



## Standard Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method<sup>1</sup>

This standard is issued under the fixed designation B 504; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the Department of Defense.*

### 1. Scope

1.1 This test method covers the determination of the thickness of metallic coatings by the coulometric method, also known as the anodic solution or electrochemical stripping method.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

#### 2.1 ISO Standard:

ISO 2177 Metallic Coatings—Measurement of Coating Thickness—Coulometric Method by Anodic Dissolution<sup>2</sup>

### 3. Summary of Test Method

3.1 The thickness of the coating is determined by measuring the quantity of electricity (coulombs) required to dissolve the coating anodically from a known and accurately defined area.

3.2 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<sup>2</sup>). 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.

3.3 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 thicknesses.

3.4 Commercial instruments using this principle are avail-

able. The method is rapid and versatile, but destructive to the coating. In general, its range is considered to be between 0.75 and 50  $\mu\text{m}$ . Chromium, gold, tin, and other coatings can be measured down to 0.075  $\mu\text{m}$ .

### 4. Significance and Use

4.1 Measurement of the thickness of a coating is essential to assessing its utility and cost.

4.2 The coulometric method destroys the coating over a very small (about 0.1 cm<sup>2</sup>) test area. Therefore its use is limited to applications where a bare spot at the test area is acceptable or the test piece may be destroyed.

### 5. Factors Affecting the Accuracy of the Method

5.1 *Composition of Electrolytes*—Electrolytes used for coulometric thickness measurements must permit the coating metal to dissolve at a constant anodic-current efficiency (preferably 100 %); they must have a negligible spontaneous chemical effect on the coating metal and must so differentiate electrochemically between the coating and the substrate that a suitably sharp and large voltage change occurs at the end point 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. Appendix X1 lists some electrolytes and coating-substrate combinations that have been used with some instruments.

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 % 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. Area variation is usually minimized by using as large an area as practical and by using a constant pressure device. If excessive pressure is applied to such a gasket, the test area may be altered undesirably.

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<sup>2</sup> Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.



5.4 *Agitation*—In most, but not all, coulometric thickness measurements, a relatively high anodic-current density is employed to shorten the test 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 as, for example, in the case of coatings applied by hot dipping, the coulometric end-point 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 that codeposit with the coating may change the effective electrochemical equivalent of the coating and also change the anodic current efficiency.

5.6.1 *Alloy Coating*—Variations in the composition of alloy coatings will change the effective electrochemical equivalent of the coating.

5.7 *Cleanliness of Test Surface*—The surface to be tested must be clean. Oil, grease, and organic coatings such as lacquer must 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 so burnished prior to testing to remove passive oxide films.

5.8 *Density of Coating*—The coulometric method intrinsically measures coating mass per unit area, the equivalent linear thickness being a function of the density of the coating. If the density 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.

5.8.1 *Density of Alloy Coatings*—Variation in the composition of alloy coatings will change the density of the coating.

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 surface.

## 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 known coating thicknesses, and adjusted to produce correct readings corresponding to the coating thicknesses of the standard.

6.3 *Calibration of Nondirect-Reading Instruments:*

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

$$C = \text{coating thickness of the standards/instrument reading} \quad (1)$$

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 specimens to be measured, and they shall have an accuracy of  $\pm 5\%$  or better.

## 7. Procedure for Making Measurements

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

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 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 abrading (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% 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 discarded and repeated.

## 8. Precision and Bias

8.1 The equipment, its calibration, and its operation shall be such that the coating thickness can be determined with an uncertainty of less than 10%.

8.2 Instruments suitable for compliance with 8.1 are available commercially. For many coating systems the instruments are capable of making measurements with an uncertainty of less than 4% (95% confidence).

8.3 Although an uncertainty of less than 10% may be achieved consistently for a great number of coating-substrate combinations, the uncertainty may be greater when the coating thickness is less than 1  $\mu\text{m}$  or exceeds 50  $\mu\text{m}$ .

8.4 The bias of a coulometric measurement is the discrepancy remaining between the measured thickness and the true thickness if all random errors are eliminated. It is, therefore, no greater than, and attributable to (1), the calibration error of the instrument and (2) the quality of the calibration standard used to calibrate the instrument.

**APPENDIX**
**(Nonmandatory Information)**
**X1. ELECTROLYTES**

X1.1 Table X1.1 lists electrolytes that have been used for coulometric thickness measurements; however, they are not necessarily suitable for use with all types of coulometric instruments.

X1.2 Use of these electrolytes is not mandatory for compliance with this method, and when commercial coulometric

instruments are used, the manufacturer's recommendations shall be followed.

X1.3 Table X1.2 lists other coating-substrate combinations that have been used successfully with commercially available electrolytes.

**TABLE X1.1 Typical Electrolytes for Electrodeposited Coatings<sup>A</sup>**

Coating	Substrate (Basis Metal)				
	Steel	Copper and Alloys (such as brass)	Nickel	Aluminum	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	...

<sup>A</sup> The numbers in the table refer to the following aqueous solutions:

- (1) 100 g KI/L, with traces of I<sub>2</sub>
- (2) 100 g Na<sub>2</sub>SO<sub>4</sub>/L
- (3) 175 mL HCl (sp gr 1.18)/L
- (4) 150 g NaOH/L
- (5) 80 g NaKC<sub>4</sub>H<sub>4</sub>O<sub>6</sub> (sodium potassium tartrate) + 100 g NH<sub>4</sub>NO<sub>3</sub>/L
- (6) 30 g NH<sub>4</sub>NO<sub>3</sub> + 30 g NaSCN/L
- (7) 100 g NaNO<sub>3</sub> + 3 ml HNO<sub>3</sub> (sp gr 1.42)/L
- (8) 180 g KSCN/L
- (9) 100 g NaCl or KCl/L
- (10) 30 g KCl + 30 g NH<sub>4</sub>Cl/L
- (11) 100 mL H<sub>3</sub>PO<sub>4</sub> (sp gr 1.75) + 10 g CrO<sub>3</sub>/L
- (12) 100 g Na<sub>2</sub>CO<sub>3</sub> [for coatings up to 5 μm (0.2 mil)]/L
- (13) 64 mL H<sub>3</sub>PO<sub>4</sub> (sp gr 1.75)/L
- (14) 800 g NH<sub>4</sub>NO<sub>3</sub> + 10 mL NH<sub>4</sub>OH (sp gr 0.88)/L
- (15) 100 g K<sub>2</sub>SO<sub>4</sub> + 20 mL H<sub>3</sub>PO<sub>4</sub> (sp gr 1.75)/L
- (16) Pure H<sub>2</sub>SiF<sub>6</sub> solution containing not less than 30 % H<sub>2</sub>SiF<sub>6</sub> (Slightly weaker acid may be used, if some MgSiF<sub>6</sub> is added to the solution.)
- (17) 200 g CH<sub>3</sub>COONa + 200 g CH<sub>3</sub>COONH<sub>4</sub>/L
- (18) 800 g NH<sub>4</sub>NO<sub>3</sub> + 3.8 g CS(NH<sub>2</sub>)<sub>2</sub> (thiourea)/L
- (19) 100 mL HCl (sp gr 1.18)/L
- (20) 100 g KNO<sub>3</sub> + 100 g KCl/L
- (21) 50 mL H<sub>2</sub>SO<sub>4</sub>(sp gr 1.84) + 5 g KF/L

**TABLE X1.2 Measurable Coating-Substrate Combinations**

Substrate Coating	Aluminum	Copper and Copper Alloys	Nickel	Steel	Magnetic Stainless Steel	Nonmetallic
Brass <sup>A</sup>	X			X		X
Cobalt		X		X		
Gold		X	X			
Indium		X	X	X		
Iron		X				
Nickel (Electrodes) <sup>A</sup>	X			X		X
Nickel-Iron <sup>A</sup>		X		X		
Tin-Lead <sup>A</sup>	X	X		X		X
Tin-Nickel <sup>A</sup>		X		X		
Tin-Zinc <sup>A</sup>		X		X		X

<sup>A</sup> The measurement accuracy of these alloy coatings is dependent on the composition of the coating.

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