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AS THE ENTIRE STANDARD
IS REVISED

FED. TEST METHOD STD. NO. 151B
November 24, 1967
CHANGE NOTICE 1
November 4, 1970

FEDERAL TEST METHOD STANDARD

METALS: TEST METHODS

The following changes in Fed. Test Method Std. No. 151B, dated November 24, 1967, have been approved by the Commissioner, Federal Supply Service, General Services Administration, for the use of all Federal Agencies.

1. Table of contents. Place an asterisk (*) after each of the following ASTM test method designations: ASTM A370, E6, E8, E10, E18, E23, E92, E140.

2. Add the following notations at the end of the table of contents: "* The test methods noted by asterisk are the subject of International Standardization Agreement, ABC-NAVY-STD. No. 32. When an amendment, revision or cancellation of this standard is proposed, which will affect or violate the International Agreement concerned, the preparing activity will take appropriate reconciliation action through International Standardization channels including departmental standardization offices if required."

3. In table titled "Industry Methods and Definitions Accepted Under This Standard", add a superscript numeral 2 after title and add the following footnote at end of table:

"2/ Methods subject to International Agreement are identified by an asterisk (*) in table of contents."

RETAIN THIS COVER PAGE AND INSERT BEFORE THE TABLE OF CONTENTS OF THIS STANDARD.

4.1.5 Procedure E, stripping with hydrochloric acid. After cleaning the test specimens as described in 3.2, weigh each specimen separately to the nearest 0.01 gram. Place specimen in a porcelain dish or a 250-ml. beaker and add 50 ml. of concentrated hydrochloric acid; for heavy roofing ternes use 100 ml. of hydrochloric acid. As soon as the coating and alloy layer have dissolved, remove the specimen from the acid and wash with a spray of water. Dry the specimen quickly by immersing in acetone and then wiping with a clean cloth. When the sample attains room temperature, reweigh. The loss in weight represents the weight of coating plus some iron dissolved from the steel sheet.

4.1.5.1 Alternate stripping method. If the antimony content of the terne metal is high, great difficulty will be experienced in dissolving the alloy layer. A long time in the acid will be required and as a result, a large amount of iron will dissolved. Less iron will be dissolved if the following procedure is used when the terne coating is high in antimony (0.5 to 1 percent). Place the cleaned and weighed sample in a 250-ml. plastic beaker that contains 125 ml. of 20 percent sodium hydroxide to which 10 ml. of 30 percent hydrogen peroxide has been added. Cover the beaker to prevent loss due to spattering. When the alkaline soluble part of the coating hass been removed, lift the disc from the liquid and hold it over a second plastic beaker while it is washed with a spray from a wash bottle.

This second beaker is used to prevent dilution of the 20 percent sodium hydroxide. Then place the disc in a glass beaker containing 125 ml. of concentrated hydrochloric acid. When it appears that all of the alloy layer has been dissolved or is adhering loosely to the base steel remove the disc from the acid, place it in a beaker of water and scrub it with a rubber policeman. Wash the disc with a jet of water from a wash bottle, immerse it in acetone and dry it with a clean cloth. After the sample attains room temperature, reweigh. The loss in weight represents the weight of coating plus some iron dissolved from the steel sheet.

4.1.5.2 Determination of iron dissolved.

4.1.5.2.1 Solutions.

(a) Stannous chloride. Dissolve 60 g. of $SnCl_2 \cdot 2H_2O$ in 600 ml. of concentrated hydrochloric acid and dilute to 1000 ml. with distilled water. Transfer to a pyrex bottle and add 1 g. of tin shot.

(b) Saturated mercuric chloride solution. Dissolve 200 g. of $HgCl_2$ in 1000 ml. of hot water and transfer to pyrex bottle. A smaller quantity in the same proportion may be prepared if desired.

(c) Potassium dichromate solution 0.10 N. Dissolve 4.903 g. of $K_2Cr_2O_7$ in 500 ml. of distilled water in a 1000-ml. volumetric flask and dilute to the mark with water. Standardize by any appropriate procedure.

(d) Diphenylamine sulfonic acid. Dissolve 0.32 g. of barium diphenylamine sulfonate in 100 ml. of water. Add 1 ml. of sulfuric acid and mix. Let the precipitated barium sulfate settle and decant the clear liquid.

4.1.5.2.2 Procedure. Dilute the stripping solution from 4.1.5 or 4.1.5.1 so that the acid concentration is approximately 1:1. Reduce the volume to 30 to 40 ml. by boiling and immediately add stannous-chloride solution (a) until the solution is decolorized and then add 4 drops in excess. Cool the solution quickly in running water and add 10 ml. of saturated mecuric-chloride solution (b) let the solution stand 2 minutes and titrate with 0.10N. potassium dichromate solution (c). Use 6 drops of phenylamine sulfonic acid (d) as an internal indicator. When near the end point add 5 ml. of 85 percent orthophosphoric acid and finish titration. Calculate the grams of iron stripped from the test specimen as follows:

Iron, grams = AB

Where:

A = ml. $K_2Cr_2O_7$ solution required to titrate sample and
B = iron equivalent (g. Fe per ml.) of standardized $K_2Cr_2O_7$ solution (for 0.10N. solution 1 ml. = 0.005584 g. Fe).

4.1.5.3 Weight of coating. Calculate the weight of coating in pounds per double base box as follows:

Weight of coating = (A-B) X 34.57

Where:

A = loss in weight of specimen, in grams (see 4.1.5 or 4.1.5.1),
B = grams of iron stripped from the test specimen (see (b) in 4.1.5.2), and
34.57 = fact to convert grams of coating on a 4-square-inch specimen to pounds per double base box.

WEIGHT AND COMPOSITION OF COATING ON SHORT TERNE PLATE (FOR MANUFACTURING PURPOSES AND FOR ROOFING)

1. SCOPE

1.1 This method covers procedures for determining the weight and compostion of coating on short terne plate by the triple-spot method and for determining composition of coating by the pot metal analysis method.

2. DEFINITIONS

2.1 Base box. The base box (bb.) is a unit of area, 112 sheets 14 in. by 20 in. or 31,360 sq. in.

2.2 Weight of coating. Coating weights are customarily expressed in pounds per double base box which is twice the area of a single base box or 62,720 sq. in.

2.3 Terne metal. Terne metal is a lead-tin alloy.

2.4 Compostion of coating. The composition of the coating consists of lead, tin, and a small amount of iron dissolved from the steel sheet.

3. PREPARATION OF SPECIMENS

3.1 Three test specimens, 2.257 ± 0.010 inch in diameter or 2.000 ± 0.010 inch square, shall be cut from each test sheet, one being cut from the center and the other two from diagonally opposite corners, adjacent to and within the rectangle formed by lines drawn 2 inches from the sides and 4 inches from the ends of the sheet as shown in figure 1. The 2.257-inch diameter disc and the 2.000 inch square are each equivalent to an area of 4 square inches. The weight of coating in grams on either of these specimens X 34.57 expresses the coating weight in pounds per double base box where 34.57 is the factor to convert grams per 4 sq. in. to pounds per double base box.

3.2 The test specimens shall be cleaned with an appropriate pertroleum solvent, rinsed in alcohol or boiling menthanol, and then dried thoroughly.

4. PROCEDURE

4.1 Weight of coating. The weight of coating shall be determined by the difference in weight of coated specimens before and after using one of the stripping procedures which follows:

Procedure A - Sulfuric acid.
Procedure B - Electrolytic.
Procedure C - Silver nitrate solution.
Procedure D - Hydorchloric acid and antimony trichloride.
Procedure E - Hydrochloric acid.

4.1.1 Procedure A, stripping with sulfuric acid. For testing procedure refer to ASTM A 309. This procedure is applicable for weight of coating on short terne plate, using specimen size and conversion factor given in 3.1. The amount of iron dissolved from the steel sheet may be determined and coating weight corrected. For details refer to sections 3, 4, 5 (a) and (b) and 6(a) of ASTM A 309.

4.1.2 Procedure B, electrolytic stripping. For testing procedure refer to ASTM A 309. This procedure is applicable for weight of coating on short terne plate, using specimen size and conversion factor given in 3.1.

4.1.3 Procedure C, stripping with silver nitrate solution. For testing procedure refer to ASTM A 309. This procedure is applicable for weight of coating on short terne plate, using specimen size and conversion factor given in 3.1.

4.1.4 Procedure D, stripping with hydrochloric acid and antimony trichloride. For testing procedure refer to ASTM A 309. This procedure is applicable for weight of coating on short terne plate, using specimen size and conversion factor given in 3.1.

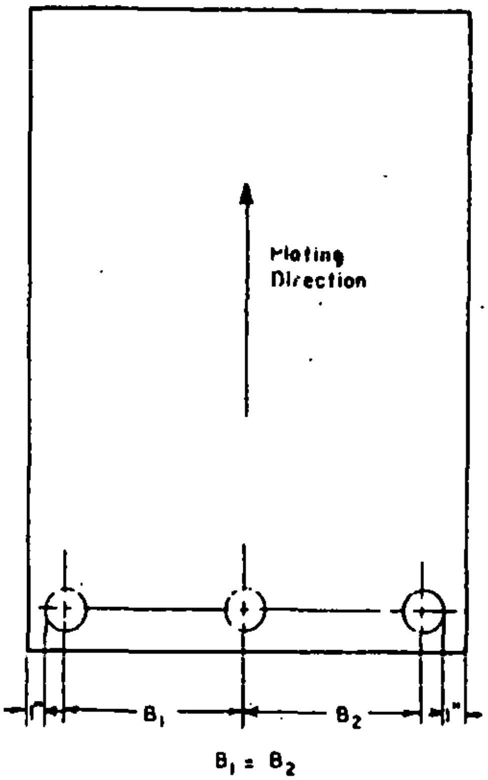

FIGURE 2. Electrolytic tinplate determination of weight of coating. Location of sample discs or spot tests.

5. REPORT OF RESULTS

5.1 Results shall be reported on forms either furnished or approved by the procuring agency. The report shall refer to the contract or purchase order and shall include all information request by the procuring agency.

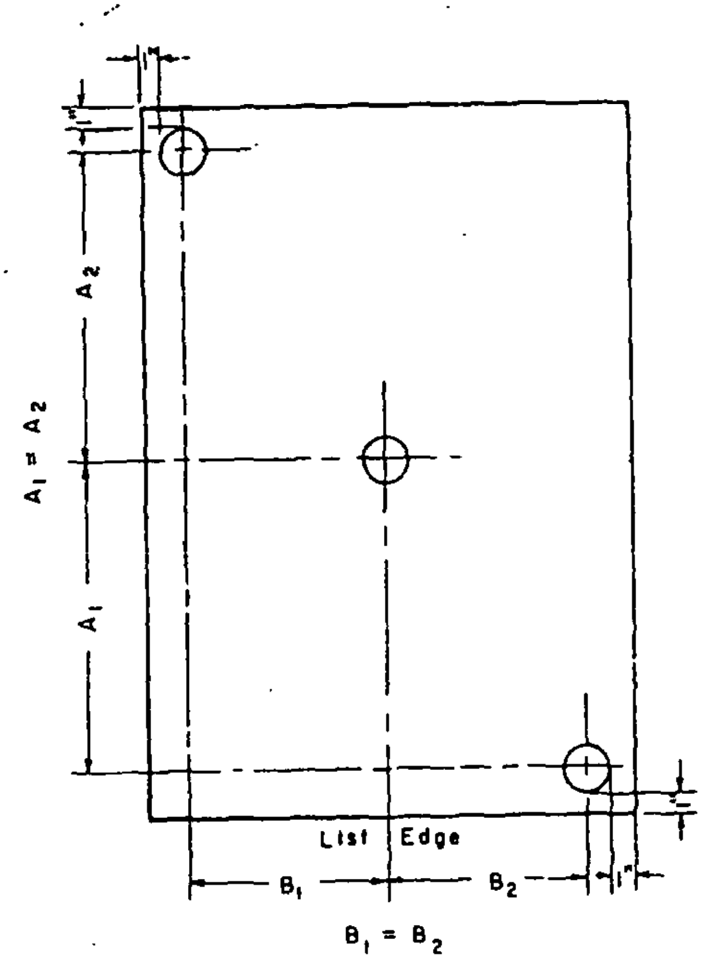

FIGURE 1. Hot-dipped tinplate determination of weight of coating. Location of samples discs or spot tests.

$$\text{Weight of coating, lb./bb.} = AB \times 17.28$$

Where:

A = ml. of iodate solution required to titrate sample,
B = tin equivalent (g. Sn per ml.) of above standard iodate solution, and
17.28 = factor to convert grams of tin on a 4-square-inch specimen to pounds per base box.

4.1.6.4 Notes.

(1) The referee method was developed by W. F. Frederick (deceased), United States Steel Corporation, H. A. Stobbs, Wheeling Steel Corporation, and D. P. Robertson, Weirton Steel Company at the request of the A.I.S.I. Technical Committee on Tin Plate. The objective was to provide an accurate method for use as a recognized referee method.

(2) The standard iodate described under "reagents" will give suitable titrant volumes when used on tinplate from 0.25 to 1.50 pounds of tin per base box. For determining tin-coating weights outside of this range an iodate solution of such a concentration should be prepared so that suitable titrants volumes are obtained (that is, iodate solution should be stronger or weaker then that indicated).

(3) It is suggested that it may be 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 result in more accurate standardization of iodate solutions and at the same time eliminate unnecessary standardizations.

(4) In making the standard tin solution up to volume, care must be employed in maintaining the temperature at which the volumetric flask was calibrated. This applies when measuring aliquots for standardization purposes. All standard solutions should be measured with a standard burette.

(5) For stripping hot dip tinplate, the coating is removed more rapidly by using 40 percent hydrochloric acid by volume and warming to approximately 70° C.

(6) The customary practice of allowing burettes to drain before reading the volume must be employed.

(7) All burette readings must be corrected in accordance with the National Bureau of Standards Certificate.

(8) If a National Bureau of Standards certified volumetric flask is not available, the flask used should be standardized with a standard burette.

4.1.7 Sellar's method. The determination of tin-coating weight by this method is based upon the oxidation of stannuous to stannic tin by the addition of an oxidizing agent. In this method the tinplate sample is dissolved in c.p. hydrochloric acid and goes into solution as iron and tin chlorides. The tin goes into solution as stannous chloride since hydrogen, a reducing agent, is evolved by the action of the acid on the tin and iron. The tin is maintained in the stannous form by carbon dioxide, a neutral gas, which is passed through the flask during solution, after solution, and cooling of the sample. The following reactions take place:

$$Sn + 2HCl = SnCl_2 + H_2$$
$$Fe + 2HCl = FeCl_2 + H_2$$

Potassium iodate is used as an oxidizing agent to titrate the stannous chloride. Since oxygen in the air will also oxidize the stannous tin, the blanket of carbon dioxide over the solution must be maintained throughout the entire determination. Any oxidation of the stannous chloride by the oxygen of the air will decrease the amount of potassium iodate necessary for the titration and cause low coating weight values. The oxidation of stannous to stannic chloride with potassium iodate is done in the presence of an excess of potassium iodide and starch solution. The starch solution serves as an indicator but the potassium iodide enters into the reaction as follows:

$$KIO_3 + 5KI + 6HCl = 6KCl + 3H_2O + 3I_2$$

As long as any stannous chloride remains it reacts as follows with the iodine liberated by the above reaction:

$$I_2 + SnCl_2 + 2HCl = SnCl_4 + 2HI$$

Both of the above reactions require an excess of hydrochloric acid which is supplied by the excess acid in the flask. When the stannous chloride has all been oxidized the iodine liberated is no longer reduced to iodide ions and reacts with the starch present to give the solution a blue color indicating the end of the reaction and titration. This method determines the total tin or the sum of the combined tin in the alloy layer and the free tin. It is not possible to differentiate between free tin and combined tin.

4.1.6.1 Apparatus.

4.1.6.1.1 Platinum contact for stripping of sample. It is suggested that approximately 14-gage platinum wire at least 24 inches long be coiled into a double spiral whose outside diameter approximates that of the specimen. Approximately 5 inches of this wire is not coiled but is bent perpendicular to the spiral. The straight length of wire facilitates handling of the sample during the stripping operation.

4.1.6.1.2 National Bureau of Standards Certified Burette. Fifty m. capacity is preferred.

4.1.6.1.3 Seller's apparatus. Sellar's apparatus as described in Scott's "Standard Methods of Chemical Analysis", fifth edition, page 966, or any similar equipment that will maintain an atmosphere of carbon dioxide in the flask may be used.

4.1.6.1.4 Reaction flask. Either a 500-ml. Erlenmeyer or Kjeldahl flask may be used.

4.1.6.2 Reagents.

4.1.6.2.1 Aluminum wire. A tin-free, relatively pure, aluminum (not less than 99.50 percent). Seven inches of 12-gauge wire weighs approximately 1 gram.

4.1.6.2.2 Tin metal. 99.95 percent tin minimum (National Bureau of Standards melting point tin is approximately 99.99 percent pure).

4.1.6.2.3 Starch solution (10 grams per liter). Prepare by making a paste of 1.0 g. of either soluble or arrowroot starch in about 5 ml. of water and add to 100 ml. of boiling water. Cool before using. If a preservative is added, it will not be necessary to prepare this solution daily.

4.1.6.2.4 Air-free water. To 3 liters of distilled water which has been boiled and cooled, add 30 g. of sodium bicarbonate and 25 ml. of concentrated hydrochloric acid.

4.1.6.2.5 Standard tin solution (1 ml. equals approximately 0.0029 g. of tin.) For standardizing the iodate solution (4.1.6.2.6), it is preferable to take aliquots from a standard master tin solution rather than weigh individual tin samples. Prepare the standard tin solution by taking approximately 2.9 g. of pure tin (4.1.6.2.2) and weighing it accurately. Dissolve the tin in 100 ml. of concentrated hydrochloric acid and then dilute to 1000 ml. in a volumetric flask, with 1:1 hydrochloric acid (see notes 4 and 8).

4.1.6.2.6 Standard potassium-iodate solution (1 ml. equals approximately 0.0029 gram of tin.) Dissolve 1.7600 g. of potassium iodate (AR), in 200 ml. of water containing 0.5 g. of potassium hydroxide (AR), and 15.0 g. of potassium iodide (AR),. When solution is complete, dilute to 1000 ml. in a volumetric flask with water. Standardize the iodate solution against standard tin solution (4.1.6.2.5) using such volume of tin solution so as to give approximately the same titration as the sample. This is essential. In standardizing the iodate, analytical reagent solution, the standard tin solution must be carried through all steps indicated in the "procedure". No provision is made for the deduction of a blank since this cancels out when the titrations for the sample and standard solutions are approximately equivalent. In some cases it may be necessary to run a series of standards containing various amounts of tin to correspond to samples of varying tin content (see notes 2 and 3).

4.1.6.3 Procedure. A 4-square-inch tinplate specimen, either a disc or a square, as described in 3.1, is placed between the spirals of the platinum contact device and immersed in 100 ml. of 1:1 hydrochloric acid contained in a 250-ml. beaker or any suitable container. When the coating, including the alloy layer, is removed, the specimen is withdrawn from the solution and any adhering solution washed into the original beaker with three portions of distilled water. The contents of the beaker are then quantitatively transferred to a 500-ml. Erlenmeyer or Kjeldahl flask, and 1 g. of aluminum wire (4.1.6.2.1) is added. The flask is connected to the Sellar's apparatus. The air in the flask is displaced with carbon dioxide and a continuous flow maintained until the flask is disconnected from the apparatus. After the aluminum is in solution, heat is applied and the solution gently boiled for approximately 15 minutes. Remove from the heat and cool with tapwater to approxiamtely 20° C. Remove the flask from the Sellar's apparatus; immediately add two or three marble chips and wash the sides of the flask with 60 ml. of air-free water (4.1.6.2.4) containing 5 ml. of starch solution (4.1.6.2.3). Titrate immediately with standard iodate solution (4.1.6.2.6) to a blue end point. Calculate the weight tin coating in pounds per base box from the ml. of standard iodate solution used in titration as follows:

4.1.3.1.1 Antimony-trichloride solution. Dissolve 120 g. of antimony-trichloride crystals in 1000 ml. of concentrated reagent grade hydrochloric acid.

4.1.3.2 Procedure. A 4-square-inch specimen, either a disc or a square as described in 3.1, is used. Clean the specimen with a good solvent as described in 3.2. Weigh it accurately to the nearest tenth of a milligram and then drop it into the stripping solution quickly (see 4.1.3.1.1). If this operation is not done quickly, the fumes of the stripping solution attack the surface of the plate and the coating will not be completely removed. Allow the sample to remain in the stripping solution for 15 to 30 seconds after the evolution of gas has ceased. Remove it from the solution and wash with cold water from a tap while rubbing with a cloth to remove the antimony adhering to the surface. Dry with a clean cloth or by immersing in acetone and then allow to air dry. Reweigh after the specimen has attained the temperature of the balance room. The loss in weight represents the amount of tin and iron-tin alloy on the specimen and this loss in weight in grams time 17.28 expresses the coating weight in lb./bb. where 17.28 is the factor to convert grams per 4 sq. in. to lb./bb.

4.1.4 Electrostripping method-constant current-acid electrolyte. This method may be used on a routine basis to determine not only the weight of tin coating but also to determine that part of the coating which is present as free tin and that present in the combined or alloyed form. It consists of stripping the tin from a 4-square-inch sample of tinplate anodically at constant current in an electrolyte of 1.0 N hydrocloric acid. The potential difference developed between the sample and a sliver reference electrode is plotted against time on a strip chart recorder. The times required for stripping the free tin and alloy tin, respectively, are read from the resulting plot. Since the stripping current has been preset, the free-tin and alloy-tin coating weights may be calculated by employing Faraday's of electrolysis.

4.1.5 Electrostripping method-variable current-caustic electrolyte. This method may be used to determine not only the weight of tin coating but also to determine that part of the coating which is present in the combined or alloyed form. Factors (counts) are obtained which are related to the coating weight with the use of standard tinplate specimens. The tin is stripped anodically from a tinplate sample 2.97 inches in diameter. A caustic electrolyte is employed. The stripping cell current is not held constant, but varies inversely as the resistance and back voltage of the cell. The magnitude of the stripping current is used to control the rate of oscillation of a vacuum tube oscillator. The higher the current the higher the oscillatory frequency. By counting the number of oscillations occurring during the stripping, a number is obtained that is directly proportional to the amount of current (coulombs) required to remove the tin. With the use of standard tinplate specimens, the counting rates can be adjusted, so that total counts read can be made to indicate the weight directly in pounds per base box. When the free-tin is being removed, the stripping current will be high, the counting rate rapid, and the voltage of the cell low. The cell voltage will increase when the alloy layer is encountered. The current will decrease, thus the counting rate will drip. This increase in voltage is used to start another counter, which counts the number of oscillations taking place during the removal of the alloy layer. When all the tin has been removed, the cell voltage will increase again to a still higher voltage. This increase in voltage at the end of the stripping is used to stop both counters. Seperately powered insoluble anodes in the cell are used to keep the dissolved tin oxidized and to keep the total tin content of the bath at a low level.

4.1.6 Referee method. This method consits of removing the tin from the steel base by stripping with hydrochloric acid, reducing it to the stannous state and then titrating with a standard iodate-iodide solution. This method is theoretically sound has been demonstrated to be the most accurate of all methods. The reproducibility of results among laboratories analyzing duplicate samples of tinplate using the referee method has been checked by statistical methods. For two different tin-coating weights, two analysts in each of three separate laboratories analyzed four sets of five sample each. The reproducibility among laboratories on duplicate samples was found 99 times out of 100 to be as shown below:

	Pounds per base box	
Specified tin-coating weight	0.25	0.75
Reproducibility among laboratories analyzing duplicated samples	±0.01459	±0.01921

Method	Recommended specimen size
Bendix	2.257-inch diameter disc or 2.000 inches square
X-ray	Full size sheet—measurements made over 4-square-inch area.
Antimony trichloride stripping	2.257-inch-diameter disc or 2.000 inches square.
Electrostripping — constant current-acid electrolyte	2.257-inch-diameter disc or 2.000 inches square.
Electrostripping — variable current-caustic electrolyte	2.97-inch-diameter disc.
Referee	2.257-inch-diameter disc or 2.000 inches square.
Sellars	2.257-inch-diameter disc or 2.000 inches square.

In cases of dispute, the determination of coating weight shall be made by the referee method at a laboratory designated by the Government inspector. The antimony trichloride stripping and referee methods are described in detail below. All of the other methods are described briefly. Complete descriptions for all of the above methods are contained in the document entitled "Methods for Determination of Coating Weights of TinPlate" published by the Amercian Iron and Steel Institute and dated December 1959. The average value for the lot obtained from testing the selected test specimens shall represent the average tin coating weight per base box.

4.1.1 Bendix method. This method consists of anodically stripping the tin from the base metal in a dilute hydrochloric-acid solution containing a measured excess of a standard potassium iodate-potassium iodide solution. The following reactions take place:

$$KIO_3 + 5KI + 6HCl \rightarrow$$
$$6KCl + 3H_2O + 3I_2$$
$$Sn + 2HCl \rightarrow Sn\,Cl2 + H_2$$
$$I_2 + SnCl_2 + 2HCl \rightarrow SnCl_4 + 2HI$$

The excess iodine is then titrated with a standard solution of sodium thiosulphate, using starch as an indicator. This reaction proceeds as follows:

$$I_2 + 2Na_2S_2O_3 \rightarrow SnCl_4 + 2HI$$

This method determines the total tin or the sum of the combined tin in the alloy layer and the free tin. It is not possible to differentiate between free tin and combined tin.

4.1.2 X-ray method. The principle of operation of the X-ray fluorenscence tin-coating thickness gage is that a beam of X-rays of a known intensity and wave length is directed at an angle to the surface of the tinplate. It penetrates the tin coating without appreciable loss of intensity, and travels to the steel base where it generates fluorescent or secondary X-rays of a different wave length. These secondary X-rays then emerge in all directions from the same side of the tinplate sheet, and in traveling through the coating are strongly absorbed by the tin. The reduction in intensity is proportional to the thickness of the tin coating. The X-ray fluorescence method permits measurement of tin coating thickness at any desired location on full-size sheets without contacting, cutting, or damaging the sheet in any way. The method determines total tin irrespective of the amount combined as iron-tin alloy, and is not affected by extraneous metallurgical variables, such as grain size and preferred orientation. Measurements are made over a four-square inch area in about thirty seconds.

4.1.3 Antimony trichloride stripping method. This method consists of stripping a weighed sample of tinplate in antimony-trichloride-hydrochloric-acid solution and again weighing. The loss in weight represents the amount of tin coating on the sample, including the alloy layer. The precision of the determination is ± 0.01 lb./bb. if it is assumed that the alloy layer is part of the coating. If it is desired to report the total tin only, the alloy layer coating weight must be determined and a correction applied for the iron present in the alloy, which theoretically is 19.05 percent of the amount of alloy present.

4.1.3.1 *Solutions.*

WEIGHT OF COATING ON HOT DIP TIN PLATE AND ELECTROLYTIC TIN PLATE

1. SCOPE

1.1 This method covers procedures for determining the weight of tin coating on tinplate by the triple-spot method.

2. DEFINITIONS

2.1 Tin-coating weights. Tin-coating weights are expressed in pounds per base box and represent the total of tin coating on both sides with the exception of the coating weight on differentially coated plate. In this latter product the coating weight equivalent represents double the coating for each side.

2.2 Base box. The base box (bb.) is a unit of area, 112 sheets of tinplate 14 by 20 inches or 31,360 square inches.

2.3 Alloy layer. In the production of tinplate a part of the tin coating is combined with iron to form an iron-tin alloy layer.

2.4 Combined tin. Combined tin is that part of the tin coating in the iron-tin alloy layer.

2.5 Free tin. Free tin is that part of the tin coating which is not combined with iron.

2.6 Weight of coating. The weight of coating is the sum of the combined tin and free tin.

3. PREPARATION OF SPECIMENS

3.1 Three test specimens, 2.257 ± 0.010 inch in diameter, 2.000 ± 0.010 inch square, or 2.97 ± 0.010 inch in diameter, shall be cut from each test sheet or strip. For hot dip tinplate, one specimen shall be cut from the center and the other two from diagonally opposite corners. In order to secure representative test specimens, the corner specimens shall be taken so that the outer edge of each sample is at least 1 inch from any edge of the plate as shown in figure 1. The specimens on cut lengths of electrolytic tinplate may either be taken in the same manner as outlined for hot dip tinplate or in a line directly across the plating direction by cutting one specimen from each edge and one specimen equidistant between the two edges. The outer edge of the two edge specimens shall be at least 1 inch from the plate edges which are parallel to the plating direction as shown in figure 2. The specimens on electrolytic coils shall be taken from a strip cut in a line directly across the plating direction with one specimen from each edge and one equidistant between the two edges. The outer edge of the two edge specimens shall be at least 1 inch from the plate edges which are parallel to the plating direction as shown in figure 2. The 2.257-inch-diameter disc and the 2.00-inch square are equivalent to an area of 4 square inches. The weight of coating in grams on either of these specimens times 17.28 expresses the coating weight in pounds per base box where 17.28 is the factor to convert grams per 4 square inches to pounds per base box. The 2.97-inch-diameter disc is the standard specimen for the electrostripping method-variable current-caustic electrolyte and the weight of coating in decigrams on this weight of tin coating in pounds per base box.

3.2 When the antimony trichloride and referee methods are used the specimens shall be cleaned with an appropriate petroleum solvent, and then alcohol followed by thorough drying. When the Bendix and the electrostripping methods are used, the specimens shall be cleaned when necessary. It is not necessary to clean the specimens when coating weights are determined by the X-Ray and Sellars methods.

4. PROCEDURE

4.1 The weight of tin coating shall be determined by any of the recognized methods which follow:

LEAK TESTING (VACUUM)

1. SCOPE

1.1 This method covers a procedure for determining the presence of leakage in a vessel. It consists of evacuating the vessel to be tested and observing the rate of pressure rise. This method will give an approximation of the leakage rate for the average pressure differential during the test period.

2. APPARATUS

2.1 Test setup (fig. 1).

3. MATERIALS

3.1 Cleaning materials (as required):

(a) Solvent, dry-cleaning (P-D-680).
(b) Remover, paint (TT-R-251 or TT-R-230).

4. PROCEDURE

4.1 Clean vessel (inside and out) of all paint, coating, and filler.

4.2 Connect vessel to test setup, and evacuate it to the vacuum specified in the detailed specification or to approximately 1 inch or mecury absolute (0.5 p.s.i.a.), if no vacuum is specified.

4.3 Close valve to vacuum, note pressure and time, and allow the vessel to remain evacuated for the specified time.

CAUTION

Keep the vessel temperature as constant as possible during the test to prevent erroneous results.

4.4 When the specified time has elapsed, note pressure. Compute the rate of pressure rise in the vessel (as a measure of leakage).

5. REPORT OF RESULTS

5.1 Report results on forms either furnished or approved by the procuring agency. Include in the report the contract or purchase order number and all information requested by the procuring agency.

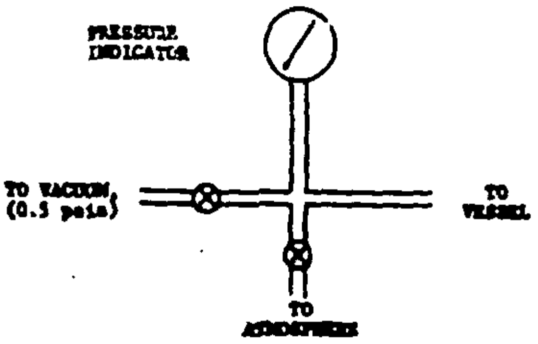

FIGURE 1. Test setup.

LEAK TESTING

(PRESSURIZED GAS)

1. SCOPE

1.1 This method covers procedures for locating leaks in vessels in which leakage is not smaller than 10^{-6} cc. per second. It consists of charging a vessel with a pressurized gas and checking for escaping gas, either visually or by use of a suitable detector.

2. APPARATUS

2.1 Detection apparatus (table I), for pressurized gas (3.1) used.

3. MATERIALS

3.1 Pressurized gas (table 1), pressure less than design pressure of vessel.

3.2 Detection materials (table 1), for pressurized gas (3.1) used.

4. PROCEDURE

4.1 Clean vessel (inside and out) to remove all oil, grease, soap solution, water, and other materials that might interfere with the test.

4.2 Charge vessel with pressurized gas.

WARNING

Jets of gas issuing from pinhole leaks in vessels containing high-pressure gas may cause injury to personnel.

4.3 Detect escaping gas by applying detector to all parts of the vessel (table I), and checking for indication of leak (table I).

4.4 Mark of leaks.

5. REPORT OF RESULTS

5.1 Report results on forms either furnished or approved by the procuring agency. Include in the report the contract or purchase order number, and all information requested by the procuring agency.

TABLE I. Leak test details

Pressurized gas	Detector		Detector application	Leak indication
	Apparatus	Materials		
Air	Tank		Submerge vessel in tank of water	Bubbles
		Soap solution	Flow on soap solution	
Ammonia, 5 percent in air		Sulfur candle Sulfur dioxide 0.1 N hydrochloric acid on swab	Probe near vessel	Wisp of white smoke
Freon, 50 percent in carbon dioxide or nitrogen	Halide torch		Probe near vessel	Change in color of flame
Freon (1 oz. per 30 cubic feet of vessel capacity) in inert gas	Halogen vapor analyzer, with sampling probe		Conduct test in clean air in draft-free enclosure. Hold sampling probe approx. 1/2 in. from vessel and move at approx. 1/2 in. per sec.	Change in halogen vapor analyzer reading

(Activities outside the Federal Government may obtain copies of Federal Specifications, Standards, and Handbooks as outlined under General Information in the Index of Federal Specifications and Standards and at the prices indicated in the Index. The Index, which includes cumulative monthly supplements as issued, is for sale on a subscription basis by the Superintendent of Documents, U. S. Government Printing Office, Washington, D. C. 20402.

(Single copies of this specification and other product specifications required by activities outside the Federal Government for bidding purposes are available without charge at the General Services Administration Regional Offices in Boston, New York, Washington, D. C., Atlanta, Chicago, Kansas City, Mo., Fort Worth, Denver, San Francisco, Los Angeles, and Seattle, Wash.

(Federal Government activities may obtain copies of Federal Specifications, Standards, and Handbooks and the Index of Federal Specifications and Standards from established distribution points in their agencies.)

MILITARY CUSTODIANS:

Army - MR
Navy - AS
Air Force - 11

Review activities:

Army - EL, MI, AT, ME,
AV, MU, WC, MR,
GL, CE
Navy - AS, OS, SH, SA,
YD, MC, CG
Air Force - 69, 11, 23

CIVIL AGENCIES INTEREST:

AGR
COM
DC
GSA
HEW
INT
JUS
VA

Preparing activity:

Army - MR

FEDERAL TEST METHOD STANDARD

METALS; TEST METHODS

GENERAL SECTION

1. SCOPE

1.1 This test method standard covers common requirements that may be omitted from the detail specifications for metals and metal products. This standard forms a part of such detailed specifications when referred to therein. Standard test methods used for measuring the properties of metals and metal products are described herein. Administrative procedures and requirements, and acceptance-inspection standards, may be found in procurement documents and specifications and are not a part of this document. Where conflict exists between this document and the procurement documents, the requirements of the contract, drawing, detail procurement specifications and this standard shall prevail in the order named.

1.2 Numbering system.

1.2.1 Federal methods. General classes of tests are assigned group numbers of whole hundreds. Properties within a class are assigned series numbers and particular tests are assigned numbers within the series.

1.2.1.1 Revision of test methods. Test method revisions will be indicated by the addition of a decimal point and number after the test method number, 111.1 being the first revision of method 111. Subsequent revisions will be numbered consecutively.

1.2.2 Industry methods. Industry methods accepted for Government use in the standard will be indicated by the industry method number and the year or revision indicator of the accepted method as for example ASTM E 95-65 or AMS 2301C.

1.2.2.1 Revision of test methods. Revisions of industry test methods will be coordinated and when accepted by the Government, a change notice to this standard will be issued.

1.2.3 Referencing of industry test methods. Industry test methods accepted in this standard should be referenced directly to the accepted test method in the detail specification and not by cross reference to this standard.

2. DUTIES AND RESPONSIBILITIES OF THE SUPPLIER

2.1 Copies of the results of specified chemical, mechanical, and metallographic tests shall be furnished by the supplier when required by the detail specifications or the purchase contract.

2.2 When material is inspected by melts, heats, or lots, the supplier shall so arrange his working, handling, and marking of the material as to maintain its proper identity. Such procedure shall be available to the Government. When doubt exists as to the identity of any portion of the material sufficient tests shall be made to establish its proper identity.

3. TEST SPECIMENS

3.1 Test coupons or test pieces shall be stamped or otherwise marked for future identification. When match marking is required, two overlapping impressions shall be stamped or marked clearly over each intersection between the material and the test pieces and between additional test pieces. Any applied marks shall remain on the coupons or test specimens until the pieces are tested and necessary records are made. Before the identification stampings or markings are effaced or removed from one location, they shall be transferred to another. The Government may waive any or all of the requirements of this paragraph in cases where they are impracticable of application, wholly or in part.

3.2 If the test metal is an integral part of the material, test pieces shall not be cut off or otherwise removed until the integral part has been subjected to all stages of material processing which may have a significant effect on the properties of the material. If the test metal is not an integral part of the material that it represents, the test coupons or pieces from which the test specimens are to be prepared shall be subjected to the same heat treatment as the material they represent. If the property under evaluation is not dependent upon complete processing, including heat treament of the product, test material can be taken at any appropriate stage in manufacture.

3.3 Test coupon or coupons, or the test specimens taken therefrom, shall not receive any treatment or working, other than by machining, that results in significant change in the properties to be evaluated by the testing, except as provided in specific test methods. Material which has been subjected to subsequent heating opeations may not yield analytical results that correctly represent the original composition.

3.4 When proof stress, elastic limit, proportional limit, precise measurement of modulus of elasticity, or 0.02 percent offset or less yield strength is specified, test specimens shall not be bent, hammered, stressed, or straightened except by removal of material by machining, except as provided for in specific test methods.

3.5 The use of chills in casting test bars shall cause the rejection of the material represented unless the castings they represent are cast in chill molds.

3.6 Replacement of test specimens. A test specimen may be discarded and a replacement test specimen selected from the same lot of material under the following conditions:

(a) Where the specimen is incorrectly machined.
(b) Where the test procedure is incorrect.
(c) Where there is malfunction of the testing equipment.
(d) Where a flaw that is not indicative of an inferior or defective lot of material develops during the test (see 3.6.1).

3.6.1 Internal flaw such as cracks, ruptures, flakes porosity and the like revealed during a a test are considered indicative of inferior or defective material and are not reasons for the selection of a replacement test specimen.

4. REJECTION AND RETESTS

4.1 *Rejection.* Where one or more test specimens fail to meet the requirements of the detail specification, the lot represented by the specimen or specimens shall be subject to rejection except as otherwise provided in a sampling plan approved by the procuring activity or in 4.2.

4.2 Retests. In event of failure of one or more representative specimens retest of additional specimens from the lot will be permitted. If one of the retest specimens fail, the lot shall be rejected with no further retesting permitted.

4.3 Resubmittal of rejected lots. Lots rejected for failure to meet the requirements of the detail specification may be resubmitted for test provided the producer has reworked the lots, as necessary, to correct the deficiencies or has removed the noncomforming material.

5. CHANGES

5.1 When a Federal agency considers that a Federal Test Method Standard does not provide for its essentail needs, written request for adding to or otherwise changing the Test Method Standard, supported by adequate justification shall be sent to the Administration. This justification shall explain wherein the Test Method Standard does not provide for essentail needs. The request shall be sent in duplicate to the General Services Administration, Federal Supply Service, Standardization Division, Washington, D. C. 20016. The Administration will determine the appropriate action to be taken and will notify the agency.

6. NOTES

6.1 Military activities may obtain copies of industry test methods from the Military control stocking point. Application for copies of ASTM methods should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pa. 19103. Application for copies of SAE-AMS methods should be addressed to the Society of Automotive Engineers, Inc., Department A2, 485 Lexington Ave., New York, N. Y. 10017. Application for copies of AMS methods should be addressed to the American Welding Society, 345 East 47th Street, New York, N. Y. 10017.

FEDERAL TEST METHOD STANDARD

METALS; TEST METHODS

Industry Methods and Definitions Accepted Under This Standard 1/

American Society for Testing and Materials (ASTM):

A 90 - Methods of Test for Weight of Coating on Zinc-Coated (Galvanized) Iron or Steel Articles.
A 219 - Methods of Test for Local Thickness of Elecrodeposited Coatings.
A 255 - Methods of End-Quench Test for Hardenability of Steel.
A 309 - Methods of Test for Weight and Composition of Coating on Long Terne Sheets by the Triple Spot Test.
A 317 - Macroetch Testing and Inspection of Steel Forgings.
A 370 - Methods and Definitions for Mechanical Testing of Steel Products.
A 393 - Recommended Practice for Conducting Acidified Copper Sulfate Test for Intergranular Attack in Austenitic Stainless Steel.
B 117 - Method of Salt Spray (Fog) Testing.
B 154 - Method of Mercurous Nitrate Test for Copper and Copper Alloys.
B 193 - Method of Test for Resistivity of Electrical Conductor Materials.
E 6 - Definitions of Terms Relating to Methods of Mechanical Testing.
E 7 - Definitions of Terms Relating to Metallography.
E 8 - Methods of Tension Testing of Metallic Materials.
E 10 - Method of Test for Brinell Hardness of Metallic Materials.
E 18 - Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials.
E 23 - Methods for Notched Bar Impact Testing of Metallic Materials.
E 45 - Recommended Practice for Determining the Inclusion Content of Steel.
E 52 - Industrial Radiographic Terminology for use in Radiographic Inspection of Castings and Weldments.
E 92 - Method of Test for Vickers Hardness of Metallic Materials.
E 94 - Recommended Practice for Radiographic Testing.
E 109 - Method for Dry Powder Magnetic Particle Inspection.
E 112 - Methods for Estimating the Average Grain Size of Metals.
E 113 - Recommended Practice for Ultrasonic Testing by the Resonance Method.
E 114 - Recommended Practice for Ultrasonic Testing by the Reflection Method, using Pulsed Longitudinal Waves Induced by Direct Contact.
E 138 - Method for Wet Magnetic Particle Inspection.
E 140 - Standard Hardness Conversion Tables for Metals (Relationship Between Brinell Hardness, Vickers Hardness, Rockwell Hardness, Rockwell superficial Hardness and Knoop Hardness).
E 142 - Method of Controlling Quality of Radiographic Testing.
E 164 - Method of Ultrasonic Contact Inspection of Weldments.
E 165 - Methods of Liquid Penetrant Inspection.
E 175 - Definitions of Terms Relating to Microscopy.
E 268 - Definitions of Terms Relating to Electromagnetic Testing.
E 269 - Definitions of Terms Relating to Magnetic Particle Inspection.
E 270 - Definitions of Terms Relating to Liquid Penetrant Inspection.
E 273 - Methods for Ultrasonic Inspection for Longitudinal and Spiral Welds of Welded Pipe and Tubing.
E 290 - Semi-Guided Bend Test for Ductility of Metallic Materials.

Aerospace Material Specifications (AMS):

2300A Premium Aircraft Quality Steel Cleanliness - Magnetic Particle Inspection Procedure.
2301C Aircraft Quality Steel Cleanliness - Magnetic Particle Inspection Procedure.

American Welding Society (AWS):

C3.2 Standard Method for Evaluating the Strength of Brazed Joints.

1/ Copies of industry methods are not contained in this standard (see section 6, General Section).

FEDERAL TEST METHOD STANDARD

METALS; TEST METHODS

Table of contents (con.)

FEDERAL TEST METHOD STANDARD

METALS; TEST METHODS

Table of contents