



Designation: D 7175 – 08

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

This standard is issued under the fixed designation D 7175; 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 D 2872, material aged in accordance with Practice D 6521, or material aged in accordance with both Test Method D 2872 and Practice D 6521. This procedure is limited to asphalt binders that contain particles with largest dimension less than 250 μm .

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

- C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials
D 140 Practice for Sampling Bituminous Materials

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.

D 2170 Test Method for Kinematic Viscosity of Asphalts (Bitumens)

D 2171 Test Method for Viscosity of Asphalts by Vacuum Capillary Viscometer

D 2872 Test Method for Effect of Heat and Air on a Moving Film of Asphalt (Rolling Thin-Film Oven Test)

D 6373 Specification for Performance Graded Asphalt Binder

D 6521 Practice for Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel (PAV)

E 77 Test Method for Inspection and Verification of Thermometers

E 563 Practice for Preparation and Use of an Ice-Point Bath as a Reference Temperature

E 644 Test Methods for Testing Industrial Resistance Thermometers

E 882 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.

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 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 für Normung e.V.), Burggrafenstrasse 6, 10787, Berlin, Germany, <http://www.en.din.de>.

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 $\pm 0.1^\circ\text{C}$ 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 D 6373 or AASHTO Standard M320.

6. Interferences

6.1 Particulate material in the asphalt binder is limited to particles with longest dimensions less than 250 μm . Particles with dimensions greater than 250 μm approach the dimensions of the gap (1000 μm). 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 $\pm 0.1^\circ\text{C}$ 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 temperature $\pm 0.1^\circ\text{C}$. 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.

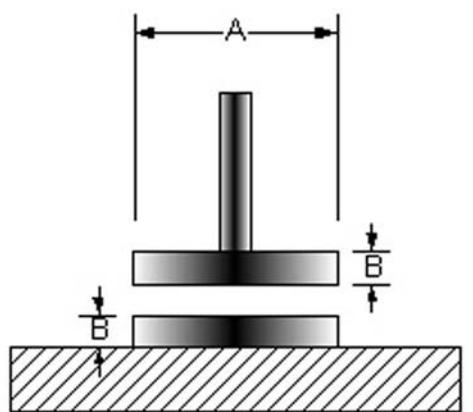


FIG. 1 Plate Dimensions

Dimension	8-mm Nominal	25-mm Nominal
A	8 ± 0.1 mm	25 ± 0.5 mm
B	> 1.5 mm	> 1.5 mm

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 percent. 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 E 77 and Practice E 563.

NOTE 4—An Optical Viewing Device is recommended as an optional viewing device for use with liquid-in-glass thermometers because it enhances readability and minimizes parallax when reading the liquid-in-glass reference thermometer.

7.4.2 Digital Temperature Measurement Device—An electronic thermometer that incorporates a thermometric device or resistive detector with an accuracy of $\pm 0.05^\circ\text{C}$ and a resolution of 0.01°C . The electronic thermometer shall be calibrated at least once per year by a commercial calibrating service using a NIST-traceable reference standard in accordance with Test Methods E 644.

7.5 Portable Temperature Measurement Device—A calibrated portable thermometer consisting of a thermometric device or resistive detector, associated electronic circuitry, and digital readout. The thickness of the detector shall be no greater than 2.0 mm such that it can be inserted between the test plates.

7.5.1 The reference temperature measurement device (see **7.4**) may be used for this purpose if its detector fits within the dummy specimen as required by **9.4.2**.

NOTE 5—Guide E 882 may be used for evaluating test data.

8. Materials

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

8.2 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.

9. Verification

9.1 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 verification: test plate diameter, DSR torque transducer, portable thermometer, and DSR test specimen temperature. Verify the DSR temperature before verifying the torque transducer.

9.2 Verification of Plate Diameter—Before first using an upper or lower test plate, measure its diameter (average of 3 different locations ~120° apart) to the nearest 0.02 mm. Verify that the plates are in compliance with the requirements specified in **7.1.1**. Maintain a log of the measured diameters so that the measurements are clearly identified with specific plates.

9.3 Verification of Portable Thermometer—Verify the portable thermometer (used to measure the temperature between the test plates) at intervals of six months or less using the laboratory reference thermometer. If the reference thermometer (see **7.4**) is also used as a portable thermometer to measure the temperature between the test plates, it shall be verified as per **9.3.1**. Electronic thermometers shall be verified using the same meters and circuitry (wiring) that are used when temperature measurements are made between the plates.

9.3.1 Recommended Verification Procedure—Bring the reference thermometer into intimate contact with the detector from the portable thermometer and place them in a thermostatically controlled and stirred water bath (see **Note 7**). Ensure that de-ionized water is used to prevent electrical conduction from occurring between electrodes of the resistive temperature sensitive element. If this is not available, encase the reference thermometer and the detector of the portable thermometer into a water proof plastic bag prior to placement into the bath. Obtain measurements at intervals of 6°C over the range of test temperatures allowing the bath to come to thermal equilibrium at each temperature. If the readings of the portable thermom-

eter and the reference thermometer differ by 0.1°C or more, record the difference at each temperature as a temperature correction and maintain the corrections in a log.

NOTE 7—A recommended procedure is to use a stirred water bath that is controlled to ± 0.1°C such as the viscosity bath used for Test Method D 2170 or Test Method D 2171. Bring the probe from the portable thermometer into contact with the reference thermometer and hold the assembly in intimate contact. A rubber band works well for this purpose. Immerse the assembly in the water bath and bring the water bath to thermal equilibrium. Record the temperature on each device when thermal equilibrium is reached.

9.4 Temperature Offset—Thermal gradients within the rheometer can cause differences between the temperature of the test specimen and the temperature indicated by the DSR thermometer (also used to control the temperature of the DSR). When these differences are 0.1°C or greater, determine a temperature correction by using a thermal detector mounted in a silicone rubber wafer (see **9.4.1**) or by placing asphalt binder (dummy sample) between the plates and inserting the detector of the portable thermometer into the asphalt binder (see **9.4.2**). The temperature offset shall be verified each time the portable thermometer is verified.

9.4.1 Method Using Silicone Rubber Wafer—Place the wafer 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 silicone rubber 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 (see **Note 8**) to completely fill any void space between the silicone rubber and the plates. Complete contact is needed to ensure proper heat transfer across the plates and silicone rubber wafer (see **Note 9**). Determine any needed temperature correction as per **9.4.3**.

NOTE 8—Anti-seize compound available by that name at hardware and the auto supply stores is much less apt to contaminate the circulating water than petroleum jelly.

NOTE 9—The currently available silicone wafer is 2 mm thick and slightly greater than 25 mm in diameter.

9.4.2 Method Using Dummy Test Specimen—The dummy test specimen shall be formed from asphalt binder, or other polymer that can be readily formed between the plates. Mount the dummy test specimen between the test plates and insert the detector (probe) of the portable thermometer into the dummy test specimen. Close the gap to the test gap (1 mm for 25-mm plates and 2 mm for 8-mm plates) keeping the detector centered vertically and radially in the dummy test specimen. Heat the plates as needed to allow the dummy test specimen to completely fill the gap between the test plates. It is not necessary to trim the dummy test specimen but avoid excessive material around the edges of the plates. Develop control charts using Guide E 882. Determine any needed temperature correction as per **9.4.3**.

NOTE 10—Silly putty can leave a residue of silicone oil on the surfaces of the plates and for this reason its use as a dummy specimen is not recommended.

9.4.3 Determination of Temperature Offset—Obtain simultaneous temperature measurements with the DSR thermometer and the portable thermometer at 6°C increments to cover the range of test temperatures. At each temperature increment,

after thermal equilibrium has been reached, record the temperature indicated by the portable thermometer and the DSR thermometer to the nearest 0.1°C. Temperature equilibrium is reached when the temperature indicated by both the DSR thermometer and the portable thermometer do not vary by more than 0.1°C over a five minute time period. Obtain additional measurements to include the entire temperature range that will be used for measuring the dynamic shear modulus.

9.4.4 Plot Offset versus Specimen Temperature (optional)— Using the data obtained in **9.4.3**, prepare a plot of the difference between the two temperature measurements versus the temperature measured with the portable thermometer, **Fig. 2**. This difference is the temperature correction that must be applied to the DSR temperature controller to obtain the desired temperature in the test specimen between the test plates. Report the temperature correction at the respective test temperature from the plot and report the corrected test temperature between the plates as the test temperature. Alternatively, the instrument software may be written to incorporate these temperature corrections.

NOTE 11—The difference between the two temperature measurements may not be a constant for a given rheometer but may vary with differences between the test temperature and the ambient laboratory temperature as well as with fluctuations in ambient temperature. The difference between the two temperature measurements is caused in part by thermal gradients in the test specimen and fixtures.

9.5 Verification of DSR—Verify the accuracy of the torque transducer and angular displacement transducer whenever the DSR is newly installed, when it is moved, every six months, each time temperature offset is verified, and/or whenever the accuracy of measurements with the DSR is suspect.

NOTE 12—A newly installed or reconditioned instrument should be verified on a weekly basis using the procedures in **9.5** until acceptable verification has been demonstrated. Maintaining the data in the form of a control chart where the verification measurements are plotted versus calendar date is recommended (see **Appendix X2**).

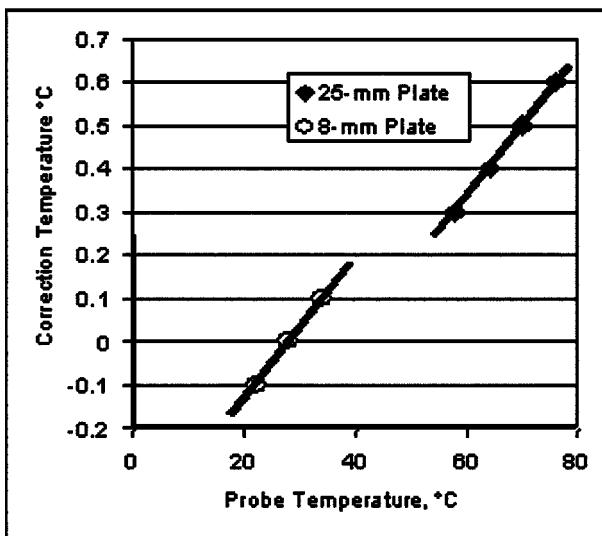


FIG. 2 Determination of Temperature Correction

9.5.1 Verification of Torque Transducer—Verify the calibration of the torque transducer using a reference fluid or manufacturer-supplied fixtures whenever the calibration of the torque transducer is suspect and/or when the dynamic viscosity, as measured for the reference fluid, indicates that the torque transducer is not in calibration.

9.5.1.1 Verification of Torque Transducer with Reference Fluid (see **Annex A1**)—The complex viscosity measured with the DSR shall be within 3 percent of the capillary viscosity as reported by the manufacturer of the reference fluid, otherwise the calibration of the torque transducer shall be considered suspect. Calculate the complex viscosity as the complex modulus, G^* divided by the angular frequency in rad/s. If the requirements of this section can not be met, discontinue use of the device and consult the manufacturer or other qualified service personnel.

NOTE 13—A suitable reference fluid is available from Cannon Instrument Company as viscosity standard number N2700000SP.

9.5.1.2 Verification of Torque Transducer with Fixtures—Verify the calibration of the torque transducer using the manufacturer-supplied fixtures in accordance with the instructions supplied by the manufacturer. Suitable manufacturer-supplied fixtures are not widely available. If suitable fixtures are not available, this requirement shall be waived.

9.5.2 Verification of Angular Displacement Transducer—If manufacturer-supplied fixtures are available, verify the calibration every six months and/or whenever the calibration of the DSR is suspect. If suitable fixtures are not available, this requirement shall be waived.

10. Preparation of Apparatus

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

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

10.3 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.

10.4 Mount Test Plates and Fixtures—Mount the test plates and fixtures in the DSR taking care to visually ensure that the plates are parallel to each other and tighten the plates and fixtures to firmly seat them into the DSR, taking care to ensure that the plates are parallel to each other.

10.5 Zero Gap—Select the testing temperature according to the expected grade of the asphalt binder or according to the pre selected testing schedule. When multiple test temperatures are used, zero the gap at the middle of the expected range of test temperatures. Allow the DSR to reach a stabilized temperature within $\pm 0.1^\circ\text{C}$ of test temperature. If the test temperature differs by more than $\pm 12^\circ\text{C}$ from the temperature at which the

gap is set, re-zero the gap. Zero the gap prior to each time a new specimen is formed between the plates.

NOTE 14—If the instrument has thermal gap compensation, the gap may be set at the first test temperature instead of in the middle of the range of test temperatures. It is very important to set and zero the gap correctly. Incorrect gap setting produces significant errors as shown in Fig. 3.

NOTE 15—Specification D 6373 and AASHTO Practice R29 provide guidance on the selection of test temperatures.

10.5.1 Determining Zero Gap—Establish the zero gap by one of three methods.

10.5.1.1 Manual Gap Setting—Spin the moveable plate, and while the moveable plate is spinning, close the gap until the movable plate touches the fixed plate. The zero gap is reached when the plate just stops spinning completely.

10.5.1.2 Normal Force Transducer—For rheometers with normal force transducers, set the zero gap by closing the gap and observing the normal force. After establishing contact between the plates, set the zero gap as the position where the normal force is approximately zero.

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

10.6 Preheating Test Plates—Once the zero gap is established as per 10.5.1, 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.

10.6.1 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.

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

NOTE 16—In order to obtain adequate adhesion between the asphalt binder and the test plates the plates must be preheated. 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.

11. Preparing Test Specimens

11.1 If unaged binder is to be sampled and tested, obtain test samples according to Practice D 140.

11.2 Annealing Asphalt Binder—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 removes reversible molecular associations (steric hardening) that occur during normal storage at ambient temperature. Cold samples must be annealed prior to testing. Structure developed during storage can result in overestimating the modulus by as much as 50 percent. During annealing process do not allow the temperature of the binder to exceed 163°C. However for some highly modified viscous binders, temperatures higher than 163°C may be required for annealing.. Minimize the heating temperature and time to avoid hardening the sample. Hot plates shall not be used to heat the asphalt binder.

NOTE 17—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 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.

11.3 Transferring Binder to Test Plate—Transfer asphalt binder to one of the test plates through pouring (see 11.3.1), direct transfer (see 11.3.2), or by use of a silicone mold (see 11.3.3).

11.3.1 Pouring—Pour the hot asphalt binder from a heated container directly onto the lower plate while it is mounted in the DSR or onto one of the plates that has been removed from the DSR. The removal of plate/plates is only allowed if the rheometer is equipped with a plate that may be removed and reinstalled without affecting the gap reference. Hold the sample container approximately 15 to 25 mm above the surface of the plate while pouring. Leave a small portion at the outside edge of the plate uncovered. Immediately after pouring the hot asphalt binder on the plate, and returning the plate to the DSR if the plate is removed for pouring, close the gap to the testing gap plus the gap required to form the bulge. Proceed immediately to 11.4 to trim the specimen and form the bulge.

11.3.2 Direct Transfer—Transfer the hot binder to one of the plates using a glass or metal rod, spatula or similar tool. Immediately after transferring the hot binder to one of the plates, proceed to 11.4 to trim the specimen and 11.5 to form the bulge.

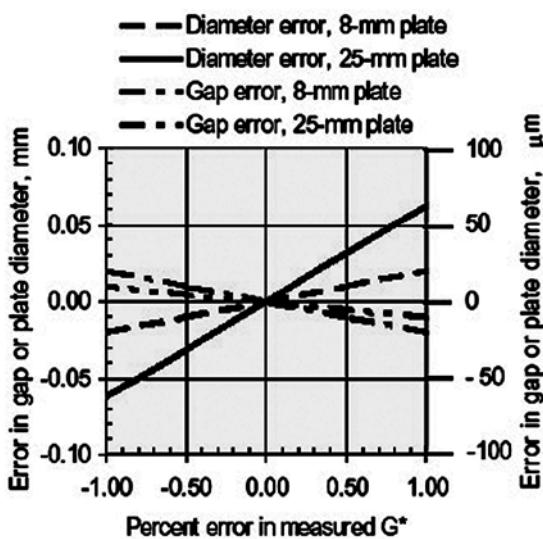


FIG. 3 Effect of Error in Gap

NOTE 18—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.

11.3.3 Silicone Mold—Pour the hot asphalt binder into a silicone mold to form a convex surface. Do not trim the surface of the binder. Allow the mold to cool to room temperature. As soon as the binder and mold have cooled to room temperature, loosen the binder from the mold by flexing the rubber mold. Gently press the convex (top) surface of the pellet against one of the preheated test plates forcing the asphalt binder to adhere to the plate. The filled mold should be cooled at room temperature by placing the mold on a flat laboratory bench surface without chilling. Cooling to below room temperature results in an unknown thermal history that may affect the measured values of modulus and phase angle. Excessive cooling 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. Pull the silicone rubber mold from the binder, close the gap to the test gap plus the gap required to form the bulge. Proceed immediately to **11.4** to trim the specimen and **11.5** to form the bulge. Testing shall be completed within two hours of pouring the hot asphalt binder into the mold. Discard any test results if the testing is not completed within two hours of being poured into the silicone mold.

NOTE 19—Solvents should not be used to clean the silicone rubber molds. Wipe the molds with a clean cloth to remove any asphalt binder residue. With repeated use the silicone molds become sticky. If sticking becomes a problem, discard the mold.

NOTE 20—Some binder grades cannot be removed from the silicone mold without cooling. Materials such as PG52-34, PG46-34, and some PG58-34 grades do not lend themselves to being removed from the mold at ambient temperature. If the binder specimen cannot be removed from the mold without cooling, it is recommended that the direct transfer or pouring method be used.

11.4 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 (see **11.5** for gap closure required to create the bulge). 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 21—The trimming tool should be at a temperature that is sufficiently hot as to allow trimming but not excessively hot as to pyrolyze the edge of the specimen.

11.5 Creating Bulge—When the trimming is complete, decrease the gap by the amount required to form a slight bulge at the outside face of the test specimen. The gap required to create a bulge is rheometer specific and depends upon factors such as the design of the rheometer and the temperature difference between the trimming temperature and test temperature. A recommended practice for verifying the gap closure required to produce an appropriate bulge is given in **Appendix X3**. Typical closure values for creating the gap are 0.05 mm for the 25-mm plate and 0.10 mm for the 8-mm plate.

NOTE 22—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 this assumption will not be valid and the modulus will be underestimated. The calculated modulus is based upon the radius of the plate raised to the fourth power. A slight bulge, equal to approximately one-quarter of the gap is recommended. A procedure for determining the closure required to form an acceptable gap is given in **Appendix X3**.

12. Test Procedure

12.1 Set the temperature controller to the temperature, including any temperature offset correction as per **9.4**, required to obtain the test temperature in the test specimen between the test plates. Allow the DSR to reach thermal equilibrium within $\pm 0.1^{\circ}\text{C}$ of test temperature. The test shall be started five to ten minutes after the test specimen has reached thermal equilibrium (see **Fig. 4**). See **Appendix X4** for a method of determining the time required to reach thermal equilibrium.

NOTE 23—It is impossible to specify a single equilibration time that is valid for DSRs produced by different manufacturers. The design (fluid bath or air oven) of the environmental control system and the starting temperature will dictate the time required to reach the test temperature. The method for determining the correct thermal equilibration time is described in **Appendix X4**.

12.1.1 When testing a binder for compliance to Specification **D 6373**, select the appropriate test temperature from Table 1 of Specification **D 6373**.

12.1.2 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.

12.1.3 Testing in Strain Control Mode—When operating in a strain-controlled mode 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.

12.1.4 Stress Control Mode—When operating in a stress-controlled mode, select an appropriate stress value from **Table 2**. Software is available with the dynamic shear rheometers that will control the stress level automatically without control by the operator.

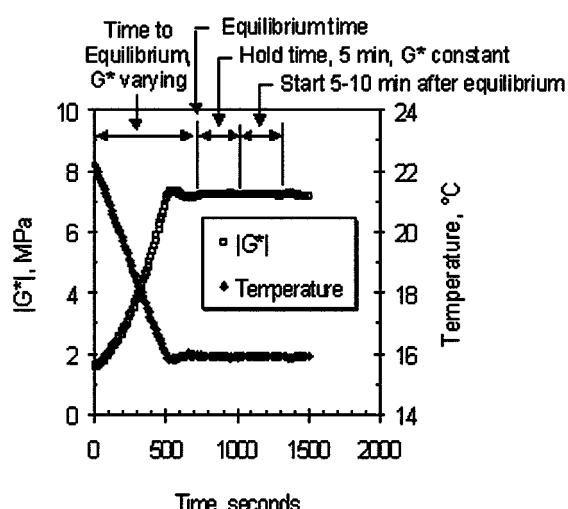


FIG. 4 Determining Thermal Equilibrium Time

TABLE 1 Target Strain Values

Material	kPa	Target Value, %	Range, %
Original Binder	1.0 G*/sin δ	12	9 to 15
RTFO Residue	2.2 G*/sin δ	10	8 to 12
PAV Residue	5000 G*·sin δ	1	0.8 to 1.2

TABLE 2 Target Stress Levels

Material to be Tested	KPa	Stress, kPa	
		Target Value	Range
Original Binder	1.00 G*/sin δ	0.120	0.090 to 0.150
RTFO Residue	2.20 G*/sin δ	0.220	0.180 to 0.260
PAV Residue	5000 G*·sin δ	500	400 to 600

12.2 Initiate the testing within five to ten minutes after reaching thermal equilibrium at each test temperature. The testing at subsequent temperatures should be done as quickly as possible to minimize the effect of molecular associations (steric hardening) that can cause an increase in modulus if the specimen is held in the rheometer for a prolonged period of time. When testing at multiple temperatures all testing should be completed within two hours of preparing the test specimen. 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.

12.3 Obtain a test measurement by averaging data for an additional 8 to 16 loading cycles using the analytical technique and software provided by the manufacturer. When conducting tests at more than one frequency, start testing at the lowest frequency and increase to the highest frequency.

NOTE 24—The standard frequency of 10 rad/s is used when testing binder for compliance with Specification D 6373.

13. Interpretation of Results

13.1 The dynamic modulus and phase angle may depend upon the magnitude of the shear strain. The modulus and phase angle for both unmodified and modified asphalt cement decrease with increasing shear strain as shown in Fig. 5. A plot such as that shown in Fig. 5 can be generated from a single test specimen by increasing the load or strain amplitude in steps, thereby producing a plot of complex shear modulus versus shear strain. It is not necessary to generate such plots during normal specification testing, however such plots are useful for verifying the limit of the linear region.

13.2 A linear region may be defined at small strains where the modulus is relatively independent of shear strain. The linear region is defined as the range in strains where the value of the complex modulus is 90 percent or more of the initial strain value as determined in accordance with Appendix X1.

14. Report

14.1 Report the following information. A recommended format for reporting the information is given in Fig. 6.

- 14.1.1 File name,
 - 14.1.2 Test Specimen ID Number,
 - 14.1.3 Sample identification information,
 - 14.1.4 Operator's name.

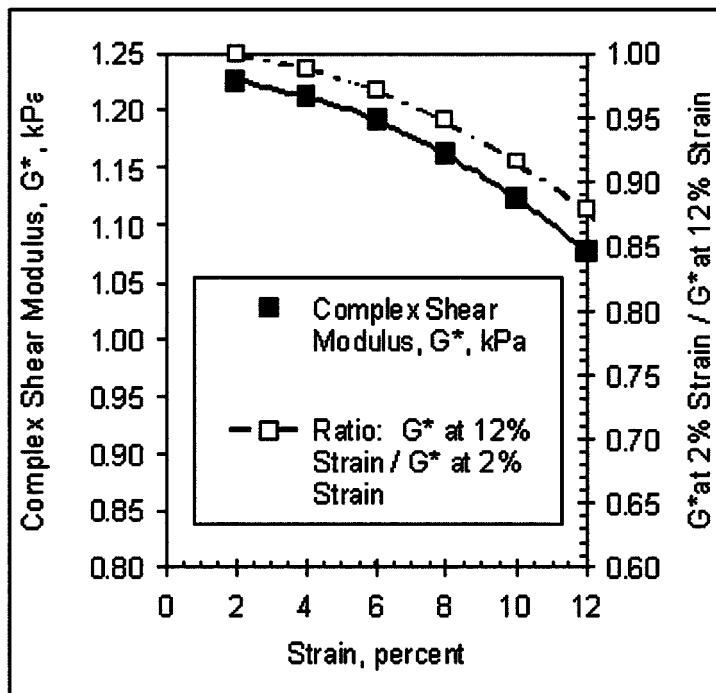


FIG. 5 Example of Strain Sweep

Instrument Serial No: XXXXXXXXXX Temp. Control: Water Bath
Summary Sheet: Software:Version 5.07
File Name: C:\XX\XXXXXXX

Parameters
Measurement Type: Intermediate Temperature Range
Target Temperature: 22.0°C
Strain Amplitude: 1.00 percent

Ancillary Information
Operator ID:
Sample ID:
Sample Type:
Test Number:

Measurement Results
Modulus (G^*):
Phase Angle (δ):
 $G^* \cdot \sin \Delta$:
Final Temperature:
Test Status:

Operator Notes

FIG. 6 Example Format for Reporting Data and Generating Electronic File

- 14.1.5 Date of test (dd/mm/yy),
 - 14.1.6 Time of test (hr:min) using 24-h clock,
 - 14.1.7 Manufacturer and Model Number of DSR, to be entered automatically by software contained within the DSR,
 - 14.1.8 Identification code that is unique to device. This is used to distinguish between multiple DSRs when more than one DSR is used in a laboratory,
 - 14.1.9 Version of software used, to be entered automatically by software contained within the DSR,

14.1.10 Test temperature to indicate the temperature of the test specimen between the DSR test plates ($^{\circ}\text{C}$ to 0.1°C),

14.1.11 Temperature correction, if a temperature offset was applied, at the test temperature ($^{\circ}\text{C}$ reported to the nearest 0.1°C),

14.1.12 Complex modulus (G^*), kPa to three significant figures,

14.1.13 The phase angle (δ), nearest 0.1 degrees,

14.1.14 Complex modulus divided by sin delta ($G^*/\sin \delta$), for unaged and RTFO aged binders,

14.1.15 Complex modulus multiplied by sin delta ($G^*\sin \delta$), for PAV aged binders,

14.1.16 Indicate whether the specimen passes or fails the specification, and

14.1.17 The strain amplitude to three significant figures.

15. Precision and Bias

15.1 *Precision*—Criteria for judging the acceptability of dynamic shear results obtained by this method are given in Table 3.

15.1.1 These criteria are based on an earlier version of this proposed standard and the procedure given in AASHTO T315. The figures in Column 2 of Table 3 are coefficients of variation that have been found to be appropriate for the conditions of test described in Column 1. The precision estimates given in Table 3 are based on the analysis of paired test results submitted by 185 to 208 laboratories testing eight pairs of AMRL proficiency samples. The analysis included five grades of asphalt binder: PG 52-34, PG 64-16, PG 64-22, PG 70-22 and PG 76-22 (SBS modified). The details of this analysis are in final report for NCHRP Project No. 9-26, Phase 3.

15.1.2 *Single-Operator Precision (Repeatability)*—Duplicate results obtained by the same operator using the same equipment in the same laboratory shall not be considered suspect unless the difference in the duplicate results, expressed as a percent of their mean, exceeds the values given in Table 3, Column 3.

15.1.3 *Multilaboratory Precision (Reproducibility)*—Two results submitted by two different operators testing the same material in different laboratories shall not be considered suspect unless the difference in the results, expressed as a percent of their mean, exceeds the values given in Table 3, Column 3.

TABLE 3 Precision Estimates

Condition	Coefficient of Variation (1s%) ^A	Acceptable Range of Two Test Results (d2s%) ^A
Single-Operator Precision:		
Original Binder: $G^*/\sin \delta$ (kPa)	2.3	6.4
RTFO/TFO Residue: $G^*/\sin \delta$ (kPa)	3.2	9.0
PAV Residue: $G^*\sin \delta$ (kPa)	4.9	13.8
Multilaboratory Precision:		
Original Binder: $G^*/\sin \delta$ (kPa)	6.0	17.0
RTFO/TFO Residue: $G^*/\sin \delta$ (kPa)	7.8	22.2
PAV Residue: $G^*\sin \delta$ (kPa)	14.2	40.2

^A These values represent the 1s% and d2s% limits described in Practice C 670.

15.2 *Bias*—Since there is no acceptable reference value the bias for this test method cannot be determined.

16. Keywords

16.1 asphalt binder; complex modulus; DSR; dynamic shear rheometer

ANNEX

(Mandatory Information)

A1. USE OF REFERENCE FLUID

A1.1 *Source of Reference Fluid*—An organic polymer produced by Cannon Instrument Company as Viscosity Standard N2700000SP has been found suitable as reference fluid for verifying the calibration of the DSR. The viscosity of the fluid, as determined from NIST-traceable capillary viscosity measurements, is approximately 270 Pa·s at 64°C . However, the viscosity of the fluid varies from one lot to the next. The lot-specific viscosity is printed on the label of the bottle.

A1.2 *Cautions in Using Reference Fluid*—Some items of caution when using the reference fluid are:

A1.2.1 The fluid cannot be used to verify the accuracy of the phase angle measurement.

A1.2.2 The fluid must not be heated as heating can degrade the fluid causing a change in its viscosity.

A1.2.3 The fluid should be used for verification only after the DSR temperature measurements are verified.

A1.2.4 The fluid cannot be used to calibrate the torque transducer. The manufacturer or other qualified service person-

nel using a calibration device designed specifically for the rheometer should perform the calibration. These calibration devices are typically not available in operating laboratories.

A1.2.5 When tested at 10 rad/s, the reference fluid should only be used between 58 and 64°C .

A1.2.6 Bubbles in the fluid will have a dramatic effect on the measured value of the complex viscosity. The fluid in the bottle should be free of bubbles and care must be taken not to introduce bubbles when preparing test specimens. Recommended procedures for preparing test specimens are given in A1.4.

A1.3 *Calculation of G^* from Steady-State Viscosity Measurements*—Among the different methods for converting between dynamic and steady-state viscosity of polymers, the most popular and most successful is the so-called Cox-Merz empirical rule. The rule leads, in simplified terms, to the following approximation (4)⁵:

$$G^*/\omega \sim \eta \quad (\text{A1.1})$$

where:

- G^* = the complex modulus,
- ω = the angular frequency in radians/s, and
- η = the shear-rate independent capillary viscosity as reported by the supplier of the reference fluid.

A1.3.1 For this rule to apply the measurements must be in the viscous region where the phase angle approaches 90°. The value of the complex modulus is then simply 10 times the value of the capillary viscosity. For example, if the capillary viscosity is 270 000 mPa·s the complex modulus is:

$$\begin{aligned} G^*, \text{kPa} &\approx (270\,000 \text{ mPa}\cdot\text{s}) (1 \text{ kPa} / 1\,000\,000 \text{ mPa}) (10 \text{ rad/s}) \\ &= 2.70 \text{ kPa}\cdot\text{rad} \end{aligned} \quad (\text{A1.2})$$

A1.3.2 The reference fluid behaves as a viscous fluid at 64°C and above and provides very accurate estimates of G^* above 64°C (3). At temperatures below 58°C the fluid gives incorrect values for G^* with the error increasing as the temperature departs from 64°C. At 64°C and above G^* divided by the frequency in radians per second should be no more than 3 % different than the viscosity printed on the bottle label. If this is not the case, then the torque calibration should be considered suspect.

A1.4 Methods for Transferring the Fluid to the Test Plates—Three different methods are recommended for transferring the fluid to the test plates: the glass rod method (see A1.4.1), the spatula method (see A1.4.2), and a direct method where a removable test plate is held in direct contact with the fluid in the bottle (see A1.4.3).

A1.4.1 Glass Rod Method (see Fig. A1.1)—In this method a glass rod is inserted into the fluid and rotated (Step 1) while in the fluid. Continue rotating the rod and pull it slowly from the fluid (Step 2) carrying a small mass of the fluid with the rod. Touch the mass to the plate (Step 3) to transfer the fluid to the plate.

A1.4.2 Spatula Method (see Fig. A1.2)—When carefully used, a spatula may be used to transfer the fluid. Special care must be taken not to trap air as the material is scooped from the



FIG. A1.1 Using a Glass Rod to Place the Reference Fluid on the Plate

bottle, Step 1. Smear the mass on the spatula onto the plate (Step 2) and cut the mass from the spatula by drawing the spatula across the edge of the plate (Step 3). This method appears to be the most difficult to implement and is the least recommended of the three methods.

A1.4.3 Direct Touch Method (see Fig. A1.3)—If the rheometer is equipped with plates that may be removed and reinstalled without affecting the gap reference, remove one of the plates and touch the surface of the plate to the surface of the fluid in the bottle (Step 1). Pull the plate from the bottle, bringing a mass of the fluid along with the plate (Step 2). Invert the plate and allow the fluid to flow out into a mushroom shape (Step 3).

A1.5 Proceed immediately to 11.4 to trim the reference fluid specimen and 11.5 to form the bulge.

A1.6 Proceed with testing the reference fluid specimen as described in Section 12.



FIG. A1.2 Using a Spatula to Place the Reference Fluid on the Plate

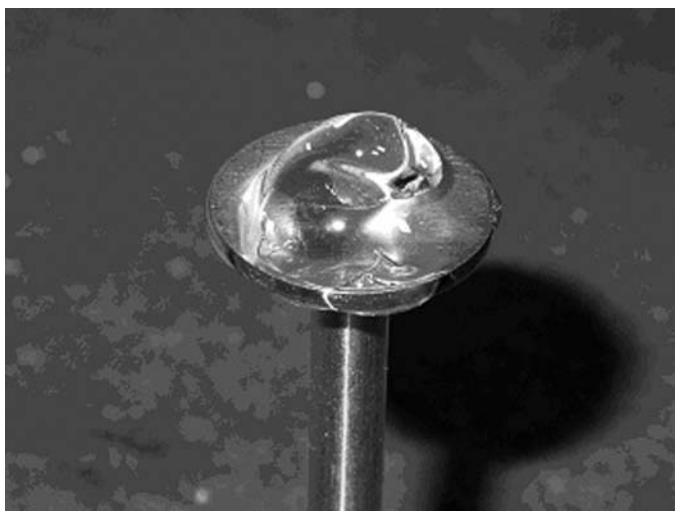


FIG. A1.3 Direct Touch Method to Place the Reference Fluid on the Plate

APPENDIXES

(Nonmandatory Information)

X1. TESTING FOR LINEARITY

X1.1 Scope—This procedure is used to determine whether an unaged asphalt binder exhibits linear or non-linear behavior at the upper grading temperature, for example, 52, 58, 64, 70, 76, or 82°C. The determination is based on the change in complex shear modulus at 10 rad/s when the strain is increased from 2 percent to 12 percent.

X1.2 Procedure:

X1.2.1 Prepare the DSR in accordance with Section 10 of this standard.

X1.2.2 Prepare a test specimen for testing with 25-mm plates as per Section 11 of this standard. Select the test temperature as the upper grading temperature for the binder in question.

X1.2.3 Determine the complex shear modulus at 2 and 12 percent strain following the test procedure described in Section 12 except as noted below. Always start with the lowest strain and proceed to the next larger strain.

X1.2.3.1 Strain Controlled Rheometers—If the software provided with the DSR will automatically conduct tests at multiple strains, program the DSR to obtain the complex shear modulus at strains of 2, 4, 6, 8, 10, and 12 percent. If this automatic feature is not available, test by manually selecting strains of 2, 4, 6, 8, 10, and 12 percent strain.

X1.2.3.2 For Stress Controlled Rheometers, compute the starting stress based on the complex shear modulus, G^*_G and shear stress, τ_G as determined at the upper grading temperature during the grading of the binder. At this temperature the complex modulus, G^*_G will be ≥ 1.00 kPa and the shear stress, τ_G will be between 0.090 and 0.150 kPa (see Table 2). Calculate the starting stress as $\tau_G/6.00$ kPa. Increase the stress in five increments of $\tau_G/6.00$ kPa.

NOTE X1.1—Sample calculation: Assume a PG 64-22 grade asphalt binder with $G^*_G = 1.29$ kPa at 64°C and the stress, $\tau_G = 0.135$ kPa. The starting stress will be $1.35 \text{ kPa}/6 = 0.225$ kPa. Test at 0.225, 0.450, 0.675, 0.900, 1.13, 1.35 kPa starting with 0.225 kPa.

X1.3 Plot of Complex Modulus versus Strain—Prepare a plot of complex shear modulus versus percent strain as shown in Fig. 6.

X1.4 Calculations—Calculate the modulus ratio as the complex shear modulus at 12 percent strain divided by the complex shear modulus at 2 percent strain.

X1.5 Report—Report the following at each stress/strain level:

X1.5.1 Complex shear modulus (G^*) to three significant figures.

X1.5.2 Strain, nearest 0.1 percent.

X1.5.3 Frequency, nearest 0.1 rad/s.

X1.5.4 The ratio calculated by dividing the modulus at 12 % strain by the modulus at 2 % strain.

X1.6 Data Interpretation—The measurement was performed in the non-linear range of the material if the modulus ratio as calculated in X1.4 is < 0.900 and linear if ≥ 0.900 . If the measurement was performed in the non-linear range of the material the results obtained under DXXX will be considered as invalid for grading a binder according to Specification D 6373.

X2. CONTROL CHARTS

X2.1 Control Charts—Control charts are commonly used by various industries, including the highway construction industry, to control the quality of products. Control charts provide a means for organizing, maintaining and interpreting test data. As such, control charts are an excellent means for organizing, maintaining, and interpreting DSR verification test data. Formal procedures based on statistical principles are used to develop control charts and the decision processes that are part of statistical quality control (2).

X2.1.1 A quality control chart is simply a graphical representation of test data versus time. By plotting laboratory measured values for the reference fluid in a control chart format it is easy to see when:

X2.1.1.1 The measurements are well controlled and both the device and the operator are performing properly.

X2.1.1.2 The measurements are becoming more variable with time, possibly indicating a problem with the test equipment or the operator.

X2.1.1.3 The laboratory measurements for the fluid are, on the average above or below the target (reference fluid) value.

X2.1.2 Many excellent software programs are available for generating and maintaining control charts. Some computer-based statistical analysis packages contain procedures that can be used to generate control charts. Spreadsheets such as Microsoft's Excel (example below) can also be used to generate control charts and, of course, control charts can be generated manually.

X2.1.3 Care in Selecting Data—Data used to generate control charts should be obtained with care. The idea of randomness is important but need not become unnecessarily complicated. An example will show why a random sample is

needed. A laboratory always measures the reference fluid at the start of the shift or workday. These measurements could be biased by start-up errors such as a lack of temperature stability when the device is first turned on. The random sample ensures that the measurement is representative of the process or the material being tested. Said another way, a random sample has an equal chance of being drawn as any other sample. A measurement or sample always taken at the start or end of the day, or just before coffee break, does not have this chance.

X2.2 Example—The power of the control chart is illustrated in Table X2.1 using the verification data obtained for the DSR. Other DSR verification data suitable for a quality control chart presentation includes measurements for determining the temperature correction, calibrating the electronic thermometer, and maintaining data from internally generated asphalt binder reference samples. For this example the reported viscosity for the reference fluid is 271 Pa·s and hence the calculated value for G^* is 2.71 kPa. This value for G^* is labeled as “ G^* from Reference Fluid” in Fig. X2.1. The laboratory should obtain this value on the average if there is no laboratory bias.

X2.2.1 Comparison of 22-week Laboratory Average for G^* with Value Calculated from Reference Fluid—The 22-week average of the laboratory measurements is labeled as “22 Week Laboratory Average” in Fig. X2.1. Over the 22 weeks for which measurements were made the average was 2.73 kPa. This compares favorably with the calculated reference value, 2.71 kPa, differing on the average by only 0.7 percent. There appears to be little laboratory bias in this data.

X2.2.2 Comparison of CV of Laboratory Measurements with Round Robin CV—From a previous round robin study, the

TABLE X2.1 Sample Test Data

Week	Measured G*, kPa
1	2.83
2	2.82
3	2.77
4	2.72
5	2.69
6	2.72
7	2.77
8	2.75
9	2.71
10	2.82
11	2.66
12	2.69
13	2.75
14	2.69
15	2.73
16	2.77
17	2.72
18	2.67
19	2.66
20	2.78
21	2.74
22	2.69
Average	2.73
Std. Dev.	0.051
CV, %	1.86

within laboratory standard deviation (d_{1s}) for the fluid was reported as 0.045 (CV = 1.67 %) (3). The 22-week standard deviation for the measured values of G^* is 0.051 (CV = 1.86 %), as compared to 0.045 (CV = 1.67 %) reported from the round robin. However, it should be pointed out that the 22-week CV, 1.86 percent, also includes day-to-day variability, a component of variability not included in the round-robin d_{1s} value. Based on this information the variability of the laboratory measurements are acceptable.

X2.2.3 Variability of Measured Values—In Fig. X2.1, the value of G^* calculated from the reference fluid is shown as a solid line. Also shown are two dotted lines that represent the G^* calculated from the reference fluid $\pm 2d_{1s}$ where d_{1s} is the value from the round robin. The calculated reference value for the fluid is 2.71 kPa and the standard deviation is 0.045. Thus, a deviation of $2d_{1s}$ gives values of (2):

$$2.71 \text{ kPa} \pm (2)(0.045) = 2.80 \text{ kPa}, 2.62 \text{ kPa} \quad (\text{X2.1})$$

X2.2.3.1 If the laboratory procedures are under control, the equipment is properly calibrated, and there is no laboratory bias, 95 percent of the measurements should fall within the limits 2.62 kPa and 2.80 kPa. Laboratory measurements outside this range are suspect and the cause of the outlier should be investigated. The outlier may be the result of either testing variability or laboratory bias. The measurement from week 10 in Fig. X2.1 falls outside the $\pm 2d_{1s}$ limits and is cause for concern such that testing procedures and verification should be investigated.

X2.2.3.2 If a measurement deviates from the target, in this case G^* from the reference fluid, by more than $\pm 3d_{1s}$, corrective action should be initiated. The $\pm 3d_{1s}$ limits 99.7 percent of the measured values if the laboratory procedures are under control and the equipment is properly calibrated.

X2.2.4 Trends in Measured Values—The control chart can also be used to identify unwanted trends in the data. For example from weeks one to five a steady decrease in the measured value is observed. This is cause for concern and the reason for the trend should be investigated. More sophisticated rules for analyzing trends in control charts can be found elsewhere (2).

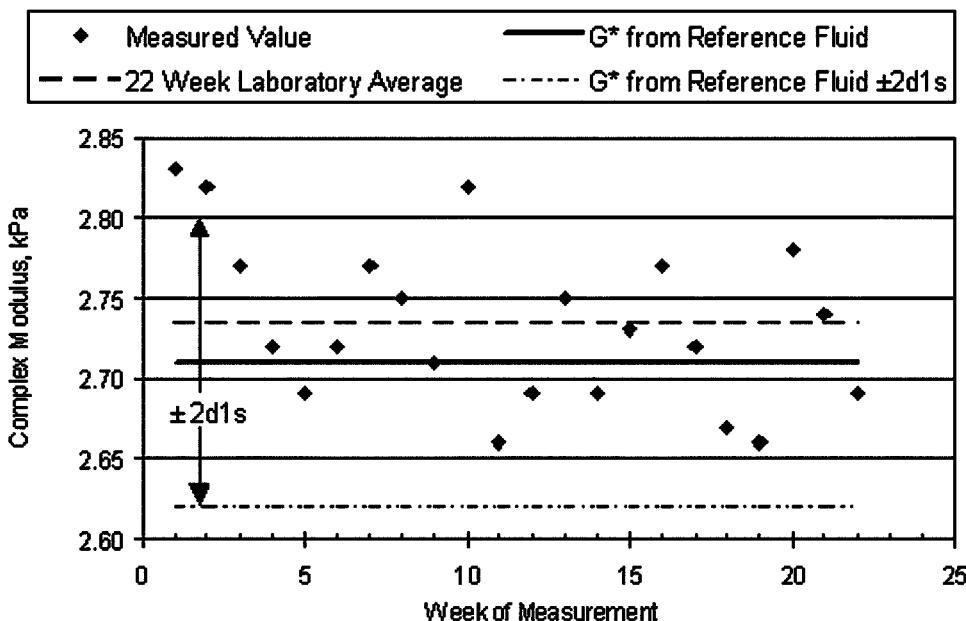


FIG. X2.1 Control Chart

X3. SELECTION OF GAP CLOSURE TO OBTAIN BULGE

X3.1 Need for Accurate Measurement of Specimen Diameter—The accuracy of the DSR measurements depends upon an accurate measurement of the diameter of the test specimen. The diameter of the test specimen is assumed equal to the diameter of the test plates. For this reason, the trimming of excess binder and the final closure of the gap to produce a slight bulge in the test specimen are critical steps in the DSR test procedure. When the gap is closed to its final dimension, the bulge must be of sufficient size to compensate for any shrinkage in the binder and consequently avoiding a concave surface as shown in Fig. X3.1. The diameter of the test specimen in Fig. X3.1 approaches d , rather than d' , the diameter of the plate. The modulus G^* is calculated according to the following equation:

$$|G^*| = (2h/\pi r^4) \cdot (\tau/\Theta) \quad (X3.1)$$

where:

G^* = complex modulus,
 τ = torque applied to test specimen,
 h = thickness of test specimen,
 Θ = angular rotation, radians, and
 r = radius of test plate.

X3.1.1 According to Eq X3.1 the modulus depends upon the radius (or diameter) raised to the fourth power. Therefore, a small concavity in the outer surface of the test specimen, as shown in Fig. X3.1, will have a large effect on the measured modulus because the actual specimen diameter will be less than the plate diameter. For a given amount of concavity, the effect on the measured modulus is greater for the 8-mm plate than the 25-mm plate. A more desirable result is a slight bulge as illustrated in Fig. X3.2. Shear stresses are not transferred directly from the plate to the overhanging binder and therefore the effect of a slight bulge on the measured modulus is much

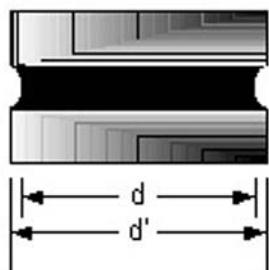


FIG. X3.1 Concave Surface Resulting from Insufficient Closure after Trimming

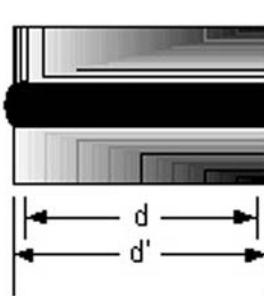


FIG. X3.2 Proper Bulge

less than a slight concavity. It should be noted that errors in the diameter of the test specimen do not affect the measured values of the phase angle.

X3.2 Recommended Gap Closure Values—Recommended values for the gap closure required to form a bulge *at the test temperature* similar to the bulge illustrated in Fig. X3.2 are given in 11.5 as 50 µm and 100 µm for the 25-mm and 8-mm plates, respectively. Although these values may be appropriate for many rheometers, they may not be appropriate for all rheometers. The applicability of these values to a specific rheometer may be determined by preparing a test specimen using the recommended closure and observing the shape of the bulge *after* the final closure of the gap and *after* the test specimen is at the test temperature. If the recommended closure values do not give an appropriate bulge, the recommended closure values should be adjusted as appropriate.

X3.2.1 Proper and improper bulges are shown in Figs. X3.3-X3.5. A magnifying glass is useful for examining the shape of the bulge. Regardless of the closure required to produce a desirable bulge, the actual gap should be used in the calculations.

X3.3 Factors Affecting Bulge Development:

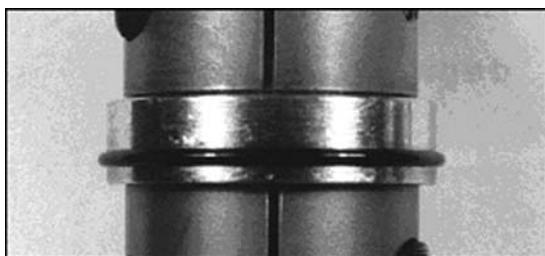


FIG. X3.3 Good Bulge Size

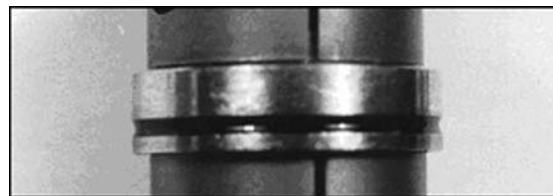


FIG. X3.4 Concave “Bulge”

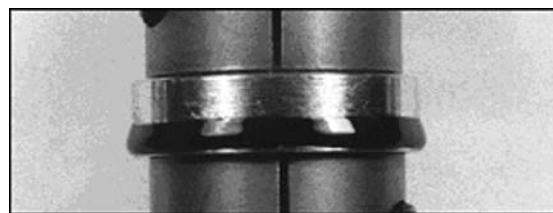


FIG. X3.5 Oversized Bulge

X3.3.1 A number of factors can affect the bulge formed at the test temperature. These include:

X3.3.1.1 The amount of closure used to create the bulge,

X3.3.1.2 The difference in temperature between the trimming temperature, the temperature at which the bulge is created, and the test temperature,

X3.3.1.3 Thermal expansion-contraction characteristics of the rheometer, and

X3.3.1.4 Thermal contraction and expansion of the asphalt binder.

X3.3.2 A concave surface is more likely to form at the intermediate temperatures than at the upper test temperatures (8-mm plate rather than the 25-mm plate). In fact, at the higher test temperatures excessive material can be squeezed from the plates as shown in Fig. X3.5. This situation should also be avoided and may require gap closures somewhat less than the recommended values.

X4. DETERMINATION OF TIME TO THERMAL EQUILIBRIUM

X4.1 Reason for Determining Time Required To Obtain Thermal Equilibrium—After the test specimen has been mounted in the DSR, it takes it some time for the asphalt binder between the test plates to reach thermal equilibrium. Because of thermal gradients within the test plates and test specimen, it may take longer for the test specimen to come to thermal equilibrium than the time indicated by the DSR thermometer. Therefore, it is necessary to experimentally determine the time required for the test specimen to reach thermal equilibrium.

X4.1.1 The time required to obtain thermal equilibrium varies for different rheometers. Factors that affect the time required for thermal equilibrium include:

X4.1.1.1 Design of the rheometer and whether air or liquid is used as a heating-cooling medium,

X4.1.1.2 Difference between ambient temperature and the test temperature, different when testing below room temperature and above room temperature,

X4.1.1.3 Difference in temperature between the trimming and test temperature, and

X4.1.1.4 Plate size, different for the 8-mm and 25-mm plate.

X4.1.2 It is not possible to specify a single time as the time required to obtain thermal equilibrium. For example, thermal equilibrium is reached much quicker with liquid-controlled rheometers than with air-cooled rheometer. This requires that the time to thermal equilibrium be established for individual rheometers, typical trimming and testing temperatures, and testing conditions.

X4.2 Method to Determine the Time Required to Obtain Thermal Equilibrium—A reliable estimate of the time required for thermal equilibrium can be obtained by monitoring the DSR temperature and the complex modulus of a sample

mounted between the test plates. Because the modulus is highly sensitive to temperature, it is an excellent indicator of thermal equilibrium.

X4.2.1 Mount a binder sample in the DSR and trim in the usual manner. Create a bulge and bring the test chamber or fluid to the test temperature.

X4.2.2 Operate the rheometer in a continuous mode at 10 rad/s using an unmodified asphalt binder—one that does not change modulus with repeated shearing. Use the smallest strain value that gives good measurement resolution.

X4.2.3 Record the modulus at 30 second time intervals and plot the modulus versus time (see Fig. 5).

X4.2.4 The time to reach thermal equilibrium is the time required to reach a constant modulus. Typically, this time will be greater than the time required to reach a constant reading on the DSR thermometer.

X4.3 Because the time required to reach thermal equilibrium will vary with the test temperature, the time to thermal equilibrium should be established separately for both intermediate and high temperature measurements.

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