under repeatability conditions that is accepted as plausible due to random causes under normal and correct operation of the test method.

13.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus 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:

4.19 % of the mean at 150 °C

NOTE 2—Reproducibility can be interpreted as the maximum difference between two results obtained under reproducibility conditions that is accepted as plausible due to random causes under normal and correct operation of the test method.

13.1.3 The precision values stated above were determined by analysis of data using Practice **D6300** from an interlaboratory study⁶ where 16 samples were measured in blind duplicate by six laboratories (192 observations in total). The viscosity of these samples ranged between 1.45 mPa·s and 5.05 mPa·s at 150 °C and $1 \times 10^6 \text{ s}^{-1}$ and consisted of four commercial engine oils, seven experimental (lower viscosity) engine oils, four Newtonian calibration oils, and one non-Newtonian calibration oil.

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 by Practice **D6708** to have no relative bias to Test Method **D4683**, the referee method in the interlaboratory study.⁶ Therefore no bias-correction is required for results reported by this test method.

14. Keywords

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

ANNEXES

(Mandatory Information)

A1. CALCULATION OF EQUATION COEFFICIENTS

A1.1 The equation:

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

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 $1.4 \times 10^6 \text{ s}^{-1}$ 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 mPa·s and 4.0 mPa·s (such as Cannon Standards HT75 or HT150).