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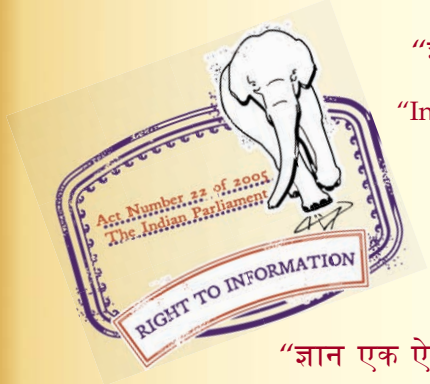

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IS : 12434 - 1988
(Reaffirmed 2004)

Indian Standard

SPECIFICATION FOR COATING/PLATING THICKNESS TESTER, DESTRUCTIVE TYPE

(First Reprint MAY 1997)

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

Gr 2

May 1989

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(Reaffirmed 1993)

Indian Standard

SPECIFICATION FOR COATING/PLATING THICKNESS TESTER, DESTRUCTIVE TYPE

0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 28 June 1988, after the draft finalized by the Industrial Process Measurement and Control Sectional Committee had been approved by the Electrotechnical Division Council.

0.2 Destructive coating/plating thickness tester is a versatile and sophisticated instrument for the measurement of coatings of metals as well as alloys applied on a substrate by electroplating or other methods. It is generally known as destructive thickness tester, coulometric type.

0.3 While preparing this standard, assistance has been derived from ASTM B 504-1982 Measurement of Thickness of Metallic Coatings by the Coulometric Method, issued by the American Society for Testing and Materials.

0.4 For the purpose of deciding whether a particular requirement of this standand is complied with, the final value, observed or calculated, expressing the result of a test, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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

1. SCOPE

1.1 This standard covers the requirements of coulometric type destructive thickness tester for measurement of coating thickness up to 100 μm.

2. OPERATING PRINCIPLE

2.1 As commonly practiced, the method employs a small metal cell which is filled with an appropriate electrolyte. The test specimen serves as the bottom of the cell, and an insulating gasket between the cell and the specimen defines the test area (about 0·1 cm^2). With the test specimen as anode and the cell as cathode, a constant direct current is passed through the cell until the coating has dissolved, at which time a sudden change in voltage occurs.

2.2 An accurate pulse generator-counter records duration of the dissolution time. The electrolytes are standard solution having nearly 100 percent anode efficiency. Vigorous agitation of the solution ensures this efficiency. Solutions shall not have any chemical attack on the coatings.

2.3 The thickness of the coating may be calculated from the quantity of electricity used (current multiplied by time), the area, the electrochemical equivalent of the coating metal, the anodic-current efficiency, and the density of the coating. Alternatively, the equipment may be calibrated against standards with known coating thickness.

3. WORKMANSHIP AND FINISH

3.1 The apparatus shall be robust in construction and have a good finish.

3.2 Paint, lacquering or any other corrosion resistant coating shall be smooth so that cleaning of accumulated dust, etc, is possible.

3.3 All exposed terminals shall be plated with thick nickel to prevent corrosion.

3.4 Cell Assembly and Etching Cell — The cell assembly shall be such as can be lowered or raised or could be moved sideways to adjust its position according to sample piece.

The cell shall have capacity to hold enough electrolyte to dissolve the coating.

4. GENERAL REQUIREMENTS

4.1 Supply — Tester shall work on ac mains or battery supply.

4.2 Weight—Weight shall not be more than 12 kg.

4.3 Accuracy — Accuracy shall not be less than the following values:

Thickness μm*	*Minimum Accuracy* percent
0 – 1	±15
2 – 15	±10
16 – 50	± 5
51 – 100	± 5

*1 μm = 1 micron.

4.4 Readability — Readability shall be atleast 0·01 μm for thickness below 10 μm and 0·1 μm above 10 μm. It is recommended that area of deplating shall approximately be 10 mm^2 (circular) and electrolyte required shall be approximately 2 cc.

4.5 Type of Coating — Tester shall be able to measure almost any metallic coating on any base/. substrate material, like thickness of nickel, tin, zinc, chromium, cadmium, copper, silver, gold on stainless steel, brass, copper, non-metal, etc.

5. FACTORS AFFECTING THE ACCURACY OF METHOD

5.1 Composition of Electrolytes—Electrolytes used for coulometric thickness measurements shall permit the coating metal to dissolve at a constant anodic-current efficiency (preferably 100 percent); they shall have a negligible spontaneous chemical effect on the coating metal and shall so differentiate electrochemically between the coating and the substrate that a suitably sharp and large voltage change occurs at the endpoint of the test.

5.1.1 Electrolytes furnished with commercial instruments may be presumed to meet these requirements; others must be evaluated before use by testing standards having known thicknesses (*see* **7.1.1**).

5.2 Current Variation — For coulometric instruments employing the constant-current technique, variation of the current during a test will result in errors. For instruments using a current-time integrator, variation of the current during a test will not result in error, unless the current change is such as to displace the anodic current density beyond the range of constant or 100 percent anodic-current efficiency.

5.3 Area Variation — The accuracy of the thickness measurement will not be better than the accuracy with which the test area is defined or known. Typically, this test area is defined by a flexible, insulating gasket. If excessive pressure is applied to such gaskets, the test area may be altered undesirable.

5.4 Agitation — In most, but not all, coulometric thickness instruments, a relatively high anodic-current density is employed to shorten the time. It is then necessary to agitate the electrolyte to maintain a constant anodic-current efficiency. Where agitation is required, insufficient agitation may result in polarization of the specimen thereby causing a premature and false endpoint.

5.5 Alloying Between Coatings and Metallic Substrates — The measurement of a coating thickness by the coulometric method implicitly assumes that a sharply defined interface exists between the coating and the substrate. If an alloy layer exists between the coating and the substrate, for example, in the case of coatings applied by hot dipping, the coulometric endpoint may occur at some point within the alloy layer, thus giving a high value of the thickness of the unalloyed coating.

5.6 Purity of Coating — Impurities or additives which co-deposit with the coating may change the effective electrochemical equivalent of the coating, and also change the anodic-current efficiency.

5.7 Cleanliness of Test Surface — The surface to be tested shall be cleaned. Oil, grease and organic coatings such as lacquer shall be removed with suitable solvents. Oxides, conversion coatings and corrosion products are preferably removed by carefully burnishing the test surface with a clean, soft pencil eraser. Tin and nickel surfaces, in particular, should be burnished prior to testing to remove passive oxide films.

5.8 Density of Coating—The coulometric method intrinsically measures coating weight per unit area, the equivalent linear thickness being a function of the density of the coating. If the denstiy of the coating tested is different from the value of the density used for the calibration, the linear thickness obtained coulometrically will be different from the actual linear thickness of the coating tested.

5.9 Number and Location of Tests — Since the coulometric test method measures, essentially, a local coating thickness, a single test may not be representative of the coating thickness over the entire significant surfaces.

6. CALIBRATION OF EQUIPMENT

6.1 The equipment shall be calibrated by means of standards having known coating thicknesses. If commercial equipment is used, the manufacturer's instructions shall be followed insofar as they are compatible with this test method.

6.2 Calibration of Direct-Reading Instruments — Direct-reading instruments shall be calibrated against standards having a known coating thickness, and adjusted to produce a correct reading corresponding to the coating thickness of the standard.

NOTE — Accuracy of such standards treacable is ± 5 percent.

6.3 Calibration of Non-Direct-Reading Instruments

6.3.1 Non-direct-reading instruments shall be calibrated against standards having a known coating thickness by using a calibration constant, C, calculated as follows:

C = coating thickness of the standard instrument reading.

6.3.2 The instrument shall be adjusted so that where standards having known coating thicknesses are tested, the correct thickness is obtained by multiplying the instrument reading by the calibration constant, C.

6.4 Thickness Standards—The thickness standards shall consist of the same type of coating and substrate as the unknown specimens to be measured and they shall have an accuracy of ±5 percent or better.

7. PROCEDURE FOR THICKNESS MEASUREMENTS

7.1 If commercial equipment is used, the manufacturers' instructions shall be followed insofar as they are compatible with this test method.

7.1.1 Recommendation for standard solutions is given in Appendix A.

7.2 The test surface shall be cleaned of all foreign material that might affect the measurement.

NOTE 1 — Certain nickel deposits, frequently dull nickel, may exhibit passivity. When such coatings are tested coulometrically, the voltage across the specimen and test cell is markedly higher (approximately 1·4 V) than normal, and the coating does not dissolve. Oxygen is evolved at the specimen and the test may continue indefinitely.

NOTE 2—Removal of the passivity may be accomplished in some cases by mildly abraiding (as with a pencil eraser) the nickel surface prior to testing. Alternatively, the specimen may be made cathodic in the coulometric electrolyte for 10 to 20 s by applying current from an external source. Allowing the nickel to be in contact with 10 percent volume hydrochloric acid for approximately 1 min prior to the test may also be used effectively.

7.3 After completion of the measurement, the test surface shall be examined visually, and if the dissolution of the coating is not virtually complete, the measurement shall be repeated.

8. MARKING

8.1 The following information shall be marked on the exposed panel or nameplate:

a) Name, grade and thickness measurement range or the instrument;

b) Manufacturers' name or trade-mark;

c) Serial Number and type number of the instrument;

d) Country of manufacture;

e) Accuracy;

f) Area of deplating (*see* Note 1);

g) Electrolyte required (*see* Note 2); and

h) Operating voltage.

NOTE 1 — Gaskets with different diameters of deplating are available. Direct reading of thickness for a different area shall be given in the operating manual.

NOTE 2 — List cannot be displayed on exposed panel too long.

8.2 Terminals shall be clearly and indelibly marked with electrical symbols or by lettering, where required [*see* IS : 1248 (Part 1)-1983*].

8.3 The manufacturer shall supply the manual for operation, maintenance and instructions for unpacking, and precautionary measures to be taken.

8.4 The apparatus may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

9. TESTS

9.1 Insulation Resistance Test — Insulation resistance between ac mains terminal and enclosure shall be not less than 50 megohms at 500 volts dc.

9.2 High Voltage Test — No breakdown, arcing or sparking shall occur when 2 000 volts rms is applied between ac mains terminals and enclosures for a period of one minute.

9.3 Accuracy Test — The coating under test shall be measured by the tester described in **6** and **7**. Accuracy shall be not less than specified in **4**.

*Direct acting indicating analogue electrical measuring instruments and their accessories: Part 1 General requirements (*second revision*).

APPENDIX A

(*Clause* 7.1.1)

RECOMMENDED ELECTROLYTES

A-1. Table 1 lists typical electrolyte that have been used for coulometric thickness measurements; however, they may not be necessarily suitable for all types of coulometric instruments.

A-2. Use of these electrolytes is not mandatory for compliance with this standard and when commercial coulometric instruments are used, the manufacturer recommendations shall be followed.

TABLE 1 TYPICAL ELECTROLYTES FOR ELECTRO-DEPOSITED COATINGS

(*Clause* A-1)

COATINGS	SUBSTRATE (BASIS METAL)				
	Steel	Copper and Alloys (such as Brass)	Nickel	Aluminium	Zinc
Cadmium	1, 10	1, 10	1	1	—
Chromium	2, 11	3, 4, 12	2, 13	2, 13	—
Copper	5, 14	—	5, 15	5	16
Lead	17	17	17	—	—
Nickel	6, 18	6, 19	—	6, 18	—
Silver	7	8	7	—	—
Tin	3, 4, 20	3, 4, 20	3, 4	2, 21	—
Zinc	9	9	9	9	—

NOTE — The numbers in the table refer to the following aqueous solutions:

1. 100 g KI/litre with traces of I_2
2. 100 g Na_2SO_4/litre
3. 175 ml HCl (sp gr 1·18)/litre
4. 150 g NaOH/litre
5. 80 g $NaKC_4H_4O_6$ (sodium potassium tartarate) +100 g NH_4NO_3/litre
6. 30 g NH_4NO_3 + 30 g NaCNS/litre
7. 100 g $NaNO_3$ + 3 ml HNO_3 (sp gr 1·42)/litre
8. 180 g KCNS/litre
9. 100 g NaCl or KCl/litre
10. 30 g KCl + 30 g NH_4Cl/litre
11. 100 ml H_3PO_4 (sp gr 1·75) + 10 g CrO_3/litre
12. 100 g Na_2CO_3 [for coatings up to 5 μm (0·2 ml)]/litre
13. 64 ml H_3PO_4 (sp gr 1·75)/litre
14. 800 g NH_4NO_3 + 10 ml NH_4OH (sp gr 0·88)/litre
15. 100 g K_2SO_4 + 20 ml H_3PO_4 (sp gr 1·75)/litre
16. Pure H_2SiF_6 solution containing not less than 30 percent H_2SiF_6 (slightly weaker acid may be used, if some $MgSiF_6$ is added to the solution)
17. 200 g CH_3COONa + 200 g CH_3COONH_4/litre
18. 800 g NH_4NO_3 + 3·8 g $CS(NH_2)_2$ (thiourea)/litre
19. 100 ml HCl (sp gr 1·18)/litre
20. 100 g KNO_3 + 100 g KCl/litre
21. 50 ml H_2SO_4 (sp gr 1·84) + 5 g KF/litre.

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