



## Standard Guide for Measurement of Electrodeposited Metallic Coating Thicknesses by the Dropping Test<sup>1</sup>

This standard is issued under the fixed designation B 555; 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.

### 1. Scope

1.1 This guide covers the use of the dropping test to measure the thickness of electrodeposited zinc, cadmium, copper, and tin coatings.

NOTE 1—Under most circumstances this method of measuring coating thicknesses is not as reliable or as convenient to use as an appropriate coating thickness gage (see Test Methods B 499, B 504, and B 568).

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 ASTM Standards:

B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section<sup>2</sup>

B 499 Test Method for Measurement of Coating Thicknesses by the Magnetic Method: Nonmagnetic Coatings on Magnetic Basis Metals<sup>2</sup>

B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method<sup>2</sup>

B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry<sup>2</sup>

D 1193 Specification for Reagent Water<sup>3</sup>

### 3. Summary of Guide

3.1 A dropping test<sup>4</sup> consists of applying dropwise a corrosive solution, at a constant rate, to the electroplated surface, and measuring the time required to penetrate the coating and expose the substrate; this time is proportional to the coating thickness.

### 4. Significance and Use

4.1 The thickness of a metal coating is often critical to its performance.

4.2 This procedure is useful for an approximate determination when the best possible accuracy is not required. For more reliable determinations, the following methods are available: Test Methods B 487, B 499, B 504, and B 568.

4.3 This test assumes that the rate of dissolution of the coating by the corrosive reagent under the specified conditions is always the same.

### 5. Factors Affecting the Accuracy

5.1 The following factors will affect the accuracy of a coating thickness measurement made by this method:

5.1.1 *Cleanliness of Surface*—Any foreign material on the surface to be tested, including lacquer, grease, corrosion products, and conversion coatings, will interfere with the test and must be removed. Tarnish and conversion coatings can often be removed by mild burnishing with a soft, clean pencil eraser.

5.1.2 *Concentration of Test Solution*—Variation from the stated concentrations will introduce an error unless the thickness factor is adjusted accordingly. Test solutions cannot be reused.

5.1.3 *Temperature*—The thickness factor for a given solution, being a function of the temperature, must be known for the temperature at which the test is made. Specimens to be tested should be allowed to reach room temperature before testing.

5.1.4 *Dropping Rate*—The thickness factor is also a function of the dropping rate. The thickness factors given in Section 10 are for a dropping rate of 100 drops/min and will remain valid for the range of 95 to 105 drops/min.

5.1.5 *Solution Drainage*—The thickness factors given in Section 10 are valid only if the test solution is rapidly drained off, as from a flat surface inclined 45° from the horizontal. The test cannot be made on a horizontal surface or at a location that does not permit rapid drainage.

5.1.6 *Drop Size*—Variation in drop size may alter the penetration rate. The tip of the dropping apparatus should conform to the dimensions given in Fig. 1 and should be kept clean.

5.1.7 *Alloy Layer*—The presence of an alloy layer at the

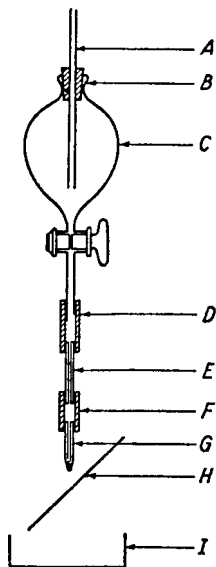
<sup>1</sup> This guide is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.10 on General Test Methods.

Current edition approved January 31, 1986. Published March 1986. Originally published as B 555 – 71. Last previous edition B 555 – 75 (1980) <sup>$\epsilon$ 1</sup>.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 02.05.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>4</sup> Such tests were described by Hull, R. O., and Strausser, P., *Monthly Review*, American Electroplaters Society, MRAEA, Vol 22, 1935, p. 9, and by Brenner, A., *Proceedings*, American Electroplaters Society, AEPPB, Vol 27, 1939, p. 204.



- A—Glass tube with one end open to atmosphere and one end submerged in the test solution in funnel, C.
- B—Rubber stopper forms air tight seal between tube, A, and funnel, C.
- C—Separatory funnel.
- D—Wide-bore rubber tubing (75 to 125 mm in length) or sealed glass joints.
- E—Capillary tubing (140 mm in length, 0.64-mm bore).
- F—Rubber tubing or sealed glass joints.
- G—Capillary glass tip (with 12.7-mm taper to outside diameter of 3.5 mm).
- H—Specimen.
- I—Collector of spent solution.

NOTE 1—Solution head is constant as long as air is being drawn through tube, A.

FIG. 1 Dropping Funnel

coating-substrate interface may obscure the end point and introduce an uncertainty as to the amount of alloy included in the thickness measurement.

5.1.8 *Detection of End Point*—The end point (the time at which the coating has been penetrated and the substrate exposed) is characterized by a change in the appearance of the surface on which drops are falling. This is a matter of personal judgment and may not always be clearly defined, depending upon the coating-substrate combination and the extent of alloying, if any. Such errors may be minimized by standardizing the test method with identical specimens with a known coating thickness determined by other means.

5.1.9 *Composition of Coating*—The dropping tests described in this document are intended for use on nominally “pure” coatings. The thickness factors listed in Section 10 are expected to vary with gross variations in the composition of the coating, as might result from codeposition of an alloying component. The specific effects, on the thickness factors, of impurities or of inclusions from brighteners or other addition agents can be appreciable. Uncertainty from these sources may be minimized by standardizing the test method against standards, prepared from the same type of plating solution, the thicknesses of which have been determined by other methods.

## 6. Apparatus

6.1 Fig. 1 illustrates one form of apparatus used for dropping tests. Equivalent apparatus are commercially available, as is a solenoid operated unit with a digital readout.

## 7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in the preparation of all test solutions.

7.2 *Purity of Water*—Water used in the preparation of test solutions shall be reagent water as defined in Specification D 1193.

## 8. Test Solutions

### 8.1 Solution for Cadmium and Zinc:

Chromic acid (CrO <sub>3</sub> )	200 g/L
Sulfuric acid (96 % H <sub>2</sub> SO <sub>4</sub> , sp gr 1.84)	50 g/L or 27 mL/L

NOTE 2—Sulfuric acid is added slowly with stirring to at least 20 times its volume of water.

### 8.2 Solution for Cadmium only:

Ammonium nitrate (NH <sub>4</sub> NO <sub>3</sub> )	110 g/L
Hydrochloric acid (37 % HCl, sp gr 1.19)	10 mL/L

### 8.3 Solution for Zinc only:

Ammonium nitrate (NH <sub>4</sub> NO <sub>3</sub> )	100 g/L
Nitric acid (70 % HNO <sub>3</sub> , sp gr 1.42)	55 mL/L

### 8.4 Solution for Tin:

Trichloroacetic acid (CHCl <sub>3</sub> COOH)	100 g/L
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### 8.5 Solution for Copper:

Hydrochloric acid (37 % HCl sp gr 1.19)	200 mL/L
Antimony trioxide (Sb <sub>2</sub> O <sub>3</sub> )	20 g/L
Glacial acetic acid (99.7 % CH <sub>3</sub> COOH)	250 mL/L
Ferric chloride (FeCl <sub>3</sub> ·6H <sub>2</sub> O)	450 g/L

NOTE 3—For ease of preparation, dissolve the antimony trioxide in the hydrochloric acid, add 150 mL of water and the glacial acetic acid, and then add the ferric chloride in small portions. Finally dilute to 1.00 L with water.

NOTE 4—Variations of ±2 % in the composition of a test solution are permissible.

## 9. Procedure

9.1 Clean the specimen and allow it and the test solution to reach room temperature, which should be recorded. Support the specimen so that the surface to be tested makes a 45° angle with the horizontal, about 13 mm below the tip of the dropping apparatus. Drop the test solution onto the specimen at a rate of 100 drops/min. Note and record the time required for the

solution to penetrate the coating at the point on which the drops fall.

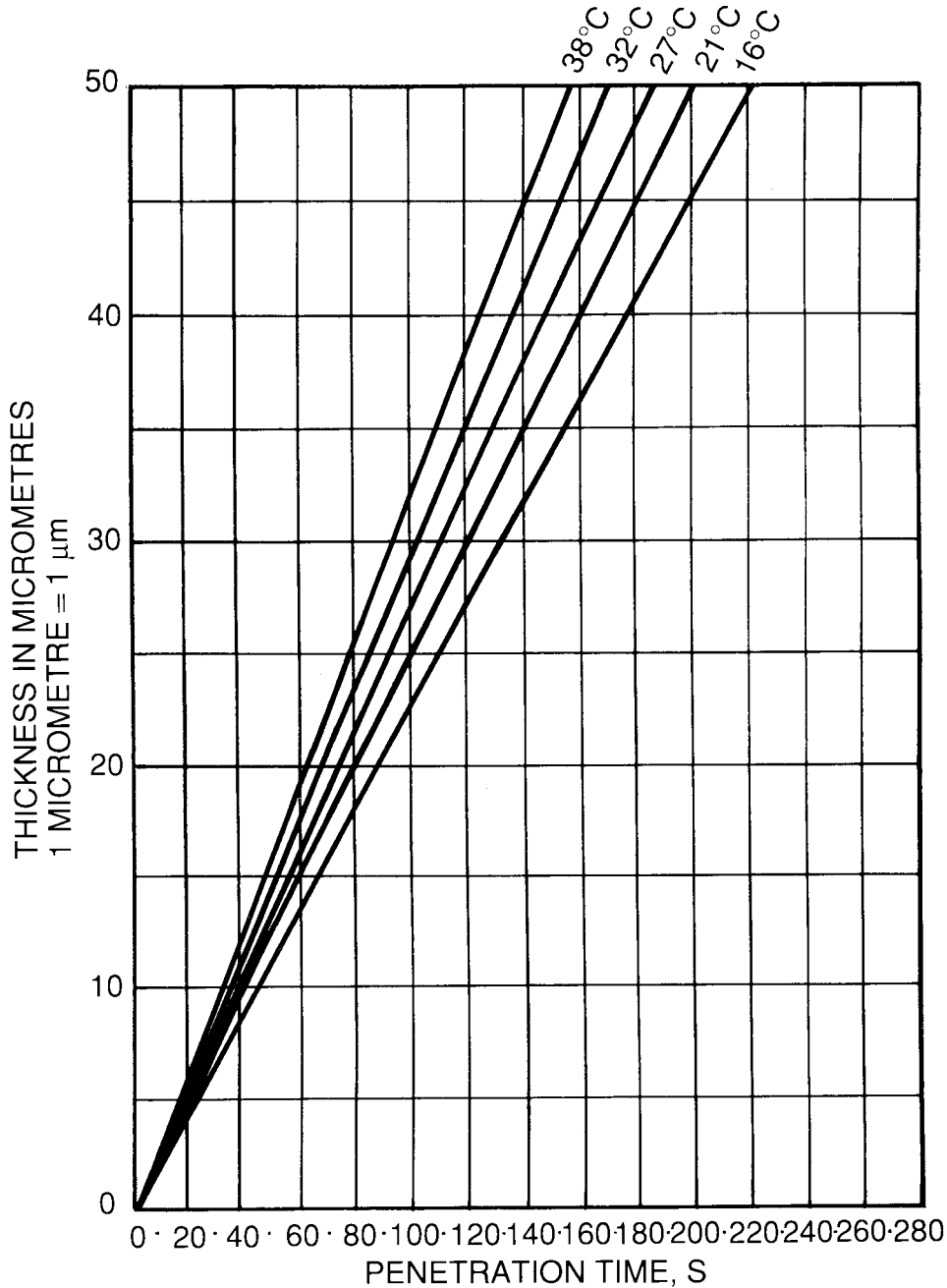
**10. Computation of Thickness**

10.1 Multiply the time required to penetrate the coating by an appropriate thickness factor to obtain the coating thickness.

10.2 Below are the thickness factors, applicable to nominally "pure" coatings (5.1.9), for the solutions given in Section 8. They are valid for a dropping rate of 100 drops/min from a capillary tip, the dimensions of which are given in Fig. 1.

Cadmium (chromic acid solution):	0.35 $\mu\text{m/s}$
Zinc (chromic acid solution):	0.26 $\mu\text{m/s}$
Cadmium (nitrate solution):	0.25 $\mu\text{m/s}$
Zinc (nitrate solution):	0.25 $\mu\text{m/s}$
Tin	0.25 $\mu\text{m/s}$
Copper	0.13 $\mu\text{m/s}$

10.2.1 The first two thickness factors for cadmium and zinc and the chromic acid solution are only valid for a temperature of 24°C; the factors for these metals and this test solution at other temperatures are given in Fig. 2 and Fig. 3.



**FIG. 2 Chart for Determining Thickness of Zinc Coating by the Chromic Acid Dropping Test**

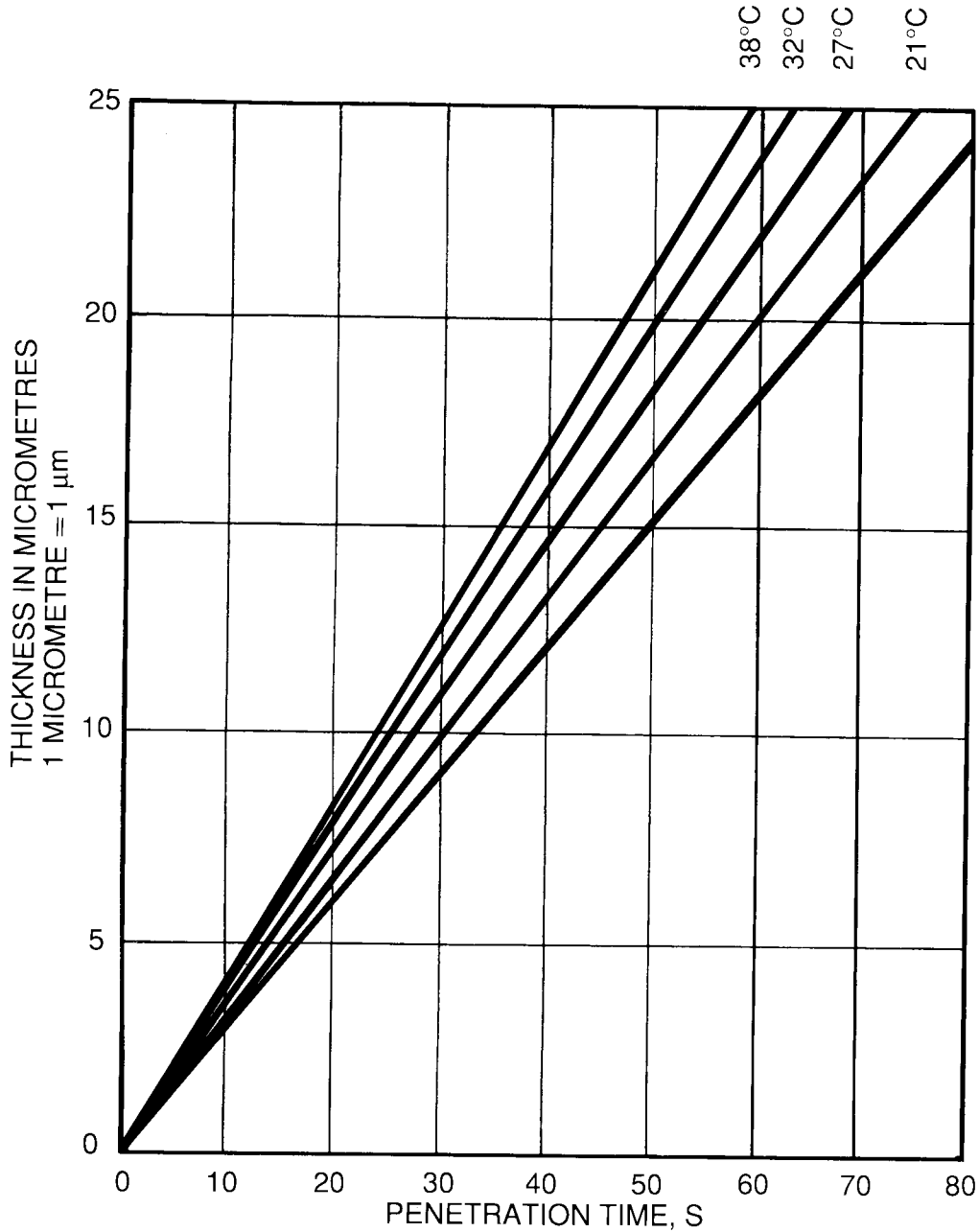


FIG. 3 Chart for Determining Thickness of Cadmium Coating by the Chromic Acid Dropping Test

10.2.2 The last four thickness factors are valid for temperatures in a range from 20 to 30°C.

**11. Reproducibility and Accuracy**

11.1 The reproducibility for a single operator is estimated to be ±2 drops (equivalent to 1.2 s) or ±5 %, whichever is greater.

11.2 The over-all accuracy may be substantially poorer than the reproducibility, because of the uncertainties discussed in

Section 5, and will depend upon the control exercised over the variables. For maximum accuracy, the test method should be standardized with specimens identical to those being tested, having known coating thicknesses determined by other methods. Sometimes a chemical can be added to the dropping solution to aid in determining the end point, for example, potassium ferricyanide ( $K_3Fe(CN)_6$ ) has been added to the test solution for zinc in 8.3.



## **B 555**

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