

FED. TEST METHOD STD. NO. 151b  
November 24, 1967  
SUPERSEDING  
Fed. Test Method Std. No. 151a  
May 6, 1959

TEST METHOD STANDARD

METALS; TEST METHODS

This standard was approved by the Commissioner, Federal Supply Service, General Services Administration, for the use of all Federal agencies.

Order for this publication are to be placed with the General Services Administration, acting as an agent for the Superintendent of Documents. Single copies of this standard are available without charge at the GSA Business Service Centers in Boston, New York, Atlanta, Chicago, Kansas City, Mo., Fort Worth, Denver, San Francisco, Los Angeles, and Seattle, Washington. Additional copies may be purchased for 25 cents each from the General Services Administration, Specifications Activity Printed Materials Supply Division, Building 197, Naval Weapons Plants, Washington, D. C. 20407.

FSC 9500

## INFORMATION SHEET

This Federal Test Method Standard is issued in loose-leaf form to permit the insertion or removal of new or revised sections and test methods.

All users of Federal Test Method Standards should keep them up to date by inserting revised or new sections and test methods as issued and removing superseded and canceled pages.

New and revised material and cancellations will be issued under Change Notices which will be numbered consecutively and will bear the date of issuance. Change Notices should be retained and filed in front of the Table of Contents until such time as they are superseded by a reissue of the entire Standard.

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Premium Aircraft Quality Steel Cleanliness Magnetic Particle Inspection Procedure	AMS 2300A	-
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Industry Methods and Definitions Accepted Under This Standard<sup>[1]</sup>

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 Electrodeposited 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 Test of Steel Products.
- B 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 Tent for Copper and Copper Alloys.
- B 193 - Method of Test for Resistivity of Electric 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.
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- 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.

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

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### METALS; TEST METHODS

#### GENERAL SECTIONS

#### 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 treatment of the product, test material can be taken at any appropriate stage in manufacture.

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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 operations may not yield analytical results that correctly represent the 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 lot of material under the following conditions:

- (a) Where the specimen is incorrectly machine.
- (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 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 lot, as necessary, to correct the deficiencies or has removed the nonconforming material.

#### 5. CHANGES

5.1 When a Federal agency considers that a Federal Test Method Standard does not provide for its essential 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 essential 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 AWS methods should be addressed to the American Welding Society, 345 East 47th Street, New York, N. Y. 10017.

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(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 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  
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Review activities:

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Preparing activity:

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Method 442.1  
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## LEAK TESTING

### (PRESSURIZED GAS)

1.1 This method covers procedures for locating leaks in vessels in which leakage is not smaller than 10-<sup>-6</sup> 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 I), pressure less than design pressure of vessel.

3.2 Detection materials (table I), 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	Probe near vessel	Wisp of white smoke

		Sulfur dioxide		
		O.I N hydrochloric acid on swab		
Freon, 50 per-cent 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

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METHOD 443.1  
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## 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, 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 mercury (0.5 p.s.i.a.), if no vacuum is specified.

4.3 Close valve to vacuum, not 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 result 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.

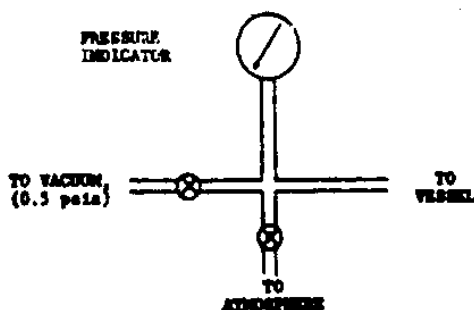


FIGURE 1. Test setup.

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METHOD 513.1  
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## 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.

2.1 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  $\pm$  0.010 inch in diameter, 2.000  $\pm$  0.010 inch square, or 2.97  $\pm$  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 "cure 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 length of electrolytic tinplate may either be taken in the 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:

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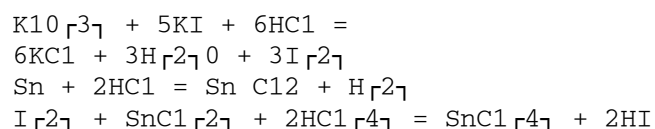
## METHOD 513.1

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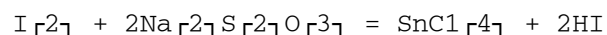
Method	Recommended specimen size
Bendix	2.257-inch diameter disc or 2.000 inches square
X-ray	Full size sheet-measurements made over 4-square-inch area.
Antimony trichloride stripping	2.257-inch-diameter disc or 2.000 inches square.
Electrostripping - constant current-acid electrolyte	2.257-inch-diameter 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 American Iron and Steel Institute 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:



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



This method determines the total tin or the sum of the combine 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 fluorescence 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, 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  $\pm 0.01$  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.

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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 hydrochloric acid. The potential difference developed between the sample and a silver 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 drop. 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. Separately 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 consist 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 or 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 samples 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

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## 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. Seller'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<sub>71</sub> 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 approximately 20 deg. 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:

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Weight of coating, lb./bb. = AB X 17.28

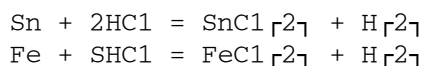
Where:

- 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.

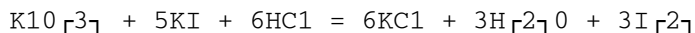
#### 4.1.6.4 Notes.

- (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 than 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 90 percent hydrochloric acid by volume and warming to approximately 70 deg. 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.

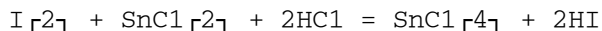
4.1.7 Sellar's method. The determination of tin-coating weight by this method is based upon the oxidation of stannous to stannic tin by the addition of an oxidizing agent. In this method the tinplate sample is dissolved in c.p. hydrochloric acid and goes into solution as iron and tin chlorides. The tin 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. following reactions take place:



Potassium iodate is used its 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 attained 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:



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



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.

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

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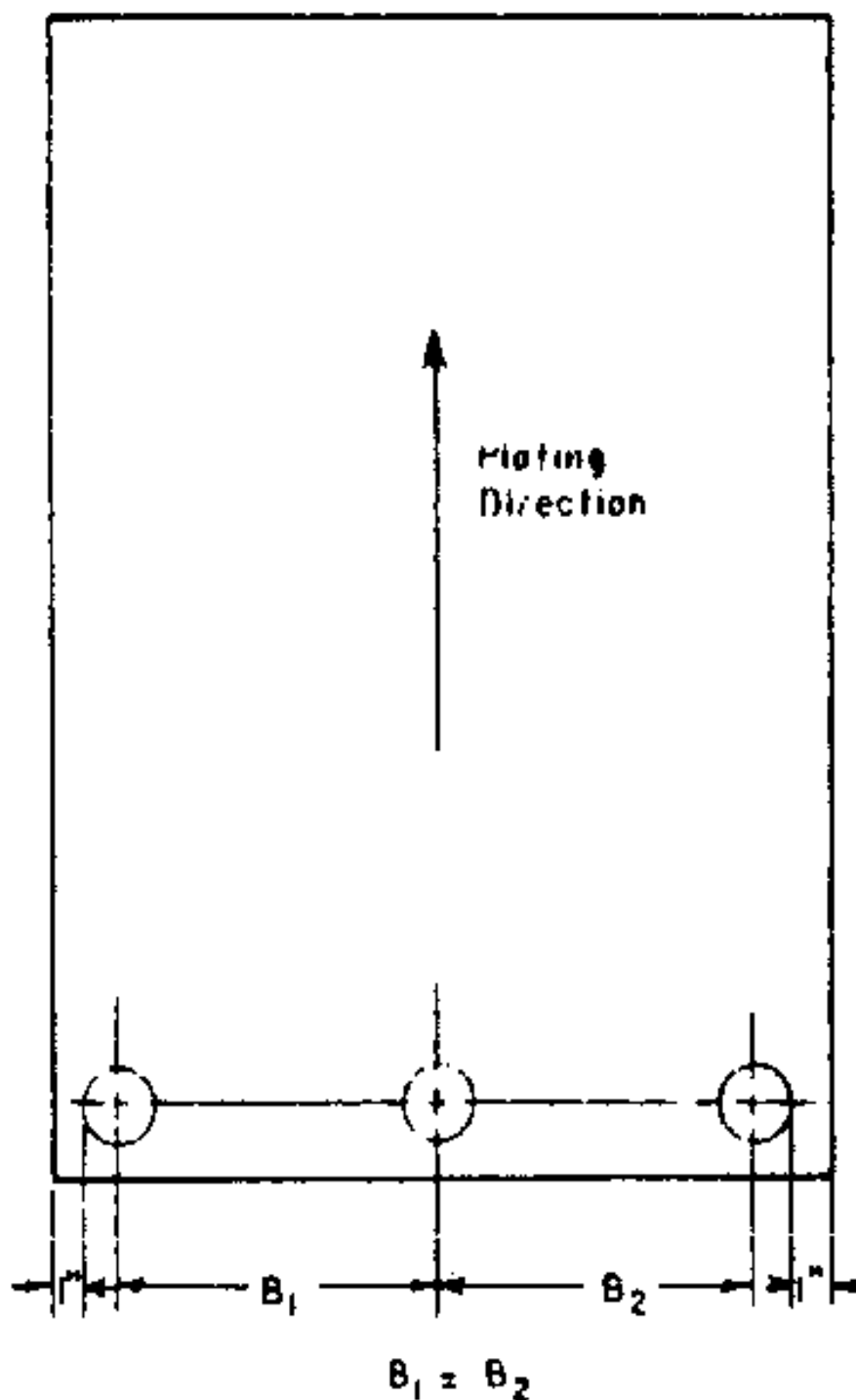


FIGURE 2. Electrolytic tinplate determination of weight of coating.  
Location of sample discs or spot tests.



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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 composition 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 Bass 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 base box or 62,720 sq. in.

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

2.4 Composition 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 petroleum solvent, rinsed in alcohol or boiling methanol, 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 - Hydrochloric 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 terns 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 tome 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.

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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 has 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 Iron the steel sheet.

## 4.1.5.2 Determination of iron dissolved.

## 4.1.5.2.1 Solutions.

- (a) Stannous chloride. Dissolve 60 g. of  $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$  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  $\text{HgCl}_2$  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  $\text{K}_2\text{Cr}_2\text{O}_7$  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 mercuric-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:

$$\text{Iron, grams} = AB$$

Where:

A = ml.  $\text{K}_2\text{Cr}_2\text{O}_7$  solution required to titrate sample and

B = iron equivalent (g. Fe per ml.) of standardized  $\text{K}_2\text{Cr}_2\text{O}_7$  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:

$$\text{Weight of coating} = (A-B) \times 34.57$$

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 = factor to convert grams of coating on a 4-square-inch specimen to pounds per double base box.

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4.2 Composition of coating. Determination of lead and tin content in the terne metal is made by analysis of the pot metal used in the manufacture of the terne plate, procedure F. When pot metal samples are not available, samples of coating stripped from the plate by procedure A (4.1.1) or procedure E (4.1.5 and 4.1.5.1) may be analyzed for lead and tin content. These procedures are given below:

4.2.1 Procedure A, determination of lead and tin in the coating. For testing procedure refer to sections 4 through 6, inclusive, of ASTM A 309. This procedure is applicable for determination of lead and tin in the coating on short terne plate.

4.2.2 Procedure E, chemical analysis of coating. Samples used for analysis of the coating may be taken from any location on the sheet except the extreme edges. The number of discs (4 sq. in.) used for analysis depends on the weight of coating.

Coating weight, pounds per double base box	Number of discs
25-----	1
15-----	2
10-----	3
5-----	7
Less than 5-----	10

The discs should be weighed before and after stripping to get the weight of sample (coating stripped from specimens plus some iron dissolved from the steel sheet). It is not necessary to weigh each specimen separately since the stripping solution from all specimens are combined as one sample. Strip the specimens separately in a 150 ml. beaker containing 100 ml. of concentrated hydrochloric acid. Successive specimens (representing one sample for chemical analysis) may be stripped in the same acid as long as it works efficiently. When all the specimens have been stripped, obtain the weight loss in grams which represents the coating and iron dissolved. Transfer all the stripping acid to a 500-ml. volumetric flask, add sufficient hydrochloric acid to make the total acid 250 ml., and then dilute to the mark with distilled water and mix thoroughly. The solution shall now be analyzed for iron, lead, and tin.

4.2.2.1 Determination of iron. Transfer a 100 ml. aliquot of the solution in the 500-ml. volumetric flask (see 4.2.2) to a 250-ml. beaker. Follow the procedure given in 4.1.5.2 and calculate the grams of iron stripped from the specimens as follows:

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

Where:

A = ml.  $\text{K}_2\text{Cr}_2\text{O}_7$  solution required to titrate the sample and  
B = iron equivalent (g. Fe per ml.) of standardized  $\text{K}_2\text{Cr}_2\text{O}_7$   
solution (for 0.10N. solution 1 ml. = 0.005584 g. Fe).

4.2.2.2 Determination of tin.

4.2.2.2.1 Solutions.

(a) Potassium iodate-potassium iodide solution 0.05 N. Dissolve 15 g.

of potassium iodide and 1.784 g. of potassium iodate in 500 ml. of water that contains 0.5 g. of sodium hydroxide. Transfer the solution to a 1,000 ml. volumetric flask and dilute to the mark with water. Standardize the solution against pure tin.

(b) Starch solution. Transfer 5 g. of soluble starch to a 600-ml. beaker and add enough cold water to make a thick slurry. Pour 400 ml. of boiling water into the slurry and cool to room temperature. Add 50 ml. of a 25 percent solution of potassium iodide and 50 ml. of a 10 percent sodium-hydroxide solution. Store in a rubber-stoppered pyrex bottle.

4.2.2.2.2 Procedure. Transfer a 100-ml. aliquot of the stripping solution (see 4.2), or more if the tin is very low, to a 500-ml. Erlenmeyer flask; Add 50 mg. of 80-mesh antimony and 1 g. of aluminum wire. Connect the flask to the tin reduction apparatus and pass  $\text{CO}_2$  through it. 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. Warm gently to effect solution

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of aluminum wire; heat to boiling and boil for 15 minutes. Cool quickly to room temperature and titrate with 0.05 N potassium iodate-iodide solution (a). Use starch (b) as an indicator. Calculate the percentage of tin in the coating as follows:

$$\text{Tin, percent} = \frac{5AB}{C - D} \times 100$$

Where:

A = ml. of potassium iodate-iodide solution required to titrate the sample,

B = tin equivalent (g. Sn per ml.) of standardized potassium iodate-iodide solution 1 ml. = 0.002968 g. Sn),

C = weight in grams of coating plus iron dissolved from steel sheet (see 4.2.2), and

D = weight in grams of iron dissolved (see 4.2.2.1).

NOTE. The factor 5 is for a one-fifth aliquot and if greater or lesser aliquot are used the correct factor should be substituted for 5.

#### 4.2.2.3 Determination of lead.

##### 4.2.2.3.1 Solutions.

(a) Lead acid. Dissolve 1 g. of lead nitrate in 700 ml. of distilled water; add 100 ml. of concentrated sulfuric acid and dilute to 2000 ml. Let the solution stand overnight and filter through a dense texture filter paper.

(b) 50 percent methyl alcohol. Mix 500 ml. of methyl alcohol with 500 ml. of distilled water.

4.2.2.3.2 Procedure. Transfer a 100-ml. aliquot of the stripping solution (set 4.2.2) to a 400-ml. beaker and add 3 ml. of bromine. Place a raised watchglass on the beaker and evaporate the solution to dryness on a steam bath. Bake the residue in the beaker on a low heat hotplate for 30 minutes. Cool and add 30 ml. of water and 30 ml. of 70 to 72 percent perchloric acid. Place the beaker on a hotplate and evaporate to fumes of perchloric acid. Cool the perchloric acid solution and add 50 ml. of water and 30 ml. of nitric acid. Add paper pulp to the solution, heat to boiling and let stand overnight. Filter through a medium texture filter paper and wash six times with 5 percent nitric acid. Evaporate the filtrate to fumes of perchloric acid. Cool the beaker, add 10 ml. of water to dissolve the salts and then add 100 ml. of lead acid (a) slowly while stirring. Place the beaker on the hotplate and digest near the boiling point for several minutes. Remove the beaker, from the hotplate and let stand for several hours at room temperature. Filter the solution through a weighed Selas crucible that was ignited at 1000 deg. F. Wash the precipitate five times with lead acid (a) and then five times with 50 percent methyl alcohol (b). Dry the crucible and contents in an oven at 110 deg. C. and then ignite in a muffle at 1000 deg. F. for 45 minutes. Cool in a desiccator and weigh. The increase in weight represents lead sulfate. Calculate the percentage of lead in the coating as follows:

$$\text{Lead, percent} = \frac{5(A-B) \times 0.6833}{C-D} \times 100$$

Where:

- A = weight in grams of Sela crucible and ignited precipitate,
- B = weight in grams of Sela crucible,
- C = weight in grams or coating plus iron dissolved from steel sheet (see 4.2.2), and
- D = weight in grams or iron dissolved (see 4.2.2.1).

#### 4.2.3 Procedure F, analysis of pot metal for lead and tin content.

4.2.3.1 Method of sampling. The terne pot metal is sampled by dipping the molten metal from the pot with a ladle that has a 1/32 inch hole drilled in the bottom. Immerse the ladle in the pot metal and hold it there until it has attained the temperature of the metal. Empty the ladle several times to remove any metal that may be adhering to it from the previous test. Then fill the ladle with the pot metal and hold it over a vessel containing water. The thin stream of metal entering the water will form wire-like sections that have a uniform chemical composition.

4.2.3.2 Determination of tin. Transfer 1 g. of the sample (see 4.2.3.1) to a 500-ml. Erlenmeyer flask and add 100 ml. of concentrated hydrochloric acid. Cover the flask with a small watchglass and allow it to stand at room temperature until the sample has dissolved. If the sample has a high antimony content, a black residue of tin-antimony alloy will remain. Add ferric chloride drop by drop until the solution has a slightly yellowish tinge and then heat gently until the tin-antimony alloy has dissolved. Add 100 ml. of water, 50 mg. of 80-mesh antimony and a 7-inch section or 12-gage

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aluminum wire (99.5 percent pure - 7 inches = about 1 gram). Connect the flask to the tin reduction apparatus and pass  $\text{CO}_2$  through it. 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 way be used. Heat gently until the aluminum has dissolved; heat to boiling and boil for 15 minutes. Cool the solution quickly in running water and titrate with 0.05 N potassium iodate-iodide solution (see (a) in 4.2.2.2.1). Use starch as an indicator as an indicator (see (b) in 4.2.2.2.1). Calculate the percentage of tin in pot metal as follows:

$$\text{Tin, percent} = \frac{AB}{C} \times 100$$

Where:

A = ml. of potassium iodate-iodide solution required to titrate the sample,  
B = tin equivalent (g. Sn per ml.) of standardized potassium iodate-iodide solution (for 0.05 N solution 1 ml. = 0.002968 g. Sn), and  
C = weight in grams of sample analyzed.

4.2.3.3 Determination of lead. Transfer 1 g. of the sample (see 4.2.3.1) to 250-ml. beaker, add 30 ml. of water and 15 g. of tartaric acid. Place on a steam bath and when the tartaric acid has dissolved, add 4 ml. of nitric acid while stirring. When the sample has completely dissolved, remove from the steam bath and cool to room temperature. Add 40 ml. of water and then while stirring add 4 ml. of methyl alcohol. Stir the solution well and let stand for one hour or more. Filter through weighed Selas crucible that was heated to 950 deg. F. for 45 minutes before weighing. Wash the precipitate with lead acid (see (a) in 4.2.2.3.1) and finally with 1:1 methyl alcohol (see (b) in 4.2.2.3.1). Heat gradually in a muffle to 950 deg. F. and hold at that temperature for 45 minutes. The alternate drying method of drying the  $\text{PbSO}_4$ , on a hotplate for 5 minutes, then transferring the crucible to a muffle heated to 1000 deg. F. for a period of 5 minutes may be used. Cool in a desiccator and weigh. The increase in weight represents lead sulfate. Calculate the percentage of load in the coating as follows:

$$\text{Lead, percent} = \frac{(A-B) \times 0.6833}{C} \times 100$$

Where:

A = weight in grams of Selas crucible and ignited precipitate,  
B = weight in grams of Sales crucible, and  
C = weight in grams of sample analyzed.

## 5. REPORT OF RESULTS

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

METHOD 514.1

November 24, 1967

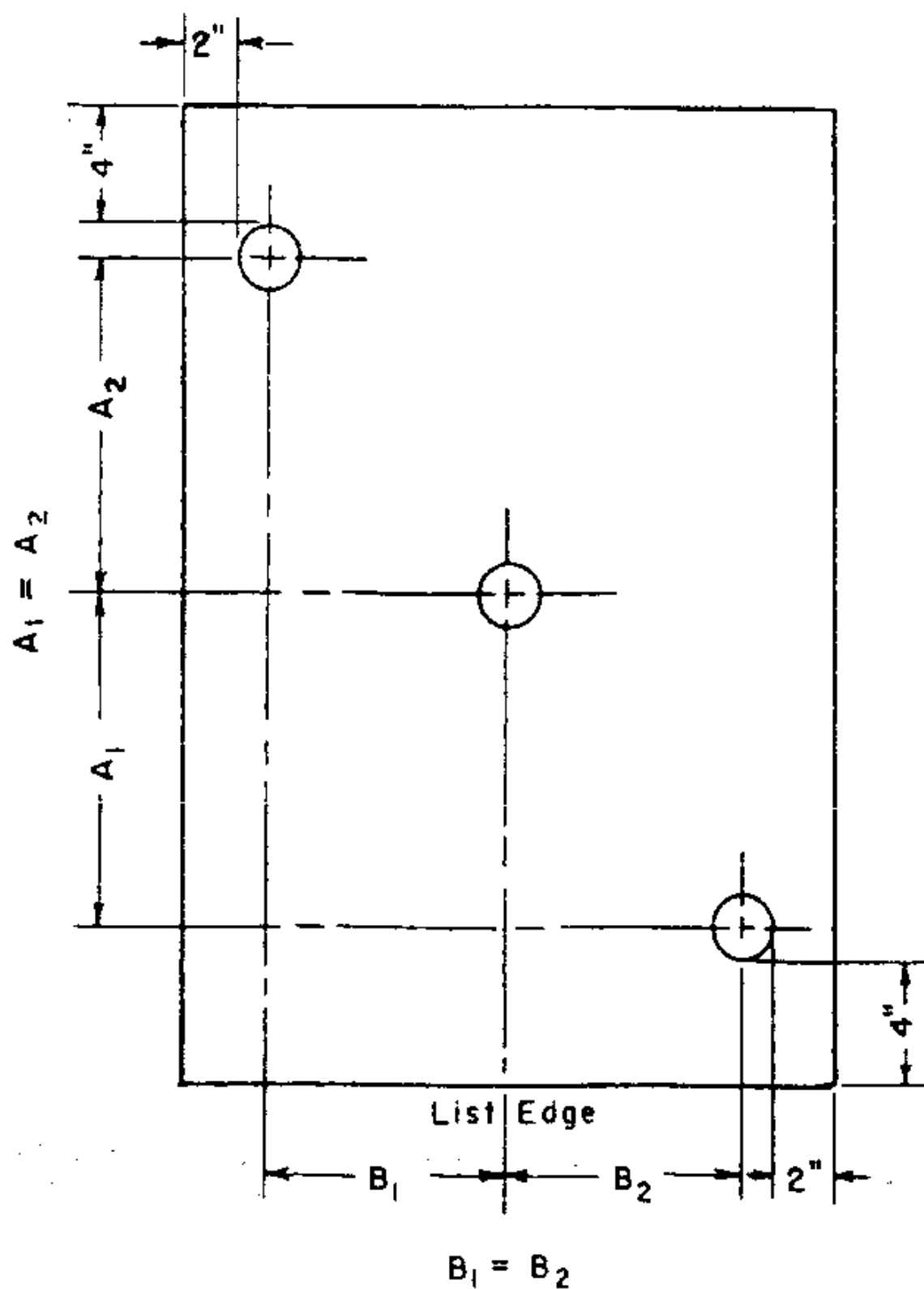


FIGURE 1. Terne plate determination of weight of coating.  
Location of sample discs or spot tests.



FED. TEST METHOD STD. NO. 151b

METHOD 520.1  
November 24, 1967

## ELECTRONIC TEST FOR LOCAL COATING THICKNESS

### 1. SCOPE

1.1 This method covers procedures for the nondestructive measurement of the thickness of metallic and nonmetallic coating on metals, and metallic coatings on nonmetals by means of electronic instruments.

### 2. DESCRIPTION

2.1 The operation of the instrument is based on the reaction of induced eddy currents to the different electrical conductivities of the coating and basis materials to indicate a measurement of the coating thickness. It consists of an instrument which generates an oscillating electrical potential and measuring probe. The oscillating electrical potential induced eddy currents into the surface of the conductive material which are measured with a probe.

### 3. APPARATUS

3.1 Coating thickness shall be determined with an electromagnetic instrument that measures the changes in apparent impedance of the coil inducing the eddy currents into the basis metal. The design of the instrument shall be such that variations in apparent impedance, produced by variations in coil to base metal spacing, can be calibrated to indicate the thickness of coatings.

3.2 Operating procedures for eddy current instruments are not included. The manufacturer's recommendations and specific instructions shall be carefully followed.

3.3 Accuracy. The instrument, its calibration, and its operation shall be such that the coating thickness can be determined within a range of plus or minus 10 percent. Accuracy is dependent upon the quality of the standards used, the type of probe used, and the proficiency of the operator.

3.3.1 The accuracy and reproducibility of thickness measurements are influenced by type of coating, basis material, and thickness, surface roughness of substrate and degree of flatness.

### 4. SPECIMEN

4.1 Various instruments differ in accuracy when measuring near the edges of specimens. There are also differences in the area of flat surface required for an accurate measurement with the various instruments. The manufacturer's instructions are specific for these points and shall be followed.

- (a) Very thin flat specimens, 0.010 inch thick or less, shall be supported by 4 flat heavy-gage plate to prevent distortion.
- (b) Curved surfaces may be measured with special probes or positioning jigs.

### 5. STANDARDS

5.1 Standard thickness samples and corresponding calibration curves are used to calibrate the instrument and determine the thickness or coating on production parts. Standard samples shall be composed of the base material and same coating material as that of the coated parts to be measured.

5.2 Standards shall be prepared specifically for the electronic instrument that is to be used and in accordance with manufacturer's recommendations.

## 6. CALIBRATION OF APPARATUS

6.1 Calibrate the eddy current instruments by first adjusting to zero on a specimen of uncoated basis material of the same alloy, temper, etc. as the specimen to be measured. Then place the probe or search unit on the standard having the same coating and basis material as the specimen to be tested. Depending on the particular instrument, make a calibration curve or adjust the instrument dial to the known thickness of the calibration standard. A typical calibration procedure is described in 8.1.

## 7. PROCEDURE

7.1 The instrument shall be calibrated and operated according to the manufacturer's instructions before any thickness measurements are made. Report the thickness for the coating as the average of at least three measurements. Recalibrate the instrument at frequent intervals to minimize errors caused by instrument drift.

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8. NOTES

8.1 Typical procedure. The following is a typical, procedure for calibrating eddy current instruments:

- (1) Obtain a production part for a standard.
- (2) Remove the coating from a portion of the part.
- (3) Metallographically determine the thickness of the coating.
- (4) After the eddy-current instrument is thoroughly warmed up and ready for operation, place the probe on the basis metal portion of the standard part and set the instrument at zero.
- (5) Place the probe on the coated portion of the standard and adjust the instrument to read the metallographically determined thickness of the coating.
- (6) Repeat steps (4) and (5) until the zero and calibrated readings have stabilized. When the readings have stabilized, the instrument is ready for measuring the thickness of coatings on production run parts.
- (7) Place the probe on a production part at a point geometrically equivalent to that used in calibrating the instrument (on the standard) and read the coating thickness.
- (8) During the course of a day's operation, the instrument should be recalibrated as many times as is necessary to assure no error in measurement caused by instrument drift.

8.2 Observe precautions as follows:

8.2.1 Do not rub or slide probe tip against any surface.

8.2.2 Do not bang probe element tip against surface being measured.

8.2.3 Do not press heavily an probe during measurement. Excessive pressure may cause inaccurate reading. A light consistent pressure is desirable, as is provided by the spring which connects the probe to the guide.

8.2.4 Keep the probe tip clean by wiping often with clean lint-free cloth.

8.2.5 For applications not outlined herein, manufacturer's instructions should be consulted.

8.3 Referee test. In case of disagreement or doubt as to results obtained by the electronic method, or when it is necessary or desirable to employ a referee test, sections 2 and 3 of ASTM A 219 is recommended.

9. REPORT OF RESULTS

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

METHOD 812.1  
November 24, 1967

## SYNTHETIC SEA-WATER SPRAY TEST

### 1. SCOPE

1.1 This method covers procedures for an accelerate-corrosion test in which specimens are exposed at a temperature of 75 deg. F. (23 deg. C.) to a fine mist of synthetic sea water. (For the salt spray test, see ASTM B 117.)

1.2 This method is recommended for testing certain steels which are subject to localized pitting attack.

### 2. APPARATUS

2.1 Stock solution. The stock solution shall consist of the following:

KCl (c.p.)-----	10 g.
KBr (c.p.)-----	45 g.
MgCl <sub>2</sub> ·6H <sub>2</sub> O(c.p.)-----	550 g.
CaCl <sub>2</sub> ·6H <sub>2</sub> O(c.p.)-----	110 g.

Sterile distilled water to make 1 liter.

2.2 Spray solution. The spray solution shall consist of the following:

NaCl (c.p.)-----	23 g.
Na <sub>2</sub> SO <sub>4</sub> ·10H <sub>2</sub> O-----	8 g.
Stock solution-----	20 g.

Distilled water to make 1 liter.

2.3 All parts of the apparatus used in the test which come in contact with the solution shall be made of materials resistant to attack or leaching by the spray or liquid to the extent that there is no detectable contamination of the liquid. No material which gives off vapor that may effect the test shall be used in the construction of the device.

2.4 The apparatus shall be so designed and constructed that the parts or the space in which the specimens are placed shall be subjected an nearly as practicable to equal conditions of fog and spray.

### 3. PREPARATION OF SPECIMENS

3.1 Specimens shall be given a minimum of handling, particularly on the significant surfaces, and shall be prepared for test immediately before exposure. Specimens for determining the corrosion resistance of metals shall be given the required surface finish (after removing any previous finish) not earlier than 4 hours before starting the test. Since all but noble metals resist corrosion by oxide films which form on their surfaces after prolonged exposure to dry air, the time elapsed since surface finishing is a factor to be considered, and uniform conditions established, when comparing the corrosion resistance of metals.

3.2 Unless otherwise specified, uncoated metallic specimens and metallic-coated specimens shall be thoroughly cleaned of oil, dirt, and grease, as necessary, until the surface is free of water break. The cleaning methods shall not include the use of corrosive solvents nor solvents that deposit either corrosive or protective films, nor the use of abrasives other than a paste of pure magnesium oxide. Specimens having an organic coating

shall not be solvent cleaned.

3.3 Unless otherwise specified, cut edges and surfaces not required to be tested and the portions of specimens which come in contact with the support shall be protected with a suitable coating of wax or similar substance impervious to moisture.

#### 4. PROCEDURE

4.1 The temperature within the exposure chamber shall be maintained at 75 deg. +/- 5 deg. F. (23 deg. +/- 3 deg. C.).

4.2 The fog or mist shall be produced by spraying the solution by means of a suitable atomizer operated by compressed air at a constant pressure obtained by passing filtered air of a substantially higher pressure through an automatic pressure regulator. Before entering the aspirator, the air shall be washed and humidified by pass a stream of fine bubbles through about 20 inches of water in a tower suitably arranged to prevent spray being carried by the outgoing air. The tubing between the washing tower and aspirator shall be short and of noncorrosive material. No warming of the wash water is needed if the air pressure is 15 pounds or lose.

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4.3 A baffle shall be placed so as to intercept direct spray from the aspirator and to prevent splashes entering the test chamber, that is, the area provided for the test specimens. The liquid which drains from the baffle should return to the reservoir provided to hold solution for feeding the aspirator, but no liquid that has settled in the test chamber shall be allowed to reach the feed reservoir.

4.4 The feed reservoir shall have ample capacity and shall be filled at the start of the test and at refilling to a volume at least twice the quantity of solution that will be used up by the time of next refilling. solution used in refilling the feed reservoir shall be of the strength specified. The liquid in the reservoir shall not be allowed to become more than one-fifth more concentrated. This may be accomplished by using an efficient atomizer, suitably humidifying the air used for aspiration when necessary, and by keeping a liberal quantity of solution in the feed reservoir or by the frequent changes of the solution.

4.5 Solution shall enter the space provided for the test specimens only as air-borne fog or mist. A thick cloud of floating fog with a minimum of precipitation and produced with a minimum of aspiratory air is desired. This condition is satisfied if objects 4 inches below the glass cover cannot be seen. This may be produced by one aspirator operating with air at 15 pounds per square inch gage pressure with an air jet of 1/64-inch bore for each cubic foot of chamber space. An adequate vent or vents for the air and means for draining off precipitated liquid shall be provided.

4.6 The apparatus shall be provided with a plate glass cover through which the conditions in the test chamber may be observed. This cover shall be set at such an angle (slope one in five suggested) that drops accumulating on its under surface will run off of its end or side and not drop onto the specimens. Solution shall not be allowed to drop or run onto the specimens from supporting rods or cords nor to creep up onto significant surfaces from liquid which collects on the bottom of the apparatus. No surface significant in the test shall be allowed to touch any other object whether similar or not unless such contact is intended as a feature of the test.

4.7 Only inert insoluble, nonleachable and electrically nonconducting materials shall be used for supports. When practicable, specimens shall be supported on posts or pedestals supported from below so that no liquid drains onto or is caused to accumulate on the specimen because of the support.

4.8 Specimens shall be set up so that the surface to be tested shall be sloped at 60 deg. from the horizontal insofar as practicable. The orientation of the tested surface is a major factor. For instance vertical and overhanging surfaces of corrosion-resistant steel are nearly immune to corrosion in the salt spray test except where solution drains down onto them from above. Horizontal surfaces are corroded erratically and indiscriminately sloping surfaces most actively and selectively.

4.9 Specimens shall be shifted in the test chamber from time to time so that each shall be exposed as nearly equally as possible notwithstanding unavoidable inequality in conditions throughout the chamber. In operating the test continuously with introduction and removal of specimens from time to time, newly entered specimens shall be placed at first in the locations nearest the source of spray so as to become wet promptly.

## 5. REPORT OF RESULTS

5.1 The following information shall be recorded, unless otherwise prescribed in the detail specification or drawing for the material or product being tested:

- (a) All reading of temperature within the exposure zone of the chamber.
- (b) Type of specimen and its dimensions, or number or description of product or tested.
- (c) Method of supporting specimen in the salt spray chamber.
- (d) Description of protection used on cut edged and other surfaces not required to be tested.
- (e) Length of exposure period.
- (f) Cause and length of interruptions during test.
- (g) Results of all inspections.

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METHOD 822.1  
November 24, 1967

## INTERGRANULAR-CORROSION TEST FOR ALUMINUM ALLOYS

### 1. SCOPE

1.1 This method covers procedures for determining the susceptibility of aluminum alloys to intergranular corrosion.

### 2. DEFINITION

2.1 Intergranular corrosion. Intergranular corrosion in aluminum alloys is a type of electro-chemical corrosion which progresses preferentially along the grain boundaries of an alloy, usually because the grain boundary regions behave anodically with respect to the grains.

### 3. TEST SOLUTIONS

3.1 The solution for etching prior to immersion in the sodium-chloride-hydrogen-peroxide solution shall be of the following composition:

Nitric acid, concentrated (70 percent)-----	50 ml.
Hydrofluoric acid (48 percent)-----	5 ml.
Distilled-----	945 ml.

3.2 The sodium-chloride-hydrogen-peroxide solution shall be of the following composition:

Sodium chloride-----	57 g.
Hydrogen peroxide, 30 percent (c.p.)-----	10 ml.

3.2.1 Dilute to 1 liter with distilled water. Use at least 30 ml. of the solution for each square inch of exposed specimen surface. In all cages, specimens shall be completely covered with solution. More than one sample of the same alloy, may be corroded in the same container provided that 30 ml. of solution are used for each square inch of specimen surface and provided the specimens do not touch each other.

3.3 The solution for etching prior to microscopic examination shall be of the following composition:

Nitric acid, concentrated (70 percent)-----	2.5 ml.
Hydrochloric acid, concentrated (c.p.)-----	1.5 ml.
Hydrofluoric acid (48 percent) (c.p.)-----	1.0 ml.
Distilled water-----	95.0 ml.

### 4. TEST SPECIMENS

4.1 The test specimens may be of any size and shape convenient for performing the required test.

### 5. PROCEDURE

5.1 In the case of clad alloys, the cladding shall be completely removed from both sides of the sample by filing or other suitable means to avoid cathodic protection of the surface being examined by cladding on adjacent areas.

5.2 Immerse samples in the nitric-acid-hydrofluoric-acid etching solution at 203 deg. F. (95 deg. C.) for one minute to produce a uniform surface

condition.

5.3 Rinse in distilled water.

5.4 Immerse in concentrated nitric acid (70 percent) at room temperature for 1 minute to remove any metallic copper that may have been plated out on the samples.

5.5 Rinse in distilled water and allow to dry.

5.6 Immerse in the sodium-chloride-hydrogen-peroxide solution at 86 deg. + 9 deg. F. (30 deg. +/- 5 deg. C.) for 6 hours. Longer exposure are permissible, but will not generally alter the results obtained in 6 hours. Prepare sodium-chloride-hydrogen-peroxide solution immediately before use.

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5.7 Wash and dry.

5.8 Prepare several small cross-sectional specimens from each sample and examine microscopically at 100X, for the presence of intergranular corrosion.

5.9 If no intergranular attack is evident, etch the specimens in the nitric-acid-hydrochloric-acid solution for 6 to 20 seconds, rinse, dry, and again examine microscopically at 100X for the presence of intergranular corrosion.

6. REPORT OF RESULTS

6.1 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 requested by the procuring agency.

METHOD 823  
November 24, 1967

STRESS-CORROSION TEST FOR ALUMINUM ALLOY PLATE,  
EXTRUSIONS, AND FORGINGS BY ALTERNATE IMMERSION

1. SCOPE

1.1 This method covers the alternate immersion test for determining the stress-corrosion susceptibility of aluminum alloy plate, extrusions, and forgings in the short transverse direction. Material must be at least 0.72 inch thick in the short transverse direction.

2. APPARATUS AND REAGENTS

2.1 Apparatus and reagents used in the alternate-immersion test shall consist of the following:

- (a) (1) Tray for specimens and reservoir for solution, air supply or pump, or (2) tank for solution and mechanism for lowering specimens into and raising them from tank, or (3) tank for solution and ferris wheel for immersing specimens.
- (b) Timer for regulating intervals or immersion for (a) (1) and (a) (2).
- (c) Test cabinet or room with means for controlling air temperatures and relative humidity.
- (d) Number 10-32 aluminum screws and nuts.
- (e) Liquid neoprene or other suitable air dried coating.
- (f) Tube micrometer, one and two inch outside micrometers.
- (g) Acetone, sodium hydroxide, nitric acid.
- (h) Test solution.
- (i) Hydrometer for determining salinity.
- (j) Microscope with range of magnification from 7X to 20X.
- (k) pH meter
- (l) Surface roughness indicator.

2.1.1 Specimen trays and tanks. Trays and tanks shall be made of plastic, hard rubber, glass or a suitably coated aluminum alloy.

2.1.2 Timers. Timers shall be of the "percentage type" with a total cycle of one hour.

2.1.3 Test solution. The test solution shall consist of 35 grams of reagent grade sodium chloride plus distilled water to make one liter. At least 200 ml. of solution shall be used for each square inch of specimen surface.

3. TEST SPECIMEN

3.1 Orientation.

3.1.1 Plate. Specimens shall be machined from plate so that the centerline of the specimen, which is in the area of maximum applied stress, represents the centerline of the plate. The orientation shall be as shown in figure 1.

3.1.2 Extrusions and forgings. The short transverse grain orientation shall be determined by examining the grain pattern of a macroetched extrusion or forging. The C-rings shall then be machined so that they represent the short transverse direction.

3.2 Specimen dimensions and finish. Specimen dimensions and surface

finish shall be as shown in figure 2. The surface shall be a machined finish. The diameter of the rings shall be determined by the material thickness, being the largest size shown in figure 2 possible to machine from the sample.

3.3 Machining. Machining practices shall be such that the metallurgical structure of the samples is not changed.

3.4 Identification of specimens. Specimens are to be identified by stamping aluminum or plastic tags, which are fastened on the stressing screw with a second nut. Marking of the specimen itself with an electric etching tool at the edge of the slot is permissible.

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## 4. TEST PROCEDURES

4.1 Dimensional measurements. Measure the outside diameter of the machined ring at points adjacent to the drilled holes and on both sides of the holes to the nearest 0.001 inch. Average the two measurements. Measure the wall thickness to the nearest 0.001 inch in at least two places along the centerline of the specimen and average the reading.

4.2 Stressing specimens. Stress the specimen by tightening the nut on the screw until the required reduction in diameter is reached. This is determined by micrometer measurements. The required reduction in diameter shall be determined by use of the formula:

$$\Delta OD = OD - [DELTA]$$

$$[DELTA] = \frac{f D^2}{4EtZ}$$

Where:

[DELTA] = change of OD required for desired stress, inches,

f = desired stress, p.s.i.,

OD = outside diameter before stressing, inches,

$OD_{\Delta}$  = diameter that gives required stress, inches,

t = wall thickness, inches,

D = mean diameter, (OD-t), inches,

E = modulus of elasticity, and

Z = function of D/t (0-94 when D/t = 11.5).

4.3 Preparation of specimens for alternate immersion. The specimens shall be cleaned in acetone prior to measuring and again after stressing. To prevent galvanic corrosion, dip the stressed specimens in liquid neoprene so that the screw, nut, and a small portion of adjacent specimen area are covered. While the neoprene is still wet, place the specimen on a plastic strip to which they adhere when the neoprene dries. Allow neoprene to dry for approximately one hour. Degradate the uncoated portion of the specimen by wining with acetone-wet cotton. Begin alternate-immersion test with no delay of 3 hours between stressing and initiation of the stress-corrosion test is permitted.

## 4.4 Details of alternate immersion.

4.4.1 Immersion cycle. The immersion cycle shall be such that the specimens are covered by the salt solution for 10 minutes of each hour and uncovered for 50 minutes.

4.4.2 Methods of cycling. Alternate immersion of the specimens may be accomplished by any of several methods: (1) The solution may be moved by air pressure or by a polyethylene pump from a tank to cover specimens which are stationary in a tray: (2) Specimens may be fixed to a corrosion-resistant rack which is lowered at intervals into a stationary tank containing the solution: or (3) Specimens may be fastened to a ferris-wheel arrangement by which they pass through a stationary tank of solution.

4.4.2.1 To prevent galvanic corrosion, specimens shall not touch one another nor any other bare metal during the alternate-immersion test period.

4.4.2.2. Specimens of alloys containing deliberate additions of copper

shall not be exposed to the same solution used for Al-Mg-Zn Alloys.

4.4.3 Replacement of water lost by evaporation. The salt solution shall be checked each working day with a hydrometer to determine the amount of water lost. The necessary amount of distilled water shall be added to bring the salt concentration to 3.5 percent.

4.4.4 Replacement of solution. Fresh solution shall be prepared weekly. It shall have a pH of 6.4 to 7.4 when prepared, and shall be maintained within that range by the addition of NaCl or HCl.

4.4.5 Temperature and relative humidity. Air temperature of the test cabinet shall be 80 deg. + 2 deg. F. and the relative humidity shall be 45 +/- 6 percent. The solution temperature shall be 75 deg. + 2 deg. F.

4.5 Test duration. The duration of the alternate-immersion test shall be as specified in the material specification. The test shall be run continuously for the time indicated or until failure has occurred with interruptions only for changing solutions or examining specimens.

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4.6 Examination of specimens. At the end of the test, specimens shall be examined while wet at a magnification of 10X for compliance with the requirements of the detail specification. If necessary to aid in examination because of discoloration of the specimens, the following cleaning procedure may be used:

Clean by a quick dip (about 3 seconds) in 10 percent NaOH solution, water rinse, remove salt by a quick dip in 50 percent  $\text{HNO}_3$ , and again water rinse.

## 5. REPORT OF RESULTS

5.1 The report shall include the following:

- (a) Producer and lot number.
- (b) Specification and contract number.
- (c) "Stress-corrosion test passed" or "stress-corrosion test failed" in accordance with results of the test.



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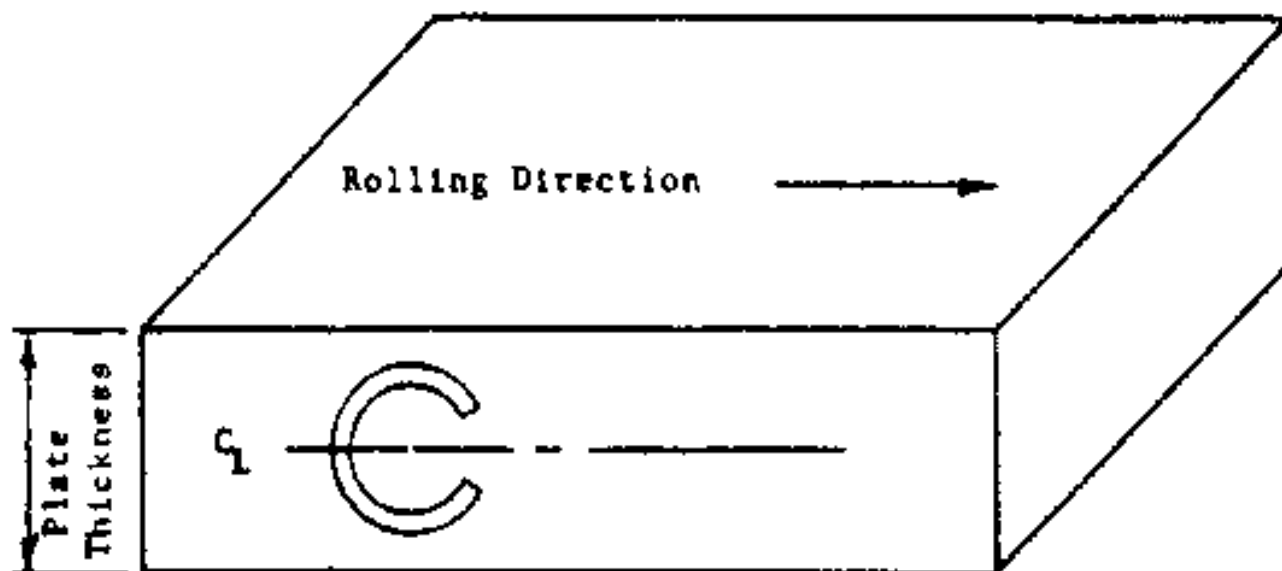
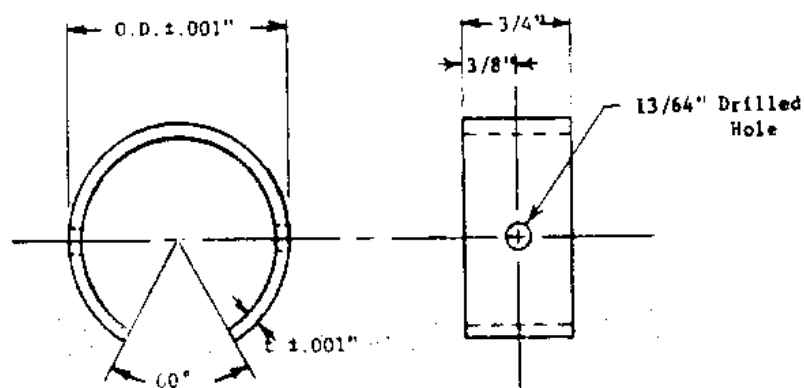


FIGURE 1. Orientation of C-ring in relation to plate.



O.D., in.	t, in.
0.700	0.056
0.750	0.060
1.250	0.100

63 —————  
Outside Machined Finish - 32

FIGURE 2. C-ring specimens.

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METHOD 111.2  
November 24, 1967

## CHEMICAL ANALYSIS

### 1. SCOPE

1.1 This method covers the sampling and analytical procedure applicable to the chemical analysis of metals. This method does not cover chemical analysis by emission methods.

### 2. DEFINITIONS

2.1 Methods of chemical analysis. Methods of chemical analysis are those in which the elements present are determined by use of reagents in solution, by combustion methods, or by other nonemission methods.

2.2 Sample. A sample is a quantity of metal, selected and removed from the material for inspection, any required fraction of which can be used for chemical analysis. A sample may include metal removed at the time of pouring.

### 3. APPARATUS AND MATERIALS

3.1 The apparatus and materials used shall be suitable for the test to be made.

### 4. SAMPLES

4.1 Selecting of samples. Samples shall be so selected as to be representative of the metal under inspection. It shall be determined that the method of selecting the sample is satisfactory for the particular metal without causing contamination and will produce a representative sample. Sample metal from any piece shall be such that it represents as nearly as possible the metal of the entire piece, including any nonhomogeneity or segregation that may be present. Samples from castings or wrought products shall consist of an appropriate piece or metal, or of drillings, millings, or clipping taken in accordance with the applicable test requirements. A milled or drilled sample representing a cross section of the material shall be taken whenever practicable. In cases where cross-section sampling is not feasible, drilled samples shall be taken from a portion midway between the outside and the center of the material. If it is known that certain parts of the material are not representative and will not be used in end items such as risers, runners, or other extraneous sections in the case of castings, samples shall not be selected from these parts. Samples may be obtained from mechanical test specimens. Procedures for obtaining check analysis samples are shown in figure 1 and in table I.

4.1.1 In addition to such analyses as may be required by the contractor, the Government may also require analyses. For this purpose, samples as required to determine conformance to the specified ladle analysis or check analysis made available to the Government on request. Each sample shall contain sufficient material for five complete Government analysis. If requested by the contractor prior to the procuring of the Government sample, a part of the Government test material shall be made available to the contractor for his check analysis.

4.2 Preparation of samples. The surface of the material from which sample metal is to be taken shall be cleaned by appropriate means to prevent contamination of the prepared sample. Surface metal be discarded if it is contaminated in any way as with corrosion products, oil, or dirt. When

practicable, drilling, milling, sawing, and other machining operations shall be performed without the use of water, oil, or other lubricants. Cutting speeds and pressures in all machining operations shall be such that no burning takes place to cause alteration of the chemical composition of the test metal. Sample metal shall be free of scale, slag, contaminated surface metal, grease, oil, dirt, paper, or other foreign substances. Nonmagnetic samples may be cleaned with a magnet. Hard metals, such as ferro-alloys, shall be crushed to suitable size in apparatus designed to withstand severe abrasive forces without contaminating the test material. When it is impossible to obtain an oil-free sample, the sample may be cleaned with a volatile solvent cleaner such as ethyl ether or acetone followed by a rinse with ethanol.

## 5. PROCEDURE

5.1 Unless a particular method of analysis is specified, any applicable ASTM method shall be acceptable. Other methods shall also be acceptable if the accuracy of the method is demonstrated by comparing its results with a cooperatively analyzed standard sample of a similar metal.

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6. NOTES

6.1 In special cases, when sampling difficulties may be encountered, suitable sampling procedures will be specified by the procuring agency.

7. REPORT OF RESULTS

7.1 The results of chemical analysis shall be determined to the number of decimal places shown in the chemical requirements specified for the material.

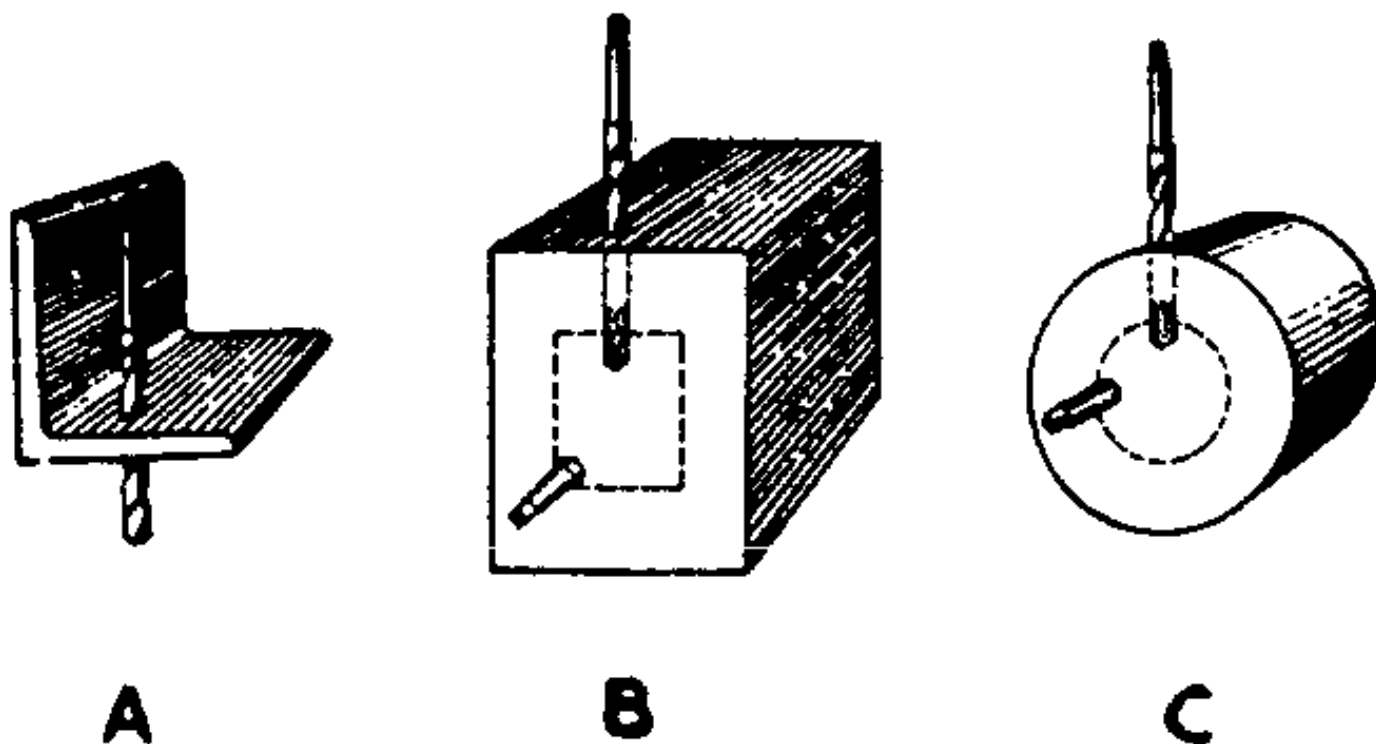


FIGURE 1 Drill locations for obtaining check analysis samples

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TABLE I. Procedures for obtaining check analysis samples

Form	Size characteristics	Cross sectional area sq. in.	Drill diameter, in. (approx)	Sampling instructions <sup>a</sup>
Bars, rounds, squares, hexagons	Small sections	Not over 0.75	---	Chips taken by milling or machining the full cross section of the piece. <sup>b</sup>
Bar-sized shapes; light flat bars	Width of cross-section greatly exceeds thickness	All	1/2	Chips taken by milling or Machining the entire cross section, or by drilling through the piece at a point midway between the outside and center as shown at A, figure 1. <sup>b</sup>
Blooms, billets, slabs, rounds, squares, shapes	---	Over 0.75 Up to 16.0	1/2	Chips taken at any point midway between the outside and center of the piece by drilling parallel to the axis. If impracticable, the piece may be drilled on the side as shown at B and C, figure 1, provided chips are not taken until they represent the portion midway between the outside and the center.
	Large sections	Over 16.0	1	
Bored forgings	---	Up to 16.0		Samples from bored forgings shall be taken midway between the inner and outer surface of the wall
		Over 16.0		
Plates	Thickness up to 2 inches	Up to 16.0	1/2	Chips taken by drilling through the thickness of the plate. <sup>b</sup>
	Thickness over 2 inches	Up to 16.0	1	Chips taken by drilling the edge of the plate at a point midway between the rolled surface and the mid-thickness. <sup>b</sup>
Shapes	---	Up to 16.0	1/2	Chips taken by drilling through the thickness of the piece from the same relative position or location as a tension

				test. <sup>LbJ</sup>
Sheet, strip	Not of the full size rolled (cut from large sheets)	Up to 16.0	1/2	Chips taken by milling or drilling entirely through the piece in a sufficient number of places so that the chips are representative of the entire sheet or strip. <sup>LbJ</sup> Sheet or strip may be folded both ways to facilitate sampling.
		Over 16.0	1	
	Rolled longitudinally	Up to 16.0	1/2	Specimen for sampling cut 2 inches in width across full width as rolled. Specimen cleaned and then folded once or more by bringing ends together and closing bends. Chips taken in middle of length by milling inside sheared edges or drilling entirely through from the flat surface. (Milling preferred.) Several light-gage pieces may be stacked together for folding simultaneously.
		Over 16.0	1	

<sup>LaJ</sup> Each sample shall consist of not less than 2 ounces of drillings, unless otherwise specified. Drillings or chips shall be taken without the application of water, oil, or other lubricant, and all chips or drillings shall be free from scale, grease, dirt or other foreign substances. Chips or drills shall not be overheated during cutting. Chips shall be well mixed and shall pass a No. 10 (2,000 microns) sieve and shall be retained on a No. 30 (590 microns) sieve.

<sup>LbJ</sup> When piece is subject to tension tests, drillings for check analysis may be taken from a tension test specimen or from the piece at the location of the tensile specimen.

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TABLE I. Procedures for obtaining check analysis samples--continued

Form	Size characteristics	Cross sectional area sq. in.	Drill diameter, in. (approx)	Sampling instructions <sup>La</sup>
Shoot, strip	Rolled transversely; sheet over 0.036 inch and all strip	Up to 16.0	1/2	Specimen shall be cut from side of piece half-way between the middle and as rolled 2 inches in 18 inches in length. Remainder of all of procedures same as for sheet and strip rolled longitudinally.
		Over 16.0	1	
Sheet	Rolled transversely; 0.036 inch or less	Up to 16.0	1/2	Specimen shall be cut from full length of sheet as rolled. Remainder of procedure same as for sheet and strip rolled longitudinally.
		Over 16.0	1	
Tubular products		Up to 16.0	1/2	Drillings taken from several points of each tube sampled as practicable. <sup>Lb</sup> Chips taken by milling or machining full cross section.
		Over 16.0	1	
Wire	---	---	---	Chips taken by milling or machining the full cross section of the wire.

<sup>La</sup> Each sample shall consist of not less than 2 ounces of drillings, unless otherwise specified. Drillings or chips shall be taken without the application of water, oil, or other lubricant, and all chips or drillings shall be free from scale, grease, dirt or other foreign substances. Chips or drillings shall not be overheated during cutting. Chips shall be well mixed and shall pass a No. 10 (2,000 micron) sieve and shall be retained on a No. 30 (590 microns) sieve.

<sup>Lb</sup> When piece is subject to tension tests, drillings for check analysis may be taken from a tension test specimen or from the piece at the location of the tensile specimen.



METHOD 112.2  
November 24, 1967

## SPECTROCHEMICAL ANALYSIS

### 1. SCOPE

1.1 Spectrochemical analysis includes all methods in which measurements of electromagnetic radiation produced or induced in the sample are employed to determine composition.

### 2. DEFINITIONS

2.1 Emission spectrochemical analysis. Emission spectrochemical analysis is the technique by which the elements present are determined by observation of spectra from test specimens excited by arcs, sparks, flames, or X-rays.

2.2 Sample. The quantity of metal, selected and removed from the material under inspection, any required fraction of which can be used for spectrochemical analysis. A sample may include metal removed at the time of pouring.

2.3 Test specimen. The portion of a sample that is used in making spectrochemical analysis.

2.4 Point-to-plane technique. This technique of analysis utilizes an electrode system consisting of a test specimen with a freshly prepared flat surface and a counter electrode of some pure conducting material such as graphite.

2.5 Rod or pin technique. This technique of analysis utilizes two electrodes one or both of which may be a rod of the metal to be analyzed.

2.6 X-ray fluorescence techniques. In these methods of analysis specimens are excited to fluorescence by X-rays and the fluorescent X-ray spectra are analyzed to determine concentrations of elements present.

2.7 Pellet technique. This technique of analysis utilizes two electrodes, one or both of which consists of briquetted sample chips, drillings, milling, turnings, or granules.

2.8 Solution technique. This technique is based on the excitation of the test specimen in solution or its dried salts.

### 3. APPARATUS AND MATERIALS

3.1 The apparatus and materials used shall be suitable for the test to be made.

### 4. SAMPLES AND TEST SPECIMENS

4.1 Selection of samples. Samples shall be so selected as to be representative of the entire quantity of metal under inspection. Samples may be taken from molten metal. If it is known that certain parts of the material are not representative and will not be used in the end items, such as risers, runners, or other extraneous sections in the case of castings, samples shall not be taken from these parts. Samples may be obtained from mechanical test specimens.

4.1.1 In addition to such analyses as may be required by the contractor the Government may also require analyses. For this purpose, samples as

required to determine conformance to the specified ladle analysis or check analysis of the detailed product specification shall be made available to the on request. Each sample shall contain sufficient material for five complete Government analysis.

4.2 Preparation of samples and test specimens. The surface of material from which sample metal is to be taken shall be cleaned by appropriate means to prevent contamination of the prepared sample. Surface metal shall be discarded if it is contaminated in any way, as with corrosion products, oil, or dirt. Cutting speeds and pressures in all machining operations shall be such that no burning takes place to cause alteration of the chemical composition of the test metal. Sample metal shall be free of scale, slag, surface metal, grease, oil, dirt, paper, or other foreign substances. Nonmagnetic samples may be cleaned with a magnet. Hard metals, such as ferro-alloys, shall be crushed to suitable size in apparatus designed to withstand severe abrasive forces without contaminating the test material. When it is impossible to obtain an oil-free sample, the sample may be cleaned with a volatile solvent cleaner such as ethyl ether or acetone followed by a rinse with ethnl.

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## METHOD 112.2

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4.2.1 Test specimens for point-to-plane technique. Test specimen shall consist of any material or item (including test specimens) which is of sufficient thickness to prevent overheating during excitation and upon which the required flat surface can be prepared. A smooth clean, flat surface of sufficient area shall be prepared on the specimen by conventional shop methods. The prepared surface shall be approximately equivalent to an intermediate ground surface or to a fine machined surface corresponding to a value of 63 micro-inches.

4.2.2 Test specimens for rod or pan technique. Test rod shall be cast, machined from sample taken from finished or semifinished material, or formed by drawing molten metal into glass tubing. Specimen shall be sound and of appropriate dimensions suitable to correct applications and usage of the method and equipment.

4.2.3 Test specimens for X-ray techniques. Test specimens shall be prepared in accordance with correct applications and usages of the methods and equipment.

4.2.4 Test specimens for pellet technique. Test specimens shall be briquetted from material representative of the test piece.

## 5. PROCEDURES

5.1 Unless a particular method analysis is specified, any applicable ASTM method shall be acceptable. Other methods shall also be acceptable if the accuracy of the method is demonstrated by comparing the results with a cooperatively analyzed standard sample of a similar metal.

## 6. NOTES

6.1 In special cases where sampling difficulties may be encountered, suitable sampling procedures will be specified by the procuring agency.

## 7. REPORT OF RESULTS

7.1 The results of spectrochemical analysis shall be determined to the number of decimal places shown in the chemical requirements specified for the material.

METHOD 441.1  
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## LEAK TESTING

### (HELIUM MASS SPECTROMETER)

#### 1. SCOPE

1.1 This method covers procedures for locating leaks and determining the rate of overall leakage in vessels in which leakage is no smaller than  $10^{-9}$  cc. per second. It consists of applying helium to the pressure side of an evacuated vessel (or a vessel in an evacuated enclosure), and checking for helium seeping into the evacuated enclosure by using a helium mass spectrometer.

#### 2. APPARATUS

2.1 Leak detector, helium mass spectrometer, sensitivity  $10^{-9}$  cc. of helium per second or better ( $10^{-6}$  cc. per second or better when used with sampling probe).

2.2 Accessories for leak detector, as specified by the required):

- (a) Calibrated leak (for calibrating leak detector).
- (b) Helium jet or sampling probe (for probe test).

2.3 Auxiliary vacuum source, 10 microns or lower.

2.4 Vessel enclosure. (See figure 1.)

#### 3. MATERIALS

3.1 Helium, water-pumped, dry (dewpoint 40 deg. F. maximum), 15 p.s.i.a. minimum.

3.2 Sealing material, easily able, such as rubber corks, screwed fittings, blind flanges with O-rings, or other acceptable vacuum seals.

#### 4. PROCEDURE

4.1 Prepare apparatus for operation in accordance with the valve to leak detector.

4.2 Prepare vessel for testing as follows:

- (a) Test vessel to locate more easily detectable leaks, using the method described in method 442. Seal all visible leaks.
- (b) Clean vessel (inside and out) to remove all oil, grease, soap solution, water, and other materials that might interfere with the test.

4.3 Determine the rate of overall leakage of the vessel as follows:

- (a) Make a hood test setup (fig. 1), make sure that the calibrated leak is as far as practical from the leak detector. Seal all unused openings in vessel and enclosure.
- (b) Subject the vessel to a vacuum as specified in the detail specification, and apply helium at atmospheric pressure to other side of the vessel wall (see fig. 1). Shut off auxiliary vacuum source.

- (c) With calibrated leak open, open the leak detector inlet valve, note the time, and allow the helium concentration indicator on the leak detector to reach a constant reading.
- (d) When the concentration becomes constant, record the reading (measured leak rate) and the elapsed (equilibrium time).
- (e) Seal the calibrated leak, and allow the equilibrium time to elapse. Note the measured leak rate.
- (f) Compute actual total leakage as follows:

Actual leakage of vessel B = A - C.

A = Measured leak rate of vessel with calibrated leak open (4.3(d)).

B = Measured leak rate of vessel alone (to be determined).

C = Known leak rate of calibrated leak.

FED. TEST METHOD STD. NO. 151b

## METHOD 441.1

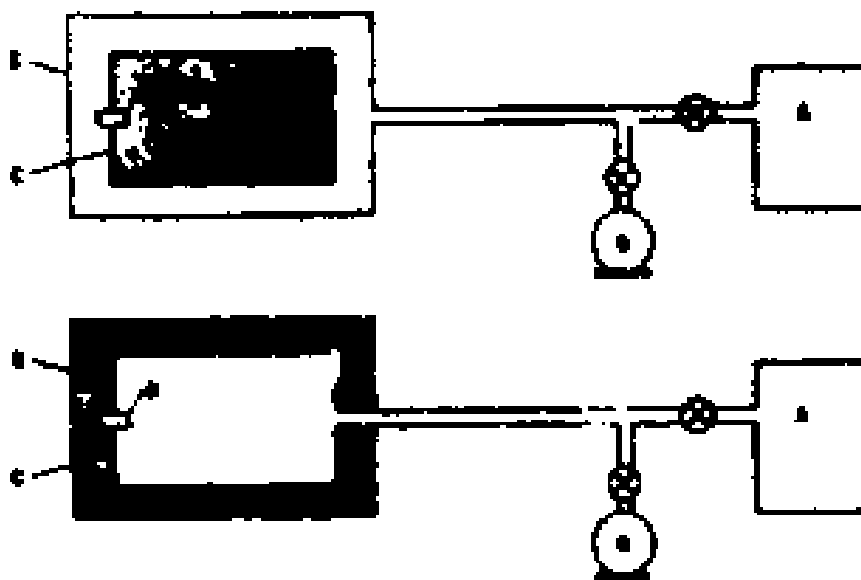
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## 4.4 Locate leaks (if required by the specification) as follows:

- (a) Make a probe test setup (fig. 2), and seal all unused openings in vessel. Test shall be conducted in a draft-free environment.
- (b) Evacuate vessel to a vacuum as specified in the detail specification or charge it with helium above atmospheric pressure.
- (c) Probe outside of vessel (using helium jet on evacuated vessel, or sampling probe on helium-charged vessel), while observing leak detector for the greatest increase in helium-charged vessel), while observing leak detector for the greatest increase in measured leak rate. Sequence of checking for leaks should be from top to bottom.
- (d) Mark and seal all leaks as they are located. Retest after repair or resealing.

## 5. METHOD 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.



- |                             |                         |
|-----------------------------|-------------------------|
| A - LEAK DETECTOR           | D - CALIBRATED LEAK     |
| B - AUXILIARY VACUUM SOURCE | E - HOOD (ENCLOSURE)    |
| C - VESSEL UNDER TEST       | - HELIUM (15 PSIA MIN.) |

FIGURE 1. Hood test setups.

FED. TEST METHOD STD. NO. 151b

CHANGE NOTICES ARE NOT  
CUMULATIVE AND SHALL BE  
RETAINED UNTIL SUCH TIME  
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:  
  
" <sup>2</sup> 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.

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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.

#### Under Table of Contents

1. Change "Microscopic Test for Local Coating Thickness ASTM A 219" to "Standard Method for Measuring Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section ASTM B 487."
2. Change "Magnetic Test for Local Coating Thickness ASTM A 219" to "Standard Method for Measurement of Coating Thicknesses by the Magnetic Method & Nonmagnetic Coatings on Magnetic Basis Metals ASTM B 499."
3. Change "Chemical Dropping Test for Local Coating Thickness ASTM A 219" to "Standard Method for Measuring the Thickness of Metallic-Coatings by the Coulometric Method ASTM B 504."

#### Under Industry Methods and Definitions Accented Under This Standard

1. Delete "ASTM A 219 Methods of Test for Local Thickness of Electrodeposited Coatings."
2. Add "ASTM B 487 Standard Method for Measuring Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section."
3. Add "ASTM B 499 Standard Method for Measurement of Coating Thicknesses by the Magnetic Method & Non-Magnetic Coatings on Magnetic Basis Metals."
4. Add "ASTM B 504 Standard Method for Measuring the Thickness of Metallic Coatings by the Coulometric Method."

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