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

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.

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

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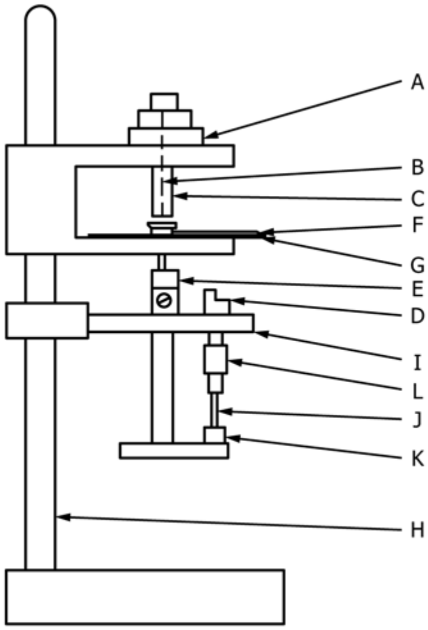
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



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

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.

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.

[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.

5.9 *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.

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:

$$\lambda = \pi^2 ml^2/3\,T^2 \tag{1}$$

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.

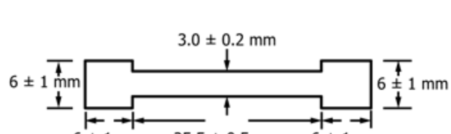


FIG. 2 Type B Specimen

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/°.

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.

10.4 After all specimens have been tested at the lowest temperature desired, increase the bath temperature by 5°C (9°F) intervals and make stiffness measurements after conditioning the specimens for 5 min at each temperature. Continue testing until a temperature is reached at which the angular twist is within 5 to 10° of the original twist at 23 ± 3°C (73.4 ± 5°F).

10.5 Increments of 10°C (18°F) instead of 5°C (9°F) may be used, if desired, for the less sensitive parts of the temperature range. The temperature rise may be accelerated by use of an electrical immersion heater. The test may be shortened by concluding the temperature rise as soon as the range of interest has been passed, as described in 13.3.

10.6 Vulcanizates of certain polymers such as dimethyl vinyl silicone and *cis*-1,4-polybutadiene are known to crystallize rapidly (over specific temperature ranges) under conditions of this test. This should be recognized in interpreting the results (see Practice D832).

11. Procedure for Stiffness Measurements in Gaseous Media (Long-Term Tests)

11.1 For long-term tests at a given temperature, the apparatus shall be used in a suitable low-temperature cabinet or cold room. Additional specimen racks are required. Mount the test specimens in racks and measure and record the pointer deflection at 23°C (73.4°F) for each specimen. Then store the racks in a low-temperature cabinet or cold room whose temperature is regulated at the desired value and measure the deflections periodically. Relevant material specifications should state the conditioning period, which should never be less than the time required for the specimens to reach thermal equilibrium with the surrounding gaseous medium. (See Fig. 3.)

12. Routine Inspection and Acceptance

12.1 For routine inspection of materials the stiffness test shall be conducted as described in Section 10 with the exceptions that only the standard wire shall be used and that the test shall be conducted at only one temperature. The test temperature, exposure time, and type of coolant shall be as stated in the relevant material specification. Unless otherwise stated in the material specification, the minimum number of angular degrees of twist exhibited by the specimens, when tested at the specified temperature, shall be as shown in Table 1.

12.2 Interpolation shall be used for those thicknesses not contained within Table 1. The angular twists shown in the table are calculated for a Young's modulus value of 69 MPa (10 000 psi) for a specimen 25 mm (1.0 in.) long (span) and 3.2 mm (0.125 in.) wide.

NOTE 8—*Example*—A specimen 2.0 mm (0.080 in.) thick, which has an angular twist of 66° or more when tested at – 55± 0.5°C (–67 ± 1°F), has a Young's modulus no greater than 69 MPa (10 000 psi) at this temperature.

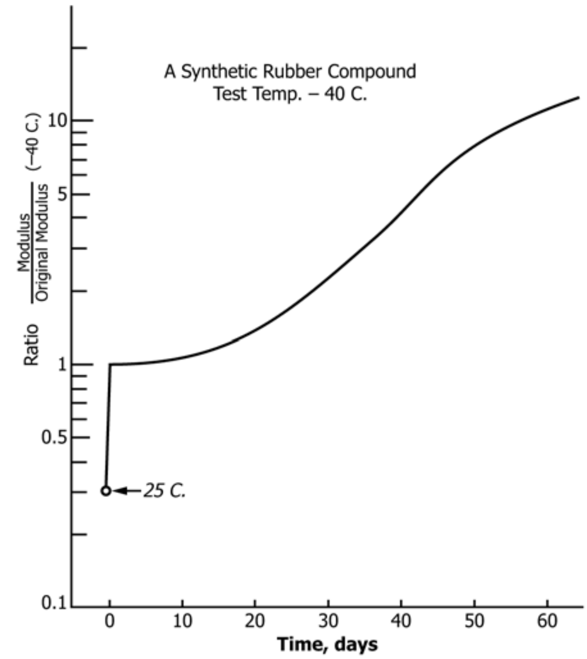


FIG. 3 Illustrative Chart for Long-Time Test

TABLE 1 Relationship Between Specimen Thickness and Angular Twist

Thickness, mm (in.)	Twist, min angular°
1.5 (0.060)	98
1.8 (0.070)	80
2.0 (0.080)	66
2.3 (0.090)	55
2.5 (0.100)	46
2.8 (0.110)	40

13. Calculation

13.1 *Twist Versus Temperature Curve*—A plot shall be made of the pointer reading (angle of twist of the test specimen) versus the temperature, as illustrated in Fig. 4. This plot can be

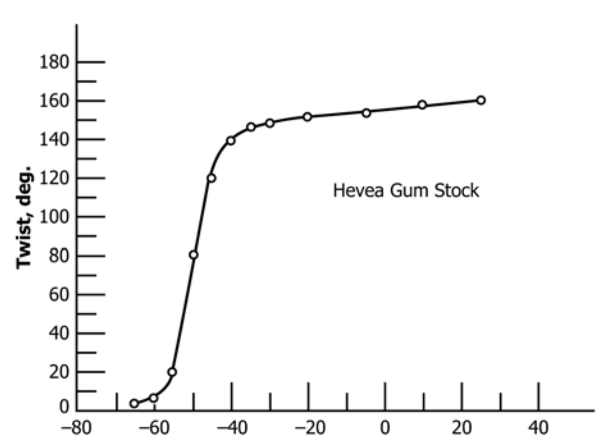


FIG. 4 Illustrative Chart of Twist Versus Temperature

used for determining the temperatures corresponding to specific relative moduli as described later.

13.2 *Modulus Proportionality Factor*—The modulus proportionality factor (MPF) of the specimen is equal to the quantity (180°-twist)/twist. The angle of twist of the test specimen at a specific test temperature is measured in degrees. Table 2 lists the value of modulus proportionality factors for every angular degree from 1 to 180.

13.3 *Relative Modulus*— The relative modulus, or torsional stiffness ratio at a specified test temperature, is the ratio of the modulus proportionality factor at the temperature to the modulus proportionality factor at 23°C (73.4°F). For example:

$$\text{Twist at } 23°\text{C} = 160° \qquad \text{Twist at } -40°\text{C} = 100°$$
$$\text{MPF} = (180–160)/160 = 0.125 \qquad \text{MPF} = (180–100)/100 = 0.800$$
$$\text{Relative Modulus or Torsional Stiffness Ratio} = 0.800/0.125 = 6.4$$

TABLE 2 Modulus Proportionality Factors

Twist, *X*,°	(180 – *X*)/*X*	Twist, *X*,°	(180 – *X*)/X	Twist, *X*,°	(180 – *X*)/X	Twist, *X*,°	(180 – *X*)/X
1	179	51	2.53	101	0.782	151	0.192
2	89	52	2.46	102	0.765	152	0.184
3	59	53	2.40	103	0.748	153	0.176
4	44	54	2.33	104	0.731	154	0.169
5	35	55	2.27	105	0.714	155	0.161
6	29	56	2.21	106	0.698	156	0.154
7	24.7	57	2.16	107	0.682	157	0.146
8	21.5	58	2.10	108	0.667	158	0.141
9	19.0	59	2.05	109	0.651	159	0.132
10	17.0	60	2.00	110	0.636	160	0.125
11	15.4	61	1.95	111	0.622	161	0.1180
12	14.0	62	1.90	112	0.607	162	0.1111
13	12.8	63	1.86	113	0.593	163	0.1043
14	11.86	64	1.81	114	0.579	164	0.0975
15	11.00	65	1.77	115	0.565	165	0.0909
16	10.25	66	1.73	116	0.552	166	0.0843
17	9.59	67	1.69	117	0.538	167	0.0778
18	9.00	68	1.65	118	0.525	168	0.0714
19	8.47	69	1.61	119	0.513	169	0.0651
20	8.00	70	1.571	120	0.500	170	0.0588
21	7.57	71	1.535	121	0.488	171	0.0527
22	7.1	72	1.500	122	0.475	172	0.0465
23	6.83	73	1.466	123	0.463	173	0.0405
24	6.50	74	1.432	124	0.452	174	0.0345
25	6.20	75	1.400	125	0.440	175	0.0286
26	5.92	76	1.368	126	0.429	176	0.0227
27	5.67	77	1.337	127	0.417	177	0.0169
28	5.43	78	1.308	128	0.406	178	0.0112
29	5.21	79	1.278	129	0.395	179	0.0056
30	5.00	80	1.250	130	0.385	180	0
31	4.81	81	1.222	131	0.374		
32	4.62	82	1.195	132	0.364		
33	4.45	83	1.169	133	0.353		
34	4.29	84	1.143	134	0.343		
35	4.14	85	1.118	135	0.333		
36	4.00	86	1.093	136	0.324		
37	3.86	87	1.069	137	0.314		
38	3.74	88	1.045	138	0.304		
39	3.62	89	1.022	139	0.295		
40	3.50	90	1.000	140	0.286		
41	3.39	91	0.978	141	0.277		
42	3.29	92	0.956	142	0.267		
43	3.19	93	0.935	143	0.258		
44	3.09	94	0.915	144	0.250		
45	3.00	95	0.895	145	0.241		
46	2.91	96	0.875	146	0.233		
47	2.83	97	0.856	147	0.224		
48	2.75	98	0.837	148	0.216		
49	2.67	99	0.818	149	0.208		
50	2.60	100	0.800	150	0.200		

13.4 *Temperature for Values of Relative Modulus*—To determine the temperature at which the relative modulus is 2, 5, 10, and 100, Table 3 shall be used in conjunction with the twist versus temperature curve for the specimen. The first column of Table 3 lists each degree in the range from 120 to 170, so that the value corresponding to the twist of the specimen at 23°C (73.4°F) can be selected. Successive columns give the twist angles which correspond to values of 2, 5, 10, and 100 for the relative modulus. The temperatures corresponding to these angles are then read from the twist versus temperature curve for the specimen and are designated at T_2 , T_5, T_{10}, and T_{100}, respectively. Table 3 can be used during a test to determine when a particular T value has been obtained so that the test may then be concluded.

NOTE 9—*Example*—The twist versus temperature curve for a hevea

TABLE 3 Twist Angles for Designated Values of the Relative Modulus

Twist at 23°C,°	Twist for RM = 2,°	Twist for RM = 5,°	Twist for RM = 10,°	Twist for RM = 100,°
120	90	51	30	3
121	91	52	31	4
122	92	53	31	4
123	93	54	32	4
124	95	55	33	4
125	96	56	33	4
126	97	57	34	4
127	98	58	35	4
128	99	59	36	4
129	101	61	36	5
130	102	62	37	5
131	103	63	38	5
132	104	64	39	5
133	105	65	40	5
134	107	66	41	5
135	108	68	42	5
136	109	69	42	5
137	111	70	43	6
138	112	71	45	6
139	113	72	46	6
140	114	74	47	6
141	116	75	48	6
142	117	77	49	7
143	119	78	50	7
144	120	80	51	7
145	121	82	53	7
146	123	83	54	7
147	124	85	55	7
148	126	87	57	8
149	127	88	58	8
150	129	90	60	9
151	130	92	62	9
152	132	94	62	9
153	133	96	65	10
154	134	97	67	10
155	136	100	69	11
156	138	102	71	11
157	139	104	73	12
158	140	106	75	12
159	142	108	78	13
160	144	111	80	13
161	146	113	82	14
162	147	116	85	15
163	149	118	88	16
164	151	121	91	17
165	152	124	94	18
166	154	126	98	19
167	156	130	101	20
168	158	133	105	22
169	159	136	109	24
170	161	139	113	26

gum compound is given in Fig. 4. From this curve the twist at 23°C (73.4°F) is found to be 160°. Referring to Table 3, the angles of twist corresponding to relative modulus values of 2, 5, 10, and 100 are, respectively, 144, 111, 80, and 13. Referring again to the curve in Fig. 4, the temperatures at which these angles of twist occur are found to be − 38°C, − 47°C, − 50°C, and − 56°C (−39°F, − 44°F, − 46°F, and − 49°F), respectively.

13.5 *Apparent Modulus of Rigidity*—Annex A1 describes the procedure for determining the apparent modulus of rigidity or torsional modulus in megapascals, and Young's Modulus, using the angular twist values determined at the test temperature, test specimen cross-sectional area measurements, and supplemental tabular information.

NOTE 10—When the computed value for apparent modulus of rigidity exceeds 69 MPa (10 000 psi), the rubber is generally considered to be too stiff to be serviceable at the specified temperature.

14. Report

14.1 Report the following information:

14.1.1 Complete identification of the material tested including type, source, manufacturer's code designation, form, date made, etc.,

14.1.2 Thickness and type of specimen,

14.1.3 Details of conditioning of specimens prior to test,

14.1.4 Torsional constant of torsion wire used,

14.1.5 Type of heat transfer medium used,

14.1.6 Exposure time,

14.1.7 Temperatures, in degrees Celsius, at which the relative modulus is 2, 5, 10, and 100. These temperatures shall be designated, respectively, as T_2, T_5, T_{10}, and T_{100}, and

14.1.8 When requested or specified, the torsional modulus or torsional stiffness ratio at a specified test temperature.

14.2 *Room-Temperature Rigidity Modulus*—The report shall also include the room-temperature rigidity modulus as calculated in the Annex. This is used as a basis for judging the actual stiffness attained at T_2, T_5, T_{10}, T_{100}.

14.3 *Long-Time Tests*— For long-time tests, the results shall be presented as plots of the ratio of modulus to original modulus at the test temperature *versus* time, the modulus ratio being plotted on a logarithmic scale, as illustrated in Fig. 3.

14.3.1 When required by control specifications or as agreed upon between the producer and the user, the results of long-time tests may be reported as the relative modulus or torsional stiffness ratio as determined according to 13.2.

14.4 *Routine Inspection and Acceptance*—For routine inspection of materials, the results shall include the test temperature, the average specimen thickness, and the average value for the twist, in angular degrees, obtained at the test temperature.

15. Precision and Bias[5]

15.1 This precision and bias section has been prepared in accordance with Practice D4483. Refer to Practice D4483 for terminology and other statistical calculation details.

15.2 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials (rubbers) used in the particular interlaboratory program as described as follows. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include this test method.

15.3 A Type 1 (interlaboratory) precision was evaluated. Both repeatability and reproducibility are short term; a period of a few days separates replicate test results. A test result is the *average value*, as specified by this test method, obtained on two determination(s) or measurement(s).

15.4 For Test Method A, four different materials were used in the interlaboratory program, these were tested in four laboratories on two different days. The results of the precision calculations for repeatability and reproducibility are given in Table 4, in ascending order of material average or level, for each of the materials evaluated.

15.5 With the approximation to 0°C of T_2 measurements of Materials 1-A and 1-B, all temperatures at the relative moduli have been transformed to the kelvin scale to avoid excessively large (r) and (R) values.

15.6 The precision of this test method may be expressed in the format of the following statements which use an *appropriate value* of r, R, (r), or (R), to be used in decisions about test results. The appropriate value is that value of r or R associated with a mean level in Table 4 closest to the mean level under consideration at any given time, for any given material, in routine testing operations.

15.7 *Repeatability*— The repeatability, r, of this test method has been established as the appropriate value tabulated in Table 4. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated r (for any given level) must be considered as derived from different or nonidentical sample populations.

15.8 *Reproducibility*— The reproducibility, R, of this test method has been established as the appropriate value tabulated in Table 4. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated R (for any given level) must be considered to have come from different or nonidentical sample populations.

15.9 Repeatability and reproducibility expressed as a percent of the mean level, (r) and (R), have equivalent application statements as above for r and R. For the (r) and (R) statements, the difference in the two single test results is expressed as a percent of the arithmetic mean of the two test results.

15.10 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for this test method since the value (of the test property) is exclusively defined by the test method. Bias, therefore, cannot be determined.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1036.

TABLE 4 Type 1 Precision for Test Method A—Amount of Twist at 23°C[A]

Material	Average Level (°)	Within Laboratories S_r	r	(r)	Between Laboratories S_R	R	(R)
1-A	152.3	0.71	2.01	1.3	2.22	6.28	4.1
1-B	155.4	0.64	1.81	1.2	1.20	3.40	2.2
3	164.0	1.02	2.89	1.8	1.17	3.31	2.0
2	168.7	1.02	2.89	1.7	1.49	4.22	2.5
4	169.1	0.75	2.12	1.3	0.75	2.12	1.3
Pooled Values	161.9	0.90	2.55	1.6	1.57	4.44	2.7

T_2, K

Material	Average Level (°)	Within Laboratories S_r	r	(r)	Between Laboratories S_R	R	(R)
4	223.2	0.81	2.31	1.0	2.13	6.02	2.7
3	230.3	0.98	2.76	1.2	0.98	2.76	1.2
2	248.9	2.30	6.51	2.6	4.58	12.96	5.2
1-B	272.6	0.14	0.41	0.2	2.40	6.80	2.5
1-A	273.5	0.55	1.56	0.7	2.54	7.19	2.6
Pooled Values	250.7	1.23	3.49	1.4	2.59	7.33	2.9

T_5, K

Material	Average Level (°)	S_r	r	(r)	S_R	R	(R)
4	218.7	0.45	1.27	0.6	2.80	7.93	3.6
3	223.3	0.22	0.62	0.3	0.48	1.37	0.6
2	240.8	0.63	1.78	0.7	0.63	1.78	0.7
1-A	267.0	0.40	1.14	0.4	1.90	5.39	2.0
1-B	267.5	0.29	0.81	0.3	0.35	0.99	0.4
Pooled Values	243.4	0.43	1.22	0.5	1.80	5.08	2.1

T_{10}, K

Material	Average Level (°)	Within Laboratories S_r	r	(r)	Between Laboratories S_R	R	(R)
4	217.1	0.35	0.98	0.5	2.65	7.49	3.5
3	219.5	0.52	1.46	0.7	2.60	7.36	3.4
2	237.8	0.33	0.93	0.4	0.33	0.93	0.4
1-A	265.1	0.40	1.12	0.4	2.39	6.77	2.6
1-B	265.9	0.27	0.78	0.3	0.27	0.78	0.3
Pooled Values	239.9	0.38	1.08	0.4	2.03	5.74	2.4

T_{100}, K

Material	Average Level (°)	Within Laboratories S_r	r	(r)	Between Laboratories S_R	R	(R)
4	212.0	0.78	2.22	1.0	2.53	7.17	3.4
3	213.5	0.37	1.04	0.5	2.45	6.94	3.3
2	230.4	0.27	0.75	0.3	2.15	6.09	2.6
1-B	259.5	0.59	1.68	0.6	1.80	5.09	2.0
1-A	259.8	0.41	1.16	0.4	2.28	6.44	2.5
Pooled Values	235.0	0.52	1.47	0.6	2.26	6.39	2.7

[A] S_r = repeatability standard deviation.
r = repeatability = 2.83 times the square root of the repeatability variance.
(r) = repeatability (as percent of material average).
S_R = reproducibility standard deviation.
R = reproducibility = 2.83 times the square root of the reproducibility variance.
(R) = reproducibility (as percent of material average).

16. Keywords

16.1 apparent modulus of rigidity; coated fabric; fabrics; flexible polymers; low temperature; low temperature modulus; low temperature test; modulus proportionality factor; MPF; polymer; relative modulus; rigidity modulus; stiffening ; stiffness; stiffness measurement in gaseous media; stiffness measurement in liquid media; subnormal temperature; torsion; twist versus temperature

ANNEX

(Mandatory Information)

A1. APPARENT MODULUS OF RIGIDITY

A1.1 *Apparent Modulus of Rigidity*—When it is desired to calculate the apparent modulus of rigidity or torsional modulus, the free length of the test specimen must be accurately measured and the following equation used (Note A1.1):

$$G = \frac{916K\,L(180 - X)}{a\,b^3\,\mu\,X} \tag{A1.1}$$

where:

G = apparent modulus of rigidity, MPa,
K = torsional constant of wire, mN·m/°,
L = measured free length (span) of the test specimen, mm,
a = width of test specimen, mm,
b = thickness of test specimen, mm,
μ = factor based on ratio of *a/b* taken from Table A1.1, and
X = angle of twist of test specimen,

To obtain Young's modulus, multiply the modulus of rigidity, G, by 3.

NOTE A1.1—There have been recent attempts to verify this equation without total success. Thus, it should be used with that knowledge.

TABLE A1.1 Values of Factor µ for Various Ratios of *a/b*

a/b	µ	*a/b*	µ
1.00	2.249	2.25	3.842
1.05	2.359	2.50	3.990
1.10	2.464	2.75	4.111
1.15	2.563	3.00	4.213
1.20	2.658	3.50	4.373
1.25	2.748	4.00	4.493
1.30	2.833	4.50	4.586
1.35	2.914	5.00	4.662
1.40	2.990	6.00	4.773
1.45	3.063	7.00	4.853
1.50	3.132	8.00	4.913
1.60	3.260	9.00	4.960
1.70	3.375	10.00	4.997
1.75	3.428	20.00	5.165
1.80	3.479	50.00	5.226
1.90	3.573	100.00	5.300
2.00	3.659		