Designation: D5018 – 18

Standard Test Method for Shear Viscosity of Coal-Tar and Petroleum Pitches[1]

This standard is issued under the fixed designation D5018; 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 covers the determination of the apparent shear viscosity of coal-tar and petroleum-based pitches having a Mettler softening point (SP) range of approximately 95 °C to 120 °C (see Test Method D3104).

1.2 This test method is applicable only for rotational viscometers.

1.3 Since this test method is based on theoretical grounds, strict adherence to details of the procedure is necessary to comply with the theoretical requirements.

1.4 The values stated in SI units are to be regarded as standard.

1.4.1 *Exception*—The values stated in conventional units (centipoise) are to be regarded as the standard for viscosity measurement only. The SI unit is the pascal second (Pa·s) and one millipascal second (mPa·s) = one centipoise (cP); centipoise is in cgs units.

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.* Specific hazard statements are given in Section 7.

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]

D3104 Test Method for Softening Point of Pitches (Mettler Softening Point Method)
D4296 Practice for Sampling Pitch
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E1953 Practice for Description of Thermal Analysis and Rheology Apparatus
E2975 Test Method for Calibration or Calibration Verification of Concentric Cylinder Rotational Viscometers

3. Summary of Test Method

3.1 The viscosity of a pitch, over the temperature range of about 40 °C to 100 °C above the SP of the material, is determined using a rotational viscometer.

3.2 The recommended specifications herein are for measuring the apparent shear viscosity of binder pitches via a concentric cylinder viscometer. Apparent shear viscosity is the ratio of shear stress to shear rate in a unidirectional simple shear flow field at steady state conditions. A concentric cylinder viscometer is useful for measuring the apparent shear viscosity, provided the sample temperature is adequately controlled, the "end-effects" are negligible, and the gap between rotor/cup is small and remains constant during the test. The extrapolated value of apparent shear viscosity at "zero" shear rate is called shear viscosity.

4. Significance and Use

4.1 This test method is useful as one element in establishing the uniformity of shipments.

4.2 Viscosity is also valuable for rheological characterization of binder pitches. Binder pitch imparts consistency to carbonaceous mixes and affects their resistance to deformation. Binder pitch viscosity is important for assessing mix consistency and for evaluating the ease of mix extrusion or molding into artifacts.

5. Apparatus

5.1 *Viscometer*—A rotational viscometer capable of measuring viscosity in the range of about 5 mPa·s (cP) to 15 000 mPa·s (cP); the viscometer should be equipped with the appropriate accessories to allow measurements up to about 230 °C. The essential instrumentation required providing the minimum rotational viscometer analytical capabilities include (see Practice E1953):

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricantsand is the direct responsibility of Subcommittee D02.05 on Properties of Fuels, Petroleum Coke and Carbon Material.

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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.

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

5.1.1 *Drive motor,* to apply a unidirectional rotational displacement to the specimen of 0.3 r/min to 60 r/min constant to ±1 %.

5.1.2 *Force sensor,* to measure the torque developed by the specimen to within ±1 %.

5.1.3 *Coupling shaft,* or other means to transmit the displacement from the motor to the specimen.

5.1.4 *Rotational element, spindle, or rotor,* of the shape shown in Fig. 1, to fix the specimen between the coupling shaft and a stationary position.

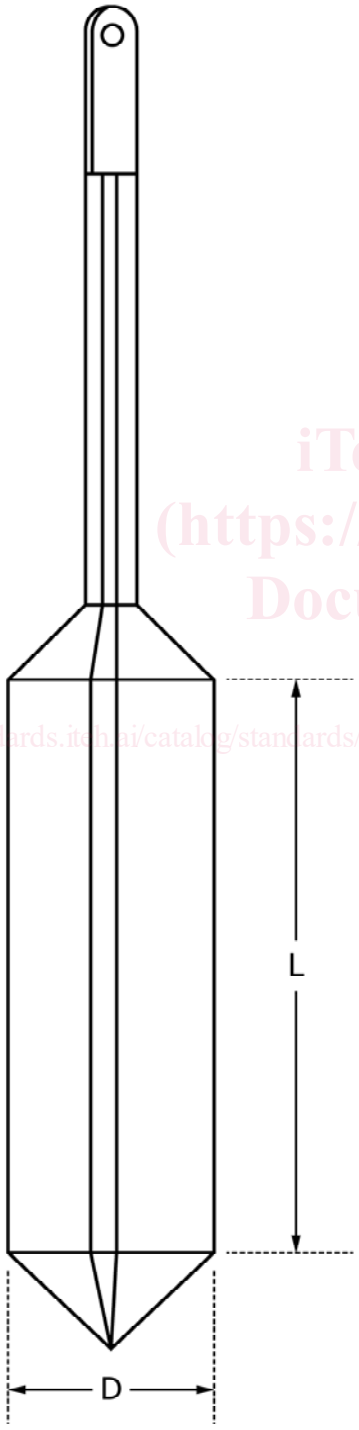

FIG. 1 Rotor Configuration

5.1.5 *Specimen container, chamber, or cup,* to contain the test specimen during testing.

NOTE 1—Rotor and cup are matched in size so that shear rate is fixed and known.

5.1.6 *Data collection device,* to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for rotational visocity are torque, rotational speed, temperature, and time.

5.1.7 Auxiliary instrumentation considered useful in conducting this test method includes:

5.1.7.1 *Data analysis capability,* to provide viscosity, stress, or other useful parameters derived from measured signals.

5.1.7.2 *Stand,* to support, level, and adjust the height of the drive motor, shaft, and rotor.

5.1.7.3 *Level,* to indicate the vertical plumb of the drive motor, shaft, and rotor.

5.2 *Sample Temperature Control System*—Any device capable of maintaining the sample test temperature within limits of ±1.0 °C while allowing viscosity measurements.

5.3 *Thermometer*—ASTM precision thermometer 2C, having a range of −5 °C to 300 °C.

5.4 *Hot Plate*[3]—Any hot plate with adjustable temperature control and surface temperature indication (to prevent sample overheating).

5.5 *Calibration Fluids*—A series of calibrated fluids that cover the viscosity range of approximately 100 mPa·s (cP) to 15 000 mPa·s (cP) at temperatures up to 150 °C.

NOTE 2—Calibration fluids are available from the rotational viscometer supplier.

6. Reagents and Materials

6.1 *Cleaning Solvent*—Any solvent capable of dissolving pitch, (suitable solvents are quinoline or creosote oils).

6.2 *Rinsing Solvents*—Toluene and acetone are used for final rinsing after initial cleaning.

7. Safety Hazards

7.1 Fumes of hot pitch or solvents, or both, should be removed from all working areas by means of proper hoods. The working area should be kept free of sparks and flames. Quinoline fumes should not be inhaled, and prolonged contact with skin should be avoided. Toluene is toxic and flammable.

8. Bulk Sampling

8.1 Take samples from shipments in accordance with Practice D4296. Samples shall be free of foreign substances. Thoroughly mix the sample immediately before removing a representative portion for the determination or for dehydration.

[3] The sole source of supply of the apparatus known to the committee at this time is the Hot plate Model 11-496-3 with 11-496-4 dial thermometer, available from Fisher Scientific, 585 Alpha Drive, Pittsburgh, PA 15238. 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.