

Standard Test Method for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer¹

This standard is issued under the fixed designation D7175; 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 dynamic shear modulus and phase angle of asphalt binders when tested in dynamic (oscillatory) shear using parallel plate geometry. It is applicable to asphalt binders having dynamic shear modulus values in the range from 100 Pa to 10 MPa. This range in modulus is typically obtained between 4 and 88°C at 10 rad/s. This test method is intended for determining the linear viscoelastic properties of asphalt binders as required for specification testing and is not intended as a comprehensive procedure for the full characterization of the viscoelastic properties of asphalt binders.

1.2 This standard is appropriate for unaged materials, material aged in accordance with Test Method D2872, material aged in accordance with Practice D6521, or material aged in accordance with both Test Method D2872 and Practice D6521. This procedure is limited to asphalt binders that contain particles with largest dimension less than 250 μ m.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 This standard may involve hazardous materials, operations, and equipment. 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.

2. Referenced Documents

2.1 ASTM Standards:²

C670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials **D140** Practice for Sampling Bituminous Materials

- D2170 Test Method for Kinematic Viscosity of Asphalts (Bitumens)
- D2171 Test Method for Viscosity of Asphalts by Vacuum Capillary Viscometer
- D2872 Test Method for Effect of Heat and Air on a Moving Film of Asphalt (Rolling Thin-Film Oven Test)
- D6373 Specification for Performance Graded Asphalt Binder
- D6521 Practice for Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel (PAV)
- E77 Test Method for Inspection and Verification of Thermometers
- **E563** Practice for Preparation and Use of an Ice-Point Bath as a Reference Temperature
- E644 Test Methods for Testing Industrial Resistance Thermometers
- E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory
- 2.2 AASHTO Standards:³
- M320 Standard Specification for Performance-Graded Asphalt Binder
- R29 Practice for Grading or Verifying the Performance Grade of an Asphalt Binder
- T315 Standard Test Method for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer
- 2.3 Deutsche Industrie Norm (DIN) Standard:⁴
- 43760 Standard for Calibration of Thermocouples

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *annealing*, n—the process of removing the effects of steric hardening by heating the binder until it is sufficiently fluid so that it can be easily poured.

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 Available from American Association of State Highway and Transportation Officials (AASHTO), 444 N. Capitol St., NW, Suite 249, Washington, DC 20001, http://www.transportation.org.

⁴ Available from Beuth Verlag GmbH (DIN-- DIN Deutsches Institut fur Normung e.V.), Burggrafenstrasse 6, 10787, Berlin, Germany, http://www.en.din.de.

3.1.2 *asphalt binder*, *n*—an asphalt-based cement that is produced from petroleum residue either with or without the addition of non-particulate modifiers.

3.1.3 *complex shear modulus (G*), n*—ratio calculated by dividing the absolute value of the peak-to-peak shear stress, τ , by the absolute value of the peak-to-peak shear strain, γ .

3.1.4 *dummy test specimen, n*—a specimen formed between the DSR test plates from asphalt binder or other polymer for the purpose of determining the temperature in the asphalt binder between the plates.

3.1.4.1 *Discussion*—The dummy test specimen is not used to measure the rheological properties of asphalt binder but is used solely to determine temperature corrections.

3.1.5 *linear viscoelastic, adj—within context of this test method,* refers to a region of behavior in which the dynamic shear modulus is independent of shear stress or strain.

3.1.6 *steric hardening*, *n*—refers to time-dependent associations that occur between asphalt binder molecules during storage at ambient temperature. The effect of molecular association or steric hardening on the dynamic shear modulus is asphalt specific and may be apparent even after a few hours of storage.

3.1.7 oscillatory shear, n—refers to a type of loading in which a shear stress or shear strain is applied to a test sample in an oscillatory manner such that the shear stress or strain varies in amplitude about zero in a sinusoidal manner.

3.1.8 *parallel plate geometry, n*—refers to a testing geometry in which the test specimen is sandwiched between two rigid parallel plates and subjected to shear.

3.1.9 *phase angle* (δ), *n*—the angle in degrees between a sinusoidally applied strain and the resultant sinusoidal stress in a controlled-strain testing mode, or between the applied stress and the resultant strain in a controlled-stress testing mode.

3.1.10 portable temperature measuring device, n—refers to an electronic device that is separate from the dynamic shear rheometer and that consists of a detector (probe containing a thermocouple or resistive element), associated electronic circuitry, and readout system.

3.1.11 *reference temperature measuring device, n*—refers to a NIST-traceable liquid-in-glass or electronic thermometer that is used as a laboratory standard.

3.1.12 *thermal equilibrium*, *n*—condition where the temperature of the test specimen mounted between the test plates is constant with time.

4. Summary of Test Method

4.1 This standard contains the procedure used to measure the complex shear modulus (G*) and phase angle (δ) of asphalt binders using dynamic shear rheometer and parallel plate geometry.

4.2 The standard is suitable for use when the dynamic shear modulus varies between 100 Pa and 10 MPa. This range in modulus is typically obtained between 4 and 88°C, depending upon the grade, test temperature, and conditioning (aging) of the asphalt binder.

4.3 Test specimens, nominally 25 mm in diameter by 1 mm thick or 8 mm in diameter by 2 mm thick, are formed between parallel metal plates.

4.4 During testing, one of the parallel plates is oscillated with respect to the other at pre-selected frequencies and angular deflection (or torque) amplitudes. The required amplitude depends upon the value of the complex shear modulus of the asphalt binder being tested. The required amplitudes have been selected so that, for most asphalt binders, the testing specified in this standard is within the region of linear behavior.

4.5 The test specimen is maintained at the test temperature ± 0.1 °C by enclosing the upper and lower plates in a thermally controlled environment or test chamber.

4.6 Oscillatory loading frequencies using this standard can range from 1 to 160 rad/s. Specification testing is performed at a test frequency of 10 rad/s. The complex modulus (G*) and phase angle (δ) are calculated automatically as part of the operation of the rheometer using proprietary computer software supplied by the instrument manufacturer.

5. Significance and Use

5.1 The test temperature for this test is related to the temperature experienced by the pavement in the geographical area for which the asphalt binder is intended to be used.

5.2 The complex shear modulus is an indicator of the stiffness or resistance of asphalt binder to deformation under load. The complex shear modulus and the phase angle define the resistance to shear deformation of the asphalt binder in the linear viscoelastic region. The complex modulus and the phase angle are used to calculate performance-related criteria in accordance with Specification D6373 or AASHTO Standard M320.

6. Interferences

6.1 Particulate material in the asphalt binder is limited to particles with longest dimensions less than 250 μ m. Particles with dimensions greater than 250 μ m approach the dimensions of the gap (1000 μ m). In order to accurately characterize a two-phase material containing particulate material it is well accepted that the thickness of the test specimen must be at least four times the maximum particle size.

6.1.1 The calculation of the complex modulus from the data obtained from the DSR is highly dependent upon an accurate measurement of the diameter of the test specimen. In the procedure, the diameter of the test specimen is assumed equal to the diameter of the test plates. This assumption is valid only if the test sample is properly trimmed.

6.1.2 The physical properties of asphalt binders are very sensitive to test temperature and thermal history. Thermal history is the number of times asphalt binder sample has been heated prior to testing. Controlling the test temperature to ± 0.1 °C and limiting the number of times the asphalt sample is heated prior to testing (only one heating is recommended) is essential in order to obtain repeatable test results within a laboratory as well as to reproduce results between laboratories.

7. Apparatus

7.1 Dynamic Shear Rheometer (DSR) Test System—A dynamic shear rheometer test system consisting of parallel metal plates, a means for controlling the temperature of the test specimen, a loading device, and a control and data acquisition system. The manufacturer of the device shall provide a certificate certifying that the frequency, deflection angle, and torque are controlled, measured, or both, with accuracy of 1 % or less in the range of this measurement.

7.1.1 *Test Plates*—Metal plates cylindrical in shape, formed from steel or aluminum, with smooth ground surfaces are required. Two plates 8.00 ± 0.02 mm in diameter and two plates 25.00 ± 0.05 mm in diameter are required. The test plates shall have a minimum thickness or raised portion of 1.5 mm to allow sufficient clearance for trimming the specimen. The plates shall be formed as an integral part of the test fixtures that are used to mount the plates in the DSR as shown in Fig. 1.

Note 1—The upper and lower plates should be concentric with each other. At the present there is no suitable procedure for the user to check the concentricity except to visually observe whether or not the upper and lower plates are centered with respect to each other. The moveable plate should rotate without any observable horizontal or vertical wobble. This may be checked visually or with a dial gage held in contact with the edge of the moveable plate while it is being rotated.

7.1.2 Environmental Chamber—A chamber for controlling the temperature of the test specimen. The medium for heating and cooling the specimen in the environmental chamber shall not affect asphalt binder properties. The temperature in the chamber may be controlled by the circulation of fluid conditioned gas, nitrogen or water is acceptable—or by a suitable arrangement of actively temperature controlled heating elements (for example, solid state Peltier elements) surrounding the sample. When laboratory air is used in a forced air oven, a suitable dryer must be included to prevent condensation of moisture on the test plates. The environmental chamber and the temperature controller shall control the temperature of the test specimen mounted between the test plates, including any thermal gradients within the test specimen, at the test

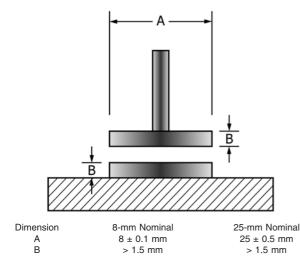


FIG. 1 Plate Dimensions

temperature $\pm 0.1^{\circ}$ C. The chamber or the water in the chamber shall completely enclose the top and the bottom plates to minimize thermal gradients within the fixtures and test specimen.

Note 2—A circulating bath unit, separate from the DSR that pumps the water through the test chamber may be required if a fluid medium is used.

7.1.2.1 *Temperature Controller*—A temperature controller capable of maintaining the temperature of the test specimen at the test temperature for the entire range of test temperatures.

7.1.3 Internal DSR Temperature Measurement Device—A platinum resistance temperature measurement device (PRT) mounted within the environmental chamber as an integral part of the DSR and in close proximity to the fixed plate, with a range of 4 to 88°C, and with a resolution of 0.1°C. This temperature measurement device shall be used to control the temperature of the test specimen between the plates and shall provide a continuous readout of temperature during the mounting, conditioning, and testing of the specimen.

7.1.4 Loading Device—The loading device shall be capable of applying a sinusoidal oscillatory load to the specimen at a frequency of 10.0 ± 0.1 rad/s. If frequencies other than 10 rad/s are used, the frequency shall be accurate to 1 %. The loading device shall be capable of providing either a stress controlled or strain controlled load within a range of stress or strain necessary to make the measurements described in this test method.

7.1.5 *Data Acquisition System*—The data acquisition system shall provide a record of temperature, frequency, deflection angle, and torque. The manufacturer of the rheometer shall provide a certificate certifying that the frequency, deflection angle, and torque are reported with an accuracy of at least 1 %.

7.2 Specimen Mold (optional)—The overall dimensions of the silicone rubber mold for forming asphalt binder test specimens may vary but the overall thickness shall be at least 5 mm thick.

Note 3—The following dimensions have been found suitable: For a 25-mm test plate with a 1-mm gap a mold cavity with a concave bottom with an approximate diameter of 18 mm and a depth of at least 2.0 mm and for an 8-mm test plate with a 2-mm gap a mold cavity with a concave bottom with an approximate diameter of 8 mm and a depth of at least 2.5 mm.

7.3 *Trimming Tool*—A tool suitable for trimming excess binder from the periphery of the test specimen to produce a smooth face on the test specimen that is parallel and coincident with the outer diameter of the upper and lower plates.

7.4 *Reference Temperature Measurement Device*—Either a NIST-traceable liquid-in-glass thermometer(s) (see 7.4.1) or NIST-traceable digital electronic thermometer (see 7.4.2) shall be maintained in the laboratory as a temperature standard.

7.4.1 *Liquid-in-Glass* Thermometer—NIST-traceable liquid-in-glass thermometer(s) with a range between 0 to 88°C and with subdivisions of 0.1°C. The thermometer(s) shall be partial immersion thermometers with an ice point. The liquid-in-glass thermometers shall be verified at least once a year in accordance with test method Test Method E77 and Practice E563.

Note 4-An Optical Viewing Device is recommended as an optional