



Standard Test Method for Measuring Apparent Viscosity by Capillary Viscometer at High-Temperature and High-Shear Rates¹

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This standard has been approved for use by agencies of the Department of Defense.

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 evolved from a cooperative program that included a wide variety of viscometer designs and procedures. Therefore, this test method is very general but its precision has not been established for any viscometer designs or procedures not evaluated in the cooperative program.

1. Scope

1.1 This test method covers the measurement of high-temperature high-shear viscosity at 150°C. The shear rate for this test method corresponds to an apparent shear rate at the wall of 10^6 s^{-1} . This is the shear rate at the wall calculated for a Newtonian oil. For non-Newtonian oils, the actual shear rate at the wall will vary from this value.

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 1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer²

D 4683 Test Method for Measuring Viscosity at High Temperature and High Shear Rate by Tapered Bearing Simulator³

D 4741 Test Method for Measuring Viscosity at High Temperature and High Shear Rate by Tapered Plug Viscometer³

3. Terminology

3.1 Definitions:

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

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

3.1.3 *kinematic viscosity*—the ratio of the viscosity to the density of the fluid. It 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*—an oil or fluid that exhibits a constant viscosity at all shear rates or shear stresses.

3.1.5 *non-Newtonian oil or fluid*—an oil or fluid 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 to the direction of flow. The derived unit of shear rate is velocity divided by length. With time in seconds and with consistent units of length, shear rate becomes reciprocal seconds, or s^{-1} . In a capillary, the significant shear rate for a Newtonian fluid is as follows:

$$S = 4Q/\pi R^3 \quad (1)$$

where:

S = shear rate, s^{-1} ,

Q = flow rate, and

R = capillary radius in consistent units.

3.1.7 *shear stress*—force per area of fluid in the direction of flow. In a capillary viscometer, the significant shear stress is the maximum shear stress at the wall, that is, the total force acting

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² *Annual Book of ASTM Standards*, Vol 05.01.

³ *Annual Book of ASTM Standards*, Vol 05.03.

on the cross section of the capillary divided by the area of the inside surface of the capillary. The SI unit for shear stress is the pascal (Pa); for practical use in this application, a submultiple, the millipascal (mPa), may be used. Mathematically, the shear stress in 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.

3.1.8 *density*—the mass per unit volume. In the SI, the unit of density is the kilogram per metre cubed (kg/m^3); the gram per cubic centimetre is often used. Density in units of g/cm^3 is multiplied by 1000 to get density in units of kg/m^3 .

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *apparent shear rate at the wall*—shear rate at the wall of the capillary calculated for a Newtonian fluid. The actual shear rate at the wall will differ for a non-Newtonian fluid.

3.2.2 *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. Calibration oils, which are Newtonian fluids, are available commercially⁴ or can be blended by the user.

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

4. Summary of Test Method

4.1 Viscosity is determined from measurements of the relationship between pressure drop and flow rate through a capillary under the desired conditions. Two types of capillary viscometers are recognized by this test method:

4.1.1 *Constant Pressure Viscometer*;—in which the pressure is set to a desired shear stress and the resulting flow rate is measured.

4.1.2 *Constant Flow-Rate Viscometer*; in which the flow rate is set to give a desired shear rate and the resulting pressure drop through the capillary is measured.

4.2 For viscometers of both types, the capillary is first calibrated by establishing the relationship between viscosity and flow rate or between viscosity and pressure drop for a series of Newtonian oils by the same procedure and under the same conditions used for test oils.

4.3 This test method determines the viscosity at 150°C and a shear rate of 10^6 s^{-1} . For constant pressure viscometers, the viscosity at 10^6 s^{-1} is determined by interpolation from viscosities measured at several shear stresses. Constant flow-rate viscometers usually can be set for a flow rate that will be at or very close to a shear rate of 10^6 s^{-1} . Operating procedures are highly dependent on the design of each particular viscometer.

5. Significance and Use

5.1 Many equipment manufacturers and lubricant specifications require a minimum high-temperature high-shear viscosity

at 150°C and 10^6 s^{-1} . The apparent shear rate at the wall for this test method is 10^6 s^{-1} . For non-Newtonian oils, the shear rate at the wall will vary from this value. For oils tested so far, this test method has shown higher results for non-Newtonian oils than have rotational viscometer methods (Test Methods D 4683 and D 4741) run at 10^6 s^{-1} . The difference has been on the order of 0.1 mPa·s. Experiments by the Subcommittee 7 high-temperature high-shear working group suggest the flow rate in this test method should be increased by 50 % for the results to correspond with the rotational methods.

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

5.3 This test method was evaluated in an ASTM cooperative program.⁵

6. Apparatus

6.1 At the time this program was conducted, no suitable capillary viscometers were available commercially. Annex A1 tabulates the characteristics of the viscometers that were used in the ASTM evaluation of this test method. The precision of this test method was calculated from data obtained with viscometers that had length-to-diameter (L/D) ratios between 120 and 280. The viscometers described in Annex A1 were all research-type instruments.

6.2 For the convenience of anyone wishing to build an HTHS capillary viscometer, working drawings, specifications, and operating procedures are available⁴ for the viscometer used by Laboratory X. This design is offered because of its availability and not because of any implied preference.

6.3 The principal components of the two types of viscometers are as follows:

6.3.1 Constant Pressure Viscometer:

6.3.1.1 Capillary.

6.3.1.2 Means to control and measure oil temperature.

6.3.1.3 Means to set pressure drop.

6.3.1.4 Means to measure flow rate.

6.3.2 Constant Flow Rate Viscometer:

6.3.2.1 Capillary.

6.3.2.2 Means to control and measure oil temperature.

6.3.2.3 Means to set flow rate.

6.3.2.4 Means to measure pressure drop.

7. Sampling

7.1 A representative sample of test oil free from suspended solid material and water is necessary to obtain valid results. If the sample is suspected to contain suspended material, filter with $10 \mu\text{m}$ or finer filter paper.

8. Calibration and Standardization

8.1 Calibration:

8.1.1 *Constant Pressure Viscometer*—Using a minimum of five Newtonian calibration oils covering the viscosity range from about 2 mPa·s (cP) to about 7 mPa·s (cP) at 150°C accurately determine viscosity as a function of flow rate (or flow time for a specified volume) for each pressure drop that is run.

⁴ Available from Cannon Instrument Co., P.O. Box 16, State College, PA 16804.

⁵ Supporting data are available from ASTM Headquarters. Request RR: D02-12.01.

NOTE 1—When data are subsequently obtained with test oils, several values of pressure drop are needed to allow interpolation to 10^6 s^{-1} shear rate. Fixing a regular schedule of pressure drops for this purpose is convenient and avoids the need to determine calibrations for every test run.

8.1.1.1 The calibration data can be presented either graphically, as plots of viscosity versus flow rate (or flow time) or mathematically. In either case, each pressure drop will have its own calibration curve. The following empirical relationship has been found to adequately describe the calibration data.

$$V = a_0 + a_1 T + a_2 T^2 + a_3 / T^3 \quad (3)$$

where:

V = viscosity, mPa·s (cP),
 T = flow time for 20 mL, s (20 mL is the flow volume that is timed), and

a_0, a_1, a_2, a_3 = constants.

8.1.1.2 The following relationship, also, has been used successfully with capillary viscometers:

$$V = c_1 t P - c_2 / t \quad (4)$$

where:

V = viscosity, mPa·s,
 t = flow time, for a calibrated volume, s,
 P = pressure, kPa (1 psi = 6.89 kPa), and
 c_1, c_2 = constants.

8.1.1.3 Constant 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 oils. In more general applications, c_2 may not be constant for all values of Reynolds Number.

8.1.2 *Constant Flow Rate Viscometer*—Using a minimum of five calibration oils covering the viscosity range from about 2 mPa·s (cP) to about 7 mPa·s (cP) at 150°C, accurately determine viscosity as a function of pressure for each flow rate that is run.

8.1.2.1 The calibration data can be presented either graphically, as plots of viscosity versus pressure, or mathematically. In either case, each flow rate will have its own calibration curve. The following empirical relationship has been found to adequately describe the calibration data:

$$V = b_0 + b_1 P \quad (5)$$

where:

V = viscosity, mPa·s,
 P = pressure, kPa, and
 b_0, b_1 = constants.

8.1.3 *Stability of Viscosity Calibration*—Check the stability of the calibration by running a calibration oil in the same manner a test oil would be run. This is done no less frequently than every twentieth run. 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 does, and if the result is confirmed by a repeat run, look for the source of the trouble, rectify it, and recalibrate if necessary. Some possible steps to find the source of the trouble are to check the system thoroughly for faults, verify the operating procedure, and check the temperature control and readout.

8.1.4 *Stability of Temperature Calibration*—Check the calibration of the temperature sensor at least once a year.

8.2 *Standardization*—Standardize each viscometer to verify its accuracy before its initial use and at least once a year thereafter. Standardization is accomplished by using the viscometer to measure the viscosity of a standard non-Newtonian reference oil. The measured viscosity must not differ from the certified viscosity of the standard by more than the reproducibility of the test (see 12.1). A standard reference oil for this purpose is commercially available.⁴

9. Procedure

9.1 Because of the diversity of viscometers that have proven satisfactory for this application, no specific test procedure can be set forth. However, certain aspects of the procedure are common to all viscometer designs and can be prescribed in general terms.

9.1.1 *Flushing*—Before the viscosity of an oil is measured, the system must be free of contamination from a previous oil tested. Every test procedure must have a provision to accomplish this and that flushing technique must be evaluated to assure its effectiveness.

9.1.2 Effectiveness of the flushing technique is checked by making a series of viscosity measurements with two oils of widely different viscosity. Typically, the sequence of the test is L L H H L L H H, where L is a low-viscosity oil and H is a high-viscosity oil. Carryover of the previous test oil is indicated by a systematic increase in viscosity between the first and second test of a back-to-back pair of tests with the high-viscosity oil, and, similarly, a systematic decrease in viscosity between the first, and second measurement in a pair of back-to-back measurements with the low-viscosity oil.

9.2 *Temperature Measurement and Control*—Close control and accurate measurement of the test oil temperature at the entrance to the capillary is essential for accurate viscosity measurements. For typical multigrade engine oils at 150°C, a 1°C temperature difference corresponds to about 1.5 % viscosity difference.

9.3 *Reynolds Number Limitation*—For any set of capillary dimensions, operating conditions should be chosen to keep the flow in the laminar regime throughout the operating range of the instrument, both for Newtonian and for nonNewtonian oils. Laminar flow is characterized by a Reynolds Number smaller than about 2000.

$$\text{Reynolds Number} = (D \times V \times d) / \eta \quad (6)$$

where:

D = diameter of capillary, mm,
 V = velocity of flow through capillary, m/s,
 d = density of fluid, kg/m^3 , and
 η = viscosity, mPa·s (1 mPa·s = 1 cP).

10. Calculation of Results

10.1 Enter the appropriate calibration curve with the observed value of the dependent variable (pressure in a constant flow rate viscometer and flow rate (or time) in a constant pressure viscometer), and read or calculate the corresponding viscosity.

11. Report

11.1 Report the viscosity in millipascal seconds (mPa·s) to two decimal places and the test temperature (°C).

12. Precision and Bias ⁵

Reproducibility = 1.08

(8)

12.1 *Precision*—The precision of the test method was determined by the statistical examination of interlaboratory test results. The analysis did not use data from Laboratories P and Q because those two laboratories did not report data for a complete class of oils in the round-robin program. The precision calculated from the test results from the other seven laboratories is as follows:

12.1.1 *Repeatability*—The ratio of successive results (larger value divided by the smaller) obtained by the same operator with the same apparatus under constant operating conditions on identical test material 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.

$$\text{Repeatability} = 1.05 \quad (7)$$

12.1.2 *Reproducibility*—The ratio between two single and independent results (the larger value divided by the smaller), obtained by different operators working in different laboratories on identical test material would, in the long run, exceed the following value only in one case in twenty.

12.2 *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.

12.2.1 No justifiable statement can be made on the bias of the procedure described in this test method since all determinations are relative to the calibration fluids.

12.2.2 However, this test method will generally give higher values (about 0.1 mPa·s higher) for non-Newtonian oils than rotational viscometer test methods (Test Methods D 4683 and D 4741). This reflects the different operating conditions of the tests, and is not a bias.

13. Keywords

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

ANNEX

(Mandatory Information)

A1. CAPILLARY VISCOMETER CHARACTERISTICS

Laboratory	X	T	U	Z	R	Q ^A	P ^A	S	W
Capillary									
Diameter, mm	0.1579	0.158	0.1534	0.194	0.194	0.100	0.10–0.16	0.1830	0.1958
Length, mm	19.61	20	20	25.4	25.4	100	100	50.8	38.1
L/D	124	127	131	131	131	1000	1000–625	278	195
Material	glass	glass	glass	stainless steel	stainless steel	stainless steel	stainless steel	stainless steel	stainless steel
Entrance	square	square	square	square	square	square	square	square	square
Heating method	air bath	air bath	liquid bath	electric coils	electric coils	liquid bath	liquid bath	electric coils	liquid bath
Pressurization method	pump	pump	nitrogen	piston	piston	pump	pump	piston	nitrogen
Dependent variable	flow rate	flow rate	flow rate	pressure	pressure	pressure	pressure	pressure	flow rate

^AData from Laboratories Q and P were not used in the calculation of precision.

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