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SPECIFICATION FOR LATEX FOAM RUBBER PRODUCTS

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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Indian Standard

SPECIFICATION FOR LATEX FOAM RUBBER PRODUCTS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 3 October 1960, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Chemical Division Council.

0.2 The Sectional Committee responsible for the preparation of this standard, after careful consideration of the views of the consumers, producers and technologists, felt that the standard should be related to the prevailing manufacturing and trade practices in the country in this field. Furthermore, due consideration had to be given to the need for international co-ordination among standards prevailing in different countries of the world. These considerations led the Sectional Committee to draw freely upon the Document ISO/TC 45 (Secretariat-327) 471 Draft ISO Proposal for Latex Foam Rubber Components for Furniture, and ASTM Designation D1055-59T Tentative Specifications and Methods of Test for Latex Foam Rubbers.

0.3 This standard requires reference to the following Indian Standard Specifications:

*IS : 264-1950 NITRIC ACID

*IS : 265-1950 HYDROCHLORIC ACID

*IS : 266-1950 SULPHURIC ACID

IS : 915-1958 ONE MARK GRADUATED FLASKS

IS : 1070-1960 WATER, DISTILLED QUALITY (*Revised*)

0.3.1 Wherever a reference to any specification mentioned under **0.3** or otherwise appears in this standard, it shall be taken as a reference to the latest version of the specification.

0.4 Metric system has been adopted in India and all quantities and dimensions appearing in this standard have been given in this system. Non-metric values to which the rubber industry has been accustomed are also given, wherever necessary, for the sake of smooth changeover by December 1966.

*Since revised.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960 Rules for Rounding Off Numerical Values (*Revised*). The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

0.6 This standard is intended chiefly to cover the technical provisions relating to latex foam rubber products, and it does not include all the necessary provisions of a contract.

1. SCOPE

1.1 This standard prescribes general requirement and methods of test for those cellular rubber products known as latex foam rubber products. It does not cover articles made from shredded latex foam, or articles consisting of a cover enclosing loose pieces of latex foam products.

1.2 In the case of conflict between the provisions of this general specification and those of detailed specifications or methods of test for a particular product, the latter shall prevail.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions shall apply.

2.1 Indentation Hardness Index — The indentation hardness index is the load in kilograms required to give an indentation in the sample equivalent to 40 percent of the original thickness of the sample under specified conditions.

2.2 Latex Foam Rubber — A cellular rubber made directly from liquid latex, and in which all the cells are intercommunicating having a vulcanized cellular structure with a porous surface. The cells shall be of a uniform character.

2.3 Lot — All latex foam rubber products of the same type, grade, size and shape drawn from single batch or consignment shall constitute the lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of latex foam rubber products in each batch shall constitute separate lots.

2.4 Sample — That part of the lot which is drawn to represent the lot.

2.5 Skin — The smooth surface of the latex foam rubber products formed by contact with the mould or lid is defined as a natural skin.

2.6 Test Piece — An appropriately shaped piece taken from the sample for use in a physical test.

3. TYPES, SIZE AND SHAPE

3.1 Types — Latex foam rubber product shall be the following types:

a) Cored, and

b) Solid.

3.2 Size and Shape — The size, shape and distribution of the coring shall be as agreed to between the supplier and the purchaser.

4. GRADES

4.1 Unless otherwise agreed to between the purchaser and the manufacturer, the latex foam rubber products shall be graded by its indentation hardness index as follows:

Grade	*Indentation Hardness Index*
A	7 to 14
B	15 „ 21
C	22 „ 28
D	29 „ 34
E	35 „ 45
F	46 „ 55
G	56 „ 65
H	66 „ 75

4.1.1 The indentation hardness index shall be determined by the method prescribed in **A-2**.

5. SAMPLING

5.1 Representative samples of latex foam rubber products shall be taken as agreed to between the purchaser and the supplier.

6. MATERIAL AND WORKMANSHIP

6.1 Latex foam rubber shall be manufactured from foamed rubber latex together with added compounding ingredients of such nature and quality that the finished product complies with the specification requirements. Any special characteristics other than those prescribed in these specifications which may be desired for specific applications shall be specified in the product specifications as they may influence the choice of ingredients used.

6.2 Due to manufacturing conditions, the material may have to be altered or repaired. The repaired or altered material shall be acceptable under the specification provided the material used in such repairs or alterations is of the same composition and quality as the original product and provided such alterations do not affect the serviceability, and size and shape beyond tolerances as provided in this specification.

6.3 Latex foam rubber products should be directly moulded or otherwise shaped or fabricated. When a product is fabricated, all joints should be at least as strong as the foam rubber itself.

7. COLOUR

7.1 The colour of the latex foam rubber products shall be as agreed between the purchaser and the supplier.

8. ODOUR

8.1 The latex foam rubber products shall have no objectionable odour.

9. DIMENSIONS

9.1 Unless otherwise agreed to between the purchaser and the supplier, the dimensions of a latex foam rubber product, when tested according to the method prescribed in **A-3**, shall be as specified by the purchaser subject to the tolerances shown in Table I and Table II.

9.1.1 The actual length and width of a latex foam rubber product should be greater than the nominal dimensions by a small amount in order to admit of the foam rubber being slightly compressed by a cover made to the nominal dimensions. It is advised that this amount should be sufficient to compensate for the negative manufacturing tolerances given in Table I.

10. FLEXING TEST

10.1 When a latex foam rubber product is submitted to the flexing test prescribed in **A-4**, its indentation hardness shall not reduce by more than 20 percent and its thickness by more than 5 percent of the original hardness and thickness respectively.

11. AGEING

11.1 When tested according to the method prescribed in **A-5**, the hardness of the sample after ageing shall not vary by more than ± 20 percent of the value obtained with unaged sample.

TABLE I TOLERANCES ON LENGTH AND WIDTH OF LATEX FOAM PRODUCTS

(*Clauses* 9.1 *and* 9.1.1)

Sl No.	Length or Width	Tolerance	
		± mm	± in
i)	Up to and including 0·2 m (or 8 in)*	3	$\frac{1}{8}$
ii)	Over 0·2 m (or 8 in) up to and including 0·4 m (or 16 in)	6	$\frac{1}{4}$
iii)	Over 0·4 m (or 16 in) up to and including 0·6 m (or 24 in)	9	$\frac{3}{8}$
iv)	Over 0·6 m (or 24 in) up to and including 0·8 m (or 31 in)	13	$\frac{1}{2}$
v)	Over 0·8 m (or 31 in) up to and including 1·0 m (or 39 in)	16	$\frac{5}{8}$
vi)	Over 1·0 m (or 39 in) up to and including 1·2 m (or 47 in)	19	$\frac{3}{4}$
vii)	Over 1·2 m (or 47 in) up to and including 1·4 m (or 55 in)	22	$\frac{7}{8}$
viii)	Over 1·4 m (or 55 in) up to and including 1·6 m (or 63 in)	25	1
ix)	Over 1·6 m (or 63 in) up to and including 1·8 m (or 71 in)	28	$1\frac{1}{8}$
x)	Over 1·8 m (or 71 in) up to and including 2·0 m (or 79 in)	32	$1\frac{1}{4}$
xi)	Over 2·0 m (or 79 in) up to and including 2·2 m (or 87 in)	35	$1\frac{3}{8}$

*For articles which are not moulded but otherwise shaped or fabricated, a minimum tolerance of ± 6 mm (or ± $\frac{1}{4}$ in) is necessary.

12. COMPRESSION SET

12.1 The compression set of the sample when determined by the method prescribed in **A-6.1** shall not exceed 15 percent.

12.2 The compression set of the sample when determined by the method prescribed in **A-6.2** shall not exceed 5 percent of the initial thickness of the sample.

TABLE II TOLERANCES ON THICKNESS OF LATEX FOAM RUBBER PRODUCTS

(*Clause* 9.1)

Sl No.	Thickness	Tolerance in mm	Tolerance in in
i)	Up to and including 25 mm (or 1 in)	$\begin{cases} +3{\cdot}0 \\ -1{\cdot}5 \end{cases}$	$\begin{cases} +\frac{1}{8} \\ -\frac{1}{16} \end{cases}$
ii)	Over 25 mm (or 1 in) up to and including 38 mm (or $1\frac{1}{2}$ in)	±3	$\pm\frac{1}{8}$
iii)	Over 38 mm (or $1\frac{1}{2}$ in) up to and including 76 mm (or 3 in)	$\begin{cases} +5 \\ -3 \end{cases}$	$\begin{cases} +\frac{3}{16} \\ -\frac{1}{8} \end{cases}$
iv)	Over 76 mm (or 3 in) up to and including 127 mm (or 5 in)	±5	$\pm\frac{3}{16}$
v)	Over 127 mm (or 5 in)	±6	$\pm\frac{1}{4}$

13. METALLIC IMPURITIES

13.1 When tested by the method prescribed in **A-7**, the sample shall not contain more than the following:

Copper, percent by weight	0·001
Manganese, percent by weight	0·005

14. PACKING AND MARKING

14.1 The latex foam rubber products shall be packed as agreed to between the purchaser and the supplier, and legibly marked with the manufacturer's name or his trade-mark, if any.

14.2 The latex foam rubber products may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

APPENDIX A

(*Clauses* 4.1.1, 9.1, 10.1, 11.1, 12.1, 12.2 *and* 13.1)

TESTING OF LATEX FOAM RUBBER PRODUCTS

A-1. PREPARATION AND CONDITIONING OF SAMPLES

A-1.1 Wherever practicable the test shall be conducted on the whole latex foam products.

A-1.2 When it is necessary to obtain test pieces from the sample, the method of cutting and the exact position from which test pieces are to be taken shall be specified.

A-1.3 When the finished product does not lend itself to testing or to the preparation of test pieces because of complicated shape, small size or other reasons, standard test slabs shall be prepared.

A-1.4 When differences due to the difficulty in obtaining suitable test pieces form the finished product arise, manufacturer and purchaser may agree on acceptable deviations. This can be done by comparing results of standard test pieces and those obtained on actual product.

A-1.5 Test pieces shall always be cut, so that the surfaces with continuous skin form the main flat surfaces, and cut surfaces form the edges of the test pieces. During this preparation, the skin shall remain undamaged. Cutting shall be carried out so that the cut ends are perpendicular to the main surface, which can be effected by either a very sharp knife or a rotary cutter.

A-1.6 Tests shall be carried out not less than 48 hours after vulcanization and drying. Samples and test pieces shall be protected from light as completely as possible and from any stress or strain wherever they are not actually in the process of being tested.

A-1.7 Aged test pieces or samples shall be tested between 16 & 96 hours after removal from the ageing oven.

A-1.8 Prior to testing, the test pieces shall be conditioned for six hours at a temperature of 27 ± 2°C and a relative humidity of 65 ± 5 percent. This conditioning period may be a part of the 48 hours referred to in **A-1.6**.

A-2. DETERMINATION OF INDENTATION HARDNESS INDEX

A-2.1 Sample—The size of the samples shall be such that a margin of not less than 5 cm shall remain outside the area immediately below the

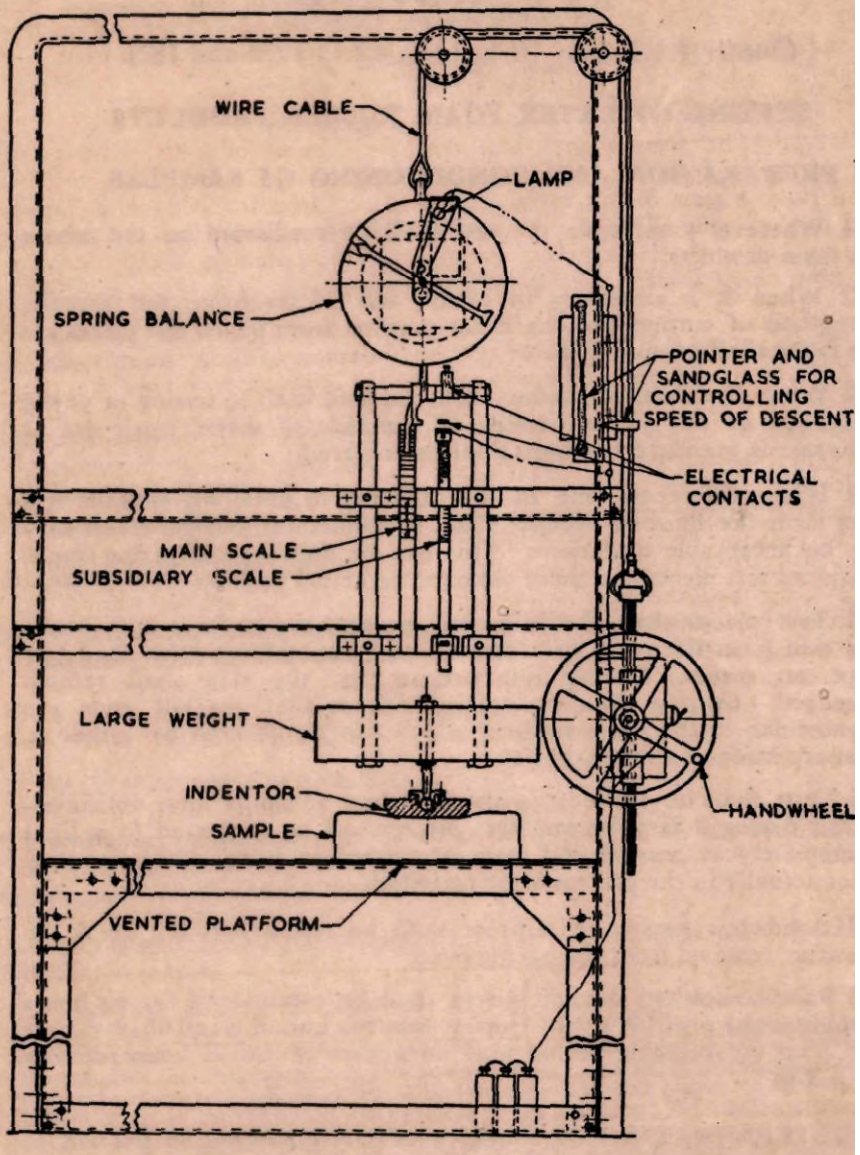


FIG. 1 APPARATUS FOR INDENTATION TEST

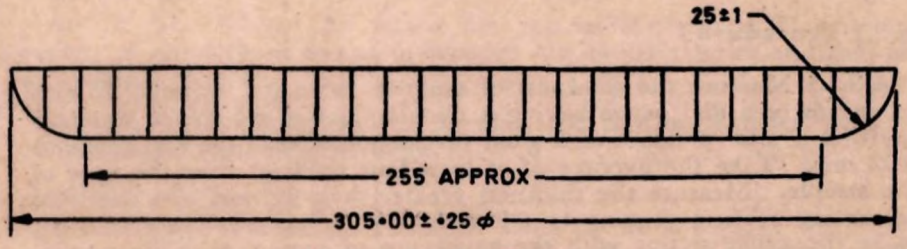
indentor. Sheets less than 20 mm thick should be super-imposed using two or more plies to bring the sample to as near to 25 mm as possible. In making comparative test, similar thickness should always be used.

A-2.2 Apparatus

A-2.2.1 The testing apparatus shall be capable of applying an indentor to the sample at a uniform speed of about 14 mm per second, and shall have means of measuring the load required to produce the specified indentation. The sample shall be supported on a smooth flat rigid surface larger than the sample, and suitably vented to allow escape of air from below the sample.

A-2.2.1.1 The essential parts of one testing apparatus (*see* Fig. 1) which has been found suitable are an indentor of dimensions specified in **A-2.2.2**, a large weight connected to the indentor by a rigid stem and ball joint, and a spring balance from which is suspended the weight and the indentor. The whole system hangs from a rigid framework by a wire cable which passes over two freely rotatable pulleys on the framework and is then attached to a hand-operated bevel worm gear on the lower part of the main framework. The whole loading system consisting of indentor, large weight, rigid stem guide rods and ball joint weighs 100 kg. The dial of the spring balance is calibrated so that when the whole weight of the loading system is taken by the rubber sample the balance reads 100 kg and when wholly taken by the suspending cable, the reading is 0 (zero) kg. The depth of indentation is measured by means of a graduated scale mounted on the main framework between the guide rods. This scale is arranged so that it measures the distance between the base plate on which the test sample is placed and the base of the indentor.

A-2.2.2 *Indentor* — The diameter of the indentor shall be 305·00 ± 0·25 mm with a 25 ± 1 mm radius at the outer edge as shown in Fig. 2.



All dimensions in millimetres.

FIG. 2 INDENTOR

A-2.3 Procedure

A-2.3.1 Carry out the test at 27 ± 2°C.

A-2.3.2 Condition the samples as described in **A-1.8**. Place the sample on an perforated plate with any cavities downwards.

A-2.3.3 Before testing, mechanically condition the test area, that is, the area of the sample which during test will be compressed by the indentor, by applying the indentor with a load at least 50 percent greater than the load which will be required in the test. After release of the mechanical conditioning load, carry out the test within five minutes.

A-2.3.4 After mechanical conditioning, measure the initial thickness of the test area under a load of 2 kg applied by the indentor. Using the apparatus described in **A-2.2**, obtain this initial thickness by lowering the indentor on to the test sample until the spring balance reads 2 kg.

A-2.3.5 Carry out the test by lowering the indentor on to the sample at the specified rate and when the specified degree of compression is reached, read the load.

NOTE — On the apparatus described above, a device is provided for indicating when this degree of compression is reached. This consists of a subsidiary adjustable scale on the side of the main framework of the machine. This subsidiary scale is pre-adjusted to the desired degree of compression and when this is reached a switch operates and brings into action an electromagnetic brake on the pointer of the spring balance. At the same time a small electric bulb is lit near the pointer.

A-2.3.6 Report the indentation hardness index as the load in kilograms applied to produce the specified indentation of 40 percent of the original thickness of the sample.

A-3. MEASUREMENTS OF DIMENSIONS

A-3.1 Procedure

A-3.1.1 Measure the thickness of samples up to and including 25 mm by means of a dial gauge having a circular foot of 6·5 cm^2 in area and exerting a total pressure of 3 g on the sample. Read the dial gauge to 0·02 mm. Take the average of at least four readings over the area of the sample. Measure the thickness greater than 25 mm by a steel rule or vernier calipers reading to 0·2 mm. When calipers are employed, make the gauge setting with the gauge out of contact with the sample. Pass the sample through the previously set gauge and assure proper setting when the measured faces of the gauge contact the surfaces of the sample without compressing it.

A-3.1.2 Measure dimensions other than thickness by means of a steel rule or vernier calipers reading to 0·2 mm. Make each measurement along a line perpendicular to opposing faces of the test sample. Take the average of at least four readings.

A-4. FLEXING TEST

A-4.1 Method—The method involves submitting a sample to a continued flexing with an indentor for 250 000 cycles at 4 cycles per second and measuring the loss in hardness and thickness.

A-4.2 Sample—Where possible, the whole sample shall be used but, if less than this is used, the size of the sample shall be such that a margin of not less than 5 cm shall remain outside the area immediately beneath the indentor.

A-4.2.1 *Samples shall not be tested less than* 48 *hours after having been vulcanized.* They shall be stored for at least 6 hours immediately before testing at a temperature of 27 ± 2°C and 65 ± 5 percent humidity. The test shall also be carried under similar atmospheric conditions.

A-4.3 Apparatus

A-4.3.1 The essential parts of one apparatus (*see* Fig. 3) which has been found suitable consist of an indentor of dimensions specified in **A-4.3.3** connected through a threaded adaptor and held by a locking nut to a push rod. This push rod is constrained to move vertically by fixed sleeves and is driven vertically by means of a motor which rotates a crank disc, the crank disc and push rod being joined up by a connecting rod. This connecting rod is adjustably mounted in a radial slit in the crank disc, the length of the stroke, therefore, being adjustable. The motor is mounted upon a steel beam above the table upon which the articles to be tested are placed, and a revolution counter is attached to the machine to record the number of flexes suffered by an article. The rate of flexing, that is, the number of flexings suffered by an article, is four per second.

A-4.3.2 The table upon which the articles are placed is suitably vented to allow rapid escape of trapped air in the cavities and cells of an article.

A-4.3.3 The indentor shall be circular, 305 mm in diameter, with a 13 mm radius on the bottom edge.

A-4.4 Procedure

A-4.4.1 Place the sample upon the table of the apparatus with any cavities facing the perforated plate on the table and flex that area of the sample which normally carries most load in use.

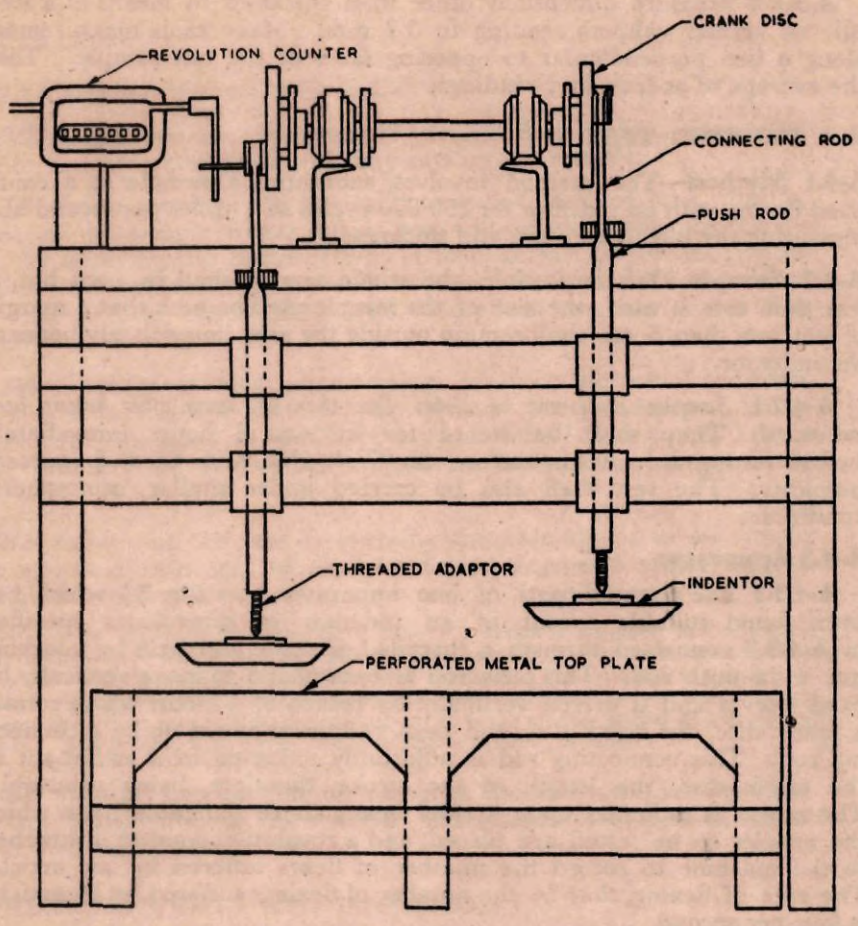


FIG. 3 APPARATUS FOR FLEXING TEST

A-4.4.1.1 Measure before test the height of the sample in the area that is to be flexed and adjust the connecting rod in the crank disc so that, in a complete revolution of the crank disc, the indentor moves a distance equal to 40 percent of the measured height of the sample.

A-4.4.2 Now adjust the adaptor in the push rod, so that at the top of its stroke the indentor just touches the top surface of the sample.

A-4.4.3 Measure the initial indentation hardness index of the sample after flexing the sample for one minute, that is, 240 flexes.

A-4.4.4 Then flex the sample for 250 000 cycles, the sample being constrained by stops from lateral movement during the test.

A-4.4.5 After 250 000 cycles, allow the sample to stand for 30 minutes and then measure any permanent change of thickness of sample and re-determine the indentation hardness index in the tested area.

A-5. AGEING

A-5.1 Method

A-5.1.1 The ageing test consists in subjecting samples to controlled deterioration by air at an elevated temperature and at atmospheric pressure, after which the physical properties are measured and compared with those of unaged samples. The deterioration is measured by the observed change in the physical properties concerned in the service application of the article or it may be determined by the visual examination. No relation between accelerated ageing tests and natural ageing is given or implied.

A-5.1.2 The sample used in any of the ageing tests shall be that required for the particular determination which is to be employed for measuring the effect of the ageing exposure. Only samples of similar dimensions and having approximately the same exposed areas shall be compared with each other. Care should be taken to ensure that the samples have a good smooth finish and are free from blemishes and other flaws.

A-5.2 Procedure

A-5.2.1 Arrange for an air-oven of such a size that the total volume of test pieces does not exceed 10 percent of the free air space in the oven. Make provision for suspending samples so that they are not within 13 mm of each other or the oven sides. Control the temperature of the oven thermostatically so that the test pieces are kept 70 ± 1°C. Place a thermometer near the centre of the samples to record the actual ageing temperature.

A-5.2.2 Place the test pieces in the oven after it has been preheated to the operating temperature. Arrange the test pieces so that they are stationary, free from strain, freely exposed to air on all sides and not exposed to light. Continue the test for 168 hours. Test the unaged test pieces within the same 24-hour period as that in which ageing commences. At the completion of the ageing period, remove the test pieces from the oven and place on a flat surface to cool to room temperature. Allow them to cool for not less than 16 hours nor more than 96 hours before their physical properties are evaluated. Avoid simultaneous ageing of different types of compound in order to safeguard migration of compounding ingredients.

A-6. TEST FOR COMPRESSION SET

A-6.1 Method A

A-6.1.1 *Outline of the Method* — The test consists of maintaining the test piece under specified conditions of time, temperature and constant deflection and noting the effect on the height of the released test piece.

A-6.1.2 *Apparatus*

A-6.1.2.1 *Compression device* — The device shall consist of two flat plates larger in dimensions than the test pieces with spacers and clamp, such that the plates are held parallel to each other and the space between the plates is adjustable to the required deflected height.

A-6.1.2.2 *Dial gauge* — Dial gauge having a circular foot of 6·5 cm^2 in area and exerting a total pressure of 3 g on the test piece. The gauge shall be capable of reading accurately up to 0·1 mm.

A-6.1.2.3 *Oven* — capable of maintaining 70 ± 1°C.

A-6.1.3 *Test Pieces* — The test pieces shall have parallel top and bottom surfaces and essentially vertical sides, free from skin, and of the dimension 5 × 5 × 2·5 cm.

A-6.1.4 *Procedure*

A-6.1.4.1 Carry out tests individually on five test pieces (**A-6.1.3**) as in **A-6.1.4.2**.

A-6.1.4.2 Measure accurately to 0·1 mm initial thickness of test piece using the dial gauge. Place the test piece in the compression device and compress it to 50 percent of its initial thickness between the parallel steel plates. Use the steel spacers between the plates, sufficient clearance being allowed for the bulging of the test piece and care being taken to avoid displacement of test piece. Within 15 minutes, place the compressed test piece in an air-oven, maintained at 70 ± 1°C and keep it there for 22 hours. Remove the test piece from the oven and release it from compression at the earliest. Allow to recover for 30 minutes at room temperature and remeasure the thickness.

A-6.1.5 *Calculation*

A-6.1.5.1 Calculate the compression set as follows:

$$\text{Compression set, percent} = \frac{T_o - T_r}{T_o} \times 100$$

where

T_o = Initial thickness of the test piece in mm, and

T_r = Thickness of the test piece in mm after recovery.

A-6.1.5.2 Report the mean value of compression set for five test pieces.

A-6.2 Method B

A-6.2.1 Measure the original thickness of the sample. Place a circular flat indentor, 305 mm in diameter and weighing 25 kg, on the middle of the sample and allow to remain there for 168 hours at room temperature and humidity. After this period, remove the load and allow the sample to recover for 30 minutes. Measure any permanent depression produced and calculate as follows:

$$\text{Compression set, percent} = \frac{\text{Change in thickness}}{\text{Initial thickness}} \times 100$$

A-7. DETERMINATION OF METALLIC IMPURITIES

A-7.0 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960) shall be employed in tests.

Note — ' Pure chemicals ' shall mean chemicals that do not contain impurities that affect the results of analysis.

A-7.1 Copper

A-7.1.0 *Method*

A-7.1.0.1 No entirely satisfactory method for the determination of normal amounts of copper in natural rubber is available at present. Until such a method is developed, the following procedure is recommended for the determination of copper in concentrations of 18 ppm and over.

A-7.1.0.2 After removal of the organic material by dry ashing in the presence of magnesium oxide, the copper is converted to the diethyldithiocarbamate complex by reacting with zinc diethyldithiocarbamate, and is determined absorptiometrically or by standard series method.

A-7.1.1 *Apparatus* — Either of the following apparatus is required:

a) *Absorptiometer*

b) *Nessler tubes* — Flat-bottomed tubes of thin colourless glass, identical in shape and graduated at 50 ml. The depth measured internally from the graduation mark to the bottom shall not vary by more than 2 mm in the tubes used for the test.

A-7.1.2 *Reagents*

a) *Light magnesium oxide*

b) *Hydrochloric acid-nitric acid mixture* — Mix two volumes of concentrated hydrochloric acid (conforming to IS : 265-1950*), one volume of concentrated nitric acid (conforming to IS : 264-1950*) and three volumes of water.

c) *Standard copper solution* — weigh 0·3928 g of copper sulphate ($CuSO_4, 5H_2O$) into a small beaker and dissolve in water. Add 5 ml of concentrated sulphuric acid (conforming to IS : 266-1950*). Transfer to a 1 000-ml graduated flask (conforming to IS : 915-1958) and dilute with water to standard volume. Pipette 10 ml of this solution into a 100-ml graduated flask (conforming to IS : 915-1958) and dilute to the mark with water. *One millilitre of this solution contains* 0·01 mg of copper (Cu).

d) *Ammonium hydroxide solution* — sp-gr 0·90.

e) *Zinc diethyldithiocarbamate reagent* — Shake one gram of sodium diethyldithiocarbamate and two grams of zinc sulphate dissolved in 100 ml of water with 100 ml of chloroform. Separate and dilute the chloroform extract to one litre with chloroform.

f) *Sodium sulphate* — Anhydrous.

g) *Ammonium citrate solution* — Dissolve 50 g of ammonium citrate in 100 ml of water.

A-7.1.3 *Procedure* — Weigh accurately about 5 g of the sample into a silica crucible containing 0·1 g of light magnesium oxide distributed over the base and partly up the side of the crucible. Support the crucible in a hole cut in an asbestos board so that at least two-thirds of the crucible projects below the asbestos. Heat gently with a small gas flame until a dry carbonaceous residue remains and then transfer the crucible to a muffle furnace to complete the ignition at a temperature not exceeding 550°C. Cool and moisten with 0·5 to 1 ml of water.

Add 10 ml of hydrochloric acid-nitric acid mixture to the moistened ash, cover with a watch glass and heat gently for 20 minutes. Wash the contents of the crucible into a 100-ml conical flask, add 5 ml of ammonium citrate solution followed by ammonium hydroxide added dropwise until the solution is just alkaline to litmus, and then cool. Transfer the solution to a 100-ml separating funnel, add a further 2 ml of ammonium hydroxide and dilute to about 40 ml with water. Immediately measure 25·0 ml of zinc diethyldithiocarbamate reagent into the solution and shake for two minutes. After separation, draw off the

*Since revised.

chloroform layer into a stoppered flask containing 0·1 g of anhydrous sodium sulphate and swirl the contents. If the cloudiness persists after two minutes add further portions of anhydrous sodium sulphate with swirling until the solution becomes clear. Complete the determination by one of the following methods.

A-7.1.3.1 *Absorptiometric method* — Decant the chloroform solution into one-centimetre cell of a photo electric absorptiometer and measure the optical density using a violet filter transmitting between about 3 850Å and 4 700Å. Correct the reading by subtracting the value of the optical density of a blank solution prepared from the same batch of reagents, and from the corrected reading and the calibration curve for the instrument, obtain the concentration of copper in the test solution.

Make up a series of solutions each containing 0·1 g of magnesium oxide dissolved in 10 ml of the hydrochloric acid-nitric acid mixture. Add to each solution 1·0, 2·0, 4·0, 6·0, 8·0 and 10·0 ml respectively of the standard copper solution, then 5 ml of ammonium citrate solution followed by slight excess of ammonium hydroxide. Concurrently with the standard solutions, prepare a blank solution using the same quantities of reagents but with no added copper. Dilute the solutions to 40 ml, extract with 25 ml of zinc diethyldithiocarbamate reagent and measure the optical density of each dried chloroformic extract. Correct the reading for the standard solutions by subtracting the blank and plot the relationship between copper concentration and optical density.

A-7.1.3.2 *Standard series method** — Pipette 20 ml of the chloroform extract into a 50-ml Nessler tube and dilute to 50 ml with chloroform.

Make up a series of solutions each containing 0·1 g of magnesium oxide dissolved in 10 ml of the hydrochloric acid-nitric acid mixture. Add to each solution 1·0, 2·0, 4·0, 6·0, 8·0 and 10·0 ml of the standard copper solution, then 5 ml of ammonium citrate solution followed by a slight excess of ammonium hydroxide. Dilute each solution to about 40 ml, extract with 25 ml of zinc diethyldithiocarbamate reagent, and, after separation, draw off the chloroform layer into a stoppered flask containing 0·1 g of anhydrous sodium sulphate. If cloudiness persists after two minutes with occasional swirling, add further portions of anhydrous sodium sulphate until the solution becomes clear.

Transfer 20 ml of each dried chloroform extract to a 50-ml Nessler tube, dilute to the mark with chloroform, and compare the colour with

*If desired, a method employing a comparator and disc may be used instead of the colour standards.

that of the test solution in order to determine the approximate copper content of the latter.

Then prepare in the same way a second series of standard solutions whose copper contents differ in amounts equivalent to 0·5 ml of the standard copper solution, and which cover a range that includes the test solution. Compare the colour of the test solution with the colours of the second set of standards in order to determine more precisely its copper content.

A-7.2 Manganese

A-7.2.0 *Method* — The manganese in the solution obtained by wet oxidation of the rubber is oxidized to permanganate by means of potassium periodate and the permanganate determined either colorimetrically or photometrically.

A-7.2.1 *Apparatus* — Same as in **A-7.1.1.**

A-7.2.2 *Reagents*

a) *Hydrogen peroxide* — 30 percent (100 volumes).

b) *Dilute sulphuric acid* — 1 : 1 by volume.

c) *Standard manganese solution*—Measure of 50 ml of 0·1 N potassium permanganate solution into a 250-ml beaker, add a few drops of dilute sulphuric acid (1 : 1). Heat to boiling and then add a saturated solution of sulphur dioxide in water, drop by drop, until the solution is just decolourized. Boil for 15 minutes and add 5 ml of dilute sulphuric acid. Cool and transfer the solution to a 500-ml graduated flask (conforming to IS : 915-1958), dilute to the mark with water and mix well. *Call this solution 'X'.* Prepare this solution freshly at least once a month. One millilitre of the solution X contains 0·11 mg of manganese.

Measure 100 ml of solution X into another 500-ml graduated flask, dilute to the mark with water and mix well. *Call this solution 'Y'.* One millilitre of solution Y contains 0·022 mg of manganese. *Solution Y shall be freshly prepared as required.*

d) *Nitric acid*—(conforming to IS : 264-1950*).

e) *Phosphoric acid*—sp gr 1·75.

f) *Potassium periodate*—solid.

g) *Potassium permanganate*—solid.

*Since revised.

h) *Stabilized distilled water*—Assemble a distillation apparatus consisting of a round-bottomed one-litre flask connected through an effcient splash head to a Liebig condenser by means of ground-glass joints. Introduce 500 to 600 ml of ordinary distilled water into the the flask, add about 0·1 g of potassium permanganate previously dissolved in 5 ml of water and a few drops of dilute sulphuric acid (1 : 1), and distil carefully into a clear receiver, rejecting the first 50 ml of distillate and taking care that there is no carry over by splashing or otherwise of liquid from the flask.

j) *Sulphuric acid*—(conforming to IS : 266-1950*).

A-7.2.3 *Prepared Sample Solution*—Weigh accurately 0·2 g of rubber into a 100-ml Kjeldahl flask of resistance glass or silica, and add 10 ml of dilute nitric acid. As soon as vigorous reaction, if any, subsides, heat gently until further vigorous reaction ceases, and then cool. Add slowly 5 ml of concentrated sulphuric acid at such a rate as not to cause excessive frothing or heating, five to ten minutes being usually required, and then heat until the liquid darkens in colour appreciably, that is, begins to char. Then add concentrated nitric acid slowly in small portions to the hot solution, heating between each addition until darkening again takes place. Heating should not be so strong that charring is excessive, and a small amount of free nitric acid shall be present throughout the procedure. Continue this treatment until the solution fails to darken in colour and remains pale yellow on prolonged heating. Note the total amount of nitric acid used. Finally clarify the solutions by boiling with successive additions, few drops at a time, of a mixture of equal volumes of hydrogen peroxide and concentrated nitric acid. Allow to cool slightly and dilute with 10 ml of water. (The solution should be quite colourless or fairly yellow if much iron is present.) Boil down again until white fumes appear, allow to cool, add further 5 ml of water, boil for five minutes and cool. Filter the solution through a good-quality filter paper (Whatman No. 1 or its equivalent) into a 100-ml graduated flask (conforming to IS : 915-1958) washing with small amounts of water, dilute to the mark and mix well. *Call this solution 'A'.*

A-7.2.4 *Blank Solution*—Prepare a blank solution by adding to a 100-ml Kjeldahl flask 5 ml of sulphuric acid and the same amount of nitric acid and hydrogen peroxide as used in the oxidation of the sample in **A-7.2.3** and boiling the mixture down to fuming. Allow to fume for about 20 minutes and evaporate twice with 5-ml portions of water. Finally, cool dilute with 25 ml of water, boil, cool, transfer to a 100-ml graduated flasl (conforming to IS : 915-1958) and dilute to the mark. *Call this solution 'B'.*

*Since revised.

A-7.2.5 *Procedure*—Transfer 50 ml of solution *A* and 50 ml of solution *B* to two separate 100-ml conical flasks, add 2 ml of phosphoric acid and evaporate to fuming. Treat with a few drops of nitric acid and hydrogen peroxide to make sure that all traces of organic matter have been destroyed, and evaporate the solution twice to fuming after adding 10 ml of stabilized distilled water each time. Dilute to 25 ml with stabilized distilled water, add 0·2 g of potassium periodate, boil for one minute and then heat at about 90°C for five minutes. Cool to room temperature, adjust the volume to 25 ml with stabililzed distilled water and mix well. Complete the determinations by one of the following methods.

A-7.2.5.1 *Absorptiometric method*—Prepare a standard series of solutions by measuring 0·0, 0·5, 1·0, 1·5, 2·0, 2·5 and 3·0 ml of standard manganese solution into 100-ml conical flasks, add 4 ml of dilute sulphuric acid and 2 ml of phosphoric acid and evaporate to fuming. Add a few drops of a mixture of equal volumes of nitric acid and hydrogen peroxide, and repeat the procedure as described in **A-7.2.5,** finally diluting to 25 ml with stabilized distilled water in 25-ml graduated flasks (conforming to IS : 915-1958). Measure the optical density of each solution on the absorptiometer using a cell of 4-cm optical depth and suitable green filters, against the first solution containing no added manganese, as reference solution. Plot a graph with parts per million of manganese (or μg Mn, if preferred) as abscissae and optical densities as ordinates. With suitable filters, this graph is a straight line. It may be considered valid for about six months, after which it should be checked.

Similarly, measure the optical density of the test solution against the blank solution as reference solution, and obtain from the curve the manganese content of the sample, corresponding to the observed reading.

A-7.2.5.2 *Standard series method*—Prepare a standard series of solutions by measuring 0·0, 1·0, 2·0, 3·0, 4·0 and 5·0 of standard manganese solution *T*, and repeating the procedure as described in **A-7.2.5.1** and finally diluting to 25 ml with stabilized distilled water.

Transfer the test, blank and standard solutions to Nessler tubes and compare visually in the usual way. Deduct the blank reading from the test reading and calculate, from the number of micrograms of manganese (Mn) present in the standard solution, of manganese content of the sample.