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Standard Method of Corrosion Testing of Decorative Electrodeposited Coatings by the Corrodkote Procedure¹

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1. Scope

1.1 This standard describes the Corrodkote² method of evaluating the corrosion performance of copper/nickel/ chromium and nickel/chromium coatings electrodeposited on steel, zinc alloys, aluminum alloys, plastics and other substrates.

NOTE 1—The following ASTM standards are not requirements. They are reference for information only: Practice B 537, Specification B 456, Test Method B 602, and Specification B 604.

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 117 Practice for Operating Salt Spray (Fog) Apparatus³
- B 456 Specification for Electrodeposited Coatings of Copper Plus Nickel Plus Chromium and Nickel Plus Chromium⁴
- B 537 Practice for Rating of Electroplated Panels Subjected to Atmospheric Exposure⁴
- B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings⁴
- B 604 Specification for Decorative Electroplated Coatings of Copper/Nickel/Chromium on Plastics⁴
- D 1193 Specification for Reagent Water⁵

3. Summary of Method

3.1 The test is conducted by applying a slurry containing corrosive salts to test specimens, allowing the slurry to dry, and exposing the specimens coated with the slurry to a high relative humidity for a specified period of time.

4. Significance and Use

4.1 Nickel/chromium and copper/nickel/chromium electrodeposited coatings are widely used for decorative and protective applications. The Corrodkote test provides a method of controlling the quality of electroplated articles and is suitable for manufacturing control, as well as research and development.

5. Apparatus

5.1 The apparatus shall consist of a humidity chamber, specimen supports, provision for heating the chamber, and provisions for air circulation in the chamber.

5.2 Drops of moisture that might accumulate on the ceiling or cover of the chamber of specimen supports shall not be permitted to fall on the specimens being tested.

NOTE 2—Suitable apparatus may be constructed from salt-spray equipment by eliminating fog-spray nozzles, substituting water for the salt solution in the reservoir, adding a manifold for bubbling air through the water in the reservoir, and adding a fan for circulating the air in the chamber with the fan discharge directed across the surface of the water in the reservoir.

5.3 Materials of construction shall not affect the corrosiveness of the test.

6. Procedure

6.1 Corrodkote Slurry—Prepare the Corrodkote slurry in a glass beaker by dissolving 0.035 g of reagent grade cupric nitrate (Cu(NO₃)₂·3H₂O), 0.165 g of ACS reagent grade ferric chloride (FeCl₃·6H₂O), and 1.0 g of ACS reagent grade ammonium chloride (NH₄Cl) in 50 mL of water conforming to Specification D 1193, Type IV. Stir 30 g of water-washed, ceramic-grade kaolin into the solution. Mix the slurry thoroughly and allow it to stand for about 2 min so that the kaolin will become saturated. Mix the slurry thoroughly just before using.

6.1.1 An alternative method of preparing the Corrodkote slurry is as follows. Weigh out 2.5 g of cupric nitrate $(Cu(NO_3)_2 \cdot 3H_2O)$, and dissolve and dilute with distilled water in a volumetric flask to exactly 500 mL. Weigh out 2.50 g of ferric chloride (FeCl₃·6H₂O), and dissolve and dilute with distilled water in a second volumetric flask to exactly 500 mL. (The ferric chloride solution shall be kept in a dark place and

¹ This method is under the jurisdiction of ASTM Committee B-8 on Metallic and Inorganic Coatingsand is the direct responsibility of Subcommittee B08.10on General Test Methods.

The original work in developing the Corrodkote procedure was initiated by the American Electroplaters' Society Research Committee, Project No. 15.

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² Bigge, D. M., "Experience with the Use of the Corrodkote Test," *Proceedings*, Am. Electroplaters' Soc., Vol. 46, 1959, p. 149.

³ Annual Book of ASTM Standards, Vol 03.02.

⁴ Annual Book of ASTM Standards, Vol 02.05.

⁵ Annual Book of ASTM Standards, Vol 11.01.

stoppered with a rubber or glass stopper when not in use.) (The ferric chloride solution shall not be more than 2 weeks old, since older solutions become unstable.) Weigh out 50.0 g of ammonium chloride (NH₄Cl) and dissolve and dilute with distilled water in a volumetric flask to exactly 500 mL. Then measure out exactly 7.0 mL of the cupric nitrate solution, 33.0 mL of ferric chloride solution, and 10.0 mL of the ammonium chloride solution; place these in a beaker to which 30.0 g of kaolin is now added. Stir with a glass stirring rod.

6.2 *Test Specimens*—The type and number of test specimens to be used, as well as the criteria for evaluation of the test results, shall be defined in the specifications covering the material or product being tested, or shall be mutually agreed upon by the manufacturer and the purchaser.

6.3 *Preparation of Test Specimens*—Metallic and metalliccoated specimens may be solvent cleaned before testing, using a suitable solvent such as alcohol, ethyl ether, acetone or petroleum ether. Solvents that are corrosive or will deposit protective films shall not be used.

6.4 *Slurry Application*—Apply the Corrodkote slurry to the specimen using a clean paint brush. Dip the brush in the Corrodkote slurry and with a circular motion, completely cover the specimen. Then smooth out the coating by brushing lightly in one direction. Allow the specimens to dry at room temperature and at a relative humidity less than 50 % for 1 h before placing them in the humidity chamber. Should it be desirable to cut test specimens from parts or from preplated, or otherwise coated steel sheet, the cut edges shall be protected by coating them with paint, wax, tape, or other effective media so that the development of a galvanic effect between such edges and the adjacent plated or otherwise coated-metal surfaces, is prevented.

6.5 *Position of Specimens During Test*—The position of the specimens in the humidity chamber during the test is not critical as long as they do not touch each other and the supports do not touch the test areas that have been coated with the Corrodkote slurry.

6.6 Conditions in the Humidity Chamber—The exposure zone of the humidity chamber shall be maintained at $38 \pm 2^{\circ}$ C. The relative humidity of the exposure zone of the chamber shall be maintained between 80 and 90 % shall not produce condensation on any of the parts.

NOTE 3—A fan or blower may be used in the chamber to maintain uniform temperature and humidity. The amount of air circulation in the chamber necessary to maintain these conditions must be determined for each chamber. An Abbeon certified hygrometer Model No. 167, or its equivalent⁶ is a suitable instrument for measuring the relative humidity of the exposure zone of the humidity chamber.

6.7 *Test Cycle*—Exposure in the humidity chamber shall be continuous for periods up to 20 h. A single test period up to 20 h shall be considered as one cycle. Continuous operation implies that the humidity chamber be closed and operated continuously except for the short interruptions necessary to place or remove test specimens. Operations shall be so scheduled that these interruptions are held to a minimum. When

more than one test cycle of 20 h or less is specified, the test specimen shall be treated as outlined in 6.9 after each test cycle. Fresh slurry shall be applied for each succeeding cycle. After the last test cycle, the test specimen shall be treated as outlined in 6.9.

6.8 *Period of Test*—The period of each test cycle as well as the number of test cycles required, shall be as specified in the specifications for the material or product being tested, or as mutually agreed upon between the manufacturer and the purchaser.

6.9 *Cleaning of Testing Specimens*—Treat the specimen after each test cycle as outlined in 6.9.1. After the last test cycle and if the specimen is to be inspected between test cycles, treat the specimens as outlined in 6.9.1 and 6.9.2.

6.9.1 Remove the specimen from the humidity chamber. Clean the specimen using fresh running water and a piece of clean cheesecloth or suitable synthetic sponge to remove all of the slurry. A mild abrasive such as a water paste of tripoli may be used to remove any adhering materials.

6.9.2 Snce the cleaning operation described in 6.9.1 will remove any corrosion products, it will be necessary to redevelop the points of failure. This may be done in several ways, such as exposure for 4 h in the salt spray cabinet described in Practice B 117, exposure for 24 h in a humidity cabinet operated at 38° C and 100 % relative humidity with condensation, or some other method that will cause corrosion of the basis metal without any significant damage to the coating.

NOTE 4—Corrosion products visible on the test specimen with the slurry in place are not necessarily caused by points of failure in the coating. Particles of metal accidentally deposited on the test specimen before humidity exposure will also cause visible corrosion products.

7. Evaluation of Results

7.1 After redevelopment of points of failure, as outlined in 6.9.2, make a careful and immediate examination for the extent of corrosion of the test specimen or for other failures as required in the specifications covering the material or product being tested or by agreement between the manufacturer and the purchaser.

8. Report

8.1 The report shall contain the following information, unless otherwise prescribed in the specifications covering the material or product being tested:

8.1.1 The title, designation, and year of issue of this method,

8.1.2 Readings of temperature within the exposure zone of the humidity chamber,

8.1.3 Readings of relative humidity within the exposure zone of the chamber,

8.1.4 Type of specimen and its dimensions or number, or description of part,

8.1.5 Exposure period for each cycle and number of cycles,

8.1.6 Results of all inspections, and

8.1.7 Method of redeveloping corrosion spots.

9. Precision and Bias

9.1 The Corrodkote test provides reliable results if the composition of the slurry, the humidity in the chamber, the temperature, and other factors discussed in this standard are

⁶ Model No. 167 hygrometer may be purchased from Sepor Laboratory Supply Co., P.O. Box 1252, 718 N. Fries, Wilmington, CA 90748.

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carefully controlled. The precision and bias are being determined in interlaboratory test programs.

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