

# Standard Test Method for Rubber Property—Vulcanization Using Oscillating Disk Cure Meter<sup>1</sup>

This standard is issued under the fixed designation D 2084; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

#### 1. Scope

1.1 This test method covers the use of the oscillating disk cure meter for determining selected vulcanization characteristics of vulcanizable rubber compounds.

1.2 ISO 3417 is very similar to this test method. It has minor technical differences that are not considered to be significant.

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

1.4 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: <sup>2</sup>

- D 1349 Practice for Rubber—Standard Temperatures for Testing
- D 3185 Test Methods for Rubber—Evaluation of SBR (Styrene-Butadiene Rubber) Including Mixtures With Oil
- D 3186 Test Methods for Rubber—Evaluation of SBR (Styrene-Butadiene Rubber) Mixed With Carbon Black or Carbon Black and Oil
- D 3187 Test Methods for Rubber—Evaluation of NBR (Acrylonitrile-Butadiene Rubber)
- D 3190 Test Method for Rubber—Evaluation of Chloroprene Rubber (CR)
- D 4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

2.2 ISO Standard:

ISO 3417 Rubber-Measurement of Vulcanization Charac-

teristics With the Oscillating Disk Rheometer<sup>3</sup>

#### 3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 The following measurements may be taken from the torque versus time curve (see Fig. 1).

3.1.2 *cure rate index*—measure of rate of vulcanization based on the difference between optimum vulcanization and incipient scorch time.

3.1.3 *peak cure rate*—measure of rate of vulcanization expressed as the maximum slope of the torque versus time curve.

3.1.4 *maximum, plateau, or highest torque*—measure of stiffness or shear modulus of the fully vulcanized test specimen at the vulcanization temperature.

3.1.5 *minimum torque*—measure of the stiffness of the unvulcanized test specimen taken at the lowest point of the curve.

3.1.6 *time to incipient cure (scorch time)*—measure of the time at which vulcanization begins.

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

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

3.1.9 *optimum cure time*—measure of the time required to reach a percentage of full cure that corresponds to a desired level of a property of the cured compound.

3.1.9.1 *Discussion*—The time to reach 90 % cure corresponds to a maximum in tensile strength for some rubber compounds. This does not apply in all cases.

## 4. Summary of Test Method

4.1 A test specimen of vulcanizable rubber compound is inserted into the cure meter test cavity and after a closure action is contained in a sealed cavity under positive pressure. The cavity is maintained at some elevated vulcanization

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<sup>&</sup>lt;sup>2</sup> 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.

<sup>&</sup>lt;sup>3</sup> Available from American National Standards Institute, 25 W. 43rd St., 4th Floor, New York, NY 10036.



Left Curve: Cure to Equilibrium Torque. Middle Curve: Cure to a Maximum Torque with Reversion. Right Curve: Cure to No Equilibrium in Maximum Torque.

FIG. 1 Types of Cure Curve

temperature. The rubber totally surrounds a biconical disk after the dies are closed (see Fig. 2). The disk is oscillated through a small rotational amplitude (1° or 3°) and this action exerts a shear strain on the test specimen. The force required to oscillate or rotate the disk to maximum amplitude is continuously recorded as a function of time, with the force being proportional to the shear modulus (stiffness) of the test specimen at the test temperature. This stiffness initially decreases as it warms up; then it increases due to vulcanization. The test is completed when the recorded torque either rises to an equilibrium or maximum value, or when a predetermined time has elapsed. The time required to obtain a cure curve is a function of the characteristics of the rubber compound and of the test temperature (see Fig. 1 for typical cure curves).

4.2 Several configurations of the oscillating disk cure meter are currently in use. Fig. 3 illustrates example shifts of the cure curves associated with the configuration differences included in this standard. Results between tests using rapid and slow temperature recovery, or between heated and unheated disks



FIG. 3 Example Cure Curves from ODR Configurations

cannot be compared without taking the heating differences into account. The differences between test curves will vary with the



compound being tested. Configurations included in this test method are listed in this section.

4.2.1 Diaphragm dies, unheated rotor, temperature recovery within 4.5 min.

4.2.2 Solid dies, unheated rotor, temperature recovery within 4.5 min.

4.2.3 Solid dies, unheated rotor, temperature recovery in less than 2 min.

4.2.4 Solid dies, heated rotor, temperature recovery in less than 2 min.

NOTE 1—Diaphragm dies are unique to cure meters developed before rapid temperature recovery and heated rotors were introduced. Diaphragm dies in combination with rapid temperature recovery or heated rotors are not a normal configuration for Oscillating Disk Cure Meters.

## 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.

## 6. Apparatus

6.1 *Cure meter*, consists of the following major components: specimen chamber and closure mechanism, temperature control system, rotor drive and torque measuring system (see Fig. 2 for a detailed drawing of cure meter assembly).

6.2 *Specimen Chamber*—Consists of platens, dies, and a biconical disk.

6.2.1 *Platens*—Two platens made of aluminum alloy, each containing an electric heater, and each having in the center, a cavity to accommodate a die and from the side, a well for inserting a temperature sensor.

6.2.2 *Dies*—Two which form a cavity when closed and which shall be fabricated from tool steel having a minimum Rockwell Hardness HRC 50. The geometry of the standard dies is shown in Figs. 4-6 with dimensions and tolerances (see Table 1). The top and bottom surfaces of the die cavity shall contain rectangular-shaped grooves arranged radially about the center and spaced at  $20^{\circ}$  intervals. Each die shall have a well or hole drilled from the side to accommodate a temperature sensor inserted through the platen. The upper die may be either solid or diaphragm type. The lower dies shall have a hole in the center to allow for the insertion of the disk shaft. A suitable low-friction seal shall be provided in this hole to prevent material leaking from the cavity.

6.2.2.1 *Diaphragm Upper Die*—Upper die manufactured so that the grooved die face is allowed to flex when closed on a specimen and then to maintain essentially constant pressure on the specimen as it shrinks slightly in volume during vulcanization. To provide thermal conduction to the metal body of the diaphragm die, an aluminum or stainless steel insert is placed in the diaphragm space with a hole designed to accommodate the temperature sensor. Fig. 5 describes the diaphragm type upper die.

6.2.2.2 *Solid Upper Die*—Upper die formed from one piece of steel, as described in Fig. 6.



6.2.3 *Disk*—The biconical disk shall be fabricated from tool steel having a minimum Rockwell Hardness of HRC 50. The disk shall be fitted with a stem that fits into the torque shaft. The disk is shown in Fig. 7 (see Table 2).

6.2.3.1 *Heated Disk*—Some manufacturers of oscillating disk cure meters offer a heated rotor as an option. If the disk is heated, both torque values and cure times may be significantly altered. The heated disk is a modification of the biconical disk shown in Fig. 7. This modification has provisions for directly controlling the disk temperature, as shown in Fig. 8. In this example, an electrical heater and temperature sensor are located in a metal tube, which is inserted in the disk through a vertical well in the disk shaft. The well is typically 0.325 cm (0.128 in.) in diameter and extends to within approximately 0.25 cm (0.100 in.) of the disk apex. The insertion tube is typically 0.0125 cm (0.005 in.) less than the well diameter to allow for easy tube removal for cleaning.

6.2.3.2 Disk wear will affect test results. A disk worn to such an extent that the disk diameter is less than the minimum diameter shown in this procedure shall not be used.

6.2.3.3 The standard frequency of the rotary oscillation of the disk shall be constant at 1.67 Hz (100 cpm)  $\pm 1$  %. Other frequencies may be used, if required.

6.2.3.4 A rotary drive system shall be provided for oscillatory rotation of the disk. The amplitude of oscillation of the unloaded disk shall be constant at  $\pm 1.00^{\circ}$  with a tolerance of  $\pm 0.03^{\circ}$  about the center position, that is, a total amplitude of 2°. Other amplitudes may be used, if specified.

NOTE 2—Disk and die surface contamination may contribute to slippage. Typically, torque values over 40 dNm may be subject to slipping,