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An American National Standard

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

This standard is issued under the fixed designation D 5018; 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.

ϵ^1 NOTE—Editorial corrections were made to Fig. 1 and in paragraph 14.2 in December 1999..

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 to 120°C.

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 conventional units (centipoise) are to be regarded as the standard. The SI unit is the pascal second (Pa·s) and one millipascal second (mPa·s) = one centipoise (cps); 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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*

D 4296 Practice for Sampling Pitch[2]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

3. Summary of Test Method

3.1 The viscosity of a pitch, over the temperature range of about 40 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 to 15 000 cps; the viscometer should be equipped with the appropriate accessories to allow measurements up to about 230°C. Two viscometers meeting these requirements are the LVT, LVF,[4] or equivalent, and the RV100, RV20,[5] or equivalent.

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. Examples are the Thermosel System[4] and the TP 24[5] with heater.

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

[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricantsand is the direct responsibility of Subcommittee D02.05.OFon Industrial Pitches.

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[2] *Annual Book of ASTM Standards*, Vol 05.02.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] Available from Brookfield Engineering Laboratories, Inc., 240 Cushing St., Stoughton, MA 02072.

[5] Available from Haake Buchler Instruments, Inc., 244 Saddle River Road, Saddle Brook, NJ 07662-6001.

5.4 *Hot Plate*[6]—Any hot plate with adjustable temperature control and surface temperature indication (to prevent sample overheating).

5.5 *Calibration Fluids*[7]—A series of calibrated fluids that cover the viscosity range of approximately 100 to 15 000 cps at temperatures up to 150°C.

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 D 4296. Samples shall be free of foreign substances. Thoroughly mix the sample immediately before removing a representative portion for the determination or for dehydration.

9. Sample Preparation

9.1 Ensure sample is dry; if there is visible moisture, dehydrate at 50°C in a forced-air or vacuum oven until dry, but no longer than 2 h. (Experience has shown that drying at temperatures in excess of 50°C increases the SP and viscosity of the pitch.)

9.2 Crush dry lumps of pitch to a size of 6 to 12 mm.

10. Charging Sample Cup

10.1 In a suitable container, or the sample cup, melt pitch with occasional stirring. Overheating of sample must be avoided as loss of volatiles may affect viscosity. (Maximum temperature should not exceed 50°C above SP. As a guide, the surface temperature of the hot plate surface should not exceed 200°C.)

10.2 Place rotor in cup and preheat both to approximately the first test temperature.

10.3 Remove rotor and transfer required amount of pitch to the level specified by the manufacturer.

10.4 Re-insert rotor, check that rotor is immersed to specified depth, and install insulating cover.

11. Viscometer Calibration

11.1 This step, required only occasionally, is designed to establish that the temperature and viscosity indicated by the appropriate measuring devices are in agreement with known standards.

11.2 Equipment is to be properly leveled and installed in accordance with manufacturer's instructions.

11.3 Use the ASTM thermometer (applying the appropriate stem correction) to correlate pitch temperature (in cup) to temperature controller/indicator. Test temperature is considered to be the actual pitch temperature and not the temperature indicated on the controller.

11.4 Calibrate viscometer using calibration fluids.

11.4.1 Use high- and low-temperature fluids; see 5.5.

11.4.2 Calibrate each rotor/cup combination and use them as a paired set.

12. Procedure for Viscosity Determination

12.1 The following general principles apply to all viscosity measurements determined by rotational methods:

12.1.1 Maximize rotor diameter.

12.1.2 Minimize gap width between rotor and cup.

12.1.3 Minimize end effects (use longest available rotor).

12.1.4 Prevent viscous heating (due to prolonged rotation of rotor at high RPM).

12.2 Select the proper rotor/cup combination that covers the anticipated viscosity range. A typical viscosity-temperature curve for various coal-tar pitches is given in Fig. 1, and a typical spindle number and RPM relationship for a LV series Brookfield[5] viscometer is given in Table 1. If there is overlap in rotor/cup selection, select the combination that best meets the criteria set forth in 12.1.1 to 12.1.4.

12.3 Determine viscosity from the lowest to the highest test temperatures.

12.4 Turn rotor at low RPM during heat-up (after sample is melted) and when changing test temperatures.

12.5 Stabilize temperature for a *minimum of 5 min but not more than 15 min* before measuring viscosity.

12.6 Determine viscosity following manufacturer's procedure. Ensure that the scale reading has stabilized before recording the value; only record values that are between 10 and 90 % of the maximum scale reading.

12.7 Increase temperature controller to next higher test temperature and repeat 12.4 through 12.7.

12.8 Record test temperature and corresponding scale readings and convert scale readings to viscosity using appropriate factors supplied by manufacturer or determined by calibration.

12.9 Pitch is generally regarded as a Newtonian liquid (the viscosity is independent of shear rate (rotational velocity or RPM)). To ensure that the pitch sample is in fact Newtonian, determine the viscosity at different RPM at a given temperature. If viscosities at different RPM are different (at same temperature), but are within 10 % of the average of the readings, report the average value. If the viscosities, at different RPM but the same temperature, differ by more than 10 % from the average, the material is non-Newtonian and the viscosity for each shear rate (RPM) should be reported.

12.10 Occasionally, the pitch volume in the cup can decrease when heated to higher temperatures due to loss of moisture or entrapped air. Consequently, the rotor will not be completely immersed in pitch and the viscosity at higher temperatures will be lower than the true value. Also, the viscosity at lower temperatures will be inaccurate due to foaming. To ensure that this did not occur, check the level in

[6] Hot plate Model 11-496-3 with 11-496-4 dial thermometer has been found suitable for this purpose. Available from Fisher Scientific, 585 Alpha Drive, Pittsburgh, PA 15238.

[7] Fluids available from Brookfield Engineering Laboratories; Cannon Instrument Co., P.O. Box 16, State College, PA 16804, have been found suitable for this purpose.