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Endorsed by American Electroplaters' Society Endorsed by National Association of Metal Finishers

# Standard Test Method for Measurement of Stain Resistance of Anodic Coatings on Aluminum<sup>1</sup>

This standard is issued under the fixed designation B136; 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  $(\varepsilon)$  indicates an editorial change since the last revision or reapproval.

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

#### 1. Scope\*

- 1.1 This test method is intended to determine whether anodic oxide coatings on aluminum and its alloys, that have undergone a sealing treatment and contact with an acid solution, are stainproof or nonadsorptive with respect to dyes.
- 1.2 Coatings that have been properly sealed should be proof against adsorption of coloring materials and, hence, "nonstaining" in many types of service.
- 1.3 This test method is applicable to anodic coatings intended for applications where they are exposed to the weather, or for protective purposes in corrosive media, and where resistance to staining is important.

Note 1—Performance in this test is predictive only of susceptibility to stain by dyes. It is not intended to be predictive of other factors in service performance such as pitting or general corrosion.

Note 2—For Aluminum Association Class I and II architectural anodic coatings that are sealed in solutions containing less than 15 ppm silicates or 3 ppm phosphates, the acid pretreatment may be omitted.

- 1.4 In the case of coatings colored in deep shades, where estimation of the intensity of any residual dye stain is difficult, interpretation of the test is based on whether or not the original color has been affected by the action of the test.
  - 1.5 This test method is not applicable to:
  - 1.5.1 Chromic acid type anodic coatings.
- 1.5.2 Anodic coatings on aluminum alloys containing more than 2 mass % Cu or 4.5 mass % Si.
- 1.5.3 Anodic coatings that have been sealed only in dichromate solutions.
- <sup>1</sup> This test method is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.07 on Conversion Coatings.
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- 1.5.4 Anodic coatings that have undergone a treatment to render them hydrophobic.
- 1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.
- 1.7 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.
- 1.8 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

#### 2. Summary of Test Method

- 2.1 The test method depends upon the observation that a nonsealed or poorly sealed anodic coating is attacked by acid and easily colored by dye, while an adequately sealed coating is not appreciably attacked and does not retain any dye stain.
- 2.2 The test method comprises contacting the test area of the anodized specimen with nitric acid solution and, after rinsing and drying, applying a special dye solution followed by rinsing and rubbing the test area with pumice powder, drying, and visual examination of the test area for retention of dye stain. Coatings that exhibit no dye stain or change in color are considered to have passed the test.

## 3. Significance and Use

3.1 This test method is intended to determine whether anodic oxide coatings on aluminum and its alloys have been properly sealed and as a result are resistant to absorbing dyes.

#### 4. Reagents

- 4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,<sup>2</sup> where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.
- 4.2 Nitric Acid Solution—Prepare a  $40 \pm 5$  mass % solution of nitric acid (HNO<sub>3</sub>) in distilled or deionized water.
- Note 3—A convenient way is to carefully add one volume of  $70\,\%$  HNO $_3$  (sp gr 1.41 at  $20^{\circ}$ C) to an equal volume of water, while stirring, observing the normal precautions for handling strong acids.
- 4.3 Special Dye Solution—Dissolve 1 g of aluminum blue 2LW dye<sup>3</sup> in 50 mL of distilled or deionized water. The pH of the dye test solution shall be adjusted to  $5.0 \pm 0.5$  and maintained at this value with additions of acetic acid or sodium hydroxide, as required.

Note 4—Dye solutions contaminated with soluble phosphates have reduced capability for staining nonsealed or poorly sealed anodic coatings. The dye test solution should have less than 20 ppm of phosphorus as soluble  ${\rm PO_4}^{3-}$ . This corresponds to a phosphorus content of less than 0.05 mass % in the dye powder. Under some conditions of storage and use, dye test solutions may become contaminated or deteriorate with age. To check the reliability of a used or aged dye test solution, it should be applied from time to time to a freshly prepared, nonsealed anodic coating on aluminum to confirm that it will produce a permanent, deeply colored stain in the anodic film.

#### 5. Procedure

- 5.1 Perform the test on an area that has not been contaminated by handling.
- 5.2 Apply a drop of HNO $_3$  (40  $\pm$  5 mass %) on the test area and allow it to remain there for 2 min  $\pm$  5 s. The temperature of the HNO $_3$  and of the test specimens shall be 25 °C  $\pm$  5 °C.
- 5.3 Wash the test area thoroughly with running water and blot dry with a clean cloth. The temperature of the water shall be 25 °C  $\pm$  5 °C.

- 5.4 Apply a drop of the dye solution to the test area and allow it to remain there for 5 min  $\pm$  10 s. The temperature of the dye solution and of the test specimen shall be 25 °C  $\pm$  5 °C.
- 5.5 Wash the test area thoroughly with running water, then rub it with pumice powder (NF or USP Fine Grade)<sup>4</sup> and a clean cloth wet with water, and finally rinse with water and blot dry with a clean cloth. The temperature of the water shall be  $25 \,^{\circ}\text{C} \pm 5 \,^{\circ}\text{C}$ .

Note 5—The pumice scrub removes sealing smut that may absorb the dye. Smut is more difficult to remove after aging than it is from freshly sealed work. Dye that has penetrated the pores of the oxide will not be removed except by removing the oxide. On sound oxides, a pumice scrub will do little more than burnish the surface, even with heavy hand pressure.

#### 6. Interpretation

- 6.1 The test specimen shall be considered to have passed the test if no color from the dye is visible in the test area. Any visible retention of dye color in the test area is considered a failure.
- 6.2 In the case of anodic coatings colored in deep shades, where visual detection of any residual dye stain is difficult or impossible, the criterion for passing the test shall be no visible change in the color of the coating from the action of the test.

Note 6—For special applications it may be found desirable to make the staining test using a solution with which the article may come in contact in service. For example, it is sometimes convenient to test anodically coated aluminum cafeteria trays with coffee. Composition of test solution and test conditions should be standardized for each application.

Note 7—Sometimes the time interval between sealing and application of the stain test influences the result. In such cases, the purchaser and seller should agree upon a time limit for application of this test after the sealing operation.

#### 7. Precision and Bias

7.1 No information is presented about either the precision or bias of Test Method B136 on anodic oxide coatings on aluminum and its alloys since the test result is nonquantitative.

### 8. Keywords

8.1 aluminum; sealing; stain

<sup>&</sup>lt;sup>2</sup> ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analar Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

<sup>&</sup>lt;sup>3</sup> A suitable dye for this test is Aluminum Blue 2LW, available from Sandoz Colors and Chemicals, East Hanover, NJ, 07936.

<sup>&</sup>lt;sup>4</sup> A suitable material is available as product No. P363 from Fisher Scientific Co., Fairlawn, NJ, 07410.