Standard Test Method for Rubber Compounds—Measurement of Unvulcanized Dynamic Strain Softening (Payne Effect) Using Sealed Cavity Rotorless Shear Rheometers[1]

This standard is issued under the fixed designation D8059; 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 use of a sealed cavity rotorless oscillating shear rheometer for the measurement of the softening effects of rising sinusoidal strain when applied to an unvulcanized rubber compound containing significant amounts of colloidal fillers (such as silica or carbon black, or both) from a rubber mixing procedure. These strain softening properties relate to mixing conditions, the composition of the rubber compound, colloidal particle (Payne Effect) characteristics of the fillers, and in some cases the degree of reaction between an organosilane and precipitated, hydrated silica during mixing. This procedure is being commonly applied to rubber reactive mixing procedures.

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1566 Terminology Relating to Rubber

3. Terminology

3.1 *Definitions:*

3.1.1 *elastic torque, S', n*—the peak amplitude torque component which is in phase with a sinusoidally applied strain.

3.1.2 *viscous torque, S", n*—the peak amplitude torque component which is 90° out of phase with a sinusoidally applied strain.

3.1.3 *complex torque, S*, n*—the peak amplitude torque response measured by a reaction torque transducer for a sinusoidally applied strain; mathematically, S* is computed by $S^* = (S'^2 + S''^2)^{1/2}$.

3.1.4 *loss angle, δ, n*—the phase angle by which the complex torque (S*) leads a sinusoidally applied strain.

3.1.5 *storage shear modulus, G', n*—the ratio of (elastic) peak amplitude shear stress to peak amplitude shear strain for the torque component in phase with a sinusoidally applied strain; mathematically, G' = [(S'/Area)/Peak Strain].

3.1.6 *loss shear modulus, G", n*—the ratio of (viscous) peak amplitude shear stress to peak amplitude shear strain for the torque component 90° out of phase with a sinusoidally applied strain; mathematically, G" = [(S"/Area)/Peak Strain].

3.1.7 *complex shear modulus, G*, n*—the ratio of peak amplitude shear stress to peak amplitude shear strain; mathematically, $G^* = [(S^*/\text{Area})/\text{Strain}] = (G'^2 + G''^2)^{1/2}$.

3.1.8 *loss factor, tan δ, n*—the ratio of loss modulus to storage modulus, or the ratio of viscous torque to elastic torque; mathematically, tan δ = G"/G' = S"/S'.

3.1.9 *Payne Effect, n*—in the low amplitude dynamic testing of filled rubbers, the decrease in modulus (G' or E') as the amplitude of deformation is increased.

3.1.9.1 *Discussion*—The effect is caused by a decrease in the additive contributions of polymer-polymer interactions, hydrodynamic effects, the polymer-filler and filler-filler interactions. **D1566**

4. Summary of Test Method

4.1 An uncured rubber compound specimen is contained in a sealed die cavity which is closed and maintained at an elevated temperature. The cavity is formed by two dies, one of which is oscillated through a rotary amplitude. This action produces a sinusoidal torsional strain in the test specimen resulting in a sinusoidal torque, which measures a viscoelastic quality of the test specimen. The test specimen can be an uncured rubber compound containing carbon black, precipitated hydrated silica, or both of these reinforcing fillers. The silica loaded compounds may also contain an organosilane

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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.

with differing degrees of reaction (silanization) from differing prior combinations of mixing time and temperature.

4.2 The Payne effect is a phenomenon in the low amplitude dynamic testing of strain dependent filled rubbers where the modulus (G' and E') decreases as the amplitude of deformation is increased. This dynamic modulus of filled elastomers and rubbers at low amplitude deformations consists of additive contributions of the polymer-polymer interaction, the hydrodynamic effect, the polymer-filler interaction, and the filler-filler network caused by the filler-filler interaction. Many factors, including the type and surface area of colloidal fillers such as carbon black and precipitated hydrated silica, the filler concentration, silanization and other treatments, can directly effect this filler-filler interaction as measured by the Payne Effect. Fig. 1 illustrates this Payne Effect.

4.3 Typically the uncured rubber specimen is sealed under pressure in the closed cavity for exactly 10 min with a very low sinusoidal oscillation frequency of 0.07 % (conditioning), followed by a broad strain sweep at the programmed processability temperature.

4.4 These viscoelastic measurements can be made from the broad strain sweep based on a strain amplitude sweep in which the applied strain is preprogrammed to start at 0.07 % strain and to increase in steps under constant frequency and temperature conditions till reaching 300 % strain at 1 Hz.

5. Significance and Use

5.1 This test method is used to measure viscoelastic properties through the strain softening effects of a strain amplitude sweep (the Payne Effect).

5.2 For the uncured state, the time conditioning and strain amplitude strain sweeps can relate to colloidal silica particle or carbon black deagglomeration from the mixing process. The profile of this Payne Effect from G' storage modulus can also be a function of loading levels and particle size of these fillers in the rubber hydrocarbon medium. In addition, with silica and an organosilane additive, this G' strain softening effect can determine if a given silanization reaction between a subject silica and an organosilane was achieved through reactive mixing. If the silanization reaction during the mixing was not achieved, the maximum G' storage modulus from the strain sweep will not be lowered and the silica particle attraction to other silica particles will still be high resulting in a more dense filler network that remains.

6. Apparatus

6.1 *Torsion Strain Rotorless Oscillating Rheometer with a Sealed Cavity*—This type of rheometer measures the elastic torque S' and viscous torque S" produced by oscillating angular strain of set amplitude and frequency in a completely closed and sealed test cavity.

6.2 *Sealed Die Cavity*—The sealed die cavity is formed by two biconical dies. In the measuring position, the two dies are fixed a specified distance apart so that the cavity is closed and sealed (see Fig. 2).

6.3 *Die Gap*—For the sealed cavity, no gap should exist at the edges of the dies. At the center of the dies, the die gap shall be set at 0.45 ± 0.05 mm.

6.4 *Die Closing Mechanism*—For the sealed cavity, a pneumatic cylinder or other device shall close the dies and hold them closed during the test with a force not less than 11 kN (2500 lbf).

6.5 *Die Oscillating System*—The die oscillating system consists of a direct drive motor which imparts a torsional oscillating movement to the lower die in the cavity plane.

6.6 *Torque Measuring System*—The torque measuring system shall measure the resultant shear torque.

6.6.1 The torque measuring device shall be rigidly coupled to one of the dies, any deformation between the die and device shall be negligibly small, and the device shall generate a signal which is proportional to the torque. The total error resulting from zero point error, sensitivity error, linearity, and %repeatability errors shall not exceed 1 % of the selected measuring range.

6.6.2 The torque recording device shall be used to record the signal from the torque measuring device and shall have a

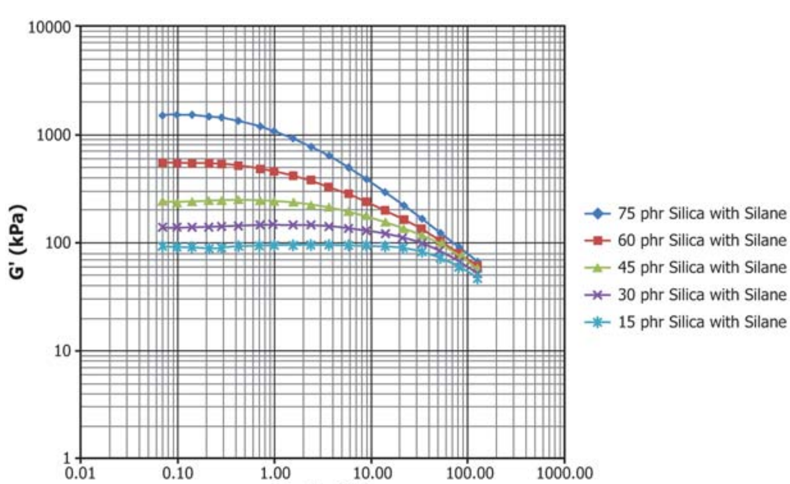

FIG. 1 Effect of Filler Loading on Measured Payne Effect

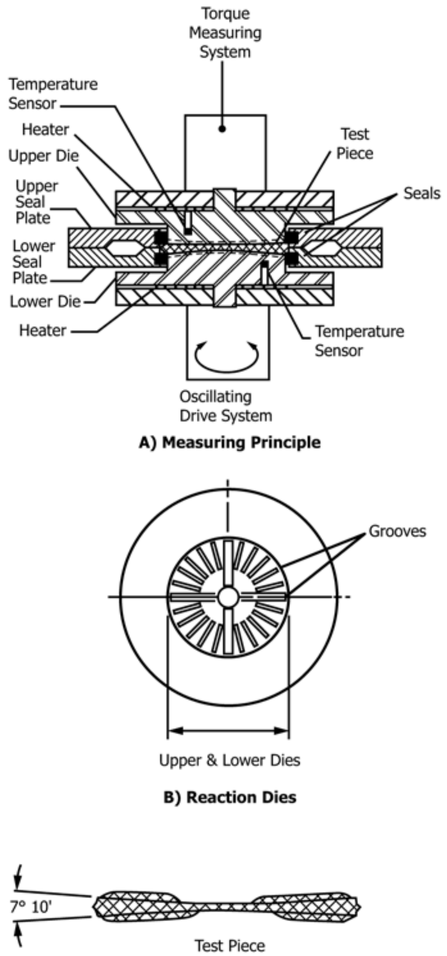

FIG. 2 Typical Sealed Torsion Shear Rotorless Rheometer with Biconical Dies

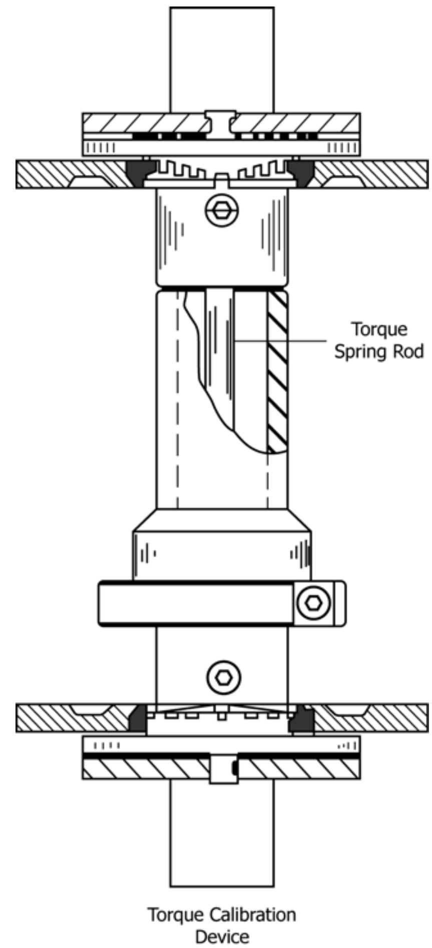

FIG. 3 Typical Torque Standard Calibration Device for Torsion Shear Curemeters

response time for full scale deflection of the torque scale of 1 s or less. The torque shall be recorded with an accuracy of ±0.5 % of the range. Torque recording devices may include analog chart recorders, printers, plotters, or computers.

6.6.3 A reference torque device is required to calibrate the torque measurement system. A torque standard may be used to calibrate the torque measuring system at the selected angular displacement by clamping a steel torsion rod to the oscillating and the torque measuring dies of the torsion shear rheometer (see Fig. 3). The reference values for angular displacement and corresponding torque shall be established by the manufacturer for each torque standard.

6.7 *Reference Test Temperature*—The standard reference test temperature for uncured rubber compounds for measuring the Payne effect is 70°C.

6.8 *Temperature Control System*—This system shall permit the reference temperature to be set to 70°C.

6.8.1 The dies shall heat to the set point temperature in 1.0 min or less from closure of the test cavity. Once the initial heating up time has been completed, die temperature shall not vary by more than ±0.3°C for the remainder of a test at a set temperature.

6.8.2 Temperature distribution within the test piece shall be as uniform as possible. Within the deformation zone, a tolerance of ±1°C of the average test piece temperature shall not be exceeded.

6.8.3 Die temperature is determined by a temperature sensor used for control. The difference between the die temperature and the average test piece temperature shall not be more than 2°C. Temperature measurement accuracy shall be ±0.3°C for the die temperature sensor.

7. Test Specimen

7.1 A test specimen taken from a sample shall be between 5 and 6 cm^3 for the sealed cavity oscillating rheometer. The specimen volume should exceed the test cavity volume by a small amount, to be determined by preliminary tests. Typically, specimen volume should be 110 to 130 % of the test cavity volume. Once a target mass for a desired volume has been established, specimen masses should be controlled to within ±0.1 g for best repeatability. The initial test specimen shape should fit well within the perimeter of the test cavity.

7.2 *Uncured Rubber Specimens*—Condition the mixed stock specimen until it has reached room temperature (23 ± 3°C (73 ± 5°F)) throughout. The uncured rubber test specimen should be tested as received, that is unmassed (not milled).

7.2.1 The rubber compound shall be in the form of a sheet, at room temperature, and as free of air as possible.

7.2.2 Rubber test specimens in a sealed cavity oscillating rheometer must be preconditioned in the instrument before rheological measurements are made to improve test repeatability. A programmed pre-conditioning step shall consist of oscillating the specimen at 0.1 Hz, ±0.07 % strain, 70°C for 10 min, as specified in Table 1. Any deviations from these standard frequency, strain and time duration can have significant effect on the Payne Plateau.

8. Procedure

8.1 Select the strain, frequency, temperature and time for the conditioning step as listed for rubber compounds in Table 1.

8.2 Select the strain steps and the frequency and temperature conditions for the amplitude strain sweep for the uncured rubber compounds as given in Table 1.

8.3 Quantitatively weigh and cut a specimen from the uncured subject rubber compound sample to within ±0.1 g of the target mass for the subject compound, which is based on the mass of the subject material at 120 % cavity fill factor (reference 7.1).

8.4 Program the test configuration to run the test.

8.5 Enter specimen identification.

8.6 Wait until both dies are at the initial test temperature. Open the test cavity and visually check both upper and lower dies for cleanliness. Clean the dies if necessary. Place a sheet of 23-micron thick Nylon 6,6 film over the lower die. Place the test specimen on the film on the center of the lower die, lay a second sheet of Nylon 6,6 film on top of the specimen, and close the dies within 20 s.

TABLE 1 Standard Test Conditions for Rotorless Shear Rheometer with Closed Biconical Die Cavity

Test Conditions	
Compound Conditioning for Payne Effect Measurement	
Temperature (ºC)	70
Strain (± %)	0.07
Frequency (Hz)	0.1
Time (min.)	10
Uncured Payne Effect Strain Sweep	
Temperature (ºC)	70
Frequency (Hz)	1.0
Strains (± %)	0.07, 0.08, 0.09, 0.10, 0.12, 0.14, 0.16, 0.18, 0.20, 0.25, 0.30, 0.40, 0.50, 0.6, 0.8, 1.0, 2.0, 4.0, 7.0, 10.0, 20.0, 50.0, 80.0,100.0, 124, 300

9. Report

9.1 Report the following information.

9.1.1 A full description of the sample or test specimen(s), or both, including their origin.

9.1.2 Type and model of oscillating rheometer.

9.1.3 The frequency, strain, temperature and time for the conditioning step.

9.1.4 The frequency in Hertz and temperature in degrees Celsius for all strain sweeps.

9.1.5 Report maximum G' in kPa units and note % strain at maximum G'.

9.1.6 Report G' in kPa units at 0.1 %, 1.0 % and 10 % strain amplitude.

10. Precision and Bias

10.1 A precision and bias estimate has not been completed for this test method at this time.

11. Keywords

11.1 loss modulus; loss tangent; Mullins Effect; Payne Effect; processability test; rheological properties; rotorless oscillating shear rheometer; storage modulus; strain softening effect; tan delta; tangent delta; VE ratio; viscosity