International Standard ISO 1432

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

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

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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

Printed in Switzerland

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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 °C rather than − 70 °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 °C.

2 Apparatus[1]

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° 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 ± 8 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.[2] 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.[3] 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 °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 ± 1,0 °C.

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

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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 ± 2,5 mm, 3 ± 0,2 mm and 2 ± 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

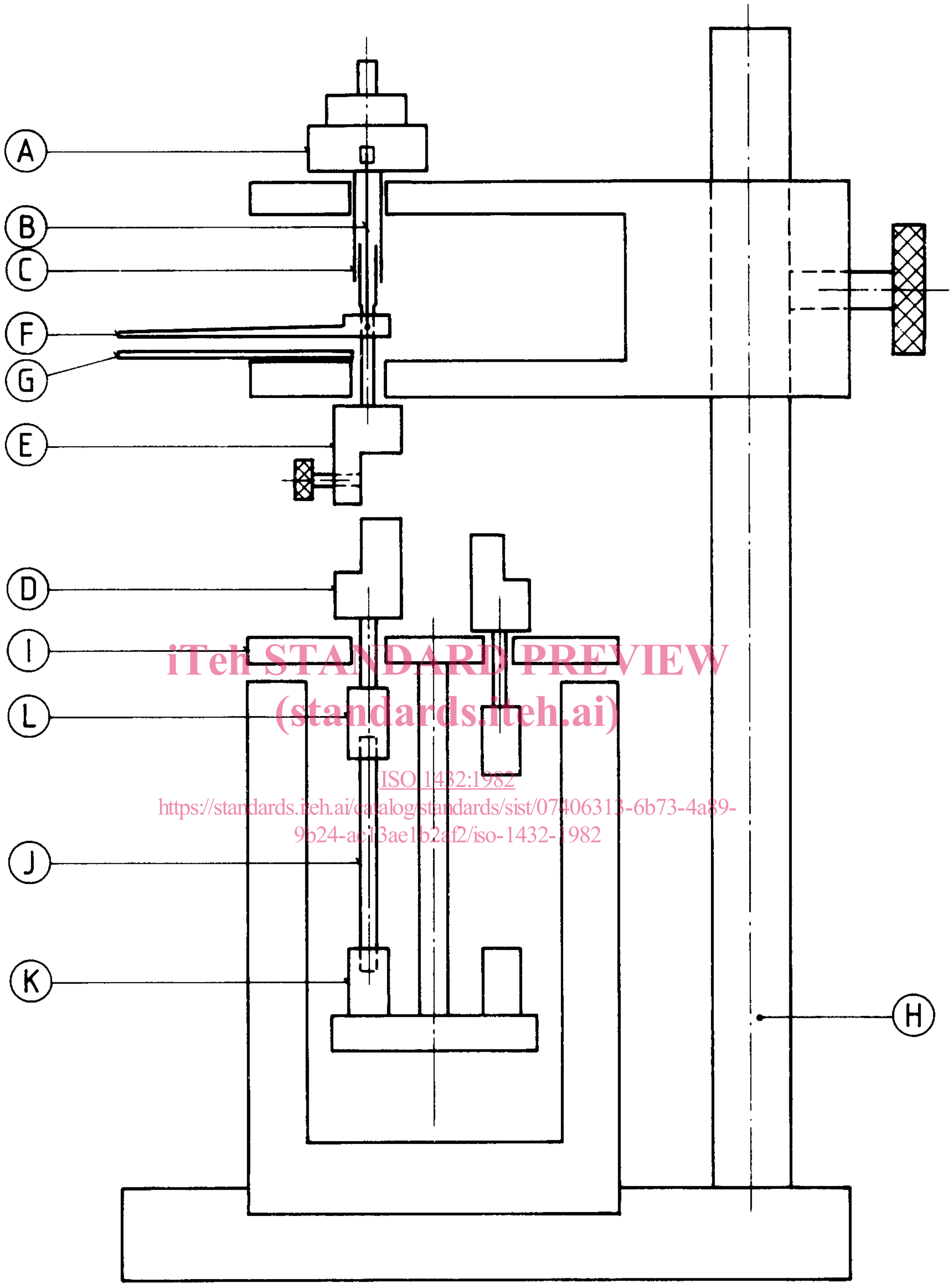
3.2.1 The minimum time between vulcanization and testing shall be 16 h.

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.



(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 ± 3 % of their specified torsional constants.

4.2 Mounting of test piece

Clamp each test piece used in such a manner that 25 ± 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 ± 2 °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° and record the torsion angle after 10 s. If the reading at 23 °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° shall be tested with a wire having a torsional constant of 0,7 mN·m. Test pieces producing twists of less than 120° shall be tested with a wire having a torsional constant of 11,24 mN·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.[1)]

Measure all the test pieces in the rack at 23 ± 2 °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 °C.[2)] 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 :

a) by stepwise 5 °C intervals, each increase being made after approximately 5 min;

b) increase the temperature continuously with a heating rate of 1 °C/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° of the twist at 23 °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.

4.4.1 Increase of temperature in steps

With the test pieces in the test chamber, adjust the temperature of the chamber to the lowest temperature desired in approximately 30 min. After this temperature has been maintained constant for 10 min, make the measurements in a similar way as in the liquid media, each test piece being tested within 2 min.

Increase the temperature of the chamber by 5 °C intervals, each increase being made in approximately 10 min, and make stiffness measurements after conditioning of the test pieces for 10 min at each temperature.

4.4.2 Continuous increase of temperature

With the test pieces in the test chamber adjust the temperature of the chamber to the lowest temperature desired, by application of a linear time programme, preferably with a rate of 3 °C/min. After this temperature has been reached, increase the temperature linearly at a rate of 1 °C/min.

Carry out measurements of the twist angle at 5 °C intervals.

4.5 Crystallization

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

5 Number of tests

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

6 Expression of results

6.1 Twist versus temperature curve

Plot a graph of the pointer readings (angles of twist of the test piece) against the temperature.

6.2 Torsional modulus

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

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

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

6.3 Relative modulus

The relative modulus at any temperature is the ratio of the torsional modulus at that temperature to the torsional modulus at 23 °C.

The value of the relative modulus for any temperature is readily determined from the angles of twist at that temperature and at 23 °C, as given by the twist versus temperature curve (6.1) and the ratio of the values of the factor $(180 - \alpha)/\alpha$ corresponding to those angles.

The temperatures at which the relative modulus is 2, 5, 10 and 100 respectively, 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 23 °C can be selected.

Successive columns give the twist angles which correspond to values 2, 5, 10 and 100 respectively, for the relative modulus. The temperatures corresponding to these angles are then read from the twist versus temperature curve for the test piece (6.1) and are designated as T_2, T_5, T_{10} and T_{100} respectively.

6.4 Apparent torsional modulus of rigidity

When it is desired to calculate the apparent torsional modulus of rigidity in pascals[1)] at various temperatures, the free length of the test piece shall be accurately measured.

The apparent torsional modulus of rigidity, G, in pascals, is given by the equation

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

where

K is the torsional constant, in newton metres, of the wire;

L is the measured free length, in metres, of the test piece;

b is the width, in metres, of the test piece;

d is the thickness, in metres, of the test piece;

μ is the factor based on the ratio b/d taken from table 2;

α is the angle of twist, in degrees, of the test piece.

1) 1 Pa = 1 N/m^2

Table 1 — Twist angles for designated values of the relative modulus (RM)

Twist angle α, in degrees at 23 °C	Twist angle α in degrees for relative modulus (RM)			
	RM = 2	RM = 5	RM = 10	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

Table 2 — Values[1] of factor μ for various ratios of b/d

b/d	μ	b/d	μ
1,00	2,25	2,25	3,84
1,05	2,36	2,50	3,99
1,10	2,46	2,75	4,11
1,15	2,56	3,00	4,21
1,20	2,66	3,50	4,37
1,25	2,75	4,00	4,49
1,30	2,83	4,50	4,59
1,35	2,91	5,00	4,66
1,40	2,99	6,00	4,77
1,45	3,06	7,00	4,85
1,50	3,13	8,00	4,91
1,60	3,26	9,00	4,96
1,70	3,38	10,00	5,00
1,75	3,43	20,00	5,17
1,80	3,48	50,00	5,23
1,90	3,57	100,00	5,30
2,00	3,66		

1) Values of μ have been rounded to two places of decimals.

7 Test report

The test report shall include the following information :

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 respectively;

c) the apparent torsional modulus of rigidity, in pascals, at room temperature;

d) when required, the apparent torsional modulus at temperatures other than room temperature;

e) when required, the temperature at which the apparent torsional modulus reaches a specified value.