



Designation: D7175 – 23

# Standard Test Method for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer<sup>1</sup>

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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the determination of the complex shear modulus and phase angle of asphalt binders when tested in dynamic (oscillatory) shear using parallel plate geometry.

1.2 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 binder.

1.3 This standard is appropriate for unaged asphalt binder, conditioned asphalt binder, and asphalt binder recovered from either asphalt mixtures or asphalt emulsions. To keep the language in this standard precise, the term “asphalt binder” is used to refer to the material being tested.

1.4 This procedure is limited to asphalt binders that contain particles with largest dimension less than 250  $\mu\text{m}$ .

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

1.6 **Warning**—Mercury has been designated by the United States Environmental Protection Agency (EPA) and many state agencies as a hazardous material that can cause central nervous system, kidney, and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury-containing products. See the applicable product Safety Data Sheet (SDS) for details and EPA’s website— [www.epa.gov/mercury/faq.htm](http://www.epa.gov/mercury/faq.htm)—for additional information. Users should be aware that selling mercury, mercury-containing products, or both, into your state may be prohibited by state law.

1.7 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.8 *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:<sup>2</sup>

- C670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials
  - D8 Terminology Relating to Materials for Roads and Pavements
  - D3666 Specification for Minimum Requirements for Agencies Testing and Inspecting Road and Paving Materials
  - D6373 Specification for Performance-Graded Asphalt Binder
  - D8239 Specification for Performance-Graded Asphalt Binder Using the Multiple Stress Creep and Recovery (MSCR) Test
  - E1 Specification for ASTM Liquid-in-Glass Thermometers
  - E77 Test Method for Inspection and Verification of Thermometers
  - E644 Test Methods for Testing Industrial Resistance Thermometers
  - E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory
- ### 2.2 AASHTO Standards:<sup>3</sup>
- R 29 Practice for Grading or Verifying the Performance Grade of an Asphalt Binder

<sup>1</sup> 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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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard’s Document Summary page on the ASTM website.

<sup>3</sup> 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>.

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2.3 *ISO Standard*.<sup>4</sup>

**ISO/IEC 17025 General Requirements for the Competence of Testing and Calibration Laboratories**

### 3. Terminology

3.1 *Definitions*—Definitions for many terms common to asphalt binder are found in Terminology **D8**.

3.2 *Definitions of Terms Specific to This Standard*:

3.2.1 *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.

3.2.2 *complex shear modulus ( $G^*$ ), n*—ratio calculated by dividing the absolute value of the peak-to-peak shear stress,  $\tau$ , by the absolute value of the peak-to-peak shear strain,  $\gamma$ .

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

3.2.4 *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.

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

3.2.6 *phase angle ( $\delta$ ), n*—the angle in degrees between the peak of a sinusoidally applied strain and the resultant peak of respective sinusoidal stress.

3.2.7 *portable thermometer, 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.

3.2.8 *reference thermometer, n*—refers to a liquid-in-glass or electronic thermometer that is used as a laboratory reference standard and is calibrated by an agency accredited to ISO/IEC 17025.

3.2.9 *steric hardening, n*—a process where molecular associations occur between asphalt binder molecules during storage at ambient temperature.

3.2.9.1 *Discussion*—Steric hardening decreases the phase angle and increases the complex shear modulus. Steric hardening is time and temperature dependent and is asphalt binder specific. It may significantly affect the test results after just a few hours of storage at ambient temperature.

3.2.10 *strain sweep*—an isothermal test procedure in which the strain is increased stepwise at a linear rate of increase.

3.2.11 *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 Test specimens are formed between parallel metal plates and one of the parallel plates is oscillated with respect to the other at pre-selected temperatures, frequencies, and rotational strain amplitudes. The required amplitudes are specified to ensure that the measurements are within the region of linear behavior.

4.2 The test specimen is maintained at the test temperature by positive heating and cooling of the upper and lower plates or by enclosing the upper and lower plates in a thermally controlled environment or test chamber.

4.3 Specification testing is performed at a test frequency of 10 rad/s. The complex modulus ( $G^*$ ) and phase angle ( $\delta$ ) 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 complex shear modulus is an indicator of the stiffness or resistance of asphalt binder to deformation under load. The phase angle is a measure of the relative portion of the response to an applied load that is elastic (recoverable) or viscous (nonrecoverable).

5.2 The test procedure is applicable to measurements in the linear region where the measured modulus and phase angle are independent of the amplitude of the strain.

5.3 The complex modulus and the phase angle are used to calculate performance-related criteria in accordance with Specification **D6373** or **D8239**.

NOTE 1—The quality of the results produced by this standard are dependent on the competence of the personnel performing the procedure and the capability, calibration, and maintenance of the equipment used. Agencies that meet the criteria of Specification **D3666** are generally considered capable of competent and objective testing, sampling, inspection, etc. Users of this standard are cautioned that compliance with Specification **D3666** alone does not completely ensure reliable results. Reliable results depend on many factors; following the suggestions of Specification **D3666** or some similar acceptable guideline provides a means of evaluating and controlling some of those factors.

### 6. Apparatus

6.1 *Dynamic Shear Rheometer (DSR) Test System*—A dynamic shear rheometer test system consisting of test plates, an environmental chamber, an internal thermometer, a loading device, and a data acquisition system as described in **6.1.1 – 6.1.5**. The manufacturer of the device shall provide a certificate certifying that the frequency, deflection angle, and torque are controlled and measured with an accuracy of 1 % or less in the range of this measurement.

6.1.1 *Test Plates*—Metal plates cylindrical in shape, formed from steel or aluminum, with smooth ground surfaces are required. Specification testing to **D6373** or **D8239** requires two plates (upper and lower)  $8.00 \pm 0.02$  mm in diameter and two plates (upper and lower)  $25.00 \pm 0.05$  mm in diameter (Item A

<sup>4</sup> Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandinnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <https://www.iso.org>.

in Fig. 1). In some cases, a laboratory may perform testing using only one plate size. In this case, only that plate size is required. The lower test plate shall have a minimum thickness or raised portion of 1.5 mm to adequately trim the specimen (Item B in Fig. 1). 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.

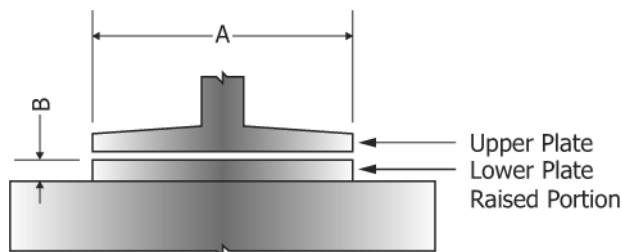


FIG. 1 Plate Diagram

NOTE 2—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.

6.1.2 *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 temperature  $\pm 0.1$  °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 3—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.

6.1.2.1 *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.

6.1.3 *Internal Thermometer*—A platinum resistance thermometer (PRT) readable to the nearest 0.1 °C. The PRT shall be mounted within the environmental chamber as an integral part of the DSR and in close proximity to the fixed plate. The internal thermometer 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. Standardize the internal thermometer and apply corrections in accordance with 8.4.1 of this standard.

6.1.4 *Loading Device*—The loading device shall be capable of applying a sinusoidal oscillatory load to the specimen at a frequency of  $10.0 \pm 0.1$  rad/s. If frequencies other than 10 rad/s are used, the frequency shall be accurate to 1 %.

6.1.5 *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 controlled and measured with an accuracy of at least 1 %.

6.2 *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 4—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.

6.3 *Trimming Tool*—A tool with a straightedge at least 4 mm wide 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.

6.4 *Reference Thermometer*—A standard reference thermometer for standardizing the portable thermometer. The reference thermometer must be calibrated annually by an agency accredited to ISO/IEC 17025. Alternatively, if the portable thermometer described in 6.5 is calibrated by an outside agency accredited to ISO/IEC 17025, the reference thermometer is not needed. The thermometer shall have a measurement temperature range covering the DSR's range of test temperatures and an accuracy of  $\pm 0.05$  °C and shall be one of the following:

6.4.1 A partial immersion liquid-in-glass thermometer readable to the nearest 0.01 °C meeting the requirements of Specification E1 readable to the nearest 0.05 °C. The thermometer shall be calibrated annually in accordance with Test Method E77.

6.4.2 A platinum resistance thermometer (PRT) readable to the nearest 0.01 °C, with a Pt 100 Class AA tolerance rating and either a three- or four-wire configuration and an overall sheath length at least 50 mm greater than the immersion depth. The thermometer shall be calibrated annually in accordance with Test Method E644. Corrections shall be applied to ensure accurate measurements within 0.05 °C.

6.4.3 A thermistor readable to the nearest 0.01 °C, calibrated annually in accordance with Test Method E644. Corrections shall be applied to ensure accurate measurements within 0.05 °C.

6.5 *Portable Thermometer*—A calibrated portable thermometer consisting of a resistive detector, associated electronic circuitry, and digital readout, fitted with a specimen-shaped wafer embedded with temperature sensor. The thickness of the sensor shall be no greater than 2.0 mm such that it can be inserted between the test plates. The portable thermometer



shall be a PRT or thermistor constructed in the same fashion as described in 6.4.2 or 6.4.3, standardized internally using the reference thermometer as described in 6.4 or externally by an outside agency accredited to ISO/IEC 17025.

6.5.1 The reference thermometer (see 6.4) may be used as the portable thermometer if its detector is fitted with a wafer embedded with a temperature sensor.

NOTE 5—Guide E882 may be used for evaluating test data.

6.6 *Micrometer*—A micrometer readable to 0.01 mm for verifying plate diameter.

## 7. Materials

7.1 *Wiping Material*—Clean cloth, paper towels, cotton swabs, or other suitable material as required for wiping the plates.

7.2 *Cleaning Solvents*—Solvents for cleaning the plates shall leave no residue on the surface of the plates.

NOTE 6—Mineral oil, citrus-based solvents, mineral spirits, toluene, or similar solvents have been found to be suitable for cleaning the plates. Organic solvent that does not leave a residue such as heptane, acetone, or ethyl alcohol can also be used for removing solvent residue from the surfaces of the plates.

7.3 *Anti-Seize Compound (optional)*—Used to ensure full contact and heat transfer between the faces of the test plates and the faces of the portable thermometer probe.

7.4 *Reference Fluid*—An organic polymer reference fluid with a known viscosity traceable to the international system of units through a national metrology institute (such as NIST) that is approximately 270 Pa·s at 64 °C, as determined through capillary viscosity measurements. The known viscosity for the production run (lot) of the reference standard shall be printed on the label of the bottle.

NOTE 7—A suitable reference fluid is available from Cannon Instrument Company as viscosity standard number N2700000SP or DSR64C. The reference fluid is reported in cP which is numerically equal to mPa·s.

## 8. Standardizations, Checks, and Verifications

8.1 Standardize, check, and verify the DSR and its components as described in this section when the DSR is newly installed, when it is moved to a new location, and/or whenever the accuracy of the DSR and/or any of its components is suspect. Four items require standardization, check, or verification: test plate diameter, portable thermometer, internal thermometer, and overall operation of the DSR. Standardize the internal thermometer before verifying the overall operation of the DSR.

8.2 *Check of Plate Diameter*—Before first using an upper or lower test plate and every six months thereafter, measure its diameter (average of three different locations ~120° apart) to the nearest 0.01 mm using the micrometer described in 6.6. Verify that the plates are in compliance with the requirements specified in 6.1.1. Maintain a log of the measured diameters so that the measurements are clearly identified with specific plates.

8.3 *Standardization of Portable Thermometer*—Standardize the portable thermometer (used to measure the temperature

between the test plates) at intervals of six months or less using the reference thermometer. If the reference thermometer (see 6.4) is also used as a portable thermometer to measure the temperature between the test plates, it shall be standardized as per 8.3. The portable thermometer shall be standardized using the same meters and circuitry (wiring) that are used when temperature measurements are made between the plates.

8.3.1 *Recommended Standardization Procedure*—Bring the reference thermometer into intimate contact with the probe of the portable thermometer and place them in a thermostatically controlled and stirred liquid bath. Obtain measurements with both thermometers at test temperatures that will be used when conducting tests with the DSR. Allow the bath to come to thermal equilibrium at each temperature. For this purpose, thermal equilibrium is defined for each thermometer as the point when three successive readings read at 1 min intervals do not change by more than 0.02 °C. Record the temperature on each thermometer when thermal equilibrium is reached. The difference between the two readings is the temperature correction that shall be applied to the portable thermometer.

8.4 *Standardization of Internal Thermometer*—The internal thermometer shall be verified at an interval of no greater than six months. When the differences between the DSR thermometer and the portable thermometer are 0.02 °C or greater, apply a temperature correction to correct to the temperature displayed by the portable thermometer.

NOTE 8—Some DSRs are programmed to automatically determine the temperature correction and software within the DSR automatically applies the correction to the reported test temperature.

8.4.1 *Internal Thermometer Standardization Procedure*—For the entire range of test temperatures used by the DSR and at intervals of 6 °C, place the portable thermometer between the 25 mm test plates and close the gap to bring the wafer into contact with the upper and lower plate so that the wafer makes complete contact with the surfaces of the upper and lower plates. If needed, apply a thin layer of petroleum grease or anti-seize compound to completely fill any void space between the wafer and the plates. Complete contact is needed to ensure proper heat transfer across the plates and wafer.

8.4.2 At each temperature verified, ensure thermal equilibrium has been reached and record the temperature indicated by the portable thermometer and the DSR thermometer to the nearest 0.01 °C. Thermal equilibrium is reached when the temperature indicated by both the portable thermometer and the DSR thermometer do not vary by more than 0.03 °C over a 3 min period.

8.4.3 *Application of Temperature Correction*—If an adjustment to the DSR thermometer is required, it is typically incorporated automatically within the software on with newer rheometers using the temperature verification data. If the DSR does not have this capability, perform a manual temperature correction as described in Appendix X1 and adjust the target test temperature in accordance with the correction such that the temperature reported by the DSR includes the correction.

8.5 *Verification of Overall Operation of DSR*—Verify the overall operation of the DSR using a reference fluid once every six months or when the accuracy of the test measurements are suspect.

8.5.1 The reference fluid shall be used only at 64 °C. Below that temperature the phase angle differs sufficiently from 90° such that the values calculated from  $G^*$  and  $\omega$  are no longer accurate. Above 64 °C the fluid tends to flow from the gap between the plates invalidating the measured value for  $G^*$ .

NOTE 9—A newly installed or reconditioned instrument should be verified on a weekly basis using the procedures in 8.5 until acceptable verification has been demonstrated.

8.5.2 *Measurement of the Viscosity of the Reference Fluid*—Measure the complex modulus,  $G^*$ , of the reference fluid at 64 °C and 10 rad/s. Additional information regarding use of the reference fluid is given in Annex A1. Precaution should be taken to ensure that there are no air bubbles in the test specimen. Newer rheometers are typically programmed to automatically calculate and report the measured viscosity,  $\eta_M$ , in units of mP-s. When making the calculation directly, convert the numerical value of  $G^*$  in kPa at 10 rad/s to viscosity in mPa-s by multiplying the numerical value of  $G^*$  by  $10^5$ . For example, if  $G^* = 2.66$  kPa,  $\eta_M = 266\,000$  mPa-s. If the units for  $G^*$  are in Pa, multiply by  $10^2$ .

NOTE 10—The reference fluid may be reported in cP or mPa-s, which are numerically equivalent; for example, 1 mPa = 1cP. The simplified conversion given above is provided to make the calculation of  $\eta_M$  straightforward for the typical user. The multipliers  $10^5$  and  $10^2$  are necessary for the proper conversion of units.

8.5.3 The viscosity,  $\eta_M$ , measured with the DSR shall be within 3 % of the viscosity,  $\eta_R$ , reported by the manufacturer of the reference fluid. Otherwise, the overall operation of the DSR shall be considered suspect. Calculate the estimated percent difference as:

$$\text{percent difference} = 100 \% \times \left[ \frac{(\eta_R - \eta_M)}{\eta_R} \right] \quad (1)$$

where:

- $\eta_R$  = the viscosity as reported by the supplier of the reference fluid, mP-s (cP), and
- $\eta_M$  = the measured viscosity, converted to mPa-s (cP).

8.5.4 If the overall DSR operation cannot be successfully verified according to 8.5, it shall not be used for testing in accordance with this standard until it has been successfully calibrated by the manufacturer or other qualified service personnel.

## 9. Preparation of Apparatus

9.1 Prepare the apparatus for testing in accordance with the manufacturer's recommendations. Specific requirements will vary for different DSR models and manufacturers.

9.2 *Inspect Test Plates*—Inspect the surfaces of the test plates and discard any plates with jagged or rounded edges or deep scratches.

9.3 *Preparation of Test Plates*—Clean any asphalt binder residue from the plates with an organic solvent such as mineral oil, mineral spirits, a citrus-based solvent, or toluene. Remove any remaining solvent residue by wiping the surface of the plates with a cotton swab or a soft cloth dampened with reagent grade organic solvent such as heptane, ethyl alcohol, or

acetone. If necessary, use a dry cotton swab or soft cloth to ensure that no moisture condenses on the plates.

9.4 *Mount Test Plates and Fixtures*—If the fixtures or plates are removed for cleaning, attach them as directed by the DSR manufacturer.

9.5 *Zero the Test Gap*—Zero the test gap in accordance with one of the three options described in 9.5.1 – 9.5.3. If the test temperature differs by more than  $\pm 12$  °C from the temperature at which the gap is set, re-zero the gap.

NOTE 11—The frame, detectors, and fixtures in the DSR change dimension with temperature, causing the zero gap to change with changes in temperature. Adjustments in the gap are not necessary when measurements are made over a limited range of temperatures. The gap should be set at the test temperature or, when tests are to be conducted over a range of temperatures, at the middle of the expected range of test temperatures. If the instrument has thermal gap compensation, the gap may be set at the first test temperature instead of the middle of the range of test temperatures.

9.5.1 *Manual Gap Setting*—Manually spin the moveable plate, and while the moveable plate is spinning, slowly close the gap. The zero gap is reached when the plate just stops spinning completely.

9.5.2 *Normal Force Transducer*—For rheometers with normal force transducers, set the zero gap by closing the gap and observing the normal force. Select the zero gap as the position where the normal force is approximately zero.

9.5.3 *Automatic Gap Setting*—Zero the gap automatically according to operating procedures specified by the instrument manufacturer.

9.6 *Preheating Test Plates*—Once the zero gap is established as per 9.5.1, move the plates apart to approximately set the test gap and preheat the plates. Preheating the plates promotes adhesion between the asphalt binder and the plates, especially at the intermediate grading temperatures.

9.6.1 *Preheating 25 mm Plate*—Bring the test plates to the test temperature or the lowest test temperature if testing is to be conducted at more than one temperature.

9.6.2 *Preheating 8 mm Plate*—Bring the plates to between 34 and 46 °C to preheat the upper and lower plates.

NOTE 12—Specifications D6373 and D8239 and AASHTO R 29 provide guidance on the selection of test temperatures.

NOTE 13—Preheating the test plates is important in order to obtain adequate adhesion between the asphalt binder and the test plates. Preheating is especially critical when the silicone mold is used to prepare the asphalt binder for transfer to the test plates and when the testing is conducted with the 8 mm plates. When the direct placement method is used, as long as the test plates are immediately brought in contact with the asphalt binder, the heat carried with the asphalt binder improves adhesion. The preheating temperature needed for proper adhesion will depend on the grade and nature of the asphalt binder and the test temperature (8 mm or 25 mm plates). For some of the stiffer binder grades, especially those with high levels of modification, heating the plates to 46 °C may not be sufficient to ensure proper adhesion of the asphalt binder to the test plates, especially if the silicone mold is used and the testing is conducted with 8 mm plates.

## 10. Preparing Test Specimens

10.1 *Annealing Asphalt Binder*—Immediately prior to forming the test specimen, anneal the asphalt binder sample from which the test specimen is prepared by heating the sample in a

container in an oven until it is sufficiently fluid to pour. Cover the sample and stir it occasionally during the heating process to ensure homogeneity and to remove air bubbles. Annealing prior to testing reverses steric hardening that occurs during normal storage at ambient temperature.

NOTE 14—Minimum pouring temperatures that produce a consistency equivalent to that of SAE 10W30 motor oil (readily pours but not overly fluid) at room temperature are recommended. Heating unaged asphalt to temperatures above 135 °C should be avoided. However, with some modified asphalts or heavily aged binders, pouring temperatures above 135 °C may be required. In all cases, heating time and temperature should be minimized. During the heating process, the sample should be covered and stirred occasionally to ensure homogeneity. Use caution during stirring to avoid trapping air bubbles in the asphalt binder.

**10.2 Transferring Binder to Test Plate**—Transfer asphalt binder to one of the test plates through direct transfer or by use of a silicone mold.

**10.2.1 Direct Transfer**—Transfer the hot binder to one of the plates by pouring from a suitable container or using a glass or metal rod, spatula, or similar tool. Immediately after transferring the hot binder to one of the plates, proceed to **10.3** to trim the specimen and **10.4** to form the bulge.

NOTE 15—A small, narrow stainless steel spatula of the type used to weigh powders on an analytical balance has been found suitable for transferring the hot binder. When using a rod, form a mass of sufficient size to form the test specimen by using a twisting motion. The twisting motion seems to keep the mass on the rod in control. A 4 to 5 mm diameter rod is suitable. The glass rod technique is especially useful for the 8 mm plate.

**10.2.2 Silicone Mold**—Pour the hot asphalt binder into a silicone mold to form a convex surface. Allow the mold to cool to room temperature. The molds shall be covered while cooling to eliminate contamination. The filled mold shall be cooled at room temperature by placing the mold on a flat laboratory bench surface without chilling. Cooling to temperatures below room temperature results in an unknown thermal history that may affect the measured values of modulus and phase angle. Chilling may also result in the formation of moisture on the surface of the specimen that will interfere with adhesion of the specimen to the plates.

**10.2.2.1 Mount the specimen on the DSR test plate.** The specimen may be mounted to either the upper or lower plate. To mount the specimen to the lower plate, remove the specimen from the mold and center the pellet on the lower plate of the DSR. Alternatively, center the specimen on the bottom plate while it is still in the rubber mold. Gently press the specimen to the bottom plate and then carefully remove the silicone rubber mold, leaving the specimen adhered to the bottom plate. To mount the specimen to the upper plate, center the specimen on the upper plate while it is still in the silicone rubber mold. Gently press the specimen to the upper plate and then carefully remove the silicone rubber mold, leaving the specimen adhered to the upper plate. Complete all testing within 4 h of pouring the specimen into the silicone rubber mold.

NOTE 16—Acetone may be used to clean the silicone rubber molds. Wipe the molds with a clean cloth or a cloth moistened with acetone to remove any asphalt binder residue. With repeated use the silicone molds become sticky. If sticking becomes a problem, discard the mold.

NOTE 17—Some softer binder grades cannot be removed from the

silicone mold without cooling below ambient temperature. If the binder specimen cannot be removed from the mold without cooling, it is recommended that the direct transfer method be used, or the filled mold may be chilled in a freezer or refrigerator for a maximum of 10 min to facilitate demolding.

**10.3 Trimming Test Specimen**—Immediately after the specimen has been placed on one of the test plates as described above, move the test plates together until the gap between the plates equals the testing gap plus the gap closure required to create the bulge. This is typically done automatically for newer rheometers. Trim excess binder by moving a heated trimming tool around the edges of the plates so that the asphalt binder is flush with the outer diameter of the plates.

NOTE 18—The trimming tool should be at a temperature that is sufficiently hot as to allow trimming but not excessively hot as to cause the specimen to emit smoke.

**10.4 Creating Testing Bulge**—Immediately after trimming is complete, decrease the gap by the amount required to form a slight bulge at the outside face of the test specimen. This is typically done automatically by newer rheometers. See **Appendix X2** if creating the testing bulge is done manually. Recommended gap closure values for creating the bulge are 0.05 mm for the 25 mm plate and 0.10 mm for the 8 mm plate.

NOTE 19—The complex modulus is calculated with the assumption that the specimen diameter is equal to the plate diameter. If the binder forms a concave surface at its outer edges during testing, this assumption will not be valid, and the modulus will be underestimated. The operator should visually verify that a proper bulge is present at the beginning and end of a test, especially when the test temperature is lower or higher than the bulge formation temperature.

## 11. Testing the Specimen

**11.1** Set the temperature controller to the temperature, including any temperature offset correction as per **8.4**, required to obtain the test temperature in the test specimen between the test plates. Allow the DSR to reach thermal equilibrium within  $\pm 0.1$  °C of test temperature.

**11.1.1** When testing a binder for compliance to Specification **D6373** or **D8239**, select the appropriate test temperature from Table 1 those standards. When testing at multiple temperatures, start at the lowest test temperature for the 25 mm plate and start at the highest test temperature for the 8 mm plate. Select an appropriate strain value from **Table 1**. Software is available with the dynamic shear rheometers that will control the strain automatically without control by the operator.

**11.2** Allow the temperature indicated by the internal thermometer to come to the target test temperature. The test shall be started only after the temperature has remained at the desired temperature  $\pm 0.1$  °C for at least 10 min. Start the application of the load and obtain a measurement of the complex modulus, phase angle, and frequency after applying 8 to 16 initial loading cycles.

**TABLE 1 Target Strain Values**

Material	kPa	Target Value, %	Range, %
Original Binder	1.0 G <sup>*</sup> /sin $\delta$	12	9 to 15
RTFO Residue	2.2 G <sup>*</sup> /sin $\delta$	10	8 to 12
PAV Residue	5000 G <sup>*</sup> sin $\delta$	1	0.8 to 1.2