Designation: D1053 − 92a (Reapproved 2018)

Standard Test Methods for Rubber Property—Stiffening at Low Temperatures: Flexible Polymers and Coated Fabrics[1]

This standard is issued under the fixed designation D1053; 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 describe the use of a torsional apparatus for measuring the relative low temperature stiffening of flexible polymeric materials and fabrics coated therewith. A routine inspection and acceptance procedure, to be used as a pass-fail test at a specified temperature, is also described.

1.2 These test methods yield comparative data to assess the low temperature performance of flexible polymers and fabrics coated therewith.

1.3 The values stated in SI units are to be regarded as 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *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]

D832 Practice for Rubber Conditioning For Low Temperature Testing

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

3. Summary of Test Method

3.1 Test Method A describes the measurement, at low temperatures, of the stiffening of flexible polymers.

3.2 Test Method B describes the measurement, at low temperatures, of the stiffening of fabrics coated with flexible polymers.

3.3 In these test methods, a specimen of flexible polymer or fabric coated with flexible polymer is secured and connected in series to a wire of known torsional constant; the other end of the wire is fastened to a torsion head to impart a twist to the wire. The specimen is immersed in a chamber filled with a heat transfer medium at a specified uniform subnormal temperature. The torsion head is then twisted 180° and in turn twists the specimen by an amount (less than 180°) that is dependent on specimen compliance or inverse stiffness. After a specified elapsed time, the amount of specimen twist is measured with a mounted protractor. The angle of twist, which is inversely related to the stiffness, is plotted versus the specified temperature. The temperature is then systematically increased in prescribed increments and the measurements repeated at each temperature, yielding a twist or inverse stiffness versus temperature profile for the test specimen. The torsional modulus of the specimen at any temperature is proportional to the quantity (180-twist)/twist.

4. Significance and Use

4.1 These test methods may be used to determine the subnormal temperature stiffening of flexible polymers or fabrics coated with flexible polymers. Temperatures at which the low temperature modulus is a specified multiple or ratio of the modulus at room temperature are interpolated from the twist versus temperature curve. These specified ratios of low-temperature modulus to room-temperature modulus are called *relative moduli*. These temperatures at the relative moduli encompass the transition region between the glassy and rubbery states of the materials tested.

4.2 These test methods offer only a general guide to stiffness characterization as service conditions of experimental materials may differ greatly from the test conditions.

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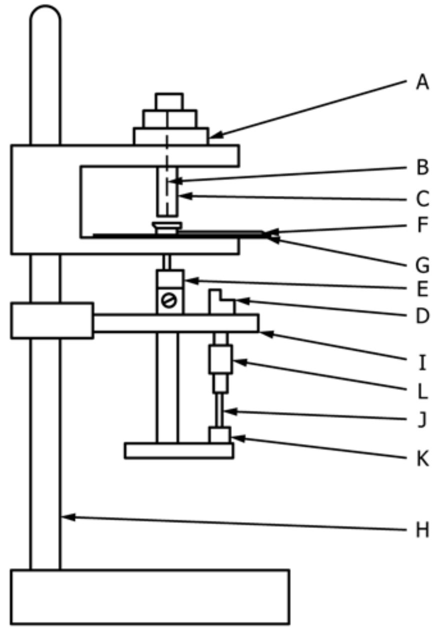
[1] These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and are the direct responsibility of Subcommittee D11.10 on Physical Testing.

Current edition approved Aug. 1, 2018. Published September 2018. Originally approved in 1943. Last previous edition approved in 2012 as D1053 – 92a (2012). DOI: 10.1520/D1053-92AR18.

[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.



A Torsion head
B Torsion wire
C Sieve
D Clamp stud
E Screw connector
F Pointer
G Movable protractor
H Supporting stand
I Specimen rack
J Test specimen
K Bottom clamp
L Top clamp

FIG. 1 Schematic Drawing of Apparatus for Low-Temperature Stiffness Test

5. Apparatus

5.1 *Torsion Apparatus*[3]—The torsion apparatus (Fig. 1) shall consist of a torsion head, *A,* capable of being turned 180 angular degrees in a plane normal to the torsion wire, *B*. The top of the wire shall be fastened to the torsion head passing through a loosely fitting sleeve, *C*. The bottom of the wire shall be fastened to the test specimen clamp stud, *D*, by means of a screw connector, *E.* A pointer, *F,* and movable protractor, *G,* shall be provided to permit convenient twist angle measurement and exact adjustment of the zero point.

5.2 *Stand*—The torsion apparatus shall be clamped to the supporting stand, *H*. It is advantageous to make the vertical portion of the stand from a poor thermal conductor.[4] The base of the stand should be of stainless steel or other corrosion-resisting material.

5.3 *Torsion Wires*— Torsion wires, made of tempered spring wire, shall be 65 ± 8 mm (2.5± 0.2 in.) long and have torsional constants (κ) of 0.0125, 0.05, and 0.2 mN·m/° of twist. The color codes for these wires are black, yellow, and white, respectively. The 0.05 mN·m/° wire (color code yellow) shall be considered standard.

5.4 *Test Specimen Rack*—A rack, *I,* made of a poor thermal conductor,[4] shall be provided for holding the test specimen, *J,* in a vertical position in the heat transfer medium (coolant). The rack shall be constructed to hold several test specimens; racks providing spaces for five or ten test specimens are commonly used. The rack shall be clamped to the stand, *H.* Two clamps, also made of a poor thermal conductor, shall be provided for holding each test specimen. The faces of these clamps shall be 6.4-mm (0.25-in.) width to facilitate proper contact with each end of the wider test specimens, that is, Type B or Type C specimens. The distance between the top and bottom clamps shall be 25 ± 2.5 mm (1.0 ± 0.1 in.) for Test Method A and 38 ± 2.5 mm (1.5 ± 0.1 in.) for Test Method B. The bottom clamp, *K,* shall be a fixed part of the test specimen rack. The top clamp, *L,* shall act as an extension of the test specimen and shall not touch the rack while the specimen is being twisted. Clearance between the top of the test specimen rack and the test specimen clamp stud is assured by inserting thin spacers between the two (Note 1). The top clamp shall be secured to a stud, *D,* which in turn shall be connected to the screw connector, *E*.

NOTE 1—Slotted TFE-fluorocarbon spacers about 1.3 mm (0.050 in.) thick and 13 mm (0.5 in.) wide have been found satisfactory. At low temperatures the test specimens stiffen in position and the spacers are removed prior to test without losing the clearance.

5.5 *Temperature Measuring Device*—A thermocouple or thermometer shall be used. Copper-constantan thermocouples, used in conjunction with a millivoltmeter or digital temperature indicator, are highly satisfactory. The thermometer, if used, shall be calibrated in 1°C divisions and shall have a range from approximately −70 to + 23°C (−95 to + 73.4°F). The thermocouple or the thermometer bulb shall be positioned as nearly equidistant from all test specimens as possible, and equidistant between the top and the bottom of the test specimens.

5.6 *Heat Transfer Media*—The heat transfer medium shall be either liquid or gaseous. Any material which remains fluid at the test temperatures and does not affect the materials being tested may be used. Among the liquids that have been found suitable for use are acetone, methyl alcohol, ethyl alcohol, butyl alcohol, silicone fluids, and normal hexane. Carbon dioxide or air are the commonly used gaseous media. Vapors of liquid nitrogen are useful for testing at very low temperatures.

NOTE 2—Specifications for materials or products requiring tests using this standard should specifically state which coolant media are acceptable for use in this test.

5.7 *Temperature Control*—Suitable means, automatic or manual, shall be provided for maintaining a uniform temperature of the heat transfer medium within ±1.0°C (1.8°F) for both liquid and gaseous media (Note 3).

5.8 *Tank or Test Chamber*—A tank for liquid heat transfer media or a test chamber for gaseous media shall be provided.

NOTE 3—Liquid medium immersion baths, low-temperature cabinets, and means for controlling temperature are described in Practice D832.

5.9 *Stirrer or Fan*—A stirrer for liquids or a fan or blower for air, which ensures thorough circulation of the heat transfer medium, shall be provided.

[3] The original apparatus was described and typical examples of the results of its use were given in a paper by Gehman, Woodford, and Wilkinson, *Industrial and Engineering Chemistry,* IECHA, Vol 39, September 1947, p. 1108.

[4] Phenolic laminate sheet has been found satisfactory for this purpose.

5.10 *Timer*—A stop watch or other timing device calibrated in seconds shall be provided.

6. Test Specimens

6.1 *Test Method A*—The test specimens shall be cut with a suitable die and shall be either Type A strips 40 ± 2.5 mm (1.5 ± 0.1 in.) long and 3.0 ± 0.2 mm (0.125 ± 0.008 in.) wide or Type B specimens of the type illustrated in Fig. 2. The standard thickness of the specimens shall be the thickness of the material undergoing test, but shall be not less than 1.5 mm (0.060 in.) nor greater than 2.8 mm (0.11 in.), and the difference between maximum and minimum thickness of each specimen shall not exceed 0.08 mm (0.003 in.). Values of thickness other than standard may be used provided it can be shown that they give equivalent results for the material being tested. When specimens taken from the finished article are not of standard thickness, it should be permissible, upon agreement between the manufacturer and the purchaser, to use a standard-size specimen, taken from a certified press-cured sheet of the same compound.

6.2 *Test Method B*—The test specimens (Type C) shall be cut with a suitable die so that the longer dimension is parallel to one of the diagonals of the fabric (on the bias). The test specimen shall be a minimum of 44 mm (1.75 in.) long and 6.3 ± 0.2 mm (0.250 ± 0.008 in.) wide. The standard thickness of the specimen shall be the thickness of the material undergoing test. The length of the test specimen shall be trimmed to fit in the specimen clamps for test.

7. Calibration of Torsion Wire

7.1 Insert one end of the torsion wire in a vertical position, in a fixed clamp, and attach the lower end of the wire at the exact longitudinal center of a circular cross-section rod of known dimension and weight. For standardization purposes, it is suggested that the rod be 200 to 250 mm (8 to 10 in.) long and about 6 mm (0.25 in.) in diameter. Initially, the rod should not be twisted through more than 90°. The rod should be allowed to oscillate freely in a horizontal plane and the time required for 20 oscillations noted in seconds. (An oscillation includes the swing from one extreme to the other and return.)

7.2 Calculate the torsional constant λ as follows:

$$\lambda = \pi^2 ml^2/3\ T^{\ 2} \quad (1)$$

where:

λ = restoring force exerted by the wire, N·m/rad of twist,
T = period of one oscillation, s,
m = mass, kg, and
l = length, m.

7.3 The torsion wires should calibrate within ±3 % of their specified torsional constants as given in 5.3.

NOTE 4—K = 17.45λ, where: K = torsional constant in mN·m/°.

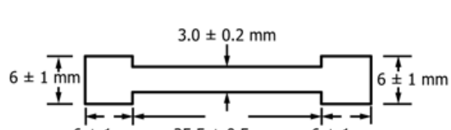


FIG. 2 Type B Specimen

8. Number of Specimens

8.1 Unless otherwise specified in the detailed specification, two specimens from each test unit shall be tested. It is good practice, however, to include a control specimen with known stiffness-temperature characteristics.

9. Mounting Test Specimens

9.1 *Test Method A*—Clamp the specimens in the testing apparatus in such a manner that 25 ± 2.5 mm (1.0 ± 0.1 in.) of each specimen is free between the clamps. For Type B specimens (see Fig. 2), make certain that the tab ends are completely within the clamps.

9.2 *Test Method B*—Clamp the specimens in the testing apparatus in such a manner that 38.0 ± 2.5 mm (1.5 ± 0.1 in.) of each specimen is free between the clamps.

10. Procedure for Stiffness Measurements in Liquid Media

10.1 Place the rack containing the test specimens in the liquid bath with a minimum of 25 mm (1 in.) of liquid covering the test specimens. Adjust the bath temperature to 23 ± 3°C (73.4 ± 5°F). Connect one of the specimens to the torsion head by means of the screw connector and the standard 0.05 mN·m/° wire. The spacer which provides clearance between the specimen rack and the specimen clamp stud need not be used for measurements made at room temperature. Adjust the pointer reading to zero by rotating the protractor scale. Turn the torsion head quickly but smoothly 180°. After 10 s as indicated by the timer, record the pointer reading. If the reading at 23°C (73.4°F) does not fall in the range from 120 to 170°, the standard torsion wire is not suitable for testing the specimen. Specimens twisting more than 170° shall be tested with a wire (black) having a torsional constant of 0.0125 mN·m/° of twist. Specimens twisting less than 120° shall be tested with a wire (white) having a torsional constant of 0.2 mN·m/° of twist.

10.2 Return the torsion head to its initial position and disconnect the specimen. Then move the test specimen rack to bring the next test specimen into position for measurement (Note 5). All test specimens in the rack shall be measured at 23 ± 3°C (73.4 ± 5°F).

NOTE 5—A modified version of the standard apparatus is now in use in which the rack is stationary while the torsion head is movable and can be positioned over the several test specimens in turn.

10.3 Insert the spacers between the specimen rack and the specimen clamp studs. Adjust the liquid bath to the lowest temperature desired (Note 6). After this temperature has remained constant within ± 1°C (± 1.8°F) for 5 min, remove one spacer and test one specimen in the same manner as was used at room temperature. Return the spacer to its original position after the specimen has been tested (Note 7).

NOTE 6—This varies with the type of material being tested since time is saved by not starting at a temperature more than 10°C (18°F) lower than the freezing point of the material. For natural rubber, the lowest temperature required is usually – 80°C (–112°F); for styrene butadiene rubber, the lowest temperature is usually – 70°C (–94°F).

NOTE 7—Movement of the spacer often tends to alter the pointer position with respect to the protractor; therefore, the pointer should be adjusted to zero after the spacer has been removed.