Designation: A309 – 01 (Reapproved 2012)

Standard Test Method for Weight and Composition of Coating on Terne Sheet by the Triple-Spot Test[1]

This standard is issued under the fixed designation A309; 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.

This standard has been approved for use by agencies of the Department of Defense.
This test method replaces Method 511.1 of Federal Test Method Standard No. 151b.

1. Scope

1.1 This test method covers the determination of the weight and composition of coating on terne sheet by the triple-spot method. The following three procedures are described:

1.1.1 *Procedure A*—Stripping with sulfuric acid.

1.1.2 *Procedure D*—Stripping with hydrochloric acid and antimony trichloride.

1.1.3 *Procedure E*—Stripping with hydrobromic acid-bromine solution.

NOTE 1—Procedure B (Electrolytic Stripping) and Procedure C (Stripping with Silver Nitrate Solution), formerly in this test method, were discontinued because lack of usage. The designation for Procedure D and Procedure E are retained to avoid future confusion when reference is made only to the procedure designation.

1.2 If the percent of tin in the coating is required, stripping with hydrobromic acid-bromine is the preferred procedure. Steel with a predeposited electrolytic nickel coating requires a two-stage stripping method to determine total tin content. If both the tin and lead percentage are required, stripping with sulfuric acid is recommended, but caution is advised since the sulfuric acid procedure has been found to produce high tin results (see Section 11).

1.3 *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.* For specific hazards statements, see Section 5, Note 2, *and Section 17.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

A308/A308M Specification for Steel Sheet, Terne (Lead-Tin Alloy) Coated by the Hot-Dip Process

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E57 Methods for Chemical Analysis of White Metal Bearing Alloys (Withdrawn 1986)[3]

E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1998)[3]

3. Significance and Use

3.1 A coating of terne metal on iron or steel articles is intended to provide drawability, solderability, or corrosion resistance, or combination thereof, which can require different amounts of coating. Specifications for terne-coated sheets frequently provide for these different classes (weights) of coating so that purchasers can select that most suitable for their needs. This test method provides a means of determining the weight of coating for comparison with the material specification requirements.

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,

[1] This is under the jurisdiction of ASTM Committee A05 on Metallic-Coated Iron and Steel Products and is the direct responsibility of Subcommittee A05.07 on Methods of Testing.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

where such specifications are available.[4] 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.

5. Hazards

5.1 For precautions to be observed in the use of certain reagents in this test method, reference shall be made to Practices E50. Particular precaution should be observed when using the hydrobromic acid-bromine stripping solution (Procedure E) as described in Section 17.

6. Test Specimens

6.1 Test specimens for weight of coating shall be obtained as prescribed in Specification A308/A308M.

6.2 Specimens shall be 2.25 ± 0.01 in. (57.15 ± 0.25 mm) square or 2.54 ± 0.01 in. (64.52 ± 0.25 mm) in diameter, except that for material narrower than 2.25 in. in width, test specimens shall be of such a length that the area of the specimen is equal to 5.08 in.^{2} (3277 mm^2). The weight of coating in grams on a specimen 5.08 in.^{2} in area is numerically equal to the weight of coating in ounces per square foot of sheet. When it is not possible to secure a specimen of 5.08 in.^{2} in area, a smaller size may be used, but it is recommended that a specimen of not less than 3 in.^{2} (2000 mm^2) be used.

NOTE 2—The area of 5.08 in.^{2} is achieved by a specimen 2.25 in. square or having a diameter of 2.54 in. within the tolerances of the procedure.

6.3 The specimens shall be clean; if necessary, they shall be washed with solvent naphtha or other suitable solvent, then with alcohol, and dried thoroughly.

PROCEDURE A—STRIPPING WITH SULFURIC ACID

7. Reagents and Materials

7.1 *Hydrochloric Acid* (sp gr 1.12)—Mix 500 mL of HCl (sp gr 1.19) with 400 mL of distilled water.

7.2 *Mercuric Chloride Solution*—Prepare a saturated solution of mercuric chloride ($HgCl_2$) in water.

7.3 *Potassium Dichromate, Standard Solution* (0.1 *N*, 1 mL = 0.00559 g Fe)—Prepare and standardize as prescribed for Reagent 10 in Practices E50.

7.4 *Potassium Iodate, Standard Solution* (0.05 *N*, 1 mL = 0.003 g Sn)—Prepare and standardize as prescribed for Reagent 12 in Practices E50, making suitable adjustments in the quantities of reagents and water used so that the solution will be 0.05 *N*.

7.5 *Sodium Bicarbonate Solution (saturated)*—Saturate freshly boiled distilled water with $NaHCO_3$.

7.6 *Sodium Bicarbonate Solution (dilute)*—Dissolve about 10 g of $NaHCO_3$ in 1 L of freshly boiled distilled water.

7.7 *Sodium Diphenylamine Sulfonate Indicator Solution*—Dissolve 0.20 g of sodium diphenylamine sulfonate in 100 mL of water. Store in a dark-colored bottle.

7.8 *Stannous Chloride, Reducing Solution*—Dissolve 100 g of $SnCl_2 \cdot 2H_2O$ in 500 mL of HCl (sp gr 1.19), dilute to 1 L, and mix. Preserve in a dark-colored bottle containing a small amount of granular or mossy tin metal.

7.9 *Starch Solution*—Prepare solution as prescribed for Reagent 110 in Practices E50.

7.10 *Sulfuric Acid* (sp gr 1.84).

7.11 *Sulfuric-Phosphoric Acid Mixture*— Pour 150 mL of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) into 300 mL of water while stirring. Cool, add 150 mL of concentrated phosphoric acid (H_3PO_4, sp gr 1.69), dilute to 1 L with water, and mix.

8. Procedures for Stripping

8.1 After cleaning as described in 6.3, weigh each test specimen separately to the nearest 0.001 g. Wrap a stiff platinum or nickel wire about each specimen in such a manner that it may be held firmly in an acid solution in a horizontal position. Using a 600-mL beaker, heat 60 mL of H_2SO_4 (sp gr 1.84) to 250°C (**Warning—**see Note 3.). Immerse each specimen for about 1½ min (see Note 4) in the hot acid; then remove and momentarily immerse in 50 mL of distilled water contained in a 600-mL beaker. Rub the surface of the specimen with a policeman while washing with about 50 mL of distilled water from a wash bottle. Dry the specimen. If the coating has not been completely removed, again immerse in the acid and repeat the procedure. Thoroughly dry and reweigh the specimen. The loss in weight represents the weight of coating together with iron dissolved from the steel sheet.

NOTE 3—A suitable face shield should be worn in order to protect the operator from accidental splashing or popping of hot sulfuric acid.

NOTE 4—The stripping must be conducted in a manner to ensure that the temperature of the stripping solution is maintained at a minimum of 250°C for at least 1½ min after immersing the test panel. Successive strippings (at 250°C) may be necessary to remove all of the coating completely.

9. Chemical Analysis

9.1 Cool the H_2SO_4 solution in which the specimen was stripped and combine with the washings obtained in the 600-mL beaker while stripping the specimen. Pour the solution into a 500-mL volumetric flask, and rinse the beaker with HCl (sp gr 1.12). Add the rinsings to the flask and dilute to the 500-mL mark with HCl (sp gr 1.12), again diluting to the mark after cooling, if necessary. (In the case of lead-coated sheets it may be necessary to dilute to 1 L with HCl (sp gr 1.12) to ensure complete solution of lead, in which case appropriate changes shall be made in the aliquot portions taken for the subsequent tests described in 9.2, 9.3, and 9.4.) The solution must now be analyzed for iron and, if desired, lead and tin.

9.2 *Determination of Iron*—Transfer a 100-mL aliquot of the solution in the 500-mL volumetric flask (see 9.1) to a

[4] *Reagent Chemicals, American Chemical Society Specifications*, 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.