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## Standard Test Method for Viscosity of Asphalts by Vacuum Capillary Viscometer<sup>1</sup>

This standard is issued under the fixed designation D2171/D2171M; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense. This test method has been approved by the sponsoring committees and accepted by the cooperating societies in accordance with established procedures.

### 1. Scope

1.1 This test method covers procedures for the determination of <u>the apparent</u> viscosity of asphalt binder (bitumen) by vacuum capillary viscometers at  $60^{\circ}C$  [140°F].  $60^{\circ}C$  [140°F]. It is applicable to materials having viscosities in the range from 0.0036 to over 20 000 Pa·s [0.036 to over 200 000 P].

Note 1—This test method is suitable for use at other temperatures, but the precision is based on determinations on asphalt binders at 60°C [140°F]. NOTE 1—This test method is suitable for use at other temperatures, but the precision is based on determinations on asphalt binders at 60°C [140°F]. NOTE 2—Modified asphalt binders or asphalt binders that have been conditioned or recovered are typically non-Newtonian under the conditions of this test. The apparent viscosity for non-Newtonian asphalt binders varies with shear rate. When the flow is non-Newtonian in a capillary tube, the shear rate determined using this test method may be invalid. The presence of non-Newtonian behavior for the test conditions of this test can be verified by measuring the viscosity with viscometers having different-sized capillary tubes or with different pressure heads. The defined precision limits in Section 11 may not be applicable to non-Newtonian asphalt binders. Test Method D4957 may be a more applicable method for testing non-Newtonian asphalts.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance nonconformance with the standard.

1.3 Warning—Mercury has been designated by the United States Environmental Protection Agency (EPA) and many state agencies as a hazardous material that can cause central nervous system, kidney, and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing mercury-containing products. See the applicable product Material Safety Data Sheet (MSDS) or Safety Data Sheet (SDS) for details and the EPA's website—http://www.epa.gov/mercury/faq.htm—for additional information. Users should be aware that selling mercury, mercury-containing products, or both, intoin your state may be prohibited by state law.

1.4 The text of this standard references notes and footnotes that provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

1.5 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 safety, health, and healthenvironmental practices and determine the applicability of regulatory limitations prior to use.

1.6 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.

#### 2. Referenced Documents

2.1 ASTM Standards:<sup>2</sup>

D8 Terminology Relating to Materials for Roads and Pavements

D3666 Specification for Minimum Requirements for Agencies Testing and Inspecting Road and Paving Materials

D4957 Test Method for Apparent Viscosity of Asphalt Emulsion Residues and Non-Newtonian Asphalts by Vacuum Capillary Viscometer

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.44 on Rheological Tests.

Current edition approved June 1, 2010 Dec. 15, 2018. Published August 2010 December 2018. Originally approved in 1963. Last previous edition approved in 20072010 as  $\frac{D2171 - 07D2171/D2171M - 10.^{e1}}{DOI: 10.1520/D2171 - 10.1520/D2171 D2171M - 18.}$ 

<sup>&</sup>lt;sup>2</sup> 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.

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E1 Specification for ASTM Liquid-in-Glass Thermometers

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E77 Test Method for Inspection and Verification of Thermometers

E879 Specification for Thermistor Sensors for General Purpose and Laboratory Temperature Measurements

E1137/E1137M Specification for Industrial Platinum Resistance Thermometers

### 3. Terminology

3.1 *Definitions: Definitions of Terms Specific to This Standard:* 

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

3.1.2 *non-Newtonian viscosity, n*—the viscosity of a liquid that is shear-rate dependent, in which the rate of shear is not linearly proportional to the shearing stress.

3.1.3 <u>viscosity</u>—<u>viscosity (coefficient of)</u>, <u>n</u>—the ratio between the applied shear stress and rate of shear is called the coefficient of viscosity. This coefficient is thus a measure of the resistance to flow of the liquid. It is commonly called the viscosity of the liquid. The SI unit of viscosity is 1 Pa·s (1 N·s/m<sup>2</sup>) and is called a Pascal-second. The cgs unit of viscosity is 1 g/cm·s (1 dyne·s/cm<sup>2</sup>) and is called a poise (P). 1 Pa·s is equivalent to 10 P.

### 3.1.3.1 Discussion—

This coefficient is a measure of the resistance to flow of the liquid. It is commonly called the viscosity of the liquid. The cgs unit of viscosity is 1 g/cm·s (1 dyne·s/cm<sup>2</sup>) and is called a poise (P). The SI unit of viscosity is 1 Pa·s (1 N·s/m<sup>2</sup>) and is called a Pascal-second. One Pa·s is equivalent to 10 P.

3.2 For definitions of other terms used in this standard, refer to Terminology D8.

### 4. Summary of Test Method

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

NOTE 2—The rate of shear decreases as the liquid moves up the tube, or it can also be varied by the use of different vacuum or different size viseometers. Thus, this method is suitable for the measurement of viscosities of Newtonian (simple) and non-Newtonian (complex) liquids.

### 5. Significance and Use/catalog/standards/sist/d580ff04-c0d8-4a80-a27c-7a97d5ef0691/astm-d2171-d2171m-18

5.1 The viscosity at  $60^{\circ}C [140^{\circ}F] \underline{60 \circ C} [140^{\circ}F]$  characterizes flow behavior and may be used for specification requirements for <u>cutbacks</u> cutback asphalt and asphalt binders.

NOTE 3—The quality of the results produced by this standard are dependent on the competence of the personnel performing the procedure and the capability, calibration, and maintenance of the equipment used. Agencies that meet the criteria of Specification D3666 are generally considered capable of competent and objective testing, sampling, inspection, etc. Users of this standard are cautioned that compliance with Specification D3666 alone does not completely ensure reliable results. Reliable results depend on many factors; following the suggestions of Specification D3666 or some similar acceptable guideline provides a means of evaluating and controlling some of those factors.

### 6. Apparatus

6.1 Viscometers, capillary-type, made of borosilicate glass, annealed, suitable for this test are as follows:

6.1.1 Cannon-Manning Vacuum Viscometer (CMVV), as described in Appendix X1.

6.1.2 Asphalt Institute Vacuum Viscometer (AIVV), as described in Appendix X2.

6.1.3 Modified Koppers Vacuum Viscometer (MKVV), as described in Appendix X3.

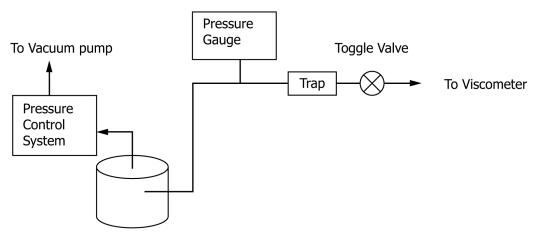
6.1.4 Calibrated viscometers are available from commercial suppliers. Details regarding calibration of viscometers are given in Appendix X4.

Note 4—The viscosity measured in a CMVV may be from 1 to 5 % lower than either the AIVV or MKVV having the same viscosity range. This difference, when encountered, may be the result of non-Newtonian flow.<sup>3</sup>

6.2 Thermometers—Calibrated liquid-in-glass thermometers<u>A calibrated thermometer</u> (see Table X5.1) of an accuracy after correction of 0.02°C [0.04°F] can be used or any other thermometric device of equal accuracy. ASTM Kinematic Viscosity Thermometers 47C and 47F conforming to Specification with suitable range and estimated measurement uncertainty of E1 are

<sup>&</sup>lt;sup>3</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D04-1003. Contact ASTM Customer Service at service@astm.org.

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1-Litre Surge Tank FIG. 1 Suggested Vacuum System for Vacuum Capillary Viscometers

suitable for the most commonly used temperature of 60°C [140°F]. 0.02 °C [0.04 °F] or less at 95 % confidence interval listed on the calibration certificate and is one of the following:

6.2.1 The specified thermometers are standardized at" total immersion," which means immersion to the top of the liquid 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 bathA liquid-in-glass thermometer that is calibrated annually. The calibration shall be verified at the ice point in accordance with Appendix X5during use, the pressure of the gas in the expansion chamber will be higher or lower than during standardization, and may cause high or low. ASTM Kinematic Viscosity Thermometers 47C and 47F conforming to Specification E1 readings on the thermometer. Thermometric devices for this test method must be standardized at least every 6 months.are suitable for the most commonly used temperature of 60 °C [140 °F].

NOTE 5—The specified thermometers in 6.2.1 are standardized as "total immersion" thermometers, which are designed to indicate temperatures correctly when just that portion of the thermometer containing the liquid is exposed to the temperature being measured and the remainder of the stem and expansion chamber at the top of the thermometer exposed to the room temperature. The practice of completely submerging the thermometer is not recommended. When a "total immersion" thermometer is 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 calibration, and may cause high or low readings on the thermometer.

6.2.2 A platinum resistance thermometer (PRT) with a sensor which conforms to the requirements of Specification E1137/E1137M. The thermometer shall be calibrated annually as a single unit and have a three- or four-wire connection configuration. The sensing element shall be immersed to the depth specified by the manufacturer adjacent to the capillary tube.

NOTE 6—A minimum of 50 mm (2 in.) of the thermometer sheath (wire-connecting side) shall not be subjected to the bath medium unless otherwise specified by the manufacturer.

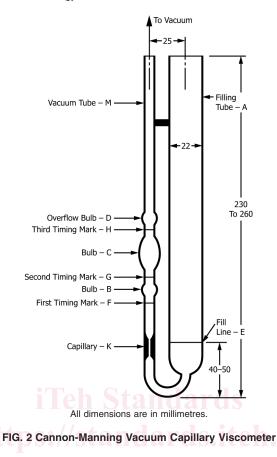
6.2.3 It is essential that liquid-in-glass thermometers be calibrated periodically using the technique given in<u>A</u> thermistor thermometer with sensor, which conforms to the requirements of Specification <u>E879</u>Test Method, calibrated <u>E77 (seeannually as Appendix X5)</u>.a single unit. The sensing element of the thermistor shall be completely immersed in the bath adjacent to the capillary tube.

NOTE 7-The use of the thermometric devices specified in 6.2.2 and 6.2.3 are understood to introduce bias in the precision in this method.

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 losses and heat input must be such that the temperature of the bath medium does not vary by more than  $\pm 0.03^{\circ}C$  [ $\pm 0.03^{\circ}C$  [ $\pm 0.05^{\circ}F$ ] over the length of the viscometer, or from viscometer to viscometer in the various bath positions.

6.4 Vacuum System—A vacuum system capable of maintaining a vacuum to within  $\pm 0.5 \text{ mm Hg}$  of the desired level up to and including 40.0 kPa [300 mm Hg]. The essential system is shown schematically in Fig. 1. Tubing of 6.35-mm [<sup>1</sup>/<sub>4</sub>-in.] inside diameter should be used, and all joints should be airtight so that when the system is closed, no loss of vacuum is indicated by the pressure gauge. A vacuum or aspirator pump is suitable for the vacuum source. The vacuum measuring system for this test method must be standardized at least once a year.

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6.5 *Timer*—A stop watch 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. Timing devices for this test method must be ealibrated standardized at least every 6twelve months.

6.5.1 *Electrical Timing Devices* may be used only on electrical circuits, the frequencies of which are controlled to an accuracy of 0.05 % or better.

6.5.1.1 Alternating currents, the frequencies of which are intermittently and not continuously controlled, as provided by some public power systems, can cause large errors, particularly over short timing intervals, when used to actuate electrical timing devices.

#### 7. Sample Preparations

7.1 Heat the sample with care to prevent local overheating until it has become sufficiently fluid to pour, occasionally stirring the sample to aid heat transfer and to <u>assureensure</u> uniformity.

7.2 Transfer a minimum of 20 mL into a suitable container and heat to  $135 \pm \frac{5.5 \text{ °C}}{5.5 \text{ °C}}$  [275  $\pm \frac{10 \text{ °F}}{10 \text{ °F}}$ , stirring occasionally to prevent local overheating and taking care to avoid the entrapment of air.

NOTE 8—If it is suspected that the sample may contain solid material, strain the melted sample into the container through a 300-µm [No. 50] sieve conforming to Specification E11.

Note 9—In the case of very viscous or modified asphalts, it may be necessary to heat in an oven at  $163 \pm \frac{5^{\circ}C}{5^{\circ}C}$  [325  $\pm \frac{10^{\circ}F}{10^{\circ}F}$  in order for the material to become sufficiently fluid to stir and pour.

#### 8. Procedure

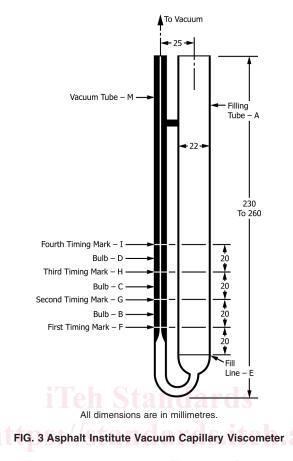
8.1 The specific details of operation vary somewhat for the various types of viscometers. See the detailed descriptions of viscometers in Appendix X1 – Appendix X3 for instructions for using the type of viscometer selected. In all cases, however, follow the general procedure described in 8.1.1 - 8.1.9.

8.1.1 Maintain the bath at the test temperature within  $\pm 0.03$  °C [0.05 °F]. within  $\pm 0.03$  °C [0.05 °F]. Apply the necessary corrections, if any, to all thermometer readings.

8.1.2 Select a clean, dry viscometer that will give a flow time greater than 60 s, and preheat to  $135 \pm \frac{5.5 \text{ °C}}{5.5 \text{ °C}}$  [275  $\pm \frac{10 \text{ °F}}{10 \text{ °F}}$ ].

8.1.3 Charge the viscometer by pouring the prepared sample to within  $\pm 2$  mm of fill line *E* (Fig. 2Figs. 2-4, Fig. 3, and Fig. 4).

## 



8.1.4 Place the charged viscometer in an oven or bath maintained at  $135 \pm \frac{5.5 \text{ °C}}{5.5 \text{ °C}}$  [275  $\pm \frac{10 \text{ °F}}{10 \text{ °F}}$ ] for a period of 10  $\pm 2 \text{ min}$ , to allow large air bubbles to escape.

8.1.5 Remove the viscometer from the oven or bath and, within 5 min, insert the viscometer in a holder, and position the viscometer vertically in the bath so that the <u>upper most uppermost timing mark</u> is at least 20 mm below the surface of the bath liquid.

8.1.6 Establish a 40.0  $\pm$  0.07 kPa [300  $\pm$  0.5 mm Hg] vacuum below atmospheric pressure in the vacuum system 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  $30 \pm 5$  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. Report the first flow time which exceeds 60 s between a pair of timing marks, noting the identification of the pair of timing marks.

8.1.9 Upon completion of the test, clean the viscometer thoroughly by several rinsings with an appropriate solvent completely miscible with the sample, followed by a completely volatile solvent. Dry the tube by passing a slow stream of filtered dry air through the capillary for 2 min, or until the last trace of solvent is removed. Alternatively, the viscometer may be cleaned in a glass-cleaning oven, at a temperature not to exceed  $500^{\circ}C$  [932°F], $500^{\circ}C$  [932 °F], followed by rinses with distilled or deionized water, residue-free acetone, and filtered dry air. Periodically, if deposits are observed, clean the instrument with a strong acid cleaning solution to remove organic deposits, rinse thoroughly with distilled or deionized water and residue-free acetone, and dry with filtered dry air.

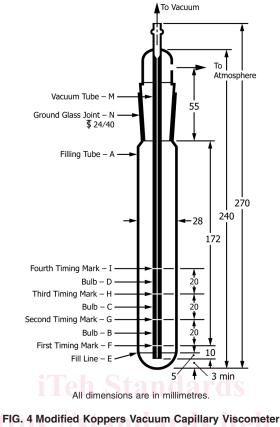
8.1.9.1 Chromic acid cleaning solution may be prepared by adding, with the usual precautions, 800 mL of concentrated sulphuric acid to a solution of 92 g of sodium dichromate in 458 mL of water. The use of similar commercially available sulphuric acid cleaning solutions is acceptable. Nonchromium-containing, strongly oxidizing acid cleaning solutions may be substituted so as to avoid the disposal problems of chromium-containing solutions.

8.1.9.2 Use of alkaline glass cleaning glass-cleaning solutions may result in a change of viscometer calibration, and is not recommended.

#### 9. Calculation

9.1 Select the calibration factor that corresponds to the pair of timing marks used for the determination, as prescribed in 8.1.8. Calculate and report the viscosity to three significant figures using the following equation:

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Viscosity,  $Pa \cdot s = (Kt)$ 

where:

K = selected calibration factor, Pa·s/s, and

t =flow time, s.

NOTE 10—If the viscometer constant or calibration factor  $(K_{cgs})$  is known in cgs units (Poise/s) calculate the calibration factor (Ksi)(Ksi) in SI units (pascal-seconds/second) as follows:

$$Ksi = (\text{Pa} \cdot s/s) = K_{cgs}/10 \text{ or } (P/s)/10$$
<sup>(2)</sup>

(1)

### 10. Report

10.1 Always report the test temperature and vacuum with the viscosity test result. For example, viscosity at 60°C [140°F] and 40.0 kPa [300 mm Hg] vacuum, in Pa-s.

Note 11-For example, viscosity at 60 °C [140 °F] and 40.0 kPa [300 mm Hg] vacuum, in Pa·s.

### 11. Precision and Bias

11.1 The following criteria (see Note 1) should be used for judging the acceptability of results (95 % probability):

11.1.1 *Repeatability*—Duplicate results by the same operator using the same viscometer should not be considered suspect unless they differ by more than 7 % of their mean.

11.1.2 *Reproducibility*—The results submitted by each of two laboratories should not be considered suspect unless the two results differ by more than 10 % of their mean.

NOTE 12—Supporting data and information for determining these estimates can be found in Manning, R. E., "Comments on Vacuum Viscometers for Measuring the Viscosity of Asphalt Cements," *Symposium on Fundamental Viscosity of Bituminous Materials*, ASTM STP 328, ASTM International, 1963, pp. 44–47.

11.2 *Bias*—The bias for this test method cannot be determined because no material with an accepted reference value is available.

#### 12. Keywords

12.1 asphalt; capillary; vacuum; viscometer; viscosity