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भारतीय मानक मसौदा

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(IS 12844 का पहला पुनरीक्षण)

Draft Indian Standard

VINYL PYRIDINE LATEX — SPECIFICATION
(*First Revision of IS 12844*)

ICS 83.040.10

Rubber and Rubber Products Sectional
Committee, PCD 13

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FOREWORD

(Formal clauses will be added later)

Vinyl pyridine latex is a terpolymer of butadiene, styrene and 2-vinyl pyridine. Vinyl pyridine latex is used in combination with resorcinol-formaldehyde resins in the formulation of dipping solution for fabric which is ultimately used in the construction of automobile tyres, rubber conveyor belting, V-belts, hoses, etc.

The polarity of vinyl pyridine is responsible for its excellent adhesive properties when used for fibres, particularly synthetic fibres like nylon, rayon, polyester, aramide, fibre glass, etc. to elastomers. In addition, the good mechanical and chemical stability results in a clear and uniform processing in a variety of applications.

This standard was originally published in 1989.

In this revision, the major changes are:

- a) Mooney viscosity range for Type 1 have been modified.
- b) Values of requirements of pH, relative density, viscosity, surface tension and mechanical stability have been modified for the two types.
- c) Requirement of Volatile unsaturates call for agreement between the purchaser and the supplier.
- d) Additionally, the various editorial corrections, and references have been updated.

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 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1 SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for vinyl pyridine latex.

2 REFERENCES

The standards listed in Annex A contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreement based on standard are encouraged to investigate the possibility of applying the most recent edition of these standards.

3 TYPES

3.1 Vinyl pyridine latex shall be of following two types depending upon the values of mooney viscosity (ML_{1+4} at 100 °C) of the contained polymer as indicated against each:

Type 1 — Mooney viscosity 30 to 45

Type 2 — Mooney viscosity 46 to 65

4 REQUIREMENTS

4.1 Physico-Chemical Requirements

The material shall conform to the requirements given in Table 1.

References to the relevant method and Annexes are given in col (5) and (6) of Table 1.

4.2 Performance Requirement

Both the types of vinyl pyridine latex, when used as a part of dipping solution for tyre cord, shall provide adhesion strength of tyre cord to vulcanized rubber as agreed to between the purchaser and the supplier when tested as per the H or T Test procedure given in Annex E of this standard.

5 PACKING AND MARKING

5.1 Packing

The latex shall be packed in epoxy coated mild steel or high density polyethylene drums or as agreed to between the purchaser and the supplier.

5.2 Marking

The containers shall be marked with the following:

- a) Name of the manufacturer or trade-mark, if any;
- b) Net, tare and gross mass in kg; and
- c) Month and year of manufacture and lot number.

6 SCALE OF SAMPLING AND CRITERIA FOR CONFORMITY

6.1 The method of drawing representative sample of the material and criteria for conformity is given in Annex F.

Table 1 Requirements for Vinyl Pyridine Latex
(Clause 4.1)

Sl. No.	Characteristics	Requirements		Methods of test reference to	
		Type 1	Type 2	Annex	IS
(1)	(2)	(3)		(4)	(5)
i)	Total solids content, percent by mass	40 to 42		—	IS 9316 (Part 4)
ii)	pH	10 to 12		—	IS 9316 (Part 6)
iii)	Relative Density at 25°C	0.980 to 0.990		—	IS 4511 (Part 2)
iv)	Viscosity, mPa.s	20 to 60		—	IS 9316 (Part 2)
v)	Volatile unsaturates, percent by mass, <i>Max</i>	As agreed between purchaser and supplier.		—	IS 4511 (Part 3)
vi)	Vinyl pyridine content of the contained polymer, percent by mass	15 ± 1		B	
vii)	Surface tension, mN/m	49 to 55		—	IS 9316 (Part 1)
viii)	Coagulum content, percent by mass, <i>Max</i>	0.05		—	IS 9316 (Part 3)
ix)	Mechanically stability, coagulum percent by mass, <i>Max</i>	0.065		—	IS 4511 (Part 6) (SBRL:11)
x)	Mooney viscosity of the contained polymer, ML ₁₊₄ at 100 °C	30 to 45	46 to 65	C	—
xi)	Particle size, A°	800 to 1000		D	—

(Clause 2)

<i>IS No.</i>	<i>Title</i>
IS 266 : 1993	Sulphuric acid — Specification (<i>third revision</i>)
IS 517 : 2020	Specification for methanol (Methyl alcohol) (<i>third revision</i>)
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 3660 (Part 7) :2013	Methods of test for natural rubber : Part 7 Determination of Mooney Viscosity
IS 3708 (Part 8): 2018 / ISO 1656 : 2014	Methods of test for natural rubber latex : Part 8 Rubber, raw natural and rubber latex, natural — Determination of nitrogen content (<i>third revision</i>)
IS 4511 (Part 2): 1986	Methods of test for styrene butadiene rubber (SBR) latices : Part 2 Determination of density [SBRL: 6] (<i>first revision</i>)
IS 4511 (Part 3):1987	Methods of test for styrene butadiene rubber (SBR) latices : Part 3 Determination of volatile unsaturates [SBRL : 8] (<i>first revision</i>)
IS 4511 (Part 6):1987	Methods of test for styrene butadiene rubber (SBR) latices : Part 6 Determination of high speed mechanical stability (SBRL:11)
IS 4905 : 2015 / ISO 24153 : 2009	Random Sampling and Randomization Procedures (<i>first revision</i>)
IS 9316 (Part 1) : 2023 / ISO 1409 : 2020	Methods of test for rubber latex : Part 1 Determination of surface tension (<i>second revision</i>)
IS 9316 (Part 2) : 1987	Methods of test for rubber latex : Part 2 Determination of viscosity (RL: 2) (<i>first revision</i>)
IS 9316 (Part 3) : 1987	Methods of test for rubber latex : Part 3 Determination of coagulum content (sieve residue) [RL : 3] (<i>first revision</i>)
IS 9316 (Part 4) : 2023 / ISO 124 : 2014	Methods of Test for Rubber Latex : Part 4 Determination of Total Solids Content (RL:4) (<i>second revision</i>)
IS 9316 (Part 5) : 2013 / ISO 123 : 2001	Methods of test for rubber latex : Part 5 Drawing of samples [RL: 5] (<i>second revision</i>)
IS 9316 (Part 6) : 2017 / ISO 976 : 2013	Methods of test for rubber latex : Part 6 Determination of pH (RL : 6) (<i>second revision</i>)
IS 1745 : 2018	Petroleum hydrocarbon solvents — Specification (<i>third revision</i>)

ANNEX B

[Table 1, Item (vi)]

DETERMINATION OF VINYL PYRIDINE CONTENT

B-1 GENERAL

B-1.1 Three methods are prescribed for determination of vinyl pyridine content. Method **B-2** is Kjeldahl procedure, method **B-3** which is an alternate to **B-2**, prescribes auto nitrogen analyser procedure and method **B-4** is a semi micro Kjeldahl procedure. Method **B-4** shall be the referee method.

B-2 KJELDAHL METHOD

B-2.1 Outline of the Method

B-2.1.1 The nitrogen content and vinyl pyridine content is estimated using Kjeldahl apparatus.

B-2 .2 Apparatus

B-2.2.1 *Kjeldahl Reflux Flasks*, 100 ml capacity

B-2.2.2 *Kjeldahl Distillation Assembly*

B-2.2.3 *Heating Mantles*, 100 ml capacity rated at 120 W, or Bunsen burners

B-2.3 Reagents

B-2.3.1 *Concentrated Sulphuric acid*, conforming to IS 266

B-2.3.2 *Sodium Hydroxide Solution*, 20 percent of sodium hydroxide in distilled water

B-2.3.3 *Sodium Thiosulphate Penta Hydrate Solution*, 25 percent solution of sodium thiosulphate in distilled water (carbon dioxide free)

B-2.3.4 *Standard Hydrochloric acid*, 0.04 N

B-2.3.5 *Mixed Indicator Solution*, prepare stock solutions of 0.1 percent bromocresol green and 0.1 percent methyl red in 95 percent alcohol or methanol. Mix 5 volume parts of bromocresol green with 1 volume part of methyl red.

B-2.3.6 *Boric Acid Solution*, dissolve 4 g boric acid in 100 ml distilled water.

B-2.3.7 *Catalyst Mixture*, prepare a finely divided and intimate mixture of the following:

Potassium sulphate anhydrous 30 parts; copper sulphate penta hydrate 4 parts; selenium 1 part or sodium selenate 2 parts.

B-2.4 Procedure

B-2.4.1 Sample Preparation

Recover the polymer from the vinyl pyridine latex by coagulating it with isopropanol. Dice the coagulated polymer in small pieces and extract repeatedly with isopropanol at ambient temperature in order to completely remove the non-polymeric ingredients like organic acid, soap and antioxidant, etc. Dry the extracted sample between 100 °C and 125 °C, avoiding under-drying and avoiding over heating for more than 5 min after reaching minimum mass.

B-2.4.1.1 Weigh accurately 50 mg to 70 mg of the dried polymer into a clean Kjeldahl flask. Add approximately 0.65 g of the catalyst mixture and 4 ml concentrated sulphuric acid.

B-2.4.2 Keep the flask alongside a blank (where all other ingredients are in same mass except the sample are taken) in the heating mantles and reflux for six hours.

B-2.4.3 Allow the flasks to cool to laboratory temperature and stopper them. Assemble Kjeldahl apparatus and steam out a Kjeldahl distillation assembly for a period of 10 min. Allow the assembly to cool by cutting off the steam and remove the condensate in distillation cup. Dissolve the contents of the Kjeldahl reflux flasks in 5 ml portions of distilled water and transfer quantitatively the solution into the distillation cup of the assembly through the funnel of the assembly. Ensure that the dissolution of the contents take place with the first 5 ml portions of distilled water, so that the remaining three portions are completely utilised for rinsing the flask. Measure out 35 ml of sodium hydroxide solution into a clean 50 ml cylinder and transfer into the flask in three or four portions for further rinsings of the flask, glass rod, and funnel of the distillation assembly. Add 5 ml of sodium thiosulphate solution measured in a clean 10 ml cylinder into the distillation cup. Close the stopper of the funnel and fill it nearly to half volume with distilled water.

B-2.4.4 Pipette 25 ml of boric acid solution into a clean dry 250 ml conical flask bearing mark at 150 ml level. Pipette also 25 ml distilled water into it and keep it underneath the condenser of the distillation assembly with the tip of the condenser atleast 0.5 cm below the surface.

B-2.4.5 Pass steam and collect the condensate until the volume reaches the level of the mark made. Remove the flask from below the condenser and allow some more drops of water to fall into the flask, while holding the tip of the condenser above the liquid level in the conical flask.

B-2.4.6 Add 0.5 ml of the mixed indicator solution to the distillate by means of a graduated pipette and titrate the solution against standard hydrochloric acid solution taken in the burette with 0.02 ml graduations, till the blue colour changes into grey with faint pinkish tinge. Note down volume of the acid added.

B-2.4.7 In parallel with the determination, carry out a blank test using the same quantities of reagents under the same operating conditions, but omitting the test portion.

B-2.5 Calculations

B-2.5.1 Calculate the vinyl pyridine content from the following formula:

$$\text{Vinyl pyridine content, percent by mass} = \frac{10.5 \times (V_2 - V_1) \times N}{m}$$

where

V_2 = volume in ml of standard hydrochloric acid required for titration;

V_1 = volume in ml of standard hydrochloric acid required for the titration in the blank test;

N = normality of standard hydrochloric acid; and

m = mass in g of the test portion.

NOTE — In all the stages of the method, any contamination especially with nitrogenous materials organic or inorganic shall be scrupulously avoided. In no case nitric acid should be employed for cleaning any part of the apparatus.

B-3 AUTO-ANALYSER PROCEDURE USING DUMA'S METHOD

B-3.1 Outline of the Method

B-3.1.1 The isopropanol extracted polymer of vinyl pyridine latex is burnt in the presence of copper oxide. The oxides of nitrogen produced during combustion are reduced to nitrogen by passing them over heated copper and swept by a carrier gas (carbon dioxide) in the nitrometer (containing 50 percent potassium hydroxide solution) where the volume of nitrogen is measured. Vinyl pyridine content is calculated from the volume of nitrogen produced and the mass of the sample taken.

B-3.2 Apparatus

B-3.2.1 *Nitrogen Analyser*

B-3.2.2 *Pure Carbon dioxide gas Source*, purity minimum 99.99 percent

B-3.2.3 *Micro-balance*, capable of weighing up to one microgram

B-3.2.4 *Vacuum Oven*

B-3.2.5 *Aluminium Boat*

B-3.2.6 *Stainless Steel Forcey*

B-3.2.7 *Micro Nitrometer*, capable of measuring the volume to the nearest of 0.001 ml.

B-3.2.8 *Combustion Tube*, preferably quartz combustion tube, 50 cm in length and 7 mm inside diameter having 1 mm to 2 mm capillary opening 1.5 cm to 2.0 cm in length.

B-3.3 Reagents

B-3.3.1 *Copper Oxide Reagent*

B-3.3.2 *Cupric Oxide*, Fines

B-3.3.3 *Copper Reagent*

B-3.3.4 *Potassium Hydroxide-Reagent*, 50 percent (w/w) solution of potassium hydroxide.

B-3.3.5 *Standard Sample of Known Nitrogen Content*

B-3.3.6 *Mercury*

B-3.4 Procedure

B-3.4.1 *Charging of Combustion Tube*

Combustion tube is permanently charged. From the capillary end charge a short wad of asbestos, 9 cm copper oxide fines, a short wad of asbestos, 4 cm of freshly activated copper and a wad of asbestos and 9 cm of cupric oxide fines. Then hold this in place with a wad of asbestos. Preheat the charged tube in a current of carbon dioxide before use.

B-3.4.2 *Nitrometer*

Deliver mercury into the nitrometer until the same covers the gas inlet of the potassium hydroxide chamber. Pour potassium hydroxide reagent in the nitrometer until the liquid level reaches the calibration mark.

B-3.4.3 *Sample Preparation*

Recover the polymer from the vinyl pyridine latex by coagulating it with isopropanol. Dice the coagulated polymer in small pieces and extract repeatedly with isopropanol at ambient temperature in order to completely remove the non-polymeric ingredients like organic acid, soap and antioxidant, etc. Dry the extracted sample between 100 °C and 125 °C, avoiding under-drying and avoiding over heating for more than 5 min. after reaching minimum mass.

B-3.4.4 *Determination of blank*

Blank is defined as the volume of unabsorbed gas which appears in the nitrometer as a result of a completed combustion cycle which originates from sources other than the sample. It is determined by completing a combustion cycle using the same amount of reagents in the aluminium boat (without sample) and cycle adjustment as shall be followed for the sample. The volume in the nitrometer thus obtained shall be taken as blank. Take atleast three constant readings of the blank.

B-3.4.5 *Sample placement*

B-3.4.5.1 Weigh accurately about 10 mg of the dry polymer to the nearest 0.1 mg in an aluminium boat and place an amount of copper oxide reagent approximately equal to about twice the sample mass over the sample in the boat. Fill the combustion tube to about two-thirds with copper oxide reagent. Slide the aluminium boat into the combustion tube with the help of forcep without spilling the contents. Fill the remainder of the combustion tube with copper oxide reagent approximately 2 cm below the end.

B-3.4.5.2 Place the combustion tube into the instrument holders and eliminate the air by passing through the system, about 100 ml of carbon dioxide, while cold.

B-3.4.5.3 At the end of the above operation which requires about 1 min., place nitrometer in its position in the instrument.

B-3.4.5.4 Adjust the carbon dioxide gas flow rate so as to maintain 31 kPa (4.5 psi) pressure in the gas regulator and 5 cc/ min gas flow rate in the flow meter. Check and ensure that there is no leak in the system.

B-3.4.5.5 Adjust the meniscus of the potassium hydroxide solution in the nitrometer to the calibration mark and start the combustion cycle by switching on heating of the furnaces to a temperature of 700 °C to 750 °C.

B-3.4.5.6 Note the initial and the final readings of the nitrometer to determine the volume of nitrogen formed during the combustion cycle.

B-3.4.5.7 Record the temperature and the pressure of the nitrogen produced.

B-3.5 Calculations

B-3.5.1 Calculate the vinyl pyridine content in the polymer as follows:

$$\text{Vinyl pyridine content, percent by mass} = \frac{P_c}{T} \times \frac{V_c}{M} \times 0.04493 \times \frac{105}{14}$$

where

V_c = corrected volume of nitrogen evolved in microliter (volume of the unabsorbed gas during the sample run - volume of the unabsorbed gas during the blank)

P_c = corrected barometric pressure [$P_o - (P_v + P_t)$]

where

P_o = barometric pressure in mm Hg,

P_v = vapour pressure of potassium hydroxide solution, and

P_t = barometric temperature correction,

T = temperature of the unabsorbed gas in K, and

M = mass of the sample in mg.

NOTES

1 Make necessary corrections for any ingredients present in the sample other than polymer in the vinyl pyridine content.

2 Always run a standard sample before analyzing the rubber sample in order to ensure that the instrument is working properly.

B-4 SEMI-MICRO KJELDAHL METHOD

B-4.1 Outline of the Method

The latex is coagulated, dried, extracted to remove non-polymeric material. A known mass of the extracted polymer is decomposed by digestion with concentrated sulphuric acid, in presence of a catalyst mixture thereby converting nitrogen compounds into ammonium hydrogen sulphate from which ammonia is distilled after making the mixture alkaline. The distilled ammonia is absorbed in standard sulphuric acid, and the excess acid is titrated with standard sodium hydroxide. From the titre value, equivalence of ammonia is calculated from the known mass of the sample. On the

basis of ammonia content, equivalent amount of vinyl pyridine content is calculated and is expressed as a percentage on the total terpolymer.

B-4.2 Apparatus

B-4.2.1 *Airoven*, capable of being maintained at 150°C.

B-4.2.2 *Soxhlet Extraction Apparatus*

B-4.2.3 *Heating Mantles*

B-4.2.4 *Wire Sieve*, non-corrodible (180 mesh to 200 mesh).

B-4.2.5 *Semi-Micro Kjeldhal Digestion Apparatus*, with digestion flash of capacity 30 ml [*see* Fig. 1, 2 and 3 of IS 3708 (Part 8)].

B-4.2.6 *Semi-Micro Kjeldahl Distillation Apparatus*, with a condenser tube made of borosilicate glass, pure tin or silver [*see* Fig. 4 to 9 of IS 3708 (Part 8)].

B-4.2.7 Burette, 5 ml, calibrated at every 0.02 ml. A burette with storage bottle and automatic zero setting is suitable.

B-4.3 Reagents

B-4.3.1 *Calcium chloride Solution*, 10 percent in distilled water.

B-4.3.2 *Solvent Mixture*

Prepare a solvent mixture by mixing 70 parts by volume of methanol with 30 parts by volume of toluene.

B-4.3.3 *Boric acid Solution*, dissolve 40 g of boric acid in water warming, if necessary, and make up the volume to two litres. Add 20 ml of indicator solution and mix well.

B-4.3.4 *Catalyst Mixture*

Prepare a finely-divided and intimate mixture of the following:

Potassium sulphate anhydrous 30 parts; copper sulphate pentahydrate 4 parts; selenium powder 1 part of sodium selenate decahydrate ($\text{Na}_2\text{SeO}_4 \cdot 10\text{H}_2\text{O}$) 5 parts.

B-4.3.5 *Mixed Indicator Solution*, 0.1 g of methyl red and 0.05 g of methylene blue in 100 ml of ethanol.

NOTE — This indicator may deteriorate on storage and in such cases shall be freshly prepared.

B-4.3.6 *Standard Sodium hydroxide Solution*, 0.02 N, carbonate free

B-4.3.7 *Concentrated Sodium hydroxide Solution*, 56 g in 100 ml of water

B-4.3.8 *Concentrated Sulphuric acid*, conforming to IS 266.

B-4.3.9 *Standard Sulphuric acid*, 0.02 N.

B-4.4 Procedure

B-4.4.1 Sample Preparation

Recover the polymer from the vinyl pyridine latex by coagulating it with isopropanol. Dice the coagulated polymer in small pieces and extract repeatedly with isopropanol at ambient temperature in order to completely remove the non-polymeric ingredients like organic acid, soap and antioxidant, etc. Dry the extracted sample between 100 °C and 125°C, avoiding underdrying and avoiding beating for more than 5 min after reaching minimum mass.

B-4.4.2 Determination of Vinyl Pyridine Content

Weigh accurately 50 mg to 70 mg of the extracted and dried coagulum and transfer into a clean Kjeldahl flask. Add about 0.65 g of catalyst mixture and 3.0 ml of concentrated sulphuric acid and determine the vinyl pyridine content following semi-micro Kjeldahl method as given in IS 3708 (Part 8).

ANNEX C

[Table 1, Item (x)]

DETERMINATION OF MOONEY VISCOSITY

C-1 OUTLINE OF THE METHOD

C-1.1 The latex is coagulated by the consecutive addition of salt and acid solutions while it is under fast agitation. The resulting crumb is filtered, washed, dried, and messed on a rubber mill. The viscosity of the contained polymer is measured in a shearing disk viscometer.

C-2 QUALITY OF REAGENTS

C-2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the result of analysis.

C-3 APPARATUS

C-3.1 Mooney Viscometer

C-3.2 Curing Press, electrically/steam heated curing press, (143 ± 15) °C.

C-3.3 Roll Mill, 15 cm × 30 cm odd speed laboratory smooth roll mill.

C-3.4 Blendor (Explosion-Proof Type)

C-3.5 Stainless Steel or Plastic Screen, 50 mesh.

C-3.6 Aluminium Foil, 0.25 mm to 0.40 mm thick.

C-3.7 Beaker, 500 ml capacity.

C-3.8 Stainless Steel Spatula or Glass Stirring Rod

C-3.9 Graduated Cylinder, 25 ml capacity.

C-4 REAGENTS

C-4.1 Methanol, conforming to IS 517.

C-4.2 Butylidene Bis (6-Tert Butyl) *m*-Cresol

C-4.3 Sodium chloride Solution, 20 percent (*m/v*)

C-4.4 Sulphuric acid, sp gr 1.84 (*see* IS 266).

C-5 PROCEDURE

C-5.1 Measure 250 ml of vinyl pyridine latex at 40 percent to 43 percent solids content into a 500 ml glass beaker. Cream the latex by slowly adding 50 ml of stock 20 percent sodium chloride solution while stirring continuously with a glass rod or spatula.

NOTE — Prepare the sodium chloride solution by dissolving 200 g of commercial sodium chloride in 800 ml of distilled water.

C-5.2 Place 250 ml of stock antioxidant solution in the blender. Place the cover on the blender and start the agitator. Slowly add the creamed latex to the blender over a period of one minute.

NOTES

1 Prepare the antioxidant solution by dissolving 7.5 g antioxidant powder in 1000 ml of methanol.

2 Care shall be taken to avoid splashing of the solution into the face and eyes.

C-5.3 Measure 7 ml of stock 18 percent sulphuric acid solution into a 25 ml graduated cylinder containing 18 ml of distilled water. Pour this acid solution slowly into the blender to complete the latex coagulation.

NOTE — Prepare the sulphuric acid solution by adding 100 ml of concentrated sulphuric acid in 900 ml of distilled water.

C-5.4 Allow the blender to run for 1 min to 2 min after the acid solution has been added, then strain the serum from the crumb by pouring (0.297 mm) through a 50 mesh screen. Mass the crumb and squeeze by hand to remove excess serum. Do not wash the crumb. Mass the crumb into a sheet or flat cake and place into the press between clean sheets of aluminum foil. Close the press until the sample is (1 cm/0.5 cm) thick. Dry the sample between 100 °C and 125 °C, avoiding under-drying and over-heating.

NOTE — Recommended drying condition for preparing the dry polymer for determination of Mooney viscosity of Type 1 latex are 125 °C for one hour.

C-5.5 Remove the sample from the press, strip off the aluminum foil and examine the sample for wet spots. Trim away wet spots before milling the sample. Pass the sample once through the

laboratory mill, cold mill with cooling water on both rolls, and (2 ± 0.1) mm opening. Fold and pass the sample twice through the mill without folding. Do not allow the sample to bend on the mill and do not make more than 10 passes. Keep the sample for about 1 h to 2 h before determining the Mooney viscosity. Determine the Mooney viscosity of the sample as given in IS 3660 (Part 7).

ANNEX D

[Table 1, Item (xi)]

DETERMINATION OF PARTICLE SIZE OF LATEX

D-1 OUTLINE OF THE METHOD

D-1.1 Particle size of latex is determined by turbidity measurements in the visible region in a UV/Visible Spectrophotometer. Turbidity of latex is proportional to the particle size in the absorption range of 0.07 to 0.1 at 700 nm.

D-2 APPARATUS

D-2.1 UV/Visible Spectrophotometer

D-2.2 80 ASTM mesh screen (or equivalent IS Sieve)

D-2.3 volumetric flasks, 100 ml

D-2.4 Balance

D-3 PROCEDURE

D-3.1 Strain latex through 80 mesh stainless screen and determine its total solids content. Prepare a one percent stock solution of a suitable dispersant (preferably sulphonate type) in distilled water. Take about 25 ml of stock solution of dispersant into a 100 ml volumetric flask and weigh accurately. Transfer about one milliliter of strained latex directly into the dispersant solution in the above flask (avoiding the sticking of latex on the walls) in order to prevent formation of skin or agglomerates. Weigh the contents of flask and determine the mass of the sample accurate to one milligram. Dilute to the mark with the stock solution of dispersant and mix thoroughly. Pipette 5 ml aliquot of the above latex solution into another clean 100 ml volumetric flask and dilute to the mark with the stock solution of dispersant. Stopper the flask and mix thoroughly.

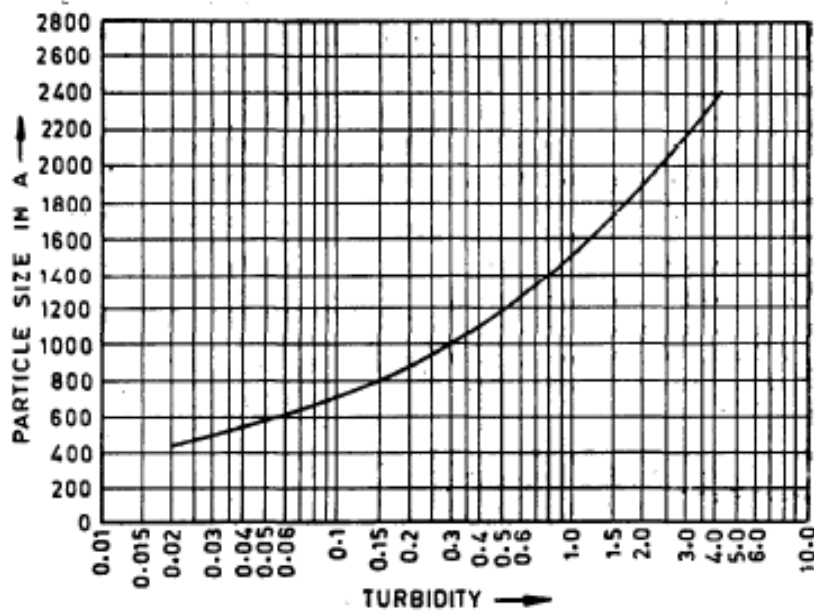
Determine absorbance at 700 nm using stock solution of dispersant as blank. If absorbance does not fall between 0.07 to 0.10, different dilutions must be tried.

D-4 CALCULATION

$$\text{Turbidity} = \frac{\text{Absorbance}}{g/1000}$$

$$= \frac{\text{Absorbance} \times 1000}{(\text{sample mass}) \times \frac{(\text{Percentage total solids})}{100} \times (\text{Volume of aliquot})}$$

D-4.1 Read particle size of latex in \AA from the graph of turbidity versus particle size using Fig. 1.



Scale of the graph on y-axis one small square = 40\AA $\lambda = 7000 \text{\AA}$

FIG. 1 FOR LOW SOLIDS LATTICES (BELOW 50 PERCENT TOTAL SOLIDS)

ANNEX E

(Clause 4.2)

STATIC ADHESION OF TEXTILE TYRE CORD TO VULCANIZED RUBBER

E-1 GENERAL

E-1.1 Two methods namely 'Method A : H-test' and 'Method B : T-Test' are prescribed in this Annex for determination of static adhesion of dipped cord to rubber.

E-2 METHOD A — H-TEST

E-2.1 Principle

Assessment of the adhesion between a rubber and textile cord is made by measuring the force required to pull a single cord from a block of cured rubber, the force being applied along with longitudinal axis of the cords and the length of cord embedded in the rubber being fixed (*see* Fig. 2). The adhesion measured is essentially a shearing force acting at the cord-to-rubber interface. The two strips of rubber and the interconnecting cord form a test piece resembling the letter 'H' from which the test derives its name.

E-2.2 Apparatus

E-2.2.1 A Suitable Mould

The dimension of the test pieces are controlled by the specifications and tolerances of the mould. The test pieces are prepared by laying strips of rubber, of thickness $Y/2$ (*see* Fig. 2, spaced Z apart, into cavities in a mould of width C . Cords are stretched over and perpendicular to the rubber strips, with a distance L between each cord. Two further strips of rubber are applied above the cord, the mould closed, put into a press, and the test pieces, vulcanized. It is common practice to use moulds which allow many identical test pieces to be produced simultaneously.

E-2.2.1.1 One example of a suitable mould is shown in Fig. 3. It is recommended that the width of the cord groove be 0.8 mm for cords of linear density 560 mg/m (tex) or less, and 1.2 mm for cords of linear density more than 560 and up to 800 mg/m (tex). Although this form of mould is simple to use, the moulding pressure tends to force excess rubber down the cord groove between the rubber strips, particularly when the cord is much narrower than the groove. This flash shall be removed from the cord by careful cutting before test to improve the reproducibility of results. The formation of this rubber flash can be almost completely eliminated by using a mould of the form shown in Fig. 4. The technique requires the cord length between the rubber strips to be held in position during cure by a deformable surface, rather than a groove, so that there are no voids into which excess rubber can flow. Two methods are show in Fig. 4. In method A, the cord between the rubber strips R_1 and R_2 and between R_3 and R_4 is held between specially prepared silicon-rubber-faced bars. A suitable method for the preparation of such bars in described in **E-3**. In method B the upper strip of the rubber is made sufficiently wide to cover the whole distance R_5 to R_6 (and R_7 to R_8) with the addition of a thin cellophane or polyester strip applied to the central portion of the rubber, which contracts the cords, to prevent the adherence of rubber to the cord in this region.

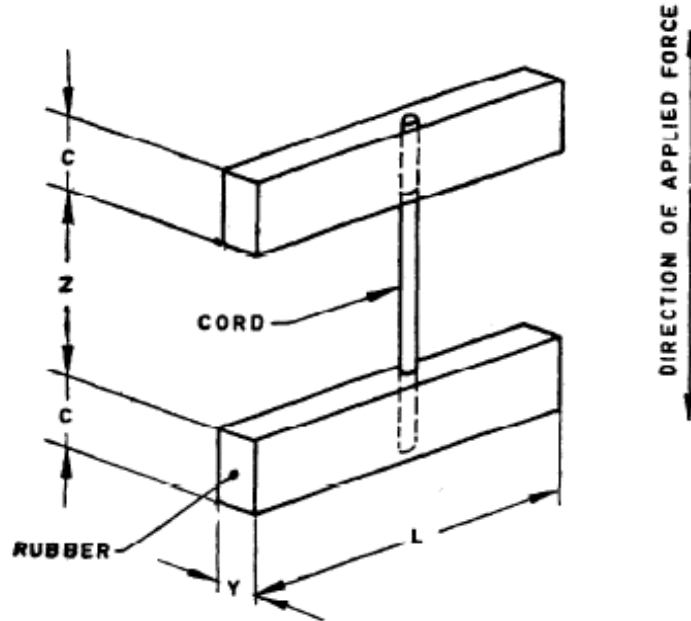
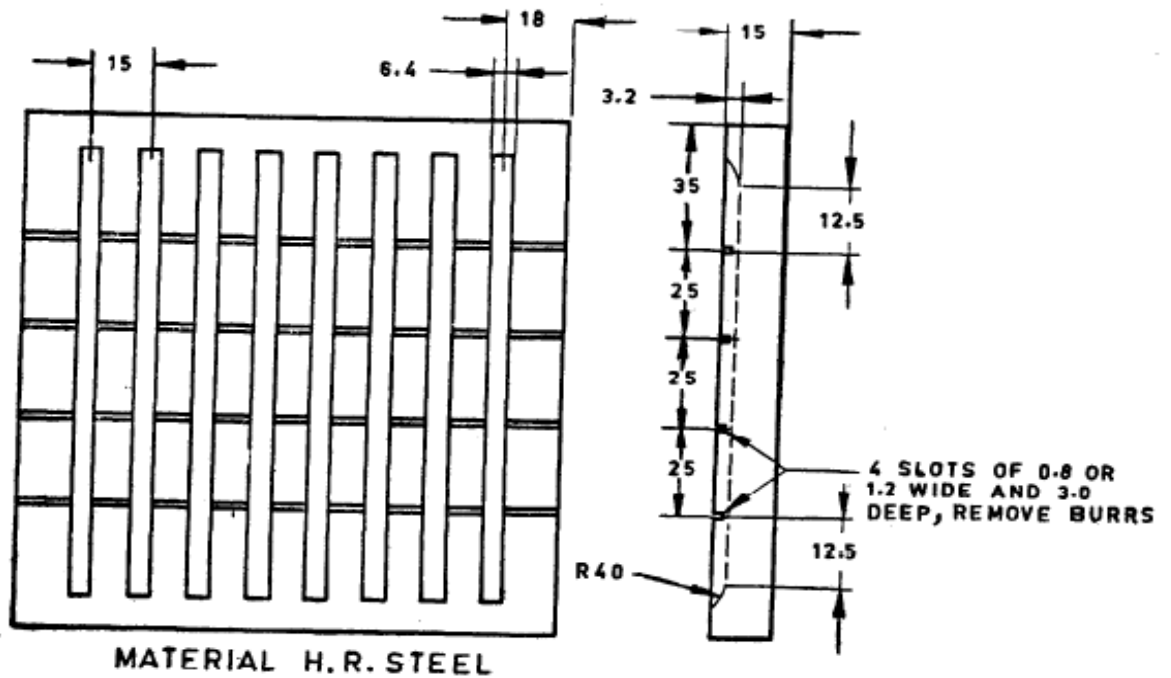


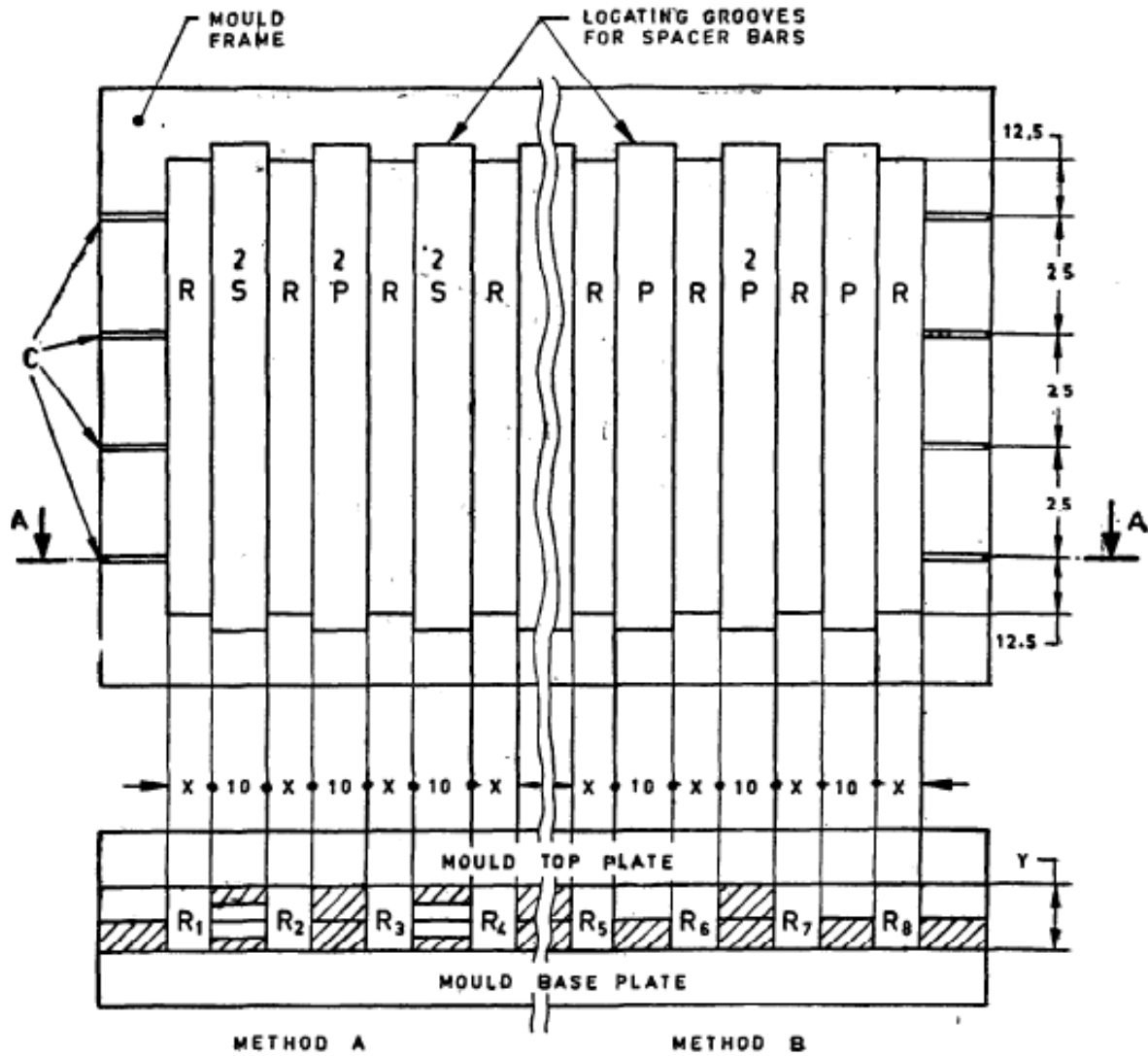
FIG. 2 TEST PIECE



NOTE — The mould as shown will produce 16 test pieces. It may be fabricated to produce a larger or smaller number but the dimensions that govern the size of the test pieces shall not be altered.

All dimensions in millimeters

FIG.3 SUITABLE MOULD FOR H-PULL TEST



SECTION AA

- R — Rubber cavity, width X and depth Y
- S — Silicon rubber faced bar
- P — Plain space bar
- C — Cord grooves, width 0.8 or 1.2

All dimensions in millimeters

FIG.4 METHODS OF PREPARATION OF TEST PIECES

E.2.2.2 A Suitable Device to Provide a Tensioning Force of $(0.49 \pm 0.1) N$.

NOTE —This may be achieved, for example, by suspending a mass of $(50 \pm 1) g$ on one end of each cord during assembly of the test piece and removing it prior to placing the mould into the curing press. The masses

may be of the hook type or designed in such a manner that they can be clamped to the cord. In any event, the total mass shall be (50 ± 1) g.

E-2.2.3 Testing Machine

It should be capable of accurately registering the applied forces during the test, while maintaining the specified constant rate of separation of the jaws, at (100 ± 10) mm/min.

NOTE — Inertia (Pendulum) type dynamometers are apt to give results which differ because of frictional and inertial effects. A low inertia type of dynamometer with a suitable recorder gives results which are free from these effects and should therefore, be preferred,

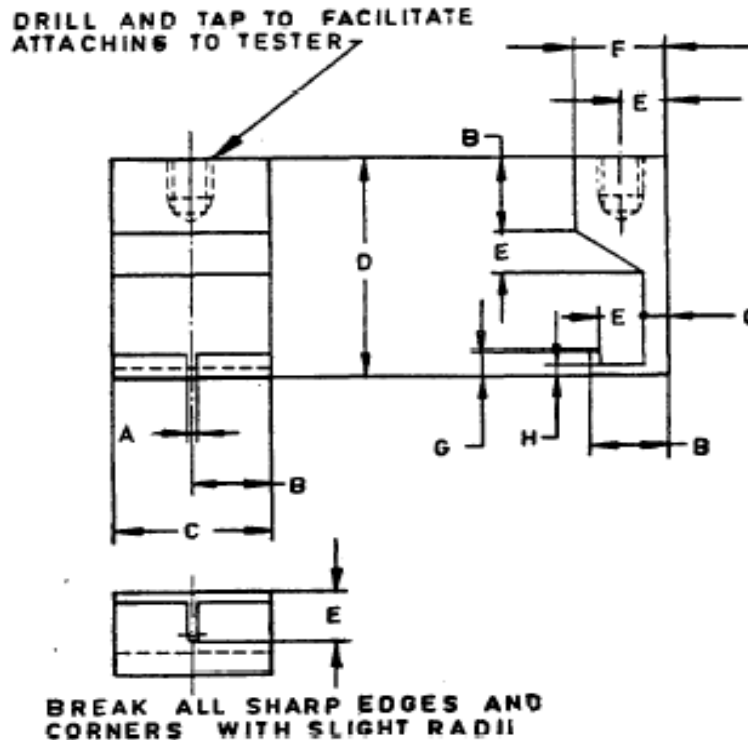
E-2.2.4 Test Piece Grips

The design of the test piece grips shall be as shown in Fig. 5 or Fig. 6. Two grips are required.

NOTE — The two types of grips do not necessarily give the same results.

E-2.3 Material

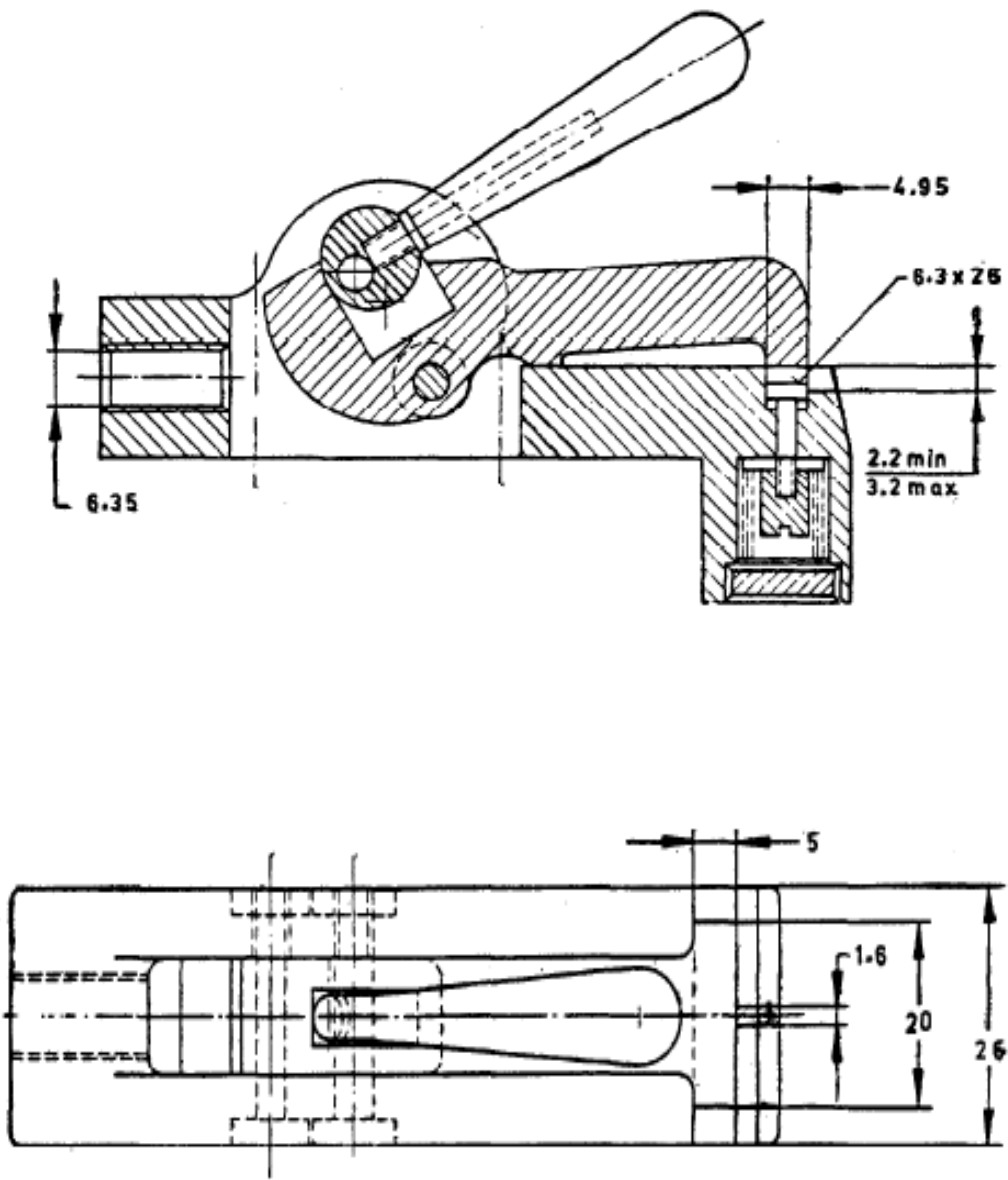
E-2.3.1 The materials comprise any combination of rubber compound, textile cord and dip solution agreed upon by both the cord user and supplier. The vulcanizing conditions, both time and temperature shall be exactly specified. The thickness of the rubber compound required to fill the mould completely shall be determined by the supplier and the purchaser.



NOTE — The important dimension is indicated by an asterisk and shall not be altered. All other dimensions are included for guidance and may be altered if desired.

Dimension	mm
A	1.6
B	12.5
C*	25.0
D	40.0
E	7.0
F	14.0
G	4.0
H	2.0

FIG.5 TEST PIECE GRIP



NOTE — The lower part of the grip is spring loader with a spring tension of between 5 N and 15 N to minimize deformation of the rubber.

All dimensions in millimeters
FIG.6 ALTERNATE TEST PIECE GRIP

NOTES

1 The decision as to which rubber compound to use is normally made by the cord user.

2 Wherever possible, the unvulcanized rubber compound shall be freshly milled. If for any reason the rubber cannot be remilled, the surface shall be freshened by wiping with heptane or SBP spirit 55/115, conforming to IS 1745. The compound shall be stored at laboratory temperature prior to use. It may be in the form of calendered sheet of suitable thickness and should be protected by a dark coloured polyethylene film.

E-2.3.2 Cotton Backing Fabric

Square-woven approximately 340 g/m² cotton fabrics or its equivalent, shall be used to support the rubber strips. This may be grey fabric or fabric that has been fractioned on one side. Alternatively, the rubber compound may be calendered to the frictioned side of the cotton fabric. The rubber surface which will be in contact with the cords shall be protected by a protective film, for example, starch paper or polyethylene.

E-2.4 Test Piece

E-2.4.1 Dimensions, The standard test piece shall be a length of cord embedded in rubber strips, nominally 6.4 mm wide and 3.2 mm thick (*see E-2.2.1*).

NOTE – Although this method specifies that the rubber strips shall be 3.2 mm thick an inter laboratory test have equivalent values for 3.2 mm and 6.4 mm thick test pieces. The embedded length of cord may be reduced to 5 mm or increased to 10 mm where the adhesion is very high or very low respectively, but the results obtained using different embedded lengths are not comparable.

E-2.4.2 Preparation of Test Pieces

E-2.4.2.1 Cut the rubber compound into strips, 6 mm wide and of a suitable length leaving the protective film attached. This may be done with scissors or with a clicker die.

E-2.4.2.2 Cut strips of cotton fabrics to the same dimensions as those of the rubber compound. If the rubber compound is calendered on to the fabric, eliminate this step.

E-2.4.2.3 If necessary, place the bottom spacer bars in the mould (Fig. 4 type mould).

E-2.4.2.4 Using the mould at room temperature, place the fabric strips in bottom of the mould cavities (*see E-2.4.2.2*).

E-2.4.2.5 Place the rubber strips in the mould cavities with the protective film side on top. If the rubber is calendered on to the fabric, the fabric side shall be on the bottom.

E-2.4.2.6 Remove the protective film from the rubber strips and immediately place the cords in the cord slots. The portion of the cord to be embedded in the rubber shall not be touched by the bare hand. The procedure for handling calendered cords shall be agreed upon by the purchaser and

the supplier. Knot each cord at one end so that it is secured firmly against the cord slot on one side of the mould. Take care to prevent the loss of cord twist. Attach a tensioning device on the other end of the cord.

E-2.4.2.7 If required, place the upper spacer bars in the mould.

E-2.4.2.8 Remove the protective film from additional strips of rubber and place them in the mould cavities on top of the cords. The side from which the protective film was removed shall be down. When preparing test pieces by Method B, these additional strips of rubber shall be 22 mm wide, with a strip of protective film or similar material replaced over the central 10 mm wide area.

E-2.4.2.9 Place strips of fabrics on top of the rubber strips. If the rubber is calendered on to the fabric, eliminate this step.

E-2.4.2.10 Identify the test piece in the mould, and cover the mould with a smooth metal plate if the upper press plate is not smooth.

E-2.4.2.11 Remove the tensioning devices from the cords and place the moulds in a press which has been preheated to the vulcanizing temperature. Adjust the pressure to minimum of 3.5 MPa with a reference to the mould surface. After vulcanizing for the specified time, immediately remove the test pad from the mould and cool at room temperature.

NOTE — A preheated mould may be used, but this will alter the vulcanizing conditions (time and temperature) of the rubber. If a preheated mould is used, the materials should exhibit sufficient green tackiness to permit the pad to be prefabricated in a cold mould and then transferred to the preheated mould.

E-2.4.2.12 Cut the pad using scissors, a sharp knife, or clicker die to produce 'H' pieces consisting of a single cord with each end embedded in the centre of a rubber tap approximately 25 mm in length. If necessary, trim off all excess rubber flash. When using method B, the trimming should be carried out with great care to avoid cutting into the test piece.

E-2.4.3 *Number of Test Pieces*

E-2.4.3.1 At least 8 test pieces shall be used.

E-2.4.4 *Time Interval Between Vulcanizing and Testing*

E-2.4.4.1 Unless otherwise specified for technical reasons, the minimum time between vulcanization and testing shall be 16 h. The maximum time between vulcanization and testing shall be four weeks, and for evaluations intended to be comparable, the test shall, as far as possible, be carried out after the same time interval.

E-2.5 Procedure

E-2.5.1 The force required to separate the cord from the rubber may be determined at room temperature or at an elevated temperature.

E-2.5.1.1 *Testing at room temperature*

Attach the test piece grips to the tension tester and set them 1 mm apart, take care to ensure axial alignment. Adjust the speed of the movable grip to (100 ± 10) mm/min. Insert the test piece in the grips and start the tester. Record to the nearest 1.0 N, the maximum force required to separate the cord from the rubber.

E-2.5.1.2 *Testing at elevated temperature*

Proceed as described in **E-2.5.1.1** but enclose the test piece grips in an oven attached to the tester. Heat the test pieces in the oven, controlled at the test temperature, for not less than 15 min and not more than 60 min total elapsed time for testing of any one test piece. Alternatively, heat the test pieces in an oven adjacent to the testing machine and then remove them, one at a time, and test within 15 s of removal. The technique for heating and testing the test pieces shall be agreed upon by the purchaser and the supplier.

E-2.5.2 *Expression of Results*

E-2.5.2.1 Record the cord adhesion value in newton and calculate the median of the results.

E-2.5.2.2 Describe the appearance of the cord indicating whether the rubber has remained adhered to it or not.

E-3 METHOD B : T-TEST

E-3.1 Principle

E-3.1.1 Assessment of the adhesion between a rubber and textile cord is made by measuring the force required to pull a single cord from a block of cured rubber. The test piece resembles the letter T, from which the test derives its name.

E-3.2 Apparatus

E-3.2.1 *A Suitable Mould*, as shown in Fig. 7.

E-3.2.2 *Testing Machine*, as described in **E-2.2.3**.

E-3.2.3 *Test Piece Grip*

The design of the test piece grip shall be as shown in Fig. 8.

E-3.3 Materials, same as in **E-2.3**.

E-3.4 Test Piece

E-3.4.1 *Dimensions*

The standard test piece shall be a length of cord embedded in rubber strips nominally 10 mm wide and 10 mm thick.

E-3.4.2 *Preparation of Test Specimens*

E-3.4.2.1 Fasten one end of the dipped cord sample to one end of the metal frame and wind the cord back and forth across the cord slots under a tensioning force of (0.49 ± 0.1) N. Wear gloves

during this operation to prevent contamination of the cord. Fasten the dip cord to the diagonally opposite end of the metal plate and cut off excess length of the dipped cord sample.

E-3.4.2.2 Cut several slabs of unvulcanized rubber, each 200 mm × 125 mm × 1.7 mm, and two sheets of cotton backing fabric, each 175 mm × 125 mm. Remove polyethylene backing film from one of the unvulcanized rubber slabs, and place one piece of cotton backing fabric on it, leaving a 25 mm strip uncovered at one edge. Repeat the procedure with the second slab of unvulcanized rubber.

E-3.4.2.3 Place the assembled slabs with a cotton backing fabric adjacent to the cord wound on the frame with the uncovered 25 mm strips touching each other. Place additional unvulcanized rubber stock (after removing the backing polythene) on both faces of the sandwich to fill the mould properly.

E-3.4.2.4 Assemble the top, frame and bottom plates, taking care to see that there is no looseness between them. Place the loaded mould between the platens of the vulcanizing press, the platens having previously been brought to the correct temperature. Adjust the pressure to a minimum of 3.5 MPa with reference to the mould surface. After curing for the specified time at the specified temperature, remove the mould immediately and cool at room temperature.

E-3.4.2.5 With the help of a precision cutter, cut through the rubber in a line perpendicular to the inner edge of the cotton backing fabrics, without damaging the cords. Repeat the operation from the other side. Cut the block of rubber left with the cords attached with it, into a 10 mm strip. The test specimen should have clean edges.

E-3.4.3 *Time Interval Between Vulcanization and Testing*

E-3.4.3.1 Same as in **E-2.4.4**.

E-3.5 Procedure

E-3.5.1 Set the tensile testing machine to rate of traverse of (100 ± 10) mm/min.

E-3.5.2 Secure the test piece grip in one jaw of the tensile testing machine. Insert the cord layer through slit 2.68 mm wide in the base of the strip and hold in the other jaw. Take care to ensure axial alignment.

E-3.5.3 Start the machine and let the cord be pulled out of the rubber stock. Record, to the nearest 1.0 N, the maximum force required to separate the cord from the rubber.

E-3.5.4 Test at least 8 specimens.

E-3.5.5 *Expression of Results*

E-3.5.5.1 Record the cord adhesion value, in newton, and calculate the median of the results.

E-3.5.5.2 Describe the appearance of the cord indicating whether the rubber has remained adhered to it or not.

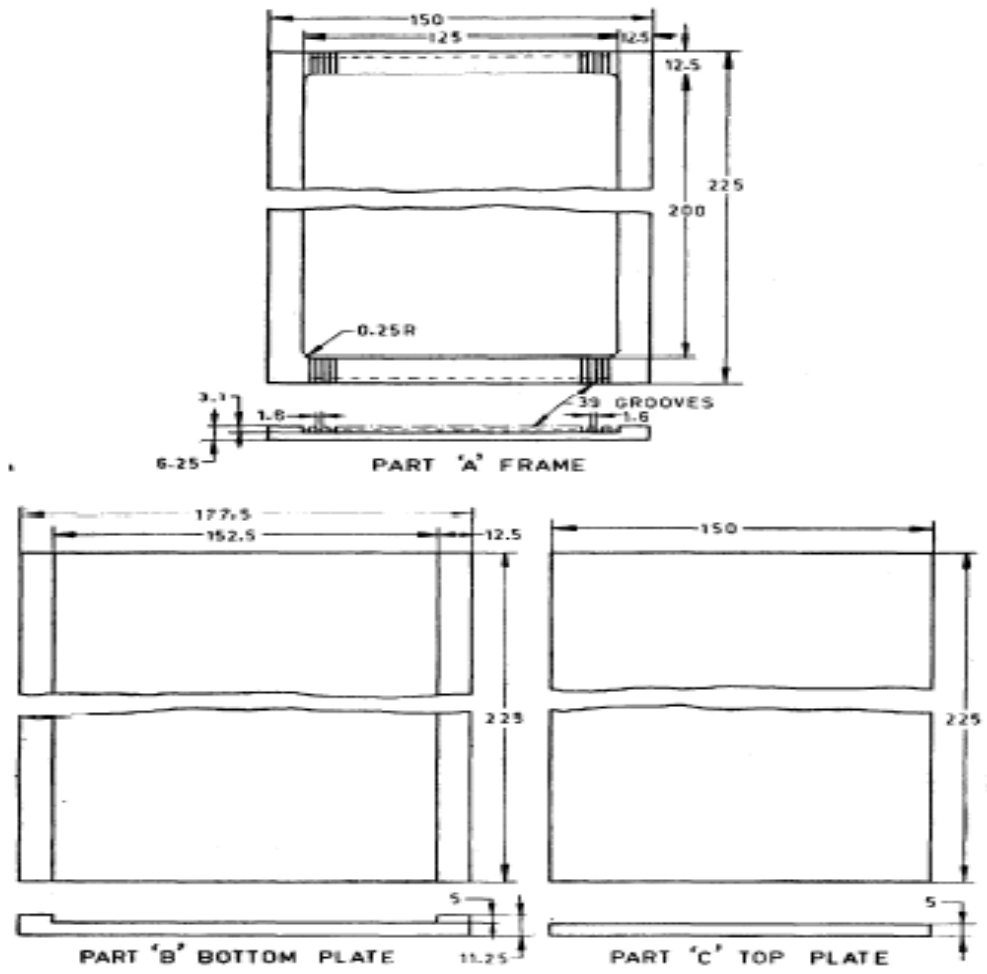
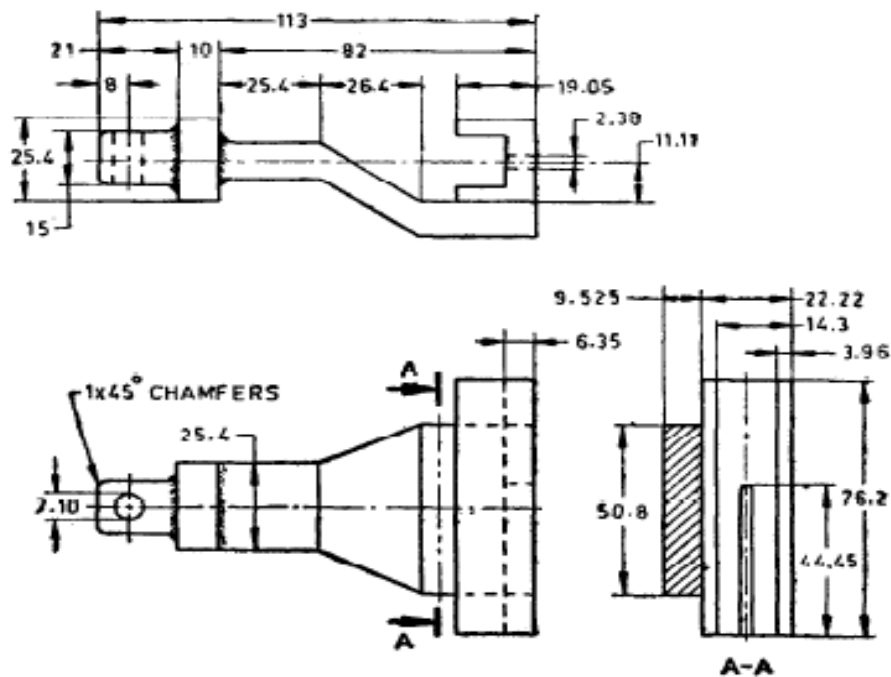


FIG. 7 Mold



All dimensions in millimetres.

FIG. 8 TEST PIECE GRIP

E-4 PREPARATION OF SILICON RUBBER FACED BARS (See E-2.2.1.1)

E-4.1 Silicon Compound Preparation

E-4.1.1 The compound used shall be a self-bonding grade of silicon rubber of hardness about 60 IRHD.

E-4.1.2 Depending on the mould plate size available, press out between sheets of polyester a 50 g to 60 g piece of the compound, using two flat mould plates to obtain as even a thickness (1.5 mm) as possible.

NOTE — The pressing is best done in a hydraulic press at very low pressure (less than 175 kPa). A hand-pump press is preferable. The platens may be heated to 50 °C to 70 °C, if preferred.

E-4.1.3 After pressing for 2 min to 3 min, examine the silicon rubber to see if the required thickness has been reached. It may not be possible to attain 1.5 mm, but up to 2 mm is permissible.

E-4.1.4 Remove the sheet of silicon rubber and store it on a flat cool surface, still protected by the polyester film.

E-4.2 Bar Preparation

E-4.2.1 Remove as much old silicon rubber as possible by scraping, mechanical wire brushing of the bars, or similar means.

E-4.2.2 Degrease the bars in a vapour degreasing bath, using trichloroethylene or perchloroethylene for 30 min to 60 min, but it is advisable to protect the non-bonding surfaces to prevent unnecessary roughening.

E-4.2.3 Abrade the bars on the bonding surfaces only using fine emery cloth. Light shot or vapour blasting may be used, but it is advisable to protect the non-bonding surfaces to prevent unnecessary roughening.

E-4.2.4 Finally clean the bars by wiping liberally with a clean cloth soaked in a petroleum solvent, the solvent being allowed to evaporate. Apply the silicon strip as soon as possible after cleaning.

E-4.3 Bonding Procedure

E-4.3.1 Cut strips from the polyester film protected silicon rubber to fit the bonding areas on the bars. If the silicon rubber is unavoidably thick, the width of the strips (normally about 10 mm) may be reduced by 1 mm to 2 mm to prevent excessive spew.

E-4.3.2 Peel the polyester film from one face of the strip, place the exposed face on the freshly cleaned bonding area and manually, lightly press into contact. Avoid contact with the exposed silicon surface and the prepared bar surface to prevent contamination and hence poor bonding.

E-4.3.3 Prepare two bars at a time. These should be used as a pair, and preferably marked for easy identification.

E-4.3.4 Place the bars in suitably mould side by side, with the silicon faces uppermost. The top layers of polyester film may now be removed if preferred, but to facilitate easy demoulding, a piece of polyester film, sufficient to cover the mould cavity, should be inserted. Alternatively, the mould lid should be sprayed with a PTFE aerosol mould lubricant.

E-4.3.5 With the lid located by the matching holes and pegs of the bars, place the moulds in a press while the platens are warming up, and use a low pressure to spread the silicon. When the full curing temperature of 160°C is reached, apply the higher possible safe pressure and vulcanize for 15 min.

E-4.3.6 Cool the bars, preferably in the moulds in the press still under pressure. If this is not possible, allow the complete mould to cool out of the press, as at this stage the silicon is still weak and prone to damage when hot.

E-4.3.7 When cool, carefully remove the faced bars from the mould. This may be facilitated by removing one of the screwed mould and stops allowing a thin lever to insert under the bars.

E-4.3.8 Excess silicon spew etc., may then be trimmed out, or this may be deferred until after the oven cure.

E-4.3.9 When all the bars to be refaced have been, press cured, store them for 18 h to 24 h at laboratory temperature. Remove excess spew, etc., if this was not carried out at the previous stage.

NOTES

1 When in use the silicon rubber may protrude beyond the edge of the bar. If a trial pull-through test piece is moulded, the amount of excess silicon may be measured by examining the test piece cross section, which

will be of H-section if the silicon protrudes. The edge of the silicon should be trimmed back from the edge of each bar by slightly less than the depth of the indentation caused in the pull-through test piece.

2 With normal usage, a minimum of 500 test mouldings can be expected before the bars need to be refaced.

ANNEX F

(Clause 5.1)

SCALE OF SAMPLING AND CRITERIA FOR CONFORMITY

F-1 TANK SUPPLIES

F-1.1 When the material is supplied in tanks or other bulk containers, each tank or bulk container shall be sampled separately.

F-1.2 A representative sample shall be drawn from each tank of bulk container according to 6 of IS 9316 (Part 5).

F-1.3 The sample shall be tested for all the requirements given in 4.2 and Table 1. The material in tank or bulk container shall be considered as conforming to this specification if the corresponding representative sample satisfy all the requirements given in the specification.

F-2 DRUM SUPPLIES

F-2.1 Lot

All the drums, in a single consignment, of the same size, same type and belong to same batch of manufacture shall constitute a lot.

F-2.2 For ascertaining the conformity of material to the requirements of this specification samples shall be tested from each lot separately.

F-2.3 The number of drums (n) to be selected for sampling shall depend on size of the lot (N) and shall be in accordance with col 1 (2) and (3) of Table 2.

Table 2 Scale of Sampling

Sl. no.	Lot Size	No. of Drums to be Selected
(1)	(N) (2)	(n) (3)
i)	Up to 25	3
ii)	26 to 50	5
iii)	51 to 100	7
iv)	101 and above	10

F-2.4 These drums shall be chosen at random from the lot. In order to ensure the randomness of selection, reference may be made to IS 4905. In case this standard is not readily available, following procedure may be adopted:

Starting from any drum in the lot, count them as 1, 2, 3,..... up to r and so on in one order, where r is the integral part of N/n . Every r^{th} drum thus counted shall be withdrawn to constitute the required sample size.

F-3 METHOD FOR TAKING SAMPLES FROM SELECTED DRUMS

F-3.1 From each of the drums selected according to **F-2.3**, a representative sample shall be drawn in accordance with the procedure prescribed in 5 of IS 9316 (Part 5).

F-4 TEST SAMPLE AND REFEREE SAMPLE

F-4.1 From the samples (**F-3.1**) representing different drums in the lot a small but approximately equal quantity of material shall be taken and thoroughly mixed to form a composite sample, not less than 600 g. The composite sample so obtained shall be divided into three equal parts, one for the purchaser, another for the supplier and third for the referee.

F-4.2 The composite sample shall be transferred to separate container. The container shall then be sealed air-tight with stoppers and marked with full details of sampling, the date of sampling, year of manufacture, batch or code number and other important particulars of the consignment.

F-4.3 The referee sample consisting of a composite sample shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. This shall be used in case of any dispute between the two.

F-5 NUMBER OF TESTS

F-5.1 Tests for all the characteristics as prescribed in this standard shall be carried out on the composite sample.

F-6 CRITERIA FOR CONFORMITY

F-6.1 The lot shall be declared as conforming to the requirements of the specification if all the test results on the composite sample meet the corresponding specification requirements.