IS : 3400 (Part XVIII) - 1976

Indian Standard

METHODS OF TEST FOR VULCANIZED RUBBERS

PART XVIII STIFFNESS AT LOW TEMPERATURE (GEHMAN TEST)

UDC 678·43:620·163

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Price Rs 7·00

November 1976

Indian Standard

METHODS OF TEST FOR VULCANIZED RUBBERS

PART XVIII STIFFNESS AT LOW TEMPERATURE (GEHMAN TEST)

Rubber Products Sectional Committee, CDC 6

Chairman	*Representing*
DR D. BANERJEE	Escon Consultants Pvt Ltd, Calcutta
Members	
SHRI M. L. BAHRANI	Ministry of Defence (R & D)
SHRI ANIL AGARWAL (*Alternate*)	
SHRI A. K. BANDYOPADHAYA	Ministry of Defence (DGI)
SHRI M. KUMARAN (*Alternate*)	
SHRI M. BANERJEE	National Rubber Manufacturers Ltd, Calcutta
SHRI J. CHATTERJEE (*Alternate*)	
SHRI A. T. BASAK	Directorate General of Supplies & Disposals, New Delhi
SHRI S. K. BOSE	National Test House, Calcutta
SHRI A. GHOSH (*Alternate*)	
DR S. N. CHAKRAVARTY	Bayer (India) Ltd, Bombay
SHRI R. R. PANDIT (*Alternate*)	
SHRI D. K. CHATTERJEE	Alkali & Chemical Corporation of India Ltd, Calcutta
DR S. K. ROY (*Alternate*)	
SHRI S. S. CHOPRA	Export Inspection Council of India, Calcutta
SHRI K. M. BIJLI (*Alternate*)	
SHRI W. G. DESAI	All India Rubber Industries Association, Bombay
SHRI K. R. SENGUPTA (*Alternate*)	
SHRI K. N. GANESH	Indian Petrochemicals Corporation Ltd, Vadodara
SHRI S. B. GANGULI	Dunlop India Ltd, Calcutta
SHRI P. N. S. MYER (*Alternate*)	
SHRI A. GEORGE JOHN	Madras Rubber Factory Ltd, Madras
SHRI K. J. ABRAHAM (*Alternate*)	
SHRI G. C. JAIN	Hindustan Steel Ltd, Ranchi
SHRI R. C. JHINGAN	Indian Oil Corporation Ltd, Bombay
SHRI LALIT MOHAN JAMNADAS	Cosmos India Rubber Works Pvt Ltd, Bombay
SHRI PULIN L. KINARIWALA (*Alternate*)	
SHRI S. V. LATHIA	Lathia Rubber Manufacturing Co Pvt Ltd, Bombay
SHRI D. P. LATHIA (*Alternate*)	

(*Continued on page* 2)

(*Continued from page* 1)

Members	*Representing*
DR S. P. MANIK	Railway Board (Ministry of Railways)
SHRI D. N. V. CHELLAM (*Alternate*)	
DR C. K. N. NAIR	Rubber Board, Kottayam
SHRI S. C. NANDY	Bata India Ltd, Calcutta
SHRI SUNIL SARKAR (*Alternate*)	
SHRI M. M. PATEL	Synthetics & Chemicals Ltd, Bombay
DR N. V. C. RAO	Directorate General of Technical Development, New Delhi
SHRI G. R. INAMDAR (*Alternate*)	
SHRI V. R. RAO	Sundaram Industries Pvt Ltd, Madurai
SHRI K. C. MADHUSUDHANAN (*Alternate*)	
SHRI R. C. SYED	Indian Rubber Manufacturers' Research Association, Bombay
SHRI D. D. TALWALKAR	All India Automobile & Ancillary Industries Association, Bombay
SHRI K. M. KHALADKAR (*Alternate*)	
DR G. M. SAXENA, Director (Chem)	Director General, ISI (*Ex-officio Member*)

Secretary

SHRI SATISH CHANDER
Deputy Director (Chem), ISI

Methods of Testing Vulcanized Rubbers Subcommittee, CDC 6:9

Convener	
SHRI M. M. PATEL	Synthetics & Chemicals Ltd, Bombay
Members	
SHRI A. D. BANDKAR (*Alternate* to Shri M. M. Patel)	
SHRI A. K. BANDYOPADHAYA	Ministry of Defence (DGI)
SHRI P. L. NAG (*Alternate*)	
SHRI M. BANERJEE	National Rubber Manufacturers Ltd, Calcutta
SHRI J. CHATTERJEE (*Alternate*)	
SHRI B. BHATTACHARYYA	Incheck Tyres Ltd, Calcutta
SHRI R. N. DUTTA (*Alternate*)	
SHRI S. K. BOSE	National Test House, Calcutta
SHRI A. GHOSH (*Alternate*)	
SHRI B. CHAKRAVARTY	Escon Consultants Pvt Ltd, Calcutta
DR S. N. CHAKRAVARTY	Bayer (India) Ltd, Bombay
DR S. K. BHATNAGAR (*Alternate*)	
SHRI D. K. CHATTERJEE	Alkali & Chemical Corporation of India Ltd, Calcutta
SHRI N. C. SAMAJDAR (*Alternate*)	
SHRI S. B. GANGULI	Dunlop India Ltd, Calcutta
SHRI A. MUKHERJEE (*Alternate*)	

(*Continued on page* 14)

Indian Standard

METHODS OF TEST FOR VULCANIZED RUBBERS

PART XVIII STIFFNESS AT LOW TEMPERATURE (GEHMAN TEST)

0. FOREWORD

0.1 This Indian Standard (Part XVIII) was adopted by the Indian Standards Institution on 8 July 1976, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Chemical Division Council.

0.2 In the preparation of this standard, assistance has been drawn from the following publications:

ISO/R 1432-1970 Determination of the stiffness of vulcanized rubber at low temperature. International Organization for Standardization

ASTM D 1053-1965 (Reapproved 1970) Measuring low-temperature stiffening of rubber and rubber-like materials by means of a torsional wire apparatus. American Society for Testing and Materials

0.3 The proposed test will not in general involve stiffening due to crystallization, as the rate at which crystallization takes place is slow compared with the speed of testing.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

---

1. SCOPE

1.1 This standard (Part XVIII) 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 — It consists of a torsion head capable of being turned through 180° in a plane normal to the torsion wire. The top of the

*Rules for rounding off numerical values (*revised*).

wire is fastened to the torsion head through a loosely fitting sleeve. The bottom of the wire is fastened to the test piece clamp stud by means of a screw connector. A pointer and a movable protractor are provided to permit convenient and exact adjustment of the zero point. The torsion apparatus is clamped to a supporting stand. 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. The apparatus is shown in Fig. 1.

2.2 Torsion Wires — These are 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 is considered the standard wire.

2.3 Test Piece Rack — It is made of material of poor thermal conductivity, for holding the test piece in a vertical position in the heat transfer medium. The rack should be constructed to hold 5 or multiples of 5 test pieces. The rack is clamped to the stand.

Two clamps shall be provided for holding each test piece. The bottom clamp shall be a fixed part of the test piece rack. The top clamp acts as an extension of the test piece and shall not touch the rack while the test piece is being twisted. Clearance between the top of the test piece rack and the test piece clamp stud is assured by inserting thin spacers between the two. The top clamp is secured to a stud which in turn is connected to the screw connector.

NOTE — Slotted PTFE or fluorecarbon spacers about 1·3 mm thick and 13 mm 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.

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 shall be positioned between two test pieces, equidistant between the top and the bottom of the test pieces. Suitable thermometer capable of measuring in the above range may also be used.

2.5 Heat Transfer Media — This may be liquid or gaseous. Any material which remains fluid at the test temperature and which will not affect 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 is the commonly used gaseous media.

Vapours of liquid nitrogen are useful for testing at very low temperature. It should be noted that stiffness measurements in gaseous media may not give in each case the same results as the measurement made in liquid media.

NOTE — Specifications for rubber products requiring tests using this procedure shall specifically state which coolant media are acceptable for use in this test.

2.6 Temperature Control — for controlling the temperatures of the heat-transfer medium within $\pm 1.0°C$.

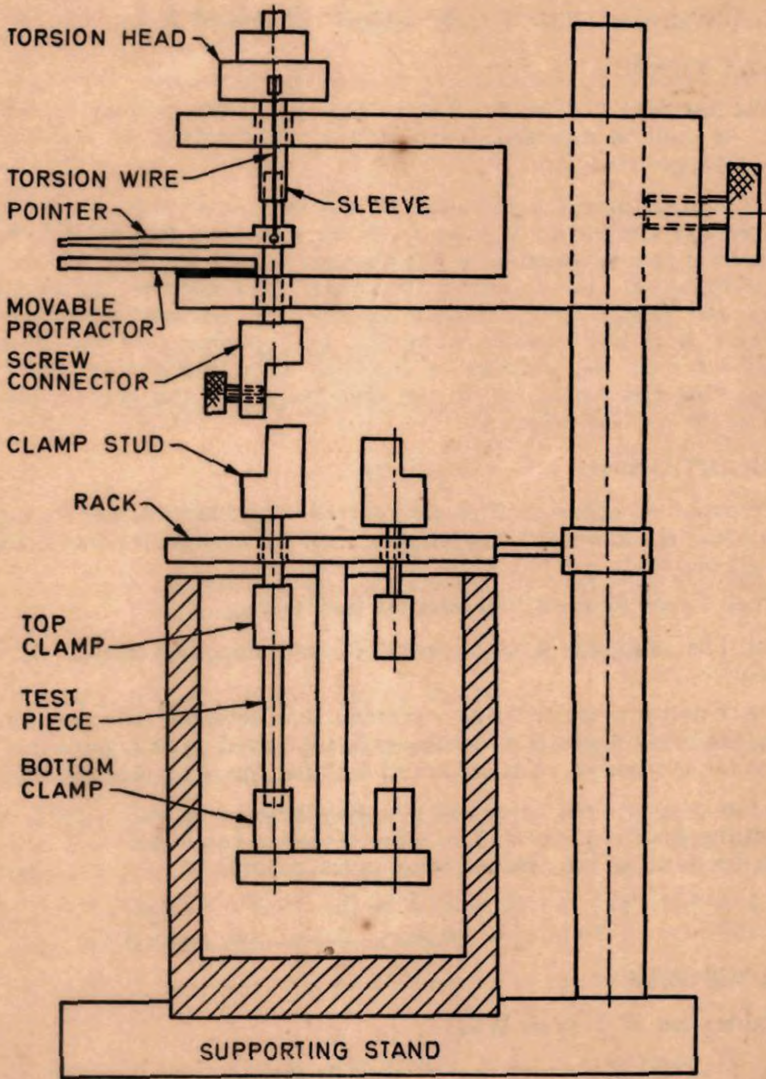

FIG. 1 SCHEMATIC DIAGRAM OF TORSION APPARATUS

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

2.8 Circulating Device — for liquids it may be stirrer and for gases a fan or blower, 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 The test piece shall be 40 ± 3 mm long, $3\pm0{\cdot}2$ mm wide, and $2\pm0{\cdot}2$ mm thick. It shall be moulded or cut with a suitable die from a vulcanized sheet of proper thickness.

3.1.1 Test specimens from vulcanized rubber product may also be tested provided that the finished dimensions of the specimens obtainable from the product are in accordance with those mentioned above. For this purpose test pieces may be cut or slit from the product to a thickness not exceeding 4 mm and finally buffed smooth carefully to the specified thickness. Wherever it is not possible to obtain test specimens of the required dimensions from the product, a moulded sheet made from the same composition and vulcanized to the same degree as the product may be provided by the manufacturer.

4. CONDITIONING

4.1 Prepared test pieces shall be conditioned immediately before testing for a minimum of 3 hours at a temperature of 27 ± 2°C and a relative humidity of 65 ± 5 percent.

4.2 Time Lapse Between Vulcanization and Testing

4.2.1 The minimum time between vulcanization and testing shall be 16 hours.

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

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

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

5. PROCEDURE

5.1 Calibration of Torsion Wire

5.1.1 One end of the torsion wire shall 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.) Initially the rod shall be twisted through an angle of not more than 90° and then released. It shall 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 shall be calculated from the following formula:

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

where

I = moment of inertia in kg·mm² of the rod,

m = mass in kg of the rod,

l = length in mm of the rod.

The torsional constant shall be calculated from the following formula:

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

where

λ = restoring torque exerted by the wire in millijoules per radian of twist,

I = moment of inertia of the rod in kg·mm², and

T = period of oscillation in seconds.

The torsion wires shall be calibrated to within ± 3 percent of their specified torsional constants.

5.2 Mounting of Test Piece — Each of the test pieces shall be clamped in such a manner that 25 ± 3 mm of the piece is free between the clamps. The test piece clamp stud shall be located with respect to a reference point on the rack in such a position that the specimen is under zero torque.

5.3 Stiffness Measurements in Liquid Media

5.3.1 The rack containing the test pieces shall be placed in the liquid bath with a minimum of 25 mm of liquid covering the test pieces. The temperature of the bath shall then be adjusted to 27°C. One of the test pieces shall be connected to the torsion head by means of the screw connector and the standard wire.

Caution shall be exercised in attaching the screw connector to the test piece clamp stud to make certain that the stud is not moved from the zero torque position. The torsion head 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.

5.3.2 The pointer reading shall be adjusted to zero by rotating the protractor scale. The torsion head shall then be turned quickly but smoothly through 180° and the pointer reading, after 10 seconds as indicated by the timer, recorded. If the reading at 27°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.

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

5.3.3 The test piece rack shall then be moved to bring the next test piece into position for measurement. All test pieces in the rack shall be measured at 27°C.

5.3.4 The spacers shall be inserted between the test piece rack and the test piece clamp studs. The test pieces shall be removed from the liquid bath and the temperature of the liquid adjusted to the lowest temperature desired (*see* Note 1). The test pieces shall be placed in the bath and allowed to remain at this temperature for approximately 15 minutes. After this, one spacer shall be removed and one test piece measured as was done at 27°C. The spacer shall be returned to its original position after the test piece has been tested (*see* Note 2). All the test pieces in the rack shall be measured in this way, taking care that the measurement of each test piece lasts approximately 2 minutes.

NOTE 1 — This varies with the type of material being tested since time is saved by not starting at a temperature more than 10°C lower than the freezing point of the material; for natural rubber stocks, the lowest temperature required is usually —70°C.

NOTE 2 — 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.

5.3.5 The bath temperature shall then be increased by 5°C increments, each increase being made in approximately 5 minutes and stiffness measurement made after conditioning the test piece for 5 minutes at each temperature. Tests shall be continued until a temperature is reached at which the angular twist is within 5 to 10° of the twist at 27°C.

5.4 Stiffness Measurements in Gaseous Media

5.4.1 Procedure in air or carbon dioxide differs from that 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.

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

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

5.5 Crystallization — When it is desired to study crystallization or plasticiser effects, the time of conditioning at the desired temperature shall be increased.

6. NUMBER OF TESTS

6.1 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.

7. EXPRESSION OF RESULTS

7.1 A plot shall 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 27°C.

The value of the relative modulus for any temperature is readily determined from the angle of twist at that temperature and at 27°C, as given by the twist versus temperature plot, and the ratio of the values of the factor $\frac{180-\alpha}{\alpha}$ corresponding to these angles.

7.2 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 27°C 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.

7.3 When it is desired to calculate the apparent torsional modulus of rigidity in megapascals at various temperatures the free length of the test piece shall be accurately measured and the following formula used:

$$G = \frac{16\,K\,L\,(180 - \alpha)}{a\,b^2\,\mu\,\alpha}$$

where

G = apparent modulus of rigidity in megapascals,

K = torsional constant of wire in millijoules per rad,

L = measured free length of the test specimen in mm,

a = width of test specimen in mm,

b = thickness of test specimen in mm,

μ = factor based on ratio of a/b taken from Table 2, and

α = angle of twist of test specimen in degrees.

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

8. TEST REPORT

8.1 The test report shall include the following particulars:

a) the heat transfer medium used;

b) the temperature 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 megapascal at room temperature;

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

TABLE 1 TWIST ANGLES FOR DESIGNATED VALUES OF THE RELATIVE MODULUS (*RM*)

(*Clause* 7.2)

Twist Angle ∝ in Degrees at 27°C	Twist Angle ∝ in Degrees for Relative Modulus (*RM*)			
	RM=2	*RM*=5	*RM*=10	*RM*=100
(1)	(2)	(3)	(4)	(5)
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

(*Continued*)

TABLE 1 TWIST ANGLES FOR DESIGNATED VALUES OF THE RELATIVE MODULUS (*RM*) — *Contd*

Twist Angle ∢ in Degrees at 27°C	Twist Angle ∢ in Degrees for Relative Modulus (*RM*)			
	RM=2	*RM*=5	*RM*=10	*RM*=100
(1)	(2)	(3)	(4)	(5)
146	123	83	54	7
147	124	85	55	7
148	126	87	57	8
149	127	88	58	8
150	129	91	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	13
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 OF FACTOR μ FOR VARIOUS RATIOS OF a/b

(*Clause* 7.3)

a/b (1)	μ (2)	a/b (1)	μ (2)
1·00	2·249	2·50	3·990
1·05	2·359	2·75	4·111
1·10	2·464	3·00	4·213
1·15	2·563	3·50	4·373
1·20	2·658	4·00	4·493
1·25	2·748	4·50	4·586
1·30	2·833	5·00	4·662
1·35	2·914	6·00	4·773
1·40	2·990	7·00	4·853
1·45	3·063	8·00	4·913
1·50	3·132	9·00	4·960
1·60	3·260	10·00	4·997
1·70	3·375	20·00	5·165
1·75	3·428	50·00	5·226
1·80	3·479	100·00	5·300
1·90	3·573		
2·00	3·659		
2·22	3·842		

(*Continued from page* 2)

Members	*Representing*
SHRI C. S. INAMDAR	Cosmos India Rubber Works Pvt Ltd, Bombay
SHRI SEN GUPTA (*Alternate*)	
SHRI A. GEORGE JOHN	Madras Rubber Factory Ltd, Madras
SHRI G. R. SANKARANARAYANAN (*Alternate*)	
SHRI G. R. KAVISHWAR	Indian Rubber Regenerating Co Ltd, Bombay
SHRI A. R. KANTAK (*Alternate*)	
DR S. P. MANIK	Railway Board (Ministry of Railways)
SHRI G. DORAISWAMY (*Alternate*)	
SHRI V. D. PENDSE	Swastik Rubber Products Ltd, Pune
SHRI K. D. DIGHE (*Alternate*)	
SHRI V. R. RAO	Sundram Industries Pvt Ltd, Madurai
SHRI K. C. MADHUSUDHANAN (*Alternate*)	
SHRI S. SARKAR	Bata India Ltd, Calcutta
SHRI S. ROY (*Alternate*)	
SHRI N. H. SIVARAMAIKRSHNAN	Hindustan Latex Ltd, Trivandrum
SHRI E. V. THOMAS	Rubber Research Institute of India, Kottayam
SHRI A. SALIM VOHRA	Premier Rubber & Cable Industries, Thane
SHRI A. D'COSTA (*Alternate*)	