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मानक

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IS 101-5-2 (1988): Methods of sampling and test for paints, varnishes and related products, Part 5: Mechanical test on paint films, Section 2: Flexibility and adhesion [CHD 20: Paints, Varnishes and Related Products]

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## Indian Standard

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METHODS OF SAMPLING AND TEST FOR PAINTS, VARNISHES AND RELATED PRODUCTS

## PART 5 MECHANICAL TESTS

Section 2 Flexibility and Adhesion

## (Third Revision)

- 1. Scope Prescribes the following four tests for determination of flexibility and adhesion:
  - a) Bend test,
  - b) Scratch hardness test,
  - c) Cupping test, and
  - d) Pull off test.

#### 2. Bend Test

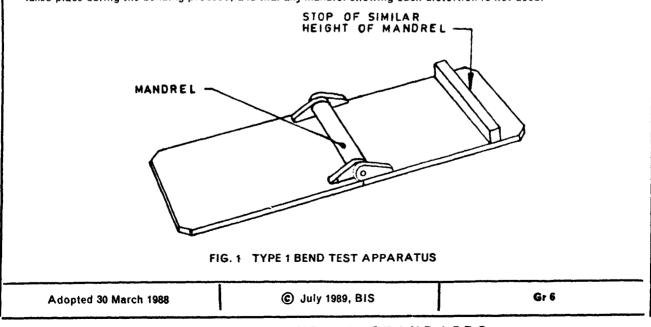
**2.1** Outline of the Method — This is an empirical test for assessing the resistance of a coating of paint, varnish, etc, to cracking and/or detachment from a metal substrate when subjected to bending round a cylindrical mandrel.

#### 2.2 Apparatus

**2.2.1** Material – In both types of apparatus described below, the mandrels shall be made of a rigid and suitably corrosion resistant material, such as stainless steel.

**2.2.2** Type 1 apparatus — A suitable apparatus is shown in Fig. 1 and 2 and is used with test panels of thickness not greater than 0.3 mm. A set of hinges is provided, each incorporating a cylindrical mandrel; the diameters of the mandrels are 2, 3, 4, 5, 6, 8, 10, 12, 16, 20, 25 and 32 mm, respectively. The dimensions of the apparatus are not critical except that the gap between the surface of the mandrel and the plates of the hinge should be  $0.55 \pm 0.05$  mm. The mandrel should be free to rotate on its pin and the apparatus shall be provided with a stop to ensure that when the test panel is bent, the two portions are parallel.

**Note** — Especially with the 2 mm diameter mandrel, it is important to ensure that no distortion of the mandrel takes place during the bending process, and that any mandrel showing such distortion is not used.





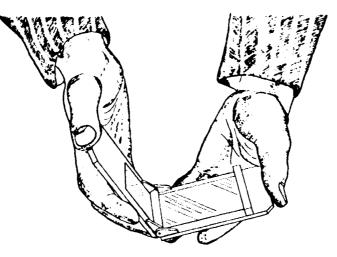


FIG. 2 TYPE 1 BEND TEST APPARATUS IN USE

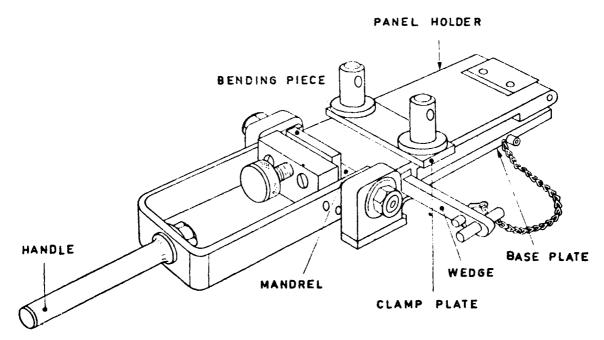
**2.2.3** Type 2 apparatus — A suitable apparatus is shown in Fig. 3 and 4 and is normally used with test panels of thickness up to 1.0 mm. With soft metals, such as, aluminium, thicker panels may be used provided there is no distortion of the mandrel. The apparatus incorporates one of a series of cylindrical mandrels of diameters 6, 10 and 13 mm.

Note — By agreement between the parties, other diameters of mandrel may be used with the Type 2 apparatus.

**2.2.4** Controlled temperature chamber — This is required for tests which are specified to be carried out at temperatures other than  $27 \pm 2^{\circ}$ C. It consists of an oven or refrigerator in which the temperature of the working chamber can be controlled to within  $\pm 1^{\circ}$ C of the required test temperature, and which is fitted with the following ancillary equipment:

- a) A circulation fan in the working chamber;
- b) A suitable remote-control device by which the panel can be bent without opening the test chamber; and
- c) A temperature indicating or recording device located with its bulb, or sensitive element, in close proximity to the coating under test.

**2.3** Test Panels — They shall be of burnished steel, burnished tinplate or soft aluminium. The panels shall be flat and free from distortion and the surface shall be free from any visible ridges or cracks.





#### AMENDMENT NO. 1 JANUARY 1998 TO

## IS 101(PART 5/SEC 2) : 1988 METHODS OF SAMPLING AND TEST FOR PAINTS, VARNISHES AND RELATED PRODUCTS

#### PART 5 MECHANICAL TESTS

#### Section 2 Flexibility and Adhesion

#### (Third Revision)

(Page 1, clause 2.2.2, line 3) — Substitute one of the diameters of the mandrels '6.25 mm' for '6 mm'.

(Page 2, clause 2.2.3, line 4) — Substitute one of the diameters of the mandrels '6.25 mm' for '6 mm'.

(Page 5, clause 3.2.3) -- Substitute the following for the existing clause:

'3.2.3 Needle — The scratching needle has a hard hemispherical tip of 1 mm of diameter. The hemispherical tip shall be firmly attached and the exposed part shall be free from any contaminants.

NOTE - Details about the tip and the needle are given in Annex A for guidance.'

(Page 5, clause 3.3) — Substitute the following for the existing clause:

'3.3 Test Panels — Unless agreed for, the panels shall be of tin plate, burnishing steel or hard aluminium prepared by acid chromating.'

(Page 12, clause 5.6.2) - Insert the following Annex at the end:

#### ANNEX A (Clause 3.2.3)

#### PROCEDURE FOUND TO BE CONVENIENT FOR MANUFACTURE AND RE-TIPPING OF SCRATCH TEST NEEDLES

#### A-1 Manufacture of New Needles

A-1.1 For a batch of shanks in the vertical position with the dimpled end uppermost by inserting them in a tray made from perforated sheet metal.

#### Amend No. 1 to IS 101 ( Part 5/Sec 2 ) : 1986

A-1.2 Place a very small quantity of a suitable solder paste on the end of each shank, and then put a steel ball in position, relying on the solder paste to hold the ball in position at this stage.

NOTE — The amount of solder paste to be used is judged by experience, insufficient will not make a firm soldered joint and too much will lead to more or less complete envelopment of the steel ball.

A-1.3 Place the tray of shanks in an oven or muffle furnace adjusted to a temperature of 210°C to 220°C for about 5 min to melt the solder and thus secure the ball in the dimple at the end of the shank.

A-1.4 Remove the needles from the source of heat, allow to cool, and wipe each ball to remove flux residues.

A-1.5 Check that the ball is securely fixed and that there is no solder on the part of the ball which will be used to make the scratch.

#### A-2 Re-tipping of Needles

A-2.1 Fix the needles in a tray made from perforated sheet metal as described in A-1.1 above.

A-2.2 Place the tray of needles in an oven or muffle furnace adjusted to a temperature of 210°C to 220°C and, as soon as the solder has softened, remove the tray and brush the end of the needles with a clean brush in order to dislodge the steel balls.

A-2.3 When the shanks have cooled, proceed as described in A-1.2 to A-1.5.

#### A-3 Guidance on Tip Material

The following tips are in common use:

a) Steel Balls	Depending on the circumstances, this type of tip may show signs of early wear. In view of the ease will which needles with steel balls can be made, it is recommended that they be used once only and then re-tipped.
b) Tungsten Carbide Tips	These tips last much longer than steel tips and can be obtained commercially.

#### Amend No. 1 to IS 101 (Part 5/Sec 2): 1988

c) Ruby Tips These (ceramic) tips last a very long time. The tips have to be attached to the shank by glueing rather than by soldering and can be obtained commercially.

In all cases, it is important that a suitable steel shank is used which will hold the tip securely in the correct position. An example of a suitable shank is given in Fig. 6 of this standard.

(CHD 020)

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## AMENDMENT NO. 2 MARCH 1999 TO IS 101 (PART 5/SEC 2) : 1988 METHODS OF SAMPLING AND TEST FOR PAINTS, VARNISHES AND RELATED PRODUCTS

#### PART 5 MECHANICAL TESTS

#### Section 2 Flexibility and Adhesion

(Third Revision)

(*Page 3, clause 2.3.3, lines 2 and 3*) — Substitute the following for the existing:

'IS 101 (Part 3/Sec 2): 1989 Methods of sampling and test for paints, varnishes and related products : Part 3 Tests on paint film formation, Section 2 Film thickness (*third revision*)'.

(*Page 7, clause 3.4.3*) — Insert the following sentence at the end of clause:

'Unless otherwise specified in the product specification, it shall be deemed to have met this requirement if it passes under a load of 1 000 g.'

(CHD 20)

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Printed at Dee Kay Printers, New Delhi

## AMENDMENT NO. 3 MAY 2006

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## IS 101 (PART 5/SEC 2): 1988 METHODS OF SAMPLING AND TEST FOR PAINTS, VARNISHES AND RELATED PRODUCTS

#### PART 5 MECHANICAL TESTS

#### Section 2 Flexibility and Adhesion

#### (Third Revision)

(Page 1, clause 1) - Substitute the following for the existing clause:

<sup>4</sup>1 Scope — Prescribes the following five tests for determination of flexibility and adhesion:

- a) Bend test,
- b) Scratch hardness test,
- c) Cupping test,
- d) Pull off test, and
- e) Cross cut adhesion test.'

(Page 12, clause 5.6.2, read with Amendment No. 1) — Add the following at the end:

#### '6 CROSS CUT ADHESION TEST

6.1 Outline of the Method — This method is applicable for the assessment of the adhesion of the coating films to metallic substrates by applying and removing the 25 mm semi-transparent pressure sensitive tape over cuts made in the film.

6.2 Test Methods — There are essentially two test methods for the determination of adhesion.

6.2.1 Test Method 1 - It is generally used for job at site and if the dry film thickness is 125 microns or more.

6.2.2 Test Method 2 — It is generally used for job in the laboratory and if the dry film thickness is less than 125 microns.

**6.2.3** These methods do not distinguish between higher levels of adhesion. These methods are not applicable in multicoated system where failure may occur between coats.

#### Amend No. 3 to IS 101 ( Part 5/Sec 2 ): 1988

#### 6.3 Test Method 1

6.3.1 Apparatus

6.3.1.1 (*'utting tool* — Sharp razor blade, scalpel, knife or other cutting edges are in good condition.

6.3.1.2 Cutting guide steel or other hard metal straightedge to ensure cuts.

**6.3.1.3** Tape -25 mm wide semi-transparent pressure sensitive tape (see IS 13262 : 1992 Pressure sensitive adhesive tapes with plastic base).

6.3.1.4 Eraser — attached at the end of the pencil.

**6.3.1.5** *Illumination* — A light source to determine whether the cuts have been made through the film to the substrate.

**6.3.1.6** Test specimen — In the field the specimen is the coated structure or article on which the adhesion is to be evaluated. In the laboratory, apply the materials to mild steel panels or panel of the composition and surface conditions on which it is desired to determine adhesion.

6.3.2 Procedure — Select a clean and dry area of film free from blemishes and minor surface imperfection. Two cuts are made in the film about 40-mm long that intersect near their middle with a smaller angle of 30° to 45°. When making the incisions, use the straightedge and cut through the coating to the substrate in one steady motion. Inspect the incisions for reflection of light from the metal substrate to establish that the coating film has been penetrated. If the substrate has not been reached make another cuts in different location. Do not attempt to deepen the previous cut as this may affect adhesion along the incision. Remove two complete laps of the pressure-sensitive tape from the roll. An additional length of 75 mm is removed at a steady rate. Place the centre of the tape at the intersection of the cuts with the tape running in the same direction as the smaller angles. Smooth the tape into place by finger in the area of the incisions and then rub firmly with the eraser on the end of the pencil. The colour under the transparent tape is useful indication of when good contact has been made. Within 90  $\pm$  30 seconds of application, remove the tape by seizing the free end by pulling it off rapidly back upon itself as close to an angle of 180° as possible. Inspect the cut area for removal of coating from the substrate or previous coating. The coated panel is considered to satisfy the test if no peeling or removal of the film is observed.

Amend No. 3 to 101 (Part 5/Sec 2) : 1988

#### 6.4 Test Method 2

#### 6.4.1 Apparatus

6.4.1.1 Cutting tool — Sharp razor blade, scalpel, knife or other cutting device having a cutting edge angle between 15 and 30 degrees that will make either a single cut or several cuts at once.

6.4.1.2 Cutting guide - Steel or other hard metal straightedge to ensure cuts.

6.4.1.3 Tape --- 25 mm wide semi-transparent pressure sensitive tape (see IS 13262 : 1992).

6.4.1.4 Eraser — attached at the end of the pencil.

6.4.1.5 *Illumination* — A light source to determine whether the cuts have been made through the film to the substrate.

#### 6.4.1.6 Magnifying glass

**6.4.1.7** Test specimen — In the field the specimen is the coated structure or article on which the adhesion is to be evaluated. In the laboratory, apply the materials to mild steel panels or panel of the composition and surface conditions on which it is desired to determine adhesion.

6.4.2 Procedure - Select a clean and dry area of film free from blemishes and minor surface imperfection. Place the panel on a firm base and under the illuminated magnifier make parallel cuts. For coatings having a drying film thickness up to and including 50 micron space the cuts 1 mm apart and make eleven cuts. For coatings having a drying film thickness between 50 micron and 125 micron space the cuts 2 mm apart and make six cuts. For film thickness greater than 125 microns this method is not applicable. Make all cuts about 20 mm long. Cut through the film to the substrate in one steady motion using just sufficient pressure on the cutting tool to have the cutting edge reach the substrate. After making the required cuts brush the film slightly with a soft brush to remove any detached flakes or ribbon of coatings. An additional number of cuts at 90° centered on the original cuts are made. Brush the area as before and inspect the incision for reflection of light from the substrate. If the metal has not been reached make another grid in a different location. Remove two complete laps of the pressure-sensitive tape from the roll. An additional length of 75 mm is removed at a steady rate. Place the centre of the tape over grid. Smooth the tape into place by finger in the area of the incisions and then

Amend No. 3 to 101 (Part 5/Sec 2) : 1988

rub firmly with the eraser on the end of the pencil. The colour under the transparent tape is useful indication of when good contact has been made.

Within 90  $\pm$  30 seconds of application, remove the tape by seizing the free end by pulling it off rapidly back upon itself as close to an angle of 180° as possible. Inspect the grid area for removal of the coating from the substrate or from a previous coating using the illuminated magnifier. The edges of the cuts should be smooth and none of the square of the lattice should be detached. The coated panel is considered to satisfy the test if no peeling or removal of the film is observed.

(CHD 20)

Reprography Unit, BIS, New Delhi, India

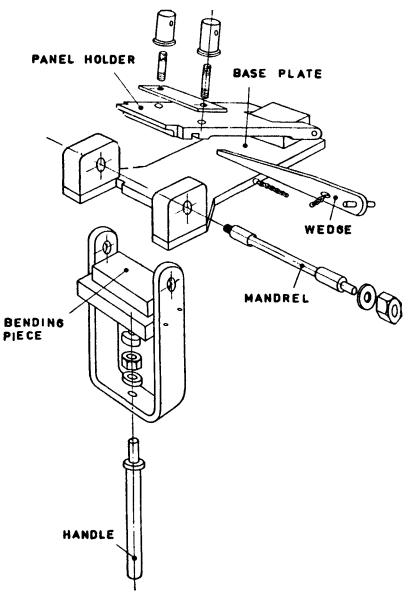


FIG. 4 DETAILS OF TYPE 2 BEND TEST APPARATUS

**2.3.1** Dimensions — The test panels shall be rectangular and approximately 100 mm  $\times$  50 mm in size and either 0.3 mm (for use with Type 1 apparatus ) or 1.0 mm (for use with Type 2 apparatus ) thick, unless otherwise specified. The test panels may be cut to size after coating and drying provided no distortion occurs. In case of aluminium panels, the longer side shall be parallel to the direction of metallurgical rolling.

2.3.2 Preparation and coating of panels — The test panels shall be prepared in accordance with IS: 101 (Part 1/Sec 3)-1986 'Methods of test for paints, varnishes and related products: Part 1 Test on liquid paints (general and physical), Section 3 Preparation of panels (*third revision*)', unless otherwise specified, and shall then be coated by the specified method with the product under test. If the product under test is applied by brushing, the direction of laying off marks shall be parallel to the longer side of the panel.

**2.3.3** Thickness of coating — The thickness in  $\mu$ m of the dry coating shall be determined by the method specified, using one of the procedures described in IS : 101 (Part 3/Sec 6) 'Methods of test for paints, varnishes and related products: Part 3 Tests on paint film formation, Section 6 Film thickness (*third revision*)'.

#### 2.4 Procedure

2.4.1 Drying the test panel — The coated test panels shall be dried (or stoved and aged) for the specified time and, unless otherwise specified, shall be conditioned at  $27 \pm 2^{\circ}$ C and  $65 \pm 5$  percent relative humidity for a minimum of 16 h. The appropriate test procedure shall then be carried out as soon as possible.

#### IS: 101 (Part 5/Sec 2) - 1988

**2.4.2** Ambient conditions — The test shall be carried out at  $27 \pm 2^{\circ}$ C and  $65 \pm 5$  percent relative humidity, unless otherwise specified.

2.4.3 Handling the test panel - Warming the test panel by undue handling shall be avoided.

**2.4.4** Procedure for a single specified size of mandrel — The appropriate procedure given in **2.4.5**, **2.4.6**, or **2.4.7** shall be carried out on two separate test panels and the panels shall then be examined as described in **2.4.8** ( if the results differ, additional tests shall be made ).

**2.4.5** Test with Type 1 apparatus at  $27 \pm 2^{\circ}$ C — Fully open the apparatus, fitted with the appropriate mandrel, and insert the panel so that it may subsequently be bent with the coated side outwards.

Close the apparatus evenly without jerking, over a period of 1 to 2 s, thus bending the panel over the mandrel through 180°.

**2.4.6** Tests with Type 2 apparatus at  $27 \pm 2^{\circ}C$  — Firmly secure the apparatus near the edge of a bench so that the handle can be operated freely. Lower the panel holder by withdrawing the wedge and use the adjusting screw to move the bending piece away from the mandrel position. Fit the apparatus with the appropriate mandrel. Lower the handle into a vertical position and then insert the panel, painted side down, between the mandrel and the bending piece until approximately 40 mm of the panel protrudes when measured from the mandrel centre line towards the bending piece. Clamp the panel firmly to the panel holder using the lock nuts and plate. Raise the panel holder by inserting the wedge into its groove until the panel just touches the mandrel. Using the adjusting screw, raise the bending piece until it just touches the panel. Lift the handle evenly through 180°C.

After bending, release the panel by moving the bending piece away from the mandrel, lowering the panel holder by removing the wedge and unscrewing the lock nuts.

**Note** — A piece of thin paper may be inserted over the coated surface between the panel holder and the bending piece to prevent the coating being scratched during the bending operation.

**2.4.7** Tests at temperatures other than  $27 \pm 2^{\circ}C$  — Open the apparatus fitted with the appropriate mandrel and place the panel in position so that it may subsequently be bent with the coated side outwards. Place the apparatus containing the panel in the test chamber previously adjusted to the specified temperature. After 2 h, with the panel still in the chamber at the specified temperature, close the apparatus evenly and without jerking by means of the remote-control device, over a period of 1 to 2 s. It is essential that the door of the chamber shall remain closed from the time of insertion of the apparatus until after the bending operation.

**2.4.8** Examination of the test panel — Examine the test panel coating immediately after bencing, and in the case of Type 1 apparatus, without removing the panel from the apparatus. Use normal corrected vision or, by agreement, a lens of  $10 \times \text{magnification}$ , and examine the coating for cracking and/or detachment from the substrate, ignoring the surface of the coating less than 10 mm from the edge of the panel.

Note - If a lens is used, it is essential to mention this fact in the test report and to avoid misleading comparisons with results obtained using normal vision only.

2.5 Procedure for Determination of Diameter of the First Mandrel to Cause Failure — Carry out the appropriate procedure given in 2.4.5, 2.4.6 and 2.4.7 on successive test panels, examining each panel as described in 2.4.8 and using mandrels of successively smaller diameter until the coating cracks and/or becomes detached from the substrate. Record the diameter of the first mandrel at which the coating cracks and/or becomes detached, after confirming the result by repeating the procedure with this size of mandrel on a fresh panel. In the event of failure not occurring with the mandrel of smallest diameter, record this fact.

#### 3. Scratch Hardness

**3.1** Outline of the Method – This is a test method for determining the resistance of a paint film to penetration by scratching with a needle.

**3.1.1** The test may be used as a GO/NO-GO test by appyling a single specified load to the needle. It may also be used by applying increasing loads to the needle to determine the maximum load at which the coating is pulled/penentrated to the substrate.

#### 3.2 Apparatus

**3.2.1** Mechanized apparatus — Figure 5 illustrates the principle of the apparatus. Figure 6 shows a suitable type of apparatus but other arrangements can be used which give a similar performance. This apparatus consists essentially of a horizontal sliding panel (A) driven by a constant-speed

## IS: 101 ( Part 5/Sec 2 ) - 1988

motor (B) at a rate of 3 to 4 cm/s beneath the point of a scratching needle (C) which is perpendicular to the film. The needle is fixed in a chuck, directly above which is a holder for weights which shall be able to take weights up to at least 2 000 g (see Note). The apparatus is adjusted so that the needle comes smoothly into contact with the film, that is, before the stop (D) reaches the bottom of the sloping ramp (E). The scratch shall be straight and not less than 60 mm in length. A ramp with an angle of 10 to 15° to the horizontal has been found to be satisfactory. An electrical indicating device based on conductivity may be used as a guide to penetration of the film, where appropriate.

Note — The maximum load for which the apparatus is designed shall be marked on the apparatus.

**3.2.2** Hand operated apparatus — A hand operated apparatus may be used except for referee tests, provided it is operated so as to give a similar performance to the mechanized apparatus specified in **3.2.1**, that is, regular movement, smooth lowering of the needle, etc.

A schematic diagram of the apparatus is as shown in Fig. 7.

**Note** — Smooth lowering may conveniently be accomplished by lowering the needle onto a safety razor blade and then allowing it to slide off the sharp edge of the blade onto the film.

**3.2.3** Needle — The scratching needle has a hardened steel hemispherical tip of 1 mm diameter, and may conveniently be made by soldering a 1 mm diameter steel ball of the type used in ball bearings (1 percent C, 1 percent Cr type steel) to a steel shank of the type illustrated in Fig. 7.

**3.3** Test Panels — The panels shall be burnished steel plate, tinplate, or acid chromate treated hard aluminium.

**3.3.1** Dimensions — The test panels shall be rectangular and approximately 125 mm  $\times$  50 mm in size. If the product under test is to be applied by brushing, the material used shall be of such a size that, after coating drying, a panel of the above dimensions can be cut from it with the brush marks parallel to the shorter side.

**3.3.2** Preparation and coating of panels — The test panels shall be prepared in accordance with IS : 101 (Part 1/Sec 3)-1986, unless otherwise specified, and shall then be coated by the specified method with the product or system under test.

#### 3.4 Procedure

**3.4.1** Drying the test panels — The coated test panels shall be dried (or stoved and aged) for the specified time, and unless otherwise specified, shall be conditioned at a temperature of  $27 \pm 2^{\circ}$ C and relative humidity of 65  $\pm$  5 percent for a minimum time of 16 h. The appropriate test procedure shall then be carried out as soon as possible.

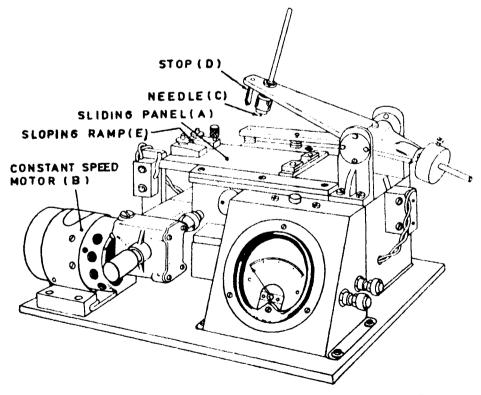


FIG. 5 SUITABLE TYPE OF MECHANIZED SCRATCH TEST APPARATUS

## IS : 101 ( Part 5/Sec 2 ) - 1988

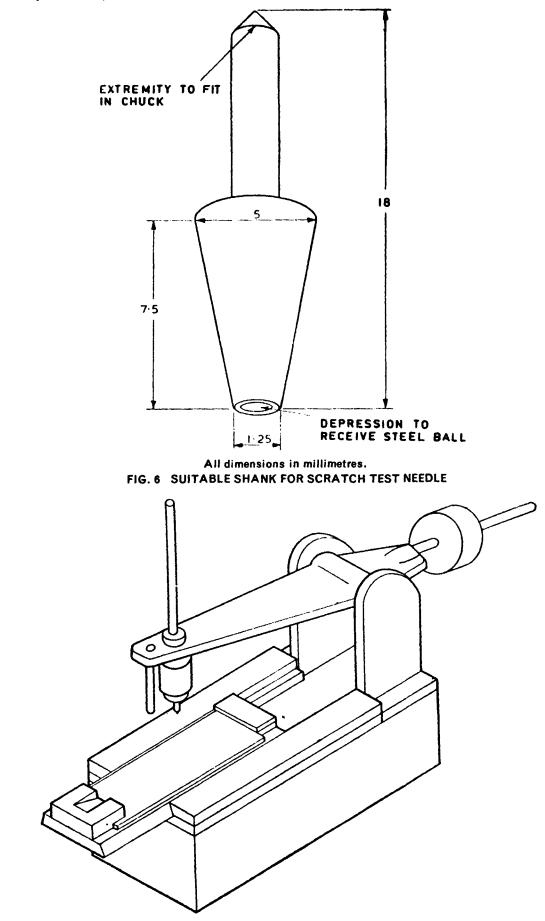


FIG. 7 DIAGRAM OF SCRATCH TEST APPARATUS

#### **3.4.2** Single specified load

**3.4.2.1** Take an unused needle and examine it under  $30 \times magnification$  to ensure that the hardened steel point is smooth, hemispherical and free from contamination.

**3.4.2.2** Fix the needle in the chuck so that the position of operation is perpendicular to the coating.

**3.4.2.3** Clamp the test panel, with the coating uppermost, to the sliding panel of the apparatus with the longer side of the panel parallel to the direction in which the scratch will be made.

3.4.2.4 Place weights on the holder above the needle to obtain the specified load.

**3.4.2.5** Start the motor of the apparatus or pull the sliding panel of the hand-operated apparatus and allow the scratch to be made on the coating.

**3.4.2.6** Remove the panel and examine the scratch to see if the coating has been penetrated to the substrate. By agreement between the interested parties, a suitable magnification may be used, in which case the degree of magnification shall be mentioned in the test report.

**3.4.3** Minimum load to cause failure — Carry out the precedure given in **3.4.2**, using a fresh portion of a test panel for each scratch, starting at a load somewhat less than that expected to cause penetration of the coating and continuing by successively increasing the load by suitable increments (for example 50 g), until the coating is penetrated to the substrate. Record the minimum load at which the needle penetrates the coating after confirming the result by repeating the procedure with this load both on the original test panel and on a fresh one.

#### 4. Cupping Test

**4.1** Outline of the Method — This is a test for assessing the paint film to cracking and/or detechment from a metal substrate when subjected to gradual deformation by indentation under specified conditions. The method may be either used as a GO/NO-GO test by testing to a specified depth of indentation to assess compliance with a particular requirement or by gradually increasing the depth of indentation to determine the minimum depth at which the film cracks and/or becomes detached from the substrate.

#### 4.2 Apparatus

4.2.1 Cupping apparatus — As shown in Fig. 8.

**4.2.1.1** Die — This is surface hardened and the surface in contact with the test panel is plane polished.

**4.2.1.2** Retaining ring — The surface in contact with the test panel is plane polished and parallel to the contacting surface of the die.

**4.2.1.3** Indentor — The part contacting the test panel is of hardened polished steel and forms a hemisphere of 20 mm diameter. During the test, the indentor shall be prevented from turning and the centre of the spherical portion shall not deviate from the axis of the die by more than 0.1 mm.

**4.2.1.4** Measuring device — This measures the depth of the indentation made by the indentor to the nearest 0.05 mm.

#### 4.3 Panels

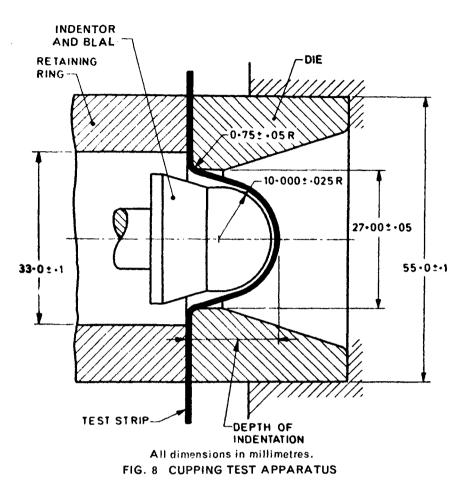
4.3.1 Dimensions — The test panel shall be rectangular with the following dimensions:

- a) Thickness Not less than 0.3 mm and not more than 1.25 mm (as specified and measured with a micrometer to the nearest 0.01 mm).
- b) Width and length At least 70 mm (upper limit depending on the capacity of the apparatus).

The test panel may be cut to size after coating and drying provided no distortion occurs.

**4.3.2** Coating of test panel — Coat the panel with material under test by the appropriate method and dry or stove in specified manner for the specified period. If normal drying conditions are specified, these shall be interpreted as a temperature of  $27 \pm 2^{\circ}$ C and a relative humidity of  $65 \pm 5$  percent with free circulation of air and not exposed to direct sunlight.

Unless otherwise specified, the test panel shall be conditioned at  $27 + 2^{\circ}C$  and  $65 \pm 5$  percent relative humidity for 16 h immediately prior to carrying out the test.



#### 4.4 Procedure

**4.4.1** Procedure for a single specified depth of indentation — The following procedure shall be carried out on two separate test panels (if the results differ, further tests shall be made).

**4.4.1.1** Hold the test panel firmly between the retaining ring and the die with the coating towards the die, and with the spherical end of the indentor in contact with the test panel (zero position of the indentor). Adjust the panel until the central axis of the indentor intersects the panel at at least 35 mm from each edge.

**4.4.1.2** Having measured the temperature and the humidity, advance the spherical end of the indentor into the test piece at a rate of  $0.2 \pm 0.1$  mm/s ( $12 \pm 6$  mm/min) until the specified depth is reached, that is until this distance has been travelled by the indentor from the zero position.

**4.4.1.3** Using normal corrected vision or, by agreement, a lens of  $10 \times$  magnification, examine the coating of the test panel for cracking and/or detachment from the substrate.

Note 1 — If a lens is used, it is essential to mention this fact in the test report and to avoid misleading comparisons with the results obtained using normal corrected vision only.

Note 2 - If the substrate shows any signs of cracking, the test should be considered as invalid.

**4.4.2** Procedure for determination of minimum depth of indentation to cause failure — Carry out the procedure given in **4.4.1** but advance the indentor until, using normal corrected vision (or, by agreement, a lens of  $10 \times$  magnification for observation), a crack first appears on the surface of the coating and/or the coating begins to become detached from the substrate.

Stop the indentor at this point and measure the depth of indentation (see Fig. 8) to the nearest 0.1 mm as the distance travelled by the indentor from the zero position. Confirm the result by repeating the determination on a fresh panel (if the results differ, further determinations shall be made).

## 5. Pull off Test

5.1 Outline of the Method — This is a pull off test on a single or multi coat system of paint, varnish or related products. One test may be carried out using different substrates.

#### 5.2 Apparatus

**5.2.1** Tensile tester—Suitable for carrying out the chosen procedure specified in **5.5**. The tensile stress shall be applied in a direction perpendicular to the coated substrate and shall be increased at a substantially uniform rate, not greater than 1 MPa/s, such that the failure of the test assembly occurs within 90 s. Suitable designs for applying the tensile stress are shown in Fig. 9.

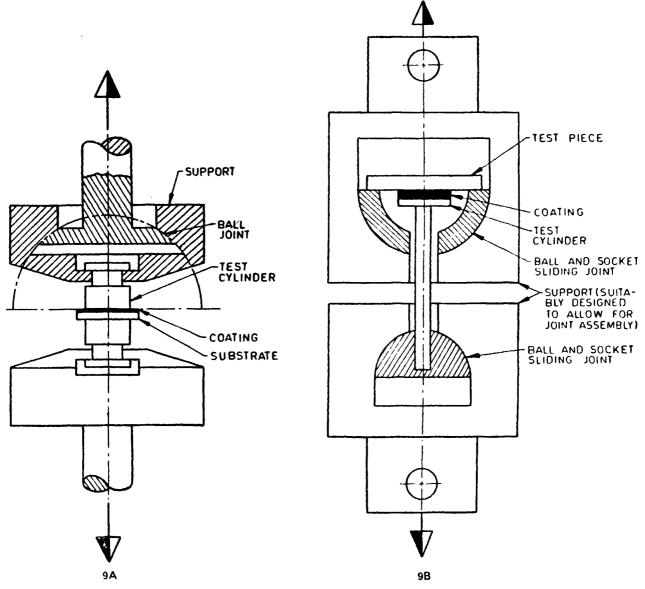


FIG. 9 SUITABLE DESIGNS FOR APPARATUS FOR PULL OFF TEST

**5.2.2** Test cylinders — Suitable for use with the tensile tester (**5.2.1**), faced, of diameter 20 mm (unless otherwise agreed) and of sufficient thickness to ensure freedom from distortion during the test. It is recommended that the length of the test cylinder should be not less than half its diameter. The faces shall be machined perpendicular to the long axis of the cylinder before use.

5.2.3 Centring device — For ensuring proper coaxial alignment of the test assembly during the adhesion process used as described in 5.5.1 and 5.5.3. A suitable design is shown in Fig. 10.

5.2.4 Cutting device — Such as a sharp knife, for cutting through cured adhesive and the paint coating to the substrate, round the circumference of the test cylinder.

#### 5.3 Materials

**5.3.1** Adhesives — Special attention is required in selecting suitable adhesives to be used in the test.

To produce failure of the coating, it is essential that the cohesive and bonding properties of the adhesive be greater than those of the coating under test.

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Preliminary screening of adhesives shall be carried out in order to determine their suitability for use. Suitable adhesives and, if applicable, their unmixed components shall cause little or no visible change in the coating under test when left in contact with the coating for a period equivalent to the curing time of the adhesive.

An adhesive may be considered suitable for a particular coating if it gives the same test result as that produced by using a different class or type of adhesive when similarly tested.

Note — In most cases, cyanoacrylate, two-component solventless epoxide and peroxide-catalyzed polyester adhesives have been found suitable. Cyanoacrylate and polyester adhesives have a short curing time and are pre-ferred for use with coatings that have been subjected to highly humid conditions.

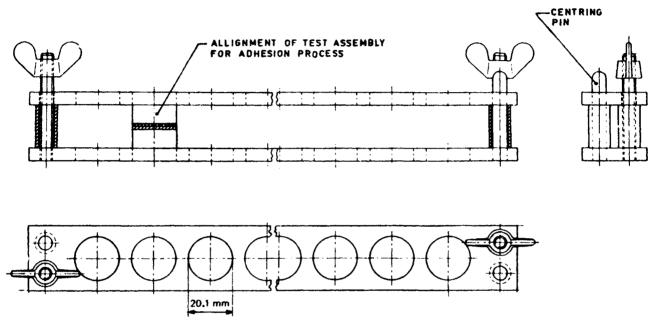


FIG. 10 SUITABLE DESIGN FOR CENTRING DEVICE FOR 20 mm DIAMETER TEST CYLINDERS

#### 5.4 Test Substrate

**5.4.1** Preparation and coating of test substrate — Prepare the specified test substrate in accordance with IS : 101 (Part 1/Sec 3)-1986 and then coat it by the specified method with the product or system under test.

**5.4.2** Drying the test coating — Dry (or stove and age) the coated test substrate for the specified time and under the specified conditions and condition it at  $27 \pm 2^{\circ}$ C and  $65 \pm 5$  percent relative humidity for a minimum of 24 h. Then carry out the appropriate test procedure as soon as possible.

5.4.3 Thickness of coating — Determine the thickness, in micrometres, of the dry coating by one of the procedures specified in IS : 101 (Part 3/Sec 6).

#### 5.5 Procedure

5.5.1 Adhesives — Prepare and apply the àdhesive in accordance with the manufacturer's instructions. Use the minimum quantity of adhesive required to produce a firm, continuous and even bond between the components of the test assembly. Remove any excess adhesive immediately, if possible.

**5.5.2** General method for testing both rigid and deformable substrates — Use as the test piece an area, cut from the coated substrate (disc of minimum diameter 30 mm or square of minimum side 30 mm). Take care not to distort the test piece. Apply the adhesive evenly to the surfaces of two freshly-cleaned test cylinders of equal diameter (see Notes 1 and 3). Place the test piece between the adhesive-coated faces of the test cylinders such that the test cylinders are coaxially aligned in the centre of the piece, as shown in Fig. 11. Align the test assembly in the centring device and maintain the alignment for a period equal to the curing time of the adhesive (see Note 2). At the end of this period, carefully use the cutting device to cut around the circumference of the test cylinders through to the substrate.

Note 1 — The adhesion at the adhesive coating interface may be improved by lightly abrading the surface of the dried coating before the application of adhesive-coated face of the test cylinder.

Note 2 - In special tests under highly humid conditions, the curing time of the adhesive shall be as short as possible.

Note 3 — In the method for deformable substrates, if a poor adhesive bond is expected between the uncoated face of the substrate and the test cylinder, coat both faces of the substrate with the product under test.

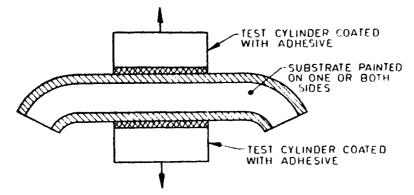


FIG. 11 TEST ASSEMBLY, SANDWICH METHOD (SUBSTRATE PAINTED ON ONE OR BOTH SIDES)

**5.5.3** Method for testing from one side only (suitable for rigid substrates only) — Apply the adhesive evenly to the uncoated, freshly-cleaned surface of a test cylinder. Place the adhesive-coated face of the test cylinder in contact with the coating (see Note 1 under **5.5.2**), for a period equal to the curing time of the adhesive (see Note 2 under **5.5.2**). At the end of this period, carefully use the cutting device (**5.2.4**) to cut round the circumference of the test cylinder through to the substrate.

Place the outer ring in position and test as indicated in Fig. 12.

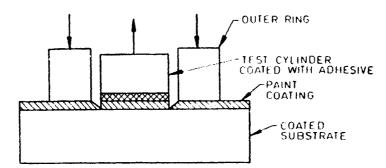
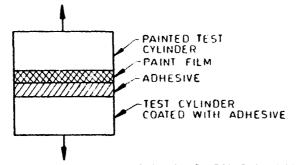


FIG. 12 TEST ASSEMBLY FOR RIGID SUBSTRATES

**5.5.4** Method using test cylinders — Apply the adhesive evenly to the uncoated, freshly-cleaned surface of a test cylinder. Place the adhesive-coated surface of the test cylinder in contact with the surface of the cylinder coated with the product under test, as shown in Fig. 13, and align the test assembly in the centring device for a period equal to the curing time of the adhesive.





**5.5.5** Measurement — Immediately after the period allowed for curing of the adhesive, place the test assembly in the tensile tester taking care to align the test cylinders so that the tensile force is applied uniformly across the test area, without the bending moment. Apply a tensile stress, increasing at a rate not greater than 1 MPa/s, perpendicular to the plane of the coated substrate such that the failure of the test assembly shall occur within 90 s of intial application of the stress.

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Record the tensile stress to break the test assembly and examine the fracture surfaces in accordance with **5.6.2**.

**5.5.6** Number of determinations — Carry out at least three determinations. For reteree purposes, carry out a minimum of five determinations. Report the results of all determinations.

#### 5.6 Results

**5.6.1** Breaking strength — The breaking strength, in megapascals, for each test assembly is given by the formula:

4F d<sup>3</sup>

where

F = the breaking force, in newtons; and

d = the diameter, in millimetres, of the test cylinder.

In the case of test cylinders of diameter 20 mm, the breaking strength, in megaposcals, is given by the formula:

$$\frac{4F}{400} = \frac{F}{100}$$

5.6.2 Nature of failure — Express the result as the percentage area and the site of fracture in the system under test in terms of adhesive, cohesive or adhesive/cohesive failure.

For convenience, the following scheme may be used to describe the results observed:

A =cohesive failure of substrate

A/B = adhesive failure between substrate and first coat

B = cohesive failure of first coat

B/C = adhesive failure between first and second coats

 $-/\gamma$  = adhesive failure between final coat and adhesive

 $\gamma$  = cohesive failure of adhesive

Y/Z = adhesive failure between adhesive and test cylinder.

#### Example:

If a paint system tested in the pull off test breaks at a tensile stress of 20 MPa and examination of the area on each side of the site of separation reveals approximately 30 percent of the test cylinder area associated with cohesive break of the first coat and 70 percent of the test cylinder area associated with intercoat adhesive break between the first and the second coats, the pull off test result is expressed as:

20 MPa, 30 percent B, 70 percent B/C

## EXPLANATORY NOTE

This is one of a series of standards on methods of sampling and test for paints, varnishes and related products. This standard supersedes clauses **15** and **16** of IS : 101-1964 'Methods of test for ready mixed paints and enamels (*second revision*)'. In the preparation of this standard, considerable assistance has been taken from ISO : 1518-1973 'Paints and varnishes — Scratch test prepared by the International Organization for Standardization (ISO).