Designation: A 309 – 01 (Reapproved 2007)

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 A 309; 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]

A 308/A 308M Specification for Steel Sheet, Terne (Lead-Tin Alloy) Coated by the Hot-Dip Process

E 50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E 57 Method for Chemical Analysis of White Metal Bearing Alloys[3]

E 173 Practices for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals[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, 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 E 50. Particular precaution should be observed when

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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] Withdrawn.

[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.

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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 A 308.

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 E 50.

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 E 50, 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 E 50.

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 400-mL beaker. Heat to boiling, rinse the cover and the sides of the beaker with water, and immediately reduce the iron by the dropwise addition of the $SnCl_2$ reducing solution. Add 3 drops in excess after the disappearance of the ferric iron color. Cool and dilute to about 175 mL with water. Add to the solution, all at once, 10 mL of the saturated $HgCl_2$ solution. Mix gently by stirring the solution. Let stand for 3 to 5 min. Add 20 mL of the sulfuric-phosphoric acid mixture and 5 to 6 drops of the sodium diphenylamine sulfonate indicator solution. Titrate with the standard $K_2Cr_2O_7$ solution to a purple end point. Calculate the grams of iron in the stripping solution as follows:

$$\text{Iron, g} = 5\,AB$$

where:

A = millilitres of $K_2Cr_2O_7$ standard solution required to titrate to a purple end point and

B = iron equivalent of the K $_2Cr_2O_7$ solution, kg/L.

9.3 *Determination of Tin*—Transfer a 200-mL aliquot of the solution in the 500-mL volumetric flask (see 9.1) to a 300-mL

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Erlenmeyer flask. Add 3 g of iron in the form of fine wire or thin sheet and 1 g of powdered antimony. Fit the flask with a one-hole rubber stopper containing a glass tube bent twice at right angles, with the end of the short bend projecting through the stopper, the other end being long enough to reach almost to the bottom of a beaker placed on a level with the flask. Pour about 300 mL of a dilute solution of $NaHCO_3$ into this beaker. Place the flask on a hot plate, with the glass tube extending into the beaker containing the dilute solution of $NaHCO_3$. After boiling the solution in the flask for about 5 min, remove the beaker containing the dilute $NaHCO_3$ solution and substitute another containing about 50 mL of a saturated solution of $NaHCO_3$. Move both the beaker and the flask to a cool place. This will cause a small amount of the saturated solution of $NaHCO_3$ to enter the flask and exclude the air. Finally cool the solution to about 10°C. Add 5 mL of starch solution and titrate with KIO_3 solution. It is desirable to run a duplicate analysis for tin, adding the KIO_3 solution quickly to a point slightly less than the end point found in the previous determination, then finishing the titration more slowly. This duplicate analysis may be run using one of the other triple-spot test specimens. Calculate the grams of tin stripped from the test specimen from the number of millilitres of KIO_3 solution used for the titration, as follows:

$$\text{Tin, g} = 5AB/2$$

where:

A = KIO_3 solution required to titrate the samples, mL and
B = tin equivalent of the KIO_3 solution, kg/L.

9.4 *Determination of Lead*—After ascertaining that all the $PbCl_2$ is in solution, remove a 100-mL aliquot from the 500-mL volumetric flask (see 9.1) and place in a 400-mL beaker. Add 10 mL of H_2SO_4(sp gr 1.84), cover, and evaporate to fumes of SO_3. Cool and dilute to 200 mL with water. Allow to settle and then filter on a weighed Gooch crucible, washing with H_2SO_4(1 + 19). Dry and ignite at a dull red heat. Cool and reweigh. Calculate the grams of lead stripped from the test specimen, as follows:

$$\text{Lead, g} = 5(A - B) \times 0.6831$$

where:

A = weight of Gooch crucible and ignited precipitate, and
B = weight of Gooch crucible.

10. Calculation

10.1 *Weight of Coating*—Calculate the weight of coating in ounces per square foot as follows:

$$\text{Weight of coating} = A - B$$

where:

A = loss in weight of specimen, g (see Section 8), and
B = iron stripped from the test specimen (see 9.2), when a specimen of 5.08 in.^{2}(3277 mm^2) in area is used, g. For specimens of other sizes, a suitable correction factor must be applied.

10.2 *Percent of Tin in Coating*—Calculate the percent of tin in the coating as follows:

$$\text{Tin, \%} = [T/(T + L)] \times 100$$

where:

T = tin stripped from the test specimen (see 9.3), g and
L = lead stripped from the test specimen (see 9.4), g.

10.3 *Percent of Lead in Coating*—Calculate the percent of lead in the coating by subtracting the percentage of tin from 100 %.

11. Precision and Bias

11.1 Due to the limited number of laboratories participating in the cooperative testing of this procedure, a precision statement conforming to the requirements of Practices E 173 cannot be furnished. Comparison of the percent tin found by the three cooperating laboratories (Table 1) with the known tin value in the samples indicates that the sulfuric acid procedure (Procedure A) yields high tin results. It is therefore advised that caution be exercised when reporting percent tin values obtained using the sulfuric acid method. The weight of coating results (Table 2) generally agreed to within 0.01 oz/ft^2 between replicate samples and between different laboratories.

PROCEDURE D—STRIPPING WITH HYDROCHLORIC ACID AND ANTIMONY TRICHLORIDE

12. Reagent

12.1 *Antimony Trichloride-Hydrochloric Acid Solution*—Dissolve approximately 40 g of $SbCl_3$ in HCl (sp gr 1.19) and dilute to 1 L with the HCl.

13. Procedure for Stripping

13.1 After cleaning as described in 6.3, weigh each test specimen separately to the nearest 0.001 g. Immerse in the cold $SbCl_3$-HCl solution until the coating is removed and all action ceases. The reaction will leave the specimen coated with antimony. Wash thoroughly to remove the loosely adherent antimony, dry, and reweigh. The loss in weight in grams is the weight of coating in ounces per square foot of sheet when a specimen of 5.08 in.^{2}(3277 mm^2) in area is used. For specimens of other sizes a suitable correction factor must be applied.

TABLE 1 Tin Determination

Sample	Assumed Tin Value[A]	Sulfuric Acid Method (Procedure A)[B]			Hydrobromic Acid-Bromine Method (Procedure E)[B]		
		Laboratory 1	Laboratory 2	Laboratory 3	Laboratory 1	Laboratory 2	Laboratory 3
A	8.6	9.9	10.2	9.2[C]	8.3	8.8	8.6
B	12.4	14.5	14.5	13.0[C]	12.2	12.7	12.2

[A] Values obtained using Method E 57.
[B] All values represent an average of three determinations.
[C] Calculated using coating weight instead of method used in 10.2.

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TABLE 2 Weight of Coating, oz/ft² of Sheet

Sample	Hydrochloric Acid-Antimony Trichloride Method (Procedure D)			Sulfuric Acid Method (Procedure A)			Hydrobromic Acid-Bromine Method (Procedure E)		
	Laboratory 1	Laboratory 2	Laboratory 3	Laboratory 1	Laboratory 2	Laboratory 3	Laboratory 1	Laboratory 2	Laboratory 3
1A	0.16	0.16	0.16	0.16	0.15	0.16	0.16	0.16	0.17
2A	0.16	0.16	0.16	0.16	0.15	0.16	0.16	0.16	0.17
1B	0.34	0.32	0.33	0.33	0.28[A]	0.31	0.35	0.33	0.35
2B	0.33	0.33	0.32	0.32	0.29[A]	0.32	0.33	0.34	0.33

[A]Procedural error—results not considered valid.

14. Precision and Bias

14.1 Due to the limited number of laboratories participating in the cooperative testing of this procedure, a precision statement conforming to the requirements of Practices E 173 cannot be furnished. Comparison of the weight of coating results, Table 2, indicates that generally the results agreed to within 0.01 oz/ft² between replicate samples and between different laboratories.

PROCEDURE E—STRIPPING WITH HYDROBROMIC ACID-BROMINE SOLUTION

15. Apparatus and Materials

15.1 *Atomic Absorption Spectrophotometer*—The instrument shall consist of an atomizer and burner, suitable pressure-regulating devices capable of maintaining constant oxidant and fuel pressure for duration of the test, a hollow cathode lamp for each metal to be tested, an optical system capable of isolating the desired line of radiation, an adjustable slit, a photomultiplier tube or other photosensitive device as a light-measuring and amplifying device, and a readout device for indicating the amount of absorbed radiation.

15.1.1 *Hollow Cathode Lamps*—A single element (tin) lamp is available and has been found satisfactory for tin analysis.

15.2 *Oxidant* :

15.2.1 *Air*—Use clean, dry air. A filter capable of removing oil, water, and other foreign substances may be used.

15.2.2 *Nitrous Oxide*—Laboratory and industrial high-purity grade nitrous oxide has been used satisfactorily.

15.3 *Fuel* :

15.3.1 *Acetylene*—Standard, commercially available acetylene is satisfactory (**Warning—**see 17.1). However, since all acetylene cylinders contain acetone, no more than three fourths of the volume of the tank should be used in order to prevent carryover of the acetone into the equipment.

15.3.2 *Pressure-Reducing Valves*—The supplies of fuel and oxidant shall be maintained at pressures somewhat higher than the controlled operating pressure of the instrument by means of suitable pressure-reducing valves.

16. Reagents

16.1 *Hydrochloric Acid (1+1)*—Mix 1 volume of concentrated hydrochloric acid (sp gr 1.19) with 1 volume of water.

16.2 *Hydrochloric Acid (1+20)*—Mix 1 volume of concentrated hydrochloric acid (sp gr 1.19) with 20 volumes of water.

16.3 *Hydrobromic Acid-Bromine Stripping Solution*—Mix 9 volumes of hydrobromic acid (sp gr 1.49) with 1 volume of bromine liquid (**Caution,** see 17.2).

16.4 *Lead Solution Stock*—Prepare stock lead solution by dissolving 1.500 g of pure lead in 50 mL of hydrobromic acid-bromine stripping solution (from 16.3). Dilute to 200 mL in a volumetric flask with the hydrobromic acid-bromine stripping solution and mix (1 mL = 7.5 mg Pb).

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

16.6 *Perchloric Acid* (sp gr 1.67) (**Caution,** see 17.3).

16.7 *Potassium Dichromate, Standard Solution* (0.1 *N*, 1 mL = 0.00559 g iron)—Prepare and standardize as prescribed for Reagent 10 in Practices E 50.

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

16.9 *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.

16.10 *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.

16.11 *Tin Standard, Stock Solution*—Prepare stock standard solution by dissolving 1.000 g of pure tin in 100 mL of HCl (1+1). Dilute to 1000 mL in a volumetric flask using HCl (1+1). Concentration: 1 mL = 1000 µg tin. Commercial tin standards are available.

17. Hazards

17.1 *Acetylene*—**Warning—**"Purified" grade acetylene contains a special proprietary solvent rather than acetone, which can weaken the walls of poly(vinyl chloride) tubing that carries acetylene to the burner, causing a potentially hazardous situation and should not be used.

17.2 *Hydrobromic Acid-Bromine Solution*:

17.2.1 The handling of bromine, either as a liquid or in the vapor form, is a *health hazard*. Therefore, the following minimum precautions should be taken:

17.2.2 Extreme care should be taken to avoid inhaling the corrosive fumes: conduct all mixing, stripping, and evaporating operations in a well-ventilated hood.

17.2.3 Avoid skin contact with the liquid or fumes: rubber gloves may be worn as protection from accidental spillage or splashes.

17.2.4 If liquid should come into contact with bare skin, immediately wash the affected area thoroughly with water. Medical attention may be necessary if burns are severe.

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17.2.5 Before disposal, neutralize all bromine-containing solutions using a 10 % solution of sodium thiosulfate ($Na_2S_2O_3$). Add thiosulfate solution and stir until the bromine color disappears.

17.3 *Perchloric Acid*—The handling and storage of perchloric acid should be conducted in accordance with the precautions given in Practices E 50, Safety Precautions.

18. Procedure for Stripping

18.1 After cleaning in accordance with 6.3, weigh the test specimen to the nearest 0.001 g. Place the specimen in a suitable stripping dish. Add 40 mL of stripping solution (**Warning—**see 17.2), and immediately begin policing both sides of the specimen. After 30 s, remove the specimen using TFE-fluorocarbon forceps and place it in a filter funnel that has been inserted into a 200-mL volumetric flask. Rinse the specimen with HCl (1+1), catching and saving all rinsings in the volumetric flask. Inspect the surface of the specimen for unremoved coating by using a "scratch" test. (Scratching the specimen surface with a fingernail leaves a noticeable metallic scratch if any coating is left on the surface. The base metal will not leave such a mark.) If the scratch test reveals residual coating, repeat the stripping (using original solution) and rinsing steps until the surface is free of coating. Rinse the stripped test specimen with water, dry, and reweigh. The loss in weight represents the weight of coating together with iron dissolved from the steel sheet.

19. Chemical Analysis

19.1 Transfer the sample stripping solution to the 200-mL flask that contains the rinsings and dilute to volume with HCl (1+1).

19.2 *Determination of Iron*—Transfer a 50-mL aliquot into a 250-mL beaker. Add 10 mL of $HClO_4$ and take to fumes on a hot plate (**Warning—**see 17.3). Cool solution, then add 100 mL of water, 5 mL of HCl, and heat to boiling. Immediately reduce the iron with addition of the $SnCl_2$ reducing solution. Add 3 drops in excess after the disappearance of the ferric iron color. Cool and dilute to about 175 mL with water. Add to the solution, all at once, 10 mL of the saturated $HgCl_2$ solution. Mix gently by stirring the solution. Let stand for 3 to 5 min. Add 20 mL of the sulfuric-phosphoric acid mixture and 5 to 6 drops of the sodium diphenylamine sulfonate indicator solution. Titrate with the standard $K_2Cr_2O_7$ solution to a purple end point. Calculate the grams of iron in the stripping solution as follows:

$$\text{Iron, g} = 4AB$$

where:

A = $K_2Cr_2O_7$ standard solution required to titrate to a purple end point, mL and

B = iron equivalent of the $K_2Cr_2O_7$ solution, kg/L.

19.3 *Determination of Tin*:

19.3.1 *Terne-Coated Sheet Without Predeposited Electrolytic Nickel Coating*—Use Procedure E.

19.3.2 *Terne-Coated Sheet With Predeposited Electrolytic Nickel Coating*:

19.3.2.1 Use a combination of Procedure E (to strip coating overlay) and an additional alloy layer stripping procedure to determine total tin content. Strip terne coating with reagent grade hydrobromic acid (48 %) containing 10 mL/L of a 10 % antimony trichloride/hydrochloric acid solution. (Dissolve 10 g antimony trichloride in 100 mL of concentrated hydrochloric acid, sp gr 1.19.) Reduce with aluminum wire and titrate with potassium iodate solution. Strip alloy layer in a 1:3 nitric acid solution, reduce with aluminum wire and titrate in potassium iodate solution. Total tin content is equal to the tin in the coating plus the tin in the alloy layer.

19.3.3 *Calibration and Standardization*:

19.3.3.1 Prepare tin standards ranging from 0 to 200 mg/L by transferring the required amounts of standard, 1000 mg/L tin solution (from 16.11) to 100-mL volumetric flasks (0, 1.0, 2.0, 5.0, 10.0, 15.0, and 20.0 mL). Add 20 mL of the stock lead solution (from 16.4) to each flask, dilute to volume with HCl (1+1), and mix.

19.3.3.2 The exact operation of different instruments varies; hence no attempt is made here to describe in detail the steps for putting the instrument into operation. However, the following parameters have been found suitable for some types of instruments.

Lamp current, mA	10
Main air supply, psi	40
Air flow, standard ft^3/h	10
Main acetylene supply, psi	10
Acetylene flow, standard ft^3/h	approximately 8
Main nitrous oxide (N_2O) supply, psi	40
Nitrous oxide (N_2O) flow, standard ft^3/h	approximately 10
Slit band width, Å	4
Wavelength, Å	2863 (0 to 200 mg/L)

Consult manufacturer's manual for the equivalent operating parameters for the instrument in use.

19.3.3.3 Atomize the standards and record the instrument readings expressed in absorbance units. Aspirate water between each standard.

19.3.3.4 Prepare a calibration curve by plotting on linear graph paper the absorbance versus standard concentration for each standard.

19.3.4 *Procedure*:

19.3.4.1 Atomize the sample stripping solution under the conditions in 19.3.3.

19.3.4.2 Determine concentration of the stripped sample solution using the calibration curve. Determine concentration in milligrams per litre.

20. Calculation

20.1 *Weight of Coating*—Calculate the weight of coating in ounces per square foot of sheet as follows:

$$\text{Weight of coating, oz/ft}^2 = A - B$$

where:

A = loss in weight of specimen, g, and

B = grams of iron stripped from the test specimen (from 19.2), when a specimen of 5.08 $in.^2$ (3277 mm^2) in area is used. For specimens of other sizes, a suitable correction factor must be applied.

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20.2 *Percentage of Tin in Coating*—Calculate the tin content as follows:

$$\text{Tin, \%} = \text{mg/L tin}/50\ [A - B]$$

where:

$A - B$ = weight of coating, oz/ft ^{2}, when a specimen of 5.08 in.^{2} (3277 mm^2) in area is used. For specimens of other sizes, a suitable correction factor must be applied.

21. Precision and Bias [5]

21.1 Due to the limited number of laboratories participating in the cooperative testing of the tin analysis procedure, a precision statement conforming to the requirements of Practices E 173 cannot be furnished. Comparison of the percentage tin found by the three cooperating laboratories (Table 1) with the known tin value in the samples indicates an error of approximately 0.2 to 0.3 % tin. Therefore, this procedure can be expected to be within 3 % of the expected tin value.

22. Keywords

22.1 coating composition; coating weight; metallic coatings; steel sheet—terne coated; terne coating

[5] Supporting data are available from ASTM Headquarters. Request RR: A05-1001.

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