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# International Standard



# 1432

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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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## Rubber, vulcanized — Low temperature stiffening (Gehman test) — Determination

*Caoutchouc vulcanisé — Rigidité à basse température (essai Gehman) — Détermination*

Second edition — 1982-12-15

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**Descriptors** : rubber, vulcanized rubber, tests, low temperature tests, stiffness tests.

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# Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 1432 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products*. The first edition (ISO 1432-1976) had been approved by the member bodies of the following countries :

|                     |                |                |
|---------------------|----------------|----------------|
| Australia           | Greece         | Poland         |
| Austria             | Hungary        | Spain          |
| Brazil              | India          | Sweden         |
| Canada              | Iran           | Switzerland    |
| Colombia            | Israel         | Thailand       |
| Czechoslovakia      | Italy          | Turkey         |
| Egypt, Arab Rep. of | Korea, Rep. of | United Kingdom |
| France              | Netherlands    | USA            |
| Germany, F. R.      | New Zealand    | USSR           |

No member body had expressed disapproval of the document.

This second edition, which cancels and replaces ISO 1432-1976, incorporates draft amendment 1, which was circulated to the member bodies in January 1980 and has been approved by the member bodies of the following countries :

|                     |                       |                |
|---------------------|-----------------------|----------------|
| Austria             | India                 | Sri Lanka      |
| Belgium             | Italy                 | Sweden         |
| Brazil              | Korea, Rep. of        | Switzerland    |
| China               | Mexico                | Thailand       |
| Denmark             | Netherlands           | Turkey         |
| Egypt, Arab Rep. of | Poland                | United Kingdom |
| Finland             | Romania               | USA            |
| Germany, F. R.      | South Africa, Rep. of | USSR           |
| Hungary             | Spain                 |                |

The member bodies of the following countries expressed disapproval of the document on technical grounds :

Canada  
France

# Rubber, vulcanized — Low temperature stiffening (Gehman test) — Determination

## 0 Introduction

This second edition of ISO 1432 differs from the first in allowing a continuous as well as a stepwise increase of temperature, and a minimum temperature of  $-150\text{ }^{\circ}\text{C}$  rather than  $-70\text{ }^{\circ}\text{C}$ .

## 1 Scope and field of application

This International Standard specifies a static procedure, known as the Gehman test, for determining the relative stiffness characteristics of vulcanized rubbers over a temperature range from room temperature to approximately  $-150\text{ }^{\circ}\text{C}$ .

## 2 Apparatus<sup>1)</sup>

**2.1 Torsion apparatus**, one possible form of which is shown in the figure. It consists of a torsion head (A), capable of being turned  $180^{\circ}$  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 device for "friction-free" indication or recording of angle by mechanical or electrical means should be provided permitting convenient and exact adjustment of the zero point; the apparatus shown in the figure has a pointer (F) and a movable protractor (G) which perform these functions. The indicating or recording system should allow reading or recording of the angle of twist to the nearest degree. 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 shall be of stainless steel or other corrosion-resistant material.

**2.2 Torsional wires (B)**, made of tempered spring wire, of length  $65 \pm 8\text{ mm}$ , and having torsional constants of 0,7; 2,81 and 11,24 mN·m.

**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 may be constructed to hold several test pieces.<sup>2)</sup> The rack is clamped to the stand (H).

Two clamps shall be provided for holding each test piece. The bottom clamp (K) shall be a fixed part of the test piece rack. The top clamp (L) acts as an extension of the test piece and shall not touch the rack.<sup>3)</sup> 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 to within  $1\text{ }^{\circ}\text{C}$  over the whole range of temperature over which the apparatus is to be used.

The sensitive element shall be positioned near a test piece equidistant from the top and bottom.

**2.5 Heat-transfer media**, which may be liquid or gaseous. Any material which remains fluid at the test temperature and which will not affect 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. Air, carbon dioxide or nitrogen are 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**, capable of maintaining the temperature of the heat-transfer medium to within  $\pm 1,0\text{ }^{\circ}\text{C}$ .

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

1) The apparatus and its use are described in : GEHMAN, S. D.; WOODFORD, D. E. and WILKINSON, C. S., Low temperature characteristics of elastomers. *Ind. and Eng. Chem.*, **39** Sept. 1947 : 1108.

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

3) 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 of thickness about 1,3 mm and width about 12 mm have been found satisfactory. At low temperatures the test pieces stiffen in position and the spacers may be removed without losing the clearance.

**2.8 Stirrer**, for liquids, or **fan** or **blower** for gases, 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 dimensions of the test piece shall be  $40 \pm 2,5$  mm,  $3 \pm 0,2$  mm and  $2 \pm 0,2$  mm. It shall be moulded or cut with a suitable die from a vulcanized sheet of suitable thickness.

#### 3.2 Conditioning of test piece

**3.2.1** The minimum time between vulcanization and testing shall be 16 h.

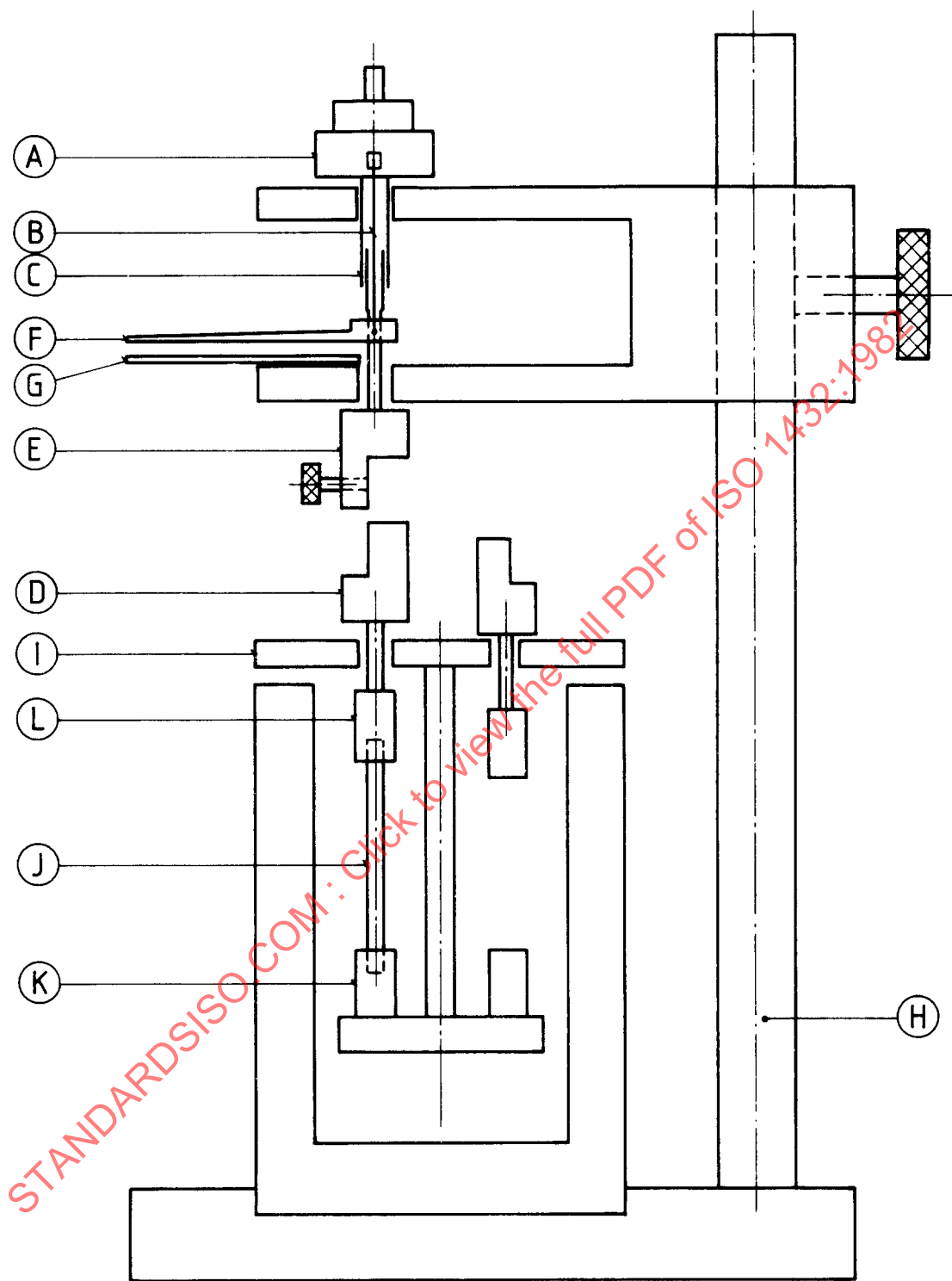
For non-product tests, the maximum time between vulcanization and testing shall be 4 weeks and, for evaluations intended to be comparable, the tests should be carried out, as far as possible, after the same time interval.

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

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

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

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|------------------|------------------------|------------------|
| (A) Torsion head | (E) Screw connector    | (I) Rack         |
| (B) Torsion wire | (F) Pointer            | (J) Test piece   |
| (C) Sleeve       | (G) Movable protractor | (K) Bottom clamp |
| (D) Clamp stud   | (H) Supporting stand   | (L) Top clamp    |

Figure — Apparatus for determination of stiffness characteristics

## 4 Procedure

### 4.1 Calibration of torsion wire

Insert one end of the torsion wire (B) in a vertical position, in a fixed clamp, and attach the lower end of the wire at the exact longitudinal centre of a rod of known dimensions and mass. (It is suggested that the length of the rod be 200 to 250 mm and the diameter about 6,4 mm).

Twist the rod through an angle of not more than 90° and then release it. Allow it to oscillate freely in a horizontal plane and note the time, in seconds, for 20 oscillations. (An oscillation includes the swing from one extreme to the other and return.)

The mass moment of inertia,  $I$ , expressed in kilogram metres squared, is given by the equation

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

where

$m$  is the mass, in kilograms, of the rod;

$L$  is the length, in metres, of the rod.

The torsional constant of the wire (i.e. the resulting torque per radian),  $K$ , expressed in newton metres, is given by the equation

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

where  $T$  is the period, in seconds, of one oscillation.

The torsion wires shall calibrate within  $\pm 3\%$  of their specified torsional constants.

### 4.2 Mounting of test piece

Clamp each test piece used in such a manner that  $25 \pm 3$  mm of the test piece is free between the clamps. The test piece clamp stud (D) shall 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

Place the rack (I) containing the test pieces in the liquid bath with a minimum of 25 mm of liquid covering the test pieces. Then adjust the temperature of the bath to  $23 \pm 2^\circ\text{C}$ . Connect one of the test pieces to the torsion head (A) by means of the screw connector (E) and the standard wire.

Take care when attaching the screw connector to the test piece clamp stud (D) not to move the stud from the zero torque

position. The position head (A) shall 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.

Adjust the angle indicating or recording device to zero. Then turn the torsion head quickly but smoothly through  $180^\circ$  and record the torsion angle after 10 s. If the reading at  $23^\circ\text{C}$  does not fall in the range of  $120$  to  $170^\circ$  the standard torsion wire is not suitable for testing the test piece. Test pieces producing twists of more than  $170^\circ$  shall be tested with a wire having a torsional constant of  $0,7\text{ mN}\cdot\text{m}$ . Test pieces producing twists of less than  $120^\circ$  shall be tested with a wire having a torsional constant of  $11,24\text{ mN}\cdot\text{m}$ .

Return the torsion head to its initial position and disconnect the test piece.

Then move the test piece rack to bring the next test piece into position zero for measurement.<sup>1)</sup>

Measure all the test pieces in the rack at  $23 \pm 2^\circ\text{C}$ .

Insert the spacers between the test piece rack and the test piece clamp studs. Remove the test pieces from the liquid bath and adjust the temperature of the liquid to the lowest temperature desired. Replace the test pieces in the bath and maintain them at this temperature for approximately 15 min. After this, remove one spacer and measure one test piece as was done at  $23^\circ\text{C}$ .<sup>2)</sup> Return the spacer to its original position after the test piece has been tested. Measure all the test pieces in the rack in this way, taking care that the measurement of each test piece lasts approximately 2 min.

Then increase the bath temperature by one of the two following methods :

- by stepwise  $5^\circ\text{C}$  intervals, each increase being made after approximately 5 min;
- increase the temperature continuously with a heating rate of  $1^\circ\text{C}/\text{min}$ .

Make the stiffness measurement in the stepwise case after conditioning of the test piece for 5 min at each temperature and in the continuous case at 5 min intervals. Continue the tests until a temperature is reached at which the angular twist is within 5 to  $10^\circ$  of the twist at  $23^\circ\text{C}$ .

### 4.4 Stiffness measurement in gaseous media

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

1) 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.

2) Movement of the spacer may alter the setting of the angle indicating or recording device; therefore, adjust this device to zero after removing the spacer.