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Designation: D 4402 – 02

## Standard Test Method for Viscosity Determinations of Unfilled Asphalts Asphalt at Elevated Temperatures Using the Brookfield Thermosel Apparatus a Rotational Viscometer<sup>1</sup>

This standard is issued under the fixed designation D 4402; 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

<sup>ε1</sup> ~~Note~~ Editorial changes were made throughout in January 2000.

*This standard has been approved for use by agencies of the Department of Defense.*

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-8 D08 on Roofing, Waterproofing, and Bituminous Materials and is the direct responsibility of Subcommittee D08.03 on Surfacing and Bituminous Materials for Membrane Waterproofing and Builtup Roofing.

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### 1. Scope

1.1 This test method outlines a procedure for measuring the apparent viscosity of asphalt from 38 to 260°C (100 to 500°F) using a rotational viscometer and a temperature-controlled thermal chamber for maintaining the Brookfield Thermosel apparatus:

1.2 test temperature.

1.2 The values stated in SI units are to be regarded as the standard. The values given in centimetre gram second (cgs) units and inch-pound units are for information only.

1.3 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. See 7.6 10.6 for specific precautionary information.

### 2. Referenced Documents

2.1 ASTM Standards:

D 92 Test Method

E 220 Method for Flash and Fire Points Calibration of Thermocouples by Cleveland Open Cup Comparison Techniques<sup>2</sup>

### 3. Terminology

3.1 Definitions:

3.1.1 *apparent viscosity, n*—the ratio of shear stress to shear rate for a Newtonian or non-Newtonian liquid.

3.1.2 *filled asphalt, n*—an asphalt blend that contains finely dispersed insoluble mineral matter.

3.1.3 *Newtonian liquid, n*—a liquid for which the rate of shear is proportional to the shearing stress. The constant ratio of the shearing stress to the rate of shear is the viscosity of the liquid. The viscosity of a Newtonian liquid is therefore not dependent on its shear rate. If the ratio is not constant, the liquid is non-Newtonian. Many liquids exhibit both Newtonian and non-Newtonian behavior, depending on the shear rate or temperature, or both.

3.1.4 *shear rate, n*—the measure of the speed at which the intermediate layers of the liquid move with respect to each other. Its unit of measure is the reciprocal second ( $\text{sec}^{-1}$ ).

3.1.5 *shear stress, n*—the force per unit area required to produce the shearing action. Its SI unit of measurement is the pascal, and its cgs unit of measurement is dynes/cm<sup>2</sup>.

3.1.6 *viscosity, n*—the ratio between the applied shear stress and the rate of shear is called the coefficient of viscosity. This coefficient is a measure of the resistance to flow of the liquid. It is commonly called the viscosity. The SI unit of viscosity is the pascal second (Pa·s). The centimetre gram second (cgs) unit of viscosity is the poise (dyne·s/cm<sup>2</sup>) and is equivalent to 0.1 Pa·s. Frequently, centipoise (cP)—equal to one millipascal second (mPa·s)—is used as the viscosity unit.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *apparatus-measuring geometry, n*—the part of the equipment that is immersed in the asphalt sample, the dimensions of which are used, in conjunction with the rotational resisting torque, to calculate the apparent viscosity. This geometry may be

<sup>2</sup> Annual Book of ASTM Standards, Vol 05.01: 14.03.

referred to by the equipment manufacturer as a spindle, bob, inner concentric cylinder, vane, and so forth.

#### 4. Summary of Test Method

##### 4.1 ~~The Brookfield Thermosel Viscometer~~

4.1 A rotational viscometer, as described in this ~~procedure can be~~ test method, is used to measure the apparent viscosity of asphalt at elevated temperatures. The torque on a ~~spindle the apparatus-measuring geometry~~, rotating in a ~~special~~ thermostatically controlled sample holder containing a ~~small~~ sample of asphalt, is used to measure the relative resistance to rotation. ~~A factor is applied to the~~ The torque ~~dial reading~~ and speed are used to ~~yield~~ determine the viscosity of the asphalt in pascal seconds, millipascal seconds, or centipoise.

#### 5. Significance and Use

5.1 This test method ~~can be~~ is used to measure the apparent viscosity of asphalts at handling, mixing, or application temperatures.

5.2 Some asphalts may exhibit non-Newtonian behavior under the conditions of this test method, or at temperatures within the range of this test method. Since non-Newtonian viscosity values are not ~~unique material properties~~ absolute properties, but reflect the behavior of the fluid ~~and within the particular~~ measurement system, it should be recognized that measurements made by this test method may not always predict field performance under the conditions of use.

5.3 Comparisons between non-Newtonian viscosity values should be made only for measurements made with similar viscometers ~~under similar conditions of shearing stress~~ temperature, shear rate, and shear history.

#### 6. Apparatus

6.1 ~~Brookfield Thermosel High Temperature Viscosity Measurement System Using a Standard Brookfield Synchron-Ectric~~ Rotational Viscometer—~~De~~, capable of measuring the torque required to rotate the selected apparatus-measuring geometry at a selected constant speed while submerged in asphalt at constant desired test temperature, and with the capability to convert the torque measurement to viscosity ~~range Model LV, RV, HA, in pascal seconds, millipascal seconds, or HB-series~~ centipoise. This calculation may need to be used.<sup>3</sup> done manually for some instruments.

6.2 ~~Apparatus-measuring geometry~~, of various shapes and sizes, for Brookfield Thermosel Viscometer. measurement of various viscosities of asphalt.

##### 6.3 ~~Thermosel System:~~

6.3.1 ~~Thermo Container and Sample~~ Temperature-Controlled Thermal Chamber:

6.3.2 ~~SCR Heater~~, for maintaining the sample of asphalt at the test temperature.

6.4 ~~Sample Chambers~~, reusable or disposable.

6.5 ~~Temperature Controller~~, capable of maintaining the specimen temperatures to  $\pm 1.0^{\circ}\text{C}$  ( $\pm 2.0^{\circ}\text{F}$ ) for test temperatures between 38 and ~~Probe:~~

6.3.3 ~~Graph Plotting Equipment:~~ 260°C (100 to 500°F).

6.6 ~~Balance~~, readable to 0.1 g, for determining the mass of asphalt sample.

6.7 ~~Calibration Device~~, in accordance with Method E 220 for calibrating the temperature controller.

#### 7. Procedure for the Brookfield Thermosel

7.1 ~~Read~~ Reagents and understand the information in the instrument manufacturer's operating instructions before proceeding.

7.2 ~~Turn on~~ Thermosel power.

7.3 Set the proportional temperature controller to desired test temperature.

7.4 Refer to the operating instructions Materials

7.1 ~~Solvents for calibration of the controller:~~

7.5 Wait 1.5 h (or until equilibrium temperature is obtained) with the selected spindle in the chamber (check control lamp).

7.6 Remove cleaning sample holder and add the volume of sample specified for the spindle to be used. Exercise caution to avoid sample overheating and to avoid ignition of sample with low flash point. Calculate the mass required from specific gravity or density data for the sample. Approximately 8 to 10 mL will be required.

7.7 Do not overfill the sample container. The sample volume is critical to meet the system calibration standard. Thoroughly stir filled asphalt coatings to obtain a representative sample.

7.8 The liquid level should intersect the spindle shaft at a point approximately 3.2 mm (1/8 in.) above the upper "conical body"—"spindle shaft" interface.

7.9 Using the extracting tool, put the loaded chamber back into the thermo container.

7.10 Lower the viscometer and align the thermo container.

7.11 Insert the selected spindle into the liquid in the chamber, apparatus-measuring geometry, and couple it to the viscometer. Proper spindle selection may require testing with more than one spindle.

7.12 Allow the asphalt to come to the equilibrium temperature (about 15 min)

7.13 Start Brookfield models RV, HA, HB viscometer at 20 rpm, LV model at 12 rpm. Observe the meter reading. If it is between 2 and 98 units, proceed with the test.

- 7.14 Record three readings 60 s apart at each test temperature.
- 7.15 Follow the procedure for each test temperature required.
- 7.16 If readings are above 98 units at the lowest test temperature, decrease the spindle rpm setting and continue with the test.
- 7.17 If the reading is above 98, use the next smaller spindle and repeat the procedure using the sample volume specified, see 7.6.
- 7.18 Multiply the viscosity factor by the Brookfield reading to obtain viscosity in centipoise.
- 7.19 Do not change the speed (rpm setting) during a viscosity measurement, as this will change the shear rate. accessories.

## 8. Report

- 8.1 Report test temperature, spindle number, and speed with results. For example, viscosity at 60°C = 105 mPa with spindle number.
- 8.2 Plot viscosity value versus actual test temperature for each Preparation of the three or more test temperatures Apparatus
- 8.1 The rotational viscometer and thermal chamber heater shall be leveled and prepawred as recommended by the instrument manufacturer.

## 9. Calibration and Standardization

- 9.1 The viscometer shall be zeroed before use, or as needed, or both, according to the manufacturer's instructions.
- 9.2 The accuracy of the viscometer shall be checked at least annually using a certified reference fluid of known viscosity at various temperatures, using the method described by the supplier of the reference fluid. The reference fluid shall be certified to be Newtonian in behavior over the full range of expected test temperatures and shear rates. The reference fluid shall be certified at a temperature within 50°C (90°F) of the temperature(s) to be used during the test. The viscosity measured shall be within  $\pm 2\%$  of the certified value, or the viscometer requires recalibration.
- 9.3 The accuracy of the temperature reading and the temperature stability of the temperature controller are to be checked at least every six months by placing an asphalt sample or high flash point oil in the test chamber, and equilibrating to a temperature within 50°C (90°F) of the temperature(s) to be used during the test. The sample temperature shall then be measured to within  $\pm 0.1^\circ\text{C}$  ( $\pm 0.2^\circ\text{F}$ ) by using a NIST traceable measuring device, as described in Method E 220. If any temperature differential is indicated, the set point of the temperature controller shall be offset accordingly.

## 10. Procedure

- 10.1 Follow the manufacturer's instructions for the operation of the instrument.
- 10.2 Allow the instrument electronics to warm up for at least five minutes before conducting any calibrations or analyses.
- 10.3 Set the temperature controller to the desired test temperature, taking into account any offset determined in 9.3.
- 10.4 Select an apparatus-measuring geometry that will develop a resisting torque between 10 and 98 % of the instrument capacity at the selected speed. Generally, measurements will be more accurate at higher torque readings.
- 10.5 Preferably, preheat the sample chamber and the selected apparatus-measuring geometry until temperature equilibrium has been obtained for at least 15 min. If filled asphalts are being measured, this step is mandatory.
- 10.6 Add the volume of sample specified by the manufacturer for the apparatus-measuring geometry to be used to the sample chamber. A convenient way for measuring the volume is by weighing out the amount calculated from approximate density data for the sample and then returning the sample chamber to the temperature controlled chamber heater. Thoroughly stir filled asphalts to obtain a representative sample before weighing.

NOTE 1—Exercise caution to avoid sample overheating, and to avoid the ignition of samples with low flash points.

- 10.7 Do not overfill the sample chamber, but ensure that the measuring portion of the apparatus-measuring geometry will be completely immersed. Follow the manufacturer's instructions. The sample volume is critical to meet the system calibration standard.
- 10.8 Insert the selected preheated apparatus-measuring geometry into the liquid in the chamber, and couple it to the viscometer, following the manufacturer's instructions for proper alignment.
- 10.9 Bring the asphalt sample to the desired temperature within 30 min and allow it to equilibrate at the desired test temperature for a minimum of 10 min before beginning the measurement. In the case of filled asphalts, start the motor rotation immediately.
- 10.10 Start the motor rotation of the viscometer at a speed that will develop a resisting torque that is between 10 and 98 % of the full-scale instrument capacity. Maintain this speed and allow the sample to equilibrate for an additional 5 min. Temperature should not deviate more than  $\pm 1.0^\circ\text{C}$  ( $\pm 2.0^\circ\text{F}$ ) during this conditioning period.
- 10.11 Measure either the viscosity or the torque at 1-min intervals for a total of three minutes. The instrument may perform this measurement automatically.
- 10.12 Repeat steps 10.9-10.11 for each test temperature required. If filled asphalts are being measured, a new, freshly stirred sample will be required for each test temperature.
- 10.13 If torque readings are above 98 % of the instrument capacity at the lowest test temperature, decrease the speed of rotation of the apparatus-measuring geometry and continue with the test, or repeat steps 10.5-10.11 with a smaller diameter geometry and the appropriate volume of sample.

10.14 If the torque reading is below 10 % of the instrument capacity at the highest test temperature, increase the speed of rotation of the apparatus geometry, or repeat steps 10.5-10.11 with a larger diameter geometry and the appropriate volume of sample.

10.15 If the instrument does not read out directly in viscosity units, multiply the torque readings by the appropriate factor to obtain the viscosity values.

## **11. Calculation**

11.1 If the instrument does not automatically average three readings, then calculate the result as the arithmetic average of the three readings taken at 1-min intervals, rounded to three significant figures. If the rotational viscometer has a digital output displaying viscosity in centipoise (cP), multiply by 0.001 to obtain the viscosity in pascal seconds (Pa·s). For instruments that offer automation, the results of a 3-min integration shall be acceptable.

## **12. Report**

12.1 Report test temperature, apparatus-measuring geometry type and size, torque in mNm or percent of instrument capacity, and speed in  $\text{sec}^{-1}$  or r/min with viscosity results in pascal seconds (Pa·s), millipascal seconds (mPa·s), or centipoise (cP). For example, Viscosity at  $135^{\circ}\text{C} = 0.455 \text{ Pa}\cdot\text{s}$  with Bohlin 25 mm bob, 8.3 mNm of torque at  $10 \text{ sec}^{-1}$  or Viscosity at  $400^{\circ}\text{F} = 240 \text{ cP}$  with Brookfield spindle number 31, 48 % torque at 60 r/min.

## **13. Precision and Bias**

913.1 The following criteria shall be used for judging the acceptability of any result (95 % confidence level).

913.1.1 *Repeatability*—Duplicate values by the same operator shall not be considered suspect unless they differ by more than 3.5 %.

913.1.2 *Reproducibility*—The values reported by each of two laboratories, representing the arithmetic average of duplicate determinations, shall not be considered suspect unless they differ by more than 14.5 %.

**NOTE 2**—Precision data was developed using unfilled asphalt, and the repeatability and reproducibility values do not apply to filled asphalt. Round robin testing is currently in progress to develop precision data for filled asphalt. In addition, since this procedure has been revised since the original precision statement was developed, the repeatability and reproducibility variability of unfilled asphalt will also be reevaluated before the next reapproval date.

913.2 *Bias*—No information can be presented on the bias of this test method for measuring apparent viscosity because no reference material having an accepted reference value is available.

## **104. Keywords**

~~10.1 asphalt; Brookfield; Newtonian Liquid; shear stress;~~

14.1 asphalt; rotational viscometer; viscosity

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