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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION
R 1432

DETERMINATION OF THE STIFFNESS OF VULCANIZED RUBBERS AT LOW TEMPERATURE (GEHMAN TEST)

1st EDITION

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For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 1432, *Determination of the stiffness of vulcanized rubbers at low temperature (Gehman test)*, was drawn up by Technical Committee ISO/TC 45, *Rubber*, the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1432, which was circulated to all the ISO Member Bodies for enquiry in July 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Hungary	Spain
Austria	India	Sweden
Brazil	Iran	Switzerland
Canada	Israel	Thailand
Colombia	Italy	Turkey
Czechoslovakia	Korea, Rep. of	U.A.R.
France	Netherlands	United Kingdom
Germany	New Zealand	U.S.A.
Greece	Poland	U.S.S.R.

No Member Body opposed the approval of the Draft.

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

ISO Recommendation   R 1432   January 1971

DETERMINATION OF THE STIFFNESS OF VULCANIZED RUBBERS AT LOW TEMPERATURE

(GEHMAN TEST)

1. SCOPE

This ISO Recommendation describes a static procedure for determining the relative stiffness characteristics of rubbers over a temperature range from room temperature to approximately – 70 °C.

2. APPARATUS*

2.1 *Torsion apparatus*, as shown in the Figure. It consists of a torsion head (A), capable of being turned 180 ° in a plane normal to the torsion wire (B). The top of the wire is fastened to the torsion head through a loosely fitting sleeve (C). The bottom of the wire is fastened to the test piece clamp stud (D) by means of a screw connector (E). A pointer (F) and a movable protractor (G) are provided to permit convenient and exact adjustment of the zero point. The torsion apparatus is clamped to a supporting stand (H). It is advantageous to make the vertical portion of the stand from material of poor thermal conductivity. The base of the stand should be of stainless steel or other corrosion resistant material.

2.2 *Torsion wires*, made of tempered spring wire, 65 ± 8 mm long, and having torsional constants of 0.70, 2.81 and 11.24 mJ/rad of twist.

The 2.81 mJ/rad wire should be considered the standard wire.

2.3 *Test piece rack*, (I), made of material of poor thermal conductivity, for holding the test piece (J) in a vertical position in the heat transfer medium. The rack should be constructed to hold several test pieces**. The rack is clamped to the stand (H).

Two clamps should be provided for holding each test piece. The bottom clamp (K) should be a fixed part of the test piece rack. The top clamp (L) acts as an extension of the test piece and should not touch the rack***. The top clamp is secured to a stud (D) which in turn is connected to the screw connector (E).

2.4 *Temperature measuring device*, capable of measuring the temperature within 1 °C over the range from approximately – 70 °C to + 30 °C. Copper-constantan thermocouples, used in conjunction with a potentiometer, are highly satisfactory.

The sensitive element should be positioned between two test pieces, equidistant between the top and the bottom of the test pieces.

* The apparatus and its use are described in "Low temperature characteristics of elastomers", S.D. Gehman, D.E. Woodford and C.S. Wilkinson, Ind. and Eng. Chem., Vol. 39, Sept. 1947, page 1108.

** Racks providing space for five or ten test pieces are commonly used.

*** Clearance between the top of the test piece rack and the test piece clamp stud is ensured by inserting thin spacers between the two. Slotted laminated plastics about 1.3 mm thick and 12 mm wide have been found satisfactory. At low temperatures the test pieces stiffen in position and the spacers may be removed without losing the clearance.

2.5 *Heat transfer media,* which may be liquid or gaseous. Any material which remains fluid at the test temperature and which will not effect the materials being tested may be used. Among the liquids that have been found suitable for use at low temperatures are acetone, methanol, ethanol, butanol, silicone fluid and *n*-hexane. Carbon dioxide or air are the commonly used gaseous media.

Vapours of liquid nitrogen are useful for testing at very low temperatures.

It should be noted that stiffness measurements in gaseous media may not give in each case the same results as the measurements made in liquid media.

2.6 *Temperature control,* for controlling the temperature of the heat-transfer medium within ± 1.0 °C.

2.7 *Tank,* for liquid heat-transfer media, or a *test chamber* for gaseous media.

2.8 *Stirrer,* for liquids, or a *fan* or *blower* for air, which ensures thorough circulation of the heat-transfer medium.

2.9 *Stop-watch,* or other timing device, calibrated in seconds.

3. TEST PIECE

3.1 Preparation of test piece

The test piece should be 40 ± 2.5 mm long, 3 ± 0.2 mm wide, and 2 ± 0.2 mm thick. It should be moulded or cut with a suitable die from a vulcanized sheet of proper thickness.

3.2 Conditioning of test piece

3.2.1 The minimum time between vulcanization and testing should be 16 hours.

For non-product tests the maximum time between vulcanization and testing should be 4 weeks and for evaluations intended to be comparable, the tests, as far as possible, should be carried out after the same time interval.

For product tests, whenever possible, the time between vulcanization and testing should not exceed 3 months. In other cases tests should be made within 2 months of the date of receipt by the customer of the product.

3.2.2 Samples and test pieces should be protected from light as completely as possible during the interval between vulcanization and testing.

3.2.3 Prepared test pieces should be conditioned immediately before testing for a minimum of 3 hours at a standard laboratory temperature, the same temperature being used throughout any one test or series of tests intended to be comparable.

4. PROCEDURE

4.1 Calibration of torsion wire

One end of the torsion wire should be inserted in a vertical position, in a fixed clamp, and the lower end of the wire attached at the exact longitudinal centre of a rod of known dimensions and mass. (It is suggested that the rod be 200 to 250 mm long and about 6.4 mm in diameter.)

The rod should be twisted through an angle of not more than 90° and then released. It 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.)

The mass moment of inertia should be calculated from the following formula :

$$I = \frac{m\,l^2}{12}$$

where

I is the moment of inertia, in kilogramme millimetres squared, of the rod;

m is the mass, in kilogrammes, of the rod;

l is the length, in millimetres, of the rod.

The torsional constant should be calculated from the following formula :

$$\lambda = \frac{\pi^2 I}{250\,T^2}$$

where

T is the period of one oscillation, in seconds;

I is the moment of inertia, in kilogramme millimetres squared, of the rod;

λ is the restoring torque, in millijoules per radian of twist, by the wire.

The torsion wires should calibrate within ± 3 % of their specified torsional constants.

4.2 Mounting of test piece

Each of the test pieces used should be clamped in such a manner that 25 ± 3 mm of the piece is free between the clamps. The test piece clamp stud (D) should be located with respect to a reference point on the rack (I) in such a position that the specimen is under zero torque.

4.3 Stiffness measurements in liquid media

The rack containing the test pieces should be placed in the liquid bath with a minimum of 25 mm of liquid covering the test pieces. The temperature of the bath should then be adjusted to 25 °C. One of the test pieces should be connected to the torsion head by means of the screw connector (E) and the standard wire.

Caution should be exercised in attaching the screw connector to the test piece clamp stud (D) to make certain that the stud is not moved from the zero torque position. The torsion head (A) should also remain in the zero position while the connector is being fastened to the stud. The spacer which provides clearance between the test piece rack and the test piece clamp stud need not be used for measurements made at room temperature.

The pointer reading should be adjusted to zero by rotating the protractor scale (G). The torsion head should then be turned quickly but smoothly 180° and the pointer reading, after 10 seconds as indicated by the timer, recorded. If the reading at 25 °C does not fall in the range of 120 to 170°, the standard torsion wire is not suitable for testing the test piece. Test pieces producing twists of more than 170° should be tested with a wire having a torsional constant of 0.70 mJ/rad of twist. Test pieces producing twists of less than 120° should be tested with a wire having a torsional constant of 11.24 mJ/rad of twist.

The torsional head should be returned to its initial position and the test piece disconnected.

The test piece rack should then be moved to bring the next test piece into position for measurement*. All test pieces in the rack should be measured at 25 °C.

The spacers should be inserted between the test piece rack and the test piece clamp studs. The test pieces should be removed from the liquid bath and the temperature of the liquid adjusted to the lowest temperature desired. The test pieces should be replaced in the bath and allowed to remain at this temperature for approximately 15 minutes. After this, one spacer should be removed and one test piece measured as was done at 25 °C**. The spacer should be returned to its original position after the test piece has been tested. All the test pieces in the rack should be measured in this way, taking care that the measurement of each test piece lasts approximately 2 minutes.

The bath temperature should then be increased by 5 °C intervals, each increase being made in approximately 5 minutes and stiffness measurement made after conditioning of the test piece for 5 minutes at each temperature. Tests should be continued until a temperature is reached at which the angular twist is within 5 to 10° of the twist at 25 °C.

4.4 Stiffness measurements in gaseous media

Procedures in air or carbon dioxide differ from those with liquid media only in that cooling is effected with the test pieces in the medium and the length of the conditioning period is different.

With the test pieces in the test chamber the temperature of the chamber should be adjusted to the lowest temperature desired in approximately 30 minutes. After this temperature has been maintained constant for 10 minutes the measurements should be made in a similar way as in the liquid media, each test piece being tested in 2 minutes.

The temperature of the chamber should be increased by 5 °C intervals, each increase being made in approximately 10 minutes, and stiffness measurements made after conditioning of the test pieces for 10 minutes at each temperature.

4.5 Crystallization

When it is desired to study crystallization or plasticizer effects, the time of conditioning at the desired temperature should be increased.

* 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 pieces in turn.

** 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.

5. NUMBER OF TESTS

At least three test pieces of each material should be tested. It is good practice to include a control rubber with known twist-temperature characteristics.

6. EXPRESSION OF RESULTS

6.1 A plot should be made of the pointer readings or angles of twist of the test piece, against the temperature.

The torsional modulus of the test piece at any temperature is proportional to the quantity

$$\frac{180 - \alpha}{\alpha}$$

where α is the angle, expressed in degrees, of the twist of the test piece

The relative modulus at any temperature is the ratio of the torsional modulus at that temperature to the torsional modulus at 25 °C.

The value of the relative modulus for any temperature is readily determined from the angles of twist at that temperature and at 25 °C, as given by the twist versus temperature plot, and the ratio of the values of the factor $\frac{180 - \alpha}{\alpha}$ corresponding to those angles.

The temperatures at which the relative modulus is 2, 5, 10 and 100, are determined by the use of Table 1 and the twist versus temperature curve for the test piece. The first column of Table 1 lists each degree of twist in the range of 120 to 170°, so that the value corresponding to the twist of the test piece at 25 °C 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 test piece and are designated as T_2, T_5, T_{10} and T_{100} respectively.

6.2 When it is desired to calculate the *apparent torsional modulus of rigidity* in newtons per square millimetre at various temperatures, the free length of the test piece should be accurately measured and the following formula used :

$$G = \frac{16 K L (180 - \alpha)}{a b^3 \mu \alpha}$$

where

- G is the modulus of rigidity, in meganewtons per square metre;
- K is the torsional constant, in millijoules per radian, of the wire;
- a is the width, in millimetres, of the test piece;
- b is the thickness, in millimetres, of the test piece;
- L is the measured free length, in millimetres, of the test piece;
- μ is the factor based on the ratio a/b taken from Table 2;
- α is the angle of twist, in degrees, of the test piece.

7. TEST REPORT

The test report should include the following particulars :

(*a*) the heat transfer medium used;

(*b*) the temperatures T_2, T_5, T_{10} and T_{100} at which the relative modulus is 2, 5, 10 and 100;

(*c*) the apparent torsional modulus of rigidity, in newtons per square millimetre, at room temperature;

(*d*) when required, the apparent torsional modulus at other temperatures than room temperature.