

Designation: D7552 - 09 (Reapproved 2014)

Standard Test Method for Determining the Complex Shear Modulus (G*) Of Bituminous Mixtures Using Dynamic Shear Rheometer¹

This standard is issued under the fixed designation D7552; 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 complex shear modulus of bituminous mixtures using torsion rectangular geometry on a dynamic shear rheometer (DSR). It is applicable to bituminous mixtures having complex shear modulus values greater than 1 × 10⁴ Pa when tested over a range of temperatures from 10°C to 76°C at frequencies of 0.01 to 25 Hz and strains of 0.001 % to 0.1 %. The determination of complex shear modulus is typically determined at 20°C to 70°C at 0.01% strain at 10 discrete frequency values covering 0.01 to 10 Hz. From these data, temperature or frequency master curves can be generated as required. This test method is intended for determining the complex shear modulus of bituminous mixtures as required for specification testing or quality control of bituminous mixture production.
- 1.2 This test method is appropriate for laboratory prepared and compacted mixtures, field produced and laboratory compacted mixtures or field cores, regardless of binder type or grade and regardless of whether RAP is used in the mixture. Due to the geometry of the specimens being tested this test method is not applicable to open-graded or SMA mixtures. It has been found to be appropriate for dense-graded mixtures, whether coarse- or fine-graded, with 19 mm or smaller nominal maximum aggregate size.
- 1.3 The between-laboratory reproducibility of this test method is being determined and will be available on or before June 2012. Therefore, this test method should not be used for acceptance or rejection of materials for purchasing purposes.
- 1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.
- 1.5 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 appro-

¹ This test method is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.26 on

Current edition approved Aug. 1, 2014. Published November 2014. Originally approved in 2009. Last previous edition approved in 2009 as D7552-09. DOI: 10.1520/D7552-09R14.

Fundamental/Mechanistic Tests.

priate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:²

C670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials

D140 Practice for Sampling Bituminous Materials

D2041 Test Method for Theoretical Maximum Specific Gravity and Density of Bituminous Paving Mixtures

D2726 Test Method for Bulk Specific Gravity and Density of Non-Absorptive Compacted Bituminous Mixtures

D3203 Test Method for Percent Air Voids in Compacted Dense and Open Bituminous Paving Mixtures

D3666 Specification for Minimum Requirements for Agencies Testing and Inspecting Road and Paving Materials

D6752 Test Method for Bulk Specific Gravity and Density of Compacted Bituminous Mixtures Using Automatic Vacuum Sealing Method

D6857 Test Method for Maximum Specific Gravity and Density of Bituminous Paving Mixtures Using Automatic Vacuum Sealing Method

D6925 Test Method for Preparation and Determination of the Relative Density of Asphalt Mix Specimens by Means of the Superpave Gyratory Compactor

D6926 Practice for Preparation of Bituminous Specimens Using Marshall Apparatus

D7175 Test Method for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer

D7312 Test Method for Determining the Permanent Shear Strain and Complex Shear Modulus of Asphalt Mixtures Using the Superpave Shear Tester (SST)

E77 Test Method for Inspection and Verification of Thermometers

E563 Practice for Preparation and Use of an Ice-Point Bath as a Reference Temperature

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.

E644 Test Methods for Testing Industrial Resistance Thermometers

2.2 Other Standards:

DIN Standard 43760 Standard for Calibration of Platinum Resistance Thermometers³

3. Terminology

- 3.1 Definitions of Terms Specific to This Standard:
- 3.1.1 *asphalt binder, n*—an asphalt-based cement that is produced from petroleum residue either with or without the addition of non-particulate modifiers.
- 3.1.2 *calibration*, *n*—a process that establishes the relationship (traceability) between the results of a measurement instrument, measurement system, or material measure and the corresponding values assigned to a reference standard. Calibration is typically performed by the manufacturer or an external commercial calibration service.
- 3.1.3 complex shear modulus (G^*) , n—a complex number that is defined by the ratio of shear stress to shear strain.
- 3.1.4 dummy test specimen, n—a rectangular prismatic or cylindrical specimen of bituminous mix prepared as discussed in Section 9.2, into which a small hole is drilled and into which a PRT wire is inserted. The dummy specimen is then mounted in the torsion fixture of the DSR for the purpose of determining the temperature in the bituminous mixture. In addition the dummy specimen can be used to ascertain the amount of time needed to bring a test specimen to the appropriate test temperature.
- 3.1.4.1 *Discussion*—The dummy test specimen is not used to measure the modulus characteristics of the bituminous mixture but is used to determine temperature corrections and equilibrium times.
- 3.1.5 *loading cycle*, *n*—refers to the application of sinusoidal stress or strain loading for a specified duration.
- 3.1.6 *shear stress*, *n*—the force per unit area that produces the flow.
- 3.1.7 portable thermometer, n—refers to an electronic device that is separate from the dynamic shear rheometer and that consists of a detector (probe containing a thermocouple or resistive element), associated electronic circuitry, and readout system.
- 3.1.8 reference thermometer, n—refers to a NIST-traceable liquid-in-glass or electronic thermometer that is used as a laboratory standard.
- 3.1.9 temperature correction, n—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.
- 3.1.10 *thermal equilibrium*, *n*—condition where the temperature of the test specimen mounted between the test plates is constant with time.

³ Available from Beuth Verlag GmbH (DIN-- DIN Deutsches Institut fur Normung e.V.), Burggrafenstrasse 6, 10787, Berlin, Germany, http://www.en.din.de.

3.1.11 *verification*, *n*—a process that establishes whether the results of a previously calibrated measurement instrument, measurement system, or material measure are stable. Usually performed internally within the operating laboratory.

4. Summary of Test Method

- 4.1 This standard contains the procedure used to measure the complex shear modulus of a bituminous mixture using a DSR in oscillatory mode and using torsional rectangular geometry. The DSR must be temperature-controlled using a forced air system.
- 4.2 The standard is suitable for use when the complex shear modulus is greater than 1×10^4 Pa at the test temperature. The complex shear modulus is typically determined at 20°C to 70°C, although other test temperatures may be used.
- 4.3 Test specimens, nominally 49 \pm 2 mm in length, 12 \pm 2 mm in width and 9 \pm 1.5 mm in thickness may be cut from gyratory or Marshall laboratory specimens or from field cores (see Figs. 1-3). Specimens can be obtained from bituminous mixture samples compacted using other devices as long as it is possible to determine the air voids of the mixture samples. The test specimens are mounted with the 49 \pm 2 mm length forming a vertical dimension in the DSR.
- 4.4 During testing, one of the fixtures⁴ is rotated with respect to the other at a pre-selected % strain and a range of frequencies at the selected temperatures. The test shall be conducted at 0.01 % strain unless otherwise stated. The % strain stipulated in this test method has been found to produce acceptable results for the bituminous materials investigated to date.

Note 1—Different strain values, within the capabilities of individual equipment, may be selected for testing materials beyond the scope of those tested to date. Regardless of % strain or test temperatures chosen or test materials investigated, the basic testing process described herein will not change.

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

5. Significance and Use

5.1 The complex shear modulus of bituminous mixtures is a fundamental property of the material. Test results at critical temperatures (T_{critical}) are used for specifications for some mixes. Mixtures with stiffer binders, aged mixtures, mixtures with higher amounts of fines (material finer than 75µ), and mixtures with lower voids all tend to have higher complex shear modulus values than mixtures with less stiff binders, unaged mixes, mixtures with low levels of fines and higher air voids. In general, mixtures with higher complex shear modulus values at a given service temperature will exhibit lower permanent deformation values than similar mixtures tested at the same temperature that have lower complex shear modulus values.

⁴ Depending upon whether a stress or strain controlled rheometer is being used, either the upper or lower fixture will be the one which is rotated. This test method is applicable to both stress and strain controlled rheometers. When a stress controlled rheometer is used, the test is performed in strain controlled mode.

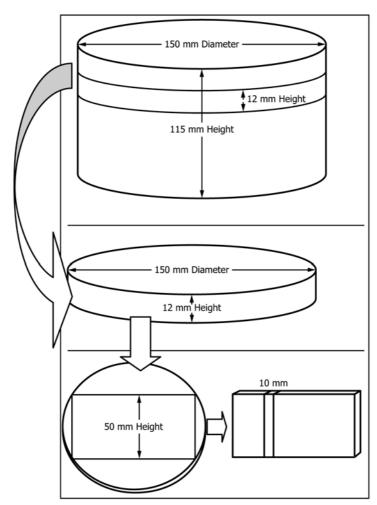


FIG. 1 Schematic of Preparing Torsion Rectangular Specimens

Note 2—The quality of the results produced by this standard are dependent on the competence of the personnel performing the procedure and the capability, calibration, and maintenance of the equipment used. Agencies that meet the criteria of Practice D3666 are generally considered capable of competent and objective testing/sampling/inspection/etc. Users of this standard are cautioned that compliance with Practice D3666 alone does not completely assure reliable results. Reliable results depend on many factors; following the suggestions of Practice D3666 or some similar acceptable guideline provides a means of evaluating and controlling some of those factors.

6. Interferences

- 6.1 Due to the nature of test geometry this test cannot be used to determine the complex shear modulus of rectangular specimens obtained from SMA (Stone Mastic or Matrix Asphalt) or OGFC (Open Graded Friction Course) mixtures. Without confining pressure these specimens fall apart when brought to test temperature. At this point in time there is no suitable method for imparting confining pressure on the test specimens.
- 6.1.1 The calculation of the complex shear modulus from the data obtained from the DSR is highly dependent upon an accurate measurement of the dimensions of the test specimen. In the procedure, the length of the test specimen is the gap distance between the mounting fixtures after the zero gap measurement of the torsion fixture has been made. Once the

test specimen is mounted in the fixture, the length of specimen between the two mounting points is the length of the specimen. The width and thickness of the specimen is determined prior to mounting the specimen in the DSR using a digital caliper and is reported to the nearest 0.01 mm. These values are entered into the software of the instrument where the test specimen dimensions are requested. Due to the potential for variability in the width and thickness due to the sample preparation procedure, the width and thickness is determined in the central portion of the test specimen.

7. Apparatus

- 7.1 The apparatus for performing the test as described in this method shall be the equipment described in Test Method D7175 under the section heading of Apparatus except as amended below.
- 7.2 Test Fixtures—Two fixtures capable of securing the rectangular test specimens with the long dimension of the test article in a vertical plane are required.
- 7.3 A torque wrench capable of applying a torque load of 0.25 N·m (250 mN·m) \pm 0.05 N·m of torque to tighten the test specimen in the mounting fixture without crushing.

Sample Preparation—Figure 2

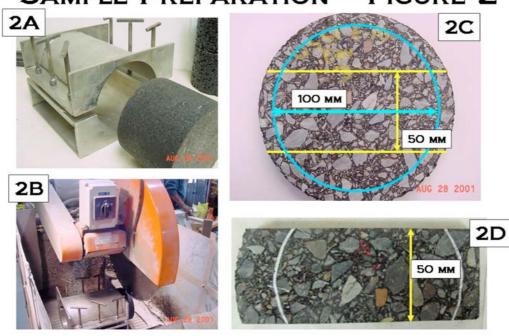


FIG. 2 Sample Preparation to Obtain 50-mm Wide by 12-mm Thick Rectangular Specimen

SAMPLE PREPARATION—FIGURE 3



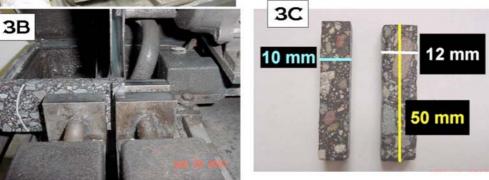


FIG. 3 Sample Preparation to Obtain 50-mm Wide by 12-mm Wide by 10-mm Thick Specimen.

7.4 Environmental Chamber—A chamber for controlling the temperature of the test specimens. The medium used to control the chamber shall be compressed laboratory air or commercially bottled air. Chilled, compressed laboratory air or liquid nitrogen (LN2) is required if testing temperatures below approximately 30°C is to be conducted. When laboratory air is

used in a forced air environmental chamber, a suitable dryer must be included to prevent condensation of moisture on the test specimen. The environmental chamber and the temperature controller shall control the temperature of the test specimen mounted between the grips, including any thermal gradients within the test specimen, at the test temperature \pm 0.1°C. Due

to the geometry and type of material being tested, water baths and Peltier fixtures cannot be used to control the test temperature of the specimens. Some companies manufacture a Peltier heated submersion cell, which uses water or some other liquid medium to condition the test specimen. Testing the mixture while submerged could introduce errors in the results due to weakening of the mix due to moisture interaction.

- 7.5 Temperature Controller—A temperature controller capable of maintaining the temperature of the test specimen at the test temperature $\pm 0.1^{\circ}$ C for test temperatures stipulated.
- 7.6 Internal DSR Thermometer—A platinum resistance thermometer (PRT) mounted within the environmental chamber as an integral part of the DSR and in close proximity to the bottom mounting fixture with a minimum range of 30°C to 82°C, and with a resolution of 0.1°C. Normally this range will be sufficient unless there is a need to determine the complex shear modulus of the mixture at temperatures below ambient. If there is a need to control test temperatures below ambient then mechanical cooling or liquid nitrogen will be needed. This thermometer shall be used to control the temperature of the test specimen and shall provide a continuous readout of temperature during the mounting, conditioning, and testing of the specimen.

Note 3—Platinum resistance thermometers (PRTs) meeting DIN Standard 43760 (Class A) or equal are recommended for this purpose. The PRT is to be calibrated as an integral unit with its respective meter or electronic circuitry.

- 7.7 Loading Device—The loading device shall at least be capable of applying a sinusoidal oscillatory load to the specimen at the following frequencies—0.01, 0.02, 0.05, 0.1, 0.2, 0.5, 1, 5, 10 and 15 Hz. The loading device shall be capable of controlling frequencies to an accuracy of 1 percent. The loading device shall be capable of providing a strain controlled load within a range of strain necessary to make the measurements described in this standard. The manufacturer of the device shall provide a certificate certifying that the frequency and strain are controlled and measured with accuracy of 1 % or less in the range of this measurement.
- 7.8 Data Acquisition System—The data acquisition system shall provide a record of temperature, frequency, deflection angle, % strain, oscillatory stress, 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 %.
- 7.9 Digital Calipers—A digital caliper with a resolution of ± 0.01 mm is required to determine the width and thickness of the test specimens.

8. Materials

- 8.1 Wiping Material—Clean cloth, paper towels, cotton swabs or other suitable material as required for wiping the mounting fixtures.
 - 8.2 Cleaning Solvents.
- 8.2.1 Mineral oil, citrus-based solvents, mineral spirits, toluene, or similar solvent, as required for cleaning the mounting clamps.

- 8.2.2 Acetone or ethanol may be used as needed for removing solvent residue from the surfaces of the mounting clamps.
- 8.3 Reference Thermometer—Either a NIST-traceable liquid-in-glass thermometer(s) (Section 8.3.1) or NIST-traceable digital electronic thermometer (Section 8.3.2) shall be maintained in the laboratory as a temperature standard. This temperature standard shall be used to verify the portable thermometer (Section 8.4).
- 8.3.1 *Liquid-in-Glass Thermometer*—NIST-traceable liquid-in-glass thermometer(s) with a suitable range and with subdivisions of 0.1°C. The thermometer(s) shall be partial immersion thermometers with an ice point and calibrated in accordance with Test Method E77. Calibration interval shall be on a 12-month interval.
- 8.3.2 Digital Electronic Thermometer—An electronic thermometer that incorporates a thermocouple or resistive detector with an accuracy of $\pm 0.05^{\circ}$ 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 Method E644.
- 8.4 Portable Thermometer—A calibrated portable thermometer consisting of a thermocouple or resistive detector, associated electronic circuitry, and digital readout. The thickness of the detector shall be no greater than 2.0 mm. The reference thermometer (See Section 8.3) may be used for this purpose if its detector fits within the dummy specimen as required by Section 9.2.1.

9. Verification and Calibration

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, or whenever the accuracy of the DSR or any of its components is suspect. Verification and calibration of the DSR required to perform solids testing follows the procedures detailed in Test Method D7175 Section 9. A DSR for which the DSR torque transducer and portable thermometer have been properly calibrated and verified requires no further calibration and verification of the torque transducer and portable thermometer.

Note 4—At this point no suitable torsional verification standard for the torque transducer has been identified. Therefore verification of the torque transducer utilizing the Cannon Instrument Company viscosity standard N2700000SP as described in Test Method D7175 Section 9.5.1.1 and Note 11 should be employed.

9.2 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). When these differences are 0.1°C, or greater, determine a temperature correction by placing a bituminous mixture specimen (dummy sample) between the torsion mounting fixtures and inserting the detector of the portable thermometer into a small hole drilled in the specimen and secured in place using commercial caulking.

Note 5—Depending upon the DSR manufacturer there may be no need to perform a separate temperature correction for solids testing. Follow the

recommendations of the DSR manufacturer to ascertain whether a separate temperature correction determination is required for solids testing.

- 9.2.1 Method Using Dummy Test Specimen—The dummy test specimen shall be a torsion rectangular bituminous mix specimen of approximate dimensions described in Section 4.3. Mount the dummy test specimen with the temperature probe wires inserted and close the environmental chamber. Determine any needed temperature correction as per Section 9.2.2.
- 9.2.2 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 five minute time period. Obtain additional measurements to include the entire temperature range that will be used for measuring the dynamic shear modulus.
- 9.2.3 Plot Correction versus Specimen Temperature—Using the data obtained in Section 9.2.2, prepare a plot of the difference between the two temperature measurements versus the temperature measured with the portable thermometer. This difference is the temperature correction that must be applied to the DSR temperature controller to obtain the desired temperature in the test specimen mounted in the torsion fixture. 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 6—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.

9.3 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, or whenever the accuracy of measurements with the DSR is suspect. Verification of torque transducer and angular displacement is performed according to Section 9.5 of Test Method D7175.

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 Fixture—Bring the upper and lower mounting fixtures together and make sure that there is not an offset. The mixture specimen, once mounted between the upper and lower fixture shall be vertical, centered within the upper and lower mounting fixtures and not subjected to torque due to offsets. If an offset between top and bottom fixtures is observed steps must be taken to correct the offset up to and including obtaining a new torsion fixture.

- 10.3 Zero Gap—Select the testing temperature according to the climatic zone in which the bituminous mix will be expected to perform or according to a pre-selected testing criteria. 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 $\pm 0.1^{\circ}$ C of test temperature. If the test temperature differs by more than $\pm 12^{\circ}$ C from the temperature at which the gap is set, re-zero the gap. Zero the gap prior to each time a new range of test temperatures is programmed or each time the upper or lower fixture is removed from the DSR. If the upper or lower fixture is not removed then the gap shall be zeroed at the beginning of each days testing.
- 10.3.1 *Determining Zero Gap*—Establish the zero gap following the procedure recommended by the DSR manufacturer.
- 10.4 Calibrating DSR—If a controlled strain rheometer is being used for testing then no additional calibration is needed on a daily basis. If a controlled stress rheometer is being used for testing then bearing friction, machine and geometry inertia calibration and mapping should be performed at least weekly or any time that the upper fixture has been removed from the machine and re-installed. These calibrations should also be performed any time there is reason to suspect a change in the instrument performance due to such factors as power outage, machine over speed or other machine errors.

Note 7—Maintaining a log of calibration results for bearing friction, machine inertia and geometry inertia can provide assistance to the operator to ascertain when the DSR may require service by the manufacturer's representative.

11. Preparing Test Specimens

- 11.1 Test specimens as diagramed in Fig. 1 and shown in Figs. 2 and 3 can be obtained from Superpave gyratory compacted specimens prepared according to Test Method D6925 or Marshall specimens prepared according to Practice D6926 or from cores cut from pavements. Slabs cut from pavements can also be used, but care should be taken to assure that damage to the mix integrity has not occurred.
- 11.1.1 Air voids of gyratory specimens or Marshall specimens shall be determined using Test Method D3203. Maximum specific gravity of Superpave gyratory or Marshall compacted specimens may be obtained using Test Method D2041 or Test Method D6857. If the air voids of the approximate 150-mm diameter by 12-mm thick slice Fig. 2(C) are to be determined then Test Method D6752 shall be used for determination of bulk specific gravity.
- 11.1.2 If the maximum specific gravity of the mix from which field cut cores or slabs is known or can be determined, then the air voids of such field cores or slabs should also be determined using the methods referenced in Section 11.1.1.

Note 8—As a matter of practice it is recommended that bulk specific gravity of field cut specimens be determined prior to preparation of the torsion rectangular specimens.

11.2 Under typical testing requirements the top 25 mm shall be sawed from the gyratory specimen and discarded to assure uniformity of air voids. An approximately 12 mm thick slice of mix shall then be sawed resulting in a disk of mix that is 150 mm in diameter and 12 mm in thickness (Figs. 1 and 2). From this disk, a rectangular portion of mix shall be sawed that is

approximately 50 mm wide (Figs. 1 and 2). This rectangular portion is then sawed (Figs. 1 and 3) into rectangular prismatic specimens that are approximately 10 mm wide. The resulting dimensions should be 9 ± 1.5 mm wide, 12 ± 2 mm thick and 49 ± 2 mm long. Since all dimensions will be individually determined for each specimen prior to testing, these dimensions are stipulated to maximize the specimen size while still assuring that the specimens will fit within the test fixtures of the DSR. If the 150 mm diameter specimens are obtained from a gyratory compactor then approximately 25 mm of each end of the rectangle shall be discarded due to variability of air voids in the mix. Air voids of the 12 mm thick by 150 mm diameter disk sawed from the gyratory or field core shall only be obtained using Test Method D6752 on specimens that have been thoroughly dried.

Note 9—All sawing is to be performed using a water cooled diamond rim saw to prevent damage to the test specimens and to assure smooth surfaces

11.3 After the torsion rectangular specimens have been cut, they should be patted to surface dryness with paper or cloth towels, air dried in front of a fan for approximately 30 minutes, and then put in a desiccator at ambient temperature overnight or for a minimum of 5 hours before testing is commenced. Specimens should be stored in such a way as to minimize exposure to ambient air and light. If testing will not be completed within 1 week the specimens shall be put into plastic bags to remove them from exposure to air.

12. Procedure

12.1 Zero the gap at the test temperature or at the mid range of the test temperatures if more than one test temperature is to be used. Open the environmental chamber and mount the test specimen at ambient temperature. Make sure the specimen is centered in the mounting fixture; obviously deformed or non straight specimens shall be discarded. Specimens exhibiting damage such as broken aggregate particles shall be discarded as well. Finger tighten the specimens in place and then carefully apply a torque of approximately 200 milli-newton meter (mN·m) to the tightening screws being careful to not introduce normal force loads on the specimen. If necessary use the normal force hold feature of the rheometer to adjust the gap to reduce normal force on the specimen. Care must be taken at this point to not damage the specimen by adjusting the gap. If necessary loosen the set screws and re-adjust the specimen to reduce the normal force. A normal force of not more than 5 N is acceptable as this force will relax when the specimen is warmed to the test temperature. Close the doors on the environmental chamber and warm the specimen to the test temperature with no further adjustment of the gap. Set the temperature controller to the beginning test temperature, including any correction as per Section 9.2.3, required to obtain the test temperature in the test specimen. Allow the DSR to reach thermal equilibrium within ± 0.1 °C of test temperature. It has been found that an equilibration time of 15 minutes is sufficient to achieve equilibrium. The test shall be started within 5 minutes of when the test specimen has reached thermal equilibrium. Perform the frequency sweep at each temperature from the highest to the lowest frequency.

- 12.1.1 Unless otherwise stipulated the complex shear modulus test shall be performed at the high PG grade temperature representative of a climatic region as determined by LTTP-BIND v3.1 without grade bumping adjustments. Therefore when a bumped PG grade of binder is used for traffic loads or other reasons, the complex shear modulus of the mix will still be determined at the actual PG grade of the region. Alternate test temperatures such as those suggested by Test Method D7312 may also be used. If a mastercurve is to be generated the mix specimens shall be tested at the high PG binder grade environmental temperature and at least at temperatures 6, 12, and 18°C below the environmental temperature.
- 12.1.2 When testing at multiple temperatures, start at the lowest test temperature.
- 12.1.3 Testing in Strain Control Mode Unless otherwise stipulated the complex shear modulus test shall be conducted in strain control mode using a strain of 0.01%. Software is available with the dynamic shear rheometers that will control the strain automatically without control by the operator.

Note 10—The complex shear modulus as determined by this procedure at a strain of 0.01 % produces results comparable to those obtained on the Superpave Shear Tester (SST) performing the Frequency Sweep at Constant Height. While other strains can be used and while the test can also be operated in stress control mode, there has been no comparative testing performed to show a similarity between the DSR complex shear modulus determined at other strain values or in stress control and results obtained from the SST.

- 12.2 For each increase in test temperature there shall be a delay of 15 minutes programmed into the test sequence before testing can be resumed to allow the test specimen to reach thermal equilibrium at the new test temperature. Start the application of the load and obtain a measurement of the complex shear 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 highest frequency and decrease to the lowest frequency. These results will typically be controlled by the individual manufacturer's software.

Note 11—The complex shear modulus of the bituminous mixture is typically reported at a frequency of 10 Hz at the stipulated test temperature.

12.4 Testing shall be conducted in triplicate. If the coefficient of variation of the complex shear modulus at 10 Hz at each test temperature is not in conformance with Table 1 a fourth specimen shall be tested. The test result from original three specimens with the greatest deviation shall be eliminated and the test result from the fourth specimen substituted. If the coefficient of variation still is not in conformance with Table 1, then another set of three specimens shall be tested. If after the second set of three specimens is tested the coefficient of variation is still not in conformance with Table 1, the results shall be reported with a notation of the actual coefficient of variation of the test results.

TABLE 1

Condition	Coefficient of Variation (1s%) ^A	Acceptable Range of Two Test Results (d2s %) ^A
Single-Operator Precision: G*(Pa) ^B Multilaboratory Precision:	15 %	42 %
G*(Pa)	not yet determined	not yet determined

^A These values represent the 1s % and d2s % limits described in Practice C670. ^B The results reported here are based on one operator testing 6 different mix samples at 10 Hz at 46°C.

13. Interpretation of Results

13.1 The dynamic complex shear modulus and phase angle may depend upon the magnitude of the shear strain. Depending on the type of mix, the test temperature, the PG grade and type of binder the test may not occur in the linear viscoelastic region. For this reason it is important that the % strain value at which the test is conducted is stipulated. Figs. 4 and 5 show typical test results for bituminous mixtures conducted at a value of 0.01% strain. Fig. 6 shows a temperature mastercurve at a reference frequency of 10 Hz. This result enables expansion of the complex shear modulus of the mix to temperatures beyond the scope of the original test. There is a peak in the phase angle plot after which the phase angle begins to decrease with increasing temperature. This is an indication that the binder is playing a decreasing role in the mixture modulus and interaction of the aggregate particles is causing a decrease in the phase angle. Fig. 7 shows a comparison between complex shear modulus frequency values at 70°C for two polymer modified mixes which use the same aggregate and binder but that were produced at two different air voids. Note that the complex shear modulus values for the lower air voids mix is greater than for the higher air voids mix.

14. Report

- 14.1 Report the following information. A recommended format for reporting the information is given in Table 2.
 - 14.1.1 File name,
 - 14.1.2 Sample identification information,
 - 14.1.3 Operator's name,
 - 14.1.4 Date of test (dd/mm/yy),
- 14.1.5 The width and thickness of the test specimen to the nearest 0.01 mm and specimen gauge length to the 0.01 mm,
 - 14.1.6 Test temperature reported to the nearest 0.1°C,
- 14.1.7 Temperature correction, if a temperature offset was applied, at the test temperature (°C reported to the nearest 0.1°C),
- 14.1.8 Strain percent value at which the test was performed reported to nearest 0.1 %,
- 14.1.9 Complex Shear modulus (G*), Pa to three significant digits,
 - 14.1.10 The phase angle (δ), to the nearest 0.1 degrees,
- 14.1.11 Test frequency for each value reported, frequency to be reported in Hertz to 2 significant digits, Test Frequencies of 0.01, 0.02, 0.05, 0.1, 0.2, 0.5, 1., 5., 10., 15. are typically used. If other frequencies are used they should be reported. For all tests a frequency of 10 Hz shall be included and reported.

15. Precision and Bias

- 15.1 *Precision*—The research required to develop estimates of precision based on the procedures described in this proposed standard has not yet been conducted.
- 15.1.1 Tentative criteria for judging the acceptability of dynamic shear results obtained by this method are given in

G* isotherm at 58°C for PG 58 binder mix 7% air voids lab compacted mix

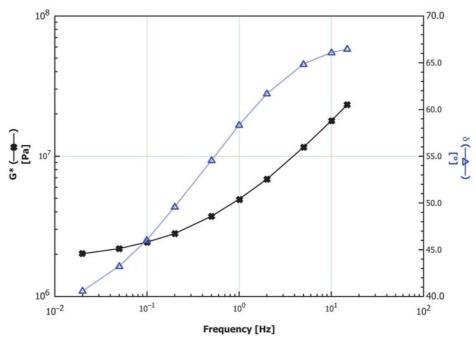


FIG. 4 Complex Shear Modulus Result for PG 58-28 Mix with 7 % Air Voids at 58°C

G* isotherms at 58°C & 70°C for PG 76 PMA binder mix from field cores

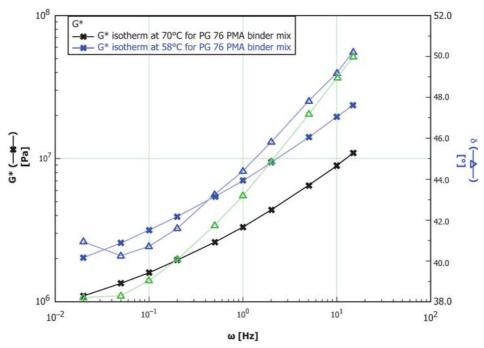


FIG. 5 Complex Shear Modulus Result for PG 76-22 Mix with 4 % Air Voids at 58°C and 70°C

Temperature MasterCurve @ 10 Hz reference frequency PG 58-28 MIX @ 7% AV

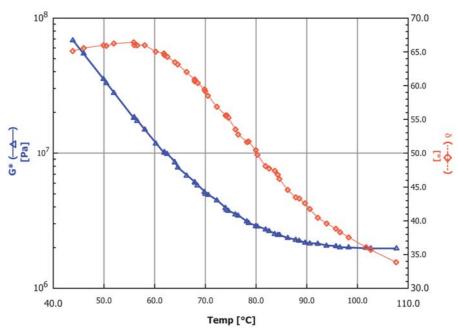


FIG. 6 Temperature Mastercurve at 10-Hz Reference Frequency for PG 58-28 Mix

Table 1. The single operator precision is based on results obtain in one laboratory performing the test on mix specimens in triplicate on a single machine. Multi-laboratory testing has not been performed.

15.1.2 *Single-Operator Precision (Repeatability)*— Triplicate results obtained by the same operator using the same

equipment in the same laboratory shall not be considered suspect unless the coefficient of variation of the triplicate results, expressed as a percent, exceeds the values given in Table 1, column 2.

15.1.3 Multilaboratory Precision (Reproducibility)—The between-laboratory reproducibility of this test method is being

COMPARE 70-28 SBS, E-1, 6.9% & 4.1% VOIDS (Temp = 70°C)

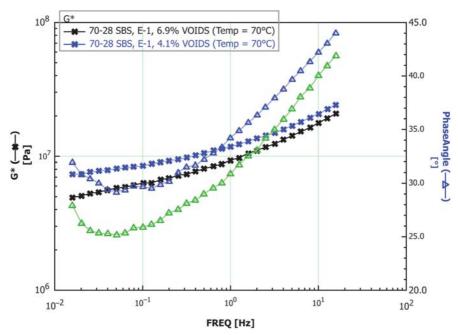


FIG. 7 Compare Complex Modulus Results for PG 70-28 Mixes with 6.9 % and 4.1 % Air Voids at 70°C

TABLE 2 Typical Report Format for Test Results

File Name:			
Sample Identifier:			
Operator:			
Test Date:			
Sample Dimensions: Width	mm, Thickness	mm, Gauge Length	_mm
Mixture Composition and Volum	etric Properties		
Mix Type	(for example dense grade	d 12.5 mm)	
Binder Grade			
Mixture Air Voids			
Mixture Source	(for example, labor	ratory specimen, field core, field QC	specime

Test Frequency,	Test Temperature,	Corrected Test	Strain, %	Complex Shear	Phase Angle, °
Hz	°C to nearest	Temperature,	to nearest 0.1%	Modulus, G*, Pa	G .
	0.01°C	if applicable			
0.01					
0.02					
0.05					
0.1					
0.2					
0.5					
1.					
5.					
10.					
15.					

determined and will be available on or before June 2012. Therefore, this test method should not be used for acceptance or rejection of materials for purchasing purposes.

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

16. Keywords

16.1 bituminous mixture; complex shear modulus; DSR; dynamic shear rheometer; torsion rectangular specimen

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