Designation: D4402/D4402M – 15

Standard Test Method for Viscosity Determination of Asphalt at Elevated Temperatures Using a Rotational Viscometer[1]

This standard is issued under the fixed designation D4402/D4402M; 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.

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

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 test temperature.

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 with the standard.

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 10.6 for specific precautionary information.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E644 Test Methods for Testing Industrial Resistance Thermometers

E1137 Specification for Industrial Platinum Resistance Thermometers

E2975 Test Method for Calibration of Concentric Cylinder Rotational Viscometers

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 (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^2.

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. 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^2) 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 referred to by the equipment manufacturer as a spindle, bob, inner concentric cylinder, vane, and so forth.

4. Summary of Test Method

4.1 A rotational viscometer, as described in this test method, is used to measure the apparent viscosity of asphalt at elevated temperatures. The torque on the apparatus-measuring geometry, rotating in a thermostatically controlled sample holder containing a sample of asphalt, is used to measure the

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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.

relative resistance to rotation. The torque and speed are used to determine the viscosity of the asphalt in pascal seconds, millipascal seconds, or centipoise.

5. Significance and Use

5.1 This test method 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 absolute properties, but reflect the behavior of the fluid 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 conditions of temperature, shear rate, and shear history.

6. Apparatus

6.1 *Rotational Viscometer,* 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 in pascal seconds, millipascal seconds, or centipoise. This calculation may need to be done manually for some instruments.

6.2 *Apparatus-measuring geometry*, of various shapes and sizes, for measurement of various viscosities of asphalt.

6.3 *Temperature-Controlled Thermal Chamber 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 ±1.0°C [±2.0°F] for test temperatures between 38 and 150°C [100 to 300°F] and to ±2.0°C [±4.0°F] for test temperatures between 150 and 260°C [300 to 500°F].

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

6.7 *Platinum Resistance Thermometer (PRT),* with a probe which conforms to the requirements of Specification E1137, for measuring the temperature of the thermal chamber. The PRT shall have a 3- or 4-wire connection configuration and overall sheath length shall be at least 50 mm [2 in.] greater than the immersion depth. Calibrate the PRT system (probe and readout) in accordance with Test Methods E644.

7. Reagents and Materials

7.1 Solvents for cleaning sample chamber, apparatus-measuring geometry, and accessories.

8. Preparation of Apparatus

8.1 The rotational viscometer and thermal chamber heater shall be leveled and prepared 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 procedure described in Test Method E2975. 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 ±2 % of the certified value, or else a calibration constant (viscosity of the calibration fluid/viscosity indicated by the apparatus) must be determined and applied.

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 ±0.1°C [±0.2°F] by using a NIST traceable measuring device, as described in Test Methods E644. 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 ±1.0°C [±2.0°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. If required, multiply the average reading by the calibration constant determined in 9.2.

12. Report

12.1 Report test temperature, apparatus-measuring geometry type and size, torque in mNm or percent of instrument capacity, and speed in 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°C = 0.455 Pa·s with Bohlin 25 mm bob, 8.3 mNm of torque at 10 sec^{-1} or Viscosity at 400°F = 240 cP with Brookfield spindle number 31, 48 % torque at 60 r/min.

13. Precision and Bias

13.1 *Unfilled Roofing Asphalt*—The following criteria shall be used for judging the acceptability of any result (95 % confidence level).

13.1.1 *Single-Operator Precision (Repeatability)*—Duplicate values by the same operator using the same test equipment, in the shortest practical period of time shall be considered not equivalent if the difference in the two results, expressed as a percent of their mean, exceeds 3.5 %.

13.1.2 *Multilaboratory Precision (Reproducibility)*—The values reported by each of two laboratories, representing the arithmetic average of duplicate determinations, shall be considered not equivalent if they differ by more than 14.5 %.

13.2 *Filled Roofing Asphalt*—An interlaboratory study was conducted in 2004 comparing three filled roofing asphalts from three different suppliers, tested in triplicate at 205°C [400°F] by nine different laboratories. The data was used to calculate precision estimates for filled asphalt. The following criteria shall be used for judging the acceptability of any result (95 % confidence level).

13.2.1 The single-operator precision (repeatability) standard deviation has been determined to be 21.0 %. Therefore, two results obtained in the same laboratory, by the same operator using the same equipment, in the shortest practical period of time, should be considered not equivalent if the difference in the two results, expressed as a percent of their mean, exceeds 59.4 %.

13.2.2 The multilaboratory precision (reproducibility) standard deviation has been determined to be 33.2 %. Therefore, two results submitted by two different operators testing the same material in different laboratories shall be considered not equivalent if the difference in the two results, expressed as a percent of their mean, exceeds 94.0 %.

13.3 *Unfilled Paving Asphalt*—The precision estimates given are based on the analysis of test results from eight pairs of AMRL proficiency samples. The data analyzed consisted of results from 142 to 202 laboratories for each of the eight pairs of samples. The details of this analysis are in NCHRP Final Report, NCHRP Project No. 9-26, Phase 3. The following criteria shall be used for judging the acceptability of any result (95 % confidence level).

13.3.1 The single-operator precision (repeatability) coefficient of variation (1s %) has been determined to be 1.2 %. Therefore, two results obtained in the same laboratory, by the same operator using the same equipment, in the shortest practical period of time, should be considered not equivalent if the difference in the two results, expressed as a percent of their mean, exceeds 3.5 % (d2s %).

13.3.2 The multilaboratory precision (reproducibility) coefficient of variation (1s %) has been determined to be 4.3 %. Therefore, two results submitted by two different operators testing the same material in different laboratories shall be considered not equivalent if the difference in the two results, expressed as a percent of their mean, exceeds 12.1 % (d2s %).

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

14. Keywords

14.1 asphalts; rotational viscometers; viscosities