Standard Method of Test for

Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)

AASHTO Designation: T 315-12

AASHTO

1. SCOPE

1.1. This test method covers the determination of the dynamic shear modulus and phase angle of asphalt binder when tested in dynamic (oscillatory) shear using parallel plate test 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 6 and 88°C at an angular frequency of 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 binder.

1.2. This standard is appropriate for unaged material or material aged in accordance with T 240 and R 28.

1.3. Particulate material in the asphalt binder is limited to particles with longest dimensions less than 250 µm.

1.4. *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user of this procedure to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use*.

2. REFERENCED DOCUMENTS

2.1. *AASHTO Standards*:

- M 320, Performance-Graded Asphalt Binder
- R 28, Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel (PAV)
- R 29, Grading or Verifying the Performance Grade (PG) of an Asphalt Binder
- R 66, Sampling Bituminous Materials
- T 240, Effect of Heat and Air on a Moving Film of Asphalt Binder (Rolling Thin-Film Oven Test)
- T 314, Determining the Fracture Properties of Asphalt Binder in Direct Tension (DT)

2.2. *ASTM Standards*:

- C670, Standard Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials
- D2170/D2170M, Standard Test Method for Kinematic Viscosity of Asphalts (Bitumens)

- D2171/D2171M, Standard Test Method for Viscosity of Asphalts by Vacuum Capillary Viscometer
- E1, Standard Specification for ASTM Liquid-in-Glass Thermometers
- E77, Standard Test Method for Inspection and Verification of Thermometers
- E563, Standard Practice for Preparation and Use of an Ice-Point Bath as a Reference Temperature
- E644, Standard Test Methods for Testing Industrial Resistance Thermometers

2.3. *Deutsche Industrie Norm (DIN) Standard*:

- 43760, Industrial Platinum Resistance Thermometers and Platinum Temperature Sensors

3. TERMINOLOGY

3.1. *Definitions*:

3.1.1. *asphalt binder*—an asphalt-based cement that is produced from petroleum residue either with or without the addition of nonparticulate organic modifiers.

3.2. *Descriptions of Terms Specific to This Standard*:

3.2.1. *annealing*—heating the binder until it is sufficiently fluid to remove the effects of steric hardening.

3.2.2. *calibration*—process of checking the accuracy and precision of a device using NIST-traceable standards and making adjustments to the device where necessary to correct its operation or precision and accuracy.

3.2.3. *complex shear modulus (G^*)*—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.

3.2.4. *dummy test specimen*—a specimen formed between the dynamic shear rheometer (DSR) test plates from asphalt binder or other polymer to measure the temperature of the asphalt binder held between the plates. The dummy test specimen is used solely to determine temperature corrections.

3.2.5. *linear viscoelastic*—within the context of this specification refers to a region of behavior in which the dynamic shear modulus is independent of shear stress or strain.

3.2.6. *loading cycle*—a unit cycle of time for which the test sample is loaded at a selected frequency and stress or strain level.

3.2.7. *loss shear modulus (G'')*—the complex shear modulus multiplied by the sine of the phase angle expressed in degrees. It represents the component of the complex modulus that is a measure of the energy lost (dissipated during a loading cycle).

3.2.8. *molecular association*—a process where associations occur between asphalt binder molecules during storage at ambient temperature. Often called steric hardening in the asphalt literature, molecular associations can increase the dynamic shear modulus of asphalt binders. The amount of molecular association is asphalt specific and may be significant even after a few hours of storage.

3.2.9. *oscillatory shear*—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 by about zero in a sinusoidal manner.

3.2.10. *parallel plate geometry*—refers to a testing geometry in which the test sample is sandwiched between two relatively rigid parallel plates and subjected to oscillatory shear.

3.2.11. *phase angle (δ)*—the angle in radians 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.

3.2.12. *portable thermometer*—an electronic device that consists of a temperature detector (probe containing a thermocouple or resistive element), required electronic circuitry, and readout system.

3.2.13. *reference thermometer*—a NIST–traceable liquid-in-glass or electronic thermometer that is used as a laboratory standard.

3.2.14. *steric hardening*—see molecular association.

3.2.15. *storage shear modulus (G′)*—the complex shear modulus multiplied by the cosine of the phase angle expressed in degrees. It represents the in-phase component of the complex modulus that is a measure of the energy stored during a loading cycle.

3.2.16. *temperature correction*—difference in temperature between the temperature indicated by the DSR and the test specimen as measured by the portable thermometer inserted between the test plates.

3.2.17. *thermal equilibrium*—is reached when the temperature of the test specimen mounted between the test plates is constant with time.

3.2.18. *verification*—process of checking the accuracy of a device or its components against an internal laboratory standard. It is usually performed within the operating laboratory.

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 a dynamic shear rheometer and parallel plate test 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 6 and 88°C at an angular frequency of 10 rad/s, dependent upon the grade, test temperature, and conditioning (aging) of the asphalt binder.

4.3. Test specimens 1 mm thick by 25 mm in diameter or 2 mm thick by 8 mm in diameter are formed between parallel metal plates. During testing, one of the parallel plates is oscillated with respect to the other at preselected frequencies and rotational deformation amplitudes (strain control) (or torque amplitudes [stress control]). The required stress or strain amplitude depends upon the value of the complex shear modulus of the asphalt binder being tested. The required amplitudes have been selected to ensure that the measurements are within the region of linear behavior.

4.4. The test specimen is maintained at the test temperature to within ±0.1°C 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.

4.5. Oscillatory loading frequencies using this standard can range from 1 to 100 rad/s using a sinusoidal waveform. 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 equipment 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.

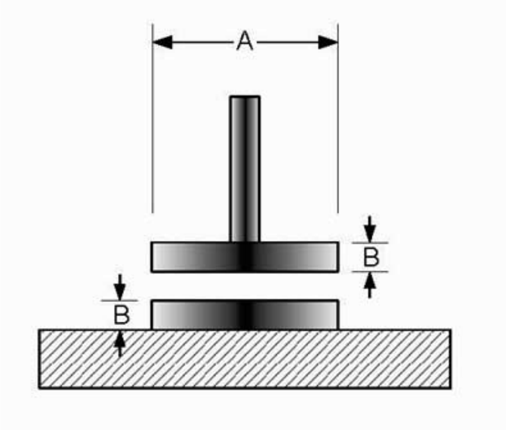
5.3. The complex modulus and the phase angle are used to calculate performance-related criteria in accordance with M 320.

6. APPARATUS

6.1. *Dynamic Shear Rheometer (DSR) Test System*—Consisting of parallel metal plates, an environmental chamber, a loading device, and a control and data acquisition system.

6.1.1. *Test Plates*—Stainless steel or aluminum plates with smooth ground surfaces. One 8.00 ± 0.02 mm in diameter and one 25.00 ± 0.05 mm in diameter (Figure 1). The base plate in some rheometers is a flat plate. A raised portion, a minimum of 1.50 mm high, with the same radius as the upper plate is required. The raised portion makes it easier to trim the specimen and may improve test repeatability.

Note 1—To obtain correct data, the upper and lower plates should be concentric with each other. At 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 operation may be checked visually or with a dial gauge held in contact with the edge of the moveable plate while it is being rotated. There are two values that determine the operating behavior of a measuring system: centricity (horizontal wobble) and runout (vertical wobble). Typically, wobble can be detected if it is greater than ±0.02 mm. For a new system, a wobble of ±0.01 mm is typical. If the wobble grows to more than ±0.02 mm with use, it is recommended that the instrument be serviced by the manufacturer.



Dimension	8-mm Nominal	25-mm Nominal
A	8 ± 0.02 mm	25 ± 0.05 mm
B	≥1.50 mm	≥1.50 mm

Figure 1—Plate Dimensions

6.1.2. *Environmental Chamber*—For controlling the test temperature, by heating or cooling (in steps or ramps), to maintain a constant specimen environment. 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 such as water, conditioned gas such as nitrogen, or by a suitable arrangement of solid-state Peltier elements surrounding the sample. When forced air is used, a suitable drier must be included to prevent condensation of moisture on the plates and fixtures and, if operating below freezing temperatures, the formation of ice. The environmental chamber and the temperature controller shall control the temperature of the specimen, including thermal gradients within the sample, to an accuracy of ±0.1°C. The chamber shall completely enclose the top and the bottom plates to minimize thermal gradients.

Note 2—A circulating bath unit, separate from the DSR, that pumps the bath fluid through the test chamber may be required if a fluid medium is used. The flow rate of the bath media should not be modified once the temperature settings have been adjusted to the desired value. Media lines should be periodically inspected and cleaned or replaced if necessary to remove obstructions

6.1.2.1. *Temperature Controller*—Capable of maintaining specimen temperatures within ±0.1°C for test temperatures ranging from 3 to 88°C.

6.1.2.2. *Internal Temperature Detector for the DSR*—A platinum resistance thermometer (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 3 to 88°C, and with a resolution of 0.1°C (see Note 3). This 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. The PRT shall be calibrated as an integral unit with its respective meter or electronic circuitry.

Note 3—PTRs meeting DIN Standard 43760 (Class A) or equal are recommended for this purpose.

6.1.3. *Loading Device*—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. If the load is strain controlled, the loading device shall apply a cyclic torque sufficient to cause an angular rotational strain accurate to within 100 μ rad of the strain specified. If the load is stress controlled, the loading device shall apply a cyclic torque accurate to within 10 mN·m of the torque specified. Total system compliance at 100 N·m of torque shall be less than 2 mrad/N·m. The manufacturer of the device shall certify that the frequency, stress, and strain are controlled and measured with an accuracy of one percent or less in the range of this measurement.

6.1.4. *Control and Data Acquisition System*—Capable of providing a record of temperature, frequency, deflection angle, and torque. Devices used to measure these quantities shall meet the accuracy requirements specified in Table 1. In addition, the system shall calculate and record the shear stress, shear strain, complex shear modulus (G^*), and phase angle (δ). The system shall measure and record G^*, in the range of 100 Pa to 10 MPa, to an accuracy of 1.0 percent or less, and the phase angle, in the range of 0 to 90 degrees, to an accuracy of 0.1 degree.

Table 1—Control and Data Acquisition System Requirements

Property	Accuracy
Temperature	0.1°C
Frequency	1%
Torque	10 mN·m
Deflection angle	100 μrad

6.2. *Specimen Mold (Optional)*—The overall dimensions of the silicone rubber mold for forming asphalt binder test specimens may vary but the thickness shall be greater than 5 mm. If the mold is a single sample mold, the following dimensions have been found suitable: For a 25-mm test plate with a 1-mm gap, a mold cavity approximately 18 mm in diameter and 2.0 mm deep. For an 8-mm test plate with a 2-mm gap, a mold cavity approximately 8 mm in diameter and 2.5 mm deep.

6.3. *Specimen Trimmer*—With a straightedge at least 4 mm wide.

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

6.5. *Cleaning Solvents*—Mineral oil, citrus-based solvents, mineral spirits, toluene, or similar solvent as required for cleaning the plates. Acetone for removing the solvent residue from the surfaces of the plates is also necessary.

6.6. *Reference Thermometer*—Either NIST–traceable liquid-in-glass thermometer(s) or NIST–traceable electronic thermometric device(s). This temperature standard shall be used to standardize the portable thermometer (Section 9.3).

6.6.1. *Liquid-in-Glass Thermometer*—NIST-traceable thermometer(s) with a suitable range and subdivisions of 0.1°C. The thermometer(s) shall be a partial immersion thermometer(s) within an ice point and standardized in accordance with ASTM E563.

6.6.1.1. *Optical Viewing Device (Optional)*—For use with liquid-in-glass thermometers that enhances readability and minimizes parallax when reading the liquid-in-glass reference thermometer.

6.6.2. *Electronic Thermometer*—Incorporating a resistive detector (Note 3) with an accuracy of ±0.05°C and a resolution of 0.01°C. The electronic thermometer shall be standardized at least once per year using a NIST–traceable reference standard in accordance with ASTM E77.

6.7. *Portable Thermometer*—A standardized portable thermometer consisting of a 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. The reference thermometer (see Section 6.6) may be used for this purpose if its detector fits within the dummy specimen as required by Section 9.4.1 or 9.4.2.

7. HAZARDS

7.1. Standard laboratory caution should be used in handling the hot asphalt binder when preparing test specimens.

8. PREPARATION OF APPARATUS

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

8.2. Inspect the surfaces of the test plates and discard any plates with jagged or rounded edges or deep scratches. 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 acetone. If necessary, use a dry cotton swab or soft cloth to ensure that no moisture condenses on the plates.

8.3. Mount the cleaned and inspected test plates on the test fixtures and tighten firmly.

8.4. Select the testing temperature according to the grade of the asphalt binder or according to the preselected testing schedule (see Note 4). Allow the DSR to reach a stabilized temperature within ±0.1°C of the test temperature.

Note 4—M 320 and R 29 provide guidance on the selection of test temperatures.

8.5. With the test plates at the test temperature or the middle of the expected testing range, establish the zero gap level (1) by manually spinning the moveable plate, and while the moveable plate is spinning, close the gap until the removable plate touches the fixed plate (the zero gap is reached when the plate stops spinning completely), or (2) for rheometers with normal force transducers, by closing the gap and observing the normal force and after establishing contact between the plates, setting the zero gap at approximately zero normal force.

Note 5—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, the gap should be set at the middle of the expected range of test temperatures. For most instruments, no gap adjustment is needed as long as the test temperature is within ±12°C of the temperature at which the gap is set. 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.

8.6. Once the zero gap is established as per Section 8.5, move the plates apart to approximately 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.

8.7. To preheat 25-mm plates, bring the test plates to the test temperature or the lowest test temperature if testing is to be conducted at more than one temperature. To preheat 8-mm plates, bring the plates to between 34 and 46°C. Move the plates apart and establish a gap setting of 1.05 mm (for 25-mm diameter test specimens) or 2.10 mm (for 8-mm diameter test specimens).

Note 6—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. For highly modified asphalt binders only, higher preheat temperatures may be used.

9. VERIFICATION AND CALIBRATION

9.1. Verify the DSR and its components at least every 6 months and when the DSR or plates are newly installed, when the DSR is moved to a new location, or when the accuracy of the DSR or any of its components is suspect. Four items require verification—the test plate diameter, DSR torque transducer, portable thermometer, and DSR test specimen temperature. Verify the DSR temperature transducer before verifying the torque transducer.

9.2. *Verification of Plate Diameter*—Measure the diameters to the nearest 0.01 mm. Maintain a log of the measured diameters as part of the laboratory quality control program so that the measurements are clearly identified with the specific plates. Enter the actual measured dimensions into the DSR software for use in calculations. If the top and bottom plates differ in diameter, enter the smaller of the two measured diameters.

Note 7—An error of ±0.05 mm in the diameter of the plate results in a 0.8 percent error in the complex modulus for the 25-mm plate. For the 8-mm plate, errors in diameter of ±0.01, ±0.02, and ±0.05 mm give respective errors in complex modulus of 0.5, 1.0, and 2.5 percent (see Figure 2).

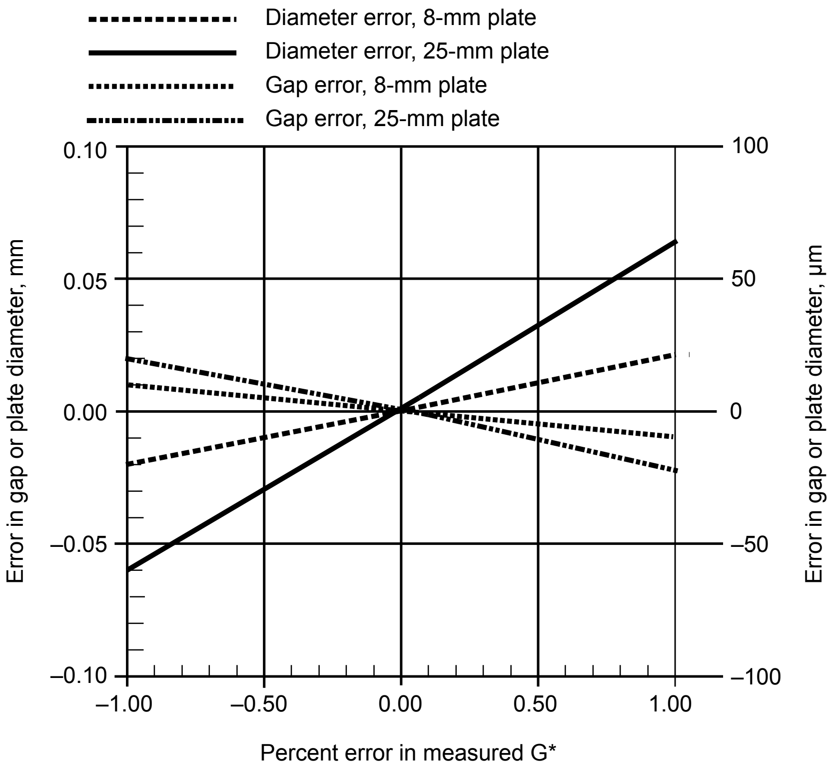


Figure 2—Effect of Error in Gap or Plate Diameter

9.3. *Verification of Portable Thermometer*—Verify the portable thermometer (used to measure the temperature between the test plates), using the laboratory reference thermometer. A portable thermometer shall be considered the combination of the meter (readout device) and the thermistor (temperature probe) as a single unit, and must be verified as such. If the reference thermometer (Section 6.6) is also used as a portable thermometer to measure the temperature between the test plates, it shall meet the requirements of Section 6.7.

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 (Note 8). Ensure that deionized water is used to prevent electrical conduction from occurring between the electrodes of the resistive temperature sensitive element. If deionized water is not available, encase the reference thermometer and detector of the portable thermometer in a waterproof plastic bag prior to placement in the bath. Obtain measurements at intervals of approximately 6°C over the range of test temperatures allowing the bath to reach thermal equilibrium at each temperature. If the readings of the portable thermometer 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 as part of the laboratory quality control program.

Note 8—A recommended procedure for the high-temperature range is to use a stirred water bath that is controlled within ±0.1°C such as the viscosity bath used for ASTM D2170/D2170M or D2171/D2171M. For a low-temperature bath, an ice bath or controlled-temperature bath may be used. 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.

Note 9—If the readings from the two devices differ by 0.5°C or more, the calibration or operation of the portable thermometer may be suspect, and it may need to be recalibrated or replaced. A continuing change in the temperature corrections with time may also make the portable thermometer suspect.

9.4. *Test Specimen Temperature Correction*—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). The DSR thermometer shall be checked at an interval no greater than six months. When these differences are 0.1°C or greater, determine a temperature correction by using a thermal detector mounted in a silicone rubber wafer (Section 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 (Section 9.4.2).

9.4.1. *Method Using Silicone Rubber Wafer*—For the entire range of test temperatures, 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 10) 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. Determine any needed temperature correction as per Section 9.4.3.

Note 10—Anti-seize compound available by that name at hardware and auto supply stores is much less apt to contaminate the circulating water than petroleum grease.

Note 11—The thickness of the silicone wafer should be measured with calipers to identify the actual thickness. The thickness can be used to set the gap for temperature.

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. Determine any needed temperature correction as per Section 9.4.3.

Note 12—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 Correction*—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 5-min period. Obtain additional measurements to include the entire temperature range that will be used for measuring the dynamic shear modulus.

9.4.4. *Plot Correction Versus Specimen Temperature*—Using the data obtained in Section 9.4, prepare a plot of the difference between the two temperature measurements versus the temperature measured with the portable thermometer (Figure 3). 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 test temperature between the plates as the test temperature. Alternatively, the instrument software may be written to incorporate these temperature corrections.

Note 13—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.

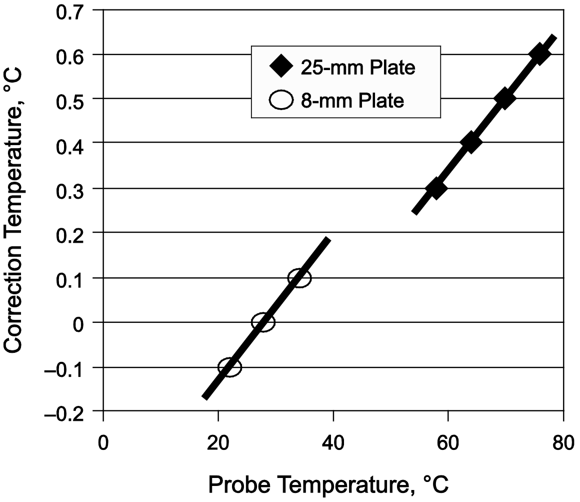


Figure 3—Determination of Temperature Correction

9.5. *Verification of DSR*—Verify the accuracy of the torque transducer and angular displacement transducer.

Note 14—A newly installed or reconditioned instrument should be verified on a weekly basis using the procedures in Section 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).

9.5.1. *Verification of Torque Transducer*—Verify the calibration of the torque transducer a minimum of once every six months using a reference fluid or manufacturer-supplied fixtures when the calibration of the torque transducer is suspect 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*—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. Recommended practice for using the reference fluid is given in Appendix X3.

$$\text{Percent Variance} = \left[\frac{(\eta a - \eta b)}{\eta a} \right] \times 100 \qquad (1)$$

where:

ηa = the standard capillary viscosity as reported by the supplier of the reference fluid; and

ηb = the measured viscosity as calculated from the complex modulus, *G**, divided by the angular frequency in rad/s.

Note 15—A suitable reference fluid is available from Cannon Instrument Company as Viscosity Standard Number N2700000SP. The viscosity of the standard is reported in mPa·s. Convert the viscosity measurements to mPa·s before calculating the percent variance.

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 or when the calibration of the DSR is suspect. If suitable fixtures are not available, this requirement shall be waived.

9.5.3. If the DSR cannot be successfully verified according to Section 9.5, 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.

10. PREPARING SAMPLES AND TEST SPECIMENS

10.1. *Preparing Test Samples*—If unaged binder is to be tested, obtain test samples according to R 66.

10.1.1. *Degassing Prior to Testing*—If the asphalt binder is also being tested according to T 314 (DT) and has been conditioned according to T 240 (RTFO) and R 28 (PAV), degas the asphalt binder as described in R 28 prior to testing. Otherwise, degassing of the asphalt binder sample is not required.

10.1.2. Anneal the asphalt binder from which the test specimen is obtained by heating until sufficiently fluid to pour the required specimens. Annealing prior to testing removes reversible molecular associations (steric hardening) that occur during normal storage at ambient temperature. Avoid heating the binder samples above a temperature of 163°C; however, with some modified or heavily aged asphalt binders, pouring temperatures above 163°C may be required. Loosely cover the sample, and stir it occasionally during the heating process to ensure homogeneity and to remove air bubbles. Minimize the heating temperature and time to avoid hardening the sample.

Note 16—For neat asphalt binders, 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.

Note 17—For PAV aged samples, asphalt binder may be placed in a vacuum oven set at a maximum of 175°C for 40 min. Due to the poor heat transfer in the vacuum oven, the asphalt binder will not be overheated.

10.1.3. Cold material from storage containers must be annealed prior to usage. Structure developed during storage can result in overestimating the modulus by as much as 50 percent.

10.2. *Preparing Test Specimens*—Zero the gap as specified in Section 8. Carefully clean and dry the surfaces of the test plates so that the specimen will adhere to both plates uniformly and strongly. Heat the chamber to 34 to 46°C when using the 8-mm specimens. Heat the chamber to the test temperature or the beginning of the range (Note 6) when using 25-mm specimens. This requirement is to preheat the upper and lower plates to allow specimen adhesion to both plates. Prepare a test specimen using one of the methods specified in Section 10.3.1, 10.3.2, or 10.3.3.

10.3. Transfer asphalt binder to one of the test plates through pouring (Section 10.3.1), direct transfer (Section 10.3.2), or by use of a silicone mold (Section 10.3.3). Use a sufficient amount of asphalt binder so that trimming is required.

Note 18—Direct transfer and pouring are the preferred methods because the test results are less likely to be influenced by steric hardening than with the silicone mold method. Direct transfer and direct pouring result in higher asphalt binder temperatures when the plates and asphalt binder are brought into contact, thereby improving adhesion. For this reason, it is also important to bring the asphalt binder and plates into contact promptly after pouring or direct transfer.

10.3.1. *Pouring*—Only when using rheometers that are designed for removal of the plates without affecting the zero setting, remove the removable plate and, while holding the sample container approximately 15 mm above the test plate surface, pour the asphalt binder in the center of the upper test plate continuously until it covers the entire plate except for an approximate 2-mm wide strip at the perimeter (Note 19). Wait only long enough for the specimen to stiffen, to prevent movement, and then mount the test plate in the rheometer for testing.

Note 19—An eye dropper or syringe may be used to transfer the hot asphalt binder to the plate.

10.3.2. *Direct Transfer*—Transfer the hot asphalt binder to one of the plates using a glass or metal rod, spatula, or similar tool. Immediately after transferring the hot asphalt binder to one of the plates, proceed to Section 10.4 to trim the specimen and form the bulge.

Note 20—A small, narrow stainless steel spatula of the type used to weigh powders on an analytical balance has been found suitable for transferring the asphalt hot binder. When using a rod, form the test specimen with a twisting motion, using a mass of sufficient size. 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.

10.3.3. *Silicone Mold*—Pour the hot asphalt binder into a silicone rubber mold that will form a pellet having dimensions as required in Section 6.2. Allow the silicone rubber mold to cool to room temperature. The molds shall be covered while cooling to eliminate contamination. 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. 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.

10.3.3.1. The filled mold should 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. 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.

Note 21—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 use, the molds will become stained from the asphalt binder, making it difficult to remove the binder from the mold. If sticking becomes a problem, discard the mold.

Note 22—Some binder grades cannot be removed from the silicone mold without cooling. Materials such as PG 52-34, PG 46-34, and some PG 58-34 grades do not lend themselves to being removed from the mold at ambient temperatures. If the binder specimen cannot be removed from the mold without cooling, the direct transfer or pouring method may be used, or the filled mold may be chilled in a freezer or refrigerator for a maximum of 10 min to facilitate demolding the specimen.

10.4. *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 Section 10.5 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 23—The trimming tool should be at a temperature that is sufficiently hot as to allow trimming but not excessively hot as to burn the edge of the specimen. The trimming tool should also not be excessively cool as to snag or damage the edge of the test specimen.

Note 24—The gap should be set at the starting test temperature (Section 11.1.1) or at the middle of the expected range of test temperatures (Section 11.1.2). See Note 5 for guidance on setting the gap. Typically, reliable test results may be obtained with a single sample using temperatures within 12°C of the temperature at which the gap is set.

10.5. *Creating Bulge*—Immediately after 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 difference between the trimming temperature and test temperature. Recommended closure values for creating the gap are 0.05 mm for the 25-mm plate and 0.10 mm for the 8-mm plate. A recommended practice for verifying the gap closure required to produce an appropriate bulge is given in Appendixes X8, X9, and X10.

Note 25—The complex modulus is calculated with the assumption that the specimen diameter is equal to the plate diameter. If the asphalt 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 Appendixes X8, X9, and X10.

11. PROCEDURE

11.1. Bring the specimen to the test temperature ±0.1°C. See Note 4.

Note 26—The gap should be set at the starting test temperature (Section 11.1.1) or at the middle of the expected range of test temperatures (Section 11.1.2). See Note 5 for guidance on setting the gap. Typically, reliable test results may be obtained with a single sample, in an 8-mm to 25-mm plate, using temperatures within 12°C of the temperature at which the gap is set.

11.1.1. When testing a binder for compliance with M 320, select the test temperature from the appropriate table in M 320.

11.1.2. When conducting a temperature sweep, start at a midrange test temperature and increase or decrease the test temperature to cover the desired range of test temperatures. (See Sections 6 and 7 in R 29.)

11.2. Set the temperature controller to the desired test temperature, including any offset as required by Section 9.4.4. Allow the temperature indicated by the RTD to come to the desired temperature. The test shall be started only after the temperature has remained at the desired temperature ±0.1°C for at least 10 min.

Note 27—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 equilibrium time is described in Appendix X12.

11.3. *Strain Control Mode*—When operating in a strain-controlled mode, determine the strain value according to the value of the complex modulus. Control the strain within 20 percent of the target value calculated by Equation 2.

$$\gamma,\ \text{percent} = \frac{12.0}{(G^*)^{0.29}} \qquad (2)$$

where:

γ = shear strain in percent, and

G^* = complex modulus in kPa.

11.3.1. When testing specimens for compliance with M 320, select an appropriate strain value from Table 2. Software is available with the dynamic shear rheometers that will control the strain automatically without control by the operator.

Table 2—Target Strain Values

		Strain, percent	
Material	kPa	Target Value	Range
Original binder	1.0 $G^*/\sin\delta$	12	9 to 15
RTFO residue	2.2 $G^*/\sin\delta$	10	8 to 12
PAV residue	5000 $G^*\sin\delta$	1	0.8 to 1.2

11.4. *Stress Control Mode*—When operating in a stress-controlled mode, determine the stress level according to the value of the complex modulus. Control the stress within 20 percent of the target value calculated by Equation 3.

$$\tau = 12.0\left(G^*\right)^{0.71} \qquad (3)$$

where:

τ = shear stress in kPa, and

G^* = complex modulus in kPa.

11.4.1. When testing specimens for compliance with M 320, select an appropriate stress level from Table 3. Software is available with the dynamic shear rheometers that will control the stress level automatically without control by the operator.

Table 3—Target Stress Levels

		Stress, kPa	
Material	kPa	Target Level	Range
Original binder	1.0 $G^*/\sin\delta$	0.12	0.09 to 0.15
RTFO residue	2.2 $G^*/\sin\delta$	0.22	0.18 to 0.26
PAV residue	5000 $G^*\sin\delta$	50.0	40.0 to 60.0

11.5. When the temperature has equilibrated, condition the specimen by applying the required strain for a recommended 10 cycles or a required range of 8 to 16 cycles at a frequency of 10 rad/s (see Note 28). Obtain a test measurement by recording data for an additional recommended 10 cycles or a range of 8 to 16 cycles. Reduce the data obtained for the second set of cycles to produce a value for the complex modulus and phase angle. Typically a Fast Fourier Transform (FFT) is used to reduce the data. Multiple measurements may be obtained to verify that the sample is properly prepared. Disbonding between the plates and the binder or fracture in the sample can result in a decrease in the modulus with repeat measurements. Some asphalt binders may exhibit a reduced modulus with continued application of shear stresses (multiple measurements). The data acquisition system automatically acquires and reduces the data when properly activated. When conducting tests at more than one frequency, start testing at the lowest frequency and increase to the highest frequency.

Note 28—The standard frequency of 10 rad/s is used when testing binder for compliance with M 320.

11.6. The data acquisition system specified in Section 6.1.4 automatically calculates *G** and δ from test data acquired when properly activated.

11.7. Initiate the testing immediately after preparing and trimming the specimen. 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 4 h.

12. INTERPRETATION OF RESULTS

12.1. The dynamic modulus and phase angle depend upon the magnitude of the shear strain; the modulus and phase angle for both unmodified and modified asphalt binder decrease with increasing shear strain as shown in Figure 4. A plot such as that shown in Figure 4 can be generated by gradually increasing the load or strain amplitude, thereby producing a strain sweep. It is not necessary to generate such sweeps during normal specification testing; however, such plots are useful for verifying the limits of the linear region.

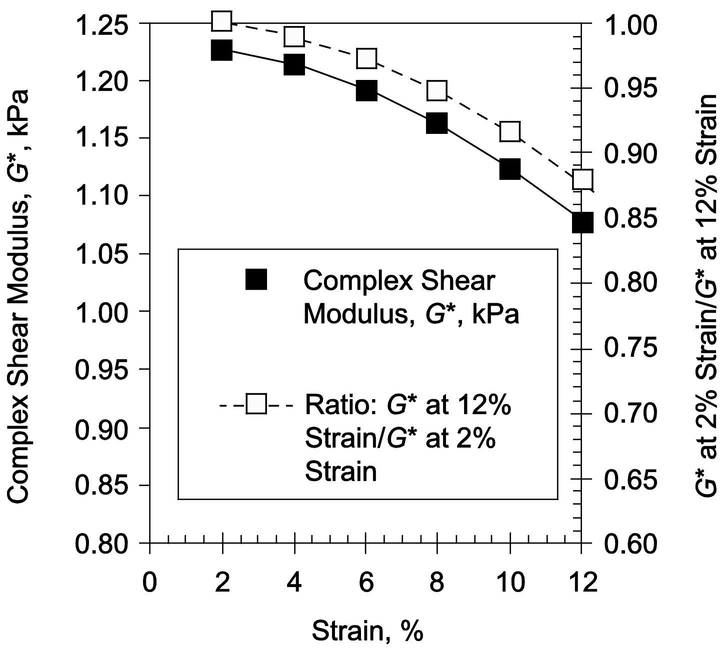


Figure 4—Example of Strain Sweep

12.2. A linear region may be defined at small strains where the modulus is relatively independent of shear strain. This region will vary with the magnitude of the complex modulus. The linear region is defined as the range in strains where the complex modulus is 95 percent or more of the zero-strain value.

12.3. The shear stress varies linearly from zero at the center of the plates to a maximum at the extremities of the plate perimeter. The shear stress is calculated from the applied or measured torque, measured or applied strain, and the geometry of the test specimen.

13. REPORT

13.1. A sample report format is given in Appendix X13. Provide a complete identification and description of the material tested including name, grade, and source.

13.2. Describe the instrument used for the test including the model number.

13.3. The strain and stress levels specified in Tables 2 and 3 have been selected to ensure a common reference point that has been shown to be within the linear region for neat and modified asphalt binders. Some systems may not be linear within this region. When this situation is observed, report the modulus at the recommended stress or strain levels but also report that the test conditions were outside the linear region.

13.4. *For each test, report the following*:

13.4.1. Test plate diameter, nearest 0.1 mm, and test gap, nearest 1 µm;

13.4.2. Test temperature, nearest 0.1°C;

13.4.3. Test frequency, nearest 0.1 rad/s;

13.4.4. Strain amplitude, nearest 0.01 percent, or torque, nearest mN·m;

13.4.5. Complex modulus (G^*) for the 10 measurements, kPa to three significant figures;

13.4.6. Phase angle (δ) for the second 10 cycles, nearest 0.1 degrees; and

13.4.7. $G^*/\sin\delta$, nearest 0.01 kPa, or $G^*\sin\delta$, nearest whole number.

14. PRECISION AND BIAS

14.1. *Precision*—Criteria for judging the acceptability of dynamic shear results obtained by this method are given in Table 4.

14.1.1. *Single-Operator Precision (Repeatability)*—The figures in Column 2 of Table 4 are the coefficients of variation that have been found to be appropriate for the conditions of test described in Column 1. Two results obtained in the same laboratory, by the same operator using the same equipment, in the shortest practical period of time, should not be considered suspect unless the difference in the two results, expressed as a percent of their mean, exceeds the values given in Table 4, Column 3.

14.1.2. *Multilaboratory Precision (Reproducibility)*—The figures in Column 2 of Table 4 are the coefficients of variation that have been found to be appropriate for the conditions of test described in Column 1. Two results submitted by two different operators testing the same material in different laboratories shall not be considered suspect unless the difference in the two results, expressed as a percent of their mean, exceeds the values given in Table 4, Column 3.

Table 4—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 Residue: $G^*/\sin\delta$ (kPa)	3.2	9.0
PAV Residue: $G^*\cdot\sin\delta$ (kPa)	4.9	13.8
Multilaboratory Precision:		
Original Binder: $G^*/\sin\delta$ (kPa)	6.0	17.0
RTFO Residue: $G^*/\sin\delta$ (kPa)	7.8	22.2
PAV Residue: $G^*\cdot\sin\delta$ (kPa)	14.2	40.2

[a] These values represent the 1s% and d2s% limits described in ASTM C670.

Note 29—The precision estimates given in Table 4 are based on the analysis of test results from eight pairs of AMRL proficiency samples. The data analyzed consisted of results from 185 to 208 laboratories for each of the eight pairs of samples. The analysis included five binder grades: PG 52-34, PG 64-16, PG 64-22, PG 70-22, and PG 76-22 (SBS modified). Average original binder results for $G^*/\sin\delta$ ranged from 1.067 kPa to 2.342 kPa. Average RTFO residue results for $G^*/\sin\delta$ ranged from 2.274 kPa to 7.733 kPa. Average PAV residue results for $G^*\cdot\sin\delta$ averaged from 1100 kPa to 4557 kPa. The details of this analysis are in the final report for NCHRP Project No. 9-26, Phase 3.

Note 30—As an example, two tests conducted on the same PAV residue yield results of 1200 kPa and 1300 kPa, respectively. The average of these two measurements is 1250 kPa. The acceptable range of results is then 13.8 percent of 1250 kPa or 173 kPa. As the difference between 1200 kPa and 1300 kPa is less than 173 kPa, the results are within the acceptable range.

14.2. *Bias*—No information can be presented on the bias of the procedure because no material having an accepted reference value is available.

15. KEYWORDS

15.1. Dynamic shear rheometer; DSR; complex modulus; asphalt binder.

APPENDIXES

(Nonmandatory Information)

X1. TESTING FOR LINEARITY

X1.1. *Scope*:

X1.1.1. This procedure is used to determine whether an unaged asphalt binder exhibits linear or nonlinear behavior at the upper grading temperature, e.g., 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 to 12 percent.

X1.2. *Procedure*:

X1.2.1. Verify the DSR and its components in accordance with Section 9 of this standard.

X1.2.2. Prepare the DSR in accordance with Section 10 of this standard.

X1.2.3. 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.

X1.2.4. 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.

X1.3. *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.

X1.4. For stress-controlled rheometers, compute the starting stress based on the complex shear modulus, G^*, and shear stress, τ, as determined at the upper grading temperature during the grading of the binder. At this temperature the complex modulus, G^*, will be greater than or equal to 1.00 kPa and the shear stress, τ, will be between 0.090 and 0.150 kPa (see Table 2). Calculate the starting stress as τ /6.00 kPa. Increase the stress in five increments of τ /6.00 kPa.

Note X1—*Sample calculation*: Assume a PG 64-22 asphalt binder with $G^* = 1.29$ kPa at 64°C and $\tau = 0.135$ kPa. The starting stress will be 1.35kPa/6 = 0.225 kPa. Test at 0.225, 0.450, 0.675, 0.900, 1.13, and 1.35 kPa, starting with 0.225 kPa.

X1.5. *Plot of Complex Modulus Versus Strain*—Prepare a plot of complex shear modulus versus percent strain as shown in Figure 4. From the plot, determine the complex shear modulus at 2 and 12 percent strain.

X1.6. *Calculations*:

X1.6.1. Calculate the modulus ratio as the complex shear modulus at 12 percent strain divided by the complex shear modulus at 2 percent strain.

X1.7. *Report*:

X1.7.1. *Report the following*:

X1.7.1.1. Complex shear modulus (G^*) to three significant figures;

X1.7.1.2. Strain, nearest 0.1 percent;

X1.7.1.3. Frequency, nearest 0.1 rad/s; and

X1.7.1.4. The ratio calculated by dividing the modulus at 12 percent strain by the modulus at 2 percent strain.

X1.8. *Data Interpretation*:

X1.8.1. The measurement was performed in the nonlinear range of the material if the modulus ratio as calculated in Section X1.6.1 is <0.900 and linear if $\geq$0.900. If the measurement was performed in the nonlinear range of the material, the results obtained under this standard will be considered as invalid for grading a binder according to M 320.

X2. CONTROL CHART

X2.1. *Control Charts*:

X2.1.1. 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.

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:

- The measurements are well controlled and both the device and the operator are performing properly.
- The measurements are becoming more variable with time, possibly indicating a problem with the test equipment or the operator.
- The laboratory measurements for the fluid are, on the average above or below the target (reference fluid) value.

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 can also be used to generate control charts and, of course, control charts can be generated manually. (See Table X3.1 as an example.)

X2.2. *Care in Selecting Data*:

X2.2.1. 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.

X3. EXAMPLE

X3.1. The power of the control chart is illustrated in Table X3.1 using the verification data obtained for the DSR. Other DSR verification data suitable for a quality control chart presentation include 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; hence, the calculated value for G^* is 2.71 kPa. This value for G^* is labeled as "G^* from Reference Fluid" in Figure X3.1. The laboratory should obtain this value on average if there is no laboratory bias.

Table X3.1—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

X3.2. *Comparison of 22-Week Laboratory Average for G* with Value Calculated from Reference Fluid*:

X3.2.1. The 22-week average of the laboratory measurements is labeled as "22-Week Laboratory Average" in Figure X3.1. Over the 22 weeks for which measurements were made, the average was 2.73 kPa. This value 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.

X3.3. *Comparison of CV of Laboratory Measurements with Round Robin CV*:

X3.3.1. From a previous round robin study, the within laboratory standard deviation (d1s) for the fluid was reported as 0.045 (CV = 1.67 percent). The 22-week standard deviation for the measured values of G^* is 0.051 CV = 1.86 percent), as compared to 0.045 (CV =1.67 percent) 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 d1s value. Based on this information the variability of the laboratory measurements are acceptable.

X3.4. *Variability of Measured Values*:

X3.4.1. In Figure X3.1, 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 ±2 d1s where d1s 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 2 d1s gives values of:

$$2.71\text{kPa} \pm (2)(0.045) = 2.80\text{ kPa}, 2.62\text{ kPa} \qquad (X3.1)$$

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 Figure X3.1 falls outside the ±2 d1s limits and is cause for concern such that testing procedures and verification should be investigated.

If a measurement deviates from the target, in this case *G** from the reference fluid, by more than ±3 d1s, corrective action should be initiated. The ±3 d1s limits 99.7 percent of the measured values if the laboratory procedures are under control and the equipment is properly calibrated.

X3.5. *Trends in Measured Value*:

X3.5.1. The control chart can also be used to identify unwanted trends in the data. For example, from Weeks 1 to 5, 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.

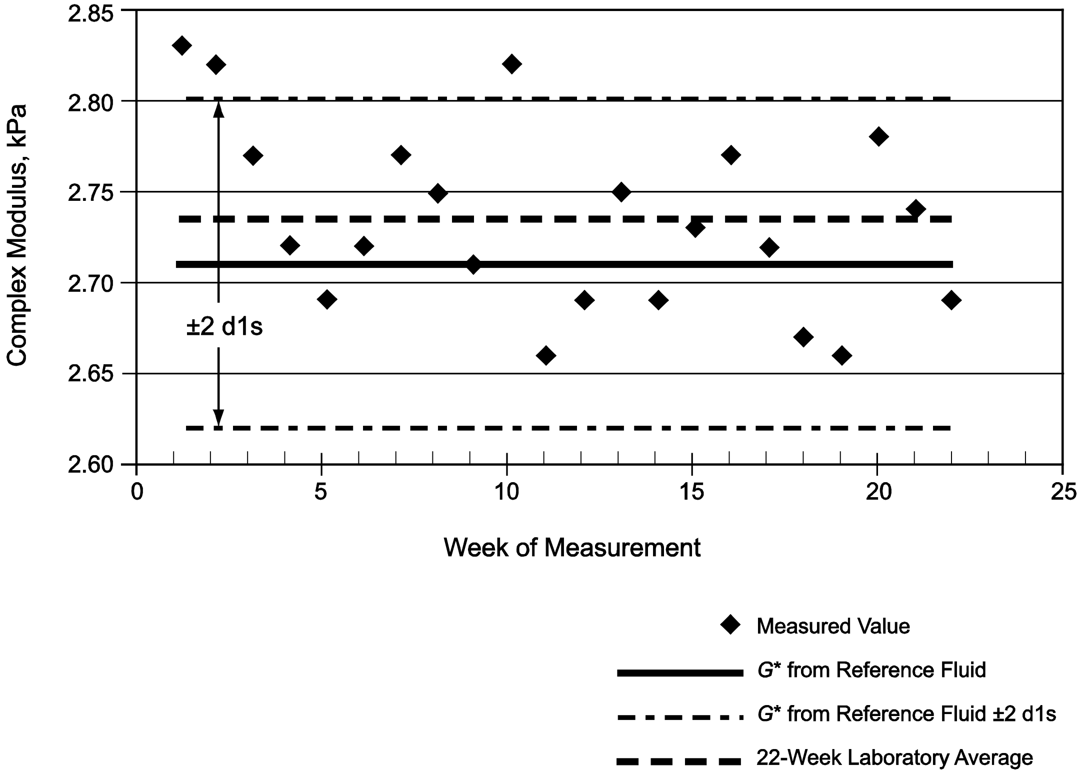


Figure X3.1—Control Chart

X4. USE OF REFERENCE FLUID

X4.1. *Source of Reference Fluid*:

X4.2. 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.

X5. CAUTIONS IN USING REFERENCE FLUID

X5.1. Some items of caution when using the reference fluid are:

- The fluid cannot be used to verify the accuracy of the phase angle measurement.
- The fluid must not be heated as heating can degrade the fluid, causing a change in its viscosity.
- The fluid should be used for verification only after the DSR temperature measurements are verified.
- The fluid cannot be used to calibrate the torque transducer. The manufacturer or other qualified service personnel using a calibration device designed specifically for the rheometer should perform the calibration. These calibration devices are typically not available in operating laboratories.
- When tested at 10 rad/s, the reference fluid should be used only at 64°C and above. At lower temperatures, the fluid is viscoelastic; hence, the viscosity, η, reported on the certificate by Cannon will not match the complex viscosity $\eta^* = G^*/10$ rad/s determined from the measurement.
- Bubbles in the fluid will have a dramatic effect on the measured value of G^*. 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 Appendix X6.

X6. CALCULATION OF *G** FROM STEADY-STATE VISCOSITY MEASUREMENTS

X6.1. 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.

$$\frac{G^*}{\omega} \sim \eta \qquad (X6.1)$$

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.

For this rule to apply the measurements must be in the viscous region where the phase angle approaches 90 degrees. 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:

$$G^*, \text{kPa} \approx (270{,}000 \text{ mPa·s})(1 \text{ kPa}/1{,}000{,}000 \text{ mPa})(10 \text{ rad/s}) = 2.70 \text{ kPa·rad} \qquad (X6.2)$$

The reference fluid behaves as a viscous fluid at 64°C and above and provides very accurate estimates of G^* above 64°C. 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 percent different than the viscosity printed on the bottle label. If this is the case, then the torque calibration should be considered suspect.

X7. METHODS FOR TRANSFERRING THE FLUID TO THE TEST PLATES

X7.1. Three different methods are recommended for transferring the fluid to the test plates:

X7.2. The glass rod method (Section X7.3), the spatula method (Section X7.4), and a direct method where a removable test plate is held in direct contact with the fluid in the bottle (Section X7.6).

X7.3. *Glass Rod Method (Figure X7.1)*:

X7.3.1. 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. See Figure X7.1.

Figure X7.1—Using a Glass Rod to Place the Reference Fluid on the Plate

X7.4. *Spatula Method (Figure X7.2)*:

X7.5. 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 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.

Figure X7.2—Using a Spatula to Place the Reference Fluid on the Plate

X7.6. *Direct Touch Method (Figure X7.3)*—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).

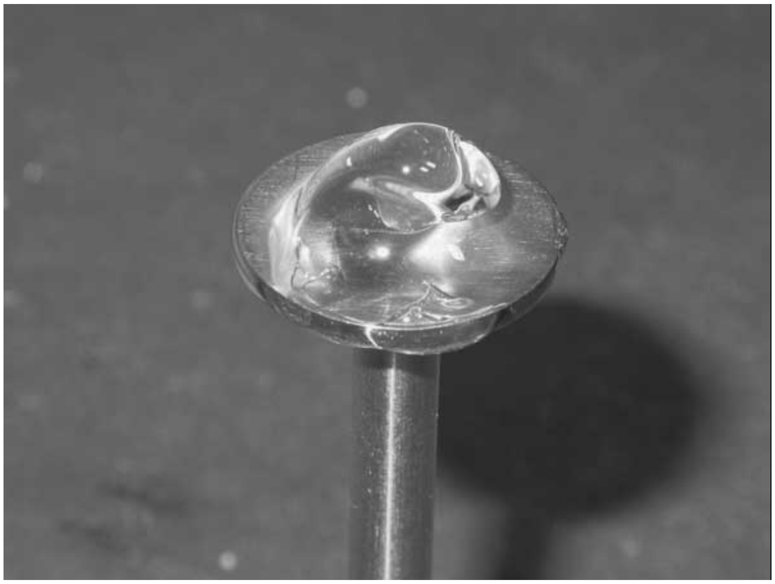


Figure X7.3—Direct Touch Method to Place the Reference Fluid on the Plate

Proceed immediately to Section 10.5 to trim the reference fluid specimen and form the bulge.

Proceed with testing the reference fluid specimen as described in Section 11.

X8. SELECTION OF GAP CLOSURE TO OBTAIN BULGE

X8.1. *Need for Accurate Measurement of Specimen Diameter*:

X8.2. 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 to be 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 Figure X8.1. The diameter of the test specimen in Figure X8.1 approaches *d*, rather than *d′*, the diameter of the plate. The modulus, *G**, is calculated according to the following equation:

$$|G^*| = \left(\frac{2h}{\pi r^4}\right)\left(\frac{\tau}{\Theta}\right) \tag{X8.1}$$

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.

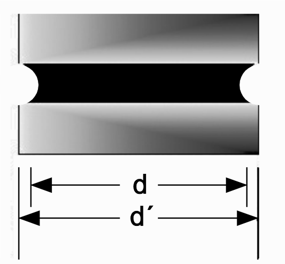


Figure X8.1—Concave Surface Resulting from Insufficient Closure after Trimming

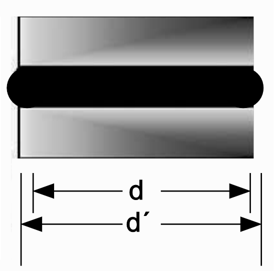


Figure X8.2—Proper Bulge

X8.3. According to Equation X8.1, 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

Figure X8.1, 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 Figure X8.2. Shear stresses are not transferred directly from the plate to the overhanging binder; therefore, the effect of a slight bulge on the measured modulus is much 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.

X9. RECOMMENDED GAP CLOSURE VALUES

X9.1. Recommended values for the gap closure required to form a bulge at the test temperature similar to the bulge illustrated in Figure X8.2 are given in Section 10.5 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.

Proper and improper bulges are shown in Figures X10.1 through X10.3. 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.

X10. FACTORS AFFECTING BULGE DEVELOPMENT

X10.1. A number of factors can affect the bulge formed at the test temperature. These include:

- The amount of closure used to create the bulge.
- The difference in temperature between the trimming temperature, the temperature at which the bulge is created, and the test temperature.
- Thermal expansion-contraction characteristics of the rheometer.
- Thermal contraction and expansion of the asphalt binder.

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 Figure X10.3. This situation should also be avoided and may require gap closures somewhat less than the recommended values.

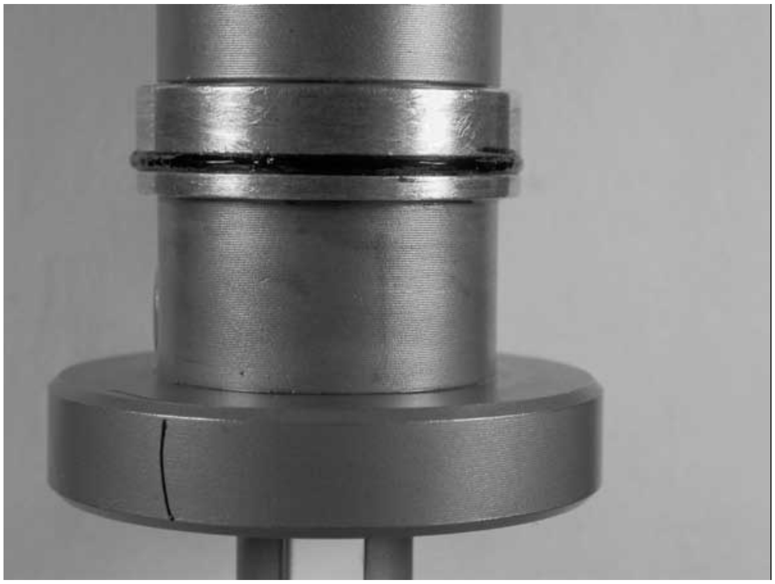


Figure X10.1—Good Bulge Size

Figure X10.2—Concave Bulge

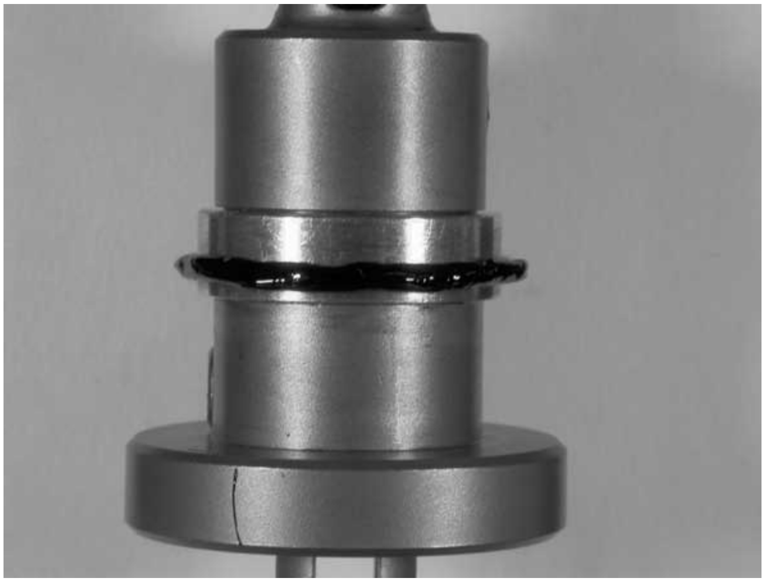


Figure X10.3—Oversized Bulge

X11. DETERMINATION OF TIME TO THERMAL EQUILIBRIUM

X11.1. *Reason for Determining Time Required to Obtain Thermal Equilibrium*:

X11.1.1. After the test specimen has been mounted in the DSR, it takes 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.

X11.1.2. The time required to obtain thermal equilibrium varies for different rheometers. Factors that affect the time required for thermal equilibrium include:

X11.1.3. Design of the rheometer and whether air or liquid is used as a heating/cooling medium;

X11.1.4. Difference between ambient temperature and the test temperature, different when testing below room temperature, and above room temperature;

X11.1.5. Difference in temperature between the trimming and test temperature; and

X11.1.6. Plate size, different for the 8-mm and 25-mm plate.

X11.2. 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.

X12. METHOD TO DETERMINE THE TIME REQUIRED TO OBTAIN THERMAL EQUILIBRIUM

X12.1. 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. The following procedure is recommended for establishing the time to thermal equilibrium:

X12.2. 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.

X12.3. 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.

X12.4. Record the modulus at 30-s time intervals, and plot the modulus versus time (Figure X12.1).

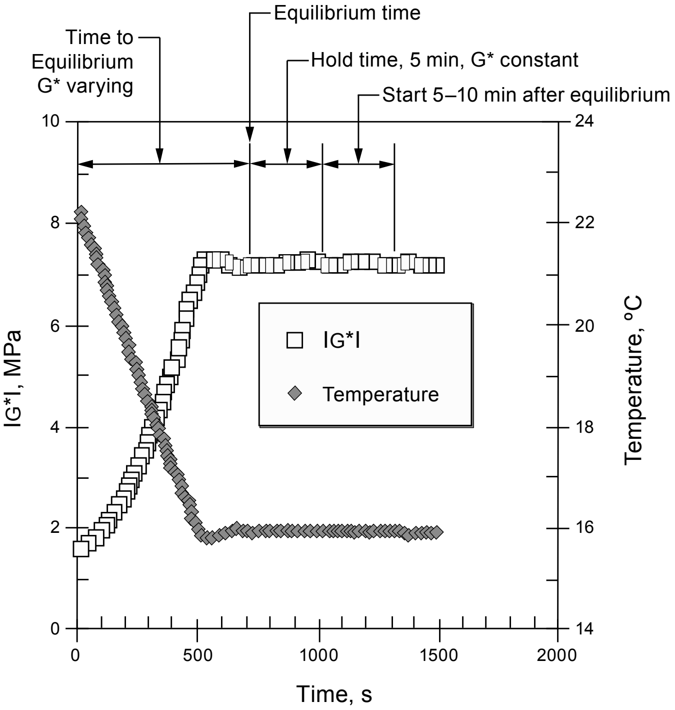


Figure X12.1—Determining Thermal Equilibrium Time

X12.5. 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.

X12.6. Because the time required to reach thermal equilibrium will vary with the test temperature and testing conditions, the time to thermal equilibrium should be established separately for both intermediate and high-temperature measurements. Once the time to thermal equilibrium has been established, it does not have to be repeated unless the test conditions change.

X13. SAMPLE REPORT

Header Information:

Item	Data Group 1	Item	Data Group 2
Operator's Name:	24 Alpha-Numeric	Date of Test (dd/mm/yy):	__/__/__
Test Specimen ID No.:	18 Alpha	Time of Test (hr:min):	__:__
Project ID No.:	12 Alpha-Numeric	DSR Manufacturer:	12 Alpha-Numeric
File Name:	12 Alpha-Numeric	DSR Model:	12 Alpha-Numeric
Test Plate Diameter, Nearest 0.01 mm:	00.00	DSR Serial Number or Other Identifying ID No.:	18 Alpha-Numeric
Test Frequency, rad/s:	0.00	Software Version:	12 Alpha-Numeric
Test Gap, 0.01 mm:	00.00		

Test Results for Grading (Use separate column for each test temperature):

Measurements	Data Group 3	Data Group 4	Data Group 5	Data Group 6
Test Specimen Temperature, 0.1°C	0.00	0.00	0.00	0.00
Temperature Correction at Test Temperature, 0.1°C	0.00	0.00	0.00	0.00
Stress Amplitude, kPa	0.00	0.00	0.00	0.00
Strain Amplitude, percent	0.00	0.00	0.00	0.00
Complex Modulus, *G**, kPa	0.00	0.00	0.00	0.00
Phase Angle, degrees	00.0	00.0	00.0	00.0
Complex Modulus/sin(Phase Angle)	0.00	0.00	0.00	0.00
Complex Modulus × sinδ (Phase Angle)	00.0	00.0	00.0	00.0
Comments generated by DSR software (Example: "This material passes.")				
At end of file, 276 alphanumeric field for operator comments and comments, if any, generated by DSR software.				

Test Results for Linearity Determination:

Measurements	Data Group 7
Test Specimen Temperature, 0.1°C	0.00
Temperature Correction at Test Temperature, 0.1°C	0.00

	Data Group 8	Data Group 9	Data Group 10	Data Group 11	Data Group 12	Data Group 13
Measurements		0.00	0.00	0.00	0.00	0.00
Stress Amplitude, kPa	0.00	0.00	0.00	0.00	0.00	0.00
Strain Amplitude, percent	0.00	0.00	0.00	0.00	0.00	0.00
Complex Modulus, *G**, kPa	0.00	00.0	00.0	00.0	00.0	00.0
Phase Angle, degrees	00.0	00.0	00.0	00.0	00.0	00.0
At end of file, 276 alphanumeric field for operator comments and comments, if any, generated by DSR software.						

X14. REFERENCES

X14.1. Anderson, D. A. and M. Marasteanu. "*Manual of Practice for Testing Asphalt Binders in Accordance with the Superpave PG Grading System.*" The Pennsylvania Transportation Institute, The Pennsylvania State University, PTI 2K07, November 1999 (Revised February 2002).

X14.2. Anderson, D. A., C. E. Antle, K. Knechtel, and Y. Liu. *Interlaboratory Test Program to Determine the Inter- and Intra-Laboratory Variability of the SHRP Asphalt Binder Tests*. FHWA, 1997.

X14.3. Cox, W. P. and E. H. Merz. Correlation of Dynamic and Steady Flow Viscosities. *Journal of Polymer Science*, Vol. 28, 1958, pp. 619–622.

X14.4. Wadsworth, H., ed. *Handbook of Statistical Methods for Engineers and Scientists.* McGraw-Hill, New York, NY, 1990.