Designation: D1646 − 19a

Standard Test Methods for Rubber—Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)[1]

This standard is issued under the fixed designation D1646; 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.

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

1. Scope

1.1 These test methods cover procedures for measuring a property called Mooney viscosity. Mooney viscosity is defined as the shearing torque resisting rotation of a cylindrical metal disk (or rotor) embedded in rubber within a cylindrical cavity. The dimensions of the shearing disk viscometer, test temperatures, and procedures for determining Mooney viscosity are defined in these test methods.

1.2 When disk rotation is abruptly stopped, the torque or stress on the rotor decreases at some rate depending on the rubber being tested and the temperature of the test. This is called "stress relaxation" and these test methods describe a test method for measuring this relaxation.

NOTE 1—Viscosity as used in these test methods is not a true viscosity and should be interpreted to mean Mooney viscosity, a measure of shearing torque averaged over a range of shearing rates. Stress relaxation is also a function of the test configuration and for these test methods the results are unique to the Mooney viscometer.

1.3 When compounded rubber is placed in the Mooney viscometer at a temperature at which vulcanization may occur, the vulcanization reaction produces an increase in torque. These test methods include procedures for measuring the initial rate of rubber vulcanization.

1.4 ISO 289 Parts 1 and 2 also describes the determination of Mooney viscosity and pre-vulcanization characteristics. In addition to a few insignificant differences there are major technical differences between ISO 289 and this test method in that ISO 289 does not provide for sample preparation on a mill, while this test method allows milling sample preparation in some cases prior to running a Mooney viscosity test. This can result in different viscosity values for some rubbers.

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

1.6 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1349 Practice for Rubber—Standard Conditions for Testing

D1418 Practice for Rubber and Rubber Latices—Nomenclature

D1485 Practice for Rubber from Natural Sources—Sampling and Sample Preparation

D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

D3185 Test Methods for Rubber—Evaluation of SBR (Styrene-Butadiene Rubber) Including Mixtures With Oil

D3186 Test Methods for Rubber—Evaluation of SBR (Styrene-Butadiene Rubber) Mixed With Carbon Black or Carbon Black and Oil

D3896 Practice for Rubber From Synthetic Sources—Sampling

[1] These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and are the direct responsibility of Subcommittee D11.12 on Processability Tests.

Current edition approved Nov. 1, 2019. Published November 2019. Originally approved in 1959. Last previous edition approved in 2019 as D1646 – 19. DOI: 10.1520/D1646-19A.

[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.

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

2.2 *ISO Standard:*[3]

ISO 289 Rubber, Unvulcanized—Determinations Using the Shearing Disk Viscometer,
Part 1 Determination of Mooney Viscosity, and
Part 2 Determination of Prevulcanization Characteristics.

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *Mooney viscosity, n*—measure of the viscosity of a rubber or rubber compound determined in a Mooney shearing disk viscometer; viscosity is indicated by the torque required to rotate a disk embedded in a rubber specimen and enclosed in the die cavity under specified conditions.

3.1.2 *pre-vulcanization characteristics, n*—for a vulcanizable compound, a measure of the time to the incipient vulcanization and the rate of cure during the early stages of vulcanization.

3.1.3 *stress relaxation, n*—response of a raw or compounded rubber to a rapid cessation of flow or a sudden deformation; specific to the use of the shearing disk viscometer, it takes the form of a decaying level of stress initiated by suddenly stopping the rotation of the disk.

3.1.4 *test temperature, n*—steady-state temperature of the closed dies with rotor in place and the cavity empty; this steady-state temperature shall be measured within the dies as described in 6.1.3.

4. Summary of Test Methods

4.1 These test methods are divided into three parts:

4.1.1 *Part A: Viscosity*—This test method describes the measurement of the Mooney viscosity. The Mooney viscosity is measured by a metal disk embedded in a rubber specimen contained in a rigid cylindrical cavity maintained at a specified pressure and temperature. The disk is slowly and continuously rotated in one direction for a specified time. The resistance to this rotation offered by the rubber is measured in arbitrary torque units as the Mooney viscosity of the specimen.

4.1.2 *Part B: Stress Relaxation*—This test method describes the procedure to measure stress relaxation. At the end of a Mooney viscosity test, the rotation of the metal disk is suddenly stopped and the rate of decrease of torque is monitored as a function of time.

4.1.3 *Part C: Pre-Vulcanization Characteristics*—This test method describes how pre-vulcanization properties may be measured. The viscosity of vulcanizable rubber compounds is recorded during heating at a specified temperature. The minimum viscosity and the times for the viscosity to increase by specified amounts are used as arbitrary measures of the start and rate of vulcanization.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

5. Significance and Use

5.1 *Viscosity*—Viscosity values determined by this test method depend on molecular structure, molecular weight, and non-rubber constituents that may be present. Since rubber behaves as a non-Newtonian fluid, no simple relationship exists between the molecular weight and the viscosity. Therefore, caution must be exercised in interpreting viscosity values of rubber, particularly in cases where molecular weight is very high. For example, as the molecular weight increases, the viscosity values for IIR polymers (butyl rubbers) reach an upper limit of about 80, at 100°C (212°F) using a large rotor at a rotation speed of 2 r/min, and may then decrease to considerably lower values. For these higher molecular weight rubbers, better correlation between viscosity values and molecular weight is obtained if the test temperature is increased.

5.2 *Stress Relaxation*—The stress relaxation behavior of rubber is a combination of both an elastic and a viscous response. Viscosity and stress relaxation behavior do not depend on such factors as molecular weight and non-rubber constituents in the same way. Thus both of these tests are important and complement each other. A slow rate of relaxation indicates a higher elastic component in the overall response, while a rapid rate of relaxation indicates a higher viscous component. The rate of stress relaxation has been found to correlate with rubber structure characteristics such as molecular weight distribution, chain branching, and gel content.

5.3 *Pre-Vulcanization Characteristics*—The onset of vulcanization can be detected with the Mooney viscometer as evidenced by an increase in viscosity. Therefore, this test method can be used to measure incipient cure (scorch) time and the rate of cure during very early stages of vulcanization. This test method cannot be used to study complete vulcanization because the continuous rotation of the disk will result in slippage when the specimen reaches a stiff consistency.

6. Apparatus

6.1 *Mooney Viscometer*—An instrument consisting of a motor-driven rotating disk within a cylindrical die cavity formed by two dies maintained at specified conditions of temperature and die closure force. The Mooney viscometer measures the effect of temperature and time on the viscosity of rubbers. If the stress relaxation test is to be performed, the instrument must be capable of quickly stopping the rotation of the disk and monitoring the relaxation of stress versus time. The die-rotor relationship of an example design is shown in Fig. 1. The Mooney viscometer shall incorporate the following components:

6.1.1 *Dies*—The dies and die holders forming the die cavity shall be fabricated from a nondeforming tool steel, shall have an unplated finish, and shall be hardened to a Rockwell hardness of 60 HRC minimum. The dimensions of the die cavity, measured from the highest surfaces, shall be 50.93 ± 0.13 mm (2.005 ± 0.005 in.) in diameter and 10.59 ± 0.03 mm (0.417 ± 0.001 in.) in depth. The surfaces of the die cavity shall either be serrated or contain V-grooves to minimize slippage of the specimen.

NOTE 2—The two types of dies may not give the same results.

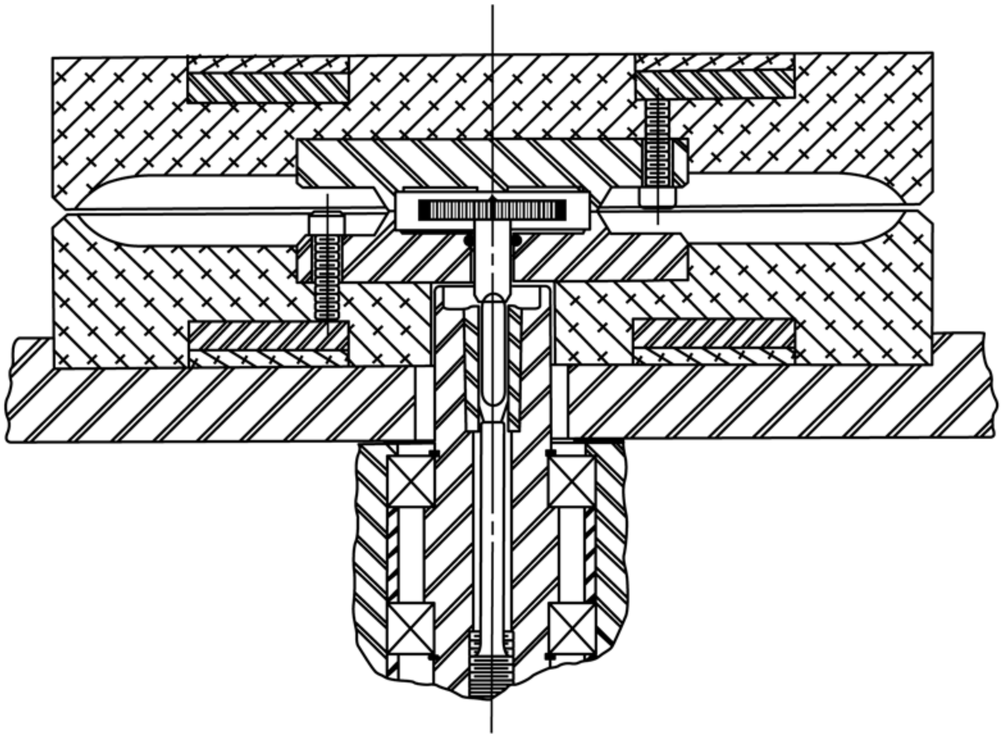

FIG. 1 Relationship of Platens, Dies, and Rotor in a Typical Shearing Disk Viscometer

6.1.1.1 *Serrated Dies*—When the cavity is formed from four pieces of steel, serrations on the surfaces of the dies and die holders are used. These serrations consist of rectangular grooves 0.8 ± 0.02 mm (0.031 ± 0.0008 in.) wide with a uniform depth of not less than 0.25 mm (0.010 in.) nor more than 0.38 mm (0.015 in.). The grooves shall be vertical and shall be cut on 1.6 ± 0.04 mm (0.063 ± 0.002 in.) centers. The serrations of the dies shall consist of two sets of such grooves at right angles to each other.

6.1.1.2 *Radial Grooved Dies*—When the die cavity is formed from two pieces of steel, radial V-grooves are used only on the flat surfaces of the die cavity. The grooves shall be spaced at 20° intervals and shall form a 90° angle in the die surfaces with the bisector of the angle perpendicular to the surface. They shall extend from the 7-mm (0.281-in.) circle to the 47-mm (1.875-in.) circle in the upper die and from the 12-mm (0.472-in.) circle to the 47-mm circle in the lower die. The grooves shall be 1 ± 0.1 mm (0.04 ± 0.004 in.) wide at the surface.

NOTE 3—Die wear can affect test results, usually to a lesser extent than rotor wear. As a general practice, many users replace dies every second time they replace worn rotors (see 6.1.2.1). This practice may not apply to all materials tested, as wear is material dependent. The ultimate way to determine if die wear has affected test results is to replace the dies with a new set and determine if the test results are changed.

6.1.1.3 *Mounting of Dies*—The dies shall be an integral part of or mounted on platens equipped with a heating device and controls capable of maintaining the die cavity at the specified test temperature with a tolerance of ±0.5°C (±1°F) at equilibrium conditions.

6.1.1.4 *Die Closure*—The viscometer shall have a suitable device for opening and closing the platens and dies and for holding them closed during a test. During a test it is extremely important that the die cavity be held closed with the correct force. To obtain the correct closing force for the mechanical-type closures, follow explicitly either the manufacturer's recommendation or other procedure of equal reliability.[4] Pneumatically closed dies shall be held closed during the test with a force of 11.5 ± 0.5 kN (2585 ± 115 lbf). A greater force may be required to close the dies when testing extremely tough stocks. At least 10 s before the motor is started, the force should be set to 11.5 ± 0.5 kN. The die closure shall be such that a piece of thin soft tissue (with a thickness not greater than 0.04 mm (0.0015 in.)) placed between the meeting surfaces will retain a continuous pattern of uniform intensity when the dies are closed upon it. A nonuniform pattern indicates wear of the die holder surface, misalignment, or distortion of dies and die holders. Any of these situations will result in undue leakage and erroneous results.

NOTE 4—For mechanical-type closure viscometers, the pressure on the die cavities may change if the viscometer is used at a different temperature than that at which it is adjusted.

6.1.2 *Rotors*—Two rotors are specified, differing only in their diameter. They shall be fabricated from a nondeforming tool steel, shall have an unplated finish and shall be hardened to a Rockwell hardness of 60 HRC minimum. The large rotor shall be 38.10 ± 0.03 mm (1.500 ± 0.001 in.) in diameter and 5.54 ± 0.03 mm (0.218 ± 0.001 in.) in thickness as measured from the highest points. The small rotor shall conform to the large rotor except the diameter shall be 30.48 ± 0.03 mm

[4] Decker, G. E., "Note on the Adjustment of the Mooney Viscometer Die Closure," *ASTM Bulletin*, No. 195, January 1954, p. 51.