CHANGE NOTICES ARE NOT CUMULATIVE AND SHALL BE RETAINED UNTIL SUCH TIME AS THE ENTIRE STANDARD IS REVISED.

RETAIN CHANGE NOTICES 1 AND 2

FED. TEST METHOD STD. NO. 601
April 12, 1955
Change Notice 3
JANUARY 30, 1962

FEDERAL TEST METHOD STANDARD

RUBBER: SAMPLING AND TESTING

The following changes in Federal Test Method Standard No 601 dated April 12, 1955, have been approved by the Commissioner, Federal Supply Service, General Services Administration, for the use of all Federal agencies

1. Revision.—The following method of this standard has been revised and supersedes the method listed:

Revision	*Superseded*
Method 5611.1	Method 5611
January 30, 1962	April 12, 1955

2. New method.—The following new method is a part of this standard:

5612—Stiffness, Torsional, Low Temperature, Gaseous Medium.

RETAIN THIS COVER PAGE AND INSERT BEFORE THE TABLE OF CONTENTS OF THIS STANDARD.

STIFFNESS, TORSIONAL, LOW TEMPERATURE, LIQUID MEDIUM

1. SCOPE

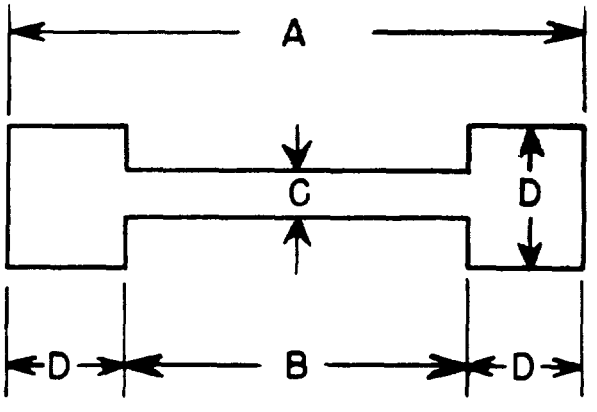
1.1 This method is intended for use in determining the stiffness of soft vulcanized rubber compounds in a liquid medium at low temperature

2. SPECIMEN

2.1 **Straight specimen.** — The specimen shall be a portion of the test unit between 35 and 40 mm in length The width shall be between 3 0 and 3 3 mm , method 2111

2.1.1 The thickness of the specimen shall be the thickness of the material undergoing test but not more than 3 mm. Thicker materials shall be reduced by cutting or buffing to a thickness between 1 5 and 3 mm. The specimen shall be uniform in thickness within 0 1 mm., method 2011.

2.2 **I-shaped specimen.** — The specimen shall be cut from the test unit with a die having the shape and dimensions given in figure 5611A. The thickness of the specimen shall be as described in 2.1.1.



DIMENSION	*MINIMUM*	*MAXIMUM*
	mm.	mm.
A	35	40
B	25	26
C	3	3.3
D	5	7

FIGURE 5611A.—*I-shaped specimen.*

3. APPARATUS

3.1 The apparatus shall be as follows:

3.1.1 *Torsion apparatus.*—The apparatus consists of a stand, specimen rack, torsion wire, and torsion head. A suitable apparatus is shown in figure 5611B.

3.1.1.1 *Stand.*—The stand, H, supports the specimen rack, I, and torsion head, A. The vertical portion of the stand shall be made of a thermal insulating material such as reinforced phenolic plastic. The base of the stand should be made of a corrosion resisting material

3.1.1.3 *Specimen rack.*—A rack, I, made of a poor thermal conductor such as reinforced phenolic plastic is provided for holding the specimen, J, in a vertical position in the heat transfer medium. The rack is usually constructed to provide space for 1 to 10 specimens. The rack is clamped to the stand, H. Two clamps are provided for holding each test specimen. The bottom clamp, K, is a fixed part of the specimen rack and the top clamp, L, acts as an extension of the specimen but must not touch the rack while the specimen is being twisted. Clearance between the top of the specimen rack and the specimen clamp stud is assured by inserting thin spacers between the two. Slotted polytetrafluoroethylene spacers about 1 mm. thick and 10 to 15 mm. wide have been found satisfactory. At low temperatures, the specimen stiffens in position and the spacers are removed prior to test without losing the clearance. The top clamp is fastened to a stud, D, which is connected to the screw connector, E.

3.1.1.3 *Torsion wire.*—One beryllium-copper wire between 60 and 68 mm. in length shall be provided. The wire shall have a torsional constant of 0.500 gram-centimer per degree of twist and shall be color coded

yellow. The wire shall be calibrated as described in 4.1 and shall be within 0.500 ± 0.015 gram-centimeter.

3.1.1.4 *Torsion head.*—The head, A, is capable of being turned 180 angular degrees in a plane normal to the torsion wire One end of a torsion wire, B, is fastened to the head through a loosely fitting sleeve, C The other end of the wire is fastened to the specimen clamp stud, D, by means of a screw connector, E. A pointer, F, and movable protractor, G, permit adjustment of the zero point.

3.1.2 *Low temperature chamber.* — The chamber consists of a thermally insulated tank containing a suitable liquid and provided with a stirrer and devices for measuring and controlling the temperature within ± 1 0° C (1.8° F.) of the specified temperature.

3.1.2.1 The dimensions and location of the tank shall be such that the specimens are immersed completely and the liquid medium covers the top of the specimen to a depth of at least 12 mm during the test.

3.1.2.2 The tank is equipped with a stirrer that insures uniformity of temperature within 0.3° C. (0.5° F.) throughout the liquid medium.

3.1.2.3 The liquid medium may be any material which is liquid at the temperature of test and does not affect the specimen Liquids that have been found satisfactory are acetone, methanol, ethanol, butanol, silicone fluids, and a solution of methyl phosphate and water in the ratio 87 to 13 by volume.

3.1.2.4 The temperature shall be measured by means of a device, such as a thermocouple, resistance thermometer, or liquid-in-glass thermometer, that is sensitive to changes in temperature of 0.2° C (0 4° F.) or less. The sensing element shall be located as nearly equidistant from the specimens as practicable, midway between the top and bottom of the specimen The temperature throughout the heat transfer medium shall be controlled automatically during the exposure period within ± 1.0° C (1 8° F.) A satisfactory control is described under apparatus in method 5311

3.1.3 *Timer.*—A stop watch or other timing device shall be provided to indicate the time of exposure in seconds.

4. PROCEDURE

4.1 **Calibration of the torsion wire.**—One end of the wire shall be inserted in a fixed clamp in a vertical position The lower end of the wire shall be attached at the exact longitudinal center of a rod of known dimensions and weight. For standardization purposes it is suggested that the rod be 20 to 30 cm long and 6 to 7 mm in diameter Initially the rod shall not be twisted through more than 90 degrees The rod shall be allowed to oscillate freely in a horizontal plane and the time required for 20 oscillations recorded in seconds An oscillation includes the swing from one extreme to the other and return The mass moment of inertia shall be calculated as follows:

$$I = \frac{ML^2}{12}$$

Where:

I is the moment of inertia, gm-cm.^{2}
M is the mass of the rod, gm
L is the length of the rod, cm.

The torsional constant shall be calculated as follows:
Where:

$$\lambda = \frac{4\pi^2 I}{T^2}$$

T is the period of one oscillation, seconds.
I is the moment of inertia of the rod, gm.-cm.^{2}.
λ is the restoring force exerted by the wire, dyne-cm. per radian of twist.

The dyne-centimeter per radian of twist, λ, shall be converted to gram-centimeter per degree as follows

$$\text{Torsional constant, K,} = \frac{\lambda \times 3.1416}{981 \times 180}$$

4.2 If the material is too thick or has a fabric backing or an uneven surface that may interfere with the test, the material shall be buffed or cut as described in method 1111. The average thickness of the specimen shall be determined as described in method 2011, to the nearest 0.02 mm or 0.001 inch.

4.3 The temperature of exposure shall be as specified in the detail specification.

4.4 The heat transfer medium shall be as specified in the detail specification.

4.5 The time of exposure before determining the stiffness of the specimen shall be 5.0 ± 0.5 minutes.

4.6 The specimen shall be clamped in the specimen rack in such a manner that the central 25 to 26 mm portion is free between the clamps. If the modulus of rigidity is required, the free length between clamps is measured to the nearest 0.5 mm.

4.7 The rack containing the specimen shall be placed in the liquid heat transfer medium so that the top of the specimen is immersed to a depth of at least 12 mm. If the rack contains more than one specimen, the spacer shall be inserted between the rack and each specimen clamp stud. The liquid transfer medium shall be adjusted to the specified temperature and maintained constant within 1.0° C. (1.8° F.) for 5 minutes before measurements are made.

4.8 If the rack contains more than one specimen, the specimen to be tested shall be connected to the torsion head by means of the screw connector and the torsion wire, and the spacer shall be removed. The pointer shall be adjusted to zero by rotating the protractor scale. The torsion head shall be turned quickly and smoothly 180 degrees and after 10 seconds as indicated by the timer, the pointer reading shall be recorded as the angular twist of the specimen.

4.9 The torsion head shall be returned to its original position and the specimen disconnected. The specimen rack or the torsion head shall be moved to bring the next specimen into position for measurement.

5. RESULTS

5.1 Unless otherwise specified in the detail specification, three specimens from each test unit shall be tested. It is considered good practice to include a control specimen with known twist-temperature characteristics.

5.2 The temperature of test shall be recorded.

5.3 The value for angular twist of each specimen tested shall be recorded to the nearest angular degree.

5.4 The average thickness of each specimen tested shall be recorded to the nearest 0.02 mm. or 0.001 inch.

5.5 The values obtained shall be compared with the minimum angular twist permitted for the thickness of the material undergoing test, shown in table I.

TABLE I.—*Relationship between specimen thickness and angular twist.*

Thickness	Minimum twist	Thickness	Minimum twist
mm.	*degrees*	*inch*	*degrees*
1.50	100	0.06	98
1.75	82	.07	80
2.00	68	.08	66
2.25	56	.09	55
2.50	48	.10	46
2.75	41	.11	40
3.00	35	.12	34

Interpolation should be used for those thicknesses not within table I. The angular twists shown in the table are calculated for a Young's modulus of 700 kg./cm.^{2} (10,000 lb./in.^{2}) for a specimen having a free span of 25 mm. (1 inch) and 3.15 mm. (⅛ inch)

wide. For example, a specimen 2 mm. thick which has an angular twist of 68 degrees or more when tested at $-55° \pm 0.5°$ C. ($-67° \pm 1°$ F.) has a modulus no greater than 700 kg./cm^2 at this temperature.

5.6 If specified, the modulus of rigidity, G, shall be calculated as follows:

$$G = \frac{AK\,(180° - x)\,l}{ab^3 \mu x}$$

Where:

a is the width of the specimen.

b is the thickness of the specimen.

l is the free length of specimen between clamps.

x is the angular degrees of twist of specimen.

μ is the factor based on ratio of a/b, table II.

K is the torsional constant of wire in gram-centimeters per degree.

A is a proportionality factor depending upon the units of measurement as follows:

A	G	a,b,l
916	kg/cm^2	millimeter
0.056	kg/cm^2	inch
13030	lb/in.^{2}	millimeter
0.795	lb/in.^{2}	inch

TABLE II.—*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		

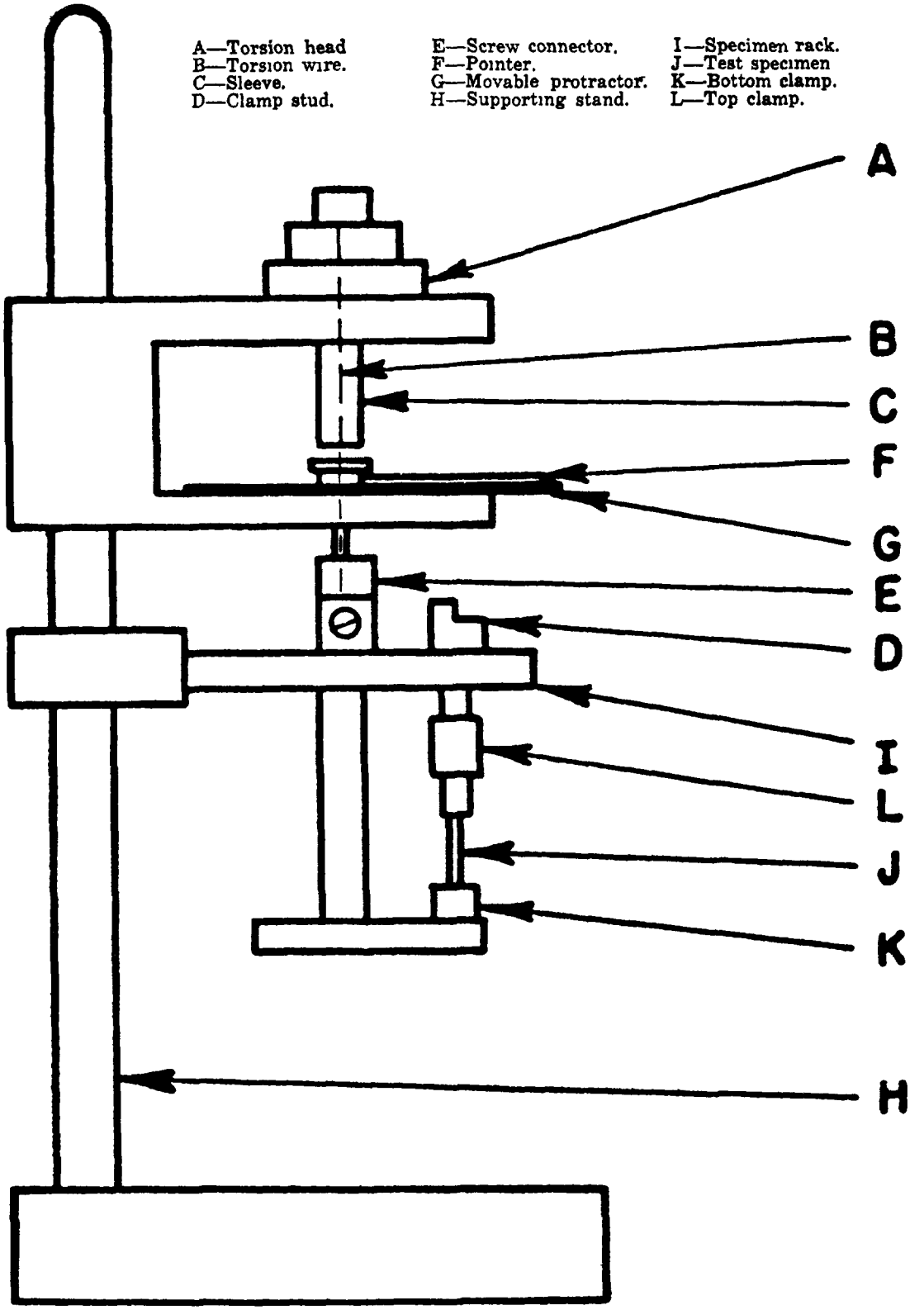


FIGURE 5611B.—*Schematic drawing of apparatus for low temperature stiffness test.*

STIFFNESS, TORSIONAL, LOW TEMPERATURE, GASEOUS MEDIUM

1. SCOPE

1.1 This method is intended for use in determining the change in stiffness of soft vulcanized rubber compounds at low temperature in a gaseous medium The ratio of the stiffness at a low temperature to the stiffness of the specimen at standard temperature is calculated

2. SPECIMEN

2.1 The specimen shall be as described in method 5611

3. APPARATUS

3.1 The apparatus shall be as follows:

3.1.1 *Torsion apparatus* —The apparatus shall be as described in method 5611 with the following exception

3.1.1.1 *Torsion wire* — Three beryllium-copper wires between 60 and 68 mm in length shall be provided The wires shall have torsional constants of 0 125, 0 500, and 2 000 gram-centimeters per degree of twist and shall be color coded, black, yellow, and white, respectively The wires shall be calibrated as described in 4.1 of 5611 and shall be within 3 percent of the specified torsional constant.

3.1.2 *Low temperature chamber.* — The chamber consists of a thermally insulated cabinet equipped with a fan or blower for circulating the gas and with devices for measuring and controlling the temperature within ± 1.0° C. (1.8° F.) of the specified temperature.

3.1.2.1 The size of the cabinet shall be such so as to house the entire torsion apparatus (3 1.1).

3.1.2.2 The gaseous medium may be air, carbon dioxide, nitrogen, or mixtures of these gases.

3.1.2.2 The temperature measuring device shall be as described in method 5611.

3.1.3 *Timer* —A stopwatch or other timing device shall be provided to indicate the time of exposure in seconds.

4. PROCEDURE

4.1 The torsion wire shall be calibrated as described in method 5611.

4.2 If the material is too thick or has a fabric backing or an uneven surface that may interfere with the test, the material shall be buffed or cut as described in method 1111. If the modulus of rigidity is desired, the width and thickness of the specimen is measured to the nearest 0 02 mm.

4.3 The temperature of exposure shall be as specified in the detail specification.

4.4 The heat transfer medium shall be as specified in the detail specification.

4.5 Unless otherwise specified in the detail specification, the time of exposure at the low temperature shall be 3 hours ± 1 hour or 166 hours ± 1 hour

4.6 The specimen shall be clamped in the specimen rack in such a manner that the central 25- to 26-mm. portion is free between the clamps If the modulus of rigidity is required, the free length between the clamps is measured to the nearest 0.5 mm.

4.7 The specimen and torsion apparatus shall be brought to standard temperature, 23° ± 1.1° C. (73.4° ± 2° F.).

4.8 The specimen to be tested shall be connected to the torsion head through the connector and the required torsion wire. The pointer shall be adjusted to zero by rotating the protractor scale. The torsion head shall be turned 180 degrees in one second or less

and the pointer reading recorded after 10 seconds as indicated by the timer. The torsion head shall be returned to its original position and the specimen disconnected. If the rack contains more than one specimen, the rack or the torsion head shall be moved to bring the next specimen into position for testing. The pointer deflection shall be measured and recorded for each specimen.

4.8.1 If the reading at standard temperature does not fall in the range of 120 to 170 degrees, wire having a torsional constant of 0.500 gram-centimeter is not suitable for testing the specimen. Specimens twisting more than 170 degrees should be tested with wire having a torsional constant of 0 125 gram-centimeter. Specimens twisting less than 120 degrees should be tested with wire having a torsional constant of 2 000 gram-centimeters.

9/10

4.9 A spacer shall be inserted between the specimen rack and each specimen clamp stud before low-temperature exposure. The low temperature chamber or cold box shall be adjusted to the specified temperature. The specimens shall be exposed at the specified temperature for the required period of time, 4 5. The torsion apparatus shall be exposed at the specified temperature for not less than 2 hours. At the end of the exposure period, 4.5, the spacer shall be removed, the pointer on the protractor adjusted to zero, each specimen measured at the temperature of exposure, and the value recorded, as described in 4.8. When it is necessary to handle the specimen at the low temperature, forceps or tongs which have been conditioned for at least 2 hours shall be used. The forceps or tongs shall be handled with gloved hands after the gloves have been conditioned for at least 5 minutes. At least 30 seconds shall elapse between the handling and testing of the specimen.

5. RESULTS

5.1 Calculations.—The torsional stiffness ratio of the specimen shall be calculated as follows:

5.1.1 The torsional stiffness factor at each temperature, 4.7 and 4.9 equals:

$$\frac{\text{180 degrees—angle of twist of the specimen}}{\text{Angle of twist of the specimen}}$$

5.1.2 The torsional stiffness ratio at the specified low temperature, 4.3, equals:

$$\frac{\text{Torsional stiffness factor at low temperature, 4 3}}{\text{Torsional stiffness factor at standard temperature, 4 7}}$$

5.2 The torsional stiffness ratio of the test unit shall be the average of the values obtained from the specimens tested.

5.3 Unless otherwise specified in the detail specification, three specimens shall be tested.

5.4 If required, the modulus of rigidity shall be calculated in accordance with 5611.

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