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Standard Guide for Determining Mass Per Unit Area of Electrodeposited and Related Coatings by Gravimetric and Other Chemical Analysis Procedures¹

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1. Scope

1.1 This guide outlines a general method for determining the mass per unit area of electrodeposited, electroless, mechanically-deposited, vacuum-deposited, anodic oxide, and chemical conversion coatings by gravimetric and other chemical analysis procedures.

1.2 This guide determines the average mass per unit area over a measured area.

1.3 The stripping methods cited are described in specifications or in the open literature or have been used routinely by at least one laboratory.

1.4 The procedures outlined can be used for many coating-substrate combinations. They cannot be used where the coating cannot be separated from the substrate by chemical or physical means as would be the case if white brass were plated with yellow brass.

1.5 In principle, these procedures can be used to measure very thin coatings or to measure coatings over small areas, but not thin coatings over large areas. The limits depend on the required accuracy. For example, 2.5 mg/cm² of coating might require 2.5 mg of coating covering 1 cm², but 0.1 mg/cm² of coating would require 25 cm² to obtain 2.5 mg of coating.

1.6 *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:

- A 90/A 90 M Test Method for Weight (Mass) of Coating on Iron and Steel Articles with Zinc or Zinc-Alloy Coatings²
- A 309 Test Method for Weight and Composition of Coating on Terne Sheet by the Triple-Spot Test²
- A 428 Test Method for Weight (Mass) of Coating on

- Aluminum-Coated Iron or Steel Articles²
- B 137 Test Method for Measurement of Coating Mass Per Unit Area on Anodically Coated Aluminum³
- B 449 Specification for Chromates on Aluminum³
- 2.2 *British Standards Institution Documents:*
 - BS 729 Hot Dip Galvanized Coatings on Iron and Steel Articles, Specification for:⁴
 - BS 1706 Electroplated Coatings of Cadmium and Zinc on Iron and Steel, Specification for:⁴
 - BS 1872 Electroplated Coatings of Tin, Specification for:⁴
 - BS 3189 Phosphate Treatment of Iron and Steel, Specification for:⁴
 - BS 3382 Electroplated Coatings on Threaded Components, Specification for:⁴
 - BS 3597 Electroplated Coatings of 65/35 Tin-Nickel Alloy, Specification for:⁴
- 2.3 *Government Standards:*
 - 2.3.1 *DOD Standard:*
 - DOD-P-16232F Phosphate Coatings, Heavy, Manganese or Zinc Base (for Ferrous Metals)⁵
 - 2.3.2 *Federal Standards:*
 - FED-STD 151b Metals; Test Methods: Test 513.1 for Weight of Coating on Hot Dip Tin Plate and Electrolytic Tin Plate⁶
 - RR-T-51D Tableware and Flatware—Silverplated⁶
 - 2.3.3 *Military Standard:*
 - MIL-M-45202C Magnesium Alloys, Anodic Treatment of⁵
- 2.4 *ISO Standards:*
 - ISO 2081 Metallic Coatings—Electroplated Coatings of Zinc on Iron or Steel⁷
 - ISO 2082 Metallic Coatings—Electroplated Coatings on Cadmium on Iron or Steel⁷
 - ISO 2093 Metallic Coatings—Electrodeposited Coatings of Tin, Annex B⁷

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² *Annual Book of ASTM Standards*, Vol 01.06.

³ *Annual Book of ASTM Standards*, Vol 02.05.

⁴ Available from British Standards Institution, Linford Wood, Milton Keynes MK14 6L3, England.

⁵ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

⁶ Available from U.S. Government Printing Office, Washington, DC 20402.

⁷ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

ISO 2106 Anodizing of Aluminum and its Alloys—Determination of Mass Per Unit Area (Surface Density) of Anodic Oxide Coatings—Gravimetric Method⁷

ISO 3892 Conversion Coatings on Metallic Materials—Determination of Coating Mass Per Unit Area—Gravimetric Methods⁷

ISO 4522/1 Metallic Coatings—Test Methods for Electrodeposited Silver and Silver Alloy Coatings—Part 1: Determination of Coating Thickness⁷

ISO 4524/1 Metallic Coatings—Test Methods for Electrodeposited Gold and Gold Alloy Coatings—Part 1: Determination of Coating Thickness⁷

3. Summary of Guide

3.1 The mass of a coating over a measured area is determined by the following:

3.1.1 Weighing the test specimen before and after dissolving the coating in a reagent that does not attack the substrate.

3.1.2 Weighing the coating after dissolving the substrate in a reagent that does not attack the coating, or

3.1.3 Dissolving both the coating and the substrate and quantitatively analyzing the resulting solution.

3.2 The mass per unit area is calculated from the mass and area measurements, the thickness from the mass, area, and density of the coating materials.

4. Significance and Use

4.1 The thickness of a coating is critical to its performance and is specified in many specifications calling for coatings.

4.2 These procedures are used for acceptance testing and appear in a few specifications.

4.3 Coating thickness instruments are often calibrated with thickness standards that are based on mass and area measurements.

4.4 The average thickness of a coating on the measured area can be calculated from its mass per unit area only if the density of the coating material is known.

5. Apparatus

5.1 In addition to normal chemical laboratory equipment for handling small amounts of corrosive and toxic chemicals, an accurate ruler or vernier caliper and a good balance are required. See sections 7 and 8.

6. Specimen Preparation

6.1 *Size*—The specimen must be large enough to permit area and mass measurement of adequate accuracy. (Section 7 and 8.2.)

6.2 *Shape*—The shape of the test specimen must be such that the surface area can be easily measured. A rectangular or circular test specimen is usually suitable.

6.3 *Edge Condition*—If the area to be measured is small and needs to be known accurately, the edges must be dressed to remove smeared coating, to remove loose burrs, and to provide well-defined and (for rectangles) straight edges. This should be considered for areas less than 100 mm². One method of dressing the edges of a rectangular specimen is to clamp the specimen between two plastic or metal blocks with the edge of

the specimen flush with the edges of the blocks and then to grind and polish the edges metallographically.

6.4 *Heat Treatment*—If the substrate is to be dissolved leaving the coating intact, it is desirable to first heat-treat the test specimen so that the coating will not curl up tightly or fall apart. Some gold deposits of 1.5 mg/cm² will fall apart when their substrates are dissolved, but after heat treatment at 120°C for 3 h will support themselves. If the thickness of a coating (instead of its mass per unit area) is being determined, one should not use a heat treatment that might change the density of the coating material.

7. Measurement of Coated Area:

7.1 *Measurement Method*—The accuracy of the area measurement must be better than the desired accuracy of the mass per unit area measurement. Hence the method of measuring the area will depend on the desired accuracy and the specimen size.

7.2 *Equipment*—The area can be measured with a planimeter, but it is usually determined by linear measurements. Often a micrometer or vernier caliper is used. For large areas, however, a ruler may do. For maximum accuracy, a measuring microscope is used.

7.3 *Number of Measurements*—Because circular or rectangular specimens will not be perfectly circular or rectangular, it is desirable to measure each dimension in three places. For a rectangle, one would measure the length of each edge and the length and width through the center and obtain an average for each dimension.

NOTE 1—In the case of a cylinder one would normally measure the diameter and length. In one specification for galvanized wire (fencing), the length of the wire specimen is not measured, but in effect is calculated from the mass (which is measured anyway), the radius, and the density of the steel substrate. ($l = m/\pi r^2 D$)

8. Gravimetric Determination of Mass of Coating:

8.1 *Specimen Size*—The accuracy of the mass measurement must be better than the desired accuracy of the mass per unit area measurement. Hence, the test specimen must be large enough that the coating can be weighed with the desired accuracy.

8.2 *Equipment*—A balance is required, but the required sensitivity of the balance depends on the size of the test specimen, the coating thickness (coating mass), and the required accuracy of the measurement. A balance that weighs to 0.01 g is sometimes satisfactory, though a good analytical balance weighing to 0.0001 g is more versatile. A microbalance is required for small specimens of thin coatings, but it is limited to small samples.

9. Procedure

9.1 The mass of coating may be determined (1) by weighing the test specimen before and after dissolving the coating (see Annex A1) and taking the difference or (2) by dissolving the substrate (see Annex A1) and weighing the coating directly.

9.1.1 *By Difference*—The test specimen is first cleaned of any foreign material and finally rinsed with alcohol, blown dry with clean air, and weighed. The specimen is immersed in the appropriate reagent (see Annex A1) to dissolve the coating,

rinsed with water, rinsed with alcohol, blown dry with clean air, and weighed again. The loss of mass is the mass of the coating. To determine if there was any dissolution of the substrate, repeat the process with the stripped substrate making sure that it is in the reagent just as long as before. Any loss of mass enables one to make a judgment of a possible error due to any dissolution of the substrate with the coating during the stripping process.

9.1.2 *By Direct Weighing*—The substrate is dissolved in the appropriate reagent (see Annex A1. The coating is rinsed with water, rinsed with alcohol, blown dry with clean air, and weighed. To determine if there was any dissolution of the coating, submit the isolated coating to the same stripping process making sure that the coating is in the stripping reagent for the same length of time as it was during the stripping process. Any loss of mass enables one to make a judgment of a possible error due to any dissolution of the coating with the substrate during the stripping process.

NOTE 2—The test procedure given at the end of 9.1.1 and 9.1.2 should be conducted to evaluate a gravimetric method the first time it is used.

9.2 *Determination of Mass of Coating by Chemical Analysis*—This method is by nature very general. Both the coating and substrate are dissolved in a suitable reagent and then the resulting solution is analyzed for the coating material. For each coating-substrate-reagent combination, there are sev-

eral analytical methods. For possible analytical methods see vols 03.05 and 03.06 of the *Annual Book of ASTM Standards*.

10. Calculation

10.1 Calculate the mass per unit area as follows:

$$\text{Mass per unit area} = m/A \text{ (mg/cm}^2\text{)} \quad (1)$$

where:

m = mass of coating (mg), and

A = area covered by coating (cm²)

10.2 Calculate the thickness as follows:

$$\text{Thickness} = 10 \times M/D \text{ (}\mu\text{m)} \quad (2)$$

where:

M = mass per unit area (mg/cm²), and

D = density (g/cm³).

NOTE 3—The density of a coating metal is usually not the same as the handbook value or the theoretical value. For example, the density of electrodeposited gold is generally less than 19.3 g/cm³ and sometimes as low or lower than 17 g/cm³. The densities of some electrodeposited metals are given by W. H. Safranek.⁸

⁸ Printed in *The Properties of Electrodeposited Metals and Alloys*, Second Edition, American Electroplaters' and Surface Finishers Society, 1986.

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ANNEX
Document Preview
(Mandatory Information)

A1. REAGENTS FOR SELECTIVE DISSOLUTION OF METAL LAYERS

NOTE A1.1—The specific issues of standards are cited in this table and included in the literature as references because they contain the information from which this table is based.

A1.1 With many of the reagents given in Table A1.1, there may be some dissolution of the layer other than the one being stripped. Often the dissolution is not significant, but the

possibility should be tested for as suggested in 9.1.1, and 9.1.2.

A1.2 Dissolution is carried out at room temperature unless otherwise indicated. All test pieces are rinsed and dried (see 9.1.1 and 9.1.2) before weighing.

TABLE A1.1 Reagents

Coating	Substrate	Reagents	Remarks—Sources
aluminum	steel	(1) 20 parts by mass NaOH, 80 parts water (2) concentrated HCl (sp gr 1.19)	Immerse a few min (avoid longer time) at about 90°C. While rinsing, scrub with a sponge to remove loose material. Drain off water, immerse 3 s in concentrated HCl at room temperature, scrub again in running water, and repeat entire process until there is no visible reaction in the HCl. Two or three cycles are required normally. A more detailed description is given in the 1981 issue of Test Method A 428.
aluminum	steel	(1) 200 g SbCl ₃ in 1L concentrated HCl (2) 100 g SnCl ₂ ·2H ₂ O in 1L concentrated HCl plus a few granules of tin	Mix equal volume of (1) and (2), immerse until evolution of hydrogen stops, about 1–4 min. Keep below 38°C, rinse and scrub with soft cloth. This test procedure appears in Ref (1) and in the 1981 issue of Test Method A 428. ⁴
anodized aluminum	aluminum	35 mL 85 % phosphoric acid plus 20 g/L CrO ₃	Immerse 5 min at 100°C, rinse, dry, weigh. Repeat cycle until weight is constant. This procedure appears in the 1945 issue of Method B 137 and the 1982 issue of ISO Standard 2106.
anodized magnesium (HAE)	magnesium	300 g/L CrO ₃	Immerse at room temperature, rinse, dry, weigh, and repeat until weight loss is less than 3.9 mg/dm ² . Keep piece of commercially pure aluminum in solution but not in contact with magnesium.