Designation: D7605 − 11 (Reapproved 2016)

Standard Test Method for Thermoplastic Elastomers—Measurement of Polymer Melt Rheological Properties and Congealed Dynamic Properties Using Rotorless Shear Rheometers[1]

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:*

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

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 δ, 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, $G'' = [(S''/Area) /Peak\ Strain]$.

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, $G' = [(S'/Area) /Peak\ Strain]$.

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

[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials 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.

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 δ, 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 ±0.5° 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

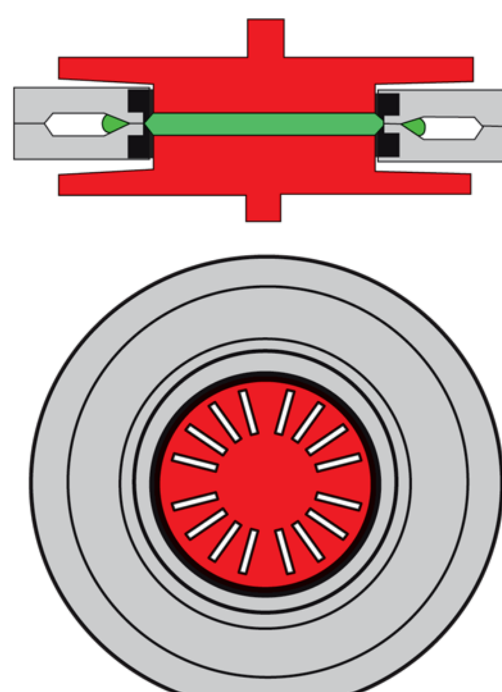

FIG. 1 Typical Sealed Torsion Shear Rotorless Rheometer with Parallel Plate Dies

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 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. 2). 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 thermoplastic elastomer polymer melts depend on the melt transition temperature of the specific class of TPE being tested. For example, measurements of the rheology of the polymer melts of thermoplastic vulcanizates based on EPDM rubber and polypropylene are commonly tested at 215ºC with dynamic property measurements of the congealed specimen performed at 60ºC. For styrenic blocked copolymer thermoplastic elastomers, measurements of the polymer melts are performed at 200ºC with dynamic property measurements of the congealed specimen performed at 60ºC. For thermoplastic vulcanizates based on polyacrylate rubber and nylon, measurements of the polymer melts are performed at 250ºC with dynamic property measurements of the congealed specimen performed at 60ºC. Tests may be carried out at other temperatures if required. Temperatures should be selected, when practical, in accordance with Practice D1349.

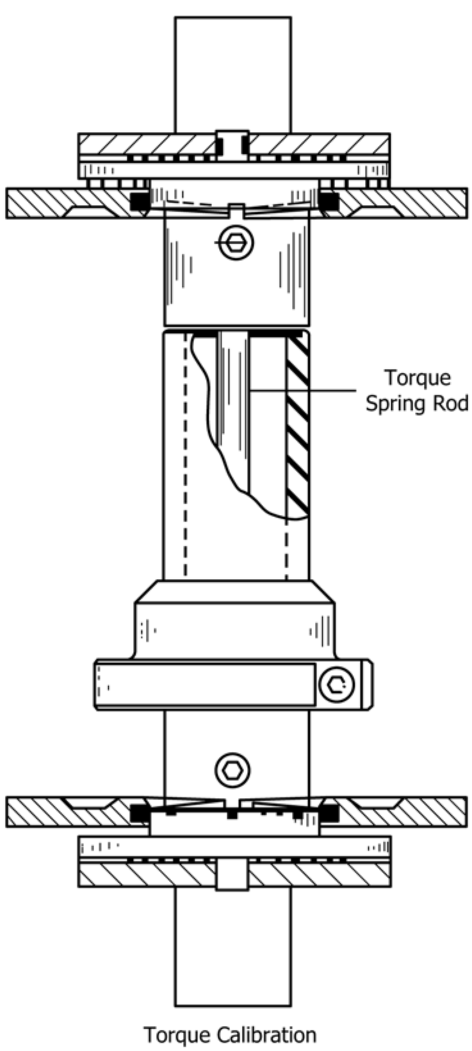

FIG. 2 Typical Torque Standard Calibration Device for Torsion Shear Curemeters

6.8 *Temperature Control System*—This system shall permit the reference temperature to be varied between 40°C and 250°C with an accuracy of ±0.3°C or better.

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. When the set temperature is changed in a programmed temperature sweep, rheological measurements should not be recorded until the die temperatures are within ±0.3°C of the new set temperature for at least 30 s.

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 carefully cut to within ±0.02 g of the target mass, which is equal to mass representing 105 % of the fill factor for the die cavity. The mass for 100 % fill factor can be empirically determined by testing a specimen from the sample that has a mass greater than the 100 % fill factor. After completing this test, the test specimen is carefully removed from the cavity, the film is carefully removed, and the flash is carefully cut away from this specimen. The sample is weighed to the nearest one hundredth of a gram. This weight is then multiplied by 1.05 to determine the target mass for all future tests for this thermoplastic elastomer series of materials. Once a target mass for a desired TPE series (all with the same specific gravity) has been established, all future specimen masses for this family of thermoplastic elastomers should be controlled to within ±0.02 g for best repeatability. The initial test specimen shape should fit well within the perimeter of the test cavity.

7.2 *Thermoplastic Elastomer Specimens*—Condition the specimen obtained in accordance with Practice D1349 until it has reached room temperature (23 ± 2.0°C (73.4 ± 3.6°F)) throughout. The thermoplastic elastomer test specimen should be tested as received, that is unmassed (not milled).

7.2.1 Thermoplastic elastomer specimens in a sealed cavity oscillating rheometer must be pre-conditioned in the instrument before rheological measurements are made to improve test repeatability. A programmed pre-conditioning step shall consist of oscillating the specimen at 0.5 Hz, ±2.8 % strain, at the predetermined polymer melt temperature for the time interval of 0.5 min.

8. Procedure

8.1 Select the frequency, strain, temperature and time for the conditioning step as listed for thermoplastic elastomer melt in Table 1.

8.2 Select the frequency steps and the strain and temperature conditions for the frequency sweep for thermoplastic elastomer melt as listed in Table 1.

8.3 Select the strain steps and the frequency and temperature conditions for the strain sweep for the thermoplastic elastomer melt in Table 1.

8.4 Select the strain steps and frequency and temperature conditions for the two "back to back" strain sweep for the congealed thermoplastic elastomer in Table 1.

8.5 Program a test configuration which incorporates all these conditions and store on the instrument computer operating system.

8.6 Quantitatively weigh and cut a specimen from the thermoplastic elastomer sample to within ±0.02 g of the target mass for the thermoplastic elastomer, which is based on the mass of the subject material at 105 % cavity fill factor (reference 7.1).

8.7 Load the test configuration to run the test.

8.8 Enter specimen identification.

8.9 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 film over the lower die (use nylon 6,6 film if the temperature is below 225°C). Place the test specimen on the film on the center of the lower die, lay a second sheet of film on top of the specimen, and close the dies within 20 s. The test shall then run as programmed.

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 strain amplitude in ± degrees of arc or ± percent strain for the frequency sweep and the strain sweeps.

9.1.5 The temperature of the frequency sweep and strain sweeps.

9.1.6 The storage shear modulus G' in kPa and the frequency in Hz for each step in the programmed frequency sweep.

9.1.7 The loss shear modulus G" in kPa and the frequency in Hz for each step in the programmed frequency sweep.

9.1.8 The dynamic complex viscosity η* in kPa-sec. and the frequency in radians per second for each step in the programmed frequency sweep.

9.1.9 The tangent delta (tan δ) and the frequency in Hz for each step in the programmed frequency sweep.

9.1.10 The storage shear modulus G' in kPa and the strain in percent for each step in the programmed strain sweeps.

TABLE 1 Standard Test Conditions for Oscillating Rheometer with Closed Parallel Plate Die Cavity

Test Conditions	EPDM/PP TPV	SBC Compounds	Nylon/ACM TPV
TPE Melt Conditioning			
Temperature (ºC)	215	200	250
Strain (± %)	2.8	2.8	2.8
Frequency (Hz)	0.5	0.5	0.5
Time (min)	0.5	0.5	0.5
TPE Melt Frequency Sweep			
Temperature (ºC)	215	200	250
Strain (± %)	7	7	7
Frequencies (Radians/Sec)	0.2,0.5,1,2,5,10,20, 50,100, and 200	0.2,0.5,1,2,5,10,20, 50,100, and 200	0.2,0.5,1,2,5,10,20, 50,100, and 200
TPE Melt Strain Sweep			
Temperature (ºC)	215	200	250
Frequency (Hz)	0.1	0.1	0.1
Strains (± %)	10,20,50,100	10,20,50,100	10,20,50,100
TPE First Congealed Strain Sweep			
Temperature (ºC)	60	60	60
Frequency (Hz)	1	1	1
Strains (± %)	0.5,1,2,5,10	0.5,1,2,5,10	0.5,1,2,5,10
TPE Second Congealed Strain Sweep			
Temperature (ºC)	60	60	60
Frequency (Hz)	1	1	1
Strains (± %)	0.5,1,2,5,10	0.5,1,2,5,10	0.5,1,2,5,10

9.1.11 The loss shear modulus G'' in kPa and the strain in percent for each step in the programmed strain sweeps.

9.1.12 The tangent δ and the strain in percent for each step in the programmed strain sweeps.

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 dynamic complex viscosity; loss modulus; processability test; rheological properties; rotorless oscillating shear rheometer; storage modulus; thermoplastic elastomer; thermoplastic vulcanizate (TPV); TPE; viscosity