

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 2  
October 7, 1971

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 Accepted Under This Standard

1. Delete "ASTM A 219 Methods of Test for Local Thickness of Electro-deposited 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 & Nonmagnetic Coatings on Magnetic Basis Metals."
4. Add "ASTM B 504 Standard Method for Measuring the Thickness of Metallic Coatings by the Coulometric Method."

FSC 9500

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

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November 24, 1967

SUPERSEDING

Fed. Test Method Std. No. 151a

May 6, 1959

FEDERAL 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.

#### 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.

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 electrochemical 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 water-----	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 cases, 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° F. (95° 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° ± 9° F. (30° ± 5° C.) for 6 hours. Longer exposures 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.

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## 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 of 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 micrometers, 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 SPECIMENS

#### 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 best 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:

$$OD_r = OD - \Delta$$

$$\Delta = \frac{r D^2}{4EtZ}$$

Where:

- $\Delta$  = change of OD required for desired stress, inches,
- $r$  = desired stress, p.s.i.,
- $OD$  = outside diameter before stressing, inches,
- $OD_r$  = 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 specimens on a plastic strip to which they adhere when the neoprene dries. Allow neoprene to dry for approximately one hour. Degrease the uncoated portion of the specimen by wiping with acetone-wet cotton. Begin alternate-immersion test with no further delay. A maximum 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  $80^\circ \pm 2^\circ$  F. and the relative humidity shall be  $45 \pm 6$  percent. The solution temperature shall be  $75^\circ \pm 2^\circ$  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 immediately 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 smut by a quick dip in 50 percent HNO<sub>3</sub>, 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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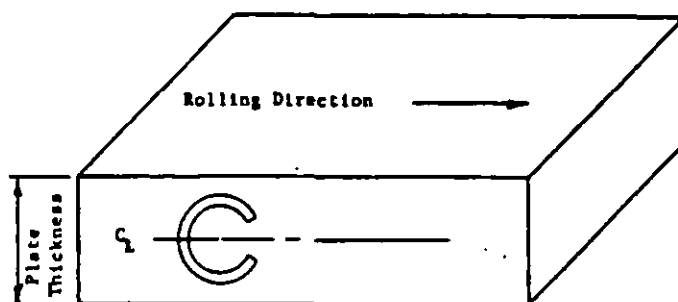
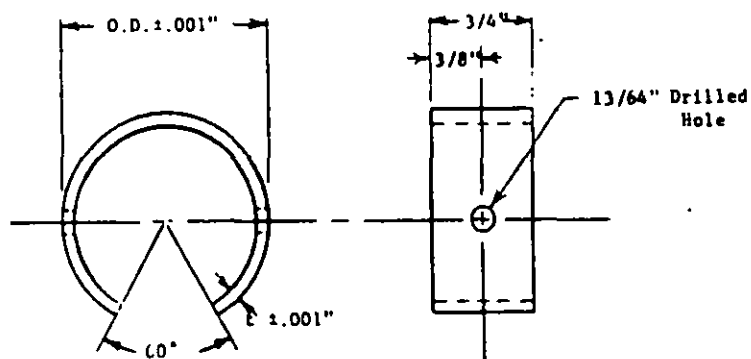


FIGURE 1. Orientation of C-ring in relation to plate.



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.

## 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 of metal, or of drillings, millings, or clippings 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 1.

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 analyses. 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 shall 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 ferroalloys, 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.

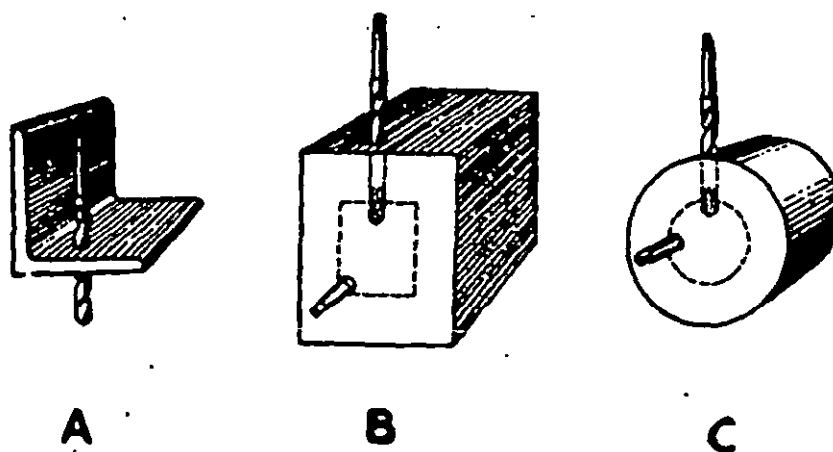


FIGURE 1. Drill locations for obtaining check analysis samples.

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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	1/2	Samples from bored forgings shall be taken midway between the inner and outer surface of the wall.
		Over 16.0	1	
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>
		Over 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>
	Thickness over 2 inches	Up to 16.0	1/2	
		Over 16.0	1	
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>b</sup>
		Over 16.0	1	
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>b</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.
		Over 16.0	1	Specimen cleaned and then folded once or more by bringing ends together and closing bend. 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.

- <sup>a</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 microns) sieve and shall be retained on a No. 30 (590 microns) sieve.
- <sup>b</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>a</sup>
Sheet, 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 halfway between the middle and end as rolled 2 inches in width and 18 inches in length. Remainder of procedure 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>b</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>a</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 microns) sieve and shall be retained on a No. 30 (590 microns) sieve.
- <sup>b</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 .-  
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## 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 item, 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 analyses.

4.2 Preparation of samples and test specimens. 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 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 ethanol.

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4.2.1 Test specimens for point-to-plane technique. Test specimens shall consist of any material or item (including cast 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 pin technique. Test rods 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  
November 24, 1967

## 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 manufacturer of the leak detector (as 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  $40^{\circ}$  F. maximum), 15 p.s.i.a. minimum.

3.2 Sealing material, easily removable, 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 manufacturer's instructions; close inlet 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), making 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 same 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.



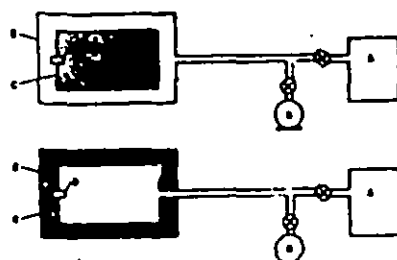
METHOD 441.1  
November 24, 1967

#### 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 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.

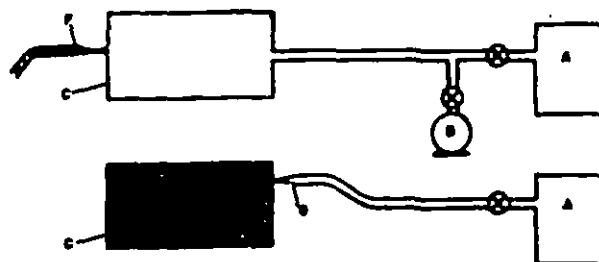
#### 5. METHOD OF RESULTS

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.



- |                             |                         |
|-----------------------------|-------------------------|
| A - LEAK DETECTOR           | F - HELIUM JET          |
| B - AUXILIARY VACUUM SOURCE | G - SAMPLING PROBE      |
| C - VESSEL UNDER TEST       | - HELIUM (15 PSIA MIN.) |

FIGURE 2. Probe test setups.

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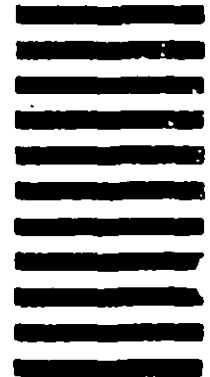
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		<input type="checkbox"/> OTHER (Specify): _____	
5. PROBLEM AREAS			
a. Paragraph Number and Wording:			
b. Recommended Wording:			
c. Reason/Rationale for Recommendation:			
6. REMARKS			
7a. NAME OF SUBMITTER (Last, First, MI) - Optional		b. WORK TELEPHONE NUMBER (Include Area Code) - Optional	
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