Designation: D4957 − 18

Standard Test Method for Apparent Viscosity of Asphalt Emulsion Residues and Non-Newtonian Asphalts by Vacuum Capillary Viscometer[1]

This standard is issued under the fixed designation D4957; 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 Method D6997. 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 asphalts 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 60 °C, but is suitable for use at other temperatures. It is applicable over the range from 5 to 50 000 Pa·s.

NOTE 1—The precision for this test method is based on determinations made at 60 °C.

1.2 The values stated in SI units are to be regarded as the standard, except in reference to viscometer constant or calibration factor (K).

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 products. See the applicable product Material Safety Data Sheets (MSDS) for details and the EPA's website (www.epa.gov/mercury/faq.htm) for additional information. Users should be aware that selling mercury, mercury-containing products, or both, in your state may be prohibited by state law.

1.4 The text of this standard references notes and footnotes which 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, health, and environmental 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:*[2]

D2171 Test Method for Viscosity of Asphalts by Vacuum Capillary Viscometer
D3666 Specification for Minimum Requirements for Agencies Testing and Inspecting Road and Paving Materials
D6997 Test Method for Distillation of Emulsified Asphalt
E1 Specification for ASTM Liquid-in-Glass Thermometers
E77 Test Method for Inspection and Verification of Thermometers
E644 Test Methods for Testing Industrial Resistance Thermometers
E1137/E1137M Specification for Industrial Platinum Resistance Thermometers
E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

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 N/m^2 by the rate of shear in reciprocal seconds is called the pascal second (Pa·s). The English unit of viscosity is the poise (P) with dimensions of $0.1 N·s/m^2$ ($dynes/cm^2/s$), and is equivalent to 0.1 Pa·s.

[1] 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.

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[2] 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.

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 asphalts. 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 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.

NOTE 3—The quality of the results produced by this standard are dependent on the competence of the personnel performing the procedure and the capacity, 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 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 0.02 °C can be used, or any other thermometric device of equal accuracy. ASTM Kinematic Viscosity Thermometers 47C conforming to Specification E1 are suitable for the most commonly used temperature of 60 °C. See Test Method D2171, Table X5.1 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

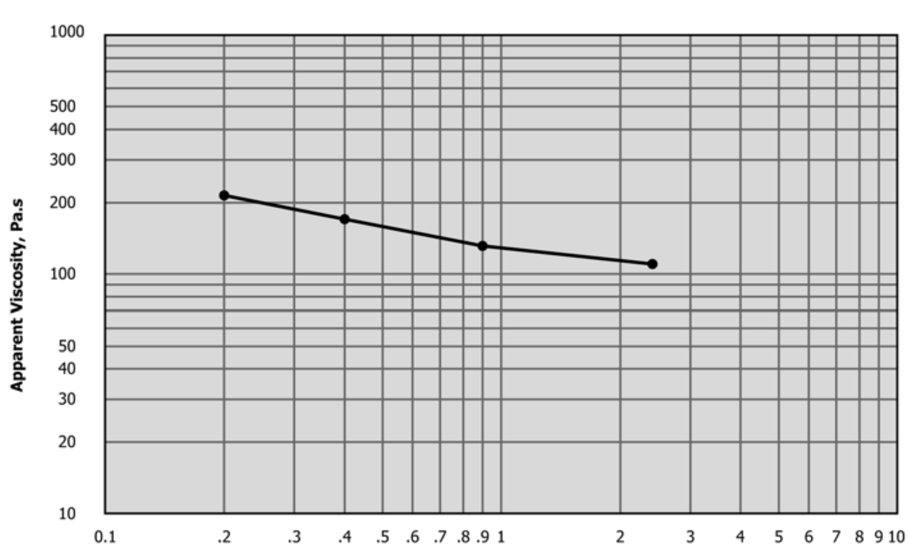

FIG. 1 Typical Log-Log Rheogram Plot with Example

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 E77 (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 0.03 °C over the length of the viscometer or from viscometer to viscometer in the various bath positions.

6.4 *Vacuum System*—A vacuum system[3] capable of maintaining a vacuum to within ±67 Pa of the desired level up to and including 40.0 kPa. The essential system is shown schematically in Fig. 2. Tubing of 6.35 mm 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 every six months.

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 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 202 ± 2 °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 260 °C, pour a suitable portion of the total residue into a 50-mL beaker and allow to cool to 180 ± 5 °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 195 ± 2 °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 4—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 5—In cases where the asphalt is not sufficiently fluid to pour at 180 ± 5 °C 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 ±0.03 °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

[3] The sole source of supply of the apparatus known to the committee at this time is Cannon Instrument Co., P.O. Box 16, State College, PA, 16801. 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.

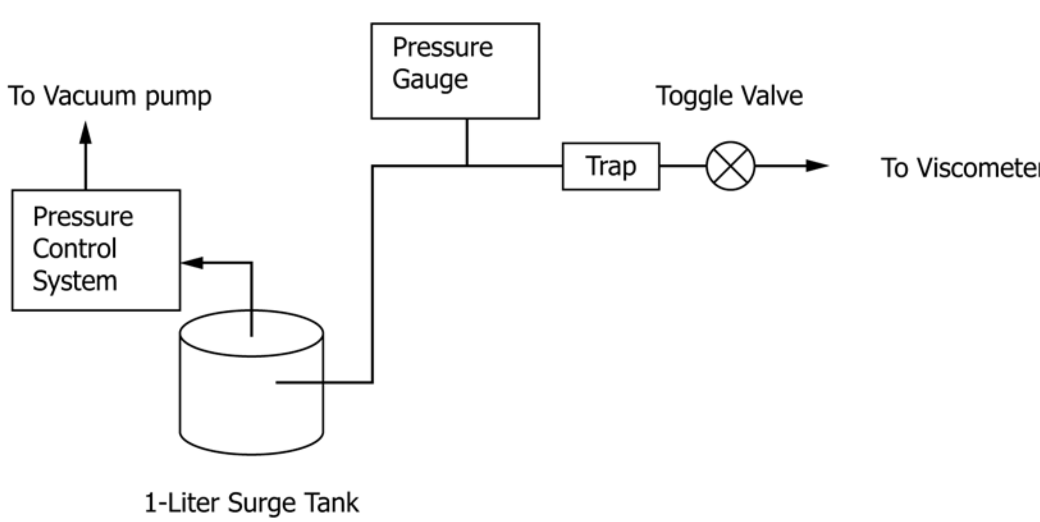

FIG. 2 Suggested Vacuum System for Vacuum Capillary Viscometers