Designation: B 556 – 90 (Reapproved 2002)

Standard Guide for Measurement of Thin Chromium Coatings by Spot Test[1]

This standard is issued under the fixed designation B 556; 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 (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers the use of the spot test for the measurement of thicknesses of electrodeposited chromium coatings over nickel and stainless steel with an accuracy of about ±20 % (Section 9). It is applicable to thicknesses up to 1.2 µm.[2]

NOTE 1—Although this test can be used for coating thicknesses up to 1.2 µm, there is evidence that the results obtained by this method are high at thicknesses greater than 0.5 µm.[3] In addition, for coating thicknesses above 0.5 µm, it is advisable to use a double drop of acid to prevent depletion of the test solution before completion of the test.

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 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method[4]

B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry[4]

B 588 Test Method for Measurement of Thickness of Transparent or Opaque Coatings by Double-Beam Interference Microscope Technique[4]

3. Summary of Guide

3.1 A drop of hydrochloric acid (test solution) is deposited on the surface of the test specimen, and the time required for the hydrochloric acid to penetrate through the chromium coating (penetration time) is measured. The coating thickness is proportional to this time.

4. Significance and Use

4.1 The thickness of a decorative chromium 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: Methods B 504, B 568, and B 588.

4.3 This test assumes that the rate of dissolution of the chromium by the hydrochloric acid under the specified conditions is always the same.

5. Test Solutions

5.1 For chromium on nickel the test solution is reagent grade hydrochloric acid having a specific gravity at 16°C of 1.180 ± 0.002. (This corresponds to 11.5 *N* ± 0.2 *N*, which may be checked by titration.) For chromium on stainless steel the test solution is 20 g/L of antimony trioxide dissolved in reagent grade hydrochloric acid having a specific gravity at 16°C of 1.160.

NOTE 2—As received, reagent grade hydrochloric acid is normally more concentrated than 11.5 *N*.

6. Preparation of Test Area

6.1 The test area must be free of foreign material. Clean by rubbing the test area with a paste of magnesium oxide, rinse it, and dry it with a clean cloth or filter paper. Draw a ring with a diameter of about 6 mm on the test area with melted paraffin or with a wax pencil.

7. Procedure

7.1 Let the test specimen, the test solution, and the dropper stand long enough to reach room temperature, which should be between 16 and 25°C. Temperatures up to 30°C are permissible, but the measurements become less reliable at the higher temperatures because of increasing sensitivity to temperature. Thin test specimens should be set on a heavy metal plate to avoid a rapid change in temperature which could be produced in such specimens by the heat of reaction.

7.2 To determine the penetration time, *t*, deposit a drop (0.03 to 0.05 mL) of the test solution inside the ring of wax, and measure the time between the beginning of the gas formation and the first appearance of nickel. Make this

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[2] Blum, W., and Olson, W. A., *Proceedings*, Am. Electroplaters Soc., Vol 28, 1940, p. 25.

[3] DuRose, A. H., and Pierce, W. J., *Metal Finishing*, Vol 57, March 1959, p. 54.

[4] *Annual Book of ASTM Standards*, Vol 02.05.

measurement to the nearest ½ s with a stop watch. If the reaction, that is, the formation of gas bubbles, does not start immediately, it can be started by touching the test surface within the ring with a fine nickel wire. The end point of the penetration is characterized by the cessation of the gassing and the appearance of the yellow color of the nickel surface. There may be an uncertainty of 2 or 3 s in determining when the end point has been reached.

7.3 If the basis metal is 18-8 stainless steel, gassing will stop at the end point; if it is 17 % chromium stainless steel, the rate of gas liberation will decrease when the end point is reached.

7.4 When using the antimony trioxide-hydrochloric acid solution, the absence of a black film will indicate that there is no chromium deposit; if a black film forms, but if there is little or no gassing, the chromium is estimated to be less than 0.1 µm thick.

7.5 Measure the room temperature near the test area to an accuracy of 0.2°C.

8. Calculation of Thickness

8.1 Calculate the thickness of the chromium coating by the equation:

$$\text{Thickness} = ut \quad (1)$$

where:

t = penetration time, and

u = speed of dissolution of the chromium coating as a function of the test temperature (see Fig. 1).

NOTE 3—Fig. 1 is applicable when chromium plate is deposited from the conventional sulfate chromic acid solution under conventional operating conditions. If these procedures are varied, it may be necessary to restandardize the test.

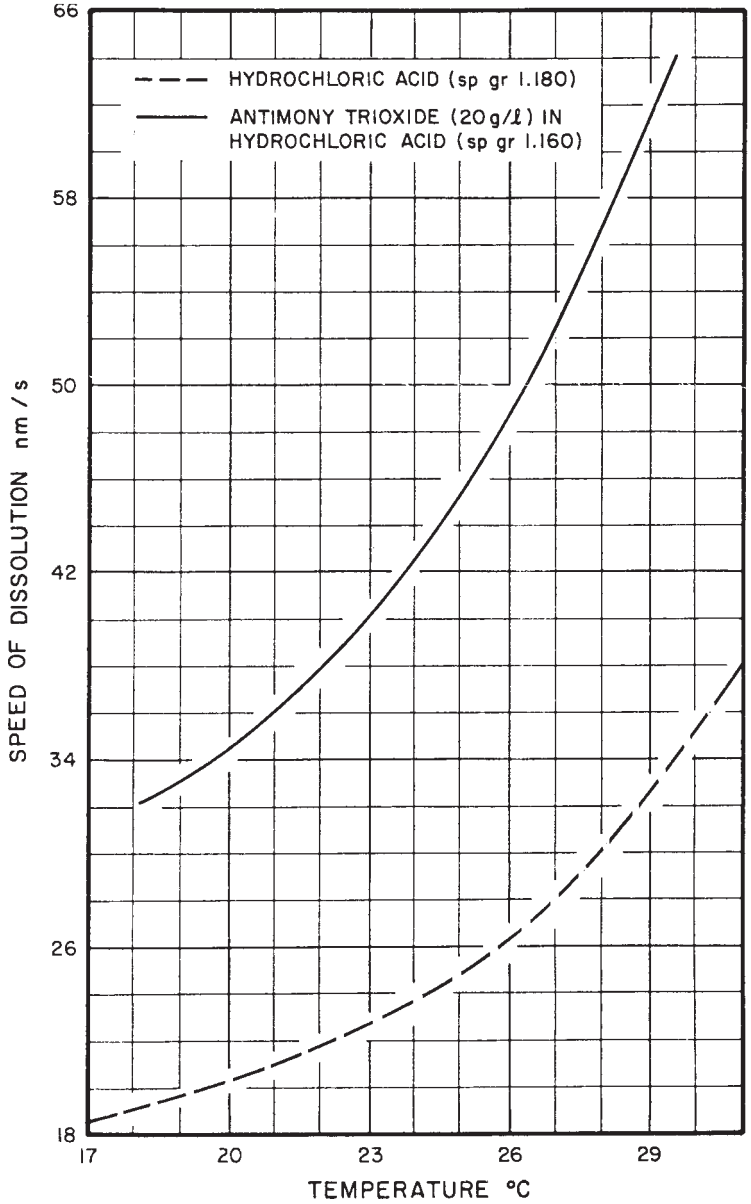

FIG. 1 Temperature Factor for Hydrochloric Acid (sp gr 1.180) and Antimony Trioxide (20 g/L) in Hydrochloric Acid (sp gr 1.160)

8.2 In case of duplex chromium coatings, the thickness will correspond to the combined thickness of the two coatings.

9. Precision and Bias

9.1 This thickness determination has an uncertainty of about ±20 %, and is, therefore, much less accurate than the coulometric method covered by Method B 504.