



Designation: D 4957 – 95

Standard Test Method for Apparent Viscosity of Asphalt Emulsion Residues and Non- Newtonian Bitumens by Vacuum Capillary Viscometer¹

This standard is issued under the fixed designation D 4957; 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.

1. Scope

1.1 This test method describes procedures primarily designed to determine the apparent viscosities of residues obtained by distilling asphalt emulsions according to Test Methods D 244. It is also recommended for use on non-Newtonian asphalts at any temperature within the capability of the apparatus. This test method is useful in characterizing rheological properties of non-Newtonian bitumens as a function of shear rate under the conditions of the test method. This test is run in straight open-end tube viscometers, normally at 140°F (60°C), but is suitable for use at other temperatures. It is applicable over the range from 50 to 500 000 P (5 to 50 000 Pa·s).

NOTE 1—The precision for this test method is based on determinations made at 140°F (60°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 244 Test Methods for Emulsified Asphalts²

D 2171 Test Method for Viscosity of Asphalts by Vacuum Capillary Viscometer²

E 1 Specification for ASTM Thermometers³

E 77 Test Method for Inspection and Verification of Thermometers³

3. Terminology

3.1 Definitions:

3.1.1 *apparent viscosity*—the determined viscosity obtained by the test method under description. Viscosity is the resistance to deformation or internal friction of a liquid expressed as the

ratio of the shear stress to shear rate, whether this ratio is constant or not. The unit of viscosity obtained by dividing the shearing stress in dynes/cm² by the rate of shear in reciprocal seconds is called the poise (P). The SI unit of viscosity is the pascal-second (Pa·s) with dimensions of N·s/m², and is equivalent to 10 P.

3.1.2 *Newtonian liquid*—a liquid in which the rate of shear is proportional to the shearing stress. The constant ratio of the shearing stress to rate of shear is the viscosity of the liquid. If the ratio is not constant, the liquid is non-Newtonian.

NOTE 2—A power law fluid is a material in which the relation between the log of the shear stress is linear with the log of the shear rate. The slope of this relation is called the shear susceptibility, *C*. If *C* is less than unity, the material is classified as pseudoplastic and the apparent viscosity decreases with increased stress. If *C* is greater than one, the material is dilatant and the apparent viscosity increases with stress. If *C* is unity the material shows Newtonian flow. Most real materials show some non-Newtonian behavior and the apparent viscosity, computed as stress divided by the shear rate, is reported.

3.1.3 *rheogram*—a rheological diagram which shows how the apparent viscosity of a material varies with the shear rate. An apparent viscosity at a specific shear rate, normally 1 s⁻¹ can be estimated from this plot. A typical rheogram with an example is shown in Fig. 1.

4. Summary of Test Method

4.1 The time is measured for a fixed volume of the liquid to be drawn up through a straight open-end capillary tube by means of vacuum, under closely controlled conditions of vacuum and temperature. The apparent viscosity in poises is calculated by multiplying the flow time in seconds by the appropriate viscometer calibration factor or calculated viscometer constant.

5. Significance and Use

5.1 This test method is useful for characterizing the flow behavior of asphalt emulsion residues and non-Newtonian bitumens. However, since non-Newtonian viscosity values depend on the level of shearing stress, its duration, and the shear history of the material, a non-Newtonian viscosity is not a unique material property. Instead, it is a parameter which is characteristic of the fluid-viscometer system under the conditions of the measurement procedure. Therefore, comparisons of

¹ This test method is under the jurisdiction of ASTM Committee D-4 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.44 on Rheological Tests.

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² Annual Book of ASTM Standards, Vol 04.03.

³ Annual Book of ASTM Standards, Vol 14.03.

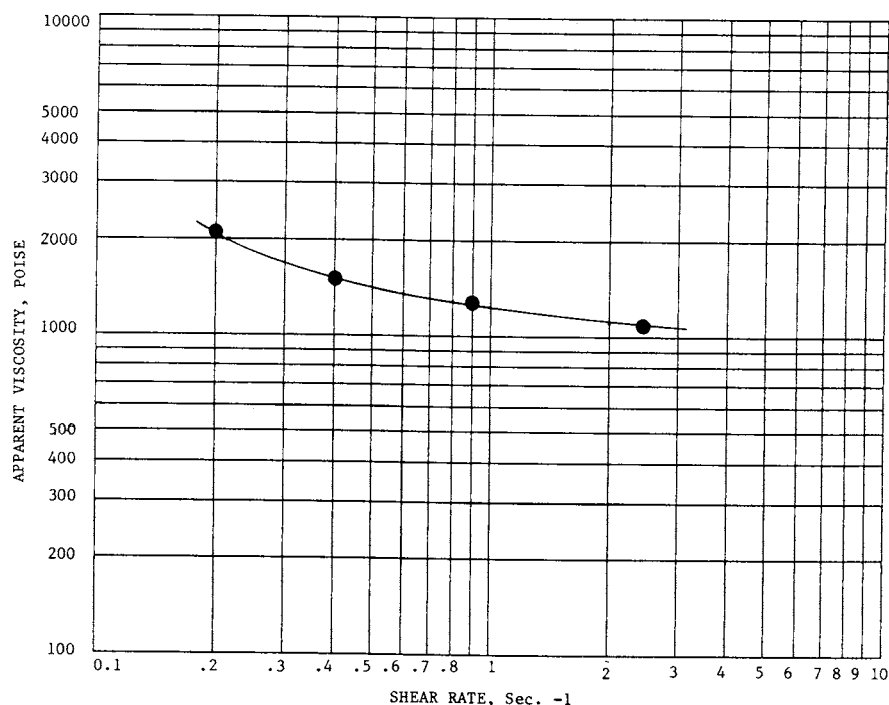


FIG. 1 Typical Log-Log Rheogram Plot with Example

non-Newtonian material behavior should only be made using apparent viscosities determined in similar viscometers under similar conditions of shearing stress and stress history. Procedures of sample preparation are especially important for repeatability or reproducibility of test results.

6. Apparatus

6.1 *Viscometers*—Capillary types, made of borosilicate glass, annealed, suitable for this test are as follows:

6.1.1 *Modified Koppers Vacuum Viscometer*, as described in Annex A1. Calibrated viscometers are available from commercial suppliers. Details regarding calibration of viscometers are given in A1.3.

6.2 *Thermometers*—Calibrated liquid in glass thermometers of an accuracy after correction of 0.04°F (0.02°C) can be used or any other thermometric device of equal accuracy. ASTM Kinematic Viscosity Thermometers 47F and 47C are suitable for the most commonly used temperature of 140°F (60°C). See Test Method D 2171, Table on Kinematic Viscosity Test Thermometers for details on specific thermometers.

6.2.1 The specified thermometers are standardized at “total immersion”, which means immersion to the top of the mercury column with the remainder of the stem and the expansion chamber at the top of the thermometer exposed to room temperature. The practice of completely submerging the thermometer is not recommended. When thermometers are completely submerged, corrections for each individual thermometer based on calibration under conditions of complete submergence must be determined and applied. If the thermometer is completely submerged in the bath during use, the pressure of the gas in the expansion chamber will be higher or lower than during standardization, and may cause high or low readings of the thermometer.

6.2.2 It is essential that liquid-in-glass thermometers be calibrated periodically using the technique given in Test Method E 77 (see Appendix X1).

6.3 *Bath*—A bath suitable for immersion of the viscometer so that the liquid reservoir or the top of the capillary, whichever is uppermost, is at least 20 mm below the upper surface of the bath liquid, and with provisions for visibility of the viscometer and the thermometer. Firm supports for the viscometer shall be provided. The efficiency of the stirring and the balance between heat loss and heat input must be such that the temperature of the bath medium does not vary by more than $\pm 0.05^\circ\text{F}$ ($\pm 0.03^\circ\text{C}$) over the length of the viscometer or from viscometer to viscometer in the various bath positions.

6.4 *Vacuum System*—A vacuum system⁴ capable of maintaining a vacuum to within ± 0.5 mm of the desired level up to and including 500 mm Hg. The essential system is shown schematically in Fig. 2. Glass tubing of 6.35 mm ($\frac{1}{4}$ in.) inside diameter should be used, and all glass joints should be airtight so that when the system is closed, no loss of vacuum is indicated by the open-end mercury manometer having 1-mm graduations. A vacuum or aspirator pump is suitable for the vacuum source.

6.5 *Timer*—A stopwatch or other timing device graduated in divisions of 0.1 s or less and accurate to within 0.05 % when tested over intervals of not less than 15 min shall be used. Electrical timing devices may be used only on electrical circuits the frequencies of which are controlled to an accuracy of 0.05 % or better.

6.5.1 Alternating-current frequencies that are intermittently and not continuously controlled, as provided by some public

⁴ The vacuum control system marketed by Cannon Instrument Co., P.O. Box 16, State College, PA, 16801, has been found satisfactory for this purpose.

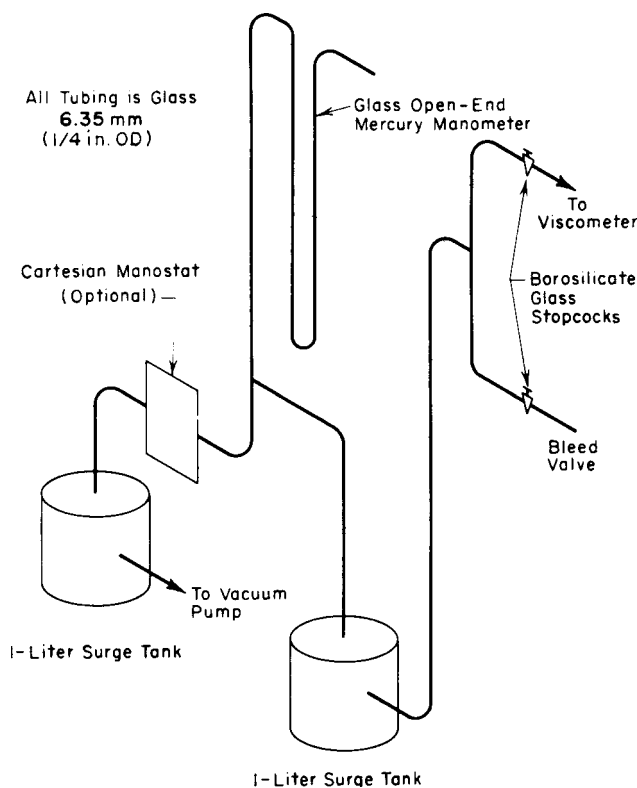


FIG. 2 Suggested Vacuum System for Vacuum Capillary Viscometers

power systems, can cause large errors, particularly over short timing intervals, when used to actuate electrical timing devices.

6.6 Sample Preparation Oven—A suitable oven for semi-continuous operations with control of temperature up to $396 \pm 4^\circ\text{F}$ ($202 \pm 2^\circ\text{C}$) is required. It should have a fast heating rate capability in order not to delay testing when needed on short notice.

7. Sample Preparation

7.1 Asphalt Emulsion Residue—If the sample is the residual product from the emulsion distillation test at 500°F (260°C), pour a suitable portion of the total residue into a 50-mL beaker and allow to cool to $356 \pm 9^\circ\text{F}$ ($180 \pm 5^\circ\text{C}$). Then stir this portion of the sample at 1 r/s for 10 s. Following this, pour the proper amount into the viscometer as in Section 8. Alternatively, allow this portion of the residue to cool completely and set aside for future testing. This material should then be handled as in 7.2.

7.2 Ambient Sample—Heat the sample in an oven maintained at $383 \pm 4^\circ\text{F}$ ($195 \pm 2^\circ\text{C}$). Stir the sample occasionally until homogeneous and pour into a 50-mL preheated beaker. Stir the sample at approximately 1 r/s for 10 s.

NOTE 3—Because of the nature of some asphalts and asphalt emulsion residues, their shear and thermal history prior to testing may cause variations in test results. Careful sample preparation is most important for consistent test results.

NOTE 4—In cases where the asphalt is not sufficiently fluid to pour at 356°F or too fluid to pour without splattering during transfer into the viscometer; other pouring temperatures may be used providing there is agreement between interested parties.

8. Procedure

8.1 Follow the general procedure described as follows, however, the specific details of the Modified Koppers viscometer are described in Annex A1.

8.1.1 Maintain the bath at the test temperatures to within $\pm 0.05^\circ\text{F}$ ($\pm 0.03^\circ\text{C}$). Apply the necessary corrections, if any, to all thermometer readings.

8.1.2 Select a clean, dry viscometer that will give a flow time between 50 and 200 s for the C zone of a Modified Koppers viscometer. Preheat the viscometer filling tube and capillary tube separately in the oven at $383 \pm 4^\circ\text{F}$ ($195 \pm 2^\circ\text{C}$) for 5 min to assist in eliminating air bubbles when the sample is poured.

8.1.3 Charge the viscometer filling tube by pouring the prepared sample to within ± 2 mm of the fill line.

NOTE 5—In cases where highly viscous materials are being tested, the possibility of air entrapment in the sample is greater. To alleviate this problem, a slight vacuum on the fill tube before the capillary tube is placed in position is suggested. This can be accomplished using a light vacuum source attached to the fill tube for 30 s. A simple cork and tube should suffice to achieve the desired effects.

8.1.4 Place the charged viscometer filling tube in an oven maintained at $383 \pm 4^\circ\text{F}$ ($195 \pm 2^\circ\text{C}$) for a period of 10 ± 2 min to allow large air bubbles to escape.

8.1.5 Remove the viscometer filling tube and capillary tube from the oven and then properly position the capillary tube into the filling tube. Insert the viscometer in a holder and position the viscometer in the bath so that the uppermost timing mark is at least 20 mm below the surface of the bath liquid.

NOTE 6—Capillary action on certain small-diameter viscometer tubes may cause a rise of the sample above the first timing mark. If this is a problem, the capillary tube should be withheld from contact with the sample for a few minutes prior to running the test, then positioned quickly and allowed to equilibrate a minute or two.

8.1.6 Establish the vacuum to within ± 0.5 mm of the desired level and connect the vacuum system to the viscometer with the toggle valve or stopcock closed in the line leading to the viscometer.

8.1.7 After the viscometer has been in the bath for 30 ± 5 min, start the flow of asphalt in the viscometer by opening the toggle valve or stopcock in the line leading to the vacuum system.

8.1.8 Measure to within 0.1 s the time required for the leading edge of the meniscus to pass between successive pairs of timing marks.

8.1.9 Upon completion of the test, clean the viscometer. There are numerous methods that can be used. Two suggested methods are given in A1.4 and A1.5.

9. Calculation

9.1 Select the appropriate viscosity constant for any set of timing marks from the calibration constants given by the manufacturer.

9.1.1 Using these appropriate viscometer constants, calculate and report the apparent viscosity to three significant figures according to the following equation:

$$\text{Apparent Viscosity, } \eta_{\text{zone}} = K_{\text{zone}} \cdot t \quad (1)$$

