Designation: A 630 – 03

Standard Test Methods for Determination of Tin Coating Weights for Electrolytic Tin Plate[1]

This standard is issued under the fixed designation A 630; 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.

INTRODUCTION

Four test methods for determination of tin coating weights are described. These are typical methods and represent those most commonly used in the tin plate industry. Publication of these test methods is not intended to preclude the use of any other methods such as X-ray fluorescence measurement systems for control purposes by the consumer or supplier. However, in case of dispute, the referee method is to be used to determine conformance to Specification A 624/A 624M and Specification A 626/A 626M.

Sampling procedures for tin coating-weight testing and applicable standards for the specific class designation are outlined in Specification A 624/A 624M and Specification A 626/A 626M.

1. Scope*

1.1 These test methods include four methods for the determination of tin coating weights for electrolytic tin plate as follows:

Test Method	Sections
A—Bendix Test Method	3 to 9
B—Constant-Current, Electrolytic Test Method (Referee Method)	10 to 18
C—Sellar's Test Method	19 to 28
D—Titration Test Method	29 to 37

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

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.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

A 599/A 599M Specification for Tin Mill Products, Electrolytic Tin-Coated, Cold-Rolled Sheet

A 623 Specification for Tin Mill Products, General Requirements

A 623M Specification for Tin Mill Products, General Requirements [Metric]

A 624/A 624M Specification for Tin Mill Products, Electrolytic Tin Plate, Single Reduced

A 626/A 626M Specification for Tin Mill Products, Electrolytic Tin Plate, Double Reduced

D 1125 Test Methods for Electrical Conductivity and Resistivity of Water

METHOD A—DETERMINATION OF THE TIN COATING WEIGHTS BY THE BENDIX TEST METHOD

3. Scope

3.1 This test method covers the determination of tin coating weights on steel plate.

4. Summary of Test Method

4.1 The procedure involves dissolution of tin from a tin plate anode in a dilute hydrochloric acid solution containing a measured excess of standard potassium iodate-potassium iodide solution. Excess iodine from the iodate-iodide solution is back titrated with standard sodium thiosulfate using a starch indicator.

[1] These test methods are under the jurisdiction of ASTM Committee A01 on Steel, Stainless Steel and Related Alloys and are the direct responsibility of Subcommittee A01.20 on Tin Plate.

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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.

***A Summary of Changes section appears at the end of this standard.**

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5. Apparatus [3]

5.1 The detinning cell consists essentially of two cathodes of carbon rod, the sample that serves as an anode, and a beaker of dilute hydrochloric acid electrolyte. The carbon rods (6 by 0.25 in. or 152 by 6.35 mm, encased in porous porcelain thimbles) are suspended from a suitable frame about 1 in. [25 mm] apart. A small glass-enclosed magnet is attached to the frame in such a manner that it will hold the sample suspended midway between the two cathodes. A movable platform permits the beaker of electrolyte to be brought up around the assembly so that the sample will be completely immersed.

5.2 A source of direct current that can be regulated to supply up to 3 A at 3 to 5 V through the deplater is required.

5.3 Although regular laboratory glassware can be used, it is advisable to use automatic dispensing pipets or burets, a motor-driven stirrer for titrations, and a timing switch when large numbers of determinations are to be made.

5.4 *Precautions*—The apparatus must be kept in continuous operation to prevent iron in the solution adhering to the porous cells from oxidizing and subsequently liberating iodine from the potassium iodate-potassium iodide solution. If the instrument has been idle for some time, it is necessary to remove the ferric iron by running a disk of tinplate through the regular procedure before test samples are run.

6. Reagents

6.1 *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.

6.2 *Water*—Deionized or distilled water having a volume resistivity greater than 1 MΩ·cm at 25°C as determined by Nonreferee Method of Test Methods D 1125.

6.3 *Hydrochloric Acid* (1.7 to 2.0 *N*)—Add 1 part of concentrated hydrochloric acid (HCl, sp gr 1.19, 36.5 to 38.0%) to 5 parts of water and mix well.

6.4 *Potassium Iodate-Potassium Iodide, Standard Solution* (0.0975 *N*)—Dissolve 3.48 g of KIO_3, 21.74 g of KI, and 1.21 g of NaOH in 1 L of distilled water. Standardize as follows: Transfer 0.2700 g of National Bureau of Standards tin to a 500-mL Erlenmeyer flask. Add 200 mL of HCl (1+1). Connect the flask to a carbon dioxide (CO_2) system and displace the air in the flask with CO_2. While continuing the flow of CO_2, heat the flask but do not boil violently. After the tin has dissolved add 0.5 g of antimony and 2 g of high-purity aluminum. Heat until the aluminum is completely dissolved and digest for an additional 10 min. Cool the flask to room temperature in running water while maintaining an atmosphere of CO_2. Disconnect from the CO_2 system and titrate with the KIO_3-KI solution using starch as an indicator. Calculate the tin titer, *T*, as follows:

$$T = A/B$$

where:

A = tin used (0.2700 g),
B = KIO_3-KI solution used for titration, mL, and
T = tin titer for above KIO_3-KI solution, (grams of tin/mL),
T = 17.28 = lb/base box (bb)/mL, for a 4-in.^{2}[25.81-cm^2] sample.

6.5 *Sodium Thiosulfate, Standard Solution* (for coatings over 0.50 lb/bb)—Dissolve 15.11 g of $Na_2S_2O_3 \cdot 5\ H_2O$ and 1.11 g of NaOH in 1 litre of distilled water in a light-proof bottle. Allow this solution to age for 72 h, remix, and standardize as follows: Connect the bottle to the Bendix apparatus and titrate 20 mL of the standardized KIO_3-KI solution with the thiosulfate solution using the same procedure as is used for making weight of coating determinations, but ignore the stripping unit. The tin equivalent of the $Na_2S_2O_3$ solution in pounds per base box is equal to: $20/A \times T \times 17.28$, where *A* = millilitres of sodium thiosulfate solution used in titrating 20 mL of standard KIO_3-KI solution. A chart can be prepared showing lb/bb/mL of thiosulfate.

6.6 *Sodium Thiosulfate, Standard Solution* (for coatings 0.50 lb/bb and under)—Dissolve 6.57 g of $Na_2S_2O_3 \cdot 5\ H_2O$ and 2.78 g of NaOH in 1 litre of distilled water in a light-proof bottle. Larger quantities in the same proportions may be prepared if desired. Allow that solution to age for 72 h, remix, and standardize by the same procedure used for the stronger $Na_2S_2O_3$ solution but use only 10 mL of potassium iodate-potassium iodide solution.

6.7 *Starch Solution*—Heat 200 mL of distilled water to boiling in a Florence flask and slowly add 2.5 g of soluble starch paste while the solution is agitated. Add the hot starch solution to 500 mL of distilled water containing 2.5 g of NaOH. Dilute to 1 L and thoroughly mix.

7. Test Sample

7.1 Tin plate samples for coating weight determinations are obtained by stamping disks 2.257 ± 0.001 in. [57.33 ± 0.02 mm] in diameter which is equivalent to 4 in.^{2}[25.81 cm^2] of area (8 in.^{2}[52 cm^2] of surface area). Recommended methods of obtaining representative samples are described in the Tin Mill Products sections of the American Iron and Steel Institute's Steel Products Manual.[5]

8. Procedure

8.1 Make required electrical connections.

8.2 Add HCl (1+1) to the porous tubes containing the carbon cathodes.

[3] Complete details and drawings of the apparatus are contained in U. S. Patent No. 2,455,726 entitled "Method for Electrolytic Stripping and Determination of Plating Metals." A suitable commercial supplier of the apparatus has been found to be the Wilkens-Anderson Company of Chicago.

[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.

[5] Available from American Iron and Steel Institute (AISI), 1101 17th St., NW, Suite 1300, Washington, DC 20036.

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8.3 Suspend the sample disk of tin plate from the magnetic holder.

NOTE 1—If it is desired to strip on one side only, mask the opposite side and reduce the current to half its normal value. If a heavy oxide film has developed on the tin plate during storage, the plate must be cathodically cleaned prior to testing.

8.4 Place a measured quantity of standardized KIO_3-KI solution into a 400-mL beaker (see 8.10). Simultaneously add 250 mL of dilute HCl and mix thoroughly.

8.5 Raise the beaker so that the sample and porous cells are immersed.

8.6 Turn on d-c current and adjust to give 0.5 A/in.^{2} of sample.

8.7 Time for complete removal of tin (see 8.10).

8.8 Remove the beaker and add approximately 5 mL of starch indicator solution.

8.9 Titrate with standardized $Na_2S_2O_3$ solution to the disappearance of the blue color.

8.10 The stripping times and amounts of KIO_3-KI solution to use are as follows:

Product	Stripping Time, s	Amount of KIO_3-KI Solution, mL
Electrolytic 100	90	20
Electrolytic 75	75	20
Electrolytic 50	60	10
Electrolytic 25	60	10
Electrolytic 10	45	10

Stripping time should not be longer than is required to remove all of the tin. Results will be high by approximately 0.01 lb/bb for each minute of over-stripping.

9. Reproducibility of Results

9.1 Arbitrary maximum spreads in intermill check tests show the reproducibility of test results by the Bendix Method to be as follows: ±0.02 lb/bb for 0.25–lb coatings, ±0.03 lb/bb for 0.75-lb coatings, and ±0.04 lb/bb for 1.25-lb coatings. Data have not been developed for 0.10-lb coatings.

METHOD B—DETERMINATION OF THE TIN COATING WEIGHTS BY THE CONSTANT CURRENT, ELECTROLYTIC METHOD (REFEREE METHOD)

10. Scope

10.1 This test method[6] may be used to determine not only the total tin coating weight but also to determine that part of the tin coating which is present as free tin and that part which is present in the alloyed form.

11. Summary of Test Method

11.1 In this test method, the tin is stripped from a sample of tin-plate anodically at constant current in an electrolyte of the hydrochloric acid. The potential difference developed between the sample and a reference electrode is plotted against time on a strip chart recorder or an electric digital readout. The time required for stripping the free tin and alloyed tin, respectively, are read from the resulting chart (see Fig. 1) or a digital readout. Since the stripping current has been preset, the free-tin and alloy-tin coating weights are calculated by employing Faraday's law of electrolysis.

12. Calibration and Standardization

12.1 Determine the weight loss of pure tin specimens electrolyzed for a given time interval, expressing the results as milligrams or pounds of tin per base box per second.

12.2 The test specimen should be a 4-in.^{2} disk of pure tin approximately 0.20 in. [5.1 mm] thick.

12.3 The milliammeter should be frequently checked using a precision milliammeter.

13. Available Constant Current Procedures

13.1 There are two commercially available constant current, electrolytic units that are in common use. Either of the following, or equivalent equipment, can be used as an acceptable referee method:

13.1.1 *Willey & Kunze Coulometric Test Method*—Supplier will provide set up, start up and running procedures.[7]

13.1.2 *The Stannomatic Test Method*—Supplier will provide set up, start up and running procedures.[8]

14. Willey & Kunze Coulometric Test Method

14.1 In this test method, the tin is electrolytically removed from a 4 in.^{2}[25.807 cm^2] circular sample of tinplate anodically at constant current in an electrolyte of 1.0 N hydrochloric acid. The potential difference developed between the sample and a glass calomel reference electrode is plotted against time on a strip chart recorder. The time required for stripping the free tin and alloy tin, respectively are read from the resulting chart (see Fig. 1) or on an electronic digital readout. Since the stripping current is constant and preset, the free tin and alloy tin coating weights are calculated by employing Faraday's law of electrolysis.

14.2 *Significance and Use*:

14.2.1 The amount of tin coating is directly associated with the economics of producing tinplate and the performance of the container or part for which such plate is used. Therefore, the test method was developed for accuracy and as a control. The amount of tin combined in the alloy layer is also essential as the continuity and amount plays an important part in its functional properties. This test method is so designed that the tin coating is determined on only one side of the specimen at a time. This is useful when determining tin coatings on differential tinplate.

14.3 *Apparatus*:

14.3.1 This instrument (Model 8014) consists of an ammeter, volt meter, constant current source and strip recorder. A cell and sample holder are provided.

[6] See Kunze, C. T., and Willey, A. R., "Electrolytic Determination of Tin and Tin-Iron Alloy Coating Weights on Tin Plate," *Journal*, Electrochemical Society, Vol 99, No. 9, September 1952, pp. 354–359; and "Methods for Determination of Coating Weights of Tin Plate," American Iron and Steel Institute, December 1959, pp. 19–26.

[7] Available from Coulometric Systems, Bergholtz, OH.

[8] Available from Products Distribution Service Division, Wilkens Anderson Co., 4525 West Division St., Chicago, IL 60651.

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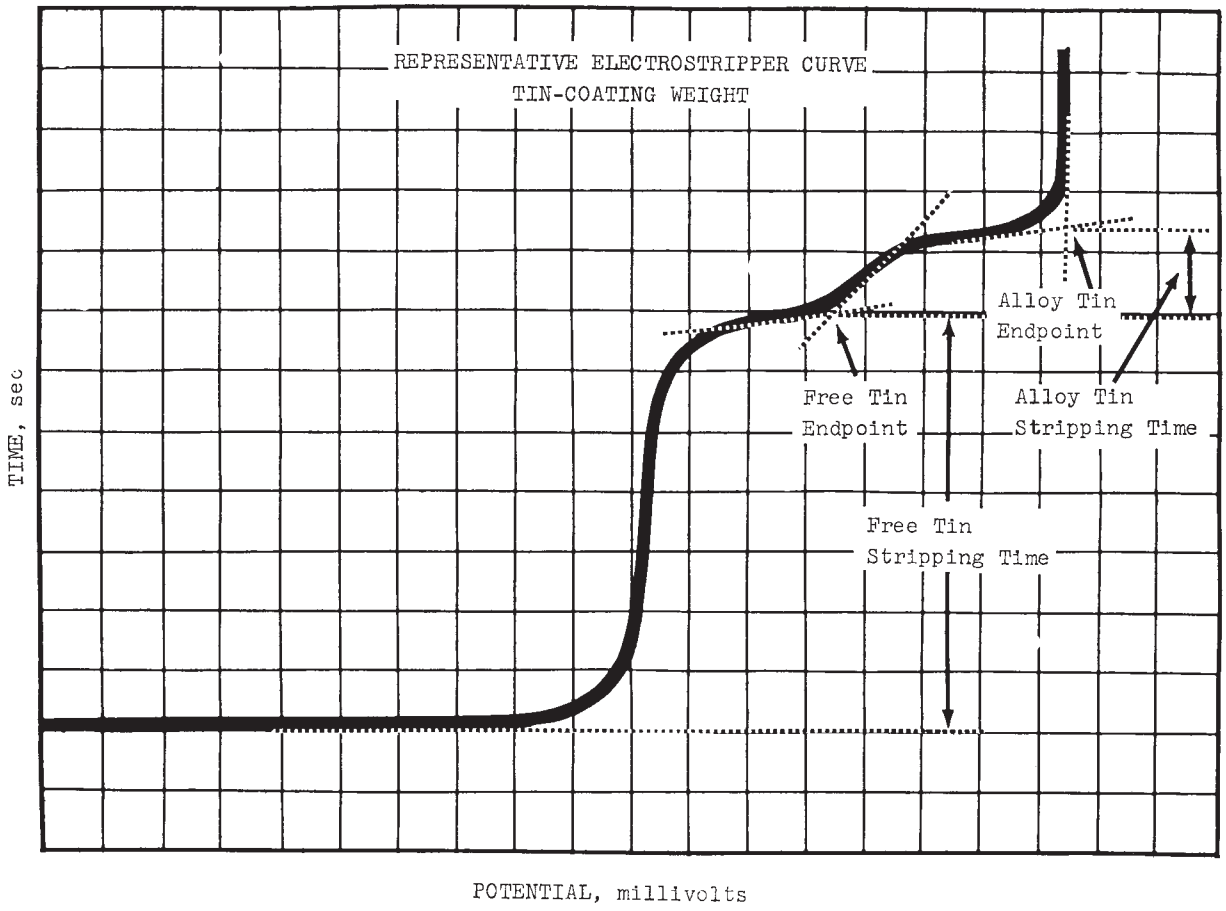


FIG. 1 Representative Electrostripper Curve

14.4 *Reagents*:

14.4.1 *Electrolyte*—1.0 N hydrochloric acid (HCl).

14.5 *Sample Size*:

14.5.1 Tinplate samples for coating weight determinations are obtained by stamping discs 2.257 ± 0.001 in. [57.33 ± 0.02 mm] in diameter. This is equivalent to 4 in.^{2}[25.81 cm^2] of area of one side. Sampling procedures for tin coating weight testing and applicable standards for the specific class designation are outlined in Specifications, A 624, A 624M, A 626, and A 626M.

14.6 *Interferences*:

14.6.1 The distance of the cathode from the sample is important and should be 0.75 ± 0.25 in. [1.90 ± 0.64 cm]. Also, it is important that the cathode be approximately the same shape as the sample so that all parts of the anode are equidistant from the cathode.

15. The Stannomatic Test Method

15.1 *Principle of Equipment Operation*:

15.1.1 The free tin and the alloy tin covering the surface of the tin plate to be examined are dissolved electrolytically. The test size is exactly defined on both sides of the sample by two precision gaskets. The free tin and iron-tin alloy results are a function of the amount of current (Coulombs) used for stripping and are displayed on a numeric readout.

15.2 *Apparatus*:

15.2.1 This instrument consists of a stripping cell with a standardized sample size control. The stripping controller has a numeric readout which displays separately free tin and alloy tin on both sides of the test sample.

15.3 *Reagent*:

15.3.1 *Electrolyte*—As specified by equipment manufacturer.

15.4 *Sample Size*:

15.4.1 Tinplate samples can be of various dimensions but must be large enough to afford a test area at least 1½ in. in diameter. The test area must be reasonably flat. Sampling procedures for tin coating weight testing and applicable standards for the specific class designations are outlined in Specifications A 624/A 624M and A 626/A 626M.

15.5 *Preparing Sample*:

15.5.1 The surface of the tested sample shall be clean. Oil, grease and organic coating such as lacquer shall be removed with suitable solvents. Remove oxides, passivation oxide coatings, and corrosion products, by carefully burnishing the test surface with a clean, soft pencil eraser. Also, cleaning with a tissue impregnated with acetone or solvent is usually sufficient. Electrochemical cleaning may be necessary occasionally.

15.6 *Special Precautions*:

15.6.1 It should be noted that the reading of the alloy counter represents the actual weight of tin contained in the $FeSn_2$ alloy. If the tinplate has not undergone heat treatment the

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alloy layer does not form. Unless the instrument is adjusted to the proper setting, the alloy counter will become inoperative, and the automatic program will stop after the first layer is completely removed.

15.6.2 Automatic prepolarization and light electro-cleaning of cathodes is performed during the first 3 s after each start of the Stannomatic III or later model.

15.6.2.1 During this initial sequence the auxiliary anodes are activated against the cathodes. This operation may be extended by pressing the start button for several seconds.

15.6.2.2 Failure of this function may cause ghost layers and subsequent premature switch over of the sequence.

15.6.3 One condition for reliable results and a well-defined inflection point displayed on the instrument meters is a uniform stripping of the whole surface.

15.6.3.1 After completion of the measurement the test surface should be examined visually. If the dissolution of the coating is not complete the measurement should be discarded and repeated.

15.6.4 The following factors influence the uniformity of stripping:

15.6.4.1 Contaminated or dirty cathode.

15.6.4.2 Cathode not well fitted and located.

15.6.4.3 Cathode and fixing part substituted with non-original parts.

15.6.4.4 Sample punched too near the edges of the metal strip may have a non-uniform coating.

15.6.4.5 Sample dirty, corroded, partially or totally coated with organic coating.

15.6.4.6 Sample with a thick passivation layer.

16. Procedure

16.1 Clean the tinplate sample disk by cathodically treating in 0.5% sodium carbonate solution for 10 s with a current density of 0.5 A/in.[3]

16.2 An alternative procedure is to wipe the surface of the sample with a clean cloth saturated with acetone, this to be followed by immersion of disk in acetone, removing, and air drying. The procedure under 16.1 is preferred as it reduces the tin oxide present on the surface to metallic tin and improves the definition of the end point.

16.3 After following steps under Section 10, the apparatus is ready for determining tin coating weight of the samples. Exactly the same procedure is used for samples with unknown tin coating weights as the "dummy" samples. The recommended recorder has a chart speed of 2 in./min and uses a chart having 10 divisions/in. Therefore, each division is equivalent to 3 s.

17. Calculation

17.1 Calculate results as follows:

$$A = ITC \quad (1)$$

$$B = 0.65\, IT_1C \quad (2)$$

$$D = A + B \quad (3)$$

where:

A = free tin coating weight, lb/bb
I = stripping current, A,
T = time required for the removal of free tin, s,
C = 0.02126 or the electrochemical equivalent of tin expressed in lb/bb for a 4-in.^{2} or 25.81-cm^2 area of tinplate,
B = alloy tin coating weight, lb/bb,
0.65 = that portion of the current required for removal of the alloyed tin. Since the tin-iron alloy composition is $FeSn_2$ and these elements enter the solution stoichiometrically with a two-electron loss for each atom of tin and iron, only two thirds of the current is dissipated for the dissolution of tin. Consequently, the total alloy time must be corrected to compensate for the electrolysis of the iron,
T_1 = time required for the removal of the alloy layer, s, and
D = total tin coating weight, lb/bb.

18. Precision and Bias

18.1 Comparison of data using the Sellar's method with that obtained using the electrolytic method shows the following expected bias: ±0.01 lb/bb for 0.50-lb coatings and ±0.02 lb/bb for 1.25-lb coatings.

METHOD C—DETERMINATION OF THE TIN COATING WEIGHTS BY THE SELLAR'S METHOD

19. Scope

19.1 This test method covers the determination of the total weight of tin carried by a unit area of tinplate.

20. Summary of Test Method

20.1 The tinplate sample is dissolved in hydrochloride acid. The tin goes into the solution as stannous chloride. Accidental air oxidation of the tin from the stannous to the stannic form is prevented by completing the analysis under an atmosphere of carbon dioxide.

20.2 The tin is titrated with a standard iodide-iodate solution, using starch as an indicator.

21. Significance and Use

21.1 This test method covers determination of the total tin in the sample tested and does not apportion the tin to one or the other side of the test specimen. The calculations appearing in Section 27 assume uniform distribution of tin over the two surfaces.

21.2 This test method does not differentiate between free tin on the tinplate surface, tin combined with iron in the intermediate alloy layer, or tin alloyed with the steel as a residual tramp element.

22. Interferences

22.1 This test method relies on a redox reaction. Therefore, any contaminant that may be reduced by the nascent hydrogen liberated as the sample dissolves in hydrochloric acid and may be subsequently oxidized by the KI-KIO_3 titrant, must be avoided. The chromate treatments commonly applied to tinplate have not been found to cause significant errors.

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23. Apparatus

23.1 *Reaction Vessel*, [9] such as a 500 mL Erlenmeyer flask, closed with a two-hole stopper. One opening in the stopper shall be connected to a source of carbon dioxide (CO_2) gas, the other, allowing escape of the hydrogen, displaced air, CO_2, and acid vapors shall be connected to a water trap. This will scrub the hydrochloric acid fumes from the emerging gas and afford a seal against air returning to the flask during cooling. The connections used should be of such length and flexibility as to permit moving the flask from the hot plate to the cooling trough without disconnecting the tubing or removing the stopper.

NOTE 2—The essential feature of the apparatus is the provision of means for passing a stream of CO_2 gas over the acid solution in which the tin is dissolving, during and after the dissolution and during the period of cooling the resultant solution to room temperature.

23.2 Any suitable buret may be used for titration, provided its accuracy is known to be adequate for the purpose. Automatic burets are convenient and those incorporating easy-read features aid in obtaining accurate readings.

24. Reagents and Materials

24.1 *Purity of Reagents*—See 6.1.

24.2 *Purity of Water*—See 6.2.

24.3 *Hydrochloric Acid* (1+1)—Add 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) to 1 volume of water and mix well.

24.4 *Potassium Iodide-Potassium Iodate Solution* (0.0975 *N*)—Place 21.8 g of KI, 3.48 g of KIO_3, and 1.2 g of NaOH in a clean bottle. Add 1 L of water and mix thoroughly.

24.4.1 Standardize the solution as follows: Place 0.2500 g of National Institute of Standards and Technology tin, 0.5 g of antimony, and 1 g of lead in a 500-mL Erlenmeyer flask. Add 200 mL of HCl (1+1). Stopper the flask (reaction vessel) with a two-hole rubber stopper and connect to the apparatus described in Section 24. Purge the air from the flask with CO_2. Place the flask on the heater and bring the solution to a boil. Boil gently until the tin is completely dissolved as shown by hydrogen no longer being evolved. When cool, titrate with the KI-KIO_3 solution, while maintaining an atmosphere of CO_2 in the flask. Calculate the weight of tin equivalent to 1 mL of iodate solution as follows:

$$\text{Grams of tin/mL solution} = A/B \quad (4)$$

where:

A = tin, g, and

B = KI-KIO_3 solution required for titration of the solution, mL

NOTE 3—The ideal concentration is one that gives a numerical value of 0.005786 g of Sn/mL of solution. Values between 0.00577 and 0.00580 are considered acceptable; otherwise, the concentration should be adjusted. Using a solution of the concentration indicated, 1 mL of solution corresponds to a coating weight of 0.1000 lb Sn/bb [1.121 g Sn/m^2] of surface when using 4 in.^{2}[25.81 cm^2] of tinplate as the sample.

24.5 *Starch Solution*—Dissolve 2.5 g of NaOH in 500 mL of water. Place about 200 mL of water in a Florence flask and bring it to a boil. Add 2.5 g of soluble starch to 10 mL of water and stir to make a smooth paste. Then add this paste cautiously to the boiling water. Remove this solution from the source of heat and shake gently. Pour the starch dispersion into the NaOH solution, dilute to 1 litre, and mix thoroughly.

25. Test Specimens or Samples

25.1 Sampling procedures for tin coating weight testing and applicable standards for the specific class designation are outlined in Specification A 624/A 624M and Specification A 626/A 626M.

25.2 The test specimens or areas to be sampled should be free of visible contamination by oil, grease, or other substance that would interfere with the dissolving of the sample. The oil coating normally applied to tinplate is not objectionable and need not be removed.

25.3 The area of the test specimen should be known to the degree of precision required in the result. An area of 4 in.^{2} [25.81 cm^2] of tinplate (8 in.^{2}[51.61 cm^2] total surface) is commonly used, and simplifies the arithmetical calculations.

26. Procedure

26.1 The following procedure applies to samples of tinplate approximately 4 in.^{2} in area. For substantially larger or smaller areas, the quantities of reagents used should be adjusted appropriately.

26.1.1 Place 200 mL of HCl (1+1) in the test flask.

26.1.2 Insert the test specimen, cutting or bending it as necessary to pass through the neck of the flask.

26.1.3 Immediately replace the two-hole stopper and purge the gas space with CO_2 gas. Permit this gas to flow during the dissolution of the sample and the cooling of the flask.

26.1.4 Place the flask on the heater and boil the solution gently. Continue heating for about 20 min until the sample has completely dissolved. At this point, the small bubbles characterizing the hydrogen evolution will be replaced by the larger steam bubbles.

26.1.5 Immediately transfer the flask from the heater to the cooling tank or trough, without removing the stopper.

26.1.6 When the solution has cooled, remove the two-hole stopper and replace with a solid rubber stopper, unless titration is to begin immediately.

26.1.7 Fill the buret with the KI-KIO_3 solution, run out a few drops to ensure that there are no air bubbles in the buret tip, *except* on automatic burets, which are required to be completely filled. Record the volume (unless 0.00 mL).

26.1.8 To the contents of the flask, add 5 mL of starch solution.

26.1.9 Proceed with the titration, swirling the contents of the flask gently until the end point is nearly reached. When nearing the end point, add the iodate solution dropwise, swirling the contents vigorously.

26.1.10 When a permanent blue color first appears in the solution in the flask, shut off the buret and record the volume used.

[9] A diagrammatic sketch of a suitable apparatus can be found in Scott, *Standard Methods of Chemical Analysis*, Fifth Edition, Vol 1, p. 967. Explanatory notes accompany the sketch.

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27. Calculation

27.1 Determine the tin coating weight in pounds of tin per base box as follows:

$$\text{Tin coating weight, lb Sn/bb} = [4.00 \times (V_2 - V_1) \times F \times 0.1]/(0.005786 \times A) \quad (5)$$

where:

V_2 = final reading of buret, mL,
V_1 = original reading of buret, mL,
F = solution factor, g of Sn/mL of solution (see 24.4.1), and
A = tinplate area, in.^{2}

27.2 When the iodate concentration has the value recommended in 36.4, the area of tinplate is the commonly used value of 4.00 in.^{2}, and an automatically zeroing buret is used, determine the weight as follows:

$$\text{Tin coating weight, lb Sn/bb} = 0.1 \times V_2.$$

28. Precision and Bias

28.1 Since the coating weight of tin on tinplate varies over the surface, it is difficult to provide identical samples for purposes of cross checking. The titration procedures are, however, sufficiently standard that the normal uncertainty of ±1 drop of titrant should be attainable, corresponding to ±0.005 lb Sn/bb.

METHOD D—DETERMINATION OF THE TIN COATING WEIGHTS BY THE TITRATION METHOD

29. Scope

29.1 This test method is normally applicable to the determination of tin coating weights in the range of 0.50 to 1.50 lb of tin/bb.

NOTE 4—For the determination of the coating weights outside of this range, an iodate solution of appropriate concentration should be prepared so that suitable titrant volumes are obtained. For the determination of low tin coating mass in the range from 0.05–0.49 lb/bb, the number of sample specimens should be adjusted appropriately to maintain a minimum total tin mass equivalent to that present on a 0.50 lb/bb specimen. This test method cannot be used to test single spot tin mass for low tin coating material.

30. Summary of Test Method

30.1 The sample is placed in a platinum-wire bracket and the tin coating is dissolved from the steel base with hydrochloric acid. The removal of the tin from the steel base is hastened by development of an electrical potential between the tin and the platinum. This assures complete dissolution of the tin before a substantial amount of iron is dissolved. Any stannic tin that has formed is reduced to the stannous state with the aid of aluminum. The tin is then titrated with a standard iodate-iodide solution.

31. Apparatus

31.1 *Platinum Contact Device for Stripping of Sample*—Take a 10-in. [254-mm] piece of platinum wire (0.075 in. [1.90 mm] in diameter), and make a loop at one end approximately 2 in. [51 mm] long and ½ in. [12.7 mm] wide. Make certain that approximately 5 in. [127 mm] of this wire is bent perpendicularly to the loop. Then take approximately 4 in. [102 mm] of fine platinum wire (0.02 in. [0.5 mm] in diameter) and coil it around opposite ends of the loop at various intervals.

31.2 *NBS Certified Buret*, 50 mL.

31.3 *Tin Reduction Apparatus*—Any equipment that will maintain an atmosphere of CO_2 in the reaction flask may be used.

32. Reagents

32.1 *Purity of Reagents*—See 6.1.

32.2 *Oxygen-Free Water*—Bubble CO_2 gas through 3 L of water for approximately 15 min.

32.3 *Aluminum Wire*, (A1), tin-free and relatively pure (not less than 99.50% Al). Seven inches [178 mm] of 12-gage [2.05-mm] wire weighs approximately 1 g.

32.4 *Potassium Iodate-Potassium Iodide Solution* (0.05 *N*) (1 mL = approximately 0.0029 g of tin)—Dissolve 1.7600 g of KIO_3 in 200 mL of water containing 0.5 g of KOH and 15.0 g of KI. When dissolution is complete, transfer to a 1-L volumetric flask, dilute to volume, and mix.

32.5 *Starch Solution* (1 g/100 mL)—Add 1 g of either soluble or arrowroot starch to 5 mL of water and stir to make a paste. Then add the paste to 100 mL of boiling water. Cool the solution before using. This solution must be prepared freshly each day.

32.6 *Standard Tin Solution* (1 mL = approximately 0.0029 g of tin)—Accurately weigh 2.9 g of pure tin in 100 mL of concentrated HCl (sp gr 1.19) at room temperature. When dissolution is complete, transfer to a 1-L volumetric flask, dilute to volume with HCl (1+1), and mix.

NOTE 5—In diluting the standard tin solution to volume, care must be employed in maintaining the temperature at which the volumetric flask was calibrated. This also applies when measuring aliquots for standardization purposes. If a National Institute of Standards and Technology certified volumetric flask is not available, the flask should be standardized with a National Institute of Standards and Technology Certified Buret.

32.7 *Tin Metal*—99.95% tin or better (National Institute of Standards and Technology melting point tin is approximately 99.99% pure).

32.8 *Antimony Metal*—80-mesh, reagent grade.

33. Test Specimen or Sample

33.1 Sampling procedures for tin coating weight testing and applicable standards for the specific class designation are outlined in Specification A 624/A 624 and Specification A 626/A 626M.

33.2 Sample specimens of tinplate for coating weight determinations are obtained by stamping disks of 2.257 ± 0.001 in. [57.33 ± 0.02 mm] in diameter which is equivalent to 4 in.^{2} [25.81 cm^2] of surface area on each face of the disk.

NOTE 6—If it is desirable to strip one side only, mask the opposite side using a suitable material, that is, a cellulose lacquer, wax, or other barrier coating. Appropriate adjustments must be made to the test method to compensate for reducing the test area.

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34. Calibration and Standardization

34.1 By means of a certified buret add directly to the reaction flask an accurately measured volume of standard tin solution (Note 6). Proceed as directed in Section 28, procedure starting with the connection of the reaction flask to the reduction apparatus. Calculate the tin titer of the iodate solution.

NOTE 7—It is preferable to analyze the unknown samples prior to standardizing the iodate solution so that suitable volumes of standard tin solution can be selected. Such a procedure will not only result in a more accurate standardization of the iodate solution but at the same time eliminate unnecessary and time-consuming standardizations.

35. Procedure

35.1 Place a 4-in.^{2}[25.81-cm^2] tinplate specimen on the platinum contact device in such a manner that the specimen rests on the loop and against the perpendicular stem and transfer to a 250-mL beaker. Add 150 mL of HCl (1+1) and strip at room temperature. After the tin coating, including the alloy layer, has been removed withdraw the specimen and rinse well with distilled water, collecting the washings in the original 250-mL beaker. Transfer the stripping solution to a 500-mL Erlenmeyer reaction flask. Add 1 g of aluminum wire and approximately 0.1 g of antimony metal (80 mesh).

NOTE 8—When stripping hot-dipped tinplate, the coating is removed more rapidly by using 100 mL of HCl (9+1) and warming to approximately 70°C.

35.2 Connect the reaction flask to the reduction apparatus. Displace the air in the flask with CO_2 and continue maintaining this flow until the flask is disconnected from the apparatus. After the aluminum wire has dissolved, heat the solution and boil gently for 15 min. Then cool the solution to approximately 20°C. Remove the flask from the reduction apparatus, add 5 mL of starch solution and immediately stopper the reaction flask with a rubber cap in which a hole has been pierced. By placing the end of the buret through the hole, titrate with standard iodate solution to a blue end point.

36. Calculation

36.1 Calculate the weight of tin per base box as follows:

$$lb\ of\ tin/base\ box = \frac{A \times B \times C}{D}$$

where:

A = standard iodate solution, mL,
B = tin titer,
C = 17.28 (factor to convert grams of tin on a 4-in.^{2} specimen to pounds per base box), and
D = number of 4-in.^{2} specimens tested (both sides stripped)

37. Precision and Bias

37.1 The following table shows the tin coating weight averages reported by ten participating laboratories. Each tabulated value was the average of 20 determinations except for Laboratory "I" in which case 12 values were reported.

Laboratory	Average lb/bb	Standard Deviation lb/bb
A	0.513	0.0022
B	0.504	0.0027
C	0.512	0.0023
D	0.515	0.0008
E	0.492	0.0033
F	0.509	0.0025
G	0.507	0.0028
H	0.517	0.0032
I	0.532	0.0056
J	0.503	0.0051
Average coating weight	0.510	
Standard deviation of all values reported		0.0013

SUMMARY OF CHANGES

Committee A01 has identified the location of selected changes to this standard since the last issue (A 630 - 98) that may impact the use of this standard.

A 630 - 03:

(*1*) Paragraph 15.3.1– Changed electrolyte from 1.N Hydrochloric acid (HCl) to "As specified by equipment manufacturer."

(*2*) Paragraph 15.6.2– Changed Stannomatic "IV" to "or later model."

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