Designation: D7552 – 22

Standard Test Method for Determining the Complex Shear Modulus (G*) of Asphalt Mixtures Using Dynamic Shear Rheometer[1]

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 asphalt mixtures using torsion rectangular geometry on a dynamic shear rheometer (DSR). It is applicable to asphalt mixtures having complex shear modulus values greater than 1×10^4 Pa when tested over a range of temperatures from –40 °C to 76 °C at frequencies of 0.01 to 25 Hz and strains of 0.0005 % to 0.1 %. The determination of complex shear modulus is typically determined at 20 °C to 70 °C at 0.01 % strain at ten 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 asphalt mixtures as required for specification testing or quality control of asphalt 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 Since a precision estimate for this standard has not been developed, the test method is to be used for research and informational purposes only. Therefore, this standard should not be used for acceptance or rejection of a material 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 The text of this standard references notes and footnotes which provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D8 Terminology Relating to Materials for Roads and Pavements

D2041/D2041M Test Method for Theoretical Maximum Specific Gravity and Density of Asphalt Mixtures

D3203 Test Method for Percent Air Voids in Compacted Asphalt Mixtures

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

D6752/D6752M Test Method for Bulk Specific Gravity and Density of Compacted Asphalt Mixtures Using Automatic Vacuum Sealing Method

D6857/D6857M Test Method for Maximum Specific Gravity and Density of Asphalt 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 Asphalt Mixture Specimens Using Marshall Apparatus

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

[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 Fundamental/Mechanistic Tests.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

D7312 Test Method for Determining the Permanent Shear Strain and Complex Shear Modulus of Asphalt Mixtures Using the Superpave Shear Tester (SST) (Withdrawn 2019)[3]

E77 Test Method for Inspection and Verification of Thermometers

E644 Test Methods for Testing Industrial Resistance Thermometers

2.2 *Other Standards:*

DIN Standard 43760 Standard for Calibration of Platinum Resistance Thermometers[4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *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.

3.1.1.1 *Discussion*—Calibration is typically performed by the manufacturer or an external commercial calibration service.

3.1.2 *complex shear modulus (G*), n*—a complex number that is defined by the ratio of shear stress to shear strain.

3.1.3 *dummy test specimen, n*—a rectangular prismatic or cylindrical specimen of bituminous mix prepared as discussed in 9.2, into which a small hole is drilled and into which a PRT wire is inserted.

3.1.3.1 *Discussion*—The dummy specimen is mounted in the torsion fixture of the DSR for the purpose of measuring the internal temperature of the asphalt mixture sample for use in determining thermal equilibrium times.

3.1.4 *loading cycle, n*—refers to the application of sinusoidal stress or strain loading for a specified duration.

3.1.5 *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.

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

3.1.7 *shear stress, n*—the force per unit area that produces the flow.

3.1.8 *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.

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

3.1.10 *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.

3.2 For definitions of other terms used in this standard, refer to Terminology D8.

4. Summary of Test Method

4.1 This standard contains the procedure used to measure the complex shear modulus of an asphalt 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 50 mm in length, 12 mm in width, and 10 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 asphalt 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 50 mm length forming a vertical dimension in the DSR.

4.4 During testing, one of the fixtures[5] is rotated with respect to the other at a pre-selected percent strain and a range of frequencies at the selected temperatures. The test shall be conducted at 0.01 % strain unless otherwise stated. The percent strain stipulated in this test method has been found to produce acceptable results for the asphalt 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 percent 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 asphalt 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 mixes, mixtures with higher amounts of fines (material finer than 75 µ), 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.

NOTE 2—The quality of the results produced by this standard are

[3] The last approved version of this historical standard is referenced on www.astm.org.

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

[5] 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.

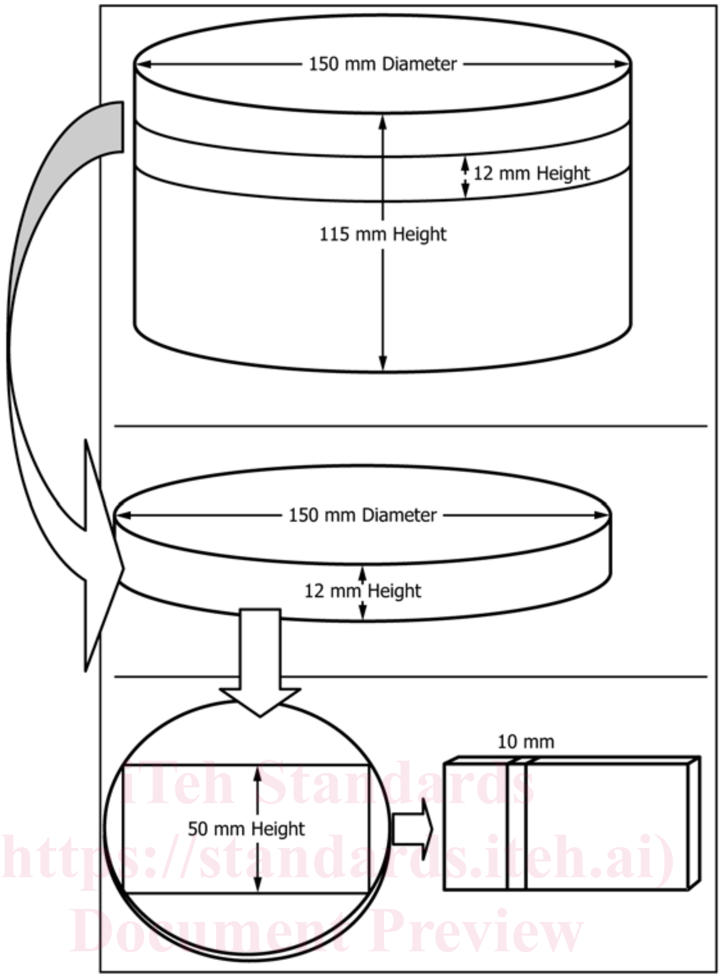

FIG. 1 Schematic of Preparing Torsion Rectangular Specimens

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

6. 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 are determined in the central portion of the test specimen.

7. Apparatus

7.1 *Saw*—The saw should be a water-cooled diamond rim saw capable of holding the specimens steady while cutting. Typically, a vise or other clamping system is required to maintain the placement of the samples while specimens are cut to size.

7.2 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.

7.3 *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.

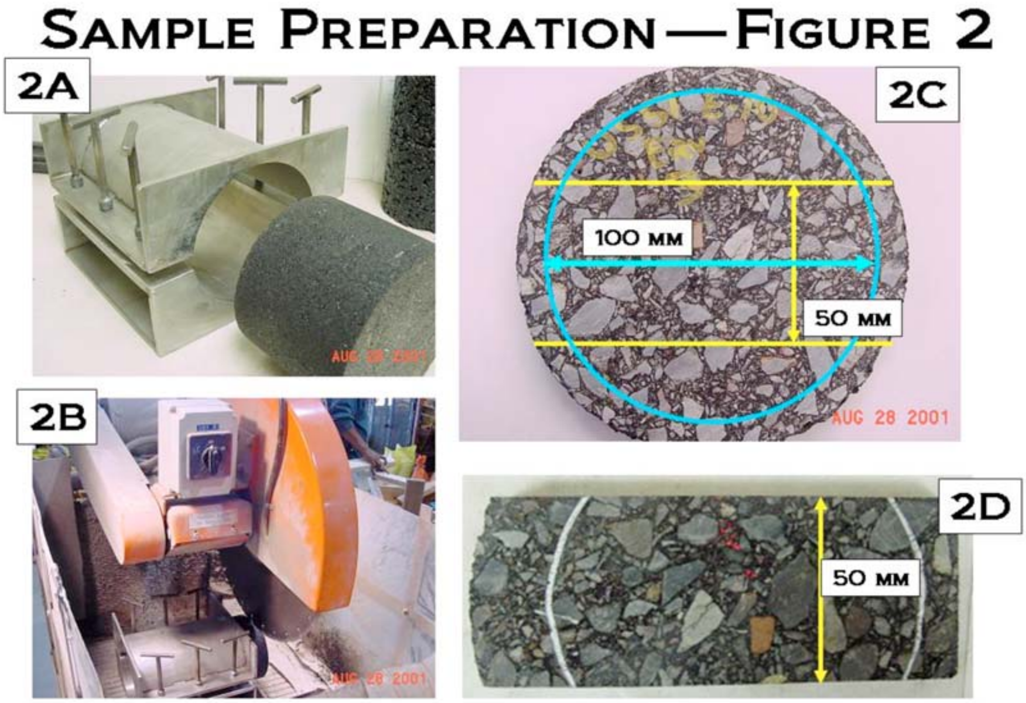

FIG. 2 Sample Preparation to Obtain 50 mm Wide by 12 mm Thick Rectangular Specimen

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

7.4 A torque wrench capable of applying a torque load of 0.25 N·m (250 mN·m) of torque to tighten the test specimen in the mounting fixture without crushing. The amount of torque applied to the sample may be adjusted based on the sample being tested to ensure a secure fit within the geometry.

7.5 *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 at 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 ± 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.

7.6 *Temperature Controller*—A temperature controller capable of maintaining the temperature of the test specimen at the test temperature ± 0.1 °C for test temperatures stipulated.

7.7 *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 20 °C to 70 °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.

7.8 *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 %. 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.

7.9 *Data Acquisition System*—The data acquisition system shall provide a record of temperature, frequency, deflection angle, percent 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 %.

7.10 *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) (8.3.1) or NIST-traceable digital electronic thermometer (8.3.2) shall be maintained in the laboratory as a reference standard. This reference standard shall be used to verify the portable thermometer (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 twelve-month interval.

8.3.2 *Digital Electronic Thermometer*—An electronic thermometer that incorporates a resistive detector with an accuracy of ±0.05 °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 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. The reference thermometer (see 8.3) may be used for this purpose if its detector fits within the dummy specimen.

9. Verification

9.1 Verify the DSR and its components as described in this section when the DSR is newly installed, when it is moved to a new location, or whenever the accuracy of the DSR or any of its components is suspect. Verification of the DSR required to perform solids testing follows the procedures detailed in the verification section of Test Method D7175.

NOTE 3—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 subsection 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.

9.2.1 *Determination of Temperature Correction*—Place the portable thermometer in the testing fixture such that the sensor is in the midpoint of the fixture where a typical sample would be positioned during a test. Obtain simultaneous temperature measurements with the internal 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 internal DSR thermometer