



Standard Test Method for Thermoplastic Elastomers—Measurement of Polymer Melt Rheological Properties and Congealed Dynamic Properties Using Rotorless Shear Rheometers¹

This standard is issued under the fixed designation D7605; 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 rotorless oscillating shear rheometer for the measurement of the flow properties of polymer melts and their respective congealed dynamic properties for thermoplastic elastomers (TPE) which includes thermoplastic vulcanizates (TPV). These flow properties and congealed dynamic properties are related to factory processing and product performance.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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

D1349 Practice for Rubber—Standard Conditions for Testing

D3896 Practice for Rubber From Synthetic Sources—Sampling

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

D6600 Practice for Evaluating Test Sensitivity for Rubber Test Methods

3. Terminology

3.1 Definitions:

¹ This test method is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.12 on Processability 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.

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

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

3.1.3 *congealed thermoplastic, n* —thermoplastic polymer that when cooled from the melt state exhibits a substantial portion of the physical properties of the solid state.

3.1.4 *dynamic complex viscosity, η^* , n* —ratio of the complex shear modulus, G^* to the oscillation frequency, ω , in radians per second.

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

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

3.1.7 *loss factor, $\tan \delta$, n* —ratio of loss modulus to storage modulus, or the ratio of viscous torque to elastic torque; mathematically, $\tan \delta = G''/G' = S''/S'$.

3.1.8 *loss shear modulus, G'' , n* —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, $G'' = [(S''/\text{Area})/\text{Peak Strain}]$.

3.1.9 *real dynamic viscosity, η' , n* —ratio of the loss shear modulus, G'' to the oscillation frequency, ω , in radians per second.

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

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

4. Summary of Test Method

4.1 A thermoplastic elastomer test 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 a thermoplastic elastomer such as a thermoplastic vulcanizate (TPV), a styrenic blocked copolymer (SBC), a copolyester, a thermoplastic polyurethane, a thermoplastic polyolefin, or other TPE forms.

4.2 These viscoelastic measurements can be made based on (1), a frequency sweep in which the frequency is programmed to change in steps under constant strain amplitude and temperature conditions, (2), a strain sweep in which the strain amplitude is programmed to change in steps under constant frequency and temperature conditions, or (3), a temperature sweep in which the temperature is programmed to either increase or decrease under constant strain amplitude and frequency conditions. A timed test may also be performed in which a sinusoidal strain is applied for a given time period under constant strain amplitude, frequency and temperature conditions.

4.2.1 For a frequency sweep test, the instrument is typically programmed to increase the frequency with each subsequent step change. For a strain sweep test, the instrument is usually programmed to increase the strain amplitude with each subsequent step change. This is done to minimize the influence of prior test conditions on subsequent test steps. For temperature sweeps, the temperature may be programmed either to increase or decrease with each subsequent step change, depending on the effects to be studied. The results from increasing frequency, strain amplitude or temperature may not be the same as results from decreasing these test parameters.

4.3 Rheological properties are measured for each set of frequency, strain and temperature conditions. These properties can be measured as combinations of elastic torque S' , viscous torque S'' , storage shear modulus, G' , loss shear modulus G'' , $\tan \delta$, complex dynamic viscosity η^* , and real dynamic viscosity η' .

5. Significance and Use

5.1 This test method is used to measure viscoelastic properties of thermoplastic elastomer polymer melts at elevated temperatures as well as the dynamic properties of the respective congealed thermoplastic elastomer specimens measured at lower temperatures. These polymer melt viscoelastic properties may relate to factory processing behavior while the dynamic properties of the respective congealed specimen may relate to product performance.

5.2 This test method may be used for quality control in thermoplastic elastomer manufacturing processes, for quality control of received shipments of thermoplastic elastomers, and for research and development testing of thermoplastic elastomers. This method may also be used for evaluating processing and product performance differences resulting from the use of different compounding materials and process conditions in the making of these thermoplastic elastomers.

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 parallel plate dies. In the measuring position, the two dies are fixed a specified distance apart so that the cavity is closed and sealed (see Fig. 1).

6.3 *Die Gap*—For the sealed cavity, no gap should exist at the edges of the dies. The die gap for these parallel plate dies shall be set at 2.58 ± 0.1 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.

6.5.1 The oscillation amplitude can be varied, but a selection of $\pm 0.5^\circ$ arc (7.0 % shear strain) is preferred for frequency sweep tests. The oscillation frequency can be varied between 0.03 Hz and 30 Hz.

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

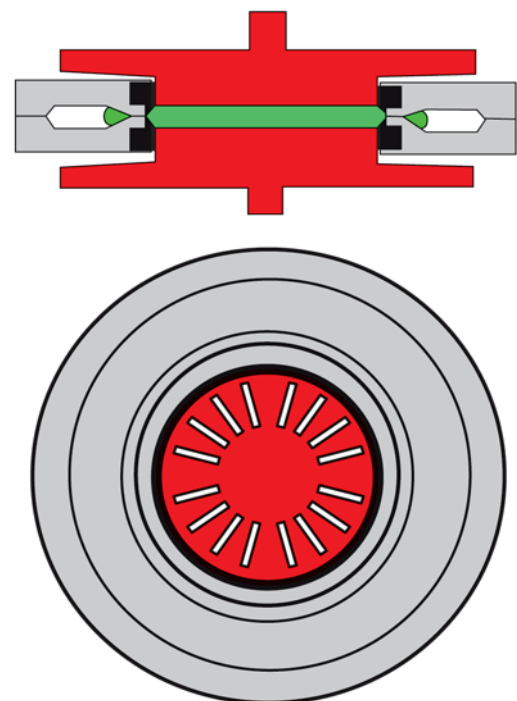


FIG. 1 Typical Sealed Torsion Shear Rotorless Rheometer with Parallel Plate Dies