



Designation: D 5289 – 06

Standard Test Method for Rubber Property—Vulcanization Using Rotorless Cure Meters¹

This standard is issued under the fixed designation D 5289; 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 a method for the measurement of selected vulcanization characteristics of rubber compounds using rotorless linear shear, unsealed torsion shear, and sealed torsion shear cure meters. The three types of instruments may not give the same results.

NOTE 1—An alternative method for the measurement of vulcanization characteristics is given in Test Method D 2084.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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

D 1349 Practice for Rubber—Standard Temperatures for Testing

D 1556 Test Method for Density and Unit Weight of Soil in Place by the Sand-Cone Method

D 2084 Test Method for Rubber Property—Vulcanization Using Oscillating Disk Cure Meter

D 4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

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 *rotorless cure meter*—a name for a class of cure meters that uses one of the two specimen shaping members or dies to sense the torque or stress during strain application. Rotorless cure meters do not have a third member in the form of a rotor (see definitions of cure meter in Test Methods D 1556 and D 2084).

3.1.2 *torque*—for an oscillating shear cure meter, the value measured by a torque transducer at the peak strain amplitude of the oscillating cycle.

3.1.3 The following measurements may be taken from the recorded curve of force or torque as a function of time (see Fig. 1):

3.1.3.1 *maximum, plateau, or highest force or torque*—measure of the stiffness or shear modulus of the vulcanized test specimen at the vulcanization temperature, measured within a specified period of time.

3.1.3.2 *minimum force or torque*—measure of the stiffness of the unvulcanized test specimen at the specified vulcanizing temperature, taken at the lowest point in the vulcanization curve.

3.1.3.3 *time to a percentage of full cure*—measure of cure based on the time to develop some percentage of the difference in force or torque from the minimum to the maximum.

3.1.3.4 *time to incipient cure (scorch time)*—measure of the time at which a specified small increase in force or torque has occurred; it indicates the beginning of vulcanization.

4. Summary of Test Method

4.1 A rubber test piece is contained in a die cavity which may be closed or almost closed and maintained at an elevated temperature. The cavity is formed by two dies, one of which is oscillated through a small linear or rotary amplitude. This action produces a sinusoidal alternating linear or torsional strain in the test piece and a sinusoidal shear force or torque which depends on the stiffness (shear modulus) of the rubber compound. The envelope curve, which is defined as the amplitude of the oscillating force or torque, is continuously recorded as a function of time (see Fig. 2).

4.2 The stiffness of the rubber test piece increases as vulcanization proceeds. The test is completed when the recorded force or torque rises to either an equilibrium or

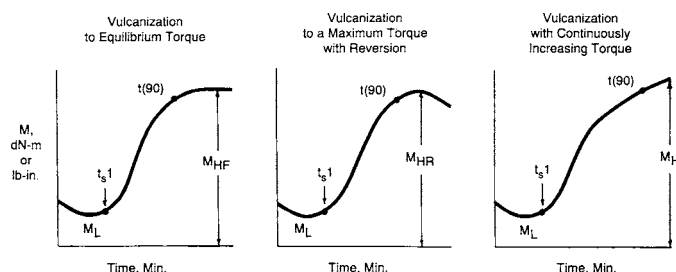
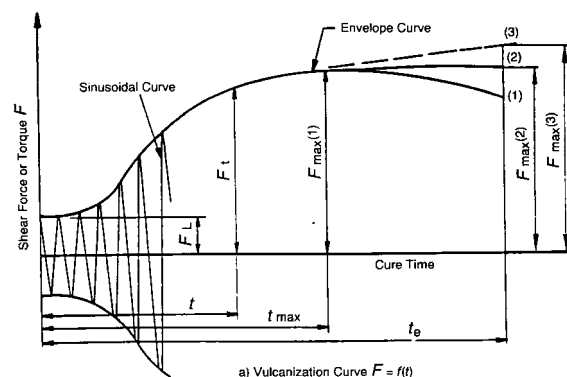


FIG. 1 Types of Vulcanization Curves



- (1) Vulcanization Curve with Maximum, $F_{\max}^{(1)}$ at Time $t_{\max}$
 (2) Vulcanization Curve with Plateau, $F_{\max}^{(2)}$
 (3) Vulcanization Curve with Steady Increase, $F_{\max}^{(3)}$ at Time t_0 at the End of Test

FIG. 2 Envelope Vulcanization Curve

maximum value, or when a predetermined time has elapsed (see Fig. 1). The time required to obtain a vulcanization curve is a function of the test temperature and the characteristics of the rubber compound.

5. Significance and Use

5.1 This test method is used to determine the vulcanization characteristics of (vulcanizable) rubber compounds.

5.2 This test method may be used for quality control in rubber manufacturing processes, for research and development testing of raw-rubber compounded in an evaluation formulation, and for evaluating various raw materials used in preparing (vulcanizable) rubber compounds.

5.3 The test piece in a rotorless cure meter approaches the test temperature in a shorter time and there is a better temperature distribution in the test piece due to the elimination of the unheated rotor found in oscillating disk cure meters.

5.4 Several manufacturers produce rotorless cure meters with design differences that may result in different torque responses and curve times for each design. Correlations of test results between cure meters of different designs should be established for each compound tested, and for each set of test conditions.

6. Apparatus

6.1 Rotorless cure meters of three types can be used. In each case, an oscillation of small amplitude is applied to one die.

6.1.1 *Linear Strain Rotorless Cure Meter*—This type of cure meter measures the force produced by a linear strain of constant amplitude (see Fig. 3(a)).

6.1.2 *Unsealed Torsion Strain Rotorless Cure Meter*—This type of cure meter measures the torque produced by an angular strain of constant amplitude in a cavity that is not completely closed (see Fig. 3(c)).

6.1.3 *Sealed Torsion Strain Rotorless Cure Meter*—This type of cure meter measures the torque produced by an angular strain of constant amplitude in a cavity that is completely closed and sealed (see Fig. 4(a)).

6.2 *Die Cavity*—The die cavity is formed by two dies. In the measuring position, the two dies are fixed a specified distance apart so that the cavity is almost closed (see Fig. 3(b) and (d)), or closed and sealed (see Fig. 4(a)).

6.2.1 The dimensions for typical linear shear curemeter dies include a diameter of 30 mm (1.18 in.), and a total depth of 4 mm (0.16 in.). The dimensions for typical torsional shear curemeters include biconical-shaped dies having a diameter of 40 ± 2 mm (1.57 ± 0.08 in.), and an angle of separation ranging from 7 to 18°, depending on the manufacturer's design. In the center of the dies, a separation equal to 0.5 mm (0.02 in.) plus the die gap should be maintained (see Fig. 3(b), Fig. 3(d), or Fig. 4(b)). Manufacturer's guidelines should be followed to determine if the dies have been excessively worn and should be replaced.

6.2.2 *Die Gap*—The gap between the edges of the dies in the closed position shall be between 0.05 and 0.20 mm (0.002 to 0.008 in.), preferably 0.1 mm (0.004 in.) for unsealed cavities. For sealed cavities, no gap should exist at the edges of the dies.

6.2.3 *Die Closing Mechanism*—A pneumatic cylinder or other device shall close the dies and hold them closed during the test with a force of not less than 8.0 kN (1820 lbf).

6.3 *Die Oscillating System*—The die oscillating system consists of a rigid eccentric drive, which imparts a linear or torsional oscillating movement to one of the dies, in the plane of the cavity.

6.3.1 The amplitude of the oscillation should be either ± 0.01 to ± 0.1 mm, preferably ± 0.05 mm (± 0.0004 to ± 0.0039 in., preferably ± 0.0020 in.) for linear shear; or $\pm 0.1^\circ$ to $\pm 3.0^\circ$, preferably $\pm 0.5^\circ$ of arc for torsional shear cure meters.

6.3.2 The frequency of oscillation should be between 0.5 and 2 Hz, preferably 1.7 ± 0.1 Hz.

6.4 *Force or Torque Measuring System*—A force or torque measuring system shall measure the resultant shear force or torque.

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

NOTE 2—The elastic deformation of the oscillating and measuring system should not be more than 1 % of the oscillating amplitude; otherwise, the curemeter curves must be corrected.

6.4.2 The force or torque recorder device shall be used to record the signal from the force or torque measuring device. It shall record the envelope (see Fig. 2) and shall have a response time for full-scale deflection on the force or torque scale of

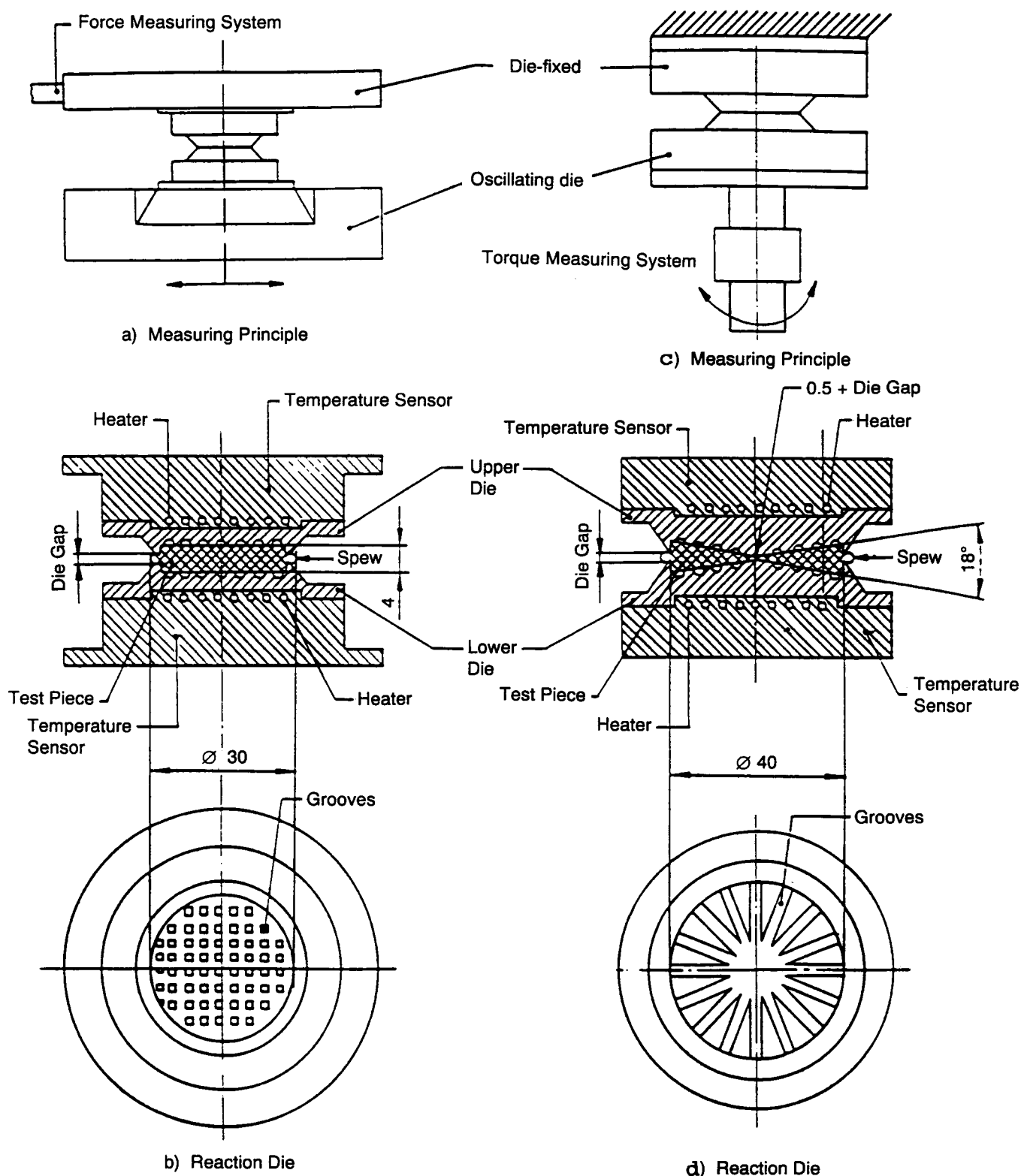


FIG. 3 (a) and (b)—Typical Linear Shear Rotorless Curemeter; (c) and (d)—Typical Unsealed Torsion Shear Rotorless Curemeter

s or less. The force or torque shall be recorded with an accuracy of $\pm 0.5\%$ of the range. Torque recording devices may include analog chart recorders, printers, plotters, or computers.

6.5 Torque or force calibration equipment is required to measure the linear or angular strain amplitude and to calibrate the force or torque measuring device. Examples of calibration