

पुनर्पुष्ट 2011
Reaffirmed 2010
पुनर्पुष्ट 2010
Reaffirmed 2010

पुनर्पुष्ट 2001
RE-AFFIRMED

पुनर्पुष्ट 2006
RE-AFFIRMED

IS 1359 : 1992
(Reaffirmed 1998)

पुनर्पुष्ट 2010
Reaffirmed 2010 भारतीय मानक

टीन के विद्युत लेपनों की विशिष्टि

(तीसरा पुनरीक्षण)

पुनर्पुष्ट 2016
Reaffirmed 2016



Indian Standard

ELECTROPLATED COATINGS OF TIN —
SPECIFICATION

(Third Revision)

First Reprint OCTOBER 2000

UDC 669.687

© BIS 1992

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

December 1992

Price Group 6

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Metallic and Non-metallic Finishes Sectional Committee had been approved by the Metallurgical Engineering Division Council.

This standard was first published in 1959 and subsequently revised in 1966 and 1971.

This standard is based on the practices generally followed in the country in this field. While reviewing the standard, need was felt to revise this standard to align various requirements in line with the International standards. In this revision, the following main modifications have been made:

- a) Service grades have been reduced to four, and
- b) Corrosion requirements of tin coatings have been incorporated.

In the formulation of this standard, assistance have been derived from ISO 2093 : 1986 'Electroplated coatings of tin', issued by the International Organization for Standardization.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (revised)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ELECTROPLATED COATINGS OF TIN — SPECIFICATION

(Third Revision)

1 SCOPE

1.1 This standard specifies the requirements for electroplated tin coatings of not less than 99.5 percent tin applied to fabricated articles of Iron, Steel, Nickel alloys and Copper and copper alloys containing more than 50 percent of copper. It also covers coatings brightened by fusion in hot oil after electrodeposition, the process being known as flow-brightening or flow melting.

1.2 This standard does not, however, cover coatings on:

- a) threaded components;
- b) tin-coated copper wire;
- c) coatings on sheet, strip or wire in unfabricated form or on articles made from them;
- d) coatings in coil springs;
- e) coatings applied by chemical means (Immersion, autocatalytic or electroless);
- f) electroplating of steels with tensile strength greater than 1 000 Mpa (or of corresponding hardness); because such steels are subject to hydrogen embrittlement (see 7.2); and
- g) aluminium and zinc alloys.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

| IS No. | Title |
|--------------------------|---|
| 193 : 1982 | Soft solder (fourth revision) |
| 265 : 1976 | Hydrochloric acid (second revision) |
| 715 (Part 2) : 1976 | Coated abrasives: Part 2 Special and mechanized application (third revision) |
| 2384 : 1963 | Tin anodes for electroplating |

3 TERMINOLOGY

For the purpose of this standard, the following definition shall apply.

3.1 Significant Surface

The surface agreed upon between the purchaser and the electroplater, and indicated on the drawing or suitably marked on a sample with

particular reference to the surface not to be plated.

NOTE — Surfaces on which a controlled deposit cannot ordinarily be obtained, such as holes, recesses, bases of angles and similar areas, are normally exempted from the requirements for significant surfaces, unless they are specially designated as such.

3.2 Flow-Melting, Fusing, Flow-Brightening, Reflowing

A process by which a coating is melted in order to impart desirable properties such as brightness or improved solderability (see 9.2.1.1).

4 INFORMATION TO BE SUPPLIED BY THE PURCHASER TO THE ELECTROPLATER

4.1 Essential Information

The following information shall be supplied by the purchaser to the electroplater:

- a) the number of this standard;
- b) the nature of the basis material (see 5.1);
- c) the service grade number (see 6.1) or the classification code of the coating required (see 6.2);
- d) the significant surface of the article to be electroplated indicated, for example, by drawings or by the provision of suitably marked samples; and
- e) the method adhesion testing to be used (see 9.3).

4.2 Additional Information

The following additional information may be required and, if so, shall be specified by the purchaser:

- a) any heat treatment required (see 7);
- b) any requirements for porosity testing (see 9.4.2),
- c) any requirement for solderability testing and the test methods and conditions to be applied (see 9.5),
- d) any special requirements for undercoats (see 8),
- e) a sample showing the required finish (see 5.3.1),

- f) any special pretreatment requirement,
- g) any specific requirements for the purity of the coating,
- h) any special packaging requirements for plated components, and
- j) any special post-plating treatment.

5 MATERIAL AND WORKMANSHIP

5.1 Basis Material

This standard specifies no requirements for the condition, finish or surface roughness of the basis material prior to electroplating.

5.2 Material

The material shall be such as will produce tin coating conforming to this specification. The anode shall be of pure tin complying with the requirements of IS 2384 : 1963.

5.3 Workmanship

Over the area of the significant surface, the plated article shall be free from visible plating defects, such as blisters, pits or unplated areas, and shall not be stained or discoloured. The plated surface shall be white, of a smooth texture, and be free from nodules. Flow-brightened coatings shall be free from de-wetted areas.

5.3.1 The article shall be clean and free from damage. When necessary, a preliminary sample showing the required finish shall be supplied or approved by the purchaser.

6 CLASSIFICATION

6.1 Service Grade Number

The service grade number indicates the severity of the service grade in accordance with the following scale:

4 : Exceptionally Severe — for example service outdoors in severe corrosive conditions or contact with food or drink where a complete cover of tin has to be maintained against corrosion and abrasion.

3 : Severe — for example service outdoors in typical temperature conditions.

2 : Moderate — for example service indoors with some condensation.

1 : Mild — for example service indoors in dry atmospheres or applications where solderability is the primary requirement.

NOTE — See 9.1 which gives guidance on the relation between service grade number and minimum thickness.

When specifying the service grade number or coating classification code, it should be noted that tin is susceptible to damage in abrasive environments or in those containing certain organic vapours.

6.2 Coating Classification Code

The coating classification code shall consist of four parts, the first two of which shall be separated by an oblique stroke, as follows:

a/b c d

where,

- a indicates the chemical symbol for the basis metal (or for the main constituent, if an alloy);
- b indicates the chemical symbol for the undercoat metal (or for the main constituent, if an alloy) followed by a figure for its minimum coating thickness, in micrometres, and is omitted if no undercoat is required [see 4.2 (d)];
- c indicates the chemical symbol for tin, Sn, followed by a figure for its minimum thickness, in micrometres;
- d indicates the surface finish, by the symbol m if the coating is matt, or b if it is bright electroplated or f if it is flow-melted.

An example is

Fe/Ni 2.5 Sn 5 f

which represents as iron or steel basis metal, with a 2.5 μm nickel undercoat, tin electroplated to a coating thickness of 5 μm and flow-melted.

7 HEAT TREATMENT

7.1 When required by the purchaser, certain steels should be treated as described in 7.2 to reduce the risk of damage due to hydrogen embrittlement.

7.1.1 Stress Relief Before Plating

Severely cold-worked steels or parts made from steel of tensile strength of 100 MN/m² or greater which have been ground or subjected to severe machining after tempering shall normally be stress-relieved by maintaining them at $200 \pm 10^\circ\text{C}$ for not less than one hour or, preferably, for 30 minutes at the highest temperature within the limit imposed by the tempering temperature.

7.1.2 Some steels which have been carburized, flame-hardened or induction-hardened and subsequently ground would be impaired by the above treatment, and shall instead be stress-relieved at $140 \pm 10^\circ\text{C}$ for not less than 5 hours.

7.2 Hydrogen Embrittlement Relief After Electroplating

Because diffusion of hydrogen through tin is very slow, heat treatment for hydrogen embrittlement relief after electroplating is impractical.

8 REQUIREMENTS FOR UNDERCOATS

8.1 Undercoats may be necessary on certain basis materials for any of the following reasons:

- to prevent diffusion,
- to retain solderability,
- to ensure adhesion, and
- to improve protection against corrosion.

8.2 Care should be taken to select an undercoat or undercoat system that will not confer undesirable properties such as embrittlement of the basis material or finished article. For example the use, of highly stressed nickel should be avoided.

8.3 If the basis material is a copper alloy containing zinc as an alloying constituent, and solderable properties are required, a nickel or copper undercoat of minimum local thickness $2.5 \mu\text{m}$ is essential in addition to the specified coating thickness of tin such coatings may also be necessary to retain good appearance and adhesion.

8.4 If an undercoat is specified, its nature and minimum local thickness (see 9.1) shall be specified by the purchaser.

The thickness of the undercoat or undercoats shall be measured by the appropriate method specified in Annex A.

9 PLATING REQUIREMENTS

9.1 Thickness

Tin coatings are classified by thickness and for each service grade (see 6.1), minimum values are specified in Table 1.

9.1.1 The thickness of the coating shall be measured over a reference area by the appropriate method given in Annex A on any part of the significant surface that can be touched with a 20 mm diameter ball. In the case of articles having a significant surface area of 100 mm^2 or

greater, the minimum thickness shall be regarded as the minimum value of local thickness. In the case of article having a significant surface area of less than 100 mm^2 , the minimum thickness shall be regarded as the minimum values of average thickness (see Annex B).

9.1.2 In the case of printed circuit boards with electroplated-through holes, the requirements shall apply to the surface within the holes, as well as to the areas that can be touched with a 20 mm diameter ball.

9.1.3 In the case of flow-melted coatings, the thickness requirements apply to the as-electroplated condition, prior to flow-melting.

9.2 Plating

9.2.1 Application

Tin shall be deposited on the articles after completing operations, such as machining, brassing, welding and forming. Tin shall be deposited directly on the basis metal without preliminary coating of other metals.

9.2.1.1 When tin is plated on the article for soft soldering purposes, it may be flow-melted at a temperature between 250°C and 260°C to overcome difficulties in soldering during long periods of storage.

9.3 Adhesion

The coating shall withstand either of the tests given in Annex C. The flaking and blistering of the coating shall be taken as evidence of unsatisfactory adhesion.

9.4 Corrosion/Porosity

If specified by the purchaser, coatings having a minimum thickness of 10 microns or greater, shall be subjected to a corrosion test for ferrous basis material and porosity test for non-ferrous basis material.

Table 1 Coating Thicknesses
(Clause 9.1)

| Service Grade Number | Copper Basis Materials ¹⁾ | | Other Basis Material | |
|----------------------|--------------------------------------|---------------------------------|---------------------------------|---------------------------------|
| | (Partial) Classification Code | Minimum Thickness μm | (Partial) Classification Code | Minimum Thickness μm |
| (1) | (2) | (3) | (4) | (5) |
| 4 | Sn 30 | 30 | Sn 30 | 30 |
| 3 | Sn 15 | 15 | Sn 20 | 20 |
| 2 | Sn 8 | 10 | Sn 12 | 12 |
| 1 | Sn 5 | 5 | Sn 5 | 5 |

¹⁾ Attention is drawn to the essential requirement in 8 for undercoats on copper alloy basis materials that contain zinc as an alloying constituent.

9.4.1 Corrosion Test

The test shall be conducted as per Annex E. The coated item shall be subjected to a 24 hours salt spray test. After removal from the salt spray (fog) cabinet examine the items. The appearance of more than 6 corroded areas that are visible to the unaided eye per sq cm of surface or any corroded areas larger than 1.6 mm in diameter shall be cause for rejection. For purpose of this requirement, a corroded area is defined as exposure or corrosion of the basis metal.

9.4.2 Porosity Test

The test shall be conducted as per Annex F.

9.5 Solderability

The samples shall be considered solderable if they have a uniform coating, free from discontinuities or breaks visible to unaided eye. When so specified by the purchaser, samples of tin coating on copper and copper alloys shall be subjected to preliminary artificial ageing treatment as given in Annex D.

NOTE — The object of this preliminary treatment is to demonstrate whether articles may be expected to retain their solderability during periods of storage. The period of storage at room temperatures which produces an effect equivalent to the test procedure can not be precisely stated but it is probably some years.

10 SAMPLING AND INSPECTION

10.1 Sampling for Production Control

Statistical quality control is recommended for controlling the quality of tin-plated articles. For

this purpose, it is recommended that samples should be taken from a portion where it is likely to give the minimum thickness of plating. The tests specified in 9 shall be conducted on such samples.

10.2 Sampling for Acceptance of a Lot

For the purpose of sampling, a lot shall be divided into sub-lots consisting of 100 articles or part thereof of only such articles as are electroplated at one time in the same bath. Two samples shall be selected from each sub-lot and subjected to the tests specified in 9.

10.2.1 Criteria for Acceptance

If these samples stand the tests, the sub-lot represented by them shall be accepted. If one or both the samples should fail, two further samples shall be selected from the same sub-lot and subjected to the tests. If these samples stand the tests, the sub-lot shall be considered as conforming to the standard.

10.3 When the consignment is made of small articles plated in a barrel, the number to be tested shall be agreed to between the plater and the purchaser.

11 MARKING

The marking related to the tin coating shall include classification number as specified in this standard and the name or trade-mark of the manufacturer.

ANNEX A

(Clauses 8.4 and 9.1 1)

METHOD FOR THE DETERMINATION OF LOCAL THICKNESS

A-1 OVERCOATING

On ferrous articles electrodeposit 1.3 μm of copper followed by a thickness of not less than 50 μm of nickel. For copper base article electrodeposit a thickness of not less than 50 μm of nickel or copper on that sample.

A-2 MOUNTING AND PREPARATION OF SECTIONS

A-2.1 Section the overcoated sample at one or more positions. Mount the section by moulding in a suitable material so that the plated surface is perpendicular to the face which is to be prepared for examination¹⁾ and so that the section is

rigidly held, there being no void between the plated surface and the mounting material.

A-2.1.1 Polish the mounted section, using successively finer abrasives, the last which shall not be coarser than IS grit No. 40 (500-micron) [see IS 715 (Part 2) : 1976]. Final polishing may be carried out on a low speed polishing wheel using suitable polishing media.

A-2.1.2 It is recommended that the surface should be lightly etched with a suitable reagent in order to improve the contrast between the deposit and the adjacent metal and to remove any soft metal which may have spread over a hard metal.

¹⁾ A deviation of 10 degrees from the perpendicular introduces an error of 2 percent in thickness.

A-3 MEASUREMENT OF THE THICKNESS OF DEPOSIT

A-3.1 Project the image of the section on to the screen of a metallographic microscope at a known and properly calibrated magnification. Measure the thickness of the deposit on the projected image accurately with a graduated linear scale and determine the actual thickness of the deposited metal by dividing this measurement by the

magnification.

A-3.1.1 Alternatively, measure the thickness with a metallurgical microscope fitted with a micrometer eye-piece which has been calibrated against an accurately graduated scale.

A-3.1.2 In either case, the magnifications shall be sufficient to allow the actual thickness of deposit to be determined to $\pm 1.2 \mu\text{m}$.

ANNEX B

(Clause 9.1.1)

METHOD FOR THE DETERMINATION OF AVERAGE THICKNESS

B-1 GENERAL

When small articles are to be tested it is convenient to perform a single determination of average thickness on a number of samples taken from a batch.

B-2 STRIPPING SOLUTION

Dissolve 20 g of antimony trioxide in 1 000 ml of cold concentrated hydrochloric acid ($d = 1.16$) (conforming to IS 265 : 1976).

B-3 PROCEDURE

Carefully clean a plated part of known area, free it from grease by means of a suitable solvent treatment, thoroughly dry it and weigh it to an accuracy of one part in 1 000. Sufficient area of sample should be taken to give a weight loss on stripping of at least 0.2 g of tin. Immerse it totally in the stripping solution and turn it over so that the liquid has free access to all surfaces. A fresh

portion of the stripping solution should be used in each test; at least 100 ml of the solution should be used to remove 1 g of tin. Allow the sample to remain in the solution for a period of one minute after gas evolution has ceased. Remove the part, wash it immediately in running water, mop it with a wet soft cloth or cotton wool swab to remove the black powdery deposit of antimony. Dry it and reweigh.

B-4 CALCULATION

Thickness of tin coating in $\mu\text{m} = \frac{137 \times 10^3 (m_1 - m_2)}{A}$ micrometres

where

m_1 = original mass of the part in g,

m_2 = final mass of the part in g, and

A = area of coating in square millimetres.

NOTE — The above calculation assumes a specific gravity of 7.30 for tin.

ANNEX C

(Clause 9.3)

ADHESION TEST

C-1 BURNISHING TEST FOR ADHESION

C-1.1 Rub an area of not more than 25 mm \times 25 mm of the plated surface, selected at the discretion of the inspector, rapidly and firmly for 15 seconds with a smooth metal implement.

C-1.1.1 A suitable burnishing implement is a copper disc, for example, a copper coin used

edgewise and broadside. Maintain a pressure sufficient to burnish the film at every stroke, but not so great as to cut the deposit.

C-2 QUENCHING TEST FOR ADHESION

C-2.1 Heat the plated articles in an oven to a temperature of $185 \pm 5^\circ\text{C}$. Quench the articles in water at room temperature.

ANNEX D

(Clause 9.5)

SOLDERABILITY TEST**D-1 TEST SAMPLE**

For small articles of suitable shape and size the whole article should be tested. For larger articles, cut a portion of suitable size of testing. A recommended panel size is 25 mm × 25 mm. For articles not falling into the categories, sampling shall be as agreed to between the plater and the purchaser.

D-2 EQUIPMENT REQUIRED**D-2.1 Solder Pot**

Electrically heated and maintained at $250 \pm 5^\circ\text{C}$.

D-2.2 Solder

Complying with Grade Sn 60 of IS 193 : 1982 in sufficient volume to ensure that the temperature remains substantially uniform when introducing the test sample.

D-2.3 Flux

Consisting of a solution containing 25 percent by weight of water white rosin and 75 percent by weight of isopropyl alcohol (99 percent pure), free from other additives.

D-3 ARTIFICIAL AGEING

D-3.1 Place the sample for test in a suitable vessel above boiling water and leave it there, with the water boiling continuously for 8 hours. Keep the vessel covered and ensure that the sample does not come into contact with the wall of the vessel and that its lower edge is not less than 50 mm or more than 100 mm above the surface of the boiling water. Arrange the cover on the vessel and the steam condensed water over the sample.

D-3.2 Disregard any discolouration of the sample occurring during this ageing treatment. After the 8 hour treatment, remove the sample from the steam and allow it to dry to air.

D-4 PROCEDURE

Coat the sample with flux by immersion. Ensure that the whole surface of the solder bath is clean and bright by skimming it with a steel scraper, and immediately lower the sample into the bath at a speed of approximately 25 mm/s. Leave it immersed for 5 seconds and then withdraw it at the rate of approximately 25 mm/s, holding at stationary just above the solder surface until the coating solidifies.

ANNEX E

(Clause 9.4.1)

CORROSION TEST**E-1 TEST SOLUTION**

The test solution shall be prepared by dissolving sodium chloride in distilled or de-ionized water to produce a concentration of 50 ± 5 g/l. The sodium chloride shall be white and shall give a colourless solution in water. It shall be substantially free from copper and nickel and shall not contain more than 0.1 % of sodium iodide and not more than 0.4 % of total impurities calculated for dry salt. If the pH of the solution as prepared is outside the range 6.0 to 7.0 the presence of undesirable impurities in the salt or the water or both shall be investigated.

E-1.1 The pH of the salt solution shall be adjusted so that the pH of sprayed solution collected within the test cabinet (see E-2.4) will be between 6.5 and 7.2. Control of pH shall be based on electrometric measurement at 25°C but a short-range pH paper which can be read in

increments of 0.3 pH unit or less and which has been calibrated against electrometric measurements may be used in routine checks. Any necessary correction shall be made by additions of solutions of hydrochloric acid or sodium hydroxide of analytical grade.

Attention is drawn to the possible changes in pH resulting from loss of carbon dioxide from the solution when it is sprayed. Such changes may be avoided by reducing the carbon dioxide content of the solution by, for example, heating it to a temperature above 35°C before it is placed in the apparatus or by making the solution from freshly boiled water.

E-1.2 The solution shall be filtered before it is placed in the reservoir of the apparatus, in order to remove any solid matter which might block the apertures of the spraying device.

E-2 APPARATUS

The apparatus shall comprise the following components.

E-2.1 Spray Cabinet made of, or lined with, material resistant to corrosion by the sprayed solution. The cabinet shall have a capacity of not less than 0.2 m³ and preferably of not less than 0.4 m³ since, with smaller volumes, difficulties are experienced in ensuring even distribution of spray. The upper parts shall be so shaped that drops of sprayed solution accumulated on them do not fall on specimens being tested.

The size and shape of the cabinet shall be such that the quantity of solution collected in the cabinet is within the limits stated in E-5.

E-2.2 Means of Supplying and Controlling Heat, adequate to maintain the cabinet and its contents at the specified temperature (see E-5). The temperature shall be controlled by a thermostat element placed either within the cabinet at least 100 mm from the walls or in a water jacket on the cabinet. In either case the thermometer, capable of being read from the outside, shall be placed within the cabinet at least 100 mm from the walls.

E-2.3 Means for Spraying the Salt Solution, comprising a supply of clean air of controlled pressure and humidity, a reservoir to contain the solution to be sprayed and one or more atomizers made of material resistant to the solution.

The compressed air supply to the atomizers shall be passed through a filter to remove all traces of oil or solid matter and shall be at a pressure of 70 to 170 kPa¹⁾. In order to prevent evaporation of water from the sprayed droplets, the air shall be humidified before entering the atomizer by passage through a saturation tower containing water at a temperature several degrees higher than that of the cabinet. The appropriate temperature depends on the pressure used and on the type of atomizer nozzle and shall be adjusted so that the rate of collection of spray in the cabinet and the concentration of the collected spray are kept within the specified limits (see E-5).

The reservoir to contain the solution to be sprayed shall be a tank made of material resistant to the solution and shall be provided with means of maintaining a constant level of solution in the reservoir.

The atomizers shall be made of inert material, for example glass or plastics material. Baffles may be used to prevent direct impingement of spray on the test specimens and the use of adjustable baffles is helpful in obtaining uniform distribution of spray throughout the cabinet.

¹⁾ 1 kPa = 1 kN/m² = 0.01 atm.

E-2.4 Suitable Collecting Devices (at least two), which shall be funnels of glass or other chemically inert material with the stems inserted into graduated cylinders or other containers. Funnels with a diameter of 100 mm have a collecting area of approximately 80 cm². The collecting devices shall be placed in the zone of the cabinet where the test specimens are placed, one close to an inlet of spray and one remote from an inlet. They shall be so placed that only spray and not liquid falling from specimens or from parts of the cabinet is collected.

NOTE — If the equipment has been used for a spray test or for any other purpose with solution differing from that specified for the test to be carried out, it shall be thoroughly cleaned before use.

E-3 TEST SPECIMENS

The number and type of test specimens, their shape and their dimensions shall be selected according to the specification covering the coating or product being tested. When not so specified, details concerning the specimens shall be mutually agreed between the interested parties.

E-3.1 The specimens shall be thoroughly cleaned before testing. The cleaning method employed shall depend on the nature of the surface and the contaminants, and shall not include the use of any abrasives or solvents which may attack the surface of the specimens.

Care shall be taken that specimens are not recontaminated after cleaning by excessive or careless handling.

E-3.2 If test specimens are cut from a larger coated article, the cutting shall be carried out in such a way that the coating is not damaged in the area adjacent to the cut. Unless otherwise specified, the cut edges shall be adequately protected by coating them with a suitable medium, stable under the conditions of the test, such as paint, wax or adhesive tape.

E-4 METHOD OF EXPOSURE OF TEST SPECIMENS

The specimens shall be so placed in the cabinet that they are not in the direct line of travel of spray from the atomizer. Baffles may be used to prevent direct impact of the sprayed solution on the specimens.

The angle at which the sample is exposed in the cabinet is very important. The surface shall, in principle, be flat and placed in the cabinet facing upwards at an angle as close as possible to 20° to the vertical. This angle shall, in all cases, be within the limits 15° to 30°.

In the case of irregular surfaces, for example entire components, these limits shall be adhered to as closely as possible.

The specimens shall be so arranged that they do not come into contact with one another or with the cabinet and that surfaces to be tested are exposed to free circulation of spray. Specimens may be placed at different levels within the cabinet as long as the solution cannot drip from specimens or their supports at one level onto other specimens placed below.

The support for the specimens shall be made of inert non-metallic material such as glass, plastics or suitably coated wood. If it is necessary to suspend test specimens, the material used shall on no account be metallic and shall be synthetic fibre, cotton thread or other inert insulating material.

E-5 OPERATING CONDITIONS

The temperature inside the spray cabinet shall be $35 \pm 2^\circ\text{C}$ with the minimum possible fluctuation throughout the cabinet during the test.

The solution collected in each of the collecting devices (see E-2.4) shall have a sodium chloride concentration of $50 \pm 10 \text{ g/l}$ and a pH value in the range 6.5 to 7.2 (see E-1.1).

The average rate of collection of solution in each device measured over a minimum period of 24 h shall be 1 to 2 ml/h for a horizontal collecting area of 80 cm^2 .

Test solution which has been sprayed shall not be reused.

E-6 DURATION OF TESTS

The period of test shall be as designed by the specification covering the coating or product being tested. When not specified, it shall be mutually agreed between the interested parties.

Recommended periods of exposure are 2 h — 6 h — 24 h — 48 h — 96 h — 240 h — 480 h — 720 h.

Spraying shall not be interrupted during the prescribed test period. The cabinet shall be opened only for brief visual inspections of the test specimens in position and for replenishing the salt solution in the reservoir if such replenishing cannot be carried out from outside the cabinet.

If the end-point of the test depends on the appearance of the first sign of corrosion, the specimens will need to be inspected frequently. For this reason such specimens shall not be tested together with other specimens requiring tests of predetermined duration.

A periodic visual examination of specimens under test for a predetermined period may be made but the surfaces under test shall not be disturbed and the period for which the cabinet is open shall be the minimum necessary to observe and record any visible changes.

E-7 CLEANING OF SPECIMENS AFTER TEST

At the end of the test period, remove the specimens from the cabinet. Allow the specimens to dry for 0.5 to 1 h before rinsing, in order to reduce the risk of removing corrosion products. Before they are examined, carefully remove residues of the spray solution from their surfaces. A suitable method is to rinse or dip the specimens gently in clean running water at a temperature not exceeding 40°C and then to dry them immediately in a steam of compressed air at a pressure not exceeding 200 kPa, at a distance to approximately 300 mm.

E-8 EVALUATION OF RESULTS

Many different criteria for the evaluation of the results of the test may be applied to meet particular requirements for example change in mass alteration revealed by micrographic examination or change in mechanical properties. Usually the appropriate criteria will be indicated in the specification for the coating or product tested. For most routine applications of the test, only the following need to be considered:

- a) appearance after test;
- b) appearance after removing superficial corrosion products;
- c) the number and distribution of corrosion defects, that is, pits, cracks, blisters; and
- d) the time elapsing before the appearance of the first sign of corrosion.

E-9 TEST REPORT

E-9.1 The test report shall indicate the outcome of the test according to the criteria for evaluation of results prescribed for the test. The result obtained for each specimen tested and, when appropriate, the average result for a group of replicate test specimens shall be reported. The report may, if required, be accompanied by photographic records of the tested specimens.

The report shall contain information about the test procedure. The information may vary according to the purposes of the test and to the direction prescribed for it but a general list of the details likely to be required is as follows:

- a) the description of the coating or product tested;
- b) dimensions and shape of the test specimen and the nature and area of the surface tested;
- c) preparation of the test specimen, including any cleaning treatment applied and any protection given to edges or other special areas;
- d) known characteristics of any coating, with an indication of the surface finish;

- e) the number of test specimens subjected to the test representing each coating or product;
- f) the method used to clean test specimens after the test with, when appropriate, an indication of the loss in mass resulting from the cleaning operation;
- g) the angle at which the tested surfaces were inclined;
- h) the test temperature;
- j) the duration of test;
- k) the properties of any test panels placed in the cabinet expressly to check the correctness of the operating conditions and the results obtained with them.

ANNEX F

(Clause 9.4.2)

POROSITY TEST

F-1 PRINCIPLE

Exposure of a test specimen to a moist atmosphere containing a low concentration of sulphur dioxide causes no corrosion of tin but causes spots of corrosion product to appear at discontinuities in the coating.

NOTE — If the sulphur dioxide concentration in the atmosphere is too high, the corrosion product formed is too fluid to permit easy observation of pore sites. The method given here ensures suitable conditions for the development of immobile corrosion products at discontinuities.

F-2 APPARATUS

F-2.1 Test Cabinet

F-2.1.1 Capacity

The capacity of the test cabinet should preferably be 300 ± 10 litres (see Note).

NOTE — Cabinet of capacities other than 300 ± 10 litres may be used provided that the other test conditions to which the test specimens as submitted are the same. The details and instructions given in this appendix are, however, appropriate to cabinets of the preferred capacity and will require corresponding modifications for cabinets of other capacities.

F-2.1.2 Construction and Components

The test cabinet shall have a door or hood capable of being closed hermetically, and shall be fitted with the following components:

- a) *temperature-controlling device*, with its actuating element placed in the upper part of the cabinet;
- b) *thermometer*, capable of being read from outside the cabinet with its bulb 150 mm from the roof and door/hood and 250 mm from one side;
- c) *inlet tube*, through which gas can be introduced into the cabinet, placed approximately 50 mm above the base;
- d) *valve*, by which excess pressure can be relieved, placed in or near the roof of the cabinet;

- e) *drain-cock*, in the floor of the cabinet; and
- f) *heating device*, capable of heating the cabinet to a temperature of $40 \pm 3^\circ\text{C}$ in 1.5 h and of maintaining the interior at this temperature.

NOTE — A 1 kW electrically-heated hotplate is suitable for the preferred size of cabinet.

Typical cabinets, with a door or hood are shown in Fig. 1 and 2 respectively.

F-2.1.3 Materials of Construction

All the materials used in the construction of the cabinet shall be resistant to the action of moist sulphur dioxide and shall themselves not emit any gas or vapour likely to influence corrosion of the test specimens.

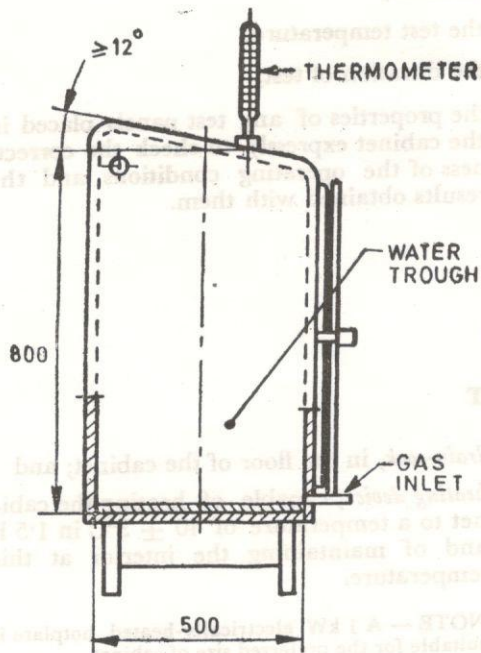
The floor and lower parts of the walls shall be capable of being heated and shall be able to retain without leakage at least 2.5 litres of water containing dissolved sulphur dioxide, this volume being required to flush the apparatus. Lead-clad constructional material is suitable for these parts and for the framework and fittings of the cabinet, but the greater part of the walls and door/hood shall be made of transparent sheet, such as glass or suitable plastics material.

F-2.1.4 Shape

Some variation in the shape of the cabinet can be tolerated, but the roof shall be shaped so that moisture condensing on it does not fall on test material in the cabinet. An inclination of the roof of approximately 12° to the horizontal provides a suitable safeguard.

F-2.1.5 Ambient Conditions

The cabinet shall be installed in a room in which there is a clean atmosphere and shall be protected from large or rapid temperature fluctuations, strong direct sunlight and draughts.



All dimensions in millimetres.

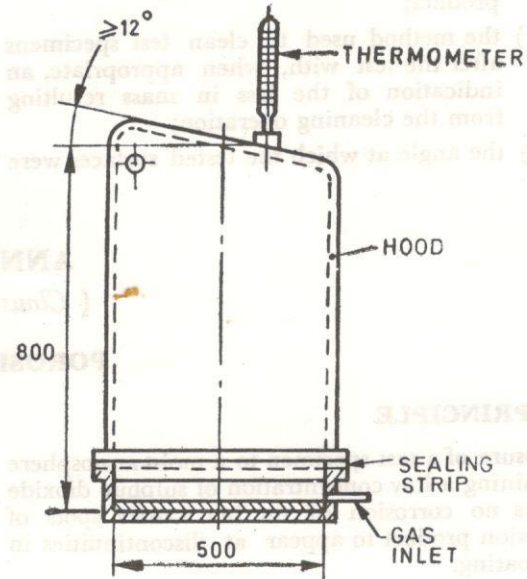
FIG. 1 CABINET WITH DOOR

F-2.1.6 Conditioning of Apparatus

To reduce risks of contamination of the atmosphere by vapours from constructional materials, a new cabinet shall be operated at least once, without introduction of test specimens, by the procedure applicable to an atmosphere containing 2 litres for sulphur dioxide, before it is brought into use for testing.

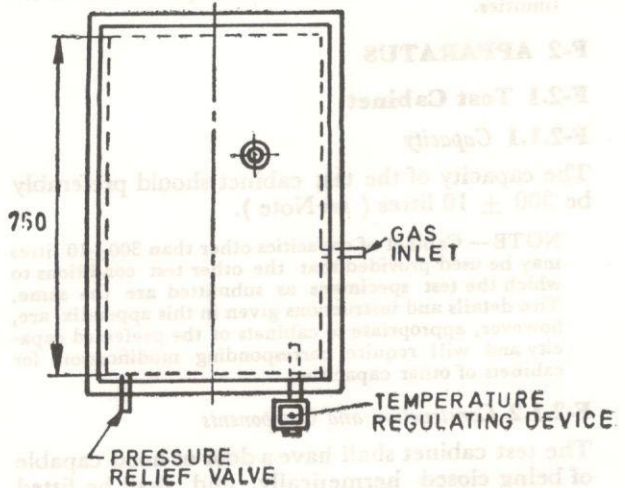
F-2.2 Test Specimen Supports

The supports for the test specimens shall be of non-metallic material such as glass, suitable



All dimensions in millimetres.

FIG. 2 HOODED CABINET



All dimensions in millimetres.

FIG. 2 HOODED CABINET

plastics material or suitably coated wood. Any material used to suspend the test specimens in the cabinet shall be of suitable synthetic fibre or other inert insulating material; metallic materials shall not be used.

F-2.3 Source and Means of Measuring Volume of Sulphur Dioxide

A container of liquid sulphur dioxide is the usual source, but the gas may be generated in a suitable apparatus by treating sodium sulphite with sulphuric acid. Sulphur dioxide shall not be

generated inside the cabinet. The volume of gas delivered into the chamber may be measured by any suitable method, for example:

- a) by means of a gas burette using viscous liquid paraffin as the pressure-controlling fluid; as the volume to be measured is usually about 0.2 litre, measures shall be taken to avoid errors from causes such as air contained in delivery tubes between the burette and the chamber;
- b) by means of a gas jar of known volume filled with sulphur dioxide and opened in the chamber;
- c) by means of a calibrated flowmeter.

F-3 CONSTRUCTION AND CLEANING OF TEST SPECIMENS

F-3.1 If test specimens are cut from a larger coated article, the cutting shall be carried out in such a way that the coating is not damaged, especially in the area adjacent to the cut. Unless otherwise specified, the cut edges shall be adequately protected by coating them with a suitable medium that is stable under the conditions of the test, such as wax or adhesive tape.

F-3.2 The test specimens shall be thoroughly cleaned before testing. The cleaning method employed shall depend on the nature of the surface and the contaminants, but shall not include the use of any abrasives, solvents or other materials that may attack the surface of test specimens. Care shall be taken that the test specimens are not recontaminated, after cleaning, by excessive or careless handling.

NOTE — The number and type of test specimens and their shape and dimensions are not specified, these should be agreed between interested parties (see also F-4.3 and F-4.4).

F-4 METHOD OF EXPOSURE OF TEST SPECIMENS

F-4.1 When in position in the cabinet, the test specimens shall be located so that no part of any specimen is within 20 mm of another specimen or within 100 mm of the walls or the roof of the cabinet or within 200 mm of the surface of the water in the base of the cabinet.

F-4.2 The test specimens shall be so arranged that any moisture condensing on any of them or on their supports does not fall on other specimens placed at lower levels.

F-4.3 The orientation of the exposed test surface is critical, and in the case of flat surfaces, the angle of inclination to the vertical, unless otherwise specified, shall be $15 \pm 2^\circ$.

F-4.4 The total exposed surface area of the specimen or specimens tested at any one time shall be substantially the same and, unless otherwise agreed, shall be $0.5 \pm 0.1 \text{ m}^2$ for the cabinet of preferred capacity (see F-2.1.1) and proportionately changed for different sizes of cabinet.

F-5 PROCEDURE

F-5.1 Introduce 2 ± 0.2 litres of distilled water into the base of the cabinet.

NOTE — The quantity of water in the cabinet depends on its size and shape. A proportional change for cabinet of different size is valid only for cabinets of similar shape.

F-5.2 Place the test specimens in position and close the door/hood of the cabinet completely.

F-5.3 Introduce 0.2 litre of the sulphur dioxide into the chamber.

F-5.4 Switch on the heater and raise the temperature inside the cabinet to $40 \pm 3^\circ\text{C}$ in approximately 1.5 h. Maintain heating, under control, so as to keep the temperature inside the cabinet at $40 \pm 3^\circ\text{C}$ for 24 h.

F-6 INSPECTION AND CLEANING OF SPECIMENS AFTER TEST

At the end of the 24 h test duration, remove the test specimens from the cabinet and, before they are examined, allow them to hang freely in a normal indoor atmosphere until any liquid corrosion products have solidified. Examine all the test specimens first with all corrosion products in position. Any cleaning subsequently carried out shall depend on the criteria laid down for evaluation of the result of the test.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act, 1986* to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc : No. MTD 20 (3572).

Amendments Issued Since Publication

| Amend No. | Date of Issue | Text Affected |
|-----------|---------------|---------------|
| | | |
| | | |
| | | |
| | | |

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 323 01 31, 323 94 02, 323 33 75

Telegrams: Manaksanstha
(Common to
all offices)

Regional Offices:

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg
NEW DELHI 110002

Telephone

{ 323 76 17
323 38 41

Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola
CALCUTTA 700054

{ 337 84 99, 337 85 61
337 86 26, 337 86 62

Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022

{ 60 38 43
60 20 25

Southern : C. I. T. Campus, IV Cross Road, CHENNAI 600113

{ 235 02 16, 235 04 42
235 15 19, 235 23 15

Western : Manakalaya, E9 MIDC, Marol, Andheri (East)
MUMBAI 400093

{ 832 92 95, 832 78 58
832 78 91, 832 78 92

Branches : AHMADABAD. BANGALORE. BHOPAL. BHUBANESHWAR.
COIMBATORE. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR.
KANPUR. LUCKNOW. NAGPUR. PATNA. PUNE. THIRUVANANTHAPURAM.