

APPENDIX OF SELECTED ASTM STANDARDS

This appendix contains the ASTM standards most often referred to in this special technical publication. These voluntary consensus standards were developed under the direct jurisdiction of ASTM Committee A-1 on Steel, Stainless Steel, and Related Alloys and Committee G-1 on Corrosion of Metals. Specific subcommittee jurisdiction is indicated in the first footnote to each standard. Membership in the Society is open to all concerned with the fields in which ASTM is active.

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The standards included in this appendix were the latest editions available at the time of publication. Because ASTM standards are subject to periodic review and revision, those who use them are cautioned to use the latest revision. A new edition of the ASTM Book of Standards is issued annually and individual standards are available as separate copies. All of the standards in this appendix are currently published in the *Annual Book of ASTM Standards*, Section 03 on Metals Test Methods and Analytical Procedures, Volume 03.02 on Metal Corrosion, Erosion, and Wear.

Precautionary Caveat

In January 1983, the Board of Directors approved the inclusion of the following precautionary caveat in ASTM standards: This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses the standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.



Standard Practices for DETECTING SUSCEPTIBILITY TO INTERGRANULAR ATTACK IN AUSTENITIC STAINLESS STEELS¹

This standard is issued under the fixed designation A 262; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

These practices have been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 These practices cover the following five tests:

1.1.1 *Practice A*—Oxalic Acid Etch Test for Classification of Etch Structures of Austenitic Stainless Steels (Sections 3 to 7, inclusive),

1.1.2 *Practice B*—Ferric Sulfate-Sulfuric Acid Test for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels (Sections 8 to 14, inclusive),

1.1.3 *Practice C*—Nitric Acid Test for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels (Sections 15 to 21, inclusive),

1.1.4 *Practice D*—Nitric-Hydrofluoric Acid Test for Detecting Susceptibility to Intergranular Attack in Molybdenum-Bearing Austenitic Stainless Steels (Sections 22 to 28, inclusive), and

1.1.5 *Practice E*—Copper-Copper Sulfate-Sulfuric Acid Test for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels (Sections 29 to 38, inclusive).

1.2 The following factors govern the application of these practices:

1.2.1 Susceptibility to intergranular attack associated with the precipitation of chromium carbides is readily detected in all five tests.

1.2.2 Sigma phase in wrought chromium-nickel-molybdenum steels, which may or may not be visible in the microstructure, can result in high corrosion rates only in nitric acid.

1.2.3 Sigma phase in titanium or columbium stabilized alloys, which may or may not be visible in the microstructure, can result in high corrosion rates in both the nitric acid and

ferric sulfate-sulfuric acid solutions.

1.3 The oxalic acid etch test is a rapid method of identifying, by simple etching, those specimens of certain stainless steel grades which are essentially free of susceptibility to intergranular attack associated with chromium carbide precipitates. These specimens will have low corrosion rates in certain corrosion tests and therefore can be eliminated (screened) from testing as “acceptable.”

1.4 The ferric sulfate-sulfuric acid test, the nitric acid test, and the nitric-hydrofluoric acid test are based on weight loss determinations and, thus, provide a quantitative measure of the relative performance of specimens evaluated. In contrast, the copper-copper sulfate-sulfuric acid test is based on visual examination of bend specimens and, therefore, classifies the specimens only as acceptable or non-acceptable.

1.5 In most cases either the 24-h copper-copper sulfate-sulfuric acid test or the 120-h ferric sulfate-sulfuric acid test, combined with the oxalic acid etch test, will provide the required information in the shortest time. All stainless grades listed in the accompanying table may be evaluated in these combinations of screening and corrosion tests, except those specimens of molybdenum-bearing grades (for

¹ These practices are under the jurisdiction of ASTM Committee A-1 on Steel, Stainless Steel and Related Alloys and are the direct responsibility of Subcommittee A01.14 on Methods of Corrosion Testing.

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example 316, 316L, 317, and 317L), which represent steel intended for use in nitric acid environments.

1.6 For AISI Grades 316, 316L, 317, and 317L only, the nitric-hydrofluoric acid test may be used to provide test results in 4 h.

1.7 The 240-h nitric acid test must be applied to stabilized and molybdenum-bearing grades intended for service in nitric acid and to all stainless steel grades which might be subject to end grain corrosion in nitric acid service.

1.8 Only those stainless steel grades are listed in Table 1 for which data on the application of the oxalic acid etch test and on their performance in various quantitative evaluation tests are available.

1.9 Extensive test results on various types of stainless steels evaluated by these practices have been published in Ref (10).²

1.10 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific precautionary statements are given in 5.6, 11.1.1, 11.1.9, and 25.1.4.1.*

2. Applicable Document

2.1 ASTM Standard:

A 370 Methods and Definitions for Mechanical Testing of Steel Products³

PRACTICE A—OXALIC ACID ETCH TEST FOR CLASSIFICATION OF ETCH STRUCTURES OF AUSTENITIC STAINLESS STEELS (1)

3. Scope

3.1 The oxalic acid etch test is used for acceptance of material but not for rejection of material. This may be used in connection with other evaluation tests to provide a rapid method for identifying those specimens which are certain to be free of susceptibility to rapid intergranular attack in these other tests. Such specimens have low corrosion rates in the various hot acid tests, requiring from 4 to 240 h of exposure. These specimens are identified by means of their etch

structures which are classified according to the following criteria:

3.2 The oxalic acid etch test may be used to screen specimens intended for testing in Practice B—Ferric Sulfate-Sulfuric Acid Test, Practice C—Nitric Acid Test, Practice D—Nitric-Hydrofluoric Acid Test, and Practice E—Copper-Copper Sulfate-Sulfuric Acid Test.

3.2.1 Each practice contains a table showing which classifications of etch structures on a given stainless steel grade are equivalent to acceptable, or possibly nonacceptable performance in that particular test. Specimens having acceptable etch structures need not be subjected to the hot acid test. Specimens having nonacceptable etch structures must be tested in the specified hot acid solution.

3.3 The grades of stainless steels and the hot acid tests for which the oxalic acid etch test is applicable are listed in Table 2.

3.4 Extra low carbon grades, and stabilized grades, such as 304L, 316L, 317L, 321, and 347, are tested after sensitizing heat treatments at 1200 to 1250°F (650 to 675°C), which is the range of maximum carbide precipitation. These sensitizing treatments must be applied before the specimens are submitted to the oxalic acid etch test. The most commonly used sensitizing treatment is 1 h at 1250°F.

4. Apparatus

4.1 *Source of Direct Current*—Battery, generator, or rectifier capable of supplying about 15 V and 20 A.

4.2 *Ammeter*—Range 0 to 30 A (Note 1).

4.3 *Variable Resistance* (Note 1).

4.4 *Cathode*—A cylindrical piece of stainless steel or, preferably, a 1-qt (0.946-L) stainless steel beaker.

4.5 *Large Electric Clamp*—To hold specimen to be etched.

4.6 *Metallurgical Microscope*—For examination of etched microstructures at 250 to 500 diameters.

4.7 *Electrodes of the Etching Cell*—The specimen to be etched is made the anode, and a stainless steel beaker or a piece of stainless steel as large as the specimen to be etched is made the cathode.

² The boldface numbers in parentheses refer to the list of references found at the end of these practices.

³ *Annual Book of ASTM Standards*, Vols 01.01 to 01.05.

4.8 *Electrolyte*—Oxalic acid, ($\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$), reagent grade, 10 weight % solution.

NOTE 1—The variable resistance and the ammeter are placed in the circuit to measure and control the current on the specimen to be etched.

5. Preparation of Test Specimens

5.1 *Cutting*—Sawing is preferred to shearing, especially on the extra-low carbon grades. Shearing cold works adjacent metal and affects the response to subsequent sensitization. Microscopical examination of an etch made on a specimen containing sheared edges, should be made on metal unaffected by shearing. A convenient specimen size is 1 by 1 in. (25 by 25 mm).

5.2 The intent is to test a specimen representing as nearly as possible the surface of the material as it will be used in service. Therefore the preferred sample is a cross section including the surface to be exposed in service. Only such surface finishing should be performed as is required to remove foreign material and obtain a standard, uniform finish as described in 5.3. For very heavy sections, specimens should be machined to represent the appropriate surface while maintaining reasonable specimen size for convenient testing. Ordinarily, removal of more material than necessary will have little influence on the test results. However, in the special case of surface carburization (sometimes encountered, for instance, in tubing or castings when lubricants or binders containing carbonaceous materials are employed) it may be possible by heavy grinding or machining to completely remove the carburized surface. Such treatment of test specimens is not permissible, except in tests undertaken to demonstrate such effects.

5.3 *Polishing*—On all types of materials, cross sectional surfaces should be polished for etching and microscopical examination. Specimens containing welds should include base plate, weld heat-affected zone, and weld metal. Scale should be removed from the area to be etched, by grinding to an 80 or 120-grit finish on a grinding belt or wheel without excessive heating, and then polishing on successively finer emery papers, No. 1, $\frac{1}{2}$, $\frac{1}{6}$, $\frac{2}{6}$, and $\frac{3}{6}$, or finer. This polishing operation can be carried out in a relatively short time since all large scratches need not be removed. Whenever practical, a polished area of 1 cm^2 or more is desirable. If any cross sectional dimension is less than 1 cm, a minimum length

of 1 cm should be polished. When the available length is less than 1 cm, a full cross section should be used.

5.4 *Etching Solution*—The solution used for etching is prepared by adding 100 g of reagent grade oxalic acid crystals ($\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$) to 900 mL of distilled water and stirring until all crystals are dissolved.

5.5 *Etching Conditions*—The polished specimen should be etched at 1 A/ cm^2 for 1.5 min. To obtain the correct current density:

5.5.1 The total immersed area of the specimen to be etched should be measured in square centimetres, and

5.5.2 The variable resistance should be adjusted until the ammeter reading in amperes is equal to the total immersed area of the specimen in square centimetres.

5.6 Etching Precautions:

5.6.1 **Caution**—Etching should be carried out under a ventilated hood. Gas, which is rapidly evolved at the electrodes with some entrainment of oxalic acid, is poisonous and irritating to mucous membranes.

5.6.2 A yellow-green film is gradually formed on the cathode. This increases the resistance of the etching cell. When this occurs, the film should be removed by rinsing the inside of the stainless steel beaker (or the steel used as the cathode) with an acid such as 30 % HNO_3 .

5.6.3 The temperature of the etching solution gradually increases during etching. The temperature should be kept below 50°C by alternating two beakers. One may be cooled in tap water while the other is used for etching. The rate of heating depends on the total current (ammeter reading) passing through the cell. Therefore, the area etched should be kept as small as possible while at the same time meeting the requirements of desirable minimum area to be etched.

5.6.4 Immersion of the clamp holding the specimen in the etching solution should be avoided.

5.7 *Rinsing*—Following etching, the specimen should be thoroughly rinsed in hot water and in acetone or alcohol to avoid crystallization of oxalic acid on the etched surface during drying.

5.8 On some specimens containing molybdenum (AISI 316, 316L, 317, 317L) which are free of chromium carbide sensitization, it may be difficult to reveal the presence of step structures by electrolytic etching with oxalic acid. In such

cases, an electrolyte of a 10 % solution of ammonium persulfate, $(\text{NH}_4)_2\text{S}_2\text{O}_8$, may be used in place of oxalic acid. An etch of 5 or 10 min at 1 A/cm² in a solution at room temperature readily develops step structures on such specimens.

6. Classification of Etch Structures

6.1 The etched surface is examined on a metallurgical microscope at 250× to 500× for wrought steels and at about 250× for cast steels.

6.2 The etched cross-sectional areas should be thoroughly examined by complete traverse from inside to outside diameters of rods and tubes, from face to face on plates, and across all zones such as weld metal, weld-affected zones, and base plates on specimens containing welds.

6.3 The etch structures are classified into the following types (Note 2):

6.3.1 *Step Structure* (Fig. 1)—Steps only between grains, no ditches at grain boundaries.

6.3.2 *Dual Structure* (Fig. 2)—Some ditches at grain boundaries in addition to steps, but no single grain completely surrounded by ditches.

6.3.3 *Ditch Structure* (Fig. 3)—One or more grains completely surrounded by ditches.

6.3.4 *Isolated Ferrite* (Fig. 4)—Observed in castings and welds. Steps between austenite matrix and ferrite pools.

6.3.5 *Interdendritic Ditches* (Fig. 5)—Observed in castings and welds. Deep interconnected ditches.

6.3.6 *End-Grain Pitting I* (Fig. 6)—Structure contains a few deep end-grain pits along with some shallow etch pits at 500×. (Of importance only when nitric acid test is used.)

6.3.7 *End-Grain Pitting II* (Fig. 7)—Structure contains numerous, deep end-grain pits at 500×. (Of importance only when nitric acid test is used.)

NOTE 2—All photomicrographs were made with specimens that were etched under standard conditions: 10 % oxalic acid, room temperature, 1.5 min at 1 A/cm².

6.4 The evaluation of etch structures containing steps only and of those showing grains completely surrounded by ditches in every field can be carried out relatively rapidly. In cases that appear to be dual structures, more extensive examination is required to determine if there are any grains completely encircled. If an encircled grain is found, the steel should be evaluated as a ditch structure. Areas near surfaces should be examined for evidence of surface carburization.

6.4.1 On stainless steel castings (also on weld

metal) the steps between grains formed by electrolytic oxalic acid etching tend to be less prominent than those on wrought materials, or are entirely absent. However, any susceptibility to intergranular attack is readily detected by pronounced ditches.

6.5 Some wrought specimens, especially from bar stock, may contain a random pattern of pits. If these pits are sharp and so deep that they appear black (Fig. 7) it is possible that the specimen may be susceptible to end grain attack in nitric acid only. Therefore, even though the grain boundaries all have step structures, specimens having as much or more end grain pitting than that shown in Fig. 7 cannot be safely assumed to have low nitric acid rates and should be subjected to the nitric acid test whenever it is specified. Such sharp, deep pits should not be confused with the shallow pits shown in Figs. 1 and 6.

7. Use of Etch Structure Classifications

7.1 The use of these classifications depends on the hot acid corrosion test for which stainless steel specimens are being screened by etching in oxalic acid and is described in each of the practices. Important characteristics of each of these tests are described below.

7.2 *Practice B—Ferric Sulfate-Sulfuric Acid Test* is a 120-h test in boiling 50 % solution that detects susceptibility to intergranular attack associated primarily with chromium carbide precipitate. It does not detect susceptibility associated with sigma phase in chromium-nickel-molybdenum stainless steels (316, 316L, 317, 317L) which is known to lead to rapid intergranular attack only in certain nitric acid environments. It does not detect susceptibility to end grain attack which is also found only in certain nitric acid environments. The ferric sulfate-sulfuric acid test does reveal susceptibility associated with a sigma-like phase constituent in stabilized stainless steels, AISI 321 and 347.

7.3 *Practice C—Nitric Acid Test* is a 240-h test in boiling, 65 % nitric acid which detects susceptibility to rapid intergranular attack associated with chromium carbide precipitate and with sigma-like phase precipitate. The latter may be formed in molybdenum-bearing and in stabilized grades of austenitic stainless steels and may or may not be visible in the microstructure. This test also reveals susceptibility to end grain attack in all grades of stainless steels.

7.4 *Practice D—Nitric-Hydrofluoric Acid*

Test is a 4-h test in a solution of 10 % nitric acid and 3 % hydrofluoric acid at 70°C. It is applicable only to molybdenum-bearing grades of austenitic stainless steels (AISI 316, 316L, 317, 317L) and detects only susceptibility to intergranular attack associated with chromium carbide precipitates. It does not detect susceptibility to intergranular attack associated with sigma phase or end grain corrosion, which, so far, are known to lead to rapid intergranular attack only in certain nitric acid environments.

7.5 Practice E—Copper-Copper Sulfate-Sulfuric Acid Test is a 24-h test in a boiling solution containing 16 % sulfuric acid and 6 % copper sulfate with the test specimen embedded in metallic copper shot or grindings, which detects susceptibility to intergranular attack associated with the precipitation of chromium-rich carbides. It does not detect susceptibility to intergranular attack associated with sigma phase, or end-grain corrosion, both of which have been observed to date only in certain nitric acid environments.

PRACTICE B—FERRIC SULFATE-SULFURIC ACID TEST FOR DETECTING SUSCEPTIBILITY TO INTERGRANULAR ATTACK IN AUSTENITIC STAINLESS STEELS (2)

8. Scope

8.1 This practice describes the procedure for conducting the boiling, 120-h ferric sulfate-50 % sulfuric acid test (Note 3) which measures the susceptibility of stainless steels to intergranular attack. The presence or absence of intergranular attack in this test is not necessarily a measure of the performance of the material in other corrosive environments. The test does not provide a basis for predicting resistance to forms of corrosion other than intergranular, such as general corrosion, pitting, or stress-corrosion cracking.

NOTE 3—See Practice A for information on the most appropriate of the several test methods available for the evaluation of specific grades of stainless steel.

8.1.1 The ferric sulfate-sulfuric acid test detects susceptibility to intergranular attack associated with the precipitation of chromium carbides in unstabilized austenitic stainless steels. It does not detect susceptibility to intergranular attack associated with sigma phase in austenitic stainless steels containing molybdenum, such as Types 316, 316L, 317, and 317L.

NOTE 4—To detect susceptibility to intergranular attack associated with sigma phase in austenitic stainless steels containing molybdenum, the nitric acid test, Practice C, should be used.

8.2 In stabilized stainless steel, Type 321 (and perhaps 347) the ferric sulfate-sulfuric acid test detects susceptibility associated with precipitated chromium carbides and with a sigma phase which may be invisible in the microstructure.

8.3 The ferric sulfate-sulfuric acid test may be used to evaluate the heat treatment accorded as-received material. It may also be used to check the effectiveness of stabilizing columbium or titanium additions and of reductions in carbon content in preventing susceptibility to rapid intergranular attack. It may be applied to wrought products (including tubes), castings, and weld metal.

8.4 Specimens of extra low carbon and stabilized grades are tested after sensitizing heat treatments at 1200 to 1250°F (650 to 675°C), which is the range of maximum carbide precipitation. The length of time of heating used for this sensitizing treatment determines the maximum permissible corrosion rate for such grades in the ferric sulfate-sulfuric acid test. The most commonly used sensitizing treatment is 1 h at 1250°F.

9. Rapid Screening Test

9.1 Before testing in the ferric sulfate sulfuric acid test, specimens of certain grades of stainless steels (see Table 3) may be given a rapid screening test in accordance with procedures given in Practice A, Oxalic Acid Etch Test for Classification of Etch Structures of Austenitic Stainless Steels. Preparation, etching, and the classification of etch structures are described therein. The use of etch structure evaluations in connection with the ferric sulfate-sulfuric acid test is specified in Table 3.

9.1.1 Corrosion test specimens having acceptable etch structures in the oxalic acid etch test will be essentially free of intergranular attack in the ferric sulfate-sulfuric acid test. Such specimens are acceptable without testing in the ferric sulfate-sulfuric acid test. All specimens having nonacceptable etch structures must be tested in the ferric sulfate-sulfuric acid test.

10. Apparatus

10.1 The apparatus (Note 6) is illustrated in Fig. 8.

10.1.1 A four-bulb Allihn or Soxhlet condenser with a 45/50 ground glass joint. Overall length: about 13 in. (330 mm), condensing section, 9½ in. (241 mm).

10.1.2 A 1-L Erlenmeyer flask with a 45/50 ground glass joint. The ground glass opening is somewhat over 1½ in. (38 mm) wide.

10.1.3 The glass cradle (Note 5) can be supplied by a glass-blowing shop. To pass through the ground glass joint on the Erlenmeyer flask, the width of the cradle should not exceed 1½ in., and the front-to-back distance must be such that the cradle will fit the 1⅓-in. (34-mm) diameter opening. It should have three or four holes to increase circulation of the testing solution around the specimen.

NOTE 5—Other equivalent means of specimen support, such as glass hooks or stirrups, may also be used.

10.1.4 Boiling chips must be used to prevent bumping.

10.1.5 A silicone grease⁴ is recommended for the ground glass joint.

10.1.6 During testing, there is some deposition of iron oxides on the upper part of the Erlenmeyer flask. This can be readily removed, after test completion, by boiling a solution of 10 % hydrochloric acid in the flask.

10.1.7 A device such as an electrically heated hot plate which provides heat for continuous boiling of the solution.

10.1.8 An analytical balance capable of weighing to the nearest 0.001 g.

NOTE 6—No substitutions for this equipment may be used. The cold-finger type of condenser with standard Erlenmeyer flasks may not be used.

11. Ferric Sulfate-Sulfuric Acid Test Solution

11.1 Prepare 600 mL of 50 % (49.4 to 50.9 %) solution as follows:

11.1.1 **Caution**—Protect the eyes and use rubber gloves for handling acid. Place the test flask under a hood.

11.1.2 First, measure 400.0 mL of distilled water in a 500-mL graduate and pour into the Erlenmeyer flask.

11.1.3 Then measure 236.0 mL of reagent grade sulfuric acid of a concentration which must be in the range from 95.0 to 98.0 % by weight in a 250-mL graduate. Add the acid slowly to the water in the Erlenmeyer flask to avoid boiling by the heat evolved.

NOTE 7—Loss of vapor results in concentration of the acid.

11.1.4 Weigh 25 g of reagent grade ferric sulfate (contains about 75 % $\text{Fe}_2(\text{SO}_4)_3$) and add to the sulfuric acid solution. A trip balance may be used.

11.1.5 Drop boiling chips into the flask.

11.1.6 Lubricate ground glass joint with silicone grease.

11.1.7 Cover flask with condenser and circulate cooling water.

11.1.8 Boil solution until all ferric sulfate is dissolved (see Note 7).

11.1.9 **Caution**—It has been reported that violent boiling resulting in acid spills can occur. It is important to ensure that the concentration of acid does not become more concentrated and that an adequate number of boiling chips (which are resistant to attack by the test solution) are present.⁵

12. Preparation of Test Specimens

12.1 A specimen having a total surface area of 5 to 20 cm² is recommended. Specimens containing welds should be cut so that no more than ½-in. (13-mm) width of base metal is included on either side of the weld.

12.2 The intent is to test a specimen representing as nearly as possible the surface of the material as used in service. Only such surface finishing should be performed as is required to remove foreign material and obtain a standard, uniform finish as specified. For very heavy sections, specimens should be machined to represent the appropriate surface while maintaining reasonable specimen size for convenience in testing. Ordinarily, removal of more material than necessary will have little influence on the test results. However, in the special case of surface carburization (sometimes encountered, for instance, in tubing or castings when lubricants or binders containing carbonaceous materials are employed) it may be possible by heavy grinding or machining to remove the carburized layer completely. Such treatment of test specimens is not permissible, except in tests undertaken to demonstrate such surface effects.

⁴ Dow Corning Stopcock Grease has been found satisfactory for this purpose.

⁵Amphoteric alundum granules, Hengar Granules, from the Hengar Co., Philadelphia, PA have been found satisfactory for this purpose.

12.3 When specimens are cut by shearing, the sheared edges should be refinished by machining or grinding prior to testing.

12.4 All surfaces of the specimen, including edges, should be finished using No. 80 or 120 grit abrasive paper. If dry abrasive paper is used, polish slowly to avoid overheating. Sand blasting should not be used.

12.5 All traces of oxide scale formed during heat treatments must be thoroughly removed. Any scale which cannot be removed by grinding, for example, in stamped numbers, must be removed by immersing the specimen in concentrated nitric acid at about 200°F (93°C). (Residual oxide scale causes galvanic action and consequent activation in the test solution.)

12.6 The specimen should be measured including the inner surfaces of any holes and the total exposed area calculated.

12.7 The specimen should then be degreased and dried using suitable nonchlorinated agents, such as soap and acetone, and then weighed to the nearest 0.001 g.

13. Procedure

13.1 Place specimen in glass cradle and immerse in boiling solution.

13.2 Mark liquid level on flask with wax crayon to provide a check on vapor loss which would result in concentration of the acid. If there is an appreciable change in the level, the test must be repeated with fresh solution and a reground specimen.

13.3 Continue immersion of the specimen for a total of 120 h, then remove specimen, rinse in water and acetone, and dry.

13.4 Weigh specimen and subtract weight from original weight.

13.5 No intermediate weighings are usually necessary. The tests can be run without interruption for 120 h. However, if preliminary results are desired, the specimen can be removed at any time for weighing.

13.6 No changes in solution are necessary during the 120-h test periods.

13.7 Additional ferric sulfate inhibitor may have to be added during the test if the corrosion rate is extraordinarily high as evidenced by a change in the color of the solution. More ferric sulfate must be added if the total weight loss of all specimens exceeds 2 g. (During the test, ferric sulfate is consumed at a rate of 10 g for each 1 g of dissolved stainless steel.)

13.8 Several specimens may be tested simultaneously. The number (3 or 4) is limited only by the number of glass cradles that can be fitted into the flask.

14. Calculation and Report

14.1 The effect of the acid solution on the material shall be measured by determining the loss of weight of the specimen. The corrosion rates should be reported as inches of penetration per month (Note 8), calculated as follows:

$$\text{Inches per month} = (287 \times W)/(A \times t \times d)$$

where:

t = time of exposure, h,

A = area, cm^2 ,

W = weight loss, g, and

d = density, g/cm^3 .

for chromium-nickel steels, $d = 7.9 \text{ g/cm}^3$

for chromium-nickel-molybdenum steels, $d = 8.00 \text{ g/cm}^3$

NOTE 8—Conversion factors to other commonly used units for corrosion rates are as follows:

inches per month $\times 12$ = inches per year

inches per month $\times 1000$ = mils per month

inches per month $\times 12,000$ = mils per year

inches per month $\times 8350 \times \text{density}$ = milligrams per square decimetre per day

inches per month $\times 34.8 \times \text{density}$ = grams per square metre per hour

$1.00 \text{ in.}^2 = 6.45 \text{ cm}^2$

PRACTICE C—NITRIC ACID TEST FOR DETECTING SUSCEPTIBILITY TO INTERGRANULAR ATTACK IN AUSTENITIC STAINLESS STEELS

15. Scope

15.1 This practice describes the procedure for conducting the boiling nitric acid test (3) as employed to measure the relative susceptibility of austenitic stainless steels to intergranular attack. The presence or absence of intergranular attack in this test is not necessarily a measure of the performance of the material in other corrosive environments; in particular, it does not provide a basis for predicting resistance to forms of corrosion other than intergranular, such as general corrosion, pitting, or stress-corrosion cracking.

15.2 The boiling nitric acid test may be used to evaluate the heat treatment accorded "as-received" material. It is also sometimes used to check the effectiveness of stabilizing elements and of reductions in carbon content in preventing susceptibility to rapid intergranular attack.

NOTE 9—Intergranular attack in nitric acid is asso-

ciated with one or more of the following: (1) intergranular precipitation of chromium carbides, (2) sigma or transition phases in molybdenum-bearing grades, and (3) sigma phase constituents in stabilized grades. The boiling nitric acid test should not be used for extra low carbon molybdenum-bearing grades unless the material tested is to be used in nitric acid service. See Practice A, Oxalic Acid Etching Test, for information on the most appropriate of the several test methods available for the evaluation of specific grades of stainless steel.

15.3 Specimens of extra low carbon and stabilized grades are tested after sensitizing heat treatments at 1200 to 1250°F (650 to 675°C), which is the range of maximum carbide precipitation. The length of time used for this sensitizing treatment determines the maximum permissible corrosion rate in the nitric acid test. The most commonly used sensitizing treatment is 1 h at 1250°F.

15.4 This practice may be applied to wrought products (including tubes), castings, and weld metal of the various grades of stainless steel (Note 9).

16. Rapid Screening Test

16.1 Before testing in the nitric acid test, specimens of certain grades of stainless steel as given in Table 1 may be given a rapid screening test in accordance with procedures given in Practice A, Oxalic Acid Etch Test for Classification of Etch Structures of Austenitic Stainless Steels. The use of the etch structure evaluations in connection with the nitric acid test is specified in Table 4.

16.1.1 Corrosion test specimens having acceptable etch structures in the oxalic acid etch test will be essentially free of intergranular attack in the nitric acid test; such specimens are acceptable without testing in the nitric acid test. All specimens having nonacceptable etch structures must be tested in the nitric acid test.

17. Apparatus

17.1 *Container*—A 1-L Erlenmeyer flask equipped with a cold finger-type condenser, as illustrated in Fig. 9, is recommended.

NOTE 10—Two other types of containers have been employed in the past and may be used if agreed upon between the supplier and purchaser. One of these consists of a 1-L Erlenmeyer flask with a ground glass joint and equipped with a 30-in. (762-mm) reflux condenser; it has been shown that results obtained with a reflux condenser tend to be somewhat higher than with the cold finger-type condenser due to greater vapor loss. The second type of container is the so called multi-sample testing apparatus (4) which was designed to

permit the testing of a large number of specimens simultaneously by providing for replacement of the acid in contact with the specimens several times per hour with redistilled acid. Because of the lesser accumulation of corrosion products in the testing solution, the rates obtained with the multi-sample tester are consistently lower than those obtained with the conventional apparatus; the differences are small on properly annealed or stabilized material which will show low rates in both types of test but can be very large for sensitized specimens. For research purposes or where results are to be compared directly, it is essential that the same type of apparatus be used for all tests.

17.2 *Specimen Supports*—Glass hooks, stirrups, or cradles for supporting the specimens in the flask fully immersed at all times during the test and so designed that specimens tested in the same container do not come in contact with each other.

17.3 *Heater*—A means for heating the test solutions and of keeping them boiling throughout the test period. An electrically heated hot plate is satisfactory for this purpose.

17.4 *Balance*—An analytical balance capable of weighing to at least the nearest 0.001 g.

18. Nitric Acid Test Solution

18.1 The test solution shall be 65 ± 0.2 weight % as nitric acid determined by analysis. This solution may be prepared by adding distilled water to concentrated nitric acid (reagent grade HNO_3 , sp gr 1.42) (Note 11) at the rate of 108 mL of distilled water per litre of concentrated nitric acid.

NOTE 11—The nitric acid used should conform to the recommended specifications for analytical reagent chemicals of the American Chemical Society (5) as follows:

Nonvolatile matter, max, %	0.0005
Sulfate (SO_4), max, %	0.0002
Arsenic, max, %	0.000003
Chlorine, max, % about	0.00007
Heavy metals, max, % about lead and iron	0.0005 0.0001

In addition, the fluorine content shall not exceed 0.0001 % and phosphate (PO_4) shall not exceed 0.00002 %.

19. Preparation of Test Specimens

19.1 The size and shape of the specimen must be considered with respect to available facilities for accurate weighing and the volume of test solution to be used. Normally, the maximum convenient weight of specimen is about 100 g. Specimens containing welds should be cut so that

no more than 1/2 in. (13 mm) width of base metal is included on either side of the weld. Furthermore, in the case of bar, wire, and tubular products, the proportion of the total area represented by the exposed cross section may influence the results. Cross-sectional areas in these products may be subject to end grain attack in nitric acid. The proportion of end grain in the specimen should therefore be kept low unless such surface is actually to be exposed in service involving nitric acid. When specimens of such products are being tested in research investigations, the ratio of the cross-sectional area exposed to the total area should be kept constant from test to test. For inspection tests, specimens cut from bars, wires, or tubes should be proportioned so that the areas of the exposed cross sections shall not exceed half the total exposed area of the specimen.

19.2 Special heat treatment of specimens prior to testing or the use of specimens which contain a weld may be specified.

19.3 When specimens are cut by shearing, the sheared edges should be refinished by machining or grinding prior to testing.

19.4 All surfaces of the specimen, including edges, should be finished using No. 80 or 120 grit abrasive paper. If dry abrasive paper is used, polish slowly to avoid overheating. Sandblasting should not be used.

19.5 The intent is to test a specimen representing as nearly as possible the surface of the material as it will be used in service. Only such surface machining should be performed as is required to remove foreign material and obtain a standard uniform finish as specified in 19.4. For very heavy sections, specimens should be machined to represent the appropriate surface while maintaining reasonable specimen size for convenience in testing. Ordinarily, removal of more material than necessary will have little influence on the test results. However, in the special case of surface carburization (sometimes encountered, for instance, in tubing or castings when lubricants or binders containing carbonaceous materials are employed), it may be possible by heavy grinding or machining to remove the carburized surface completely. Such treatment of test specimens is not permissible except in tests undertaken to demonstrate such surface effects.

19.6 The specimen should be measured including the inner surfaces of any holes and the total exposed area calculated.

19.7 The specimen should then be degreased and dried using suitable nonchlorinated agents, such as soap and acetone (Note 12), and then weighed to the nearest 0.001 g (see 17.4).

NOTE 12—The cleaning treatment described may be supplemented by immersing the specimen in nitric acid (for example, 20 weight % at 120 to 140°F (49 to 60°C) for 20 min), followed by rinsing, drying, and weighing. In the case of small-diameter tubular specimens which cannot be conveniently resurfaced on the inside, it is desirable to include in the preparation an immersion in boiling nitric acid (65 %) for 2 to 4 h using the same apparatus as for the actual test. The purpose of these treatments is to remove any surface contamination that may not be accomplished by the regular cleaning method and which may increase the apparent weight loss of the specimen during the early part of the test.

19.8 It is common practice to test only one specimen of each material or lot of material, as defined by those using the test for specification purposes. However, the use of at least two specimens for check purposes is recommended.

20. Procedure

20.1 Use a sufficient quantity of the nitric acid test solution to cover the specimens and to provide a volume of at least 125 mL/in.² (20 mL/cm²) of specimen surface. Normally, a volume of about 600 cm³ is used.

20.2 The best practice is to use a separate container for each test specimen.

NOTE 13—For routine evaluations, it is acceptable to test as many as three specimens in the same container provided that they all are of the same grade and all show satisfactory resistance to corrosion. If more than one of the specimens tested in the same container fail to pass the test, it is necessary to retest all specimens in separate containers, since excessive corrosion of one specimen may result in accelerated corrosion of the other specimens tested with it. Excessive corrosion may often be detected by changes in the color of the test solution, and it may be appropriate to provide separate containers for such specimens without waiting until the end of the test period. A record should be made showing which specimens were tested together.

NOTE 14—If the multi-sample testing apparatus (see Note 10) is employed, a large number of specimens may be tested in the large container provided.

20.3 After the specimens have been placed in the acid in the container, pass cooling water through the condenser and bring the acid to a boil on the hot plate and then keep boiling throughout the test period (Note 15). After each test period, rinse the specimens with water and treat by scrubbing with rubber or a nylon brush under running water to remove any adhering

corrosion products, after which they should be dried and weighed. Drying may be facilitated, if desired, by dipping the specimens in acetone after they are scrubbed.

NOTE 15—Care should be taken to prevent contamination of the testing solution, especially by fluorides, either before or during the test. Experience has shown that the presence of even small amounts of hydrofluoric acid will increase the corrosion rate in the nitric acid test. It is not permissible, for example, to conduct nitric-hydrofluoric acid tests in the same hood with nitric acid tests.

20.4 For most consistent results, the test should consist of five boiling periods of 48 h each (Note 16) with a fresh test solution being used in each period.

NOTE 16—For specification purposes, those experienced in the use of the test may, by mutual agreement, shorten the standard test to three 48-h boiling periods. However, if with this shorter test procedure the rate of attack in the third period should exceed that in either the first or second periods to some previously agreed-upon extent, then the test should be continued for a total of five periods. As an alternative, when the test is being used for inspection prior to approval of steel for shipment, a procedure may be agreed upon by the purchaser and the manufacturer whereby the material will be released for shipment following satisfactory performance in three 48-h boiling periods with final acceptance being dependent upon satisfactory performance in the longer test of five 48-h boiling periods. Also, by mutual agreement, a combination of one 48-h period and two 96-h periods (not necessarily in that order) instead of five 48-h test periods may be acceptable for routine evaluations.

21. Calculation and Report

21.1 Calculation—The effect of the acid on the material shall be measured by determining the loss of weight of the specimen after each test period and for the total of the test periods. Such weight-loss determinations should be made with the accuracy prescribed in 17.1.4. The corrosion rates are usually reported as inches per month (Note 17), calculated in the following rate of corrosion equation:

$$\text{Inches per month} = (287 \times W)/(A \times d \times t)$$

where:

- t = time of exposure, h,
- A = total surface area, cm²,
- W = weight loss, g, and
- d = density of the sample, g/cm³.

NOTE 17—Conversion factors to other commonly used units for corrosion rates are as follows:

- inches per month $\times 12$ = inches per year
- inches per month $\times 1000$ = mils per month

- inches per month $\times 12 \ 000$ = mils per year
- inches per month $\times 8350 \times \text{density}$ = milligrams per square decimetre per day
- inches per month $\times 34.8 \times \text{density}$ = grams per square metre per hour
- 1.00 in.² = 6.45 cm²

21.2 Report—Results should be reported for the individual periods, as well as the average for the three or five test periods.

PRACTICE D — NITRIC - HYDROFLUORIC ACID TEST FOR DETECTING SUSCEPTIBILITY TO INTERGRANULAR ATTACK IN MOLYBDENUM-BEARING AUSTENITIC STAINLESS STEELS (6)

22. Scope

NOTE 18—See Practice A for information on the most appropriate of the several test methods available for the evaluation of specific grades of stainless steel.

22.1 This practice describes the procedure for conducting the 70°C 4-h, 10 % nitric-3 % hydrofluoric acid test as employed to measure the susceptibility of molybdenum-bearing stainless steels to intergranular attack. The presence or absence of intergranular attack in this test is not necessarily a measure of the performance of the material in other corrosive environments. The test does not provide a basis for predicting resistance to forms of corrosion other than intergranular, such as general corrosion, pitting, or stress-corrosion cracking.

22.1.1 The 10 % nitric-3 % hydrofluoric acid test detects susceptibility to intergranular attack associated with the precipitation of chromium carbides in molybdenum-bearing stainless steels (Types 316, 317, 316L, and 317L). It does not detect susceptibility to intergranular attack associated with sigma phase in these same types of stainless steel.

22.2 The 10 % nitric-3 % hydrofluoric acid test may be used to evaluate the heat treatment accorded as-received material (Types 316 and 317 stainless steel). It may also be used to check the effectiveness of reduction in carbon content in preventing susceptibility to rapid intergranular attack (Types 316L and 317L). It may be applied to wrought products (including tubes), castings, and weld metal.

22.3 Specimens of extra low carbon grades (Types 316L and 317L) are tested after sensitizing heat treatments at 1200 to 1250°F (650 to 675°C), which is the range of maximum carbide precipitation. The length of time of heating used for this

sensitizing treatment will generally be 1 or 2 h. The most commonly used sensitizing treatment is 1 h at 1250°F.

23. Summary of Practice

23.1 Types 316 and 317 Stainless Steel—The material submitted for evaluation is tested in each of two conditions of heat treatment: (1) as-received (commercially annealed) and (2) laboratory-annealed for 1 h at 1900 to 2000°F (1040 to 1095°C), and water quenched.

23.2 Types 316L and 317L Stainless Steel—The material submitted for evaluation test is tested in each of two conditions of heat treatment: (1) a sensitized specimen, and (2) a baseline specimen, which usually is the as-received (commercially annealed) specimen. If the as-received specimen does not show a step structure in the oxalic acid etch test, a portion of it must be laboratory annealed to give a step structure and that sample used as the baseline specimen (see footnote C of Table 5).

24. Rapid Screening Test

24.1 Before testing in the 10 % nitric-3 % hydrofluoric acid test, specimens of the stainless steels as given in Table 5 may be given a rapid screening test in accordance with procedures given in Practice A, Oxalic Acid Etch Test for Classification of Etch Structures of Austenitic Stainless Steels. Preparation, etching, and the classification of etch structures are described therein. The use of etch-structure evaluations in connection with the 10 % nitric-3% hydrofluoric acid test is specified in Table 5.

24.1.1 Corrosion test specimens having acceptable etch structures in the Oxalic Acid Etch Test will be largely free of intergranular attack in the 10 % nitric-3% hydrofluoric acid test. Such specimens are acceptable without testing in the 10 % nitric-3 % hydrofluoric acid test. All specimens having nonacceptable etch structures must be tested in the 10 % nitric-3 % hydrofluoric acid test.

25. Apparatus

25.1 The apparatus is illustrated in Fig. 9.

25.1.1 Test Cylinders (Note 19)—The tests are conducted in cylinders of poly(vinyl chloride) (PVC) as shown in Fig. 10. The test cylinders can be made from 12-in. (305-mm) lengths of 1¼-in. PVC pipe (¾-in. (4.76 mm) wall) by either of

the following two techniques: (1) one end of each pipe length is plugged with a disk of ¾-in. PVC sheet and the disk is then heat-welded in place with PVC filler rod, or (2) one end of each pipe length is plugged by solvent-welding a 1¼-in. NPS (Schedule 80) socket-type PVC cap onto the end of the pipe.

25.1.2 Specimen Holders (Note 19)—The specimens may be suspended in the test solution by means of either a specimen holder (as described in this paragraph) or a TFE-fluorocarbon string through an appropriate hole drilled in one end of the specimen. The specimen holders may be made in either of two ways: (1) a 1-in. (25-mm) length of TFE-fluorocarbon tubing (1-in. inside diameter) is drilled at one end to accommodate a ¾-in. (4.76-mm) TFE-fluorocarbon rod. The holders are flattened into an elliptical shape which is maintained by inserting the rod through the two holes and upsetting the ends of the rod with a hammer, or (2) a ½-in. NPS (Schedule 80) socket-type PVC cap is machined to reduce the outer diameter of the cap to 1¼ in. (27.0 mm). Holes ¼ in. (6.35 mm) in diameter are then drilled in the bottom and sides of the machined PVC caps to allow free circulation of the test solution. Smaller holes are drilled at the top of the PVC cap to attach the TFE-fluorocarbon string. A loop of the same string is attached to each specimen holder and used to suspend it in the test cylinder.

NOTE 19—All poly(vinyl chloride) materials should be specified on the order as Schedule 80, rigid unplasticized normal impact PVC.

25.1.3 Constant-Temperature Bath—The desired solution test temperature of 70°C is obtained by placing the PVC cylinders within a rack in a constant-temperature water bath. The temperature of the bath is maintained at 72 to 73°C to offset the low thermal conductivity of the poly(vinyl chloride).

25.1.4 10 % Nitric-3 % Hydrofluoric Acid Test Solution:

25.1.4.1 Caution—The 10 % nitric-3 % hydrofluoric acid solution will cause severe burns if it comes into contact with the skin. Therefore, extreme care should be exercised in handling this solution. Rubber gloves should be worn. Spilled acid should immediately be washed from the skin with an excess of water and emergency first-aid treatment obtained.

25.1.4.2 A 10 % nitric-3 % hydrofluoric acid

solution (by weight) is prepared by mixing 111 mL of 65 % nitric acid (sp gr 1.39), 54 mL of 48 % hydrofluoric acid (sp gr 1.16), and 784 mL of distilled water in a polyethylene carboy. Fresh test solution should be made up daily to avoid changes in concentration due to evaporation.

26. Preparation of Test Specimens

26.1 A specimen having a total surface area of 5 to 20 cm² is recommended. Specimens containing welds should be cut so that no more than a 1/2-in. (13-mm) width of base metal is included on either side of the weld.

26.2 The intent is to test a specimen representing as nearly as possible the surface of the material as it will be used in service. Only such finishing should be performed as is required to remove foreign material and obtain a standard, uniform finish as specified. For very heavy sections, specimens should be machined to represent the appropriate surface while maintaining reasonable specimen size for convenience in testing. Ordinarily, removal of more material than necessary will have little influence on the test results. However, in the special case of surface carburization (sometimes encountered, for instance, in tubing or castings when lubricants or binders containing carbonaceous materials are employed), it may be possible by heavy grinding or machining to remove the carburized layer completely. Such treatment of test specimens is not permissible.

26.3 When specimens are cut by shearing, the sheared edges should be refinished by machining or grinding prior to testing.

26.4 All surfaces of the specimen, including edges, should be finished using No. 80 or 120 grit abrasive paper. If dry abrasive paper is used, polish slowly to avoid overheating. Sand blasting should not be used.

26.5 The specimen should be measured and the total exposed area, including the inner surfaces of any holes, calculated in square centimetres.

26.6 The specimen should then be degreased and dried using suitable nonchlorinated agents, such as soap and acetone, and then weighed to the nearest 0.001 g.

27. Procedure

27.1 Types of Test Specimens:

27.1.1 *Types 316 and 317*—Test two specimens, one representing the as-received condition

and one representing the laboratory-annealed condition. (The laboratory-annealed specimen must show a step structure in the oxalic acid etch test.)

27.1.2 *Types 316L and 317L*—Test two specimens, one representing the sensitized condition and the other a base-line specimen, which is usually the as-received (commercially annealed) specimen. If the as-received specimen does not show a step structure in the oxalic acid etch test, laboratory anneal a portion of it to give a step structure and use that sample as the base-line specimen (see footnote C of Table 5).

27.2 Fill the PVC test cylinders with 200 mL of 10 % nitric-3 % hydrofluoric acid solution and then heat in the constant temperature water bath until the solution temperature, as measured with a thermometer, is $70 \pm 0.5^\circ\text{C}$.

NOTE 20—It is important that the test solution be maintained at $70 \pm 0.5^\circ\text{C}$ because small changes in solution temperature produce large changes in the corrosion rate.

27.3 When the solution temperature is $70 \pm 0.5^\circ\text{C}$, lower the specimens into the solution by means of the specimen holders or string. After a 2-h exposure, remove the specimens from the test, wash in distilled water and acetone, dry, and weigh. Then expose the two specimens for an additional 2-h test period in fresh test solutions at $70 \pm 0.5^\circ\text{C}$.

27.4 Test only one specimen in each cylinder. In addition, it is preferable to test simultaneously the two specimens representing each of the two conditions of heat treatment for each material evaluated.

28. Calculation and Report

28.1 *Calculation of Individual Corrosion Rates*—The effect of the acid solution on each of the two specimens of each material shall be measured by determining the loss of weight of the specimen. The average corrosion rate in inches per month (Note 21), based on the 4-h test exposure, is calculated for each specimen as follows:

$$\text{Inches per month} = (287 \times W)/(A \times t \times d)$$

where:

t = time of exposure, h,

A = area, cm²,

W = weight loss, g, and

d = density, g/cm³, for chromium-nickel-molybdenum steels, $d = 8.0 \text{ g/cm}^3$.

28.2 Calculations of Corrosion-Rate Ratios:

28.2.1 Types 316 and 317 Stainless Steel—

The ratio of the corrosion rate for the as-received specimen to the corrosion rate for the laboratory-annealed specimen is determined.

28.2.2 Types 316L and 317L Stainless Steel—

The ratio of the corrosion rate for the sensitized specimen to the corrosion rate for the as-received specimen is determined.

28.3 Significance of Corrosion Rate Ratios—

A value of 1.5 or less for the above ratios indicates that the degree of intergranular attack in the 10 % nitric-3 % hydrofluoric acid test was not significant. A ratio of greater than 1.5 indicates that significant intergranular corrosion has occurred in the 10 % nitric-3 % hydrofluoric acid test.

NOTE 21—Conversion factors to other commonly used units for corrosion rates are as follows:

inches per month $\times 12$ = inches per year

inches per month $\times 1000$ = mils per month

inches per month $\times 12\ 000$ = mils per year

inches per month $\times 8350 \times \text{density}$ = milligrams per square decimetre per day

inches per month $\times 34.8 \times \text{density}$ = grams per square metre per hour

$1.00\text{ in.}^2 = 6.45\text{ cm}^2$

PRACTICE E—COPPER-COPPER SULFATE-SULFURIC ACID TEST FOR DETECTING SUSCEPTIBILITY TO INTERGRANULAR ATTACK IN AUSTENITIC STAINLESS STEELS (7) (8)

29. Scope

29.1 This practice describes the procedure by which the copper-copper sulfate-sulfuric acid test is conducted to determine the susceptibility of austenitic stainless steels to intergranular attack. The presence or absence of intergranular corrosion in this test is not necessarily a measure of the performance of the material in other corrosive media. The test does not provide a basis for predicting resistance to other forms of corrosion, such as general corrosion, pitting, or stress-corrosion cracking.

29.2 The copper-copper sulfate-sulfuric acid test indicates susceptibility to intergranular attack associated with the precipitation of chromium-rich carbides. It does not detect susceptibility associated with sigma phase. This test may be used to evaluate the heat treatment accorded as-received material. It may also be used to evaluate the effectiveness of stabilizing element additions (Cb, Ti, etc.) and reductions in carbon

content to aid in resisting intergranular attack.

29.3 All wrought products and weld material of austenitic stainless steels can be evaluated by this test.

30. Rapid Screening Test

30.1 Before testing in the copper-copper sulfate-sulfuric acid test, specimens of certain grades of stainless steel (see Table 6) may be given a rapid screening test in accordance with the procedures given in Practice A (Sections 3 through 7). Preparation, etching, and the classification of etch structures are described therein. The use of etch-structure evaluations in connection with the copper-copper sulfate-sulfuric acid test is specified in Table 6.

30.1.1 Corrosion test specimens having acceptable etch structures in the oxalic acid etch test will be essentially free of intergranular attack in the copper-copper sulfate-sulfuric acid test. Such specimens are acceptable without testing in the copper-copper sulfate-sulfuric acid test. All specimens having nonacceptable etch structures must be tested in the copper-copper sulfate-sulfuric acid test.

31. Summary of Practice

31.1 A suitable sample of an austenitic stainless steel, embedded in copper shot or grindings, is exposed to boiling acidified copper sulfate solution for 24 h. After exposure in the boiling solution, the specimen is bent. Intergranular cracking or crazing is evidence of susceptibility.

32. Apparatus

32.1 A 1-L glass Erlenmeyer flask with a ground 45/50 glass joint and four-bulb Allihn condenser with 45/50 ground glass joint (as in 10.1.1 and 10.1.2 and Fig. 8) are required. A silicone grease is recommended for the ground glass joint.

32.2 *Specimen Supports*—An open glass cradle capable of supporting the specimens and copper shot or grindings in the flask is recommended.

NOTE 22—It may be necessary to embed large specimens, such as from heavy bar stock, in copper shot on the bottom of the test flask. A copper cradle may also be used.

32.3 *Heat Source*—Any gas or electrically heated hot plate may be utilized for heating the

test solution and keeping it boiling throughout the test period.

33. Acidified Copper Sulfate Test Solution

33.1 Dissolve 100 g of copper sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in 700 mL of distilled water, add 100 mL of sulfuric acid (H_2SO_4 , cp, sp gr 1.84), and dilute to 1000 mL with distilled water.

NOTE 23—The solution will contain approximately 6 weight % of anhydrous CuSO_4 and 16 weight % of H_2SO_4 .

34. Copper Addition

34.1 Electrolytic grade copper shot or grindings may be used. Shot is preferred for its ease of handling before and after the test.

34.2 A sufficient quantity of copper shot or grindings is to be used to cover all surfaces of the specimen whether it is in a vented glass cradle or embedded in a layer of copper shot on the bottom of the test flask.

34.3 The amount of copper used, assuming an excess of metallic copper is present, is not critical. The effective galvanic coupling between copper and the test specimen may have importance (9).

34.4 The copper shot or grindings may be reused if they are cleaned in warm tap water after each test.

35. Specimen Preparation

35.1 The size of the sample submitted for test and the area from which it is to be taken (end or middle of coil, midway surface and center, etc.) is generally specified in the agreement between the purchaser and the seller. The testing apparatus dictates the final size and shape of the test specimen. The specimen configuration should permit easy entrance and removal through the neck of the test container.

35.1.1 Table 7 may be used as a guide to determine acceptable specimen sizes. There may be restrictions placed on specimen size by the testing apparatus.

35.1.2 Specimens obtained by shearing should have the sheared edges machined or ground off prior to testing. Care should be taken when grinding to avoid overheating or "burning." A "squared" edge is desirable.

35.2 Any scale on the specimens should be removed mechanically unless a particular surface finish is to be evaluated. Chemical removal of scale is permissible when this is the case. Me-

chanical removal of scale should be accomplished with 120-grit iron-free aluminum oxide abrasive.

35.2.1 Each specimen should be degreased using a cleaning solvent such as acetone, alcohol, ether, or a vapor degreaser prior to being tested.

35.3 All austenitic material in the "as-received" (mill-annealed) condition should be capable of meeting this test.

35.3.1 Specimens of extra low carbon and stabilized grades are tested after sensitizing heat treatments at 1200 to 1250°F (650 to 675°C), which is the range of maximum carbide precipitation. The most commonly used sensitizing treatment is 1 h at 1250°F. Care should be taken to avoid carburizing or nitriding the specimens. The heat treating is best carried out in air or neutral salt.

NOTE 24—The sensitizing treatment (1250°F) is performed to check the effectiveness of stabilized and 0.03 % maximum carbon materials in resisting carbide precipitation, hence, intergranular attack.

36. Test Conditions

36.1 The volume of acidified copper sulfate test solution used should be sufficient to completely immerse the specimens and provide a minimum of 50 mL/in.² (8 mL/cm²) of specimen surface area.

36.1.1 As many as three specimens can be tested in the same container. It is ideal to have all the specimens in one flask to be of the same grade, but it is not absolutely necessary. The solution volume-to-sample area ratio is to be maintained.

36.1.2 The test specimen(s) should be immersed in ambient test solution which is then brought to a boil and maintained boiling throughout the test period. Begin timing the test period when the solution reaches the boiling point.

NOTE 25—Measures should be taken to minimize bumping of the solution when glass cradles are used to support specimens. A small amount of copper shot (8 to 10 pieces) on the bottom of the flask will conveniently serve this purpose.

36.1.3 The time of the test shall be a minimum of 24 h unless a longer time is agreed upon between the purchaser and the producer. If not 24 h, the test time shall be specified on the test report. Fresh test solution would not be needed if the test were to run 48 or even 72 h. (If any adherent copper remains on the specimen, it may

be removed by a brief immersion in concentrated nitric acid at room temperature.)

NOTE 26—Results in the literature indicate that this test is more sensitive if it is run for longer times (11), (12).

37. Bend Test

37.1 The test specimen shall be bent through 180° and over a diameter equal to the thickness of the specimen being bent (see Fig. 11). In no case shall the specimen be bent over a smaller radius or through a greater angle than that specified in the product specification. In cases of material having low ductility, such as severely cold worked material, a 180° bend may prove impractical. Determine the maximum angle of bend without causing cracks in such material by bending an untested specimen of the same configuration as the specimen to be tested.

37.1.1 Duplicate specimens shall be obtained from sheet material so that both sides of the rolled samples may be bent through a 180° bend. This will assure detection of intergranular attack resulting from carburization of one surface of sheet material during the final stages of rolling.

NOTE 26—Identify the duplicate specimen in such a manner as to ensure both surfaces of the sheet material being tested are subjected to the tension side of the 180° bends.

37.1.2 Samples machined from round sections or cast material shall have the curved or original surface on the outside of the bend.

37.1.3 The specimens are generally bent by holding in a vise and starting the bend with a hammer. It is generally completed by bringing the two ends together in the vise. Heavy specimens may require bending in a fixture of suitable design. An air or hydraulic press may also be used for bending the specimens.

37.1.4 Tubular products should be flattened in accordance with the flattening test, prescribed in Methods and Definitions A 370.

37.1.5 When agreed upon between the purchaser and the producer, the following shall apply to austenitic stainless steel plates 0.1875 in. (4.76 mm) and thicker:

37.1.5.1 Samples shall be prepared according to Table 7.

37.1.5.2 The radius of bend shall be two times the sample thickness, and the bend axis shall be perpendicular to the direction of rolling.

37.1.5.3 Welds on material 0.1875 in. (4.76 mm) and thicker shall have the above bend radius, and the weld-base metal interface shall be located approximately in the centerline of the bend.

37.1.5.4 Face, root, or side bend tests may be performed, and the type of bend test shall be agreed upon between the purchaser and the producer. The bend radius shall not be less than that required for mechanical testing in the appropriate material specification (for base metal) or in ASME Code Section IX (for welds).

38. Evaluation

38.1 The bent specimen shall be examined under low (5 to 20×) magnification (see Fig. 12). The appearance of fissures or cracks indicates the presence of intergranular attack (see Fig. 13).

38.1.1 When an evaluation is questionable (see Fig. 14), presence or absence of intergranular attack shall be determined by metallographic examination of a longitudinal section of the specimen at a magnification of 100 to 250×.

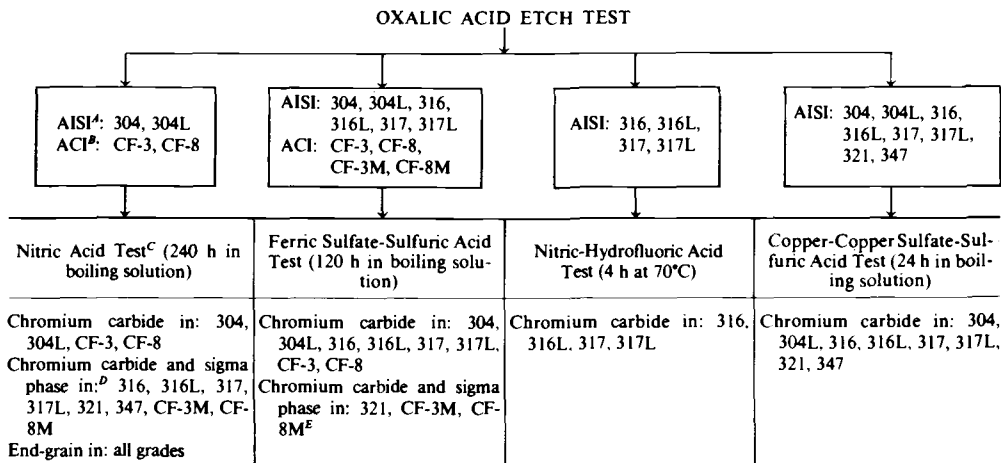
NOTE 27—Cracking that originates at the edge of the specimen should be disregarded. The appearance of deformation lines, wrinkles, or “orange peel” on the surface, without accompanying cracks or fissures, should be disregarded also.

NOTE 28—Cracks suspected as arising through poor ductility may be investigated by bending a similar specimen which was not exposed to the boiling test solution. A visual comparison between these specimens should assist in interpretation.

TABLE 1 Application of Evaluation Tests for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels

NOTE 1—For each corrosion test, the types of susceptibility to intergranular attack detected are given along with the grades of stainless steels in which they may be found. These lists may contain grades of steels in addition to those given in the rectangles. In such cases, the acid corrosion test is applicable, but not the oxalic acid etch test.

NOTE 2—The oxalic acid etch test may be applied to the grades of stainless steels listed in the rectangles when used in connection with the test indicated by the arrow.



^A AISI: American Iron and Steel Institute designations for austenitic stainless steels.

^B ACI: Alloy Casting Institute designations.

^C The nitric acid test may be also applied to AISI 309, 310, 348, and AISI 410, 430, 446, and ACI CN-7M.

^D Must be tested in nitric acid test when destined for service in nitric acid.

^E To date, no data have been published on the effect of sigma phase on corrosion of AISI 347 in this test.

TABLE 2 Applicability of Etch Test

	AISI Grade No.	ACI Grade No.
Practice B—Ferric Sulfate-Sulfuric Acid Test	304, 304L, 316, 316L, 317, 317L	CF-3, CF-8, CF-3M, CF-8M
Practice C—Nitric Acid Test	304, 304L	CF-8, CF-3
Practice D—Nitric Hydrofluoric Acid Test	316, 316L, 317, 317L	...
Practice E—Copper-Copper Sulfate-Sulfuric Acid Test	304, 304L, 316, 316L, 317, 317L, 321, 347	...

TABLE 3 Use of Etch Structure Classifications from the Oxalic Acid Etch Test with Ferric Sulfate-Sulfuric Acid Test

NOTE—Grades AISI 321 and 347 cannot be screened because these grades may contain a type of sigma phase which is not visible in the etch structure but which may cause rapid corrosion in the ferric sulfate-sulfuric acid test.

Grade	Acceptable Etch Structures	Nonacceptable Etch Structures ^A
AISI 304	Step, Dual, End Grain, I & II	Ditch
AISI 304L	Step, Dual, End Grain, I & II	Ditch
AISI 316	Step, Dual, End Grain, I & II	Ditch
AISI 316L	Step, Dual, End Grain, I & II	Ditch
AISI 317	Step, Dual, End Grain, I & II	Ditch
AISI 317L	Step, Dual, End Grain, I & II	Ditch
AISI 321	None	...
ACI CF-3	Step, Dual, Isolated Ferrite Pools	Ditch, Interdendritic Ditches
ACI CF-8	Step, Dual, Isolated Ferrite Pools	Ditch, Interdendritic Ditches
ACI CF-3M	Step, Dual, Isolated Ferrite Pools	Ditch, Interdendritic Ditches
ACI CF-8M	Step, Dual, Isolated Ferrite Pools	Ditch, Interdendritic Ditches

^A Specimens having these structures must be tested in the ferric sulfate-sulfuric acid test.

TABLE 4 Use of Etch Structure Classification from Oxalic Acid Etch Test with Nitric Acid Test

NOTE—AISI 316, 316L, 317, 317L, 347, and 321 cannot be screened because these steels may contain sigma phase not visible in the etch structure. This may cause rapid intergranular attack in the nitric acid test.

Grade	Acceptable Etch Structures	Nonacceptable Etch Structures ⁴
AISI 304	Step, Dual, End Grain I	Ditch, End Grain II
AISI 304L	Step, Dual, End Grain I	Ditch, End Grain II
ACI CF-8	Step, Dual, Isolated Ferrite Pools	Ditch, Interdendritic Ditches
ACI CF-3	Step, Dual, Isolated Ferrite Pools	Ditch, Interdendritic Ditches

⁴ Specimens having these structures must be tested in the nitric acid test.

TABLE 5 Use of Etch Structure Classifications from the Oxalic Acid Etch Test with 10 % Nitric-3 % Hydrofluoric Acid Test

Grade	Condition of Heat Treatment	Acceptable Etch Structures	Nonacceptable Etch Structures ⁴
AISI 316	As-received ^B	Step, Dual, End Grain I and II	Ditch
AISI 317	As-received ^B	Step, Dual, End Grain I and II	Ditch
AISI 316L	As-received ^C	Step, End Grain I and II	Dual, Ditch
	Sensitized	Step, Dual, End Grain I and II	Ditch
AISI 317L	As-received ^C	Step, End Grain I and II	Dual, Ditch
	Sensitized	Step, Dual, End Grain I and II	Ditch

⁴ Specimens having these structures must be tested in 10 % nitric-3 % hydrofluoric acid test.

^B If the as-received specimen shows a ditch structure and the HNO₃-HF test is to be applied, then a duplicate specimen must be laboratory-annealed in order to produce a base-line specimen having a step structure. The laboratory-annealed specimen (step structure) along with the original as-received specimen (ditch structure) is subjected to the HNO₃-HF test. The final criterion is then the ratio of the corrosion rate for the as-received specimen to the corrosion rate for the laboratory-annealed specimen.

^C When the oxalic acid etch test is used with the HNO₃-HF test for Types 316L and 317L, the as-received specimens should show a step structure (free from precipitated carbides). If it does not, a portion of the as-received specimen must be laboratory annealed to produce a base-line specimen having a step structure and that sample used for the HNO₃-HF test. This requirement is necessary because the base-line specimen must be completely free of intergranular attack in the HNO₃-HF test in order to obtain an indicative ratio of the corrosion rate of the sensitized specimen to the corrosion rate of the base-line specimen.

TABLE 6 Use of Etch Structure Classifications from the Oxalic Acid Etch Test with the Copper-Copper Sulfate-Sulfuric Acid Test

Grade	Acceptable Etch Structures	Non-acceptable Etch Structures ⁴
AISI 304	Step, Dual, End Grain I and II	Ditch
AISI 304L	Step, Dual, End Grain I and II	Ditch
AISI 316	Step, Dual, End Grain I and II	Ditch
AISI 316L	Step, Dual, End Grain I and II	Ditch
AISI 317	Step, Dual, End Grain I and II	Ditch
AISI 317L	Step, Dual, End Grain I and II	Ditch
AISI 321	Step, Dual, End Grain I and II	Ditch
AISI 347	Step, Dual, End Grain I and II	Ditch

⁴ Specimens having these structures must be tested in the copper-copper sulfate-sulfuric acid test.

TABLE 7 Sizes of Test Specimens

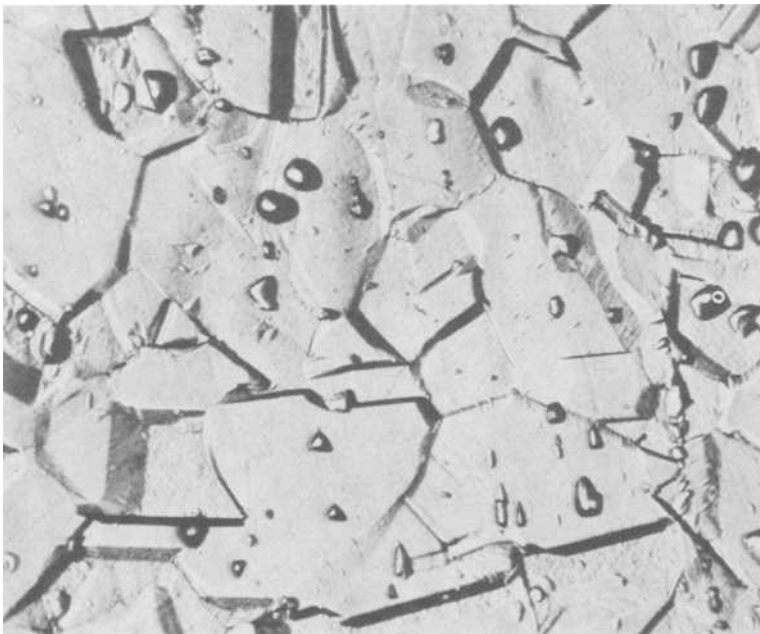
Type of Material	Size of Test Specimen
<i>Wrought wire or rod:</i> Up to $\frac{1}{4}$ in. in diameter, incl	Full diameter by 3 in. (min) long
Over $\frac{1}{4}$ in. in diameter	Cylindrical segment $\frac{1}{4}$ in. thick by 1 in. (max) wide by 3 to 5 in. long ^a
<i>Wrought sheet, strip, plates, or flat rolled products:</i> Up to $\frac{3}{16}$ in. thick, incl	Full thickness by $\frac{3}{4}$ to 1 in. wide by 3 in. (min) long
Over $\frac{3}{16}$ in. thick	$\frac{3}{16}$ to $\frac{1}{2}$ in. thick by $\frac{3}{4}$ to 1 in. wide by 3 in. (min) long ^b
<i>Tubing:</i> Up to $1\frac{1}{2}$ in. in diameter, incl	Full ring, 1 in. wide ^c
Over $1\frac{1}{2}$ in. in diameter	A circumferential segment 3 in. (min) long cut from a 1-in. wide ring ^d

^a When bending such specimens, the curved surface shall be on the outside of the bend.

^b One surface shall be an original surface of the material under test and it shall be on the outside of the bend. Cold-rolled strip or sheets may be tested in the thickness supplied.

^c Ring sections are not flattened or subjected to any mechanical work before they are subjected to the test solution.

^d Specimens from welded tubes over $1\frac{1}{2}$ in. in diameter shall be taken with the weld on the axis of the bend.

FIG. 1 Step Structure (500 $\times$) (Steps between grains, no ditches at grain boundaries)

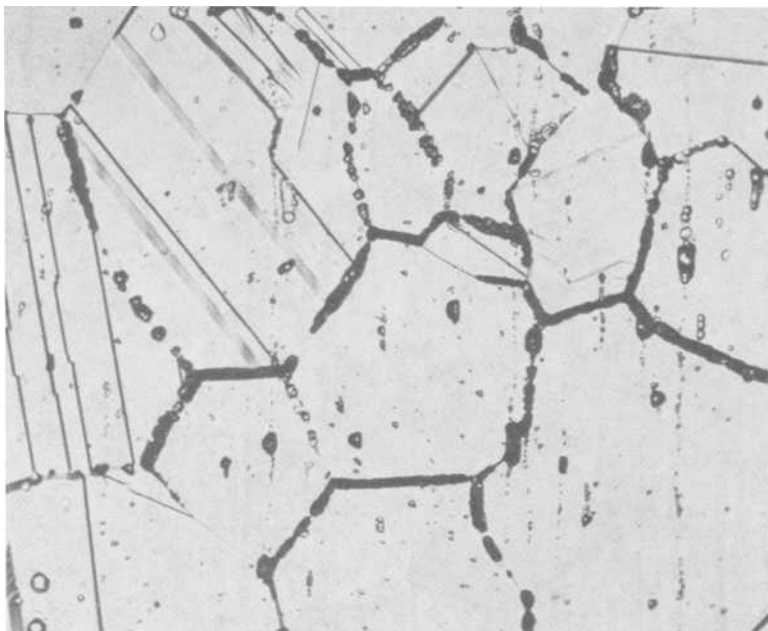


FIG. 2 Dual Structure (250×) (Some ditches at grain boundaries in addition to steps, but no one grain completely surrounded)

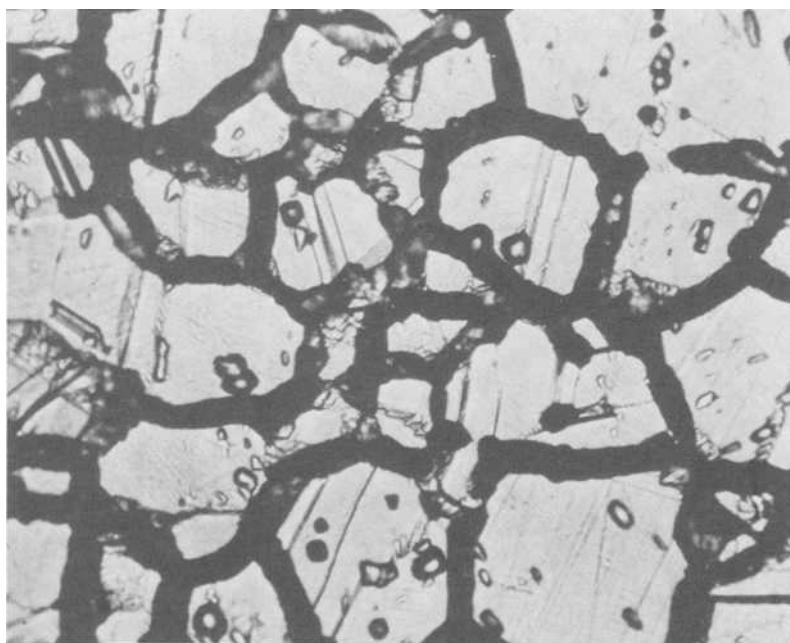


FIG. 3 Ditch Structure (500×) (One or more grains completely surrounded by ditches)

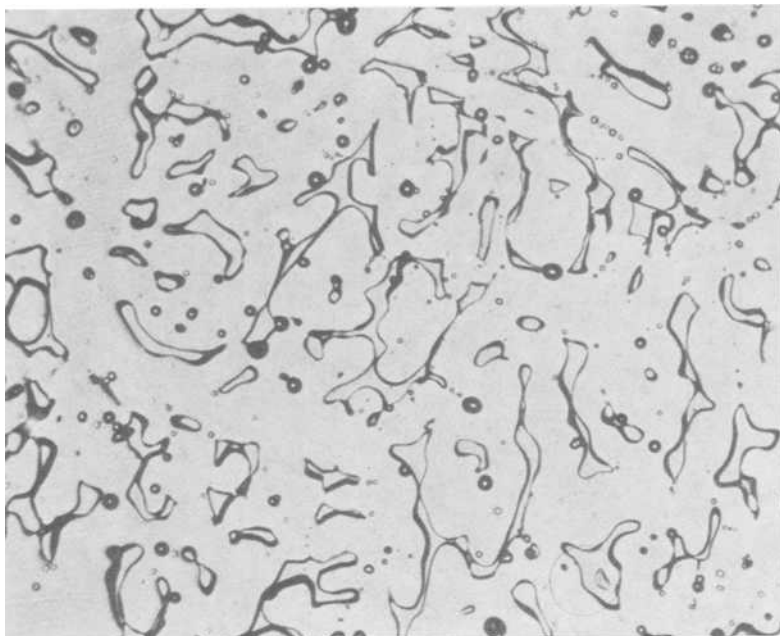


FIG. 4 Isolated Ferrite Pools (250 $\times$) (Observed in castings and welds. Steps between austenite matrix and ferrite pools)

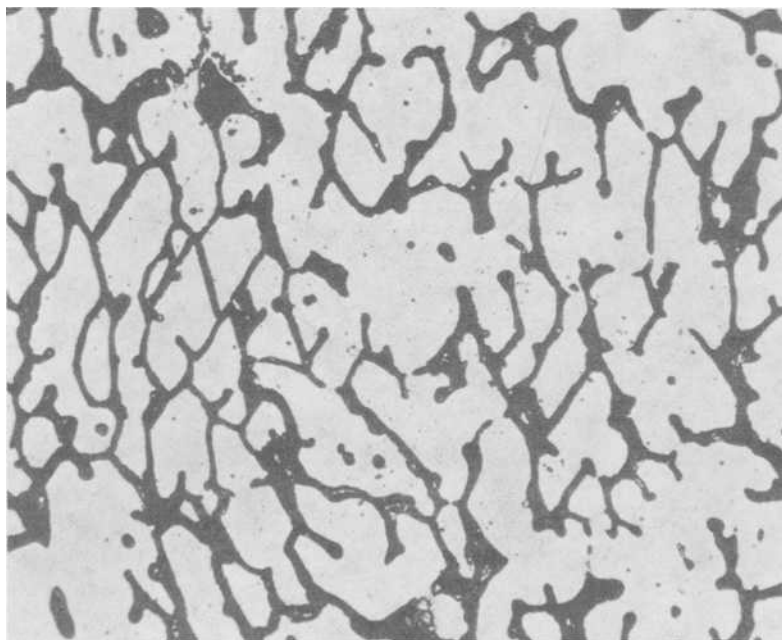
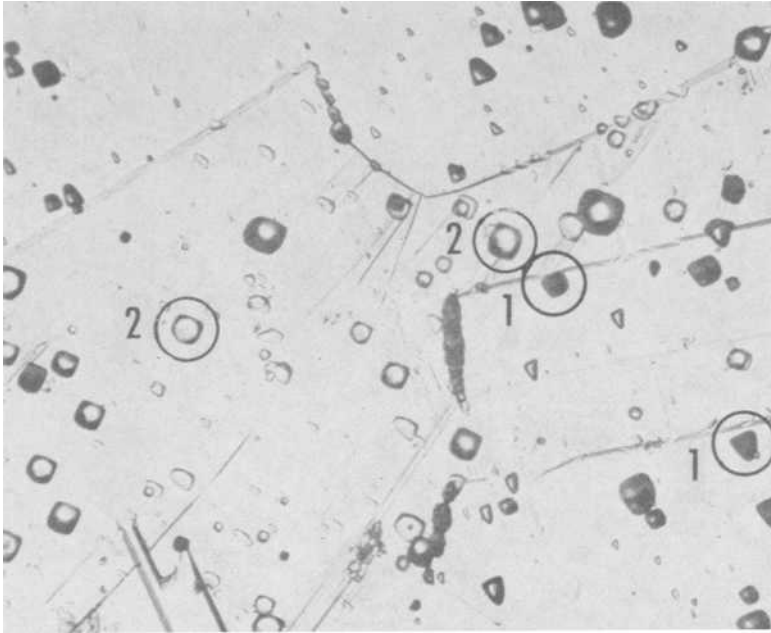
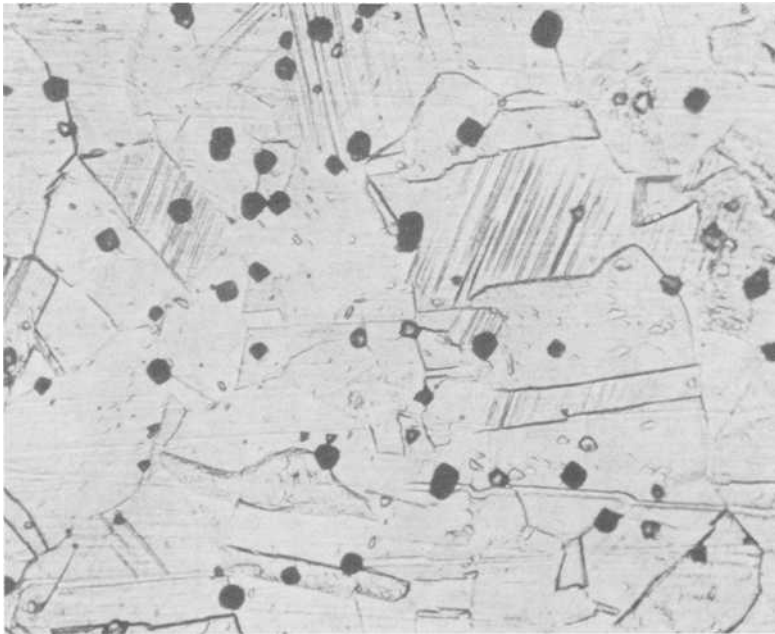


FIG. 5 Interdendritic Ditches (250 $\times$) (Observed in castings and welds. Deep interconnected ditches)



To differentiate between the types of pits, use a magnification of 500× and focus in the plane of etched surface. The pits which now appear completely black are end grain pits.

FIG. 6 End Grain Pitting I (500×) (A few deep end grain pits (see 1 in figure) and shallow etch pits (2))



This or a greater concentration of end grain pits at 500× (using standard etching conditions) indicates that the specimen must be tested when screening is for nitric acid test.

FIG. 7 End Grain Pitting II (500×)

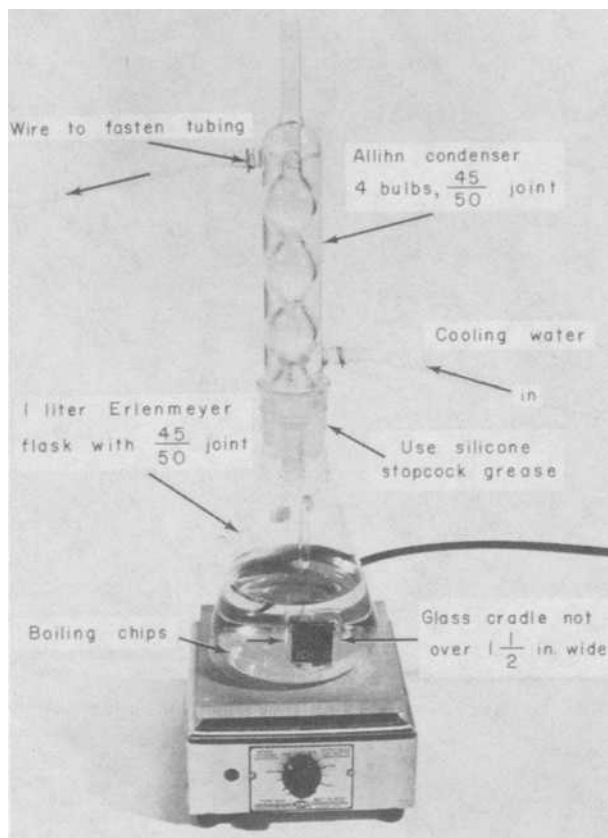


FIG. 8 Apparatus for Ferric Sulfate-Sulfuric Acid Test

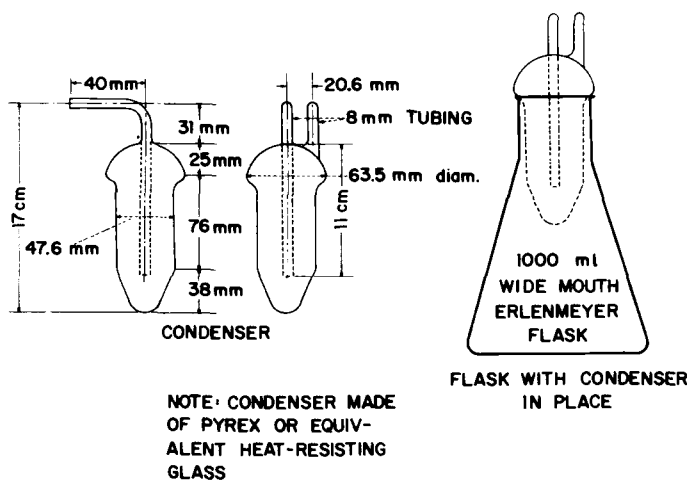


FIG. 9 Flask and Condenser for Nitric Acid Test

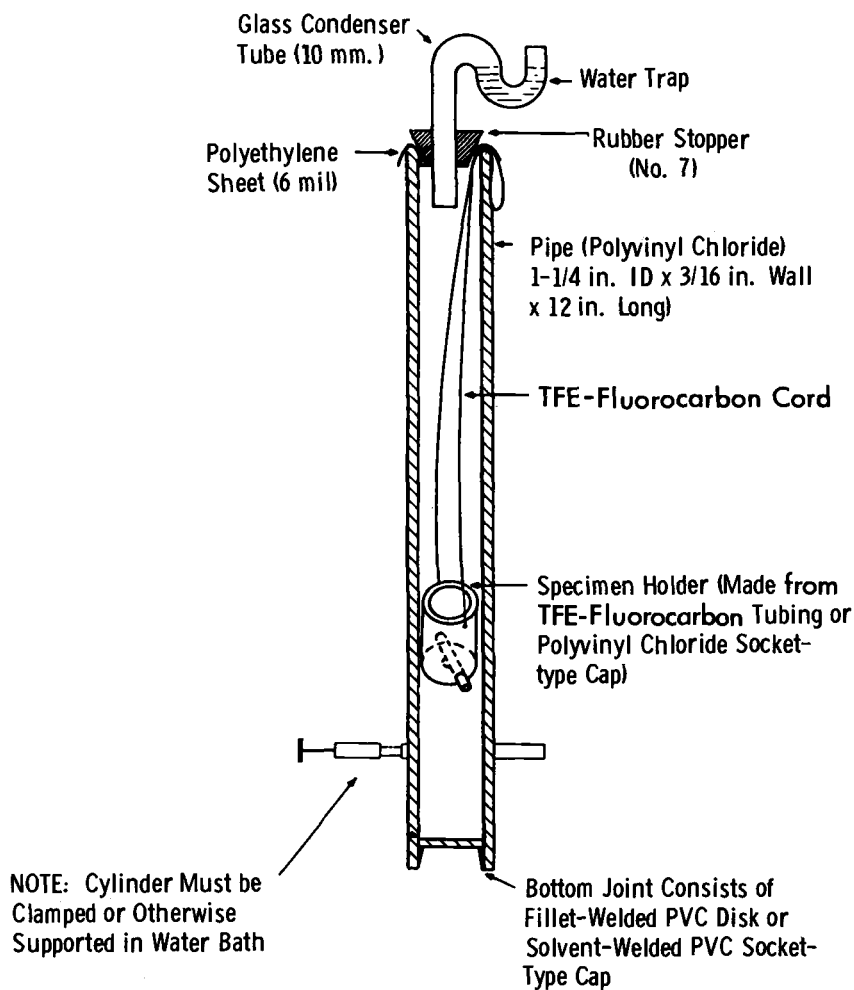


FIG. 10 Apparatus for Nitric-Hydrofluoric Acid Test

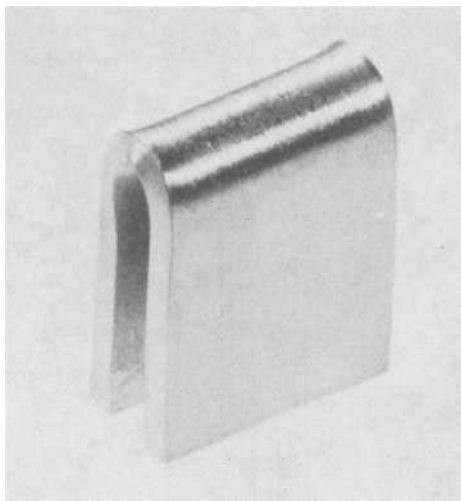


FIG. 11 A Bent Copper-Copper Sulfate-Sulfuric Acid Test Specimen

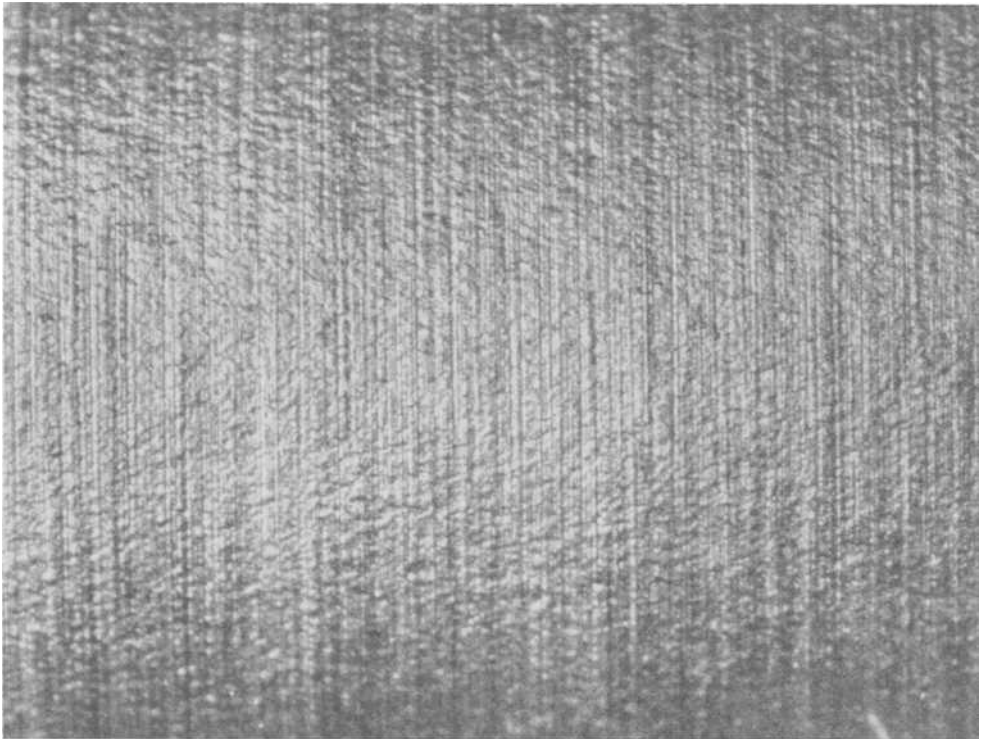


FIG. 12 Passing Test Specimen—View of the Bent Area (20× magnification before reproduction)

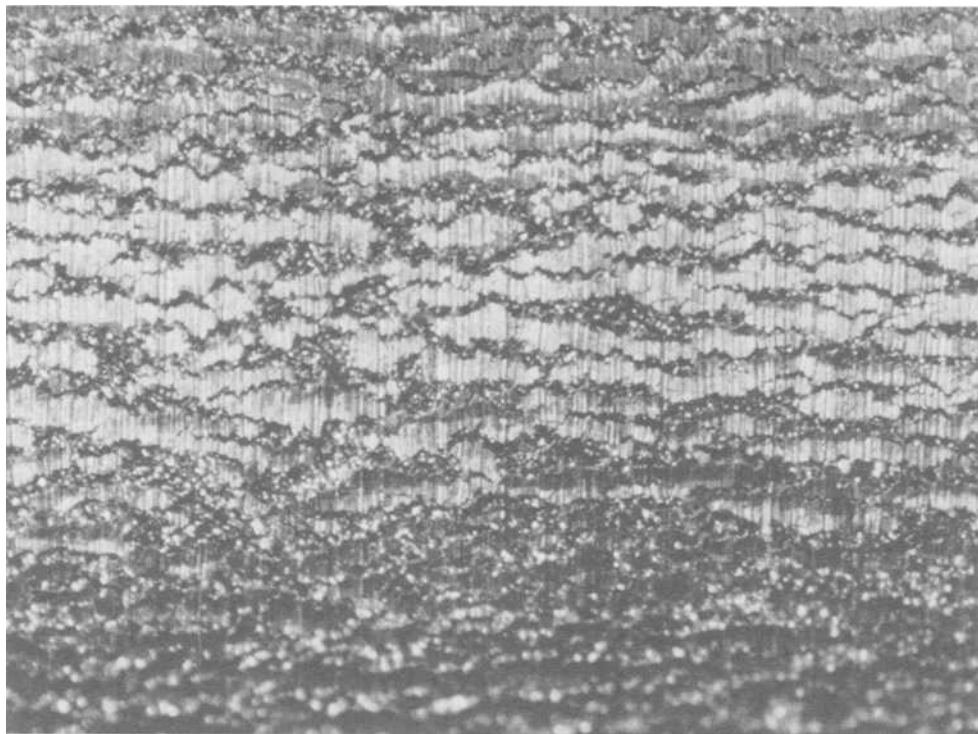


FIG. 13 Failing Test Specimen (Note the many intergranular fissures. Bent area at 20× magnification before reproduction.)

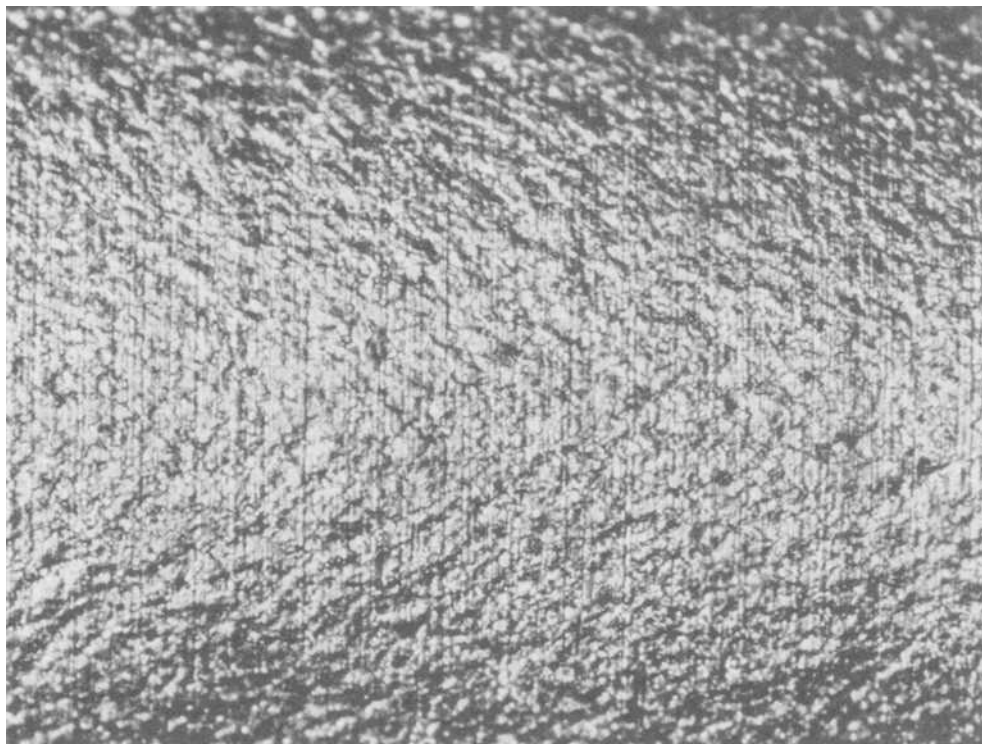


FIG. 14 Doubtful Test Result (Note the traces of intergranular fissures and "orange-peel" surface. Bent area at 20 $\times$ magnification before reproduction.)

REFERENCES

- (1) For original descriptions of the use of etch structure classifications, see Streicher, M. A., "Screening Stainless Steels from the 240-h Nitric Acid Test by Electrolytic Etching in Oxalic Acid," *ASTM Bulletin*, No. 188, February 1953, p. 35; also "Results of Cooperative Testing Program for the Evaluation of the Oxalic Acid Etch Test," *ASTM Bulletin*, No. 195, January 1954, p. 63.
- (2) For original description of ferric sulfate-sulfuric acid test, see Streicher, M. A., "Intergranular Corrosion Resistance of Austenitic Stainless Steels: A Ferric Sulfate-Sulfuric Acid Test," *ASTM Bulletin*, No. 229, April 1958, (STP 95), pp. 77-86.
- (3) For original descriptions of the boiling nitric acid test, see Huey, W. R., "Corrosion Test for Research and Inspection of Alloys," *Transactions, Am. Soc. Steel Treating*, Vol 18, 1930, p. 1126; also, "Report of Subcommittee IV on Methods of Corrosion Testing," *Proceedings, ASTM*, Vol 33, Part I, 1933, p. 187.
- (4) For details, see DeLong, W. B., "Testing Multiple Specimens of Stainless Steels in a Modified Boiling Nitric Acid Test Apparatus," *Symposium on Evaluation Tests for Stainless Steels, ASTM STP 93*, ASTM, 1950, p. 211.
- (5) See *Industrial and Engineering Chemistry*, Vol 17, 1925, p. 756; also, "A.C.S. Analytical Reagents. Specifications Recommended by Committee on Analytical Reagents," *Am. Chem. Soc.*, March 1941.
- (6) For original description of 10 % nitric-3 % hydrofluoric acid test, see Warren, D., "Nitric-Hydrofluoric Acid Evaluation Test for Type 316L Stainless Steel," *ASTM Bulletin*, No. 230, May 1958, pp. 45-56.
- (7) The use of copper to accelerate the intergranular corrosion of sensitized austenitic stainless steels in copper sulfate-sulfuric acid was first described by H. J. Rocha in the discussion of a paper by Brauns, E., and Pier, G., *Stahl und Eisen*, Vol 75, 1955, p. 579.
- (8) For original evaluation of the copper-copper sulfate-sulfuric acid test, see Scharfstein, L. R., and Eisenbrown, C. M., "An Evaluation of Accelerated Strauss Testing," *ASTM STP 369*, ASTM, 1963, pp. 235-239.
- (9) Subtle effects due to variations in copper surface areas, galvanic contact, condenser design, etc., are described by Herbsleb, G., and Schwenk, W., "Untersuchungen zur Einstellung des Redoxpotentials der Straussschen Lösung mit Zusatz von Metallischem Kupfer," *Corrosion Science*, Vol 7,

1967, pp. 501–511.

- (10) Brown, M. H., “Behavior of Austenitic Stainless Steels in Evaluation Tests for the Detection of Susceptibility to Intergranular Corrosion,” *Corrosion*, Vol 30, January 1974, pp. 1–12.
- (11) Streicher, M. A., “Theory and Application of Evaluation Tests for Detecting Susceptibility to Intergranular Attack in Stainless Steels and Related Alloys-Problems and Opportunities,” *Inter-*

granular Corrosion of Stainless Alloys, ASTM STP 656, R. F. Steigerwald ed, ASTM, 1978, 3–84.

- (12) Walker, W. L., “Variations in the Evaluation of ASTM A262, Practice E, Results (ASTM Subcommittee A01.14 Round Robin),” *Intergranular Corrosion of Stainless Alloys*, ASTM STP 656, R. F. Steigerwald ed; 1978, pp. 146–153.

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Standard Method of SALT SPRAY (FOG) TESTING¹

This standard is issued under the fixed designation B 117; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This method has been approved for use by agencies of the Department of Defense to replace Method 811.1 of Federal Test Method Standard No. 151b and for listing in DoD Index of Specifications and Standards.

1. Scope

1.1 This method sets forth the conditions required in salt spray (fog) testing for specification purposes. Suitable apparatus which may be used to obtain these conditions is described in Appendix X1. The method does *not* prescribe the type of test specimen or exposure periods to be used for a specific product, nor the interpretation to be given to the results. It should be noted that there is seldom a direct relation between salt spray (fog) resistance and resistance to corrosion in other media, because the chemistry of the reactions, including the formation of films and their protective value, frequently varies greatly with the precise conditions encountered. Comments on the use of the test in research will be found in Appendix X2.

NOTE 1—This method is applicable to salt spray (fog) testing of ferrous and non-ferrous metals, and is also used to test inorganic and organic coatings, etc., especially where such tests are the basis for material or product specifications.

1.2 The values stated in SI units are to be regarded as standard. The inch-pound units in parentheses are provided for information.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Applicable Documents

2.1 ASTM Standards:

B 287 Method of Acetic Acid-Salt Spray (Fog) Testing²

B 368 Method for Copper-Accelerated Acetic Acid-Salt Spray (Fog) Testing (CASS Test)³

D 609 Method for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products⁴

D 1193 Specification for Reagent Water⁵

D 1654 Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments⁴

E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode⁶

3. Apparatus

3.1 The apparatus required for salt spray (fog) testing consists of a fog chamber, a salt solution reservoir, a supply of suitably conditioned compressed air, one or more atomizing nozzles, specimen supports, provision for heating the chamber, and necessary means of control. The size and detailed construction of the apparatus are optional, provided the conditions obtained meet the requirements of this method.

3.2 Drops of solution which accumulate on the ceiling or cover of the chamber shall not be permitted to fall on the specimens being tested.

¹ This method is under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals, and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.

Current edition approved April 26, 1985. Published August 1985. Originally published as B 117 – 39 T. Last previous edition B 117 – 64.

² *Annual Book of ASTM Standards*, Vols 02.05 and 03.02.

³ *Annual Book of ASTM Standards*, Vol 02.05.

⁴ *Annual Book of ASTM Standards*, Vol 06.01.

⁵ *Annual Book of ASTM Standards*, Vol 11.01.

⁶ *Annual Book of ASTM Standards*, Vol 15.05.

3.3 Drops of solution which fall from the specimens shall not be returned to the solution reservoir for respraying.

3.4 Material of construction shall be such that it will not affect the corrosiveness of the fog.

4. Test Specimens

4.1 The type and number of test specimens to be used, as well as the criteria for the evaluation of the test results, shall be defined in the specifications covering the material or product being tested or shall be mutually agreed upon by the purchaser and the seller.

5. Preparation of Test Specimens

5.1 Metallic and metallic-coated specimens shall be suitably cleaned. The cleaning method shall be optional depending on the nature of the surface and the contaminants, except that it shall not include the use of abrasives other than a paste of pure magnesium oxide nor of solvents which are corrosive or will deposit either corrosive or protective films. The use of a nitric acid solution for the chemical cleaning, or passivation, of stainless steel specimens is permissible when agreed upon by the purchaser and the seller. Care shall be taken that specimens are not recontaminated after cleaning by excessive or careless handling.

5.2 Specimens for evaluation of paints and other organic coatings shall be prepared in accordance with applicable specification(s) for the material(s) being tested, or as agreed upon by the purchaser and supplier. Otherwise, the test specimens shall consist of steel meeting the requirements of Methods D 609 and shall be cleaned and prepared for coating in accordance with applicable procedure of Method D 609.

5.3 Specimens coated with paints or nonmetallic coatings shall not be cleaned or handled excessively prior to test.

5.4 Whenever it is desired to determine the development of corrosion from an abraded area in the paint or organic coating, a scratch or scribed line shall be made through the coating with a sharp instrument so as to expose the underlying metal before testing. The conditions of making the scratch shall be as defined in Method D 1654, unless otherwise agreed upon between the purchaser and seller.

5.5 Unless otherwise specified, the cut edges of plated, coated, or duplex materials and areas

containing identification marks or in contact with the racks or supports shall be protected with a suitable coating stable under the conditions of the test, such as ceresin wax.

NOTE 2—Should it be desirable to cut test specimens from parts or from preplated, painted, or otherwise coated steel sheet, the cut edges shall be protected by coating them with paint, wax, tape, or other effective media so that the development of a galvanic effect between such edges and the adjacent plated or otherwise coated metal surfaces, is prevented.

6. Position of Specimens During Test

6.1 The position of the specimens in the salt spray chamber during the test shall be such that the following conditions are met:

6.1.1 Unless otherwise specified, the specimens shall be supported or suspended between 15 and 30° from the vertical and preferably parallel to the principal direction of horizontal flow of fog through the chamber, based upon the dominant surface being tested.

6.1.2 The specimens shall not contact each other or any metallic material or any material capable of acting as a wick.

6.1.3 Each specimen shall be so placed as to permit free settling of fog on all specimens.

6.1.4 Salt solution from one specimen shall not drip on any other specimen.

NOTE 3—Suitable materials for the construction or coating of racks and supports are glass, rubber, plastic, or suitably coated wood. Bare metal shall not be used. Specimens shall preferably be supported from the bottom or the side. Slotted wooden strips are suitable for the support of flat panels. Suspension from glass hooks or waxed string may be used as long as the specified position of the specimens is obtained, if necessary by means of secondary support at the bottom of the specimens.

7. Salt Solution

7.1 The salt solution shall be prepared by dissolving 5 ± 1 parts by weight of sodium chloride in 95 parts of water conforming to Type IV water in Specification D 1193. The salt used shall be sodium chloride substantially free of nickel and copper and containing on the dry basis not more than 0.1 % of sodium iodide and not more than 0.3 % of total impurities. Some salts contain additives that may act as corrosion inhibitors; careful attention should be given to the chemical content of the salt. Upon agreement between purchaser and seller, analysis may be required and limits established for elements or com-

pounds not specified in the chemical composition given above.

7.2 The pH of the salt solution shall be such that when atomized at 35°C (95°F) the collected solution will be in the pH range of 6.5 to 7.2 (Note 4). Before the solution is atomized it shall be free of suspended solids (Note 5). The pH measurement shall be made electrometrically at 25°C (77°F) using a glass electrode with a saturated potassium chloride bridge in accordance with Method E 70, or colorimetrically using bromothymol blue as indicator, or short range pH paper which reads in 0.2 or 0.3 of a pH unit (Note 6).

NOTE 4—Temperature affects the pH of a salt solution prepared from water saturated with carbon dioxide at room temperature and pH adjustment may be made by the following three methods:

(1) When the pH of a salt solution is adjusted at room temperature, and atomized at 35°C (95°F), the pH of the collected solution will be higher than the original solution due to the loss of carbon dioxide at the higher temperature. When the pH of the salt solution is adjusted at room temperature, it is therefore necessary to adjust it below 6.5 so the collected solution after atomizing at 35°C (95°F) will meet the pH limits of 6.5 to 7.2. Take about a 50-mL sample of the salt solution as prepared at room temperature, boil gently for 30 s, cool, and determine the pH. When the pH of the salt solution is adjusted to 6.5 to 7.2 by this procedure, the pH of the atomized and collected solution at 35°C (95°F) will come within this range.

(2) Heating the salt solution to boiling and cooling to 95°F for maintaining it at 95°F for approximately 48 h before adjusting the pH produces a solution the pH of which does not materially change when atomized at 35°C (95°F).

(3) Heating the water from which the salt solution is prepared to 35°C (95°F) or above, to expel carbon dioxide, and adjusting the pH of the salt solution within the limits of 6.5 to 7.2 produces a solution the pH of which does not materially change when atomized at 35°C (95°F).

NOTE 5—The freshly prepared salt solution may be filtered or decanted before it is placed in the reservoir, or the end of the tube leading from the solution to the atomizer may be covered with a double layer of cheesecloth to prevent plugging of the nozzle.

NOTE 6—The pH can be adjusted by additions of dilute cp hydrochloric acid or cp sodium hydroxide solutions.

8. Air Supply

8.1 The compressed air supply to the nozzle or nozzles for atomizing the salt solution shall be free of oil and dirt (Note 7) and maintained between 69 and 172 kN/m² (10 and 25 psi) (Note 8).

NOTE 7—The air supply may be freed from oil and dirt by passing it through a water scrubber or at least 610 mm (2 ft) of suitable cleaning material such as sheep's wool, excelsior, slag wool, or activated⁷ alumina.

NOTE 8—Atomizing nozzles may have a "critical pressure" at which an abnormal increase in the corrosiveness of the salt fog occurs. If the "critical pressure" of a nozzle has not been established with certainty, control of fluctuation in the air pressure within plus or minus 0.7 kN/m² (0.1 psi), by installation of a suitable pressure regulator valve⁸ minimizes the possibility that the nozzle will be operated at its "critical pressure."⁹

9. Conditions in the Salt Spray Chamber

9.1 *Temperature*—The exposure zone of the salt spray chamber shall be maintained at 35 + 1.1 – 1.7°C (95 + 2 – 3°F). The temperature within the exposure zone of the closed cabinet shall be recorded at least twice a day at least 7 h apart (except on Saturdays, Sundays, and holidays when the salt spray test is not interrupted for exposing, rearranging, or removing test specimens or to check and replenish the solution in the reservoir).

NOTE 9—A suitable method to record the temperature is by a continuous recording device or by a thermometer which can be read from outside the closed cabinet. The recorded temperature must be obtained with the salt spray chamber closed to avoid a false low reading because of wet-bulb effect when the chamber is open.

9.2. *Atomization and Quantity of Fog*—At least two clean fog collectors shall be so placed within the exposure zone that no drops of solution from the test specimens or any other source shall be collected. The collectors shall be placed in the proximity of the test specimens, one nearest to any nozzle and the other farthest from all nozzles. The fog shall be such that for each 80 cm² of horizontal collecting area there will be collected in each collector from 1.0 to 2.0 mL of solution per hour based on an average run of at least 16 h (Note 10). The sodium chloride concentration of the collected solution shall be 5 ±

⁷ Registered U. S. Patent Office.

⁸ The Nullmatic pressure regulator (or equivalent) manufactured by Moore Products Co., H and Lycoming Sts., Philadelphia, PA 19124, is suitable for this purpose.

⁹ It has been observed that periodic fluctuations in air pressure of ±3.4 kN/m² (0.5 psi) resulted in about a twofold increase in the corrosivity of the fog from a nozzle which was being operated at an average pressure of 110 kN/m² (16 psi). Controlling the fluctuations within ±0.7 kN/m² (0.1 psi), however, avoided any increase in the corrosivity of the salt fog. See Darsey, V. M. and Cavanagh, W. R., "Apparatus and Factors in Salt Fog Testing," *Proceedings, ASTM*, Vol 48, 1948, p. 153.

1 weight % (Note 11). The pH of the collected solution shall be 6.5 to 7.2. The pH measurement shall be made electrometrically or colorimetrically using bromothymol blue as the indicator.

NOTE 10—Suitable collecting devices are glass funnels with the stems inserted through stoppers into graduated cylinders, or crystallizing dishes. Funnels and dishes with a diameter of 10 cm have an area of about 80 cm².

NOTE 11—A solution having a specific gravity of 1.0255 to 1.0400 at 25°C (77°F) will meet the concentration requirement. The concentration may also be determined as follows: Dilute 5 mL of the collected solution to 100 mL with distilled water and mix thoroughly; pipet a 10-mL aliquot into an evaporating dish or casserole; add 40 mL of distilled water and 1 mL of 1 % potassium chromate solution (chloride-free) and titrate with 0.1 N silver nitrate solution to the first appearance of a permanent red coloration. A solution that requires between 3.4 and 5.1 mL of 0.1 N silver nitrate solution will meet the concentration requirements.

9.3 The nozzle or nozzles shall be so directed or baffled that none of the spray can impinge directly on the test specimens.

10. Continuity of Test

10.1 Unless otherwise specified in the specifications covering the material or product being tested, the test shall be continuous for the duration of the entire test period. Continuous operation implies that the chamber be closed and the spray operating continuously except for the short daily interruptions necessary to inspect, rearrange, or remove test specimens; to check and replenish the solution in the reservoir, and to make necessary recordings as described in Section 9. Operations shall be so scheduled that these interruptions are held to a minimum.

11. Period of Test

11.1 The period of test shall be as designated by the specifications covering the material or product being tested or as mutually agreed upon between the purchaser and the seller.

NOTE 12—Recommended exposure periods are to be as agreed upon by the purchaser and seller, but exposure periods of multiples of 24 h are suggested.

12. Cleaning of Tested Specimens

12.1 Unless otherwise specified in the specifications covering the material or product being tested, specimens shall be treated as follows at the end of the test:

12.1.1 The specimens shall be carefully re-

moved.

12.2 Specimens may be gently washed or dipped in clean running water not warmer than 38°C (100°F) to remove salt deposits from their surface, and then immediately dried. Drying shall be accomplished with a stream of clean, compressed air.

13. Evaluation of Results

13.1 A careful and immediate examination shall be made for the extent of corrosion of the dry test specimens or for other failure as required by the specifications covering the material or product being tested or by agreement between the purchaser and the seller.

14. Records and Reports

14.1 The following information shall be recorded, unless otherwise prescribed in the specifications covering the material or product being tested:

14.1.1 Type of salt and water used in preparing the salt solution,

14.1.2 All readings of temperature within the exposure zone of the chamber,

14.1.3 Daily records of data obtained from each fog-collecting device including the following:

14.1.3.1 Volume of salt solution collected in milliliters per hour per 80 cm²,

14.1.3.2 Concentration or specific gravity at 35°C (95°F) of solution collected, and

14.1.3.3 pH of collected solution.

14.4 Type of specimen and its dimensions, or number or description of part,

14.5 Method of cleaning specimens before and after testing,

14.6 Method of supporting or suspending article in the salt spray chamber,

14.7 Description of protection used as required in 5.5,

14.8 Exposure period,

14.9 Interruptions in test, cause and length of time, and

14.10 Results of all inspections.

NOTE 13—If any of the atomized salt solution which has not contacted the test specimens is returned to the reservoir, it is advisable to record the concentration or specific gravity of this solution also.

15. Precision and Bias

15.1 The reproducibility of results in the salt spray test is highly dependent on the type of

specimens tested and the evaluation criteria selected as well as the control of the operating variables. In any testing program, sufficient replicates should be included to establish the variability of the results. Substantial variability is often observed when similar specimens are tested in different fog chambers even though the testing conditions are nominally similar and within the ranges specified in this method.

15.2 The salt spray (fog) test is intended to reproduce the corrosion that occurs in atmos-

pheres containing salt spray or splash. It has been widely observed, however, that rankings of different alloys or coating systems, or both, do not necessarily fall in the same order as atmospheric tests in marine or road salt splash environments. This test has been more useful in rating the relative resistance of a specific type of protective coating, for example, hot-dip zinc coatings on steel. Interpretation of the results of this method beyond this purpose must be verified by actual exposure tests.

APPENDIXES

(Nonmandatory Information)

XI. CONSTRUCTION OF APPARATUS

X1.1 Cabinets

X1.1.1 Standard salt spray cabinets are available from several suppliers, but certain pertinent accessories are required before they will function according to this method and provide consistent control for duplication of results.

X1.1.2 The salt spray cabinet consists of the basic chamber, an air-saturator tower, a salt solution reservoir, atomizing nozzles, specimen supports, provisions for heating the chamber, and suitable controls for maintaining the desired temperature.

X1.1.3 Accessories such as a suitable adjustable baffle or central fog tower, automatic level control for the salt reservoir, and automatic level control for the air-saturator tower are pertinent parts of the apparatus.

X1.1.4 The cabinet should be of sufficient size to test adequately the desired number of parts without overcrowding. Small cabinets have been found difficult to control and those of less than 0.43-m³ (15-ft³) capacity should be avoided.

X1.1.5 The chamber may be made of inert materials such as plastic, glass, or stone, but most preferably is constructed of metal and lined with impervious plastics, rubber, or epoxy-type materials or equivalent.

X1.2 Temperature Control

X1.2.1 The maintenance of temperature within the salt chamber can be accomplished by several methods. It is generally desirable to control the temperature of the surroundings of the salt spray chamber and to maintain it as stable as possible. This may be accomplished by placing the apparatus in a constant-temperature room, but may also be achieved by surrounding the basic chamber of a jacket containing water or air at a controlled temperature.

X1.2.2 The use of immersion heaters in an internal salt solution reservoir or of heaters within the chamber is detrimental where heat losses are appreciable, because of solution evaporation and radiant heat on the specimens.

X1.2.3 All piping which contacts the salt solution or spray should be of inert materials such as plastic. Vent piping should be of sufficient size so that a minimum of back pressure exists and should be installed so that no solution is trapped. The exposed end of the vent pipe should be shielded from extreme air currents that may cause fluctuation of pressure or vacuum in the cabinet.

X1.3 Spray Nozzles

X1.3.1 Satisfactory nozzles may be made of hard rubber, plastic, or other inert materials. The most commonly used type is made of plastic. Nozzles calibrated for air consumption and solution atomized are available. The operating characteristics of a typical nozzle are given in Table X1.1.

X1.3.2 It can readily be seen that air consumption is relatively stable at the pressures normally used, but a marked reduction in solution sprayed occurs if the level of the solution is allowed to drop appreciably during the test. Thus, the level of the solution in the salt reservoir must be maintained automatically to ensure uniform fog delivery during the test.¹⁰

X1.3.3 If the nozzle selected does not atomize the salt solution into uniform droplets, it will be necessary to direct the spray at a baffle or wall to pick up the larger drops and prevent them from impinging on the test specimens. Pending a complete understanding of air-pressure effects, etc., it is important that the nozzle selected shall produce the desired condition when operated at the air pressure selected. Nozzles are not necessarily located at one end, but may be placed in the center and can also be directed vertically up through a suitable tower.

¹⁰ A suitable device for maintaining the level of liquid in, either the saturator tower, or reservoir of test solution may be designed by a local engineering group, or may be purchased from manufacturers of test cabinets as an accessory.

X1.4 Air for Atomization

X1.4.1 The air used for atomization must be free of grease, oil, and dirt before use by passing through well-maintained filters. Room air may be compressed, heated, humidified, and washed in a water-sealed rotary pump, if the temperature of the water is suitably controlled. Otherwise cleaned air may be introduced into the bottom of a tower filled with water, through a porous stone or multiple nozzles. The level of the water must be maintained automatically to ensure adequate humidification. A chamber operated according to this method and Appendix will have a relative humidity between 95 and 98 %. Since salt solutions from 2 to 6 % will give the same results (though for uniformity the limits are set at 4 to 6 %), it is preferable to saturate the air at temperatures well above the chamber temperature as insurance of a wet fog. Table X1.2 shows the temperatures, at different pressures, that are required to offset the cooling effect of expansion to atmospheric pressure.

X1.4.2 Experience has shown that most uniform

spray chamber atmospheres are obtained by increasing the atomizing air temperature sufficiently to offset heat losses, except those that can be replaced otherwise at very low-temperature gradients.

X1.5 Types of Construction

X1.5.1 A modern laboratory cabinet is shown in Fig. X1.1. Walk-in chambers are not usually constructed with a sloping ceiling due to their size and location. Suitably located and directed spray nozzles avoid ceiling accumulation and drip. Nozzles may be located at the ceiling, or 0.91 m (3 ft) from the floor directed upward at 30 to 60° over a passageway. The number of nozzles depends on type and capacity and is related to the area of the test space. A 11 to 19-dm³ (3 to 5-gal) reservoir is required within the chamber, with the level controlled. The major features of a walk-in type cabinet, which differs significantly from the laboratory type, are illustrated in Fig. X1.2. Construction of a plastic nozzle, such as is furnished by several suppliers, is shown in Fig. X1.3.

TABLE X1.1 Operating Characteristics of Typical Spray Nozzle

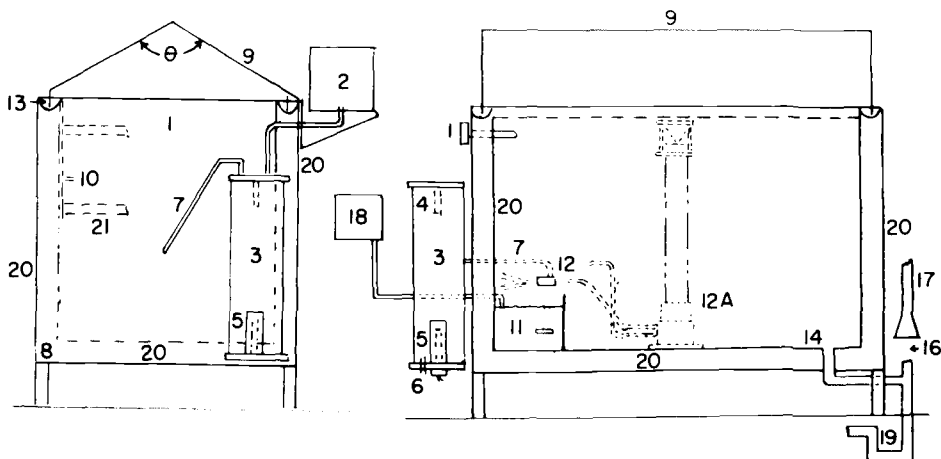
Siphon Height, in.	Air Flow, L/min				Solution Consumption, mL/h			
	Air Pressure, psi				Air Pressure, psi			
	5	10	15	20	5	10	15	20
4	19	26.5	31.5	36	2100	3840	4584	5256
8	19	26.5	31.5	36	636	2760	3720	4320
12	19	26.5	31.5	36	0	1380	3000	3710
16	19	26.6	31.5	36	0	780	2124	2904

Siphon Height cm	Air Flow, dm ³ /min				Solution Consumption, cm ³ /h			
	Air Pressure, kPa				Air Pressure, kPa			
	34	69	103	138	34	69	103	138
10	19	26.5	31.5	36	2100	3840	4584	5256
20	19	26.5	31.5	36	636	2760	3720	4320
30	19	26.5	31.5	36	0	1380	3000	3710
40	19	26.6	31.5	36	0	780	2124	2904

TABLE X1.2 Temperature and Pressure Requirements for Operation of Test at 95°F

	Air Pressure, psi			
	12	14	16	18
Temperature, °F	114	117	119	121

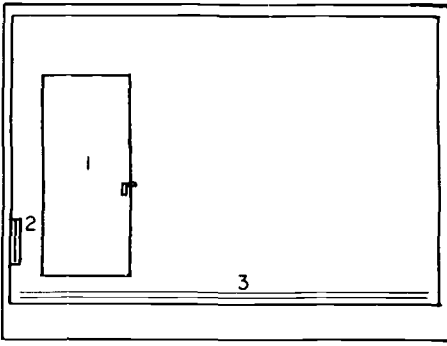
	Air Pressure, kPa			
	83	96	110	124
Temperature, °C	46	47	48	49



θ —Angle of lid, 90 to 125°

- 1—Thermometer and thermostat for controlling heater (Item No. 8) in base
- 2—Automatic water levelling device
- 3—Humidifying tower
- 4—Automatic temperature regulator for controlling heater (Item No. 5)
- 5—Immersion heater, non-rusting
- 6—Air inlet, multiple openings
- 7—Air tube to spray nozzle
- 8—Strip heater in base
- 9—Hinged top, hydraulically operated, or counterbalanced
- 10—Brackets for rods supporting specimens, or test table
- 11—Internal reservoir
- 12—Spray nozzle above reservoir, suitably designed, located, and baffled
- 12A—Spray nozzle housed in dispersion tower located preferably in center of cabinet
- 13—Water Seal
- 14—Combination drain and exhaust. Exhaust at opposite side of test space from spray nozzle (Item 12), but preferably in combination with drain, waste trap, and forced draft waste pipe (Items 16, 17, and 19).
- 16—Complete separation between forced draft waste pipe (Item 17) and combination drain and exhaust (Items 14 and 19) to avoid undesirable suction or back pressure.
- 17—Forced draft waste pipe.
- 18—Automatic levelling device for reservoir
- 19—Waste trap
- 20—Air space or water jacket
- 21—Test table or rack, well below roof area

FIG. X1.1 Typical Salt Spray Cabinet.



NOTE—The controls are the same, in general as for the laboratory cabinet (Fig. X1.1), but are sized to care for the larger cube. The chamber has the following features:

- (1) Heavy insulation,
- (2) Refrigeration door with drip rail, or pressure door with drip rail, inward-sloping sill,
- (3) Low-temperature auxiliary heater, and
- (4) Duck boards on floor, with floor sloped to combination drain and air exhaust.

FIG. X1.2 Walk-in Chamber, 1.5 by 2.4 m (5 by 8 ft) and Upward in Over-all Size

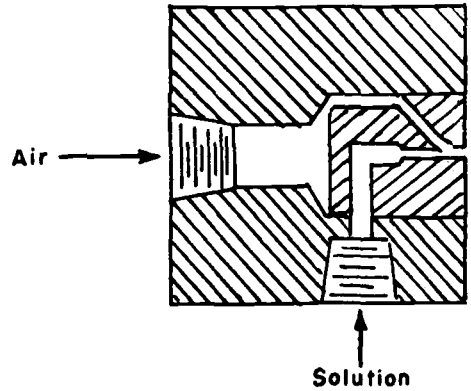


FIG. X1.3 Typical Spray Nozzle

X2. USE OF THE SALT SPRAY (FOG) TEST IN RESEARCH

X2.1 The detailed requirements of this method are primarily for quality acceptance and should not be construed as the optimum conditions for research studies. The test has been used to a considerable extent for the purpose of comparing different materials or finishes with an acceptable standard. The recent elimination of many cabinet variables and the improvement in controls have made the three ASTM Salt Spray Tests: Method B 117, B 287, and B 368, into useful tools for many industrial and military production and qualification programs.

X2.2 The test has been used to a considerable extent for the purpose of comparing different materials or finishes. It should be noted that there is seldom a direct relation between salt spray (fog) resistance and resistance to corrosion in other media, because the chemistry of the reactions, including the formation of films and their protective value, frequently varies greatly with the precise conditions encountered. Informed personnel are aware of the erratic composition of basic alloys, the possibility of wide variations in quality and thickness of plated items produced on the same racks at the same time, and the consequent need for a mathematical determination of the number of specimens required to constitute an adequate sample for test purposes. In this connection it is well to point out that Method B 117 is not applicable to the study or testing of decorative chromium plate (nickel-chromium or copper-nickel-

chromium) on steel or on zinc-base die castings or of cadmium plate on steel. For this purpose Methods B 287 and B 368 are available, which are also considered by some to be superior for comparison of chemically-treated aluminum (chromated, phosphated, or anodized), although final conclusions regarding the validity of test results related to service experience have not been reached. Method B 117 is considered to be most useful in estimating the relative behavior of closely related materials in marine atmospheres, since it simulates the basic conditions with some acceleration due to either wetness or temperature or both.

X2.3 When a test is used for research, it may prove advantageous to operate with a different solution composition or concentration or at a different temperature. In all cases, however, it is desirable to control the temperature and humidity in the manner specified, and to make certain that the composition of the settled fog and that of the solution in the reservoir are substantially the same. Where differences develop, it is necessary to control conditions so that the characteristics of the settled fog meet the specified requirements for the atmosphere.

X2.4 Material specifications should always be written in terms of the standard requirements of the appropriate salt spray method, thereby making it possible to test a variety of materials from different sources in the same equipment.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Practice for PREPARING, CLEANING, AND EVALUATING CORROSION TEST SPECIMENS¹

This standard is issued under the fixed designation G 1; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice gives suggested procedures for preparing bare, solid metal specimens for laboratory corrosion tests, for removing corrosion products after the test has been completed, and for evaluating the corrosion damage that has occurred. Emphasis is placed on procedures related to the evaluation of corrosion by mass-loss and pitting measurements.

NOTE 1: **Caution**—In many cases the corrosion product on the reactive metals titanium and zirconium is a hard and tightly bonded oxide that defies removal by chemical or ordinary mechanical means. In many such cases, corrosion rates are established by weight gain rather than weight loss.

2. Applicable Documents

2.1 *ASTM Standards*:

A 262 Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels²

D 1384 Method for Corrosion Test for Engine Coolants in Glassware³

G 31 Recommended Practice for Laboratory Immersion Corrosion Testing of Metals⁴

G 46 Recommended Practice for Examination and Evaluation of Pitting Corrosion⁴

3. Significance and Use

3.1 A significance and use statement is not applicable to this practice.

4. Sampling

4.1 The bulk sampling of products is outside the scope of this practice.

5. Methods for Preparing Specimens for Test

5.1 *Surface Condition*:

5.1.1 For laboratory corrosion tests that simulate exposure to service environments, a commercial surface, closely resembling the one that would be used in service, will yield the most significant results.

5.1.2 For more searching tests of either the metal or the environment, standard surface finishes may be preferred. A suitable procedure might be:

5.1.2.1 Degrease in an organic solvent or hot alkaline cleaner.

NOTE 2—Hot alkalies and chlorinated solvents may attack some metals.

NOTE 3—Ultrasonic cleaning may be beneficial in both pre-test and post-test cleaning procedures.

5.1.2.2 Pickle in an appropriate solution (in some cases the chemical cleaners described in Section 7 will suffice) if oxides or tarnish are present.

5.1.2.3 Abrade with a slurry of an appropriate abrasive or with an abrasive paper (see Practices A 262, D 1384, and G 31). The edges as well as the faces of the specimens should be abraded to remove burrs.

5.1.2.4 Rinse thoroughly and dry.

5.2 *Metallurgical Condition*—When speci-

¹ This practice is under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.

Current edition approved April 24, 1981. Published July 1981. Originally published as G 1 – 67. Last previous edition G 1 – 72 (1979).

² *Annual Book of ASTM Standards*, Vols 01.03 and 03.02.

³ *Annual Book of ASTM Standards*, Vol 15.05.

⁴ *Annual Book of ASTM Standards*, Vol 03.02.

men preparation changes the metallurgical condition of the metal, other methods should be chosen or the metallurgical condition must be corrected by subsequent treatment. For example, shearing a specimen to size will cold work and may possibly fracture the edges. Edges should be machined or the specimen annealed.

5.3 The clean, dry specimens should be measured and weighed. Dimensions determined to the third significant figure and mass determined to the fifth significant figure are suggested.

6. Method for Electrolytic Cleaning After Testing

6.1 Electrolytic cleaning is a satisfactory method for many common metals.

6.1.1 The following method is typical; after scrubbing to remove loosely attached corrosion products, electrolyze the specimen as follows:

Sulfuric acid (H_2SO_4 , sp gr 1.84)	28 ml
Organic inhibitor	2 ml (see Note 4)
Water to make	1 litre
Temperature	75 C (167 F)
Time	3 min
Anode	carbon or lead (see Note 5)
Cathode	test specimen
Current density	20 A/dm ²

NOTE 4—Instead of using 0.2 volume percent of any proprietary inhibitor, about 0.5 g/litre of such inhibitors as diorthotolyl thiourea, quinoline ethiodide, or betanaphthol quinoline may be used.

NOTE 5—If lead anodes are used, lead may deposit on the specimen and cause an error in the mass loss. If the specimen is resistant to nitric acid, the lead may be removed by a flash dip in 1 + 1 nitric acid. Except for this possible source of error, lead is preferred as an anode as it gives more efficient corrosion-product removal.

6.2 It should be noted that this electrolytic treatment may result in the redeposition of metal, such as copper, from reducible corrosion products and, thus, lower the apparent mass loss.

7. Methods for Chemical Cleaning After Testing

NOTE 6: **Caution**—These methods may be hazardous to personnel. They should not be carried out by the uninitiated or without professional supervision.

7.1 Copper and Nickel Alloys—Dip in:

Hydrochloric acid (HCl, sp gr 1.19)	500 ml
Water to make	1 litre
Temperature	room
Time	1 to 3 min

NOTE 7—To avoid metal loss during cleaning,

deacerate solution with purified nitrogen before cleaning specimens.

7.2 *Aluminum Alloys*—The following methods are suitable:

7.2.1 Dip in:

Chromic acid (CrO_3)	20 g
Phosphoric acid (H_3PO_4 , sp gr 1.69)	50 ml
Water to make	1 litre
Temperature	80°C (176°F)
Time	5 to 10 min, or until clean

7.2.1.1 If a film remains, dip in:

Nitric acid (HNO_3 , sp gr 1.42)	...
Temperature	room
Time	1 min

7.2.2 Dip in:

Nitric acid (HNO_3 , sp gr 1.42)	...
Temperature	room
Time	15 min, or until clean

NOTE 8: **Caution**—Remove extraneous deposits as well as bulky corrosion products before the HNO_3 treatment to avoid violent reactions that may result in attack of the aluminum alloy (see 7.9).

7.3 Tin Alloys—Dip in:

Trisodium phosphate (Na_3PO_4)	150 g
Water to make	1 litre
Temperature	boiling
Time	10 min

7.4 Lead Alloys—Suitable methods include:

7.4.1 Preferably, use the electrolytic cleaning procedure of Section 6.

7.4.2 Dip in:

Acetic acid (99.5 percent)	10 ml
Water to make	1 litre
Temperature	boiling
Time	5 min

7.4.3 Alternatively dip in:

Ammonium acetate	50 g
Water to make	1 litre
Temperature	hot
Time	5 min

7.4.3.1 This removes lead oxide (PbO) and lead sulfate ($PbSO_4$).

7.5 *Zinc*—The following methods are suitable:

7.5.1 Dip in:

Ammonium hydroxide (NH_4OH , sp gr 0.90)	150 ml
Water to make	1 litre
Temperature	room
Time	several minutes

7.5.2 Then dip in:

Chromic acid (CrO_3)	50 g
Silver nitrate ($AgNO_3$)	10 g
Water to make	1 litre
Temperature	boiling
Time	15 to 20 s

NOTE 9—In making up the chromic acid solution, it is advisable to dissolve the silver nitrate separately and add it to the boiling chromic acid to prevent excessive crystallization of the silver chromate. The chromic acid must be free from sulfate to avoid attack on the zinc.

7.5.2 Dip in:

Hydriodic acid (HI, sp gr 1.5)	85 ml
Water to make	1 litre
Temperature	room
Time	15 s

7.5.2.1 This procedure dissolves a little zinc and corrections must be made as noted in 8.1.

7.5.6 Magnesium Alloys—Dip in:

Chromic acid (CrO_3)	150 g
Silver chromate (Ag_2CrO_4)	10 g
Water to make	1 litre
Temperature	boiling
Time	1 min

7.7 Iron and Steel—The following methods are suitable:

7.7.1 The hot sodium hydride method is excellent for cleaning iron and steel both from the point of view of ease of removal of corrosion products and minimum attack on the metal.⁵ Because of the hazard involved and the somewhat more sophisticated equipment required, other methods may be preferred. An alternative choice is electrolytic cleaning (see Section 6).

7.7.2 Dip in Clarke's solution:

Hydrochloric acid (HCl, sp gr 1.19)	1 litre
Antimony trioxide (Sb_2O_3)	20 g
Stannous chloride (SnCl_2)	50 g
Temperature	room
Time	up to 25 min

7.7.2.1 Solution should be vigorously stirred or the specimen should be rubbed with a non-abrasive implement of wood or rubber.

7.7.3 Dip in:

Sulfuric acid (H_2SO_4 , sp gr 1.84)	100 ml
Organic inhibitor	1.5 ml
Water to make	1 litre
Temperature	50 C (120 F)

7.8 Stainless Steels:

7.8.1 Methods in 7.7.1 are also applicable.

7.8.2 Dip in:

Nitric acid (HNO_3 , sp gr 1.42)	100 ml
Water to make	1 litre
Temperature	60 C (140 F)
Time	20 min

7.8.3 Alternatively dip in:

Ammonium citrate	150 g
Water to make	1 litre

Temperature
Time

70 C (158 F)
10 to 60 min

7.9 Mechanical cleaning in some cases may be used in place of, or as supplement to, chemical treatments to remove bulky adherent deposits. A relatively soft scraper or stiff fiber bristle brush should be used to avoid scratching or removal of metal from the surface of the specimen. However, this method may not remove all the products from pits.

NOTE 10—Such vigorous mechanical cleaning is applicable when mass losses are large and hence errors in mass loss will produce only small errors in corrosion rates. Blank corrections will be difficult to apply.

7.10 In all the foregoing methods, specimens should be rinsed following cleaning and scrubbed lightly with a bristle brush under running water. The cleaning dip may be repeated as necessary. After the final rinse, specimens should be dried and weighed.

8. Calculation of Corrosion Rate

8.1 Whatever cleaning method is used, the possibility of removal of solid metal is present; this results in error in the determination of the corrosion rate. To check this, one or more cleaned and weighed specimens may be re-cleaned by the same method and reweighed. Loss due to this second weighing may be used as an approximate correction to the first one (see Appendix X2 for a more exact method).

8.2 The initial total surface area of the specimen (making allowances for the change in area due to mounting holes) and the mass lost during the test are determined. The average corrosion rate may then be obtained as follows:

$$\text{Corrosion rate} = (K \times W)/(A \times T \times D)$$

where:

K = a constant (see 8.2.1),

T = time of exposure in hours to the nearest 0.01 h,

A = area in cm^2 to the nearest 0.01 cm^2 ,

W = mass loss in g, to nearest 1 mg, and

D = density in g/cm^3 (see Appendix X1).

8.2.1 Many different units are used to express corrosion rates. Using the above units for T , A , W , and D the corrosion rate can be

⁵ Technical Information Bulletin SP29-370 "DuPont Sodium Hydride Descaling Process Operating Instructions," available from E. I. duPont de Nemours & Co. (Inc.), Electrochemicals Dept., Wilmington, Del. 19898.

calculated in a variety of units with the following appropriate value of *K*:

Corrosion Rate Units Desired	Constant (<i>K</i>) in Corrosion Rate Equation
mils per year (mpy)	3.45×10^6
inches per year (ipy)	3.45×10^3
inches per month (ipm)	2.87×10^2
millimetres per year (mm/y)	8.76×10^4
micrometres per year ($\mu\text{m/y}$)	8.76×10^7
picometres per second (pm/s)	2.78×10^6
grams per square metre per hour ($\text{g/m}^2 \cdot \text{h}$)	$1.00 \times 10^4 \times D^A$
milligrams per square decimetre per day (mdd)	$2.40 \times 10^6 \times D^A$
micrograms per square metre per second ($\mu\text{g/m}^2 \cdot \text{s}$)	$2.78 \times 10^6 \times D^A$

^A Density is not needed to calculate the corrosion rate in these units; the density in the constant *K* cancels out the density in the corrosion rate equation.

NOTE 11—If desired, these constants may also be used to convert corrosion rates from one set of units to another. To convert a corrosion rate in units *X* to a rate in units *Y*, multiple by K_Y/K_X for example:

$$15 \text{ mpy} = 15 \times (2.78 \times 10^6)/(3.45 \times 10^6) \text{ pm/s}$$

8.3 Corrosion rates calculated from mass losses can be misleading when deterioration is highly localized, as in pitting or crevice corrosion. If corrosion is in the form of pitting, it may be measured with a depth gage or micrometer calipers with pointed anvils (see Recommended Practice G 46). Microscopical methods will determine pit depth by focusing from top to bottom of the pit, when it is viewed from above (using a calibrated focusing knob) or by examining a section that has been mounted and metallographically polished. The pitting factor is the ratio of the deepest metal penetration to

the average metal penetration (as measured by mass loss).

8.4 Other methods of assessing corrosion damage are:

8.4.1 *Appearance*—The degradation of appearance by rusting, tarnishing, or oxidation.

8.4.2 *Mechanical Properties*—An apparent loss in tensile strength will result if the cross-sectional area of the specimen (measured before exposure to the corrosive environment) is reduced by corrosion. Loss in tensile strength will result if a metasomatic change, such as parting has taken place. Loss in tensile strength and elongation will result from localized attack, such as cracking.

8.4.3 *Electrical Properties*—Loss in apparent conductivity will result from cracking or pitting.

8.4.4 *Microscopical Examination*—Parting, exfoliation, cracking, or intergranular attack may be measured by metallographic examination of suitably prepared sections.

9. Report

9.1 The report should include the compositions and sizes of specimens, their metallurgical conditions, surface preparations, and post-corrosion cleaning methods, as well as measures of corrosion damage such as corrosion rates (calculated from mass losses), maximum depths of pitting, or losses in mechanical properties.

10. Precision and Accuracy

10.1 A precision and accuracy statement cannot be made for this practice.

APPENDIXES

XI. DENSITIES FOR A VARIETY OF METALS AND ALLOYS

Aluminum Alloys	Density g/cm^3	Copper Alloys	Density g/cm^3
1100, 3004	2.72	Copper	8.94
1199, 5005, 5357, 6061, 6062, 6070, 6101	2.70	Brasses:	
2024	2.77	Commercial bronze 220	8.80
2219, 7178	2.81	Red brass 230	8.75
3003, 7079	2.74	Cartridge brass 260	8.52
5050	2.69	Muntz metal 280	8.39
5052, 5454	2.68	Admiralty 442, 443, 444, 445	8.52
5083, 5086, 5154, 5456	2.66	Aluminum brass 687	8.33
7075	2.80		

Aluminum Alloys	Density g/cm ³	Aluminum Alloys	Density g/cm ³
Bronzes:		Carpenter Stainless No. 20 Cb 3	8.05
Aluminum bronze, 5 percent 608	8.16	Lead	
Aluminum bronze, 8 percent 612	7.78	Antimonial	10.80
Composition M	8.45	Chemical	11.33
Composition G	8.77		
Phosphor bronze, 5 percent 510	8.86	Nickel Alloys	
Phosphor bronze, 10 percent 524	8.77	Nickel 200	8.89
85-5-5-5	8.80	Monel Alloy 400	8.84
Silicon bronze 655	8.52	Inconel Alloy 600	8.51
Copper nickels 706, 710, 715	8.94	Incoloy Alloy 825	8.14
Nickel silver 752	8.75	Illium G	8.31
Ferrous Metals		Hastelloy B	9.24
Gray cast iron	7.20	Hastelloy C	8.93
Carbon steel	7.86	Hastelloy G	8.27
Silicon iron	7.00		
Low alloy steels	7.85	Other Metals	
Stainless steels:		Magnesium	1.74
Types 201, 202, 302, 304, 304L, 321	7.94	Molybdenum	10.22
Types 309, 310, 311, 316, 316L, 317, 329, 330	7.98	Platinum	21.45
Type 347	8.03	Silver	10.49
Type 410	7.70	Tantalum	16.60
Type 430	7.72	Tin	7.30
Type 446	7.65	Titanium	4.54
Type 502	7.82	Zinc	7.13
Durimet 20	8.02	Zirconium	6.53

X2. METHOD FOR DETERMINING MASS LOSS WHERE CLEANING MAY ATTACK THE BASE METAL OF THE SPECIMEN

X2.1 Repeat the cleaning procedure a number of times. Weigh after each cleaning and plot the mass loss against the total time of cleaning or the number of cleanings, see Fig. X2.1. The ordinate at the inter-

section of the two lines is the mass loss caused by removal of corrosion products alone. The method is particularly applicable to electrolytic cleaning, see Section 6.

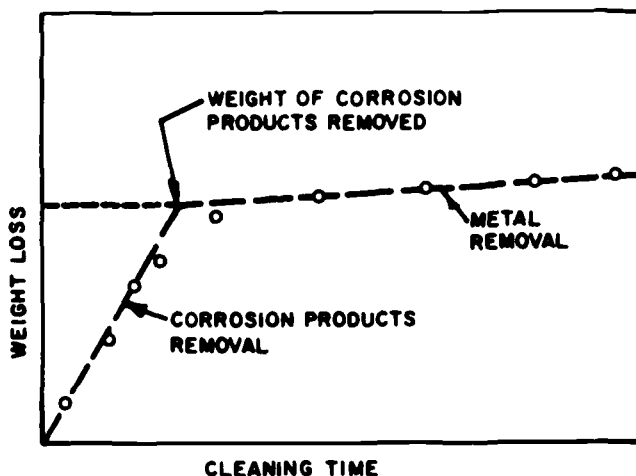


FIG. X2.1 Mass Loss Versus Exposure Time for Specimens During Cleaning

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, Pa. 19103.



Standard Practice for STANDARD REFERENCE METHOD FOR MAKING POTENTIOSTATIC AND POTENTIODYNAMIC ANODIC POLARIZATION MEASUREMENTS¹

This standard is issued under the fixed designation G 5; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

¹ NOTE—A revised Fig. 4B was included editorially in July 1983. (See Note in Fig 4B.)

1. Scope

1.1 This practice describes an experimental procedure which can be used to check one's experimental technique and instrumentation. If followed, this practice will provide repeatable potentiostatic and potentiodynamic anodic polarization measurements that will reproduce data determined by others at other times and in other laboratories.

1.2 Standard potentiostatic and potentiodynamic polarization plots are included. These reference data are based on the results from different laboratories (15 for the potentiostatic and 12 for the potentiodynamic) that followed the standard procedure, using a specific ferritic Type 430 stainless steel in 1.0 N H₂SO₄. Maximum and minimum current values are shown at each potential to indicate the acceptable range of values.

1.3 Deviations from the standard reference plots and the causes for such deviations are discussed in the Appendix.

1.4 Samples of the standard ferritic Type 430 stainless steel used in obtaining the standard reference plots are available for those who wish to check their own test procedure and equipment.²

1.5 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Applicable Document

2.1 *ASTM Standard:*

G 3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing³

3. Apparatus

3.1 The test cell should be constructed to allow the following items to be inserted into the solution chamber: the test electrode, two counter electrodes, a Luggin capillary with salt-bridge connection to the reference electrode, inlet and outlet for an inert gas, and a thermometer. The test cell shall be constructed of materials that will not corrode, deteriorate, or otherwise contaminate the test solution.

NOTE 1—Borosilicate glass and TFE-fluorocarbon have been found suitable.

3.1.1 A suitable cell is shown in Fig. 1 (1).⁴ A 1-L, round-bottom flask has been modified by the addition of various necks to permit the introduction of electrodes, gas inlet and outlet tubes, and a thermometer. The Luggin probe-salt bridge separates the bulk solution from the saturated calomel reference electrode, and the

¹ This practice is under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals.

Current edition approved Sept. 24, 1982. Published December 1982. Originally published as G 5 – 69. Last previous edition G 5 – 78.

² These standard samples are available from ASTM Headquarters. Generally, one sample can be repolished and reused for many runs. This procedure is suggested to conserve the available material. Order PCN 12-700050-00.

³ *Annual Book of ASTM Standards*, Vol 03.02.

⁴ The boldface numbers in parentheses refer to the list of references at the end of this practice.

probe tip can be easily adjusted to bring it in close proximity with the working electrode.

3.2 Electrode Holder (1):

3.2.1 The auxiliary and working electrodes are mounted in the type of holder shown in Fig. 2. A longer holder is required for the working electrode than for the auxiliary electrode. A leak-proof assembly is obtained by the proper compression fit between the electrode and a TFE-fluorocarbon gasket. (Too much pressure may cause shielding of the electrode or breakage of the glass holder, and too little pressure may cause leakage.)

3.3 Potentiostat (Note 2):

3.3.1 A potentiostat that will maintain an electrode potential within 1 mV of a preset value over a wide range of applied currents should be used. For the type and size of standard specimen supplied, the potentiostat should have a potential range of -0.6 to 1.6 V and an anodic current output range of 1.0 to 10^5 μ A. Many commercial potentiostats meet the specific requirements for these types of measurements.

3.4 Potential-Measuring Instruments (Note 2):

3.4.1 The potential-measuring circuit should have a high input impedance on the order of 10^{11} to 10^{14} Ω to minimize current drawn from the system during measurements. Such circuits are provided with most modern potentiostats. Instruments should have sufficient sensitivity and accuracy to detect a change of 1.0 mV over a potential range between -0.6 and 1.6 V.

3.5 Current-Measuring Instruments (Note 2):

3.5.1 An instrument that is capable of measuring a current accurately to within 1 % of the absolute value over a current range between 1.0 and 10^5 μ A for the approximately 5-cm^2 specimen of Type 430 stainless steel that is supplied should be used. Many commercial instruments are satisfactory for this purpose.

3.6 Anodic Polarization Circuit:

3.6.1 A schematic potentiostatic anodic polarization wiring diagram (2) is illustrated in Fig. 3. Note that the milliammeter is placed between the potentiostat and the auxiliary electrode. The potentiometer-electrometer combination provides the high impedance necessary for potential measurement. Most modern potentiostats have outputs of potential and current as a voltage for connection to appropriate recorders. This makes the use of a milliammeter, potentiometer, and electrometer null detec-

tor shown in Fig. 3 unnecessary.

3.6.2 A scanning potentiostat is used for potentiodynamic measurements. A record of the potential and current is plotted continuously using such instruments as an X - Y recorder and a logarithmic converter incorporated into the circuit described in 3.6.1. Some potentiostats have an output of the logarithm of the current as a voltage, which allows direct plotting of the potential log current curve using an X - Y recorder.

NOTE 2—The instrumental requirements are based upon values typical of the instruments in the 15 laboratories that provided the data used in determining the standard polarization plot.

3.7 Electrodes:

3.7.1 *Working Electrode*, prepared from a $\frac{1}{2}$ -in. (12.7-mm) length of $\frac{3}{8}$ -in. (9.5-mm) diameter rod stock. Each electrode is drilled, tapped, and mounted in the manner discussed in 3.2.1.

NOTE 3—If specimen forms are used other than those called for by this practice, for example, flat sheet specimen, care should be taken since it was shown that crevices may be introduced which can lead to erroneous results (see Fig. X1).

3.7.1.1 The standard AISI Type 430 stainless steel should be used if one wishes to reproduce the standard reference plot. This material was prepared from a single heat of metal that was mill-annealed for $\frac{1}{2}$ h at 1500°F (816°C) and air cooled. The chemical composition of the standard stainless steel is shown in Table 1.

3.7.2 *Platinum Electrodes*—Two platinum auxiliary electrodes are prepared from high-purity rod stock. Each electrode is drilled, tapped, and mounted with a TFE-fluorocarbon gasket in the same manner as the working electrode. A large platinum sheet sealed into a glass holder is also acceptable.

3.7.2.1 A platinized surface is recommended because of the increased surface area. This may be accomplished by cleaning the surface in hot aqua regia (3 parts concentrated HCl and 1 part concentrated HNO_3), washing, and then drying. Both electrodes are platinized by immersing them in a solution of 3 % platinum chloride and 0.02 % lead acetate and electrolyzing at a current density of 40 to 50 mA/cm^2 for 4 or 5 min (1,3). The polarity is reversed every minute. Occluded chloride is removed by electrolyzing in a dilute (10 %) sulfuric acid solution for several minutes with a reversal in polarity every minute. Electrodes are rinsed thoroughly and stored in distilled water until ready for use. Since certain ions can poison these

electrodes, periodic checks of platinized platinum potentials against a known reference electrode should be made.

3.7.2.2 Alternatively, graphite auxiliary electrodes can be used, but material retained by the graphite may contaminate subsequent experiments. This contamination can be minimized by using high-density graphite or avoided by routinely replacing the graphite electrode.

3.8 *Calomel Electrode (4)*—A saturated calomel electrode with a controlled rate of leakage (about 3 $\mu\text{L/h}$) is recommended. This type of electrode is durable, reliable, and commercially available. The normal precautions should be taken to ensure that it is maintained in the proper condition. The potential of the calomel electrode should be checked at periodic intervals to ensure the accuracy of the electrode.

4. Standard Experimental Procedure

4.1 *Test Specimen Preparation:*

4.1.1 Prepare the surface within 1 h of the experiment. Wet grind with 240-grit SiC paper, wet polish with 600-grit SiC paper until previous coarse scratches are removed, rinse, and dry. (Drilled and tapped specimens can be threaded onto an electrode holder rod and secured in a lathe or electric drill for this operation.)

4.1.2 Determine the surface area by measuring all dimensions to the nearest 0.01 mm, subtracting the area under the gasket (usually 0.20 to 0.25 cm^2).

4.1.3 Mount the specimen on the electrode holder as described in 3.2.1. Tighten the assembly by holding the upper end of the mounting rod in a vise or clamp while tightening the mounting nut until the gasket is properly compressed.

4.1.4 Clean the specimen just prior to immersion by degreasing for 5 min in boiling benzen (Caution: Use under hood), followed by rinsing in distilled water.

4.2 Prepare 1 L of 1.0 *N* H_2SO_4 from reagent grade acid and distilled water, using 27.2 mL of 98 % H_2SO_4 /L of solution. Transfer 900 mL of solution to clean polarization cell.

4.3 Bring the temperature of the solution to $30 \pm 1^\circ\text{C}$ by immersing the test cell in a controlled-temperature water bath or by other convenient means.

4.4 Place the platinized auxiliary electrodes, salt-bridge probe, and other components in the test cell and temporarily close the center open-

ing with a glass stopper. Fill the salt bridge with test solution.

NOTE 4—The levels of the solution in the reference and polarization cells should be the same to avoid siphoning. If this is impossible, a closed solution-wet (not greased) stopcock can be used in the salt bridge to eliminate siphoning.

4.5 Purge the solution prior to immersion of the test specimen, for a minimum of $\frac{1}{2}$ h with oxygen-free hydrogen gas at the rate of 150 cm^3/min to remove oxygen from solution.

4.6 Transfer the specimen to the test cell and adjust the salt-bridge probe tip so it is about 2 mm from the specimen electrode.

4.7 Record the open-circuit specimen potential, that is, the corrosion potential, after 55 min immersion. Record the platinized platinum potential 50 min after immersion of the specimen.

4.8 *Potential Scan:*

4.8.1 Start the potential scan 1 h after specimen immersion, beginning at the corrosion potential (E_{corr}) for potentiodynamic measurements and the nearest 50-mV increment above E_{corr} for the potentiostatic measurements. Proceed through +1.60 V versus saturated calomel electrode (SCE) (active to noble).

4.8.2 Use a potentiostatic potential step rate of 50 mV every 5 min, recording the current at the end of each 5-min period at potential.

4.8.3 Use a potentiodynamic potential sweep rate of 0.6 V/h ($\pm 5\%$) recording the current continuously with change in potential.

4.9 Plot anodic polarization data on semi-logarithmic paper in accordance with Practice G 3, (potential-ordinate, current density-abscissa). If a potentiostat with a logarithmic converter is used, this plot can be produced directly during the measurement.

5. Standard Reference Plots

5.1 Standard polarization plots based on potentiostatic data from 15 different laboratories and potentiodynamic data from 12 different laboratories are shown in Figs. 4A and 4B (5). The plots show a range of acceptable current density values at each potential. The average corrosion potential is -0.52 V, and the average platinized platinum potential is -0.26 V.

5.2 These plots were prepared from data obtained by following the standard procedure discussed in this practice.

5.3 Typical deviations from the standard potentiostatic plot are shown and discussed in Appendix X1. Reference to this discussion may be helpful in determining the reasons for dif-

ferences between an experimental curve and the standard plots.

5.4 The potentiodynamic standard curve shows good agreement with the potentiostatic standard curve determined at an equivalent overall polarization rate.

6. Reproducibility

6.1 It is possible to show excellent repeatability in the same laboratory when following a set procedure. However, these data may not agree with that in another laboratory. An example of repeatable data is shown in Fig. 5, but the curve does not agree completely with the

standard plot in that the secondary current density maximum occurs at a potential of about 100 mV more active than the standard.

6.2 The small spread in data obtained from a number of laboratories and used in the preparation of the standard plot demonstrated that good reproducibility is possible when a standard procedure is followed.

6.3 The availability of a standard procedure, standard material, and a standard plot should make it easy for an investigator to check his techniques. This should lead to polarization curves in the literature which can be compared with greater confidence than is possible now.

TABLE 1 Chemical Composition of Standard Type 430 Stainless Steel, Weight Percent.

Chromium	16.52
Carbon	0.052
Manganese	0.40
Silicon	0.49
Phosphorus	0.013
Sulfur	0.009
Nickel	0.27
Molybdenum	0.03
Copper	0.07
Iron	balance

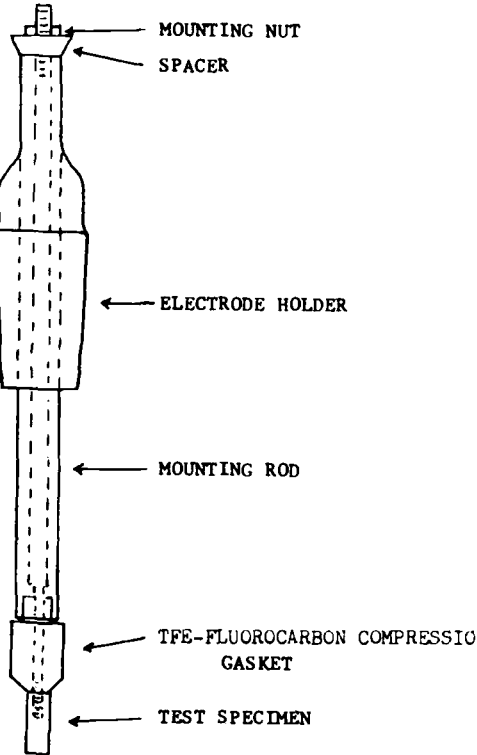


FIG. 2 Specimen Mounted on Electrode Holder

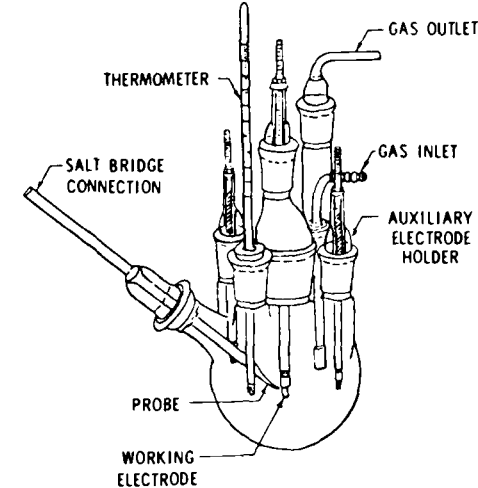


FIG. 1 Schematic Diagram of Polarization Cell (1)

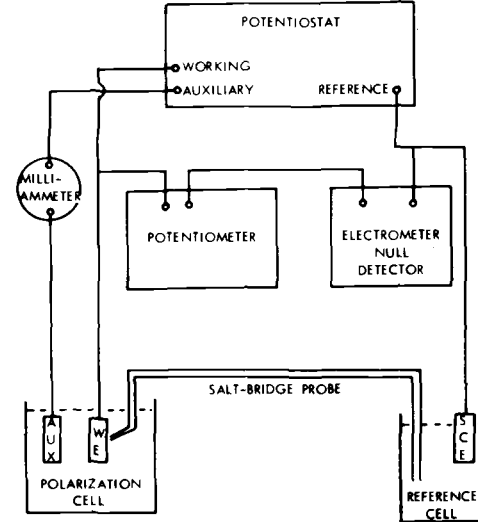


FIG. 3 Schematic Potentiostatic Anodic Polarization Wiring Diagram (2)

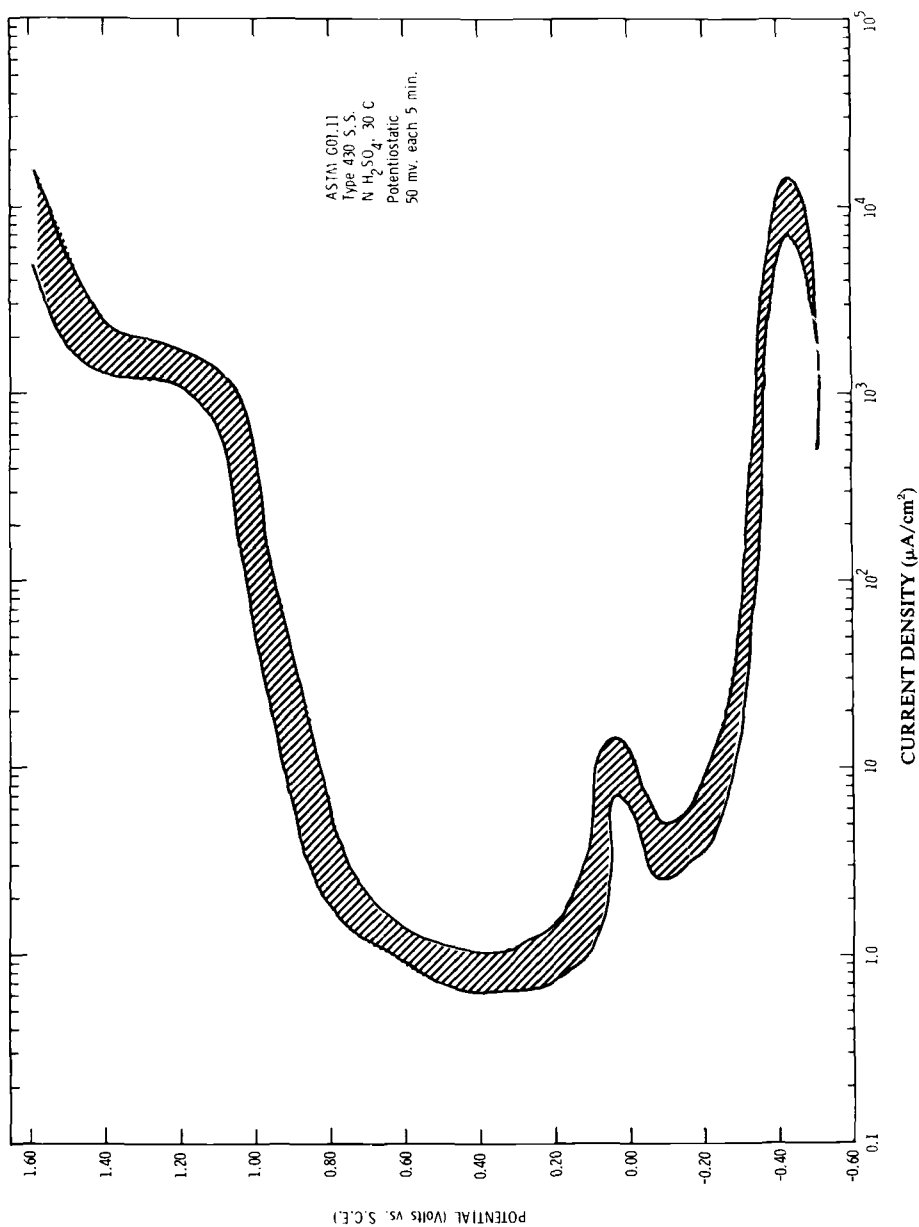
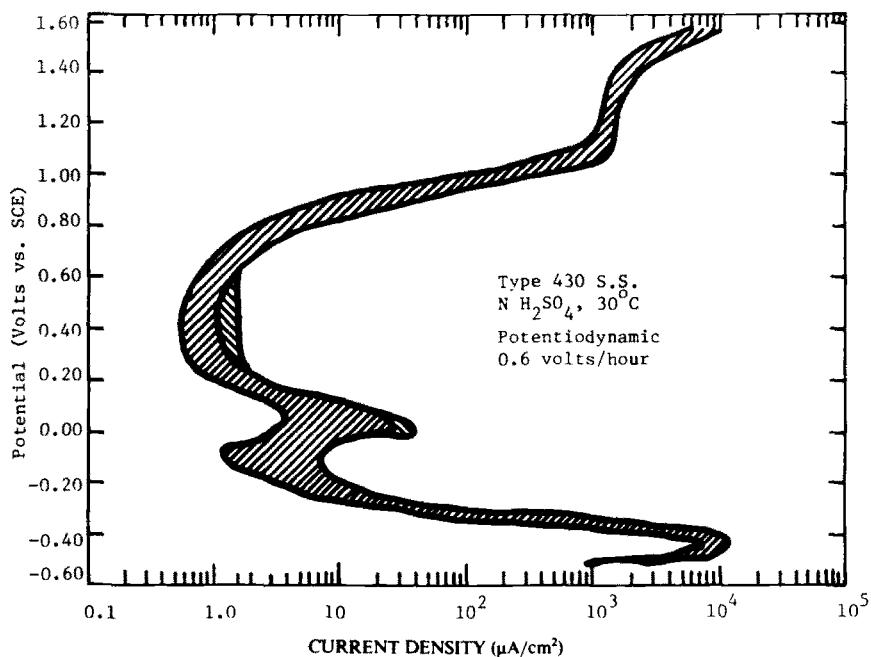


FIG. 4A Standard Potentiostatic Anodic Polarization Plot



NOTE—This practice is being revised within Subcommittee G01.11. A new heat of Type 430 S.S. is being used for standard material, and the standard curve has been shown through round-robin testing to be slightly off in terms of the maximum passive current density and the maximum secondary peak current density for this new material. This plot shows the standard curve and the revised areas (crosshatched in the opposite direction). This revised curve has not been balloted and is offered for information only for your convenience until a revised practice appears.

FIG. 4B Standard Potentiodynamic Anodic Polarization Plot

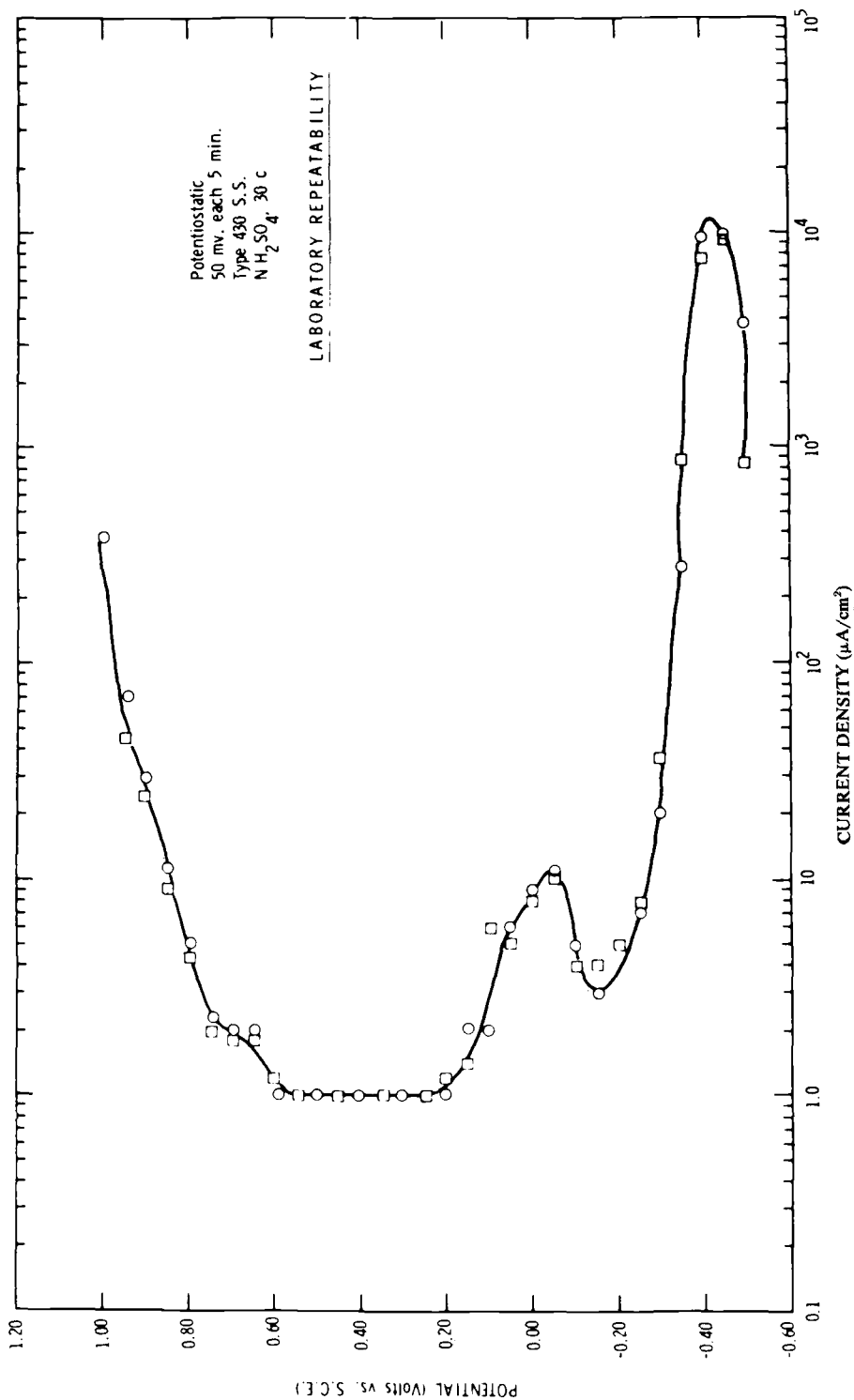


FIG. 5 Laboratory Repeatability of Potentiostatic Anodic Polarization Curve

APPENDIX

(Nonmandatory Information)

X1. DEVIATIONS FROM STANDARD POLARIZATION PLOTS

X1.1 High Passive Current Densities (Crevice Effect)

X1.1.1 Examples of passive current densities which are greater than those for the standard potentiostatic plot are shown in Fig. X1. This effect is attributable to a crevice between the specimen and mounting material (6). The crevice may be the result of the mounting technique or the material used for mounting.

X1.1.2 The potential drop along the narrow path of the electrolyte within the crevice between the specimen and the mounting material prevents this area from passivating. Although the face of the specimen passivates, the high current density associated with the active crevice contributes to an increase in the measured current density. Specimen electrodes for polarization measurements must be mounted without crevice sites to avoid such erroneous passive current densities.

X1.2 Low Passive Current Densities (Instrumental Effect)

X1.2.1 The low passive current densities shown in

Fig. X2 are undoubtedly the result of instrumental problems. This effect can be eliminated by calibrating the current over the entire range of interest before conducting an experiment.

X1.3 Cathodic Currents During Anodic Polarization (Oxygen Effect)

X1.3.1 The "negative loop" at potentials between -0.350 V and -0.050 V, shown by dashed lines in Fig. X3, occurs when the total cathodic current exceeds the total anodic current. Such results are characteristic of oxygen being present in the solution (7). This effect can be anticipated if the recorded platinum potential is considerably more noble than -0.26 V. The hydrogen purge should remove oxygen from the system, but there may be an air leak or the hydrogen may be contaminated with oxygen. It is necessary to take extreme care in the design of glassware equipment and to ensure a high order of purity in the gas that is used to avoid oxygen contamination.

REFERENCES

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- (3) Mellon, M. G., *Quantitative Analysis*, Thomas Y. Crowell Co., New York, 1955.
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- (6) Greene, N. D., France, Jr., W. D., and Wilde, B. E., "Electrode Mounting for Potentiostatic Anodic Polarization Studies," *Corrosion, CORRA*, Vol 21, 1965, p. 275.
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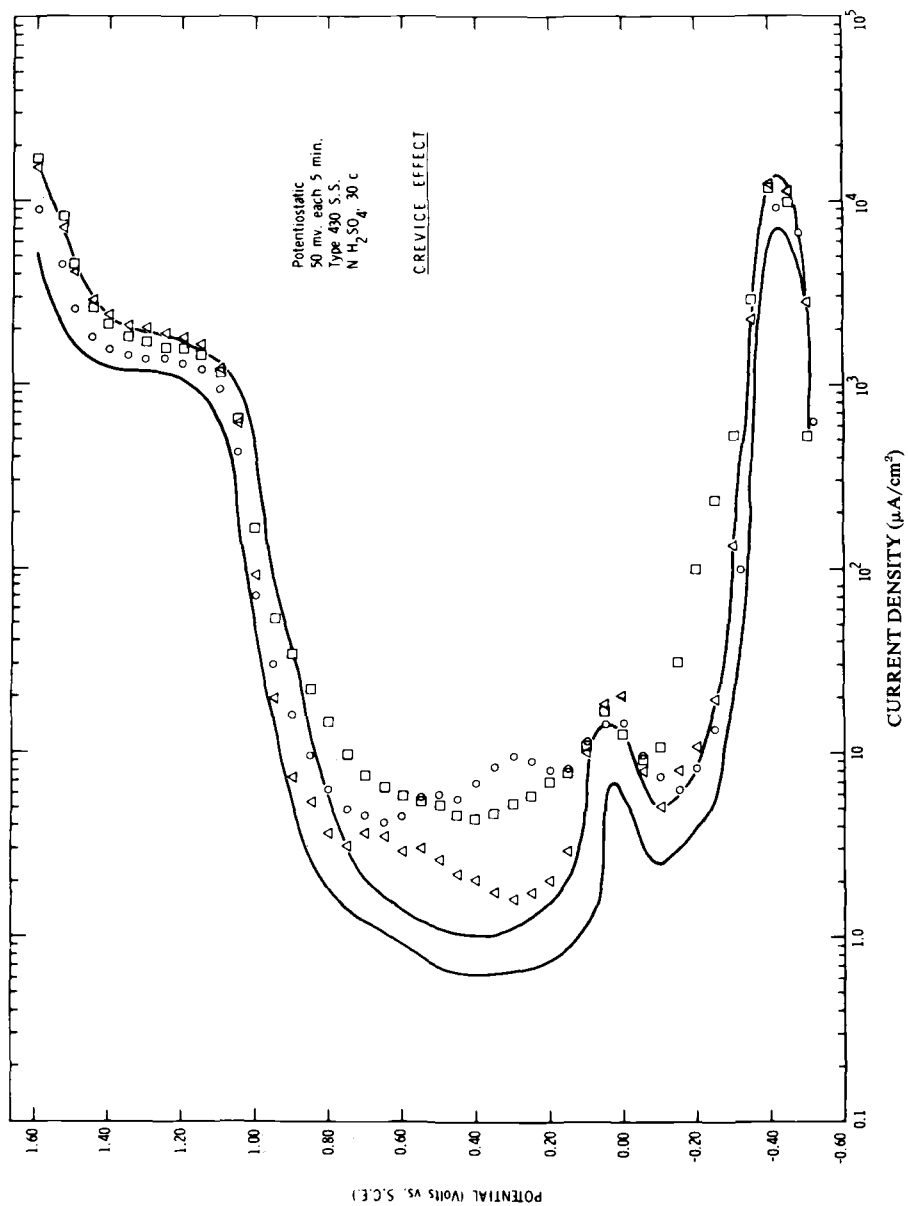


FIG. X1 Crevice Effect During Potentiostatic Anodic Polarization

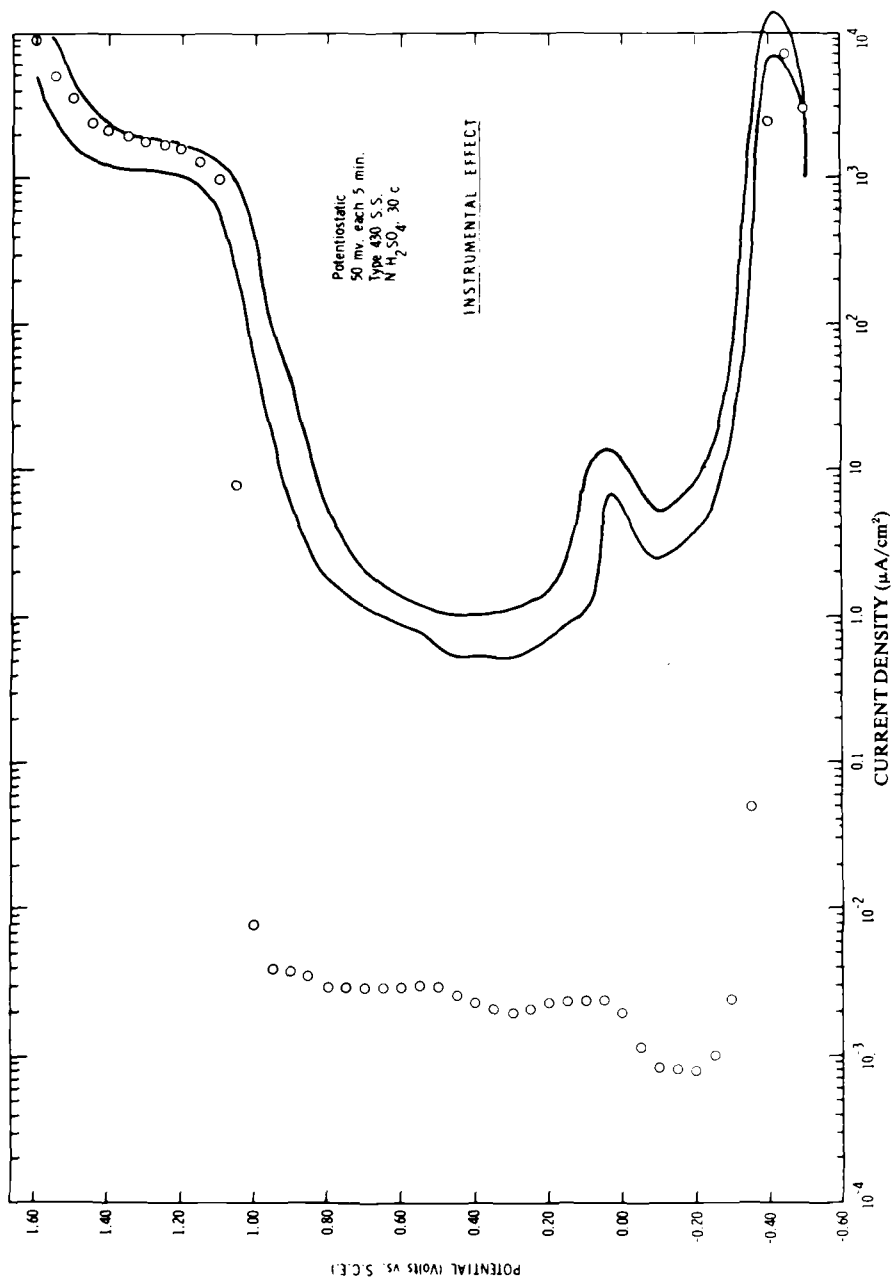


FIG. X2 Instrumental Effect During Potentiostatic Anodic Polarization

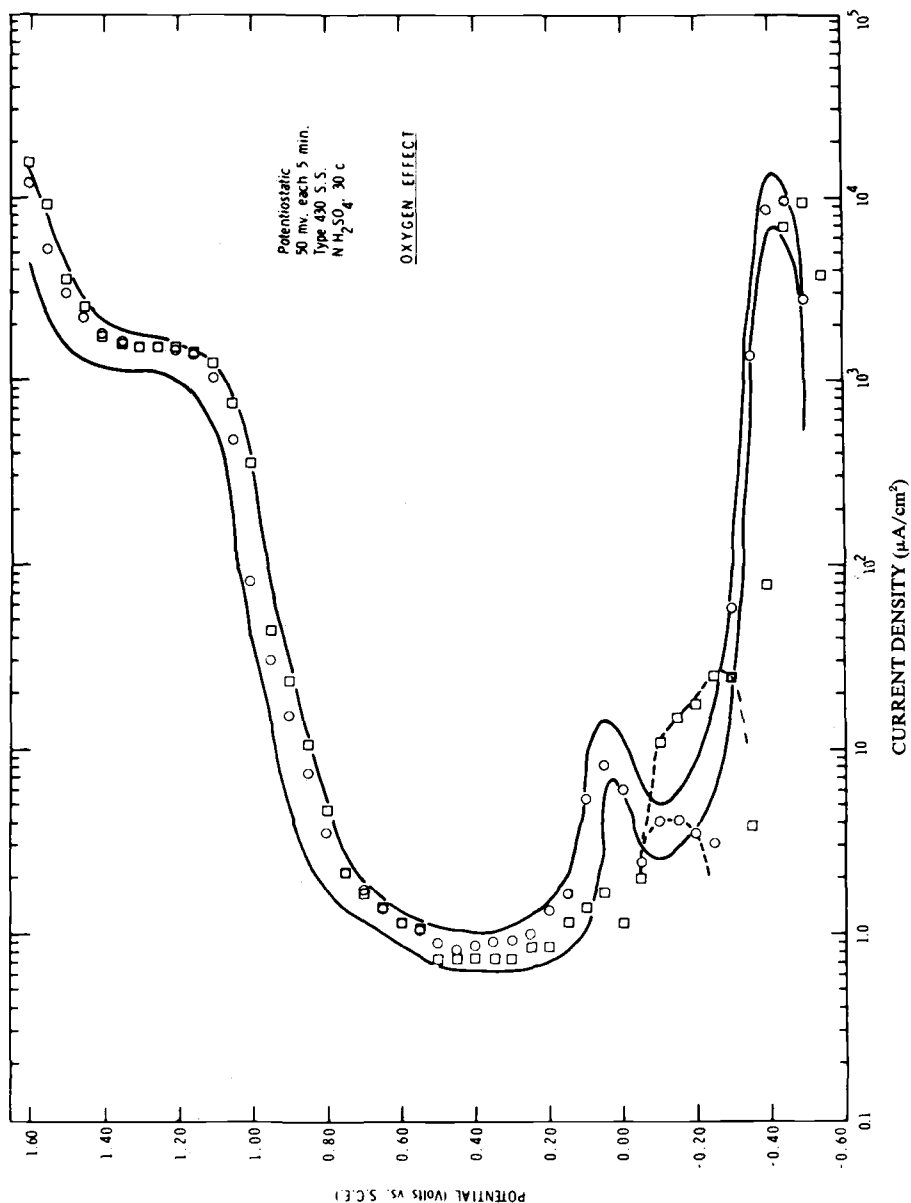


FIG. X3 Oxygen Effect During Potentiostatic Anodic Polarization

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Standard Definitions of Terms Relating to CORROSION AND CORROSION TESTING¹

This standard is issued under the fixed designation G 15; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

These definitions have been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

- active**—the negative direction of electrode potential. (Also used to describe corrosion and its associated potential range when an electrode potential is more negative than an adjacent depressed corrosion rate [passive] range.)
- anion**—a negatively charged ion.
- anode**—the electrode of an electrolytic cell at which oxidation is the principal reaction. (Electrons flow away from the anode in the external circuit. It is usually the electrode where corrosion occurs and metal ions enter solution.)
- anode corrosion efficiency**—the ratio of the actual corrosion (weight loss) of an anode to the theoretical corrosion (weight loss) calculated by Faraday's law from the quantity of electricity that has passed.
- anodic inhibitor**—an inhibitor of metal corrosion that reduces the rate of the anodic or oxidation reaction.
- anodic polarization**—the change of the electrode potential in the noble (positive) direction due to current flow. (See **polarization**.)
- anodic protection**—a technique to reduce the corrosion rate of a metal by polarizing it into its passive region where dissolution rates are low.
- anolyte**—the electrolyte adjacent to the anode of an electrolytic cell.
- auxiliary electrode**—the electrode in an electrochemical cell that is used to transfer current to or from a test electrode.
- breakdown potential**—the least noble potential where pitting or crevice corrosion, or both, will initiate and propagate.
- cathode**—the electrode of an electrolytic cell at which reduction is the principal reaction. (Electrons flow toward the cathode in the external circuit.)
- cathodic corrosion**—corrosion of a metal when it is a cathode. (It usually happens to metals because of a rise in pH at the cathode or as a result of the formation of hydrides.)
- cathodic inhibitor**—an inhibitor of metal corrosion that reduces the rate of the cathodic or reduction reaction.
- cathodic polarization**—the change of the electrode potential in the active (negative) direction due to current flow. (See **polarization**.)
- cathodic protection**—a technique to reduce the corrosion rate of a metal surface by making it the cathode of an electrochemical cell.
- catholyte**—the electrolyte adjacent to the cathode of an electrolytic cell.
- cation**—a positively charged ion.
- caustic cracking**—stress corrosion cracking of metals in caustic solutions. (See also **stress corrosion cracking**.)
- caustic embrittlement**—see **caustic cracking**.
- cavitation**—the formation and rapid collapse within a liquid of cavities or bubbles that contain vapor or gas or both.
- cavitation damage**—the degradation of a solid body resulting from its exposure to cavitation. (This may include loss of material, surface deformation, or changes in properties or appearance.)
- cavitation-erosion**—progressive loss of original material from a solid surface due to continuing exposure to cavitation.

¹ These definitions are under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals and are the direct responsibility of Subcommittee G01.02 on Nomenclature.

Current edition approved March 29, and April 26, 1985. Published July 1985. Originally published as G 15 – 71. Last previous edition G 15 – 83a.

- chemical conversion coating**—a protective or decorative nonmetallic coating produced *in situ* by chemical reaction of a metal with a chosen environment. (It is often used to prepare the surface prior to the application of an organic coating.)
- concentration cell**—an electrolytic cell, the emf of which is caused by a difference in concentration of some component in the electrolyte. (This difference leads to the formation of discrete cathode and anode regions.)
- corrosion**—the chemical or electrochemical reaction between a material, usually a metal, and its environment that produces a deterioration of the material and its properties.
- corrosion fatigue**—the process in which a metal fractures prematurely under conditions of simultaneous corrosion and repeated cyclic loading at lower stress levels or fewer cycles than would be required in the absence of the corrosive environment.
- corrosion fatigue strength**—the maximum repeated stress that can be endured by a metal without failure under definite conditions of corrosion and fatigue and for a specific number of stress cycles and a specified period of time.
- corrosion potential**—the potential of a corroding surface in an electrolyte relative to a reference electrode measured under open-circuit conditions.
- corrosion rate**—the amount of corrosion occurring in unit time. (For example, mass change per unit area per unit time; penetration per unit time)
- counter electrode**—See **auxiliary electrode**.
- crazing**—a network of checks or cracks appearing on a coated surface.
- crevice corrosion**—localized corrosion of a metal surface at, or immediately adjacent to, an area that is shielded from full exposure to the environment because of close proximity between the metal and the surface of another material.
- critical anodic current density**—the maximum anodic current density observed in the active region for a metal or alloy electrode that exhibits active-passive behavior in an environment.
- critical humidity**—the relative humidity above which the atmospheric corrosion rate of some metals increases sharply.
- critical pitting potential**—the least noble potential where pitting corrosion will initiate and propagate. (See breakdown potential.)
- current density**—the current flowing to or from a unit area of an electrode surface.
- current efficiency**—the ratio of the electrochemical equivalent current density for a specific reaction to the total applied current density.
- deactivation**—the process of prior removal of the active corrosive constituents, usually oxygen, from a corrosive liquid by controlled corrosion of expendable metal or by other chemical means, thereby making the liquid less corrosive.
- dealloying**—see **parting**.
- depolarization**—not a preferred term. (See **polarization**.)
- deposit corrosion**—localized corrosion under or around a deposit or collection of material on a metal surface. (See also **crevice corrosion**.)
- dezincification**—See **parting**; specific to copper-zinc alloys.
- differential aeration cell (oxygen concentration cell)**—a concentration cell caused by differences in oxygen concentration along the surface of a metal in an electrolyte. (See **concentration cell**.)
- diffusion limited current density**—the current density, often referred to as limiting current density, that corresponds to the maximum transfer rate that a particular species can sustain due to the limitation of diffusion.
- electrochemical cell**—an electrochemical system consisting of an anode and a cathode in metallic contact and immersed in an electrolyte. (The anode and cathode may be different metals or dissimilar areas on the same metal surface.)
- electrochemical potential (electrochemical tension)**—the partial derivative of the total electrochemical free energy of the system with respect to the number of moles of the constituent in a solution when all other factors are constant. (Analogous to the chemical potential of the constituent except that it includes the electrical as well as the chemical contributions to the free energy.)
- electrode potential**—the potential of an electrode in an electrolyte as measured against a reference electrode. (The electrode potential does not include any resistance losses in potential in either the solution or external cir-

- cuit. It represents the reversible work to move a unit charge from the electrode surface through the solution to the reference electrode.)
- electrolysis**—production of chemical changes of the electrolyte by the passage of current through an electrochemical cell.
- electrolytic cleaning**—a process of removing soil, scale, or corrosion products from a metal surface by subjecting it as an electrode to an electric current in an electrolytic bath.
- Electromotive Force Series (EMF Series)**—a list of elements arranged according to their standard electrode potentials, with “noble” metals such as gold being positive and “active” metals such as zinc being negative.
- embrittlement**—the severe loss of ductility or toughness or both, of a material, usually a metal or alloy.
- equilibrium (reversible) potential**—the potential of an electrode in an electrolytic solution when the forward rate of a given reaction is exactly equal to the reverse rate. (The equilibrium potential can only be defined with respect to a specific electrochemical reaction.)
- erosion**—the progressive loss of material from a solid surface due to mechanical interaction between that surface and a fluid, a multi-component fluid, or solid particles carried with the fluid.
- erosion-corrosion**—a conjoint action involving corrosion and erosion in the presence of a moving corrosive fluid, leading to the accelerated loss of material.
- exchange current density**—the rate of charge transfer per unit area when an electrode reaches dynamic equilibrium (at its reversible potential) in a solution; that is, the rate of anodic charge transfer (oxidation) balances the rate of cathodic charge transfer (reduction).
- exfoliation**—corrosion that proceeds laterally from the sites of initiation along planes parallel to the surface, generally at grain boundaries, forming corrosion products that force metal away from the body of the material, giving rise to a layered appearance.
- external circuit**—the wires, connectors, measuring devices, current sources, etc., that are used to bring about or measure the desired electrical conditions within the test cell.
- filiform corrosion**—corrosion that occurs under some coatings in the form of randomly distributed threadlike filaments.
- fretting corrosion**—the deterioration at the interface between contacting surfaces as the result of corrosion and slight oscillatory slip between the two surfaces.
- galvanic corrosion**—accelerated corrosion of a metal because of an electrical contact with a more noble metal or nonmetallic conductor in a corrosive electrolyte.
- galvanic couple**—a pair of dissimilar conductors, commonly metals, in electrical contact. (See **galvanic corrosion**.)
- galvanic current**—the electric current that flows between metals or conductive nonmetals in a galvanic couple.
- Galvanic Series**—a list of metals and alloys arranged according to their relative corrosion potentials in a given environment.
- galvanostatic**—an experimental technique whereby an electrode is maintained at a constant current in an electrolyte.
- grain dropping**—the dislodgement and loss of a grain or grains (crystals) from a metal surface as a result of intergranular corrosion.
- graphitic corrosion**—the deterioration of metallic constituents in gray cast iron, which leaves the graphitic particles intact. (The term “graphitization” is commonly used to identify this form of corrosion but is not recommended because of its use in metallurgy for the decomposition of carbide to graphite.)
- hot corrosion**—an accelerated corrosion of metal surfaces that results from the combined effect of oxidation and reactions with sulfur compounds and other contaminants, such as chlorides, to form a molten salt on a metal surface which fluxes, destroys, or disrupts the normal protective oxide.
- hydrogen blistering**—the formation of blisters on or below a metal surface from excessive internal hydrogen pressure. (Hydrogen may be formed during cleaning, plating, corrosion, etc.)
- hydrogen embrittlement**—hydrogen-induced cracking or severe loss of ductility caused by the presence of hydrogen in the metal.
- immunity**—a state of resistance to corrosion or anodic dissolution of a metal caused by thermodynamic stability of the metal.
- impingement corrosion**—a form of erosion-corrosion generally associated with the local

- impingement of a high-velocity, flowing fluid against a solid surface.
- impressed current**—an electric current supplied by a device employing a power source that is external to the electrode system. (An example is d-c current for cathodic protection.)
- inhibitor**—a chemical substance or combination of substances that, when present in the proper concentration and forms in the environment, prevents or reduces corrosion.
- intensistatic**—See **galvanostatic**.
- intercrystalline corrosion**—See **intergranular corrosion**.
- intergranular corrosion**—preferential corrosion at or adjacent to the grain boundaries of a metal or alloy.
- internal oxidation**—the formation of isolated particles of corrosion products beneath the metal surface. (This occurs as the result of preferential oxidation of certain alloy constituents by inward diffusion of oxygen, nitrogen, sulfur, etc.)
- knife-line attack**—intergranular corrosion of an alloy, usually stabilized stainless steel, along a line adjoining or in contact with a weld after heating into the sensitization temperature range.
- local action corrosion**—corrosion caused by local corrosion cells on a metal surface.
- local corrosion cell**—an electrochemical cell created on a metal surface because of a difference in potential between adjacent areas on that surface.
- localized corrosion**—corrosion at discrete sites, for example, pitting, crevice corrosion, and stress corrosion cracking.
- long-line current**—current that flows through the earth from an anodic to a cathodic area of a continuous metallic structure. (Usually used only where the areas are separated by considerable distance and where the current results from concentration-cell action.)
- Luggin probe or Luggin-Haber capillary**—a device used in measuring the potential of an electrode with a significant current density imposed on its surface. (The probe minimizes the IR drop that would otherwise be included in the measurement and without significantly disturbing the current distribution on the specimen.)
- metal dusting**—accelerated deterioration of metals in carbonaceous gases at elevated temperatures to form a dust-like corrosion product.
- metallizing**—See **thermal spraying**.
- microbial corrosion**—corrosion that is affected by the action of microorganisms in the environment.
- mixed potential**—the potential of a specimen when two or more electrochemical reactions are occurring simultaneously.
- noble**—the positive (increasingly oxidizing) direction of electrode potential.
- noble metal**—a metal with a standard electrode potential that is more noble (positive) than that of hydrogen.
- open-circuit potential**—the potential of an electrode measured with respect to a reference electrode or another electrode when no current flows to or from it.
- overvoltage**—the change in potential of an electrode from its equilibrium or steady state value when current is applied.
- oxidation**—loss of electrons by a constituent of a chemical reaction. (Also refers to the corrosion of a metal that is exposed to an oxidizing gas at elevated temperatures.)
- parting**—the selective corrosion of one or more components of a solid solution alloy.
- parting limit**—the minimum concentration of a more noble component in an alloy, above which parting does not occur in a specific environment.
- passivation**—the process in metal corrosion by which metals become passive. (See **passive**.)
- passivator**—a type of inhibitor which appreciably changes the potential of a metal to a more noble (positive) value.
- passive**—the state of the metal surface characterized by low corrosion rates in a potential region that is strongly oxidizing for the metal.
- passive-active cell**—a corrosion cell in which the anode is a metal in the active state and the cathode is the same metal in the passive state.
- pitting**—corrosion of a metal surface, confined to a point or small area, that takes the form of cavities.
- pitting factor**—ratio of the depth of the deepest pit resulting from corrosion divided by the average penetration as calculated from weight loss.
- polarization**—the change from the open-circuit electrode potential as the result of the passage of current.
- polarization admittance**—the reciprocal of po-

- larization resistance (di/dE).
- polarization resistance**—the slope (dE/di) at the corrosion potential of a potential (E)–current density (i) curve. (It is inversely proportional to the corrosion current density when the polarization resistance technique is applicable.)
- potentiodynamic (potentiokinetic)**—the technique for varying the potential of an electrode in a continuous manner at a preset rate.
- potentiostat**—an instrument for automatically maintaining an electrode in an electrolyte at a constant potential or controlled potentials with respect to a suitable reference electrode.
- potentiostatic**—the technique for maintaining a constant electrode potential.
- poultice corrosion*—see **deposit corrosion**.
- Pourbaix diagram (electrode potential-pH diagram)**—a graphical representation showing regions of thermodynamic stability of species in metal-water electrolyte systems.
- primary passive potential (passivation potential)**—the potential corresponding to the maximum active current density (critical anodic current density) of an electrode that exhibits active-passive corrosion behavior.
- protection potential**—the most noble potential where pitting and crevice corrosion will not propagate.
- redox potential**—the potential of a reversible oxidation-reduction electrode measured with respect to a reference electrode, corrected to the hydrogen electrode, in a given electrolyte.
- reduction**—the gain of electrons by a constituent of a chemical reaction.
- rest potential*—See **open-circuit potential**.
- rust**—a corrosion product consisting primarily of hydrated iron oxide. (A term properly applied only to ferrous alloys.)
- season cracking*—See **stress-corrosion cracking**.
- stray current corrosion**—the corrosion caused by electric current from a source external to the intended electrical circuit, for example, extraneous current in the earth.
- stress-corrosion cracking**—a cracking process that requires the simultaneous action of a corrodent and sustained tensile stress. (This excludes corrosion-reduced sections which fail by fast fracture. It also excludes intercrystalline or transcrystalline corrosion which can disintegrate an alloy without either applied or residual stress.)
- subsurface corrosion*—See **internal oxidation**.
- sulfidation**—the reaction of a metal or alloy with a sulfur-containing species to produce a sulfur compound that forms on or beneath the surface of the metal or alloy.
- Tafel slope**—the slope of the straight line portion of a polarization curve, usually occurring at more than 50 mV from the open-circuit potential, when presented in a semi-logarithmic plot in terms of volts per logarithmic cycle of current density (commonly referred to as volts per decade).
- thermal spraying**—a group of processes wherein finely divided metallic or nonmetallic materials are deposited in a molten or semimolten condition to form a coating. (The coating material may be in the form of powder, ceramic rod, wire, or molten materials.)
- thermogalvanic corrosion**—the corrosive effect resulting from the galvanic cell caused by a thermal gradient across the metal surface.
- transpassive region**—the region of an anodic polarization curve, noble to and above the passive potential range, in which there is a significant increase in current density (increased metal dissolution) as the potential becomes more positive (noble).
- tuberculation**—the formation of localized corrosion products that appear on a surface as knoblike prominences (tubercles).
- uniform corrosion**—corrosion that proceeds at about the same rate over a metal surface.
- weld decay**—not a preferred term. Intergranular corrosion, usually of stainless steels or certain nickel-base alloys, that occurs as the result of sensitization in the heat-affected zone during the welding operation.
- working electrode**—the test or specimen electrode in an electrochemical cell.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, Pa. 19103.



Standard Test Methods of DETECTING SUSCEPTIBILITY TO INTERGRANULAR ATTACK IN WROUGHT, NICKEL-RICH, CHROMIUM-BEARING ALLOYS¹

This standard is issued under the fixed designation G 28; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover two tests as follows:

1.1.1 *Method A*—This method covers the procedures for the boiling ferric sulfate—50 % sulfuric acid test which applies to nine alloys and measures the susceptibility of certain nickel-rich, chromium-bearing alloys to intergranular attack, which may be encountered in certain service environments.

1.1.2 *Method B*—This method covers the procedures for a test in boiling 23 % H_2SO_4 + 1.2 % HCl + 1 % FeCl_3 + 1 % CuCl_2 that applies to one alloy for measuring the susceptibility to intergranular attack, which may be encountered in certain service environments.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. (For more specific safety precautionary statements see 5.1.1, 5.1.9, 12.1.1, and 12.1.10.)*

2. Applicable Document

2.1 ASTM Standard:

A 262 Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels²

METHOD A—Ferric Sulfate—50 % Sulfuric Acid Test

3. Significance and Use

3.1 The boiling ferric sulfate-sulfuric acid test

may be applied to the following alloys in the wrought condition:

Alloy	Testing Time, h
N10276	24
N06455	24
N06007	120
N06985	120
N08020	120
N00600	24
N06625	120
N08800	120
N08825 ⁴	120

⁴ While the ferric sulfate-sulfuric acid test does detect susceptibility to intergranular attack in Alloy N08825, the boiling 65 % nitric acid test, ASTM Practice A 262, Part C, for detecting susceptibility to intergranular attack in stainless steels is more sensitive.

3.2 Nominal compositions of these alloys are given in Table 1.

3.3 This method may be used to evaluate as-received material and to evaluate the effects of subsequent heat treatments. In the case of nickel-rich, chromium-bearing alloys, the method may be applied only to wrought products. The test is not applicable to cast products.

4. Apparatus (See Fig. 1.)

4.1 *Allihn or Soxhlet Condenser*, 4-bulb,³ with a 45/50 ground-glass joint, overall length about

¹ These test methods are under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.

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² *Annual Book of ASTM Standards*, Vol 03.02.

³ To avoid frequent chipping of the drip-tip of the condenser during handling, the modified condenser described by Streicher, M. A., and Sweet, A. J., *Corrosion*, Vol 25, 1969, pp. 1, may be used.

330 mm, condensing section about 240 mm.

4.2 *Erlenmeyer Flask*, 1-L, with a 45/50 ground-glass joint. The ground-glass opening shall be 40 mm wide.

4.3 *Glass Cradle* (Fig. 2)—This can be supplied by a glass blowing shop. To pass through the ground-glass joint on the Erlenmeyer flask, the width of the cradle should not exceed 40 mm and the front-to-back distance must be such that the cradle will fit the 40-mm diameter opening. It should have three or four holes to increase circulation of the test solution around the specimen.

NOTE 1—Other equivalent means of specimen support, such as glass hooks or stirrups, may also be used.

4.4 *Boiling Chips*, or some other boiling aids must be used to prevent bumping.

4.5 *Silicone Grease*⁴ is recommended for the ground-glass joint.

4.6 *Electrically Heated Hot Plate*, or equivalent to provide heat for continuous boiling of the solution.

4.7 *Analytical Balance*, capable of weighing to the nearest 0.001 g.

5. Test Solution

5.1 Prepare 600 mL of 50 % (49.4 to 50.9 %) solution as follows:

5.1.1 **Caution**—Protect the eyes and use rubber gloves for handling acid. Place the test flask under a hood.

5.1.2 First, measure 400.0 mL of distilled water in a 500-mL graduate and pour into the Erlenmeyer flask.

5.1.3 Then measure 236.0 mL of reagent-grade sulfuric acid (H_2SO_4) of a concentration which must be in the range from 95.0 to 98.0 weight % in a 250-mL graduate. Add the acid slowly to the water in the Erlenmeyer flask to avoid boiling by the heat evolved. Externally cooling the flask with water during the mixing will also reduce overheating.

NOTE 2—Loss of vapor results in concentration of the acid.

5.1.4 Weigh 25 g of reagent grade ferric sulfate (contains about 75 % $\text{Fe}_2(\text{SO}_4)_3$) and add to the H_2SO_4 solution. A trip balance may be used.

NOTE 3—Ferritic sulfate is a specific additive that establishes and controls the corrosion potential. No substitutions are permitted.

5.1.5 Add boiling aids.

5.1.6 Lubricate the ground glass of the con-

denser joint with silicone grease.

5.1.7 Cover the flask with the condenser and circulate cooling water.

5.1.8 Boil the solution until all ferritic sulfate is dissolved.

5.1.9 **Caution**—It has been reported that violent boiling resulting in acid spills can occur. It is important to ensure that the concentration of acid does not become more concentrated and that an adequate number of boiling chips (which are resistant to attack by the test solution) are present.⁵

6. Test Specimens

6.1 A specimen having a total surface area of 5 to 20 cm^2 is recommended.

6.2 The intent is to test a specimen representing as nearly as possible the material as used in service. The specimens should be cut to represent the grain flow direction that will see service, for example, specimens should not contain large cross-sectional areas unless it is the intent of the test to evaluate these. Only such surface finishing should be performed as is required to remove foreign material and obtain a standard, uniform finish as specified in 6.4. For very heavy sections, specimens should be machined to represent the appropriate surface while maintaining reasonable specimen size for convenience in testing. Ordinarily, removal of more material than necessary will have little influence on the test results. However, in the special case of surface decarburization or of carburization (the latter is sometimes encountered in tubing when lubricants or binders containing carbonaceous materials are employed), it may be possible by heavy grinding or machining to remove the affected layer completely. Such treatment of test specimens is not permissible, except in tests undertaken to demonstrate such surface effects.

6.3 When specimens are cut by shearing, the deformed material should be removed by machining or grinding prior to testing.

6.4 All surfaces of the specimen, including edges, should be finished using wet No. 80- or day No. 120-grit abrasive paper. If dry abrasive paper is used, polish slowly to avoid overheating. Sand blasting should not be used.

6.5 Residual oxide scale causes galvanic ac-

⁴ Stopcock grease has been found satisfactory for this purpose.

⁵ Amphoteric alundum granules, Hengar Granules, from the Hengar Co., Philadelphia, PA have been found satisfactory for this purpose.

tion and consequent activation in the test solution. Therefore, the formation of oxide scale in stamped codes must be prevented, and all traces of oxide scale formed during heat treatment must be thoroughly removed.

6.6 The specimen dimensions should be measured including the edges and inner surfaces of any holes and the total exposed area calculated.

6.7 The specimen should then be degreased and dried using suitable nonchlorinated agents such as soap and acetone and then weighed to the nearest 0.001 g.

7. Procedure

7.1 Place the specimen in the glass cradle, remove the condenser, immerse the cradle by means of a hook in the actively boiling solution (Fig. 1), and immediately replace the condenser. A fresh solution should be used for each test.

7.2 Mark the liquid level on the flask with wax crayon to provide a check on vapor loss which would result in concentration of the acid. If there is an appreciable change in the level (a 0.5-cm or more drop), repeat the test with fresh solution and with a fresh specimen or a reground specimen.

7.3 Continue immersion of the specimen for the length of time specified in Section 2, then remove the specimen, rinse in water and acetone, and dry.

7.4 Weigh the specimen and subtract this mass from the original mass.

7.5 No intermediate weighing are necessary, except as noted in 7.7. The tests can be run without interruption. However, if preliminary results are desired, the specimen can be removed at any time for weighing.

7.6 No replacement of acid is necessary during the test periods.

7.7 If the corrosion rate is extraordinarily high in Method A, as evidenced by a change in color (green) of the solution, additional ferric sulfate must be added during the test. The amount of ferric sulfate that must be added if the total mass loss of all specimens exceeds 2 g, as indicated by an intermediate weight, is 10 g for each 1 g of dissolved alloy. This does not apply to Method B.

7.8 In Method A, several specimens of the same alloy may be tested simultaneously. The number (3 or 4) is limited only by the number of glass cradles that can be fitted into the flask and the consumption of ferric sulfate. Only one

sample of Alloy N10276 should be tested in a flask for Method B.

7.9 During testing, there is some deposition of iron oxides on the upper part for the Erlenmeyer flask. This can be readily removed after test completion by boiling a solution of 10 % hydrochloric acid (HCl) in the flask.

8. Calculation and Interpretation of Results

8.1 *Calculation*—Measure the effect of the acid solution on the material by determining the loss of mass of the specimen. Calculate the corrosion rates as follows:

$$\text{Corrosion Rate} = (K \times W)/A \times T \times D$$

where

K = a constant (see the following paragraph),
 T = time of exposure, h, to the nearest 0.01 h,
 A = area, cm^2 , to the nearest 0.01 cm^2 ,
 W = mass loss, g, to the nearest 0.001 g, and
 D = density, g/cm^3 (see Table 2).

Many different units are used to express corrosion rates. Using the above units for T , A , W , and D , the corrosion rate can be calculated in a variety of units with the following appropriate value of K :

Corrosion Rate Units Desired	Constant K in Corrosion Rate Equation (A)
mils per year (mpy)	3.45×10^6
inches per year (ipy)	3.45×10^3
inches per month (ipm)	2.87×10^2
millimeters per year (mm/Y)	8.76×10^4
micrometers per year ($\mu\text{m}/\text{Y}$)	8.76×10^7
picometers per second (pm/s)	2.78×10^6
grams per square meter-hour ($\text{g}/\text{m}^2\text{-h}$)	$1.00 \times 10^4 \times D^B$
milligrams per square decimeter-day (mdd)	$2.40 \times 10^6 \times D^B$
micrograms per square meter-second ($\mu\text{g}/\text{m}^2\text{-s}$)	$2.78 \times 10^6 \times D^B$

^A If desired, these constants may also be used to convert corrosion rates from one set of units to another. To convert a corrosion rate in units X to a rate in units Y , multiply by K_Y/K_X . For example:

$$15 \text{ mpy} = 15 \times [(2.78 \times 10^6)/(3.45 \times 10^6)] \text{ pm/s} \\ = 12.1 \text{ pm/s}$$

^B Density is not needed to calculate the corrosion rate in these units. The density in the constant K cancels out the density in the corrosion rate equation.

8.2 *Interpretation of Results*—The presence of intergranular attack is usually determined by comparing the calculated corrosion rate to that for properly annealed material. Even in the absence of intergranular attack, the rate of general or grain-face corrosion of properly annealed material will vary from one alloy to another. These

differences are demonstrated in Refs. (1-7).⁶

8.3 As an alternative or in addition to calculating a corrosion rate from mass loss data, metallographic examination may be used to evaluate the degree of intergranular attack. However, metallography is of limited use for evaluating the detrimental effects of prior thermal treatment when the grain size is very small or when there is considerable precipitation of deleterious phases within the grains themselves. In these cases, high corrosion rates may be associated with a general roughening of the surface rather than the more typical undermining and dislodging of grains.

9. Precision and Bias

9.1 The precision and bias for this method are discussed in the references for Method A (1-7) and are dependent on the alloy examined by the method.

METHOD B—23 % H₂SO₄ + 1.2 % HCl + 1 % FeCl₃ + 1 % CuCl₂ Test

10. Significance and Use

10.1 The boiling 23 % H₂SO₄ + 1.2 % HCl + 1 % FeCl₃ + 1 % CuCl₂ acid mixture test may be applied to the following alloy in the wrought condition:

Alloy	Testing Time, h
N10276	24

10.2 Nominal composition of this alloy is given in Table 1.

10.3 This practice may be used to evaluate as-received material and to evaluate the effects of subsequent heat treatments. In the case of nickel-rich, chromium-bearing alloys, the method may be applied only to wrought products. The test is not applicable to cast products.

11. Apparatus

11.1 See Section 3.

12. Test Solution

12.1 23 % H₂SO₄ + 1.2 % HCl + 1 % FeCl₃ + 1 % CuCl₂ Acid Mixture Test—Prepare 600 mL of the solution as follows:

12.1.1 **Caution**—Protect the eyes and use rubber gloves for handling acid. Place the test flask under a hood.

12.1.2 First, weigh 10 g of reagent-grade ferric chloride (FeCl₃ 6H₂O) and put into Erlenmeyer flask.

12.1.3 Then weigh 7.2 g of reagent grade cupric chloride (CuCl₂ 2H₂O) and add to Erlenmeyer flask.

12.1.4 Measure 475 mL of distilled water in a 500-mL graduate and pour into the Erlenmeyer flask.

12.1.4 Then measure 90 mL of reagent-grade sulfuric acid (H₂SO₄) of a concentration that must be in the range from 95.0 to 98.0 weight % in a 100-mL graduate. Add the acid slowly to the water in the Erlenmeyer flask to avoid boiling by the heat evolved. Externally cooling the flask with water during the mixing will reduce overheating.

NOTE 4—Loss of vapor results in concentration of the acid.

12.1.5 Measure 18 mL of reagent-grade hydrochloric acid (HCl) of a concentration that must be in the range from 36.5 to 38 weight % in a 25-mL graduate. Add the acid slowly to the solution to avoid over heating and vapor loss.

12.1.6 Add boiling aids.

12.1.7 Lubricate the ground glass of the condenser joint with silicone grease.

12.1.8 Cover the flask with the condenser and circulate cooling water.

12.1.9 Boil the solution until all ferric chloride and cupric chloride are dissolved.

12.1.10 **Caution**—It has been reported that violent boiling resulting in acid spills can occur. It is important to ensure that the concentration of acid does not become more concentrated and that an adequate number of boiling chips (which are resistant to attack by the test solution) are present.⁵

13. Test Specimens

13.1 See Section 5.

14. Procedure

14.1 See Section 6.

15. Calculation and Interpretation of Results

15.1 See Section 7.

16. Precision and Bias

16.1 The precision and bias for this method are discussed in the references of Method B (6, 7).

⁶ The boldface numbers refer to the list of references at the end of these test methods.

TABLE 1 Limiting Compositions of Nickel-Rich, Chromium-Bearing Alloys

Composition, Weight %																
	Nickel	Chro- mium	Iron	Molyb- denum	Manga- nese, max	Car- bon, max	Copper	Sili- con, max	Sulfur, max	Alumi- num	Tita- nium	Colum- bium and Tanta- lum	Tung- sten	Phos- phorus, max	Vana- dium, max	Co- balt, max
Hastelloy Alloy C	balance	14.5 to 16.5	4.0 to 7.0	15.0 to 17.0	1.0	0.08	...	1.0	0.030	...	...	...	3.0 to 4.5	0.040	0.35	2.5
Hastelloy Alloy C-276	balance	14.5 to 16.5	4.0 to 7.0	15.0 to 17.0	1.0	0.02	...	0.05	0.030	...	...	...	3.0 to 4.5	0.040	0.35	2.5
Hastelloy Alloy G	balance	21.0 to 23.5	18.0 to 21.0	5.5 to 7.50	1.0 to 2.0	0.05	1.50 to 2.50	1.00	0.030	...	...	1.75 to 2.50	1.0 max	0.040	...	2.5
Carpenter Stainless No. 20 Cb-3	32.0 to 38.0	19.0 to 21.0	balance	2.0 to 3.0	2.0	0.07	3.0 to 4.0	1.0	0.035	...	...	8xC to 1.00	...	0.045	...	...
Inconel Alloy 600	72.0 ^A min	14.0 to 17.0	6.0 to 10.0	...	1.0	0.15	0.5	0.5	0.015	...	...	...	...	...	...	...
Inconel Alloy 625	balance	20.0 to 23.0	5.0 max	8.0 to 10.0	0.50	0.10	...	0.5	0.015	0.40	0.40	3.15 to 4.15	...	0.015	...	1.0 ^B max
Incoloy Alloy 800	30.0 to 35.0	19.0 to 23.0	balance	...	1.5	0.10	0.75	1.0	0.015	0.15 to 0.60	0.15 to 0.60	...	...	...	...	...
Incoloy Alloy 825	38.0 to 46.0	19.5 to 23.5	balance	2.5 to 3.5	1.0	0.05	1.5 to 3.0	0.5	0.03	0.2	0.6 to 1.2	...	...	...	...	...

^A = cobalt counting as nickel.^B = if determined.

TABLE 2 Density

Alloy	Density, g/cm ³
N10276	8.92
N06455	8.92
N06007	8.28
N06985	8.28
N08020	8.05
N06600	8.41
N06625	8.44
N08800	8.03
N08825	8.14

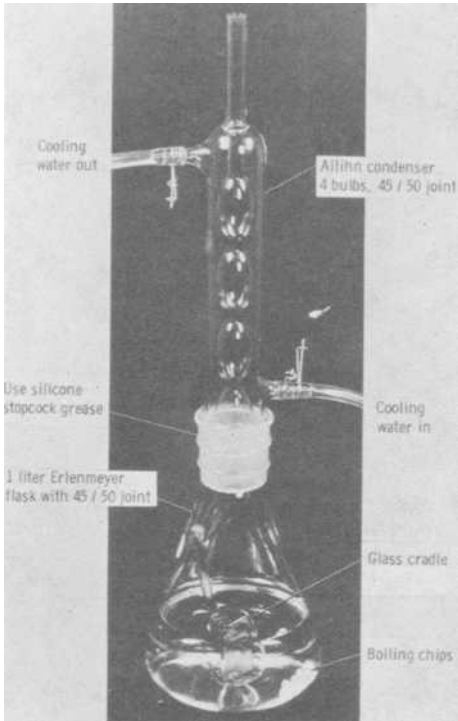


FIG. 1 Apparatus for Ferric Sulfate-Sulfuric Acid Test

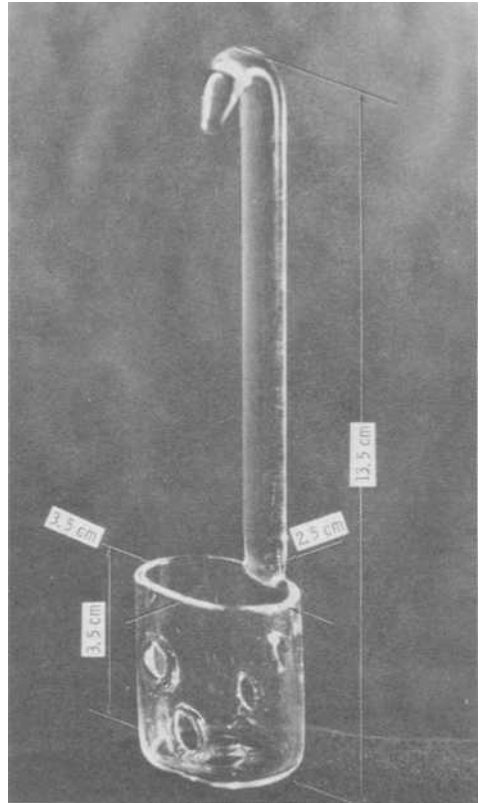


FIG. 2 Glass Cradle

REFERENCES

- (1) For original descriptions of the use of the ferric sulfate-sulfuric acid test for nickel-rich, chromium-bearing alloys, see Streicher, M. A., "Relationship of Heat Treatment and Microstructure to Corrosion Resistance in Wrought Ni-Cr-Mo Alloys," *Corrosion*, Vol 19, 1963, pp. 272-284.
- (2) For application of the ferric sulfate-sulfuric acid test to other nickel-rich, chromium-bearing alloys, see Brown, M. H., "Relationship of Heat Treatment to the Corrosion Resistance of Stainless Alloys," *Corrosion*, Vol 25, 1969, pp. 438-443.
- (3) For data on HASTELLOY Alloy C-276, see Leonard, R. B., "Thermal Stability of HASTELLOY Alloy C-276," *Corrosion*, Vol 25, 1969, pp. 222-228.
- (4) For data on Carpenter 20CB-3, see Henthorne,

- M. and DeBold, T. A., "Intergranular Corrosion Resistance of Carpenter 20CB-3," *Corrosion*, Vol 27, 1971, pp. 255-262.
- (5) Streicher, M. A., "Effect of Composition and Structure on Crevice, Intergranular, and Stress Corrosion of Some Wrought Ni-Cr-Mo Alloys," *Corrosion*, Vol 32, 1976, pp. 79-93.
- (6) Manning, P. E., "An Improved Intergranular Corrosion Test for HASTELLOY alloy C-276," *ASTM International Symposium on Laboratory Corrosion and Standards*, Bal Harbour, FL, November 14-16, 1983.
- (7) ASTM Research Report No. G-1-1002, "Round Robin on New Immersion Test for Detection of Intergranular Corrosion of HASTELLOY alloy C-276."

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Standard Practice for LABORATORY IMMERSION CORROSION TESTING OF METALS¹

This standard is issued under the fixed designation G 31; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{ε1} NOTE—Editorial changes were made throughout in May 1985.

1. Scope

1.1 This practice² describes accepted procedures for and factors that influence laboratory immersion corrosion tests, particularly mass loss tests. These factors include specimen preparation, apparatus, test conditions, methods of cleaning specimens, evaluation of results, and calculation and reporting of corrosion rates. This practice also emphasizes the importance of recording all pertinent data and provides a checklist for reporting test data. Other ASTM procedures for laboratory corrosion tests are tabulated in the Appendix.

NOTE 1: **Caution**—In many cases the corrosion product on the reactive metals titanium and zirconium is a hard and tightly bonded oxide that defies removal by chemical or ordinary mechanical means. In many such cases, corrosion rates are established by weight gain rather than weight loss.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Applicable Documents

2.1 ASTM Standards:

A 262 Practices for Detecting Susceptibility to Intergranular Attack in Stainless Steels^{3,4}

E 8 Methods for Tension Testing of Metallic Materials⁵

G 1 Practice for Preparing, Cleaning, and Eval-

uating Corrosion Test Specimens⁴

G 4 Method for Conducting Corrosion Coupon Tests in Plant Equipment⁴

G 16 Practice for Applying Statistics to Analysis of Corrosion Data⁴

G 46 Recommended Practice for Examination and Evaluation of Pitting Corrosion⁴

3. Significance and Use

3.1 Corrosion testing by its very nature precludes complete standardization. This practice, rather than a standardized procedure, is presented as a guide so that some of the pitfalls of such testing may be avoided.

3.2 Experience has shown that all metals and alloys do not respond alike to the many factors that affect corrosion and that “accelerated” corrosion tests give indicative results only, or may even be entirely misleading. It is impractical to propose an inflexible standard laboratory corrosion testing procedure for general use, except for material qualification tests where standardization is obviously required.

3.3 In designing any corrosion test, consideration must be given to the various factors dis-

¹ This practice is under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.

Current edition approved May 30, 1972. Published July 1972.

² This practice is based upon NACE Standard TM-01-69, “Test Method-Laboratory Corrosion Testing of Metals for the Process Industries”, with modifications to relate more directly to Practices G 1 and G 31 and Method G 4.

³ *Annual Book of ASTM Standards*, Vol 01.03.

⁴ *Annual Book of ASTM Standards*, Vol 03.02.

⁵ *Annual Book of ASTM Standards*, Vol 03.01.

cussed in this practice, because these factors have been found to affect greatly the results obtained.

4. Interferences

4.1 The methods and procedures described herein represent the best current practices for conducting laboratory corrosion tests as developed by corrosion specialists in the process industries. For proper interpretation of the results obtained, the specific influence of certain variables must be considered. These include:

4.1.1 Metal specimens immersed in a specific hot liquid may not corrode at the same rate or in the same manner as in equipment where the metal acts as a heat transfer medium in heating or cooling the liquid. If the influence of heat transfer effects is specifically of interest, specialized procedures (in which the corrosion specimen serves as a heat transfer agent) must be employed (1).⁶

4.1.2 In laboratory tests, the velocity of the environment relative to the specimens will normally be determined by convection currents or the effects induced by aeration or boiling or both. If the specific effects of high velocity are to be studied, special techniques must be employed to transfer the environment through tubular specimens or to move it rapidly past the plane face of a corrosion coupon (2). Alternatively, the coupon may be rotated through the environment, although it is then difficult to evaluate the velocity quantitatively because of the stirring effects incurred.

4.1.3 The behavior of certain metals and alloys may be profoundly influenced by the presence of dissolved oxygen. If this is a factor to be considered in a specific test, the solution should be completely aerated or deaerated in accordance with 8.7.

4.1.4 In some cases, the rate of corrosion may be governed by other minor constituents in the solution, in which case they will have to be continually or intermittently replenished by changing the solution in the test.

4.1.5 Corrosion products may have undesirable effects on a chemical product. The amount of possible contamination can be estimated from the loss in mass of the specimen, with proper application of the expected relationships among (1) the area of corroding surface, (2) the mass of the chemical product handled, and (3) the duration of contact of a unit of mass of the chemical

product with the corroding surface.

4.1.6 Corrosion products from the coupon may influence the corrosion rate of the metal itself or of different metals exposed at the same time. For example, the accumulation of cupric ions in the testing of copper alloys in intermediate strengths of sulfuric acid will accelerate the corrosion of copper alloys, as compared to the rates that would be obtained if the corrosion products were continually removed. Cupric ions may also exhibit a passivating effect upon stainless steel coupons exposed at the same time. In practice, only alloys of the same general type should be exposed in the testing apparatus.

4.1.7 Coupon corrosion testing is predominantly designed to investigate general corrosion. There are a number of other special types of phenomena of which one must be aware in the design and interpretation of corrosion tests.

4.1.7.1 Galvanic corrosion may be investigated by special devices which couple one coupon to another in electrical contact. The behavior of the specimens in this galvanic couple are compared with that of insulated specimens exposed on the same holder and the galvanic effects noted. It should be observed, however, that galvanic corrosion can be greatly affected by the area ratios of the respective metals, the distance between the metals and the resistivity of the electrolyte. The coupling of corrosion coupons then yields only qualitative results, as a particular coupon reflects only the relationship between these two metals at the particular area ratio involved.

4.1.7.2 Crevice corrosion or concentration cell corrosion may occur where the metal surface is partially blocked from the corroding liquid as under a spacer or supporting hook. It is necessary to evaluate this localized corrosion separately from the overall mass loss.

4.1.7.3 Selective corrosion at the grain boundaries (for example, intergranular corrosion of sensitized austenitic stainless steels) will not be readily observable in mass loss measurements unless the attack is severe enough to cause grain dropping, and often requires microscopic examination of the coupons after exposure.

4.1.7.4 Dealloying or "parting" corrosion is a condition in which one constituent is selectively removed from an alloy, as in the dezincification

⁶ The boldface numbers in parentheses refer to the list of references at the end of this practice.

of brass or the graphitization of cast iron. Close attention and a more sophisticated evaluation than a simple mass loss measurement is required to detect this phenomenon.

4.1.7.5 Certain metals and alloys are subject to a highly localized type of attack called pitting corrosion. This cannot be evaluated by mass loss alone. The reporting of nonuniform corrosion is discussed below. It should be appreciated that pitting is a statistical phenomenon and that the incidence of pitting may be directly related to the area of metal exposed. For example, a small coupon is not as prone to exhibit pitting as a large one and it is possible to miss the phenomenon altogether in the corrosion testing of certain alloys, such as the AISI Type 300 series stainless steels in chloride contaminated environments.

4.1.7.6 All metals and alloys are subject to stress-corrosion cracking under some circumstances. This cracking occurs under conditions of applied or residual tensile stress, and it may or may not be visible to the unaided eye or upon casual inspection. A metallographic examination may confirm the presence of stress-corrosion cracking. It is imperative to note that this usually occurs with no significant loss in mass of the test coupon, although certain refractory metals are an exception to these observations. Generally, if cracking is observed on the coupon, it can be taken as positive indication of susceptibility, whereas failure to effect this phenomenon simply means that it did not occur under the duration and specific conditions of the test. Separate and special techniques are employed for the specific evaluation of the susceptibility of metals and alloys to stress corrosion cracking (see Ref. (3)).

5. Apparatus

5.1 A versatile and convenient apparatus should be used, consisting of a kettle or flask of suitable size (usually 500 to 5000 mL), a reflux condenser with atmospheric seal, a sparger for controlling atmosphere or aeration, a thermowell and temperature-regulating device, a heating device (mantle, hot plate, or bath), and a specimen support system. If agitation is required, the apparatus can be modified to accept a suitable stirring mechanism, such as a magnetic stirrer. A typical resin flask setup for this type test is shown in Fig. 1.

5.2 The suggested components can be modified, simplified, or made more sophisticated to

fit the needs of a particular investigation. The suggested apparatus is basic and the apparatus is limited only by the judgment and ingenuity of the investigator.

5.2.1 A glass reaction kettle can be used where the configuration and size of the specimen will permit entry through the narrow kettle neck (for example, 45/50 ground-glass joint). For solutions corrosive to glass, suitable metallic or plastic kettles may be employed.

5.2.2 In some cases a wide-mouth jar with a suitable closure is sufficient when simple immersion tests at ambient temperatures are to be investigated.

5.2.3 Open-beaker tests should not be used because of evaporation and contamination.

5.2.4 In more complex tests, provisions might be needed for continuous flow or replenishment of the corrosive liquid, while simultaneously maintaining a controlled atmosphere.

6. Sampling

6.1 The bulk sampling of products is outside the scope of this practice.

7. Test Specimen

7.1 In laboratory tests, uniform corrosion rates of duplicate specimens are usually within $\pm 10\%$ under the same test conditions. Occasional exceptions, in which a large difference is observed, can occur under conditions of borderline passivity of metals or alloys that depend on a passive film for their resistance to corrosion. Therefore, at least duplicate specimens should normally be exposed in each test.

7.2 If the effects of corrosion are to be determined by changes in mechanical properties, untested duplicate specimens should be preserved in a noncorrosive environment at the same temperature as the test environment for comparison with the corroded specimens. The mechanical property commonly used for comparison is the tensile strength. Measurement of percent elongation is a useful index of embrittlement. The procedures for determining these values are shown in detail in Methods E 8.

7.3 The size and shape of specimens will vary with the purpose of the test, nature of the materials, and apparatus used. A large surface-to-mass ratio and a small ratio of edge area to total area are desirable. These ratios can be achieved through the use of square or circular specimens

of minimum thickness. Masking may also be used to achieve the desired area ratios but may cause crevice corrosion problems. Circular specimens should preferably be cut from sheet and not bar stock, to minimize the exposed end grain. Special coupons (for example, sections of welded tubing) may be employed for specific purposes.

7.3.1 A circular specimen of about 38-mm (1.5-in.) diameter is a convenient shape for laboratory corrosion tests. With a thickness of approximately 3 mm (0.125-in.) and an 8-mm ($\frac{5}{16}$ -in.) or 11-mm ($\frac{7}{16}$ -in.) diameter hole for mounting, these specimens will readily pass through a 45/50 ground-glass joint of a distillation kettle. The total surface area of a circular specimen is given by the following equation:

$$A = \pi/2(D^2 - d^2) + \pi t D + \pi t d$$

where:

t = thickness,

D = diameter of the specimen, and

d = diameter of the mounting hole.

7.3.1.2 If the hole is completely covered by the mounting support, the last term ($\pi t d$) in the equation is omitted.

7.3.2 Strip coupons 50 by 25 by 1.6 or 3 mm (2 by 1 by $\frac{1}{16}$ or $\frac{1}{8}$ in.) may be preferred as corrosion specimens, particularly if interface or liquid line effects are to be studied by the laboratory tests (see Fig. 1), but the evaluation of such specific effects are beyond the scope of this practice.

7.3.3 All specimens should be measured carefully to permit accurate calculation of the exposed areas. A geometric area calculation accurate to $\pm 1\%$ is usually adequate.

7.4 More uniform results may be expected if a substantial layer of metal is removed from the specimens to eliminate variations in condition of the original metallic surface. This can be done by chemical treatment (pickling), electrolytic removal, or by grinding with a coarse abrasive paper or cloth such as No. 50, using care not to work harden the surface (see 5.7). At least 0.0025 mm (0.0001 in.) or 1.55 to 2.33 mg/cm.² (10 to 15 mg/in.²) should be removed. (If clad alloy specimens are to be used, special attention must be given to ensure that excessive metal is not removed.) After final preparation of the specimen surface, the specimens should be stored in a desiccator until exposure, if they are not used immediately. In special cases (for example, for aluminum and certain copper alloys), a mini-

mum of 24 h storage in a desiccator is recommended. The choice of a specific treatment must be considered on the basis of the alloy to be tested and the reasons for testing. A commercial surface may sometimes yield the most significant results. Too much surface preparation may remove segregated elements, surface contamination, etc., and therefore not be representative.

7.5 Exposure of sheared edges should be avoided unless the purpose of the test is to study effects of the shearing operation. It may be desirable to test a surface representative of the material and metallurgical conditions used in practice.

7.6 The specimen can be stamped with an appropriate identifying mark. If metallic contamination of the stamped area may influence the corrosion behavior, chemical cleaning must be employed to remove any traces of foreign particles from the surface of the coupon (for example, by immersion of stainless steel coupons in dilute nitric acid following stamping with steel dies).

7.6.1 The stamp, besides identifying the specimen, introduces stresses and cold work in the specimen that could be responsible for localized corrosion or stress-corrosion cracking, or both.

7.6.2 Stress-corrosion cracking at the identifying mark is a positive indication of susceptibility to such corrosion. However, the absence of cracking should not be interpreted as indicating resistance (see 4.1.7.6).

7.7 Final surface treatment of the specimens should include finishing with No. 120 abrasive paper or cloth or the equivalent, unless the surface is to be used in the mill finished condition. This resurfacing may cause some surface work hardening, to an extent which will be determined by the vigor of the surfacing operation, but is not ordinarily significant. The surface finish to be encountered in service may be more appropriate for some testing.

7.7.1 Coupons of different alloy compositions should never be ground on the same cloth.

7.7.2 Wet grinding should be used on alloys which work harden quickly, such as the austenitic stainless steels.

7.8 The specimens should be finally degreased by scrubbing with bleach-free scouring powder, followed by thorough rinsing in water and in a suitable solvent (such as acetone, methanol, or a mixture of 50 % methanol and 50 % ether), and air dried. For relatively soft metals (such as aluminum, magnesium, and copper), scrubbing with abrasive powder is not always needed and

can mar the surface of the specimen. Proper ultrasonic procedures are an acceptable alternate. The use of towels for drying may introduce an error through contamination of the specimens with grease or lint.

7.9 The dried specimens should be weighed on an analytical balance to an accuracy of at least ± 0.5 mg. If cleaning deposits (for example, scouring powder) remain or lack of complete dryness is suspected, then recleaning and drying is performed until a constant mass is attained.

7.10 The method of specimen preparation should be described when reporting test results, to facilitate interpretation of data by other persons.

7.11 The use of welded specimens is sometimes desirable, because some welds may be cathodic or anodic to the parent metal and may affect the corrosion rate.

7.11.1 The heat-affected zone is also of importance but should be studied separately, because welds on coupons do not faithfully reproduce heat input or size effects of full-size weldments.

7.11.2 Corrosion of a welded coupon is best reported by description and thickness measurements rather than a mils per year rate, because the attack is normally localized and not representative of the entire surface.

7.11.3 A complete discussion of corrosion testing of welded coupons or the effect of heat treatment on the corrosion resistance of a metal is not within the scope of this practice.

8. Test Conditions

8.1 Selection of the conditions for a laboratory corrosion test will be determined by the purpose of the test.

8.1.1 If the test is to be a guide for the selection of a material for a particular purpose, the limits of the controlling factors in service must be determined. These factors include oxygen concentration, temperature, rate of flow, pH value, composition, and other important characteristics of the solution.

8.2 An effort should be made to duplicate all pertinent service conditions in the corrosion test.

8.3 It is important that test conditions be controlled throughout the test in order to ensure reproducible results.

8.4 The spread in corrosion rate values for duplicate specimens in a given test probably

should not exceed $\pm 10\%$ of the average when the attack is uniform.

8.5 Composition of Solution:

8.5.1 Test solutions should be prepared accurately from chemicals conforming to the Specifications of the Committee on Analytical Reagents of the American Chemical Society⁷ and distilled water, except in those cases where naturally occurring solutions or those taken directly from some plant process are used.

8.5.2 The composition of the test solutions should be controlled to the fullest extent possible and should be described as completely and as accurately as possible when the results are reported.

8.5.2.1 Minor constituents should not be overlooked because they often affect corrosion rates.

8.5.2.2 Chemical content should be reported as percentage by weight of the solutions. Molarity and normality are also helpful in defining the concentration of chemicals in some test solutions.

8.5.3 If problems are suspected, the composition of the test solutions should be checked by analysis at the end of the test to determine the extent of change in composition, such as might result from evaporation or depletion.

8.5.4 Evaporation losses may be controlled by a constant level device or by frequent addition of appropriate solution to maintain the original volume within $\pm 1\%$. Preferably, the use of a reflux condenser ordinarily precludes the necessity of adding to the original kettle charge.

8.5.5 In some cases, composition of the test solution may change as a result of catalytic decomposition or by reaction with the test coupons. These changes should be determined if possible. Where required, the exhausted constituents should be added or a fresh solution provided during the course of the test.

8.5.6 When possible, only one type of metal should be exposed in a given test (see 4.1.6).

8.6 Temperature of Solution:

8.6.1 Temperature of the corroding solution should be controlled within $\pm 1^\circ\text{C}$ ($\pm 1.8^\circ\text{F}$) and

⁷ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

must be stated in the report of test results.

8.6.2 If no specific temperature, such as boiling point, is required or if a temperature range is to be investigated, the selected temperatures used in the test, and their respective duration, must be reported.

8.6.3 For tests at ambient temperature, the tests should be conducted at the highest temperature anticipated for stagnant storage in summer months. This temperature may be as high as from 40 to 45°C (104 to 113°F) in some areas. The variation in temperature should be reported also (for example, $40 \pm 2^\circ\text{C}$).

8.7 *Aeration of Solution:*

8.7.1 Unless specified, the solution should not be aerated. Most tests related to process equipment should be run with the natural atmosphere inherent in the process, such as the vapors of the boiling liquid.

8.7.2 If aeration is employed, the specimen should not be located in the direct air stream from the sparger. Extraneous effects can be encountered if the air stream impinges on the specimens.

8.7.3 If exclusion of dissolved oxygen is necessary, specific techniques are required, such as prior heating of the solution and sparging with an inert gas (usually nitrogen). A liquid atmospheric seal is required on the test vessel to prevent further contamination.

8.7.4 If oxygen saturation of the test solution is desired, this can best be achieved by sparging with oxygen. For other degrees of aeration, the solution should be sparged with air or synthetic mixtures of air or oxygen with an inert gas. Oxygen saturation is a function of the partial pressure of oxygen in the gas.

8.8 *Solution Velocity:*

8.8.1 The effect of velocity is not usually determined in normal laboratory tests, although specific tests have been designed for this purpose.

8.8.2 Tests at the boiling point should be conducted with the minimum possible heat input, and boiling chips should be used to avoid excessive turbulence and bubble impingement.

8.8.3 In tests below the boiling point, thermal convection generally is the only source of liquid velocity.

8.8.4 In test solutions with high viscosity, supplemental controlled stirring with a magnetic stirrer is recommended.

8.9 *Volume of Test Solution:*

8.9.1 The volume of the test solution should be large enough to avoid any appreciable change in its corrosivity during the test, either through exhaustion of corrosive constituents or by accumulation of corrosion products that might affect further corrosion.

8.9.2 Two examples of a minimum "solution volume-to-specimen area" ratio are 20 mL/cm² (125 mL/in.²) of specimen surface (Practice A 262), and 40 mL/cm² (250 mL/in.²).

8.9.3 When the test objective is to determine the effect of a metal or alloy on the characteristics of the test solution (for example, to determine the effects of metals on dyes), it is desirable to reproduce the ratio of solution volume to exposed metal surface that exists in practice. The actual time of contact of the metal with the solution must also be taken into account. Any necessary distortion of the test conditions must be considered when interpreting the results.

8.10 *Method of Supporting Specimens:*

8.10.1 The supporting device and container should not be affected by or cause contamination of the test solution.

8.10.2 The method of supporting specimens will vary with the apparatus used for conducting the test, but should be designed to insulate the specimens from each other physically and electrically and to insulate the specimens from any metallic container or supporting device used within the apparatus.

8.10.3 Shape and form of the specimen support should assure free contact of the specimen with the corroding solution, the liquid line, or the vapor phase as shown in Fig. 1. If clad alloys are exposed, special procedures will be required to ensure that only the cladding is exposed, unless the purpose is to test the ability of the cladding to protect cut edges in the test solution.

8.10.4 Some common supports are glass or ceramic rods, glass saddles, glass hooks, fluorocarbon plastic strings, and various insulated or coated metallic supports.

8.11 *Duration of Test:*

8.11.1 Although duration of any test will be determined by the nature and purpose of the test, an excellent procedure for evaluating the effect of time on corrosion of the metal and also on the corrosiveness of the environment in laboratory tests has been presented by Wachter and Treseder (4). This technique is called the "planned interval test," and the procedure and evaluation of results

are given in Table 1. Other procedures that require the removal of solid corrosion products between exposure periods will not measure accurately the normal changes of corrosion with time.

8.11.2 Materials that experience severe corrosion generally do not ordinarily need lengthy tests to obtain accurate corrosion rates. However, there are cases where this assumption is not valid. For example, lead exposed to sulfuric acid corrodes at an extremely high rate at first, while building a protective film; then the rates decrease considerably so that further corrosion is negligible. The phenomenon of forming a protective film is observed with many corrosion-resistant materials. Therefore, short tests on such materials would indicate a high corrosion rate and be completely misleading.

8.11.3 Short-time tests also can give misleading results on alloys that form passive films, such as stainless steels. With borderline conditions, a prolonged test may be needed to permit breakdown of the passive film and subsequent more rapid attack. Consequently, tests run for long periods are considerably more realistic than those conducted for short durations. This statement must be qualified by stating that corrosion should not proceed to the point where the original specimen size or the exposed area is drastically reduced or where the metal is perforated.

8.11.4 If anticipated corrosion rates are moderate or low, the following equation gives the suggested test duration:

$$\text{Hours} = 2000/(\text{corrosion rate in mpy})$$

where mpy = mils per year (see 11.2.1 and Note 2 for conversion to other units).

8.11.4.1 *Example*—Where the corrosion rate is 10 mpy, the test should run for at least 200 h.

8.11.4.2 This method of estimating test duration is useful only as an aid in deciding, after a test has been made, whether or not it is desirable to repeat the test for a longer period. The most common testing periods are 48 to 168 h (2 to 7 days).

8.11.5 In some cases, it may be necessary to know the degree of contamination caused by the products of corrosion. This can be accomplished by analysis of the solution after corrosion has occurred. The corrosion rate can be calculated from the concentration of the matrix metal found in the solution and it can be compared to that determined from the mass loss of the specimens.

However, some of the corrosion products usually adhere to the specimen as a scale and the corrosion rate calculated from the metal content in the solution is not always correct.

8.12 The design of corrosion testing programs is further discussed in Practice G 16.

9. Methods of Cleaning Specimens after Test

9.1 Before specimens are cleaned, their appearance should be observed and recorded. Location of deposits, variations in types of deposits, or variations in corrosion products are extremely important in evaluating localized corrosion, such as pitting and concentration cell attack.

9.2 Cleaning specimens after the test is a vital step in the corrosion test procedure and if not done properly, can cause misleading results.

9.2.1 Generally, the cleaning procedure should remove all corrosion products from specimens with a minimum removal of sound metal.

9.2.2 Set rules cannot be applied to specimen cleaning, because procedures will vary, depending on the type of metal being cleaned and on the degree of adherence of corrosion products.

9.3 Cleaning methods can be divided into three general categories: mechanical, chemical, and electrolytic.

9.3.1 Mechanical cleaning includes scrubbing, scraping, brushing, mechanical shocking, and ultrasonic procedures. Scrubbing with a bristle brush and mild abrasive is the most popular of these methods. The others are used principally as a supplement to remove heavily encrusted corrosion products before scrubbing. Care should be used to avoid the removal of sound metal.

9.3.2 Chemical cleaning implies the removal of material from the surface of the specimen by dissolution in an appropriate chemical solution. Solvents such as acetone, carbon tetrachloride, and alcohol are used to remove oil, grease, or resin and are usually applied prior to other methods of cleaning. Chemicals are chosen for application to a specific material. Methods for chemical cleaning after testing of specific metals and alloys are described in Practice G 1.

9.3.3 Electrolytic cleaning should be preceded by scrubbing to remove loosely adhering corrosion products. A method of electrolytic cleaning is described in Practice G 1.

9.3.3.1 Precautions must be taken to ensure good electrical contact with the specimen, to avoid contamination of the solution with easily

reducible metal ions, and to ensure that inhibitor decomposition has not occurred.

9.4 Whatever treatment is used to clean specimens after a corrosion test, its effect in removing metal should be determined and the mass loss should be corrected accordingly. A “blank” specimen should be weighed before and after exposure to the cleaning procedure to establish this mass loss (see also Practice G 1). Careful observation is needed to ensure that pitting does not occur during cleaning.

9.4.1 Following removal of all scale, the specimen should be treated as discussed in 5.8.

9.4.2 The description of the cleaning method should be included with the data reported.

10. Interpretation of Results

10.1 After corroded specimens have been cleaned, they should be reweighed with an accuracy corresponding to that of the original weighing. The mass loss during the test period can be used as the principal measure of corrosion.

10.2 After the specimens have been reweighed, they should be examined carefully for the presence of any pits. If there are any pits, the average and maximum depths of pits are determined with a pit gage or a calibrated microscope which can be focused first on the edges and then on the bottoms of the pits. The degree of lateral spreading of pits may also be noted.

10.2.1 Pit depths should be reported in millimeters or thousandths of an inch for the test period and not interpolated or extrapolated to millimeters per year, thousandths of an inch per year, or any other arbitrary period because rarely, if ever, is the rate of initiation or propagation of pits uniform.

10.2.2 The size, shape, and distribution of pits should be noted. A distinction should be made between those occurring underneath the supporting devices (concentration cells) and those on the surfaces that were freely exposed to the test solution (see Recommended Practice G 46).

10.3 If the material being tested is suspected of being subject to dealloying forms of corrosion such as dezincification or to intergranular attack, a cross section of the specimen should be microscopically examined for evidence of such attack.

10.4 The specimen may be subjected to simple bending tests to determine whether any embrittlement attack has occurred.

10.5 It may be desirable to make quantitative

mechanical tests, comparing the exposed specimens with uncorroded specimens reserved for the purpose, as described in 7.2.

11. Calculating Corrosion Rates

11.1 Calculating corrosion rates requires several pieces of information and several assumptions:

11.1.1 The use of corrosion rates implies that all mass loss has been due to general corrosion and not to localized corrosion, such as pitting or intergranular corrosion of sensitized areas on welded coupons. Localized corrosion is reported separately.

11.1.2 The use of corrosion rates also implies that the material has not been internally attacked as by dezincification or intergranular corrosion.

11.1.3 Internal attack can be expressed as a corrosion rate if desired. However, the calculations must not be based on weight loss (except in qualification tests such as Practice A 262), which is usually small but on microsections which show depth of attack.

11.2 Assuming that localized or internal corrosion is not present or is recorded separately in the report, the average corrosion rate can be calculated by the following equation:

Corrosion rate = (K × W)/(A × T × D)

where:

K = a constant (see below)

T = time of exposure in hours to the nearest 0.01 h,

A = area in cm² to the nearest 0.01 cm²,

W = mass loss in g, to nearest 1 mg (corrected for any loss during cleaning (see 9.4)), and

D = density in g/cm³, (see Appendix X1 of Practice G 1).

11.2.1 Many different units are used to express corrosion rates. Using the above units for T, A, W, and D, the corrosion rate can be calculated in a variety of units with the following appropriate value of K:

Corrosion Rate Units Desired	Constant (K) in Corrosion Rate Equation
mils per year (mpy)	3.45 × 10 ⁶
inches per year (ipy)	3.45 × 10 ³
inches per month (ipm)	2.87 × 10 ²
millimetres per year (mm/y)	8.76 × 10 ⁴
micrometres per year (µm/y)	8.76 × 10 ⁷
picometres per second (pm/s)	2.78 × 10 ⁶

Corrosion Rate Units Desired	Constant (K) in Corrosion Rate Equation
grams per square metre per hour (g/m ² ·h)	$1.00 \times 10^4 \times D^4$
milligrams per square decimetre per day (mdd)	$2.40 \times 10^6 \times D^4$
micrograms per square metre per second (μg/m ² ·s)	$2.78 \times 10^6 \times D^4$

⁴ Density is not needed to calculate the corrosion rate in these units. The density in the constant *K* cancels out the density in the corrosion rate equation.

NOTE 2—If desired, these constants may also be used to convert corrosion rates from one set of units to another. To convert a corrosion rate in units *X* to a rate of units *Y*, multiply by K_Y/K_X for example:

$$15 \text{ mpy} = 15 \times [(2.78 \times 10^6)/(3.45 \times 10^6)] \text{ pm/s} \\ = 12.1 \text{ pm/s}$$

12. Reporting the Data

12.1 The importance of reporting all data as completely as possible cannot be overemphasized.

12.2 Expansion of the testing program in the future or correlating the results with tests of other investigators will be possible only if all pertinent information is properly recorded.

12.3 The following checklist is a recommended guide for reporting all important information and data.

12.3.1 Corrosive media and concentration (any changes during test).

12.3.2 Volume of test solution.

12.3.3 Temperature (maximum, minimum, average).

12.3.4 Aeration (describe conditions or technique).

12.3.5 Agitation (describe conditions or technique).

12.3.6 Type of apparatus used for test.

12.3.7 Duration of each test.

12.3.8 Chemical composition or trade name of metals tested.

12.3.9 Form and metallurgical conditions of specimens.

12.3.10 Exact size, shape, and area of specimens.

12.3.11 Treatment used to prepare specimens for test.

12.3.12 Number of specimens of each material tested, and whether specimens were tested separately or which specimens tested in the same container.

12.3.13 Method used to clean specimens after exposure and the extent of any error expected by this treatment.

12.3.14 Initial and final masses and actual mass losses for each specimen.

12.3.15 Evaluation of attack if other than general, such as crevice corrosion under support rod, pit depth and distribution, and results of microscopical examination or bend tests.

12.3.16 Corrosion rates for each specimen.

12.4 Minor occurrences or deviations from the proposed test program often can have significant effects and should be reported if known.

12.5 Statistics can be a valuable tool for analyzing the results from test programs designed to generate adequate data. Excellent references for the use of statistics in corrosion studies include Ref. (5) through (7) and in Practice G 16.

REFERENCES

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- (2) U.S. Patent 3,228,236, 1969.
- (3) "Stress Corrosion Test Environments and Test Durations," *Symposium on Stress Corrosion Testing*, ASTM STP 425, ASTM, 1967.
- (4) Wachter, A. and Treseder, R. S., "Corrosion Testing Evaluation of Metals for Process Equipment," *Chemical Engineering Progress*, Vol 43, June 1947, pp. 315-326.
- (5) Mickley, H. S., Sherwood, T. K., and Reed, C. E. editors, *Applied Mathematics in Chemical Engineering* 2nd Edition, McGraw-Hill Book Co., New York, NY 1957.
- (6) Youden, W. J., *Experimentation and Measurement*, National Science Teachers Assn., Washington, DC, 1962.
- (7) Booth, F. F. and Tucker, G. E. G., "Statistical Distribution of Endurance in Electrochemical Stress-Corrosion Tests," *Corrosion*, Vol 21, No. 5, May 1965, pp. 173-177.
- (8) Champion, F. A., *Corrosion Testing Procedures*, 2nd Edition, John Wiley & Sons, Inc., New York, NY, 1965.

TABLE 1 Planned Interval Corrosion Test

(Reprinted by permission from *Chemical Engineering Progress*, June 1947.)

Identical specimens all placed in the same corrosive fluid. Imposed conditions of the test kept constant for entire time $t + 1$. Letters, A_1 , A_t , A_{t+1} , B , represent corrosion damage experienced by each test specimen. A_2 is calculated by subtracting A_t from A_{t+1} .

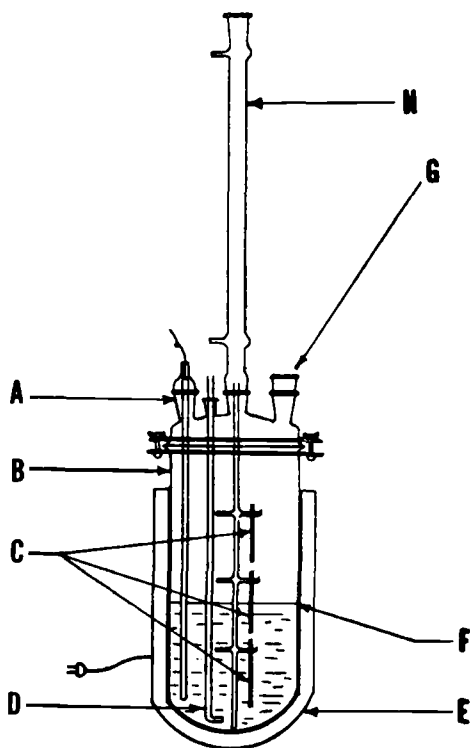
Occurrences During Corrosion Test		Criteria
Liquid corrosiveness	unchanged	$A_1 = B$
	decreased	$B < A_1$
	increased	$A_1 < B$
Metal corrodibility	unchanged	$A_2 = B$
	decreased	$A_2 < B$
	increased	$B < A_2$
Combinations of Situations		
Liquid corrosiveness	Metal corrodibility	Criteria
1. unchanged	unchanged	$A_1 = A_2 = B$
2. unchanged	decreased	$A_2 < A_1 = B$
3. unchanged	increased	$A_1 = B < A_2$
4. decreased	unchanged	$A_2 = B < A_1$
5. decreased	decreased	$A_2 < B < A_1$
6. decreased	increased	$A_1 > B < A_2$
7. increased	unchanged	$A_1 < A_2 = B$
8. increased	decreased	$A_1 < B > A_2$
9. increased	increased	$A_1 < B < A_2$

Example: Conditions: Duplicate strips of low-carbon steel, each $\frac{3}{4}$ by 3 in., immersed in 200 mL of 10 % AlCl_3 -90 % SbCl_3 mixture through which dried HCl gas was slowly bubbled at atmospheric pressure. Temperature 90°C .

	Interval, days	Mass Loss, mg	Penetration, mils	Apparent Corrosion Rate, mpy
A_1	0-1	1080	1.69	620
A_t	0-3	1430	2.24	270
A_{t+1}	0-4	1460	2.29	210
B	3-4	70	0.11	40
A_2	calc. 3-4	30	0.05	18

Example: $A_2 < B < A_1$
 $0.05 < 0.11 < 1.69$

Therefore, liquid markedly decreased in corrosiveness during test, and formation of partially protective scale on the steel was indicated.



NOTE—The flask can be used as a versatile and convenient apparatus to conduct simple immersion tests. Configuration of top to flask is such that more sophisticated apparatus can be added as required by the specific test being conducted. *A* = thermowell, *B* = resin flask, *C* = specimens hung on supporting device, *D* = air inlet, *E* = heating mantle, *F* = liquid interface, *G* = opening in flask for additional apparatus that may be required, and *H* = reflux condenser.

FIG. 1 Typical Resin Flask

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for EXFOLIATION CORROSION SUSCEPTIBILITY IN 2XXX AND 7XXX SERIES ALUMINUM ALLOYS (EXCO TEST)¹

This standard is issued under the fixed designation G 34; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This method has been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 This method describes a procedure for constant immersion exfoliation corrosion (EXCO) testing of high-strength 2XXX and 7XXX series aluminum alloys.

1.2 This method applies only to wrought products such as sheet, plate, extrusions, and forgings.

1.3 This method can be used with any form of specimen or part that can be immersed in the test solution.

2. Applicable Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

E 3 Methods of Preparation of Metallographic Specimens³

G 15 Definitions of Terms Relating to Corrosion and Corrosion Testing⁴

3. Summary of Method

3.1 This method provides an accelerated exfoliation corrosion test for 2XXX and 7XXX series aluminum alloys that involves the continuous immersion of test materials in a solution containing 4 M sodium chloride, 0.5 M potassium nitrate, and 0.1 M nitric acid at $25 \pm 3^\circ\text{C}$ ($77 \pm 5^\circ\text{F}$). The susceptibility to exfoliation is determined by visual examination, with performance ratings established by reference to standard photographs.

4. Significance and Use

4.1 Use of this method provides a useful prediction of the exfoliation corrosion behavior of these alloys in various types of outdoor service, especially in marine and industrial en-

vironments.⁵ The test solution is very corrosive and represents the more severe types of environmental service, excluding, of course, unusual chemicals not likely to be encountered in "natural" environments.

4.2 The exfoliation ratings were arbitrarily chosen to illustrate a wide range in resistance to exfoliation in this test. However, it remains to be determined whether correlations can be established between EXCO test ratings and realistic service conditions for a given alloy. It is an ongoing activity of the Joint Task Group on Exfoliation Corrosion of Aluminum Alloys (Aluminum Association and ASTM G01.05.02.08 Task Group) to maintain outdoor exposure tests for this purpose. For example, it has been reported⁶ that samples of Al-Zn-Mg-

¹ This method is under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.

Current edition approved April 27, 1979. Published June 1979. Originally published as G 34 – 72. Last previous edition G 34 – 72.

² *Annual Book of ASTM Standards*, Vols 02.05, 05.05, 06.03, 09.01, 10.01, 10.02, 10.05, 11.01, and 15.09.

³ *Annual Book of ASTM Standards*, Vol 03.03.

⁴ *Annual Book of ASTM Standards*, Vol 03.02.

⁵ Ketcham, S. J., and Jeffrey, P. W., "Exfoliation Corrosion Testing of 7178 and 7075 Aluminum Alloys" (Report of ASTM G01.05 Interlaboratory Testing Program in Cooperation with the Aluminum Association); and Sprowls, D. O., Walsh, J. D., and Shumaker, M. B., "Simplified Exfoliation Testing of Aluminum Alloys," *Localized Corrosion—Cause of Metal Failure*, ASTM STP 516, Am. Soc. Testing Mats., 1972.

⁶ Sprowls, D. O., Summerson, T. J., and Loftin, F. E., "Exfoliation Corrosion Testing of 7075 and 7178 Aluminum Alloys—Interim Report on Atmospheric Exposure Tests" (Report of ASTM G01.05.02 Interlaboratory Testing Program in Cooperation with the Aluminum Association); and Lifka, B. W. and Sprowls, D. O., "Relationship of Accelerated Test Methods for Exfoliation Resistance in 7XXX Series Aluminum Alloys With Exposure to a Seacoast Atmosphere," *Corrosion in Natural Environments*, ASTM STP 558, Am. Soc. Testing Mats., 1974.

Cu alloys rated EA in a 48-h EXCO test did not develop more than a slight amount of incipient exfoliation (EA) during a four-year exposure in a seacoast atmosphere, whereas, ED rated materials in most cases developed severe exfoliation within a year in the seacoast atmosphere. It is anticipated that additional comparisons will become available as the outdoor tests are extended.

5. Definitions

5.1 *exfoliation*—corrosion that proceeds laterally from the sites of initiation along planes parallel to the surface, generally at grain boundaries, forming corrosion products that force metal away from the body of the material giving rise to a layered appearance (Definitions G 15).

6. Apparatus

6.1 Any suitable glass, plastic, or similarly inert container can be used to contain the solution and specimens during the period of test. Depending upon the shape and size of the specimens, rods or racks of glass, plastic, or any inert substance shall be used to support the specimen above the bottom of the container. The container should be fitted with a loose-fitting cover to reduce evaporation.

7. Reagents

7.1 *Purity of Reagents*—The test solution shall be prepared with reagent grade sodium chloride (NaCl), potassium nitrate (KNO_3), and nitric acid (HNO_3).

7.2 *Purity of Water*—Distilled or deionized water conforming to Specification D 1193, Type IV, shall be used to prepare the test solution.

7.3 A test solution of the following composition shall be used:

NaCl	(4.0 M)
KNO_3	(0.5 M)
HNO_3	(0.1 M)

Dissolve 234 g of NaCl, 50 g of KNO_3 in water, and add 6.3 ml of concentrated HNO_3 (70 weight %). Dilute to 1 litre. This solution has an apparent pH of 0.4.

7.4 The solution shall be maintained at a temperature of $25 \pm 3^\circ\text{C}$ ($77 \pm 5^\circ\text{F}$).

8. Sampling

8.1 Sampling procedures are not considered

applicable to this standard, as they are often covered by product specifications. It is assumed that the test specimens are removed from representative samples of materials.

9. Test Specimens

9.1 Specimens may be of any practical size or shape. Nevertheless, for the results to be of most significance a specimen size of at least 50 by 100 mm (2 by 4 in.), or the equivalent, is recommended.

9.2 The edges of sawed specimens need not be machined, but specimens obtained by blanking or shearing shall have edges dressed by machining or filing to a depth equal to the thickness of the specimen to remove cold-worked metal.

9.3 Remove the cladding of alclad sheet by machining the test surface; remove or mask the cladding on the back side (non-test surface) also.

9.4 When removing test specimens from extrusions and forgings, take care to avoid specimen locations underneath flanges, ribs, etc., where the grain structure is usually variable.

10. Standardization

10.1 To provide an indication when some inadvertent deviation from the correct test conditions occurs, it is necessary to expose to the test at regular intervals a control specimen of a material with known resistance. This control should exhibit the same degree of exfoliation each time it is included in the test.

10.2 The control may be any material of the alloy type included in the scope of this standard, preferably one with an intermediate degree of susceptibility (Figs. 4 and 5).

11. Procedure

11.1 Degrease the specimens with a suitable solvent.

11.2 Mask the back surfaces of the specimens to minimize corrosion of non-test areas. Protective coatings must have good adherence to avoid crevice corrosion beneath the coating; also, they should not contain leachable ions or protective oils that will influence the corrosion of the test surface.⁷

⁷ Coatings that have been satisfactorily used include: No. 35 Vinyl Plastic Electrical Tape or No. 470 Electroplaters Tape made by the Minnesota Mining and Mfg. Co., St. Paul, Minn.

11.3 Use the solution in sufficient quantity to provide a volume-to-metal surface area ratio of 10 to 30 ml/cm² (65 to 200 ml/in.²). Include all exposed metal area in the determination of total surface area.

11.4 Use fresh solution at the start of each test. Do not change the solution even though the pH increases during the test. It is normal for the pH to increase from the initial apparent value of 0.4 to about 3 during the first several hours depending upon the amount of corrosion that occurs.

11.5 Immerse the specimens in the solution using rods or racks of inert material to support the specimens above the bottom of the container. Place the test surface upward in a horizontal position to prevent loss of exfoliated metal from the surface of the specimen. Do not concurrently immerse in the same container alloys containing less than 0.25 % copper with those containing greater amounts of copper.

11.6 The following maximum periods of exposure are recommended for testing the alloy types indicated:

2XXX Series	96 h
7XXX Series	48 h

The length of time to develop exfoliation in material of a given alloy and temper may vary with the mill product form, with some materials developing severe exfoliation in much shorter periods than those listed. Therefore, inspect test specimens in place and rate in accordance with Section 12 at periods such as 5, 24, 48, and 72 h, and discontinue the exposure of a specimen when it has developed the most severe exfoliation rating (Fig. 6).

11.7 Rate the performance of test specimens in accordance with Section 12 immediately after discontinuation of the exposure while the specimen is still wet or moist, taking into account all loose products of exfoliation lying on the test specimen or on the bottom of the container.

11.8 Clean exposed test specimens by rinsing in water and soaking in concentrated nitric acid only after the specimens have been inspected and rated.

12. Interpretation of Results

12.1 The following codes and classifications shall be used when reporting the visual rating of corroded specimens:

Classification	Code
No appreciable attack	N
Pitting	P
Exfoliation	EA through ED

12.2 Descriptions of the various classifications, which are illustrated in Figs. 1 through 6,⁸ are as follows:

12.2.1 *N*—No appreciable attack: Surface may be discolored or etched, but no evidence of pitting or exfoliation.

12.2.2 *P*—Pitting: Discrete pits, sometimes with a tendency for undermining and slight lifting of metal at the pit edges (Figs. 1a and 2).

12.2.3 *EA through ED*—Exfoliation:

12.2.3.1 Visible separation of the metal into layers manifested in various forms, such as blisters, slivers, flakes, fairly continuous sheets, and sometimes granular particles resulting from disintegration of thin layers, depending upon the grain morphology of the sample. Various degrees of exfoliation with increasing penetration and loss of metal are illustrated in Figs. 3 through 6.

12.2.3.2 The formation of tiny “pit-blisters” or the dislodgement of an extremely thin surface layer of metal after only a few hours of exposure may resemble superficial exfoliation (EA), but can in fact result from undermining pitting. If continued exposure to the recommended periods in 11.6 produces more corrosion but no evidence of advancing delamination, metallographic examination (see Methods E 3) will be required to determine whether the initial effect was truly exfoliation (Fig. 1b) or undermining pitting (Fig. 1a); in the latter case the rating should be P.

12.2.3.3 When exfoliation occurs in isolated sites, rate the attack in the individual sites rather than the relative number of sites.

12.3 The visual ratings are intended to be finite indications of the resistance to exfoliation, and care should be taken when rating a series of test specimens to compare them with the photographs and captions in Figs. 2 through 6 rather than with each other. The final rating of a specimen shall be determined by the poorest classification observed during the exposure.

⁸ Enlarged glossy prints of Figs. 1 through 6 are available from ASTM Headquarters. Order PCN 12-700340-22.

13. Report

13.1 The report should contain the following essential information:

13.1.1 Alloy and temper of the material tested,

13.1.2 Mill product, section thickness, and the surface tested, including reference to applicable product specification,

13.1.3 Sampling procedure if other than that specified in referenced product specification,

13.1.4 A rating of the test specimens using the codes and classifications in Section 12, and

13.1.5 Notation of any deviation in test procedure from that set forth in preceding paragraphs.

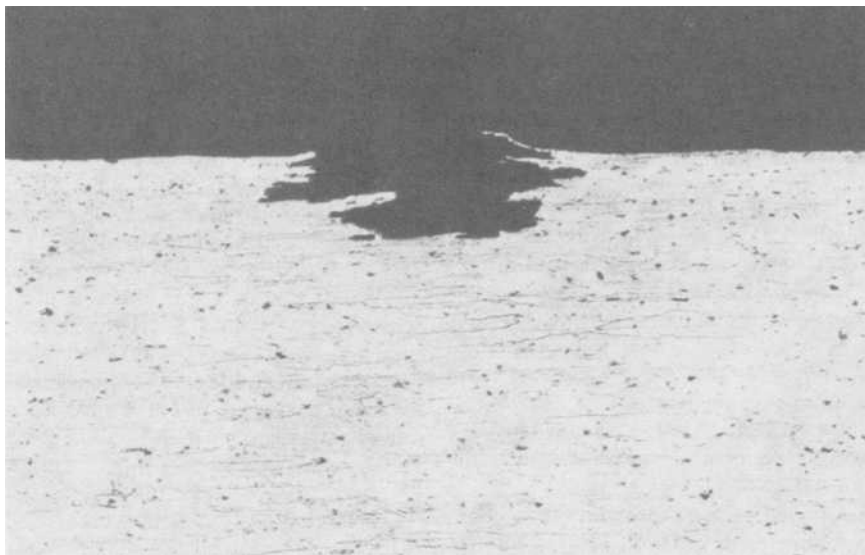
13.2 Other information that may be desirable for certain types of reports includes:

13.2.1 Size, type, and number of replicate specimens; method of edge preparation, and

13.2.2 Volume to surface ratio.

14. Precision and Accuracy

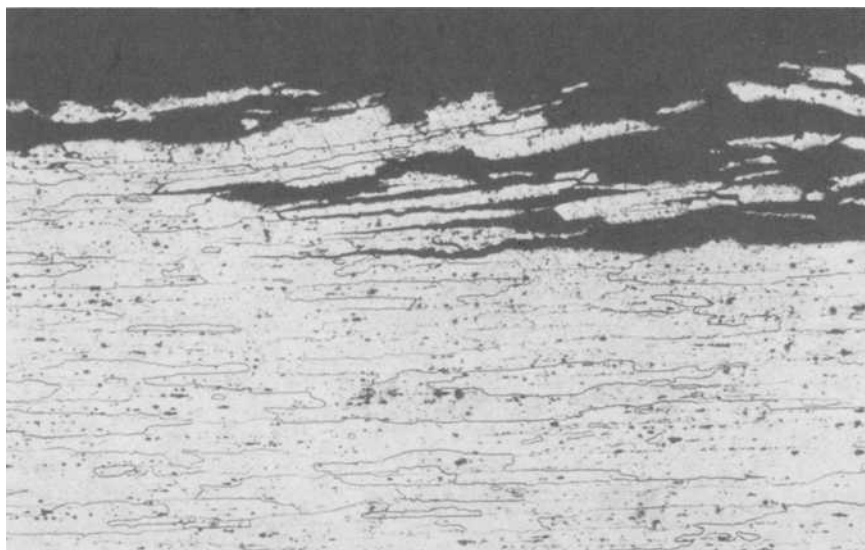
14.1 Exfoliation ratings obtained by the visual interpretation guidelines in Section 12 are, at best, judgments by the inspector. Hence, they are subject to variation among inspectors, but experience has shown that differences greater than one rating letter are rare among trained inspectors.



1a— Undermining pitting that may from the surface give the appearance of incipient exfoliation.

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1b— Exfoliation resulting from rapid lateral attack of selective boundaries or strata forming wedges of corrosion product that force layers of metal upward giving rise to a layered appearance.

FIG. 1 Metallographic Sections Illustrating Two Different Types of Attack (Keller's Etch; 100×).

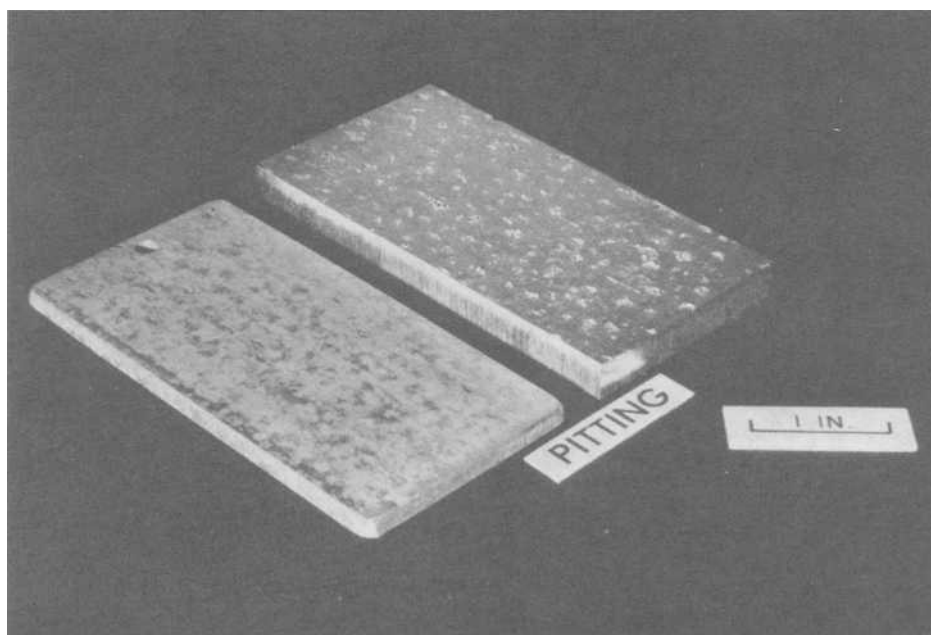
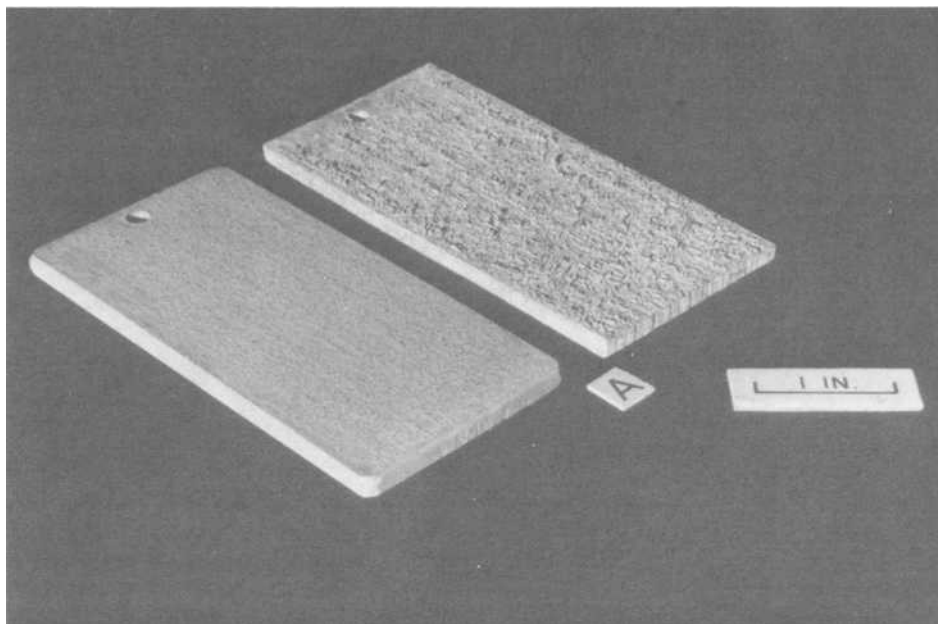
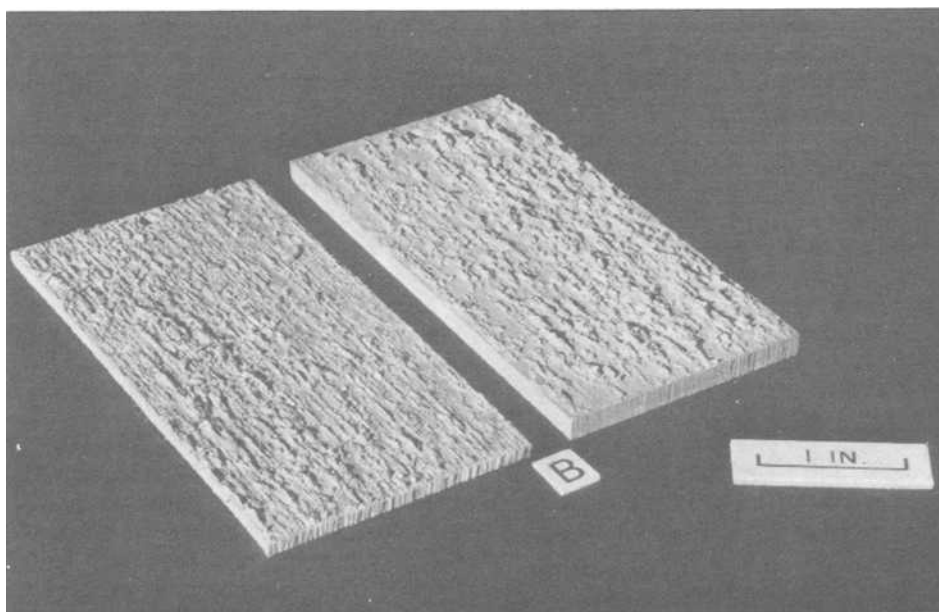


FIG. 2 Examples of Pitting Corrosion.



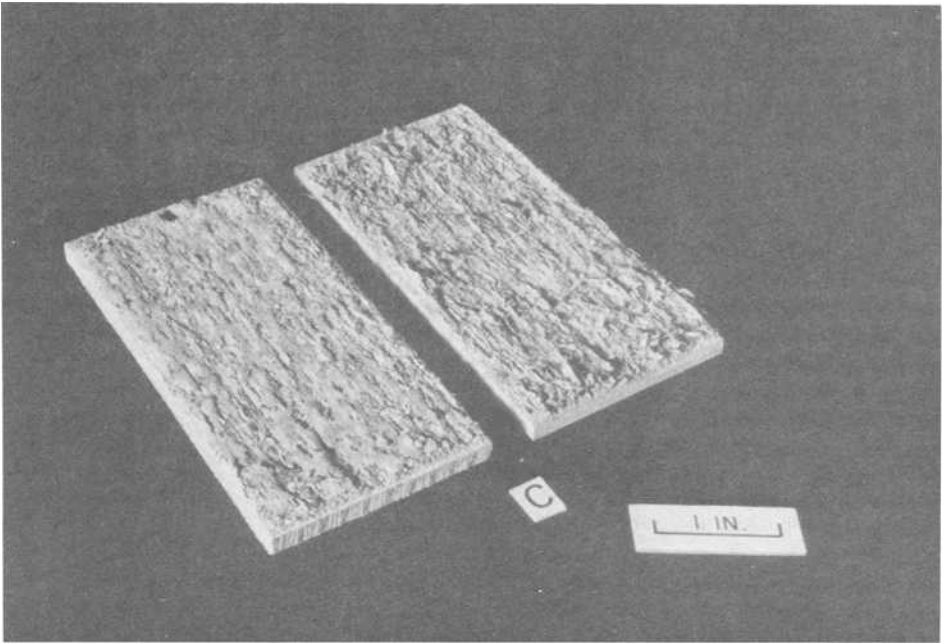
NOTE—The panel on the right illustrates an extreme condition, and a worse appearing panel should be given a less favorable rating.

FIG. 3 Examples of Exfoliation Rating EA (Superficial): Tiny Blisters, Thin Slivers, Flakes or Powder, with only Slight Separation of Metal.



NOTE—The panel on the right illustrates an extreme condition.

FIG. 4 Example of Exfoliation Rating EB (moderate); notable layering and penetration into the metal.



NOTE The panel on the right illustrates an extreme condition.

FIG. 5 Examples of Exfoliation Rating EC (severe): Penetration to a Considerable Depth into the Metal.

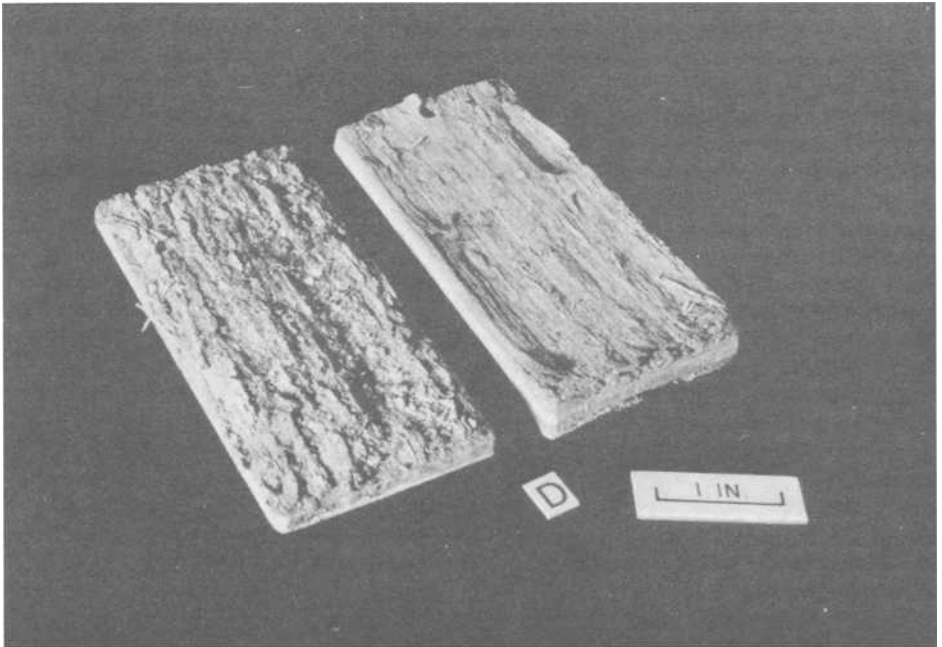


FIG. 6 Examples of Exfoliation rating ED (Very Severe) (similar to EC except for much greater penetration and loss of metal).



Standard Recommended Practice for EXAMINATION AND EVALUATION OF PITTING CORROSION¹

This standard is issued under the fixed designation G 46; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This recommended practice is a guide to assist in the selection of procedures that can be used in the identification and examination of pits and in the evaluation of pitting corrosion to determine the extent of its effect.

2. Applicable Documents

2.1 *ASTM Standards:*

E 3 Methods of Preparation of Metallographic Specimens²

G 1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens³

G 16 Practice for Applying Statistics to Analysis of Corrosion Data³

2.2 *National Association of Corrosion Engineers Standard:*

RP-01-73 Collection and Identification of Corrosion Products⁴

3. Significance

3.1 It is important to be able to determine the extent of pitting, either in a service application where it is necessary to predict the remaining life in a metal structure, or in laboratory test programs that are used to select the most pitting-resistant materials for service.

4. Definition

4.1 *pitting*—the localized corrosion of a metal surface, confined to a point or small area, that takes the form of cavities.

5. Identification and Examination of Pits

5.1 *Visual Inspection*—A visual examination of the corroded metal surface is usually beneficial, and this is done under ordinary light, with or without the use of a low-power magnifying glass, to determine the extent of

corrosion and the apparent location of pits. It is often advisable to photograph the corroded surface at this point so that it can be compared with the clean surface after the removal of corrosion products.

5.1.1 If the metal specimen has been exposed to an unknown environment, the composition of the corrosion products may be of value in determining the cause of corrosion. Follow recommended procedures in the removal of particulate corrosion products and reserve them for future identification (see NACE Standard RP-01-73).

5.1.2 To expose the pits fully, use recommended cleaning procedures to remove the corrosion products and avoid solutions that attack the base metal excessively (see Practice G 1). It may be advisable during cleaning to probe the pits with a pointed tool to determine the extent of undercutting or subsurface corrosion (Fig. 1). However, scrubbing with a stiff bristle brush will often enlarge the pit openings sufficiently by removal of corrosion products, or undercut metal to make the pits easier to evaluate.

5.1.3 Examine the cleaned metal surface under ordinary light to determine the approximate size and distribution of pits. Follow this procedure by a more detailed examination through a microscope using low magnification ($20\times$).

5.1.4 Determine the size, shape, and den-

¹ This recommended practice is under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals, and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.

Current edition approved April 9, 1976. Published July 1976.

² *Annual Book of ASTM Standards*, Vol 03.03.

³ *Annual Book of ASTM Standards*, Vol 03.02.

⁴ Insert in *Materials Protection and Performance*, Vol 12, June 1973, p. 65.

sity of pits.

5.1.4.1 Pits may have various sizes and shapes. A visual examination of the metal surface may show a round, elongated, or irregular opening, but it seldom provides an accurate indication of corrosion beneath the surface. Thus, it is often necessary to cross section the pit to see its actual shape and to determine its true depth. Several variations in the cross-sectioned shape of pits are shown in Fig. 1.

5.1.4.2 It is a tedious job to determine pit density by counting pits through a microscope eyepiece, but the task can be made easier by the use of a plastic grid. Place the grid, containing 3 to 6-mm squares, on the metal surface. Count and record the number of pits in each square, and move across the grid in a systematic manner until all the surface has been covered. This approach minimizes eye-strain because the eyes can be taken from the field of view without fear of losing the area of interest.

5.1.5 *Metallographic Examination*—Select and cut out a representative portion of the metal surface containing the pits and prepare a metallographic specimen in accordance with the recommended procedures given in Method E 3. Examine microscopically to determine whether there is a relation between pits and inclusions or microstructure, or whether the cavities are true pits or might have resulted from metal dropout caused by intergranular corrosion, dealloying, etc.

5.2 *Nondestructive Inspection*—A number of techniques have been developed to assist in the detection of cracks or cavities in a metal surface without destroying the material (1).⁵ These methods are less effective for locating and defining the shape of pits than some of those previously discussed, but they merit consideration because they are often used *in situ*, and thus are more applicable to field applications.

5.2.1 *Radiographic*—Radiation, such as X rays, are passed through the object. The intensity of the emergent rays varies with the thickness of the material. Imperfections may be detected if they cause a change in the absorption of X rays. Detectors or films are used to provide an image of interior imperfections. The metal thickness that can be inspected is dependent on the available energy

output. Pores or pits must be as large as $1/2\%$ of the metal thickness to be detected. This technique has only slight application to pitting detection, but it might be a useful means to compare specimens before and after corrosion to determine whether pitting has occurred and whether it is associated with previous porosity. It may also be useful to determine the extent of subsurface and undercutting pitting (Fig. 1).

5.2.2 *Electromagnetic*:

5.2.2.1 Eddy currents can be used to detect defects or irregularities in the structure of electrically conducting materials. When a specimen is exposed to a varying magnetic field, produced by connecting an alternating current to a coil, eddy currents are induced in the specimen, and they in turn produce a magnetic field of their own. Materials with defects will produce a magnetic field that is different from that of a reference material without defects, and an appropriate detection instrument is required to determine these differences.

5.2.2.2 The induction of a magnetic field in ferromagnetic materials is another approach that is used. Discontinuities that are transverse to the direction of the magnetic field cause a leakage field to form above the surface of the part. Ferromagnetic particles are placed on the surface to detect the leakage field and to outline the size and shape of the discontinuities. Rather small imperfections can be detected by this method. However, the method is limited by the required directionality of defects to the magnetic field, by the possible need for demagnetization of the material, and by the limited shape of parts that can be examined.

5.2.3 *Sonics*:

5.2.3.1 In the use of ultrasonics, pulses of sound energy are transmitted through a couplant, such as oil or water, onto the metal surface where waves are generated. The reflected echoes are converted to electrical signals that can be interpreted to show the location of flaws or pits. Both contact and immersion methods are used. The test has good sensitivity and provides instantaneous information about the size and location of flaws. However, reference standards are required

⁵ The boldface numbers in parentheses refer to the list of references at the end of this recommended practice.

for comparison, and training is needed to interpret the results properly.

5.2.3.2 An alternative approach is to use acoustic emissions in detecting flaws in metals. Imperfections, such as pits, generate high-frequency emissions under thermal or mechanical stress. The frequency of emission and the number of occurrences per unit time determine the presence of defects.

5.2.4 *Penetrants*—Defects opening to the surface can be detected by the application of a penetrating liquid that subsequently exudes from the surface after the excess penetrant has been removed. Defects are located by spraying the surface with a developer that reacts with a dye in the penetrant, or the penetrant may contain a fluorescent material that is viewed under black light. The size of the defect is shown by the intensity of the color and the rate of bleed-out. This technique provides only an approximation of the depth and size of pits.

5.2.5 None of these nondestructive test methods provide satisfactory detailed information about pitting. They can be used to locate pits and to provide some information about the size of pits, but they generally are not able to detect small pits, and confusion may arise in attempting to differentiate between pits and other surface blemishes. Most of these methods were developed to detect cracks or flaws in metals, but with more refined development they may become more applicable to pitting measurements.

6. Extent of Pitting

6.1 *Weight Loss*—Metal weight loss is not ordinarily recommended for use as a measure of the extent of pitting unless general corrosion is slight and pitting is fairly severe. If uniform corrosion is significant, the contribution of pitting to total metal loss is small, and pitting damage cannot be determined accurately from weight loss. In any case, weight loss can only provide information about total metal loss due to pitting but nothing about depth of penetration. However, weight loss should not be neglected in every case because it may be of value; for example, weight loss along with a visual comparison of pitted surfaces may be adequate to evaluate the pitting resistance of alloys in laboratory tests.

6.2 *Pit Depth Measurement:*

6.2.1 *Metallographic*—Pit depth can be determined by sectioning vertically through a preselected pit, mounting the cross-sectioned pit metallographically, and polishing the surface. The depth of the pit is measured on the flat, polished surface by the use of a microscope with a calibrated eyepiece. The method is very accurate, but it requires good judgment in the selection of the pit and good technique in cutting through the pit. Its limitations are that it is time consuming, the deepest pit may not have been selected, and the pit may not have been sectioned at the deepest point of penetration.

6.2.2 *Machining (2, 3):*

6.2.2.1 This method requires a sample that is fairly regular in shape, and it involves the destruction of the specimen. Measure the thickness of the specimen between two areas that have not been affected by general corrosion. Select a portion of the surface on one side of the specimen that is relatively unaffected; then machine the opposite surface where the pits are located on a precision lathe, grinder, or mill until all signs of corrosion have disappeared. (Some difficulty from galling and smearing may be encountered with soft metals, and pits may be obliterated.) Measure the thickness of the specimen between the unaffected surface and subtract from the original thickness to give the maximum depth of pitting. Repeat this procedure on the unmachined surface unless the thickness has been reduced by 50% or more during the machining of the first side.

6.2.2.2 This method is equally suitable for determining the number of pits with specific depths. Count the visible pits; then machine away the surface of the metal in measured stages and count the number of visible pits remaining at each stage. Subtract the number of pits at each stage from the count at the previous stage to obtain the number of pits at each depth of cut.

6.2.3 *Micrometer or Depth Gage:*

6.2.3.1 This method is based on the use of a pointed needle attached to a micrometer or calibrated depth gage to penetrate the pit cavity. Zero the instrument on an unaffected area at the lip of the pit. Insert the needle in the pit until it reaches the base where a new measurement is taken. The distance traveled by the needle is the depth of the pit. It is best to use

constant-tension instruments to minimize metal penetration at the base of the pit. It can be advantageous to use a stereomicroscope in conjunction with this technique so that the pit can be magnified to ensure that the needle point is at the bottom of the pit. The method is limited to pits that have a sufficiently large opening to accommodate the needle without obstruction; this eliminates those pits where undercutting or directional orientation has occurred.

6.2.3.2 In a variation of this method, attach the probe to a spherometer and connect through a microammeter and battery to the specimen (3, 4). When the probe touches the bottom of the pit, it completes the electrical circuit, and the probe movement is a measurement of pit depth. This method is limited to very regularly shaped pits because contact with the side of the pit would give a false reading.

6.2.4 *Microscopical*—This method is particularly valuable when pits are too narrow or difficult to penetrate with a probe type of instrument. The method is amenable to use as long as light can be focused on the base of the pit, which would not be possible in the case of example (e) in Fig. 1.

6.2.4.1 Use a metallurgical microscope with a magnification range from 50 to 500X and a calibrated fine-focus knob (for example, 1 division = 0.001 mm). If the latter is not available, a dial micrometer can be attached to the microscope in such a way that it will show movement of the stage relative to the microscope body.

6.2.4.2 Locate a single pit on the metal surface and center under the objective lens of the microscope at low magnification (for example, 50X). Increase the objective lens magnification until the pit area covers most of the field under view. Focus the specimen surface at the lip of the pit, using first the coarse and then the fine-focusing knobs of the microscope. Record the initial reading from the fine-focusing knob. Refocus on the bottom of the pit with the fine-focusing knob and record the reading. The difference between the initial and the final readings on the fine-focusing knob is the pit depth.

6.2.4.3 Repeat the steps in 6.2.4.2 to obtain additional measurements or until satisfactory duplication has been obtained. The re-

peatability of pit depth measurements on a single pit at four magnifications is shown in Annex A1.

6.2.4.4 A variation of the microscopical technique employs the use of an interference microscope. A beam of light is split, and one portion is projected on the specimen and the other on a reference mirror surface. The reflected light from these two surfaces is recombined, and interference fringes are formed that provide a topographical map of the specimen surface. These fringes can be used to measure vertical deviations on the metal surface. However, the method is limited to the shallower pits, that is, less than 25 μm , because the number of fringes increases to the point where they are difficult to count.

7. Evaluation of Pitting

7.1 There are several ways in which pitting can be described, given a quantitative expression to indicate its significance, or used to predict the life of a material. Some of the more commonly used methods are described in this section, although it is often found that no single method is sufficient by itself.

7.2 *Standard Charts (3):*

7.2.1 Rate the pits in terms of density, size, and depth on the basis of standard charts, such as those shown in Fig. 2. Columns A and B relate to the extent of pitting at the surface of the metal (that is, Column A is a means for rating the number of sites per unit area and Column B a means for showing the average size of these sites). Column C rates the intensity or average depth of attack. A typical rating might be A-3, B-2, C-3, representing a density of 5×10^4 pits/ m^2 , an average pit opening of 2.0 mm^2 , and an average pit depth of 1.6 mm.

7.2.2 This method offers an effective means of communication between those who are familiar with the charts, and it is a simple means for storing data for comparison with other test results. However, it is tedious and time consuming to measure all pits, and the time is usually not justified because maximum values (for example, pit depths) usually have more significance than average values.

7.3 *Metal Penetration:*

7.3.1 Measure the deepest pits and express metal penetration in terms of the maximum pit depth or the average of the ten deepest

pits, preferably both. This type of measurement is particularly significant when the metal is associated with an enclosure for a gas or liquid, and a hole could lead to a loss of fluid.

7.3.2 Metal penetration can also be expressed in terms of a pitting factor. This is the ratio of the deepest metal penetration to the average metal penetration, determined from weight loss, as shown in the following relationship:

$$\text{Pitting Factor} = \frac{\text{deepest metal penetration}}{\text{average metal penetration}}$$

A pitting factor of one represents uniform corrosion; the larger the number, the greater the depth of penetration. The factor does not apply in those cases where pitting or general corrosion is very small because values of zero or infinity can readily be obtained when dealing with a ratio.

7.4 Statistical:

7.4.1 The application of statistics to the analysis of corrosion data is covered in detail in Practice G 16. The subject is discussed briefly in this standard to show that statistics have a bearing on the evaluation of pitting data; more detailed information can be obtained from other publications.

7.4.2 The probability that pits will initiate on a metal surface is dependent on a number of factors, such as the pitting tendency of the metal, the corrosivity of the solution, the specimen area, and the time of exposure. A pitting probability test can be conducted to determine the susceptibility of metals to pitting, but it will not provide information about the rate of propagation, and the results are only applicable to the conditions of exposure. The pitting probability (P) in % after the exposure of a number of specimens to a particular set of conditions can be expressed as follows (5, 6):

$$P = \frac{N_p}{N} \times 100$$

where:

N_p = number of specimens that pit, and
 N = total number of specimens.

7.4.2 The relationship between pit depth and area or time of exposure may vary with the environment, the metal exposed, and other variables. The relationships cited in 7.4.2.1 and 7.4.2.2 are examples that have

been found to apply under certain exposure conditions.

7.4.2.1 The following relationship was found between the maximum pit depth (D) and the area (A) of a pipeline exposed to soil (7, 8, 9):

$$D = bA^a$$

where a and $b > 0$, and a and b are constants that were derived from the slope and the y-intercept of a straight line curve obtained when the logarithms of the mean pit depth for successively increasing areas on the pipe were plotted against the logarithms of the corresponding areas. The dependence on area is attributed to the increased chance for the deepest pit to be found when the size of the sample of pits is increased through an increased area of corroded surface.

7.4.2.2 The maximum pit depth (D) of aluminum exposed to various waters was found to vary as the cube root of time (t), as shown in the following relationship (5, 10):

$$D = Kt^{1/3}$$

K is a constant that is a function of the composition of the water and alloy. This relationship has been found to apply to several aluminum alloys exposed to different waters.

7.4.3 Extreme value probability statistics (11, 12) have been applied successfully to maximum pit depth data to estimate the maximum pit depth of a large area of material on the basis of examination of a small portion of that area (3, 5, 10). The procedure is to measure maximum pit depths on several replicate specimens that have pitted, and then arrange the pit depth values in order of increasing rank. A plotting position for each order of ranking is obtained by substituting in the relation, $M/(n+1)$, where M = order of ranking, and n = total number of specimens or values. For example, the plotting position for the second value out of 10 would be $2/(10+1) = 0.1818$. These values are plotted on the ordinate of extreme value probability paper versus their respective maximum pit depths. If a straight line is obtained, it shows that extreme value statistics apply. Extrapolation of the straight line can be used to determine the probability that a specific depth will occur or the number of observations that must be made to find a particular pit depth.

7.5 *Loss in Mechanical Properties* — If pitting is the predominant form of corrosion and the density of pitting is relatively high, the change in a mechanical property may be used advantageously to evaluate the degree of pitting. Typical properties that are considered for this purpose are tensile strength, elongation, fatigue strength, impact resistance, and burst pressure (13, 14).

7.5.1 The precautions that must be taken in the application of these mechanical test procedures are covered in most standard methods, but it must be stressed that it is important to use as nearly replicate specimens as possible for both the exposed and unexposed specimens. Thus, consideration should be given to edge effects, direction of rolling, surface conditions, etc.

7.5.2 Representative specimens of the metal are exposed to the same conditions except for the corrosive environment. The mechanical properties of the exposed and unexposed specimens are measured after the exposure; the difference between the two results is attributed to corrosion.

7.5.3 Some of these methods are more properly suited to the evaluation of other forms of localized corrosion, such as intergranular or stress corrosion, so their limitations must be considered. The often erratic

nature of pitting and the location of pits on the specimen can affect results. In some cases the change in mechanical properties due to pitting may be too small to provide meaningful results. Probably one of the most difficult problems is to separate the effects due to pitting from those caused by some other form of corrosion.

8. Report

8.1 The report should include as much detailed information as possible, such as the following:

8.1.1 Metallurgical treatment of the metal, surface preparation, and final surface finish before exposure to test,

8.1.2 Environmental conditions and duration of exposure,

8.1.3 Appearance of the corroded surface before and after cleaning,

8.1.4 Identification of corrosion products,

8.1.5 Characterization of pits to include: size, shape, density, uniformity of distribution, depth (average and maximum), and location of pits with reference to microstructure, face, edge, crevice, etc.,

8.1.6 Change in mechanical properties as the result of corrosion, and the method by which determined, and

8.1.7 Statistical information.

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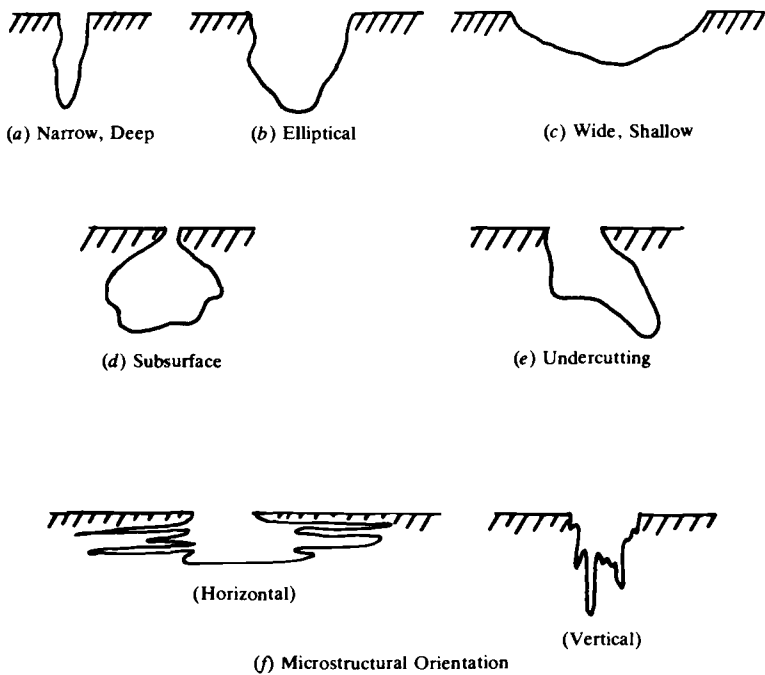


Fig. 1 Variations in the Cross-Sectional Shape of Pits.

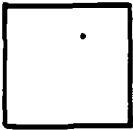
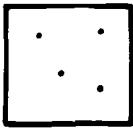
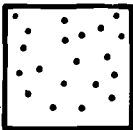
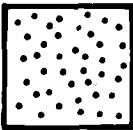
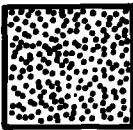
	<u>A</u> <u>DENSITY</u>	<u>B</u> <u>SIZE</u>	<u>C</u> <u>DEPTH</u>
1	 $2.5 \times 10^3 / \text{m}^2$	 0.5 mm^2	 0.4 mm
2	 $1 \times 10^4 / \text{m}^2$	 2.0 mm^2	 0.8 mm
3	 $5 \times 10^4 / \text{m}^2$	 8.0 mm^2	 1.6 mm
4	 $1 \times 10^5 / \text{m}^2$	 12.5 mm^2	 3.2 mm
5	 $5 \times 10^5 / \text{m}^2$	 24.5 mm^2	 6.4 mm

Fig. 2 Standard Rating Charts for Pits.

ANNEX

A1. REPEATABILITY OF MICROSCOPICAL PIT DEPTH MEASUREMENTS

A1.1 Repeatability of pit depth measurements on a single pit at four magnifications is shown in Table A1.

A1.2 The data in Table A1 indicate that as the

magnification was increased (that is, from 65 to 370 $\times$), the average pit depth that was measured decreased from 0.174 mm to 0.151 mm. Repeatability of measurement improved with magnification,

and, as will be shown in A1.3, accuracy also showed marked improvement.

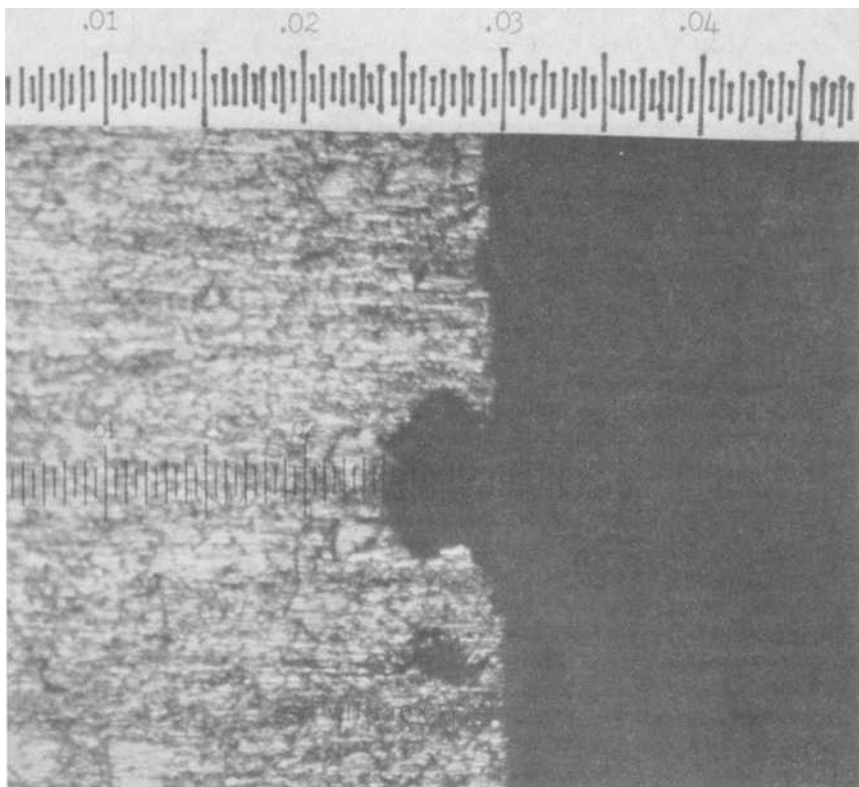
A1.3 The pit used for the measurements in Table A1 was cross sectioned and photographed at 100× through a microscope with a micrometer reticle. As shown in Fig. A1, the depth measured in cross section is 0.152 mm. This result is in excellent

agreement with that found under high magnification, and shown in Table A1.

A1.4 Pit depth measurements have been made over the range from 0.04 to 0.34 mm. The only limitation to this method is that associated with the range of movement of the calibrated focusing knob on the microscope.

TABLE A1 Microscopical Pit Depth Measurements

Magnification	Pit Depth, mm
65	0.183
	0.159
	0.179
	0.174 avg
132	0.159
	0.160
	0.155
	0.159 avg
200	0.149
	0.157
	0.150
	0.153 avg
370	0.151
	0.151
	0.152
	0.151 avg



NOTE—Use with 10× F.F. Objective.

FIG. A1 Cross Section of Pit Used for Depth Measurements in Table A1 (Each Scale Division Equals 0.0005 in. (13 μm)).

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Standard Test Methods for PITTING AND CREVICE CORROSION RESISTANCE OF STAINLESS STEELS AND RELATED ALLOYS BY THE USE OF FERRIC CHLORIDE SOLUTION¹

This standard is issued under the fixed designation G 48; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

⁽¹⁾NOTE—Editorial changes were made throughout in November 1980.

1. Scope

1.1 These methods cover procedures for the determination of pitting and crevice corrosion resistance of stainless steels and related alloys when exposed to oxidizing chloride environments. Two procedures are described and identified as Methods A and B:

1.1.1 *Method A*—Total immersion ferric chloride test.

1.1.2 *Method B*—Ferric chloride crevice test.

1.2 Method A is designed to determine the relative pitting resistance of stainless steels and nickel-base, chromium-bearing alloys, whereas Method B can be used for determining both the pitting and crevice corrosion resistance of these alloys.

1.3 These tests may be used to determine the effects of alloying additives, heat treatment, and surface finishes on pitting and crevice corrosion resistance.

2. Applicable Document

2.1 *ASTM Standard*:

G 46 Recommended Practice for Examination and Evaluation of Pitting Corrosion²

3. Ferric Chloride Test Solution

3.1 Dissolve 100 g of reagent grade ferric chloride, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, in 900 ml of distilled water (about 6% FeCl_3 by weight). Filter through glass wool or filter paper to remove insoluble particles.

4. Test Specimens

4.1 A test specimen 25 by 50 mm (1 by 2 in.)

is recommended as a standard size, although various shapes and sizes can be tested by this method. All specimens in a test series should have the same dimensions when comparisons are to be made. Unless end-grain pitting is an integral part of the evaluation, the proportion of end-grain surface to specimen surface should be kept small because of the susceptibility of end-grain surfaces to pitting.

NOTE 1—The thickness of the specimen can influence the tightness of the crevice and the test results.

4.2 When specimens are cut by shearing, the deformed material should be removed by machining or grinding prior to testing unless the sheared edges are being evaluated.

4.3 All surfaces of the specimen should be polished to a uniform finish. A 120-grit abrasive paper has been found to provide a satisfactory standard finish. Wet polishing is preferred, but if dry polishing is used, it should be done slowly to avoid overheating. Sharp edges of the specimen should be rounded to avoid cutting rubber band or O-ring.

NOTE 2—While a polished surface is preferred for uniformity, the test may be varied at the discretion of the investigator to evaluate other surface finishes, such as a mill finish.

4.4 Measure the dimensions of the specimen

¹ These methods are under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals, and are the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests. Current edition approved June 11, 1976. Published August 1976.

² *Annual Book of ASTM Standards*, Vol 03.02.

and calculate the total exposed area of interest.

4.5 Clean specimen surfaces with magnesium oxide paste or equivalent, rinse well with water, dip in acetone or methyl alcohol, and air dry.

4.6 Weigh each specimen to the nearest 0.001 g or better and store in a desiccator until ready for use.

5. Method A—Total Immersion Ferric Chloride Test

5.1 Apparatus:

5.1.1 *Glass Beakers*, 1000-ml, tall-form, or Erlenmeyer flasks, 1000-ml, wide neck.

5.1.2 *Glass Cradles* (Fig. 1)—The dimensions of the cradle shall be restricted to those that will permit its passage through the test container opening, a diameter of about 40 mm (1.6 in.) in the case of the Erlenmeyer flask.

5.1.3 *Water or Oil Bath*, constant-temperature.

5.2 Procedure:

5.2.1 Pour 600 ml of the ferric chloride test solution into the test container. If specimens larger than the standard are used, provide a solution volume of at least 20 ml/cm² (125 ml/in.²) of surface area. Transfer the container to a constant-temperature bath and allow to come to the equilibrium temperature of interest. Suitable temperatures for evaluation are 22 ± 2°C and 50 ± 2°C.

5.2.2 Place the specimen in a glass cradle and immerse in the test solution after it has reached the desired temperature. Maintain temperature throughout the test.

5.2.3 Cover the container with a watch glass. A reasonable test period is 72 h, although variations may be used at the discretion of the investigator and depend on the materials being evaluated.

5.2.4 Remove the specimens, rinse with water, and scrub with a nylon bristle brush under running water to remove corrosion products, dip in acetone or methanol, and dry. Ultrasonic cleaning may be used as a substitute method in those cases where it is difficult to remove corrosion products from deep pits.

5.2.5 Weigh each specimen to 0.001 g or better and reserve for examination.

6. Method B—Ferric Chloride Crevice Test³

6.1 Apparatus:

6.1.1 *Cylindrical TFE-fluorocarbon Blocks*,

two for each test specimen. Each block shall be 12.7 mm (0.5 in.) in diameter and 12.7 mm high, with perpendicular grooves 1.6 mm (0.063 in.) wide and 1.6 mm deep cut in the top of each cylinder for retention of the O-rings or rubber bands. Blocks can be machined from bar or rod stock.

6.1.2 *Fluorinated Elastomer⁴ O-Rings or Rubber Bands* (low sulfur (0.02% max)), two for each test specimen.

NOTE 3—It is good practice to use all O-rings or all rubber bands in a given test program.

6.1.2.1 O-rings shall be 1.75 mm (0.070 in.) in cross section; one ring with an inside diameter of about 20 mm (0.8 in.) and one with an inside diameter of about 30 mm (1.2 in.). Rubber bands should be one No. 12 (38 mm (1.5 in.) long) and one No. 14 (51 mm (2 in.) long).

NOTE 4—Rubber bands or O-rings can be boiled in water prior to use to ensure the removal of water-soluble ingredients that might affect corrosion.

6.1.3 *Test Tube*, 38 mm (1.5 in.) in diameter and 300 mm (11.5 in.) long.

6.1.4 *Rubber Stopper*, No. 8, with center hole for insertion of 6.4-mm (0.25-in.) diameter, 102-mm (4-in.) long glass tube (medicine dropper tube may be conveniently used) to serve as a vent and condenser.

6.1.5 *Water or Oil Bath*, thermostated.

6.2 Procedure:

6.2.1 Add 150 ml of ferric chloride solution to each test tube, insert a rubber stopper, and place the tube in a thermostated bath until it comes to the equilibrium temperature of interest. Suitable temperatures for evaluation are 22 ± 2°C and 50 ± 2°C.

6.2.2 Fasten two TFE-fluorocarbon blocks to the test specimen with O-rings or a double loop of each of two rubber bands as shown in Fig. 2. Use plastic gloves to avoid hand contact with metal surfaces during this operation. Use the small O-ring or the No. 12 rubber band for the 25-mm (1-in.) dimension and the large O-ring or the No. 14 rubber band for the 50-mm (2-in.) dimension.

6.2.3 After the test solution has reached the desired temperature, tilt the tube at a 45-deg angle and slide the test specimen to the bottom

³ Streicher, M. A., "Development of Pitting Resistant Fe-Cr-Mo Alloys," *Corrosion*, SBI/A, Vol 30, No. 77, March 1974.

⁴ Viton®, a trademark of DuPont, has been found acceptable.

of the tube, replace the stopper, and return the tube to the bath.

6.2.4 A reasonable test period is 72 h, although variations may be used at the discretion of the investigator and depend on the materials being evaluated. Specimens may be inspected at intervals by decanting the test solution into a clean beaker and sliding the specimen from the tube. Rinse the specimen in water and examine under four O-ring or rubber band crevices and TFE-fluorocarbon blocks. If further exposure is required, return the specimen and solution to the tube without allowing the specimen to dry and replace the tube in the bath.

NOTE 5—The removal of specimens for inspection may affect the rate of corrosion, and caution should be observed when comparing these results with those obtained from constant immersion tests.

6.2.5 After the test has been completed, remove the test specimens, rinse with water, and scrub with a nylon bristle brush under running water to remove corrosion products, dip in acetone or methanol, and dry. Ultrasonic cleaning may be used in those cases where it is difficult to remove corrosion products from deep pits or crevices.

6.2.6 Weigh each specimen to 0.001 g or better and reserve for examination.

7. Examination and Evaluation

7.1 A visual examination (Note 4) and photographic reproduction of specimen surfaces, along with specimen weight losses, are often sufficient to characterize the pitting and crevice resistance of different materials. A more detailed examination will include the measurement of maximum pit depth, average pit depth, pit density, and crevice depth (see Recommended Practice G 46).

NOTE 6—It is often desirable to probe pit sites on the metal surface with a needle to expose subsurface attack.

NOTE 7—A test shall be disregarded if the rubber band or O-ring breaks during exposure.

7.2 Examine planar surfaces for pits under low-power (for example, 20 $\times$) magnification. (Disregard end pits unless this is an important phase of test, recognizing that these pits may affect pitting on planar surfaces.)

7.3 Measure the deepest pits with the appropriate technique; for example, needle point micrometer gage or microscope with calibrated fine-focus knob. It may be necessary to probe some pits to ensure exposure of the cavity. Measure a significant number of pits to determine the deepest pit and the average of the ten deepest pits. Do not measure the depth of pits that intersect the edge of the specimen.

7.4 Count the number of pits on the planar surface under low-power magnification (for example, 20 $\times$) to determine pit density. A clear plastic grid, divided in centimetres, may be helpful, or the surface can be subdivided by scribing with light lines.

7.5 Determine crevice attack (Method B) under O-rings or rubber bands and TFE-fluorocarbon blocks. Measure the depth of attack (open notch) at O-ring or rubber band contact areas using a microscope with a calibrated eyepiece or other suitable technique. Measure the depth of attack under TFE-fluorocarbon blocks in a manner similar to that used for determining depth of pits.

8. Report

8.1 Record the test procedure used, specimen size and surface preparation, time of test, and temperature.

8.2 Calculate the specimen weight loss and record as grams per square metre.

8.3 Record the maximum pit depth and the average of the ten deepest pits in micrometres and pit density in pits per square metre for both front and back faces of the specimen. Record the maximum pit depth of edges if end grain attack is of interest.

8.4 Record the average depth of crevice attack (Method B) in micrometres under elastic contact at vertical edges and horizontal edges and under blocks at faces.

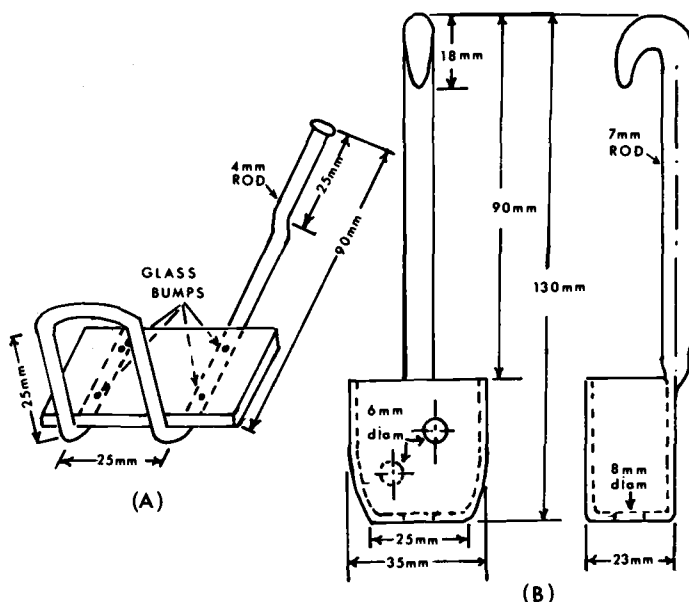
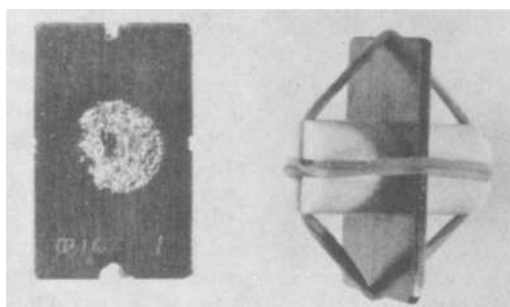


FIG. 1 Examples of Glass Cradles that Can Be Used to Support the Specimen



(a) Specimen After Test with Attack at Four Crevices Under Rubber Bands and Under One Block

(b) Assembled Crevice Test Specimen

FIG. 2 Ferric Chloride Crevice Test Specimen

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, Pa. 19103.



Standard Practice for CONDUCTING CYCLIC POTENTIODYNAMIC POLARIZATION MEASUREMENTS FOR LOCALIZED CORROSION¹

This standard is issued under the fixed designation G 61; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice gives a suggested procedure for conducting cyclic potentiodynamic polarization measurements to determine susceptibility to localized corrosion (pitting and crevice corrosion). If followed, this practice will provide repeatable cyclic potentiodynamic anodic polarization measurements that will reproduce data determined by others at other times in other laboratories. The procedure is preferably used for iron, nickel, or cobalt-based alloys.

1.2 A standard potentiodynamic polarization plot is included. These reference data are based on the results from five different laboratories that followed the standard procedure, using specific alloys of Type 304 stainless steel and Hastelloy C-276. Curves are included which have been constructed using statistical analysis to indicate the acceptable range of polarization curves.

1.3 Samples of the standard Type 304 stainless steel and the Hastelloy C-276 used in obtaining the standard reference plot are available for those who wish to check their own test procedure and equipment.²

2. Applicable Documents

2.1 *ASTM Standards:*

G3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing³

G5 Practice for Standard Reference Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements³

3. Apparatus

3.1 The polarization cell should be similar to the one described in Practice G 5. Other polarization cells may be equally suitable.

3.1.1 The cell should have a capacity of about 1 litre and should have suitable necks or seals to permit the introduction of electrodes, gas inlet and outlet tubes, and a thermometer. The Luggin probe-salt bridge separates the bulk solution from the saturated calomel reference electrode. The probe tip should be adjustable so that it can be brought into close proximity with the working electrode.

3.2 *Specimen Holder:*

3.2.1 Specimens should be mounted in a suitable holder designed for flat strip, exposing 1 cm² to the test solution (Fig. 1). Such specimen holders have been described in the literature.^{4,5} It is important that the circular TFE-fluorocarbon gasket be drilled and machined flat in order to minimize crevices.

3.3 *Potentiostat* (Note 1)—A potentiostat

¹ This practice is under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals, and is the direct responsibility of Subcommittee G01.11 on Electrochemical Measurements in Corrosion Testing.

Current edition approved Dec. 29, 1978. Published April 1979.

² These standard samples are available as a set of one of each type from ASTM Headquarters. Order PCN 12-700610-22.

³ *Annual Book of ASTM Standards*, Vol 03.02.

⁴ France, W. D., Jr., *Journal of the Electrochemical Society*, JESOA, Vol 114, 1967, p. 818.

⁵ Myers, J. R., Gruewlar, F. G., and Smulczynski, L. A., *Corrosion*, CORRA, Vol 24, 1968, p. 352.

that will maintain an electrode potential within 1 mV of a preset value over a wide range of applied currents should be used. For the type and size of standard specimen supplied, the potentiostat should have a potential range of -1.0 to +1.6 V and an anodic current output range of 1.0 to $10^5 \mu\text{A}$. Most commercial potentiostats meet the specific requirements for these types of measurements.

NOTE 1—These instrumental requirements are based upon values typical of the instruments in the five laboratories that have provided the data used in determining the standard polarization plot.

3.4 Potential-Measuring Instruments (Note 1)—The potential-measuring circuit should have a high input impedance on the order of 10^{11} to $10^{14} \Omega$ to minimize current drawn from the system during measurements. Instruments should have sufficient sensitivity and accuracy to detect a change in potential of ± 1 mV, usually included in commercial potentiostats. An output as a voltage is preferred for recording purposes.

3.5 Current-Measuring Instruments (Note 1)—An instrument that is capable of measuring a current accurately to within 1 % of the absolute value over a current range between 1.0 and $10^5 \mu\text{A}$ should be used. Many commercial units have a built-in instrument with an output as a voltage, which is preferred for recording purposes. For the purpose of the present test a logarithmic output is desirable.

3.6 Anodic Polarization Circuit—A scanning potentiostat is used for potentiodynamic measurements. Potential and current are plotted continuously using an X-Y recorder and a logarithmic converter (contained in the potentiostat or incorporated into the circuit) for the current. Commercially available units are suitable.

3.7 Electrodes:

3.7.1 The standard Type 304 stainless steel and Hastelloy Alloy C-276 should be machined into flat 0.625-in. (14-mm) diameter disks. The chemical composition of these alloys is listed in Table 1.

3.7.2 Counter Electrodes—The counter electrodes may be prepared as described in Practice G 5 or may be prepared from high-purity platinum flat stock and wire. A suitable method would be to seal the platinum wire in glass tubing and introduce the platinum electrode assembly

through a sliding seal. Counter electrodes should have an area at least twice as large as the test electrode.

3.7.3 Reference Electrode⁶—A saturated calomel electrode with a controlled rate of leakage (about $3 \mu\text{l/h}$) is recommended. This type of electrode is durable, reliable, and commercially available. Precautions should be taken to ensure that it is maintained in the proper condition. The potential of the calomel electrode should be checked at periodic intervals to ensure the accuracy of the electrode.

4. Standard Experimental Procedure

4.1 Test Specimen Preparation:

4.1.1 Wet grind with 240-grit SiC paper, wet polish with 600-grit SiC paper until previous coarse scratches are removed, rinse, and dry.

4.1.2 Prior to assembly of the specimen holder, ultrasonically degrease the specimen for 5 min in detergent and water, rinse thoroughly in distilled water, and dry.

4.1.3 Mount the specimen in the electrode holder. Tighten the assembly until the TFE-fluorocarbon gasket is sufficiently compressed to avoid leakage in the gasket.

4.2 Prepare a 3.56 % (by weight) sodium chloride solution by dissolving 34 g of reagent grade NaCl in 920 ml of distilled water.

4.3 Assemble the electrode holder and place in the polarization cell. Transfer 900 ml of test solution to the polarization cell, ensuring that the specimen remains above the solution level.

4.4 Bring the temperature of the solution of $25 \pm 1^\circ\text{C}$ by immersing the test cell in a controlled-temperature water bath or by other convenient means.

4.5 Place the platinum auxiliary electrodes, salt-bridge probe, and other components in the test cell. Fill the salt bridge with test solution and locate the probe tip approximately 1 mm from the working electrode.

NOTE 2—The levels of the solution in the reference and polarization cells should be the same. If this is impossible, a closed solution-wet (not greased) stopcock can be used in the salt bridge to eliminate siphoning.

4.6 Purge the solution sufficiently with an appropriate gas to remove oxygen before specimen immersion (minimum of 1 h).

⁶ Ives, D. J., and Janz, G. J., *Reference Electrodes, Theory and Practice*, Academic Press, New York, N.Y., 1961.

4.7 Immerse the specimen for 1 h before initiating polarization. A sliding seal can be used to ensure that an oxygen-free environment is maintained while the specimen is lowered. It is important that all oxygen be removed by purging prior to polarization, otherwise, more noble initial corrosion potential values will be observed.

4.8 Record the platinum potential 50 min after immersion of the specimen. Record the open-circuit specimen potential, that is, the corrosion potential, the instant before beginning polarization.

4.9 *Potential Scan*—Start the potential scan 1 h after specimen immersion, beginning at the corrosion potential (E_{corr}), and scan in the more noble direction at a scan rate of 0.6 V/h ($\pm 5\%$). Record the current continuously with change in potential on an X-Y recorder using semilogarithmic paper.

4.9.1 The onset of localized corrosion is usually marked by a rapid increase of the anodic current at potentials below the oxygen-evolution potential. When the current reaches 5 mA ($5 \times 10^3 \mu\text{A}$), reverse the scanning direction (toward more active potentials).

4.9.2 Continue the reverse scan until the hysteresis loop closes or until the corrosion potential is reached.

4.10 Plot anodic polarization data on semilogarithmic paper in accordance with Practice G 3 (potential-ordinate, current density-abscissa). A plot of representative polarization curves generated by the practice is shown in Fig. 2.

5. Standard Reference Plot

5.1 A standard polarization plot, based on the potentiodynamic data from five different laboratories, has been prepared. The plot has been separated into the forward (Fig. 3) and reverse (Fig. 4) scans for clarity. These plots show the mean values and a range of ± 2 standard deviations.

5.2 These plots were prepared from data obtained by following the standard procedure discussed in this practice.

5.3 A curve obtained by the potentiodynamic technique described above should show good agreement with the standard plots.

6. Reproducibility

6.1 The spread in data obtained from a number of laboratories and used in the preparation of the standard plot (Fig. 3 and 4) demonstrates the reproducibility that is possible when a standard procedure is followed.

6.2 The availability of a standard procedure, standard material, and standard plots should make it easy for an investigator to check his techniques to evaluate susceptibility to localized corrosion. An investigator's data should fall within the range of ± 2 standard deviations since this includes 95 % of all data, provided random variations are the only source of error.

7. Evaluation of Results

7.1 In general, localized corrosion can propagate at potentials more noble than that at which the hysteresis loop is completed. In these cases the more noble this potential, the less likely E_{corr} will exceed it, and the less likely that localized corrosion will occur.

7.2 An indication of the susceptibility to initiation of localized corrosion is given by the potential at which the anodic current increases rapidly (see Figs. 2 and 3). The more noble this potential, the less susceptible is the alloy to initiation of localized corrosion.

7.3 The polarization curves shown in Figs. 2, 3, and 4 indicate that initiation and propagation of localized corrosion occurs at potentials below the oxygen-evolution potential in Type 304 stainless steel. The curve for Hastelloy Alloy C-276 is not a result of localized corrosion but of uniform corrosion in the transpassive or oxygen evolution region. Since the corrosion potentials (E_{corr} values) for Hastelloy Alloy C-276 and Type 304 stainless steel are usually similar, these curves indicate that Hastelloy Alloy C-276 is more resistant to initiation and propagation of localized corrosion than Type 304 stainless steel.

**TABLE 1 Chemical Composition of Standard Alloys,
Weight %**

Element	Hastelloy C-276	Type 304 Stainless Steel
Carbon	0.003	0.060
Chromium	15.29	18.46
Cobalt	2.05	...
Columbium	...	0.11
Copper	...	0.17
Iron	5.78	balance
Manganese	0.48	1.43
Molybdenum	16.03	0.17
Nickel	balance	8.74
Phosphorus	0.018	0.029
Silicon	0.05	0.60
Sulfur	0.006	0.014
Vanadium	0.20	...
Tungsten	3.62	...

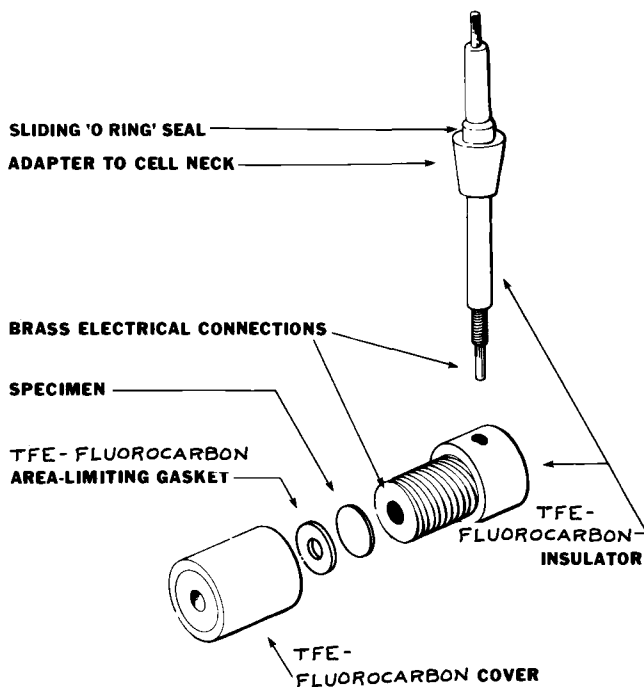


FIG. 1 Schematic Diagram of Specimen Holder.^{4,5}

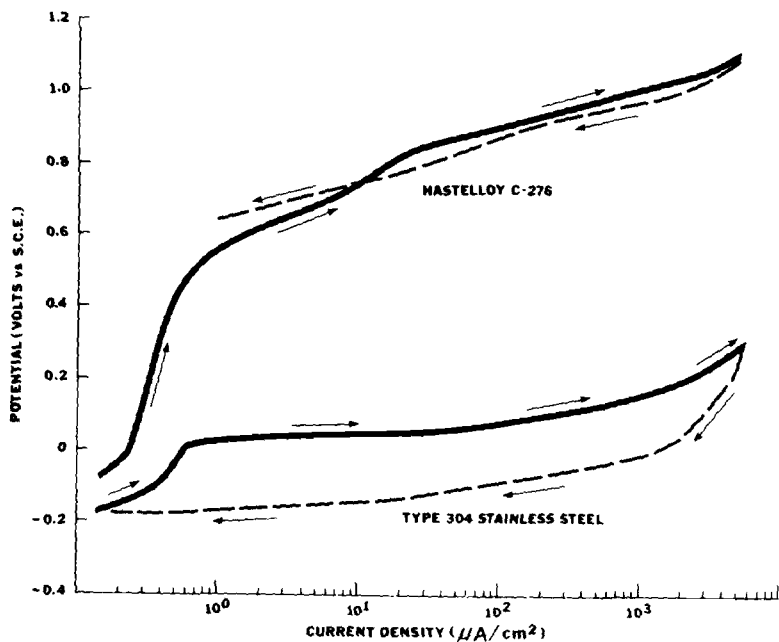


FIG. 2 Representative Cyclic Potentiodynamic Polarization Curves.

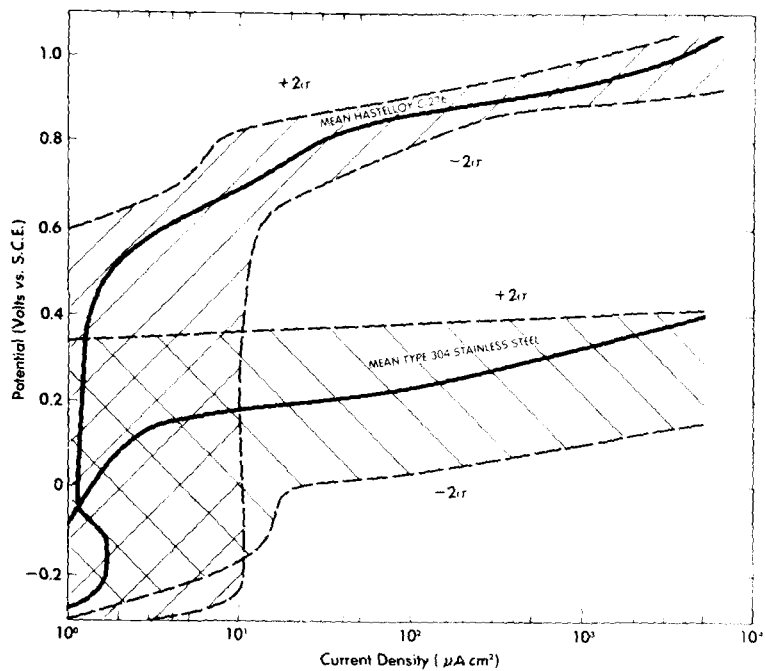


FIG. 3 Standard Potentiodynamic Polarization Plot (Forward Scan).

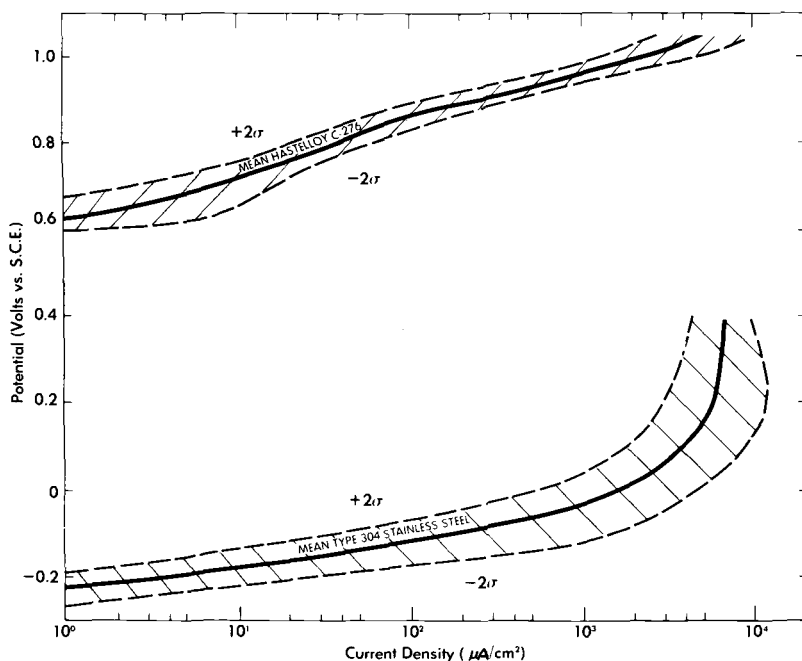


FIG. 4 Standard Potentiodynamic Polarization Plot (Reverse Scan).

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Standard Practice for CONDUCTING AND EVALUATING GALVANIC CORROSION TESTS IN ELECTROLYTES¹

This standard is issued under the fixed designation G 71; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice is an outline or guide for galvanic corrosion tests to characterize the behavior of two dissimilar metals in electrical contact in an electrolyte under low-flow conditions. It can be adapted to wrought or cast metals and alloys.

1.2 This practice covers the selection of materials, specimen preparation, test environment, method of exposure, and method for evaluating the results to characterize the behavior of galvanic couples in an electrolyte.

NOTE—Additional information on galvanic corrosion testing and examples of the conduct and evaluation of galvanic corrosion tests in electrolytes are given in Refs (1)² through (7).

2. Applicable Documents

2.1 *ASTM Standards:*

G 1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens³

G 3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing³

G 4 Method for Conducting Corrosion Coupon Tests in Plant Equipment⁴

G 16 Practice for Applying Statistics to Analysis of Corrosion Data³

G 31 Practice for Laboratory Immersion Corrosion Testing of Metals⁴

G 46 Recommended Practice for Examination and Evaluation of Pitting Corrosion³

3. Significance and Use

3.1 Use of this practice is intended to provide information on the galvanic corrosion of metals in electrical contact in an electrolyte that does not have a flow velocity sufficient to cause erosion-corrosion or cavitation.

3.2 This practice is presented as a guide for conducting galvanic corrosion tests in liquid electrolyte solutions, both in the laboratory and in service environments. Adherence to this practice will aid in avoiding some of the inherent difficulties in such testing.

4. Test Specimens

4.1 *Material*—Test specimens should be manufactured from the same material as those used in the service application being modeled. Minor compositional or processing differences between materials or between different heats can greatly affect the results in some cases.

4.2 *Size and Shape:*

4.2.1 The size and shape of the test specimens are dependent on restrictions imposed by the test location. When determining material behavior in the laboratory, it is advisable to use the largest specimens permissible within the constraints of the test equipment. In general, the ratio of surface area to metal volume should be large in order to favor maximum corrosion loss per weight. Sufficient thickness should be employed, however, to minimize the possibility of perforation of the specimens during the test exposure. When modeling large components, the size of the specimens should be as large as practical. When modeling smaller components, specimen size should be as close as possible to that of the application being modeled. Surface

¹ This practice is under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.07 on Galvanic Corrosion.

Current edition approved Nov. 27, 1981. Published January 1982.

² The boldface numbers in parentheses refer to the list of references appended to the practice.

³ *Annual Book of ASTM Standards*, Vol 03.02.

⁴ *Annual Book of ASTM Standards*, Vols 01.03 and 03.02.

area ratio in the test should be identical to the application being modeled. This ratio is defined as the surface area of one member of the couple divided by the surface area of the other member of the couple. Only the area in contact with the electrolyte (wetted area) is used in this calculation. In low-resistivity electrolytes, maintaining proximity between the materials being coupled may be more important than maintaining the exact area ratio. Also, with some couples, such as copper coupled to aluminum, there may be effects of corrosion products washing from one electrode to another which may have to be considered in determining specimen placement.

4.2.2 Laboratory tests are normally performed on rectangular plates or on cylinders. When modeling service applications, the shapes of the couple members should approximate the shapes in the application. Frequently complex shapes are simplified for testing purposes. The shape of the specimen is more important in electrolytes of low conductivity, where voltage drop in the electrolyte is significant. In highly conductive electrolytes, the shapes of the couple members may therefore deviate somewhat from the shapes in the application.

4.3 Specimen Preparation:

4.3.1 The edges of the test specimens should be prepared so as to eliminate all sheared or cold-worked metal except that cold-working introduced by stamping for identification. Shearing will, in some cases, cause considerable attack. Therefore, specimens having sheared edges should not be used. The edges should be finished by machining or polishing. The slight amount of cold working resulting from machining will not introduce any serious error.

4.3.2 Specimens should be cleaned in accordance with Practice G 1, or else the specimen surface condition should be similar to the application being modeled. The metallurgical condition of the specimens should be similar to the application being modeled. In all cases surface contamination, such as dirt, grease, oil, and thick oxides, should be removed prior to weighing and exposure to the test environment.

4.3.3 The specimen identification system must be one that will endure throughout the test period. Edge notches, drilled holes, stamped numbers, and tags are some of the methods used for identification. The identification system must not induce corrosion attack

in any way.

4.4 Number of Specimens:

4.4.1 The number of galvanic couples to be tested will be determined by whether or not one or more periodic specimen removals are scheduled during the course of the test. As a minimum, duplicate and preferably triplicate specimens should be tested for any given test period to determine the variability in the galvanic corrosion behavior. The effect of the number of replications on the application of the results is set forth in Practice G 16.

4.4.2 Control specimens should also be tested to provide corrosion rates of the individual metals and alloys without coupling for comparisons. These specimens should be of the same alloys, shapes, sizes, and metallurgical conditions as the materials in the couple.

5. Test Environment

5.1 Laboratory Tests:

5.1.1 In the laboratory, the test solution should closely approximate the service environment. The amount of test solution used depends on the size of the test specimens. A good rule of thumb is to use 40 cm³ of test solution for every 1 cm² of exposed surface area of both members of the couple. The volume of test solution may be varied to closely approximate the service application.

5.1.2 Galvanic corrosion tests conducted for an extensive period of time may exhaust important constituents of the original solution. Some accumulated corrosion products may act as corrosion accelerators or inhibitors. These variables may greatly change the end results, and replenishment of the solution should be chosen to be representative of the service application. A test system using continuously replenished test electrolytes is often the only solution to this problem.

5.1.3 Periodic measurements of the test environment should be made when the test duration in a fixed volume solution is for periods of several days or longer. These observations may include temperature, pH, O₂, H₂S, CO₂, NH₃, conductivity, and pertinent metal ion content.

5.2 Field Tests—Field testing should be performed in an environment similar to the service environment. Periodic measurements of those environmental variables which could vary with

time, such as temperature, dissolved O₂, etc., should be made.

6. Procedure

6.1 *Laboratory Versus Field Testing:*

6.1.1 Galvanic corrosion tests are conducted in the laboratory for several purposes: (1) inexpensive screening to reduce expensive field testing, (2) study of the effects of environmental variables, and (3) study of the corrosion accelerating or protective effects of various anode/cathode surface area ratios.

6.1.2 The materials proven in the laboratory to be the most promising should also be tested in the field, since it is frequently impossible to duplicate the actual service environment in the laboratory.

6.2 *Test Procedure:*

6.2.1 Specimens should be electrically joined before exposure. There are a number of methods for joining the specimens. Laboratory testing generally employs external electrical connection through wires such as to allow current measurement, (see Fig. 1). Field tests frequently employ direct contact physical bonding by threaded rods as in Fig. 2, soldering, brazing, etc. Prime considerations are that the electrical bond to the specimen will not corrode, which could result in decoupling, that the method of joining will not in itself be a galvanic couple or introduce other corrosion mechanisms (crevice, etc.), and that the resistance of the electrical path be small compared to the polarization resistance of the couple materials. Soldering or brazing will prevent the use of mass measurements for calculating corrosion rates. A coating may be applied to the electrical connections to prevent electrolyte access as in Fig. 2, provided the coating does not result in other corrosion phenomena, such as crevice attack, and is sufficiently resistant to the environment.

6.2.2 The physical relationship between the members of each couple should approximate that of the service situation being modeled. This is particularly important in electrolytes with low conductivity, since the effect of IR drops will be more noticeable. The specimens may be positioned by the use of nonconductive holders, provided that these do not result in other corrosion phenomena (crevice, etc.). A discussion of the mounting of specimens is included in Method G 4. The supporting device should not be affected by or cause contamination of the test

solution.

6.2.3 The coupled assembly is next immersed in the test electrolyte for the period of exposure. Exposure duration should be sufficient to allow prediction of the behavior for the entire service duration. If the service duration is long, corrosion data can be taken as a function of time until a curve can be developed that can be extrapolated to the service duration, provided that steady-state conditions have been reached and that no transient environmental conditions are expected in service to affect this steady state.

6.2.4 Specimen removal should be based on a preplanned removal schedule.

7. Evaluation of Test Specimens

7.1 *Measurements During Exposure*—Data recorded during exposures may include galvanic current measurements and couple and control specimen potentials measured relative to a suitable reference half-cell as recommended in Practice G 3. Current data can then be converted into a theoretical corrosion rate based on Faraday's law.

7.2 *Measurements After Removal:*

7.2.1 After removal, samples of corrosion products may be obtained for chemical and physical analysis. The specimens should then be cleaned of deposits (such as biofouling from fresh or seawater) by scraping or brushing with a wooden scraper or soft bristle brush. Visual observations should be recorded before and after this initial cleaning operation. Color photographs may be taken of each specimen before and after cleaning. Final cleaning of specimens should be in accordance with Practice G 1 after which the specimens should be weighed to determine galvanic corrosion weight loss which can be converted to corrosion as set forth in Practice G 31. Additional recommendations for specimen cleaning may be found in Method G 4 and Practice G 31.

7.2.2 In some cases, mass loss measurements will not be possible or meaningful. For example, soldered assemblies cannot be separated into their components without introducing extra mass due to the remaining solder. In this case, corrosion evaluation of the end product configuration must be based on visual assessments, thickness loss measurements, or on other techniques. Materials suffering localized cor-

rosion such as pitting may be analyzed using Recommended Practice G 46, and those suffering crevice corrosion should have the depth of attack measured and described in detail, with attention to changes at the edges as well as the surfaces. In addition, changes in physical properties such as breaking strength can also be measured. Metallographic examination of specimen cross sections may be necessary to determine parting corrosion depth.

7.2.3 Regardless of the method of assessment, the behavior of the coupled materials should be compared to that of the uncoupled controls. Subtracting control values from values of coupled specimens yields the increase in corrosion due to coupling. A ratio of couple data to the uncoupled data has been used to determine a percentage change in corrosion due to the couple (acceleration factor).

7.2.4 Where replicate couples are exposed, statistical analysis of the data, as set forth in Practice G 16, may be applied to generate confidence intervals for predictive purposes.

8. Report

8.1 The report should include detailed descriptions of the exposed specimens including wetted areas, pertinent data on exposure conditions including the geometry used, the deposits formed, and results of the corrosion evaluation.

8.2 Data for the exposed specimens should include physical dimensions, chemical compo-

sition, metallurgical history, surface preparation, and after-exposure cleaning methods.

8.3 Details of exposure conditions should include location, dates, and periods of exposure and description of the environmental conditions prevailing during the exposure period, including electrolyte conductivity.

8.4 The results of the tests may be expressed as corrosion rate in penetration per unit time (for example, millimetres per year) or loss in thickness or mass during the exposure period. Rates for both control (uncoupled) and coupled samples should be reported, with the change in rate due to the coupling reported as either the difference between control and coupled samples or as the coupled rate divided by the control rate (acceleration factor).

8.5 In cases where corrosion is in the form of pitting or crevice corrosion, a pitting factor should be reported in accordance with Recommended Practice G 46. Depths of pitting should be recorded. Pitting factors or depths can then be used to determine the change in corrosion due to coupling.

8.6 If any physical property of the specimens is measured after exposure, the change in that property should be reported and these values can be compared between control and coupled samples in the same manner as corrosion rates or pitting factors.

8.7 Changes in the physical appearance of the specimens during the exposure period should be reported.

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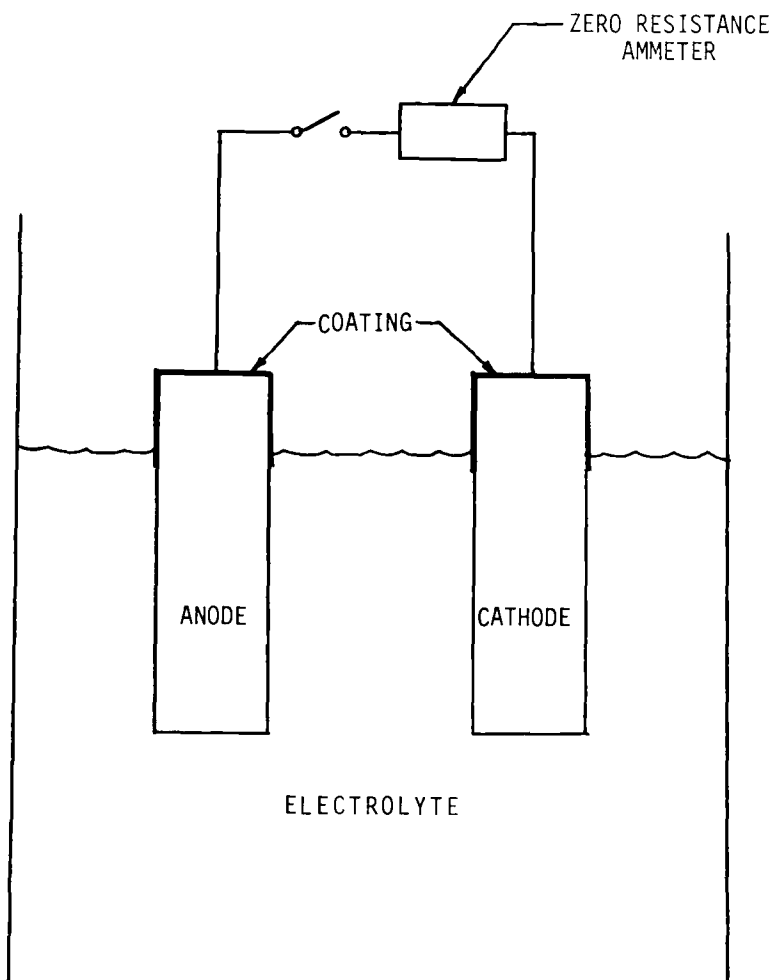
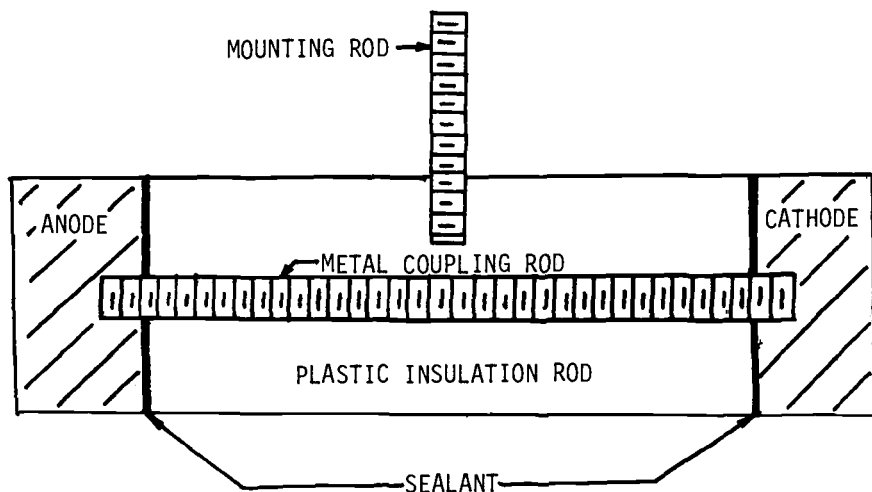


FIG. 1 Laboratory Galvanic Corrosion Test Setup With Facility for Measuring Galvanic Current



NOTE—The length of the plastic insulation rod should approximate the distance between the anode and the cathode of the final product.

FIG. 2 Specimen Configuration for Galvanic Corrosion Tests of Bar Stock Material

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, Pa. 19103.



Standard Practice for MODIFIED SALT SPRAY (FOG) TESTING¹

This standard is issued under the fixed designation G 85; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

NOTE—Corrections were made editorially to A2.2.1 and the designation date was changed on Jan. 3, 1985.

1. Scope

1.1 This practice sets forth conditions for four modifications in salt spray (fog) testing for specification purposes. These are in chronological order of their development:

1.1.1 *Annex A1*, acetic acid-salt spray test, continuous Method B 287.

1.1.2 *Annex A2*, cyclic acidified salt spray test.

1.1.3 *Annex A3*, seawater acidified test, cyclic (SWAAT), Method G 43.

1.1.4 *Annex A4*, SO₂ salt spray test, cyclic.

1.2 Suitable apparatus that may be used to obtain these conditions is described in Annex A1 of Method B 117.

1.3 This practice does not prescribe the type of modification, test specimen or exposure periods to be used for a specific product, nor the interpretation to be given to the results.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Applicable Documents

2.1 *ASTM Standards:*

B 117 Method of Salt Spray (Fog) Testing²

B 287 Method of Acetic Acid-Salt Spray (Fog) Testing²

D 609 Method for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products³

D 1141 Specification for Substitute Ocean Water⁴

D 1193 Specification for Reagent Water⁵

D 1654 Method for Evaluation of Painted or

Coated Specimens Subjected to Corrosive Environments³

E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode⁶

G 43 Method of Acidified Synthetic Sea Water (Fog) Testing²

3. Significance and Use

3.1 This practice is applicable to ferrous and nonferrous metals; also organic and inorganic coatings. The variations described herein are useful when a more corrosive environment than the salt fog described in Method B 117 is desired.

4. Apparatus

4.1 *Cabinet:*

4.1.1 The apparatus required for salt spray (fog) testing consists of a fog chamber, a salt solution reservoir, a supply of suitably conditioned compressed air, one or more atomizing nozzles, specimen supports, provision for heating the chamber, and necessary means of control. The size and detailed construction of the cabinet are optional, provided the conditions obtained meet the requirements of this practice. The material of construction shall be such that it will not affect the corrosiveness of the fog. Suitable apparatus that may be used to obtain these conditions is described in Appendix A1 of Method B 117.

4.1.2 Design the cabinet so that drops of so-

¹ This practice is under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.

Current edition approved Jan. 3, 1985. Published August 1985.

² *Annual Book of ASTM Standards*, Vols 02.05 and 03.02.

³ *Annual Book of ASTM Standards*, Vol 06.01.

⁴ *Annual Book of ASTM Standards*, Vol 11.02.

⁵ *Annual Book of ASTM Standards*, Vol 11.01.

⁶ *Annual Book of ASTM Standards*, Vol 15.05.

lution that accumulate on the ceiling or cover of the chamber do not fall on the specimens being tested. Do not return drops of solution that fall from the specimens to the solution reservoir for respraying.

4.1.3 Equip the cabinet with one or more timing devices to provide for intermittent spraying or periodic introduction of a gas, or both.

4.2 Air Supply:

4.2.1 Make sure the compressed air supply to the nozzle or nozzles for atomizing the salt solution is free of oil and dirt and maintain the air supply between 69 and 172 kPa (10 and 25 psi).

NOTE 1—The air supply may be freed of oil and dirt by passing it through a water scrubber or at least 610 mm (2 ft) of suitable cleaning material such as asbestos, sheep's wool, or activated alumina.

4.2.2 Temperature in the saturator tower (bubble tower) varies depending on the test method used.

4.3 Conditions in Salt-Spray Chamber:

4.3.1 *Temperature*—The temperature in the exposure zone varies with the test method used. For recommended exposure zone temperatures for the various methods see the Annex. Record the temperature within the exposure zone of the closed cabinet at least twice a day at least 7 h apart (except on weekends and holidays, when the salt spray test is not interrupted for exposing, rearranging, or removing test specimens or to check and replenish the solution in the reservoir).

NOTE 2—Suitable methods to record the temperature are a continuous recording device or a thermometer which can be read from outside the closed cabinet. Obtain the recorded temperature with the salt spray chamber closed to avoid a false low reading because of wet-bulb effect when the chamber is open.

4.3.2 *Atomization and Quantity of Fog*—Place at least two clean fog collectors within the exposure zone so that no drops of solution from the test specimens or any other source can be collected. Position the collectors in the proximity of the test specimens, one nearest to any nozzle and the other farthest from all nozzles. Make sure that for each 80 cm² of horizontal-collecting area fog accumulates in each collector from 1.0 to 2.0 mL of solution per hour based on an average run of at least 16 h continuous spray.

NOTE 3—Suitable collecting devices are glass funnels with the stems inserted through stoppers into graduated cylinders or crystallizing dishes. Funnels and dishes with a diameter of 100 mm have an area of about 80 cm².

4.3.3 Direct or baffle the nozzle or nozzles so that none of the spray can impinge directly on the test specimens.

5. Test Specimens

5.1 Define the type and number of test specimens to be used, as well as the criteria for the evaluation of the test results in the specifications covering the material or product being tested or upon mutual agreement between the purchaser and the seller.

5.2 Preparation of Test Specimens:

5.2.1 Clean metallic and metallic-coated specimens. The cleaning method is optional depending on the nature of the surface and the contaminants; however, when using a cleaning method do not include in the contents abrasives other than a paste of pure magnesium oxide nor of solvents which may form corrosive or inhibitive films. The use of nitric acid solution for the chemical cleaning, or passivation, of stainless steel specimens is permissible when agreed upon between the purchaser and the seller. Take care that specimens are not recontaminated after cleaning by excessive or careless handling.

5.2.2 Prepare specimens for evaluation of paints and other organic coatings in accordance with applicable specification(s) for the material(s) being tested, or as agreed upon between the purchaser and supplier. Otherwise, make sure the test specimens consist of steel meeting the requirements of Methods D 609; clean and prepare the specimens for coating in accordance with applicable procedure of Methods D 609.

5.2.4 Whenever it is desired to determine the development of corrosion from an abraded area in the paint or organic coating, make a scratch or scribed line through the coating with a sharp instrument so that the underlying metal is exposed before testing. Use the conditions of making the scratch as defined in Method D 1654, unless otherwise agreed upon between the purchaser and seller.

5.2.5 Protect the cut edges of plated, coated, or duplex materials and areas that contain identification marks or that are in contact with the racks or supports with a suitable coating that is stable under the conditions of the test, such as ceresin wax, unless otherwise specified.

NOTE 4—Should it be desirable to cut test specimens from parts or from preplated, painted, or otherwise coated-steel sheet, protect the cut edges by coating them

with paint, wax, tape, or other effective media so that the development of preferential attack or a galvanic effect between such edges and the adjacent plated or otherwise coated-metal surfaces, is prevented.

6. Salt Solutions

6.1 Make the salt solutions by using either synthetic sea salt in accordance with Specification D 1141 or sodium chloride in accordance with Method B 117. Make-up water shall be distilled or deionized water conforming to Type III reagent water described in Specification D 1193.

6.2 *Synthetic Sea Salt Solution:*

6.2.1 Make the salt solution so that it consists of 42 g of synthetic sea salt in accordance with Specification D 1141 per litre of solution (see Note 5).

6.3 *Sodium Chloride Solution:*

6.3.1 Prepare the salt solution by dissolving 5 ± 1 parts by weight of sodium chloride in 95 parts of distilled water or water containing not more than 200 ppm of total solids (see Note 5). The sodium chloride shall be substantially free of nickel and copper and shall contain on the dry basis not more than 0.1 % of sodium iodide and not more than 0.3 % of total impurities. Some salts contain additives that may act as corrosion inhibitors; careful attention should be given to the chemical content of the salt. Upon agreement between purchaser and seller, analysis may be required and limits established for elements or compounds not specified in the chemical composition given above.

NOTE 5—A solution having a specific gravity of 1.0255 to 1.0400 at 25°C (77°F) will meet the concentration requirement of 6.2.1 and 6.3.1. It is suggested that a daily check be made.

6.4 The pH of the salt solutions will vary depending on the test method used. Before the solution is atomized, free it of suspended solids (see Note 6). Take the pH measurements electrometrically at 25°C (77°F) using a glass electrode with a saturated potassium chloride bridge in accordance with Test Method E 70.

NOTE 6—The freshly prepared salt solution may be filtered or decanted before it is placed in the reservoir, or the end of the tube leading from the solution to the atomizer may be covered with a double layer of cheesecloth or suitable nonmetallic filter cloth to prevent plugging of the nozzle.

7. Procedure

7.1 *Position of Specimens During Test:*

7.1.1 Unless otherwise specified, support or

suspend the specimens between 6 and 45° from the vertical, and preferably parallel to the principal direction of horizontal flow of fog through the chamber, based upon the dominant surface being tested. Note that test severity increases as angle from the vertical increases.

7.1.2 Do not allow contact of the specimens between each other, between any metallic material, or between any material capable of acting as a wick.

7.1.3 Place each specimen so as to permit free settling of fog on all specimens. A minimum spacing between specimens of 30 mm is recommended.

7.1.4 Do not permit the salt solution from one specimen to drip on any other specimen.

7.1.5 It is recommended that placement of replicate specimens be randomized to avoid possible bias caused by difference in spray patterns. Individual specimens may also be rotated daily for the same reason.

7.1.6 Suitable materials for the construction or coating of racks and supports are glass, rubber, plastic, or suitably coated wood. Do not use bare metal. Support specimens preferably from the bottom or the side. Slotted wooden, laminated plastic, or inert plastic strips are suitable for the support of flat panels. Suspension from glass hooks or waxed string may be used as long as the specified position of the specimens is obtained, and, if necessary, by means of secondary support at the bottom of the specimens.

7.2 *Continuity of Test:*

7.2.1 Unless otherwise specified, in the specifications covering the material or product being tested, allow the test to be continuous for the duration of the entire test period. Continuous operation implies that the chamber be closed except for the short daily interruptions necessary to inspect, rearrange, or remove test specimens, and to check and replenish the solution in the reservoir. Schedule operations so that these interruptions are held to a minimum.

7.3 *Period of Test:*

7.3.1 Designate the period of test in accordance with the specifications covering the material or product being tested or as mutually agreed upon between the purchaser and the seller. Exposure periods of multiples of 24 h are suggested.

7.4 *Cleaning of Tested Specimens:*

7.4.1 Unless otherwise specified in the specifications covering the material or product being tested, at the end of the test, specimens may be

gently washed or dipped in clean running water no warmer than 38°C (100°F) to remove salt deposits from their surface, and then immediately dried. Dry with a stream of clean, compressed air.

NOTE 7—Drying with compressed air may not be desirable for aluminum specimens to be tested for exfoliation corrosion resistance.

8. Evaluation of Results

8.1 Make a careful and immediate examination for the extent of corrosion of the dry test specimens or for other failure as required by the specifications covering the material or product being tested or by agreement between the purchaser and the seller.

9. Records and Reports

9.1 Record the following information, unless otherwise prescribed in the specifications covering the material or product being tested:

9.1.1 Type of salt and water used in preparing the salt solution,

9.1.2 All readings of temperature within the exposure zone of the chamber,

9.1.3 Weekly records of data obtained from each fog-collecting device including the following:

9.1.3.1 Volume of salt solution collected in millilitres per hour per 80 cm²,

9.1.3.2 Concentration or specific gravity at 35°C (95°F) of solution collected, (see Note 8), and

9.1.3.3 pH of collected solution.

9.1.4 Type of specimen and its dimensions, or number or description of part,

9.1.5 Method of cleaning specimens before and after testing,

9.1.6 Method of supporting or suspending article in the salt spray chamber,

9.1.7 Description of protection used as required in 5.2.5,

9.1.8 Exposure period,

9.1.9 Interruptions in test, cause and length of time, and

9.1.10 Results of all inspections.

NOTE 8—It is also advisable to record the concentration or specific gravity of any atomized salt solution that has not made contact with the test specimen and that was returned to the reservoir.

ANNEX

Mandatory Information

A1. ACETIC ACID-SALT SPRAY (FOG) TESTING—(METHOD B 287)

A1.1 Salt Solution:

A1.1 A sodium chloride solution made in accordance with 6.3.

A1.1.2 Adjust the pH of this solution measured in accordance with Test Method E 70 to range from 3.1 to 3.3 by the addition of acetic acid.

NOTE A1—The initial solution may be adjusted to pH of 3.0 to 3.1 with the expectation that the pH of the collected fog will be within the specified limits. Base the adjustment of the initial pH for make-up solution upon the requirements to maintain the required pH of the collected samples. If less than 0.1 or more than 0.3 % of the glacial acetic acid is required to attain the specified pH, the purity of the water or salt, or both may not be satisfactory.

A1.2 Conditions in Saturator Tower:

A1.2.1 Make sure the temperature in the saturator tower (bubble tower) is $47 \pm 1^\circ\text{C}$ ($117 \pm 2^\circ\text{F}$).

A1.3 Conditions in the Salt Spray Chamber:

A1.3.1 Temperature—Maintain the exposure zone of the acetic acid-salt spray fog chamber at 35 ± 1.1 or -1.7°C (95 ± 2 or -3°F).

NOTE A2—This test is particularly applicable to research studies that have the effect of altering parameters of the electroplating process in connection with decorative chromium plating on steel or zinc die-cast base as well as for the evaluation of the quality of the product. This is true because of the normal duration of the test, which may be as brief as 16 h, but normally runs for 144 to 240 h or more giving ample opportunity for observations at practical intervals of the effects of minor parameter changes.

A2. CYCLIC ACIDIFIED SALT FOG TESTING

A2.1 Salt Solution:

A2.1 Use a sodium chloride solution made in ac-

cordance with 6.3.

A2.1.2 Adjust the pH of this solution to range from

2.8 to 3.0 by the addition of acetic acid.

A2.2 Conditions in Saturator Tower:

A2.2.1 Make sure the temperature in the saturator tower (bubble tower) is $57 \pm 1^\circ\text{C}$ ($135 \pm 2^\circ\text{F}$).

A2.3 Conditions in Salt-Spray Chamber:

A2.3.1 *Temperature*—Maintain the temperature in the exposure zone of the salt spray chamber at 49 ± 1.1 or -1.7°C ($120^\circ\text{F} + 2$ or -3°F).

A2.3.2 *Humidity*—Although the humidity limits for optimum test conditions have not been determined, operate the salt spray chamber under wet bottom conditions (that is, make sure an inch or so of water is present in the bottom of the box) for most testing. This ensures that the interior of the box does not become dry, a condition that decreases corrosion rate. (The dry bottom is recommended, however, for testing 2000-series aluminum alloys and paint coatings that require a less aggressive environment.)⁷

A2.3.3 *Cabinet*—Equip the chamber with a timing device that can be used for the following 6-h repetitive cycles: $\frac{3}{4}$ -h spray; 2-h dry-air purge; and $\frac{3}{4}$ -h soak at high relative humidity.

A2.3.4 *Purge*—Purging of the fog atmosphere immediately after spraying is the most unique feature of

this test. Dry all droplets of water on the specimens and dry the corrosion products so that they are of a white, rather than a damp gray appearance. Perform this by electrically switching the air-flow to by-pass the saturator tower and aspirator nozzle and allowing it to enter directly into the test chamber for 120 min at an angle that sweeps the fog out of the peaked lid of the cabinet. This reduces the relative humidity from 40 to 7 %, depending on the climatic conditions of the ambient air. After purging, the specimens remain in the closed cabinet until the next spray cycle. Since most testing requires a wet bottom, the humidity gradually increases from 65 to 95 % during this period.

A2.3.5 *Atomization and Quantity of Fog*—Collect the fog in a special continuous spray run periodically between test runs. Determine the proper consumption of solution by monitoring solution level in the glass reservoir.

⁷ Personal communication, Alcoa Technical Center, New Kensington, PA.

A3. ACIDIFIED SYNTHETIC SEA WATER (FOG) TESTING (METHOD G 43)

A3.1 Salt Solution:

A3.1.1 Use a synthetic sea salt solution made in accordance with 6.2 with the addition of 10 mL of glacial acetic acid per litre of solution.

A3.1.2 Adjust the pH of the salt solution between 2.8 and 3.0.

A3.2 *Conditions in Saturator Tower*—Make sure the temperature in the saturator tower (bubble tower) is $47 \pm 1^\circ\text{C}$ ($117 \pm 2^\circ\text{F}$) if cabinet temperature is 35°C (95°F); and $57 \pm 1^\circ\text{C}$ ($135 \pm 2^\circ\text{F}$) if cabinet temperature is 49°C (120°F).

A3.3 Conditions in Salt Spray Chamber:

A3.3.1 *Temperature*—The temperature in the exposure zone of the salt spray chamber may vary to suit the material being tested. The specifications that cover the material or product being tested define the temperature or the temperature may be mutually agreed upon between the purchaser and the seller. See Note A3 for recommended exposure zone temperatures for some materials.

It is also applicable to developmental studies of varying heat treatment parameters to determine effect on corrosion behavior. For this purpose, a temperature of 49°C (120°F) is recommended for the exposure zone. For testing organic coatings on various metallic substrates, an exposure zone temperature of 24 to 35°C (75 to 95°F) may be used since temperatures in excess of 35°C frequently result in paint blistering.

A3.3.2 *Humidity*—Although the humidity limits for optimum test conditions have not been determined, results of an interlaboratory testing program indicate that it is necessary to operate under wet bottom conditions (that is, an inch or so of water should always be present in the bottom of the box). This ensures that the interior of the box does not become dry, a condition that will decrease the corrosion rate.

A3.3.3 *Cabinet*—Equip the cabinet with a timing device that can be used for the following cycle: 30-min spray followed by 90-min soak at above 98 % relative humidity.

⁸ Ketcham, S. J., and Jeffrey, P. W., "Localized Corrosion—Cause of Metal Failure." *ASTM STP 516*. ASTM, 1973, pp. 273–302.

NOTE A3—This test is particularly applicable to production control of exfoliation-resistant heat treatments for the 2000, 5000, and 7000-series aluminum alloys.⁸

A4. SALT/SO₂ SPRAY (FOG) TESTING

A4.1 This test consists of spraying salt fog with introduction of SO₂ gas directly into the chamber periodically.

A4.2 Salt Solution:

A4.2.1 Define the salt solution by using the specifications covering the material or product being tested or upon mutual agreement between the purchaser and the seller.

A4.2.2 If synthetic sea salt is specified, prepare it in accordance with 6.2.

A4.2.3 If sodium chloride is specified, prepare it in accordance with 6.3.

A4.3 Conditions in Saturator Tower:

A4.3.1 Make sure the temperature in the saturator tower (bubble tower) is $47 \pm 1^\circ\text{C}$ ($117 \pm 2^\circ\text{F}$).

A4.4 Conditions in the Salt Spray Chamber:

A4.4.1 *Temperature*—Maintain the exposure zone of the salt spray chamber at 35 ± 1.1 , -1.7°C (95 ± 2 , -3°F).

A4.4.2 *SO₂ Gas*—Equipment and materials required for addition of SO₂ to cabinet are as follows:

A4.4.2.1 Cylinder of SO₂ gas.

A4.4.2.2 Flowmeter capable of measuring SO₂ gas flow of $1 \text{ cm}^3/\text{min} \cdot \text{ft}^3$ of cabinet volume ($35 \text{ cm}^3/\text{min} \cdot \text{m}^3$); also constructed of materials inert to SO₂ gas.

A4.4.2.3 Timer.

A4.4.2.4 Two-way solenoid valve fabricated of materials inert to SO₂ gas.

A4.4.2.5 Tubing and fittings for SO₂ line of materials inert to SO₂ gas. A schematic of the SO₂ line is shown in Fig. A4.1.

NOTE A4—It is highly desirable to have the SO₂ gas introduced into the chamber in such a way that a uniform dispersion throughout the interior will result.

If the cabinet is equipped with a central dispersion tower, holes can be drilled in the poly(methyl methacrylate) (PMMA) baffle at the top of the tower so that the gas comes out of eight uniformly spaced ports. There are undoubtedly other means for accomplishing a uniform dispersion of the gas. However, avoid introducing the gas into the chamber through one or two tubes at the side.

A4.4.3 The pH of the collected solution range from 2.5 to 3.2.

A4.4.4 *Cycle*—Define the cycle to be used by using the specifications covering the material or product being tested or upon mutual agreement between the purchaser and the seller. Examples of some possible cycles are as follows:

A4.4.4.1 Constant spray with introduction of SO₂ gas for $1 \text{ h } 4 \times \text{a day}$ (every 6 h).

A4.4.4.2 $\frac{1}{2}$ -h salt spray, $\frac{1}{2}$ -h SO₂, 2-h soak.

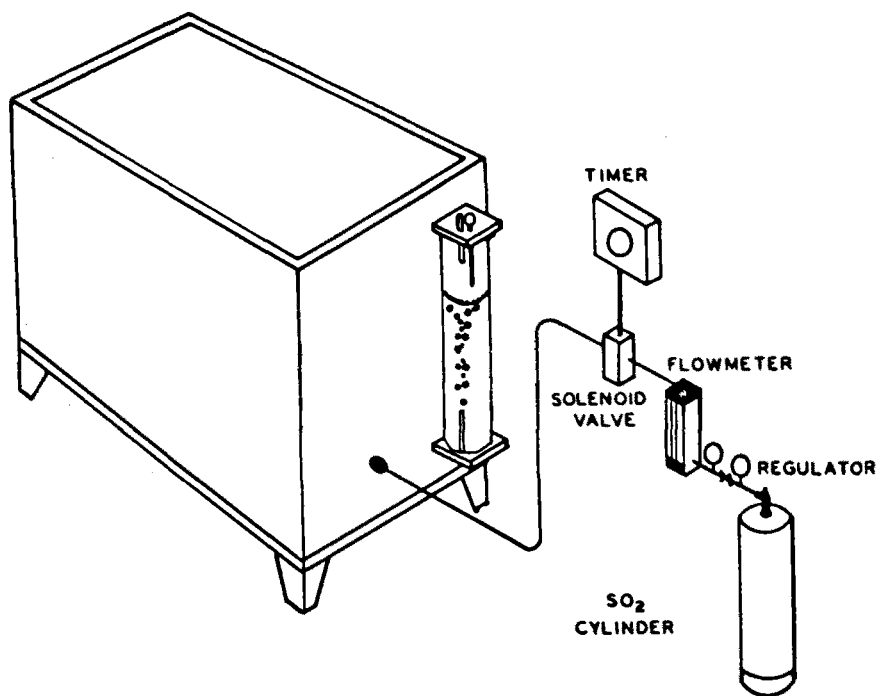


FIG. A4.1 Schematic of SO₂ Line into Salt Fog Cabinet

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Standard Practice for CONDUCTING MOIST SO₂ TESTS¹

This standard is issued under the fixed designation G 87; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice describes the apparatus and procedure to be used in conducting qualitative assessment tests in accordance with the requirements of material or product specifications by means of specimen exposure to condensed moisture containing sulfur dioxide.

1.2 The exposure conditions may be varied to suit particular requirements and this practice includes provisions for use of different concentrations of sulfur dioxide and for tests either running continuously or in cycles of alternate exposure to the sulfur dioxide containing atmosphere and to the ambient atmosphere.

1.3 The variant of the test to be used, the exposure period required, the type of test specimen, and the criteria of failure are not prescribed by this practice. Such details are provided in appropriate material and product purchase specifications.

1.4 The values stated in SI units are to be regarded as standard. The inch-pound values in parentheses may be approximate.

1.5 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* (For specific precautionary statement, see 9.1.1.)

2. Applicable Documents

2.1 ASTM Standards:

D 714 Method of Evaluating Degree of Blistering of Paints²

D 1193 Specification for Reagent Water³

D 1654 Method for Evaluation of Painted or

Coated Specimens Subjected to Corrosive Environments²

3. Significance and Use

3.1 Moist air containing sulfur dioxide quickly produces easily visible corrosion on many metals in a form resembling that occurring in industrial environments. It is therefore a test medium well suited to detect pores or other sources of weakness in protective coatings and deficiencies in corrosion resistance associated with unsuitable alloy composition or treatments.

3.2 The results obtained in the test should not be regarded as a general guide to the corrosion resistance of the tested materials in all environments where these materials may be used. Performance of different materials in the test should only be taken as a general guide to the relative corrosion resistance of these materials in moist SO₂ service.

4. Apparatus

4.1 The apparatus required for moist SO₂ testing consists of a test chamber having an internal capacity of 300 L (10.6 ft³), a supply of sulfur dioxide with metering device, specimen supports, provisions for heating the chamber, and necessary means of control. The size and detailed construction of the apparatus are optional, provided the conditions obtained meet the requirements of this method. Suitable apparatus which may be used to obtain these conditions is described in Appendix X1.

¹ This practice is under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.

Current edition approved April 27, 1984. Published July 1984.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vol 11.01.

4.2 Drops of condensing moisture which accumulate on the ceiling of the chamber shall not be permitted to fall on the specimens being tested.

5. Sampling

5.1 The specific location of samples in a mill product, the number of samples that should be tested, and other factors concerning sampling, are not within the scope of this practice. These factors should be mutually agreed upon between purchaser and supplier (see 6.1).

6. Test Specimens

6.1 Select the number and type of test specimens, and their shape and dimensions according to the specification covering the product or material being tested or agreed upon between purchaser and supplier.

6.2 The total combined exposed surface area of the material tested at any one time should be substantially the same and unless otherwise agreed upon, it shall be $0.5 \pm 0.1 \text{ m}^2$.

6.3 To obtain quantitative corrosion-rate data, only materials with similar reactivities should be included in a test run.

6.4 Preparation of Test Specimens:

6.4.1 Suitably clean the specimens before testing. Unless otherwise agreed upon the cleaning method shall be optional depending on the nature of the surface and of likely contamination except that it shall not include the use of abrasives (other than a paste of pure magnesium oxide) or of solvents that are corrosive or that may deposit either corrosion-promoting or protective films. Take care that specimens are not recontaminated by excessive or careless handling after cleaning.

6.4.2 If test specimens are cut from a larger coated article, carry out the cutting in such a way that coating damage is minimized in the area adjacent to the cut. Unless otherwise specified, adequately protect the cut edges by coating them with a suitable medium, stable under the conditions of the test, such as wax or tape. High-quality platers tape or microcrystalline wax are generally suitable.

6.5 Position of Specimens During Test:

6.5.1 Place the test specimens in the cabinet so that no part of any specimen is within 20 mm (5 in.) of another or within 100 mm (25 in.) of the walls or the ceiling or within 200 mm (50 in.) of the surface of the water in the base of the

chamber.

6.5.2 Arrange the specimens so that moisture which may condense on any of them or their supports will not fall on other specimens placed at lower levels.

6.5.3 Unless otherwise agreed upon, the angle of inclination of test surfaces to the vertical is optional. However, a near vertical orientation (0 to 10° from vertical) is suggested.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available⁴. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV of Specification D 1193.

7.3 A commercially available source of bottled SO_2 gas having a minimum purity of 99.9 % (liquid phase) with proper regulator, and means of measuring the volumes of gas required for delivery into test chamber shall be provided.

7.3.1 **Caution**—Suck-back into cylinder may cause explosion. Always use a check valve, vacuum break, or other protective apparatus in any line or piping from cylinder to test chamber to prevent suck-back.

7.4 The volume of gas delivered into test chamber shall be measured by means of either a properly calibrated flowmeter (rotameter type) with metering valve, or in a gas buret using viscous paraffin oil as the pressure controlling fluid.

7.4.1 The volume to be measured may be as small as 0.2 L, and measures should be taken to avoid errors from such as air contained in delivery tubes between flowmeter or gas burette and test chamber.

⁴"Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

8. Conditioning

8.1 Operate a new chamber for at least ten 24-h cycles without introduction of any test material by the procedure applicable to an atmosphere containing an addition of 2 L of sulfur dioxide before it is brought into use for testing. This should reduce any risks of contamination of the atmosphere by vapors from construction materials of chamber.

9. Procedure

9.1 Introduce 2 ± 0.2 L of distilled water into the base of chamber.

9.2 Place the test specimens in position and close the door of the chamber.

9.3 Introduce the volume of sulfur dioxide, required by the governing materials specification, into the chamber through the inlet pipe. Usually this volume will be 0.2, 1, or 2 L.

9.4 Switch on the heater below the chamber, and raise the temperature inside the chamber to $40 \pm 3^\circ\text{C}$ ($104 \pm 5.4^\circ\text{F}$) in about 1.5 h. Make sure the heating is under control to keep the temperature inside the chamber at $40 \pm 3^\circ\text{C}$ ($104 \pm 5.4^\circ\text{F}$) for the specified period.

9.5 One test cycle is 24 h and may be either continuous exposure of the specimens or alternate exposure for 8 h followed by drying in the ambient atmosphere inside the chamber for 16 h. For either test cycle, replace the water in the chamber and the sulfur dioxide in the air of the chamber before each 24-h cycle begins.

9.5.1 For a test of continuous exposure in the chamber for 24 h, the replacement of water and sulfur dioxide for a second period of test shall be made with a minimum of disturbance of the test specimens.

9.5.2 For specimens exposed to ambient conditions for part of the test cycle, such conditions shall be an indoor atmosphere with no unusual pollution other than that arising from the test cabinet, a temperature in the range of 20 to 30°C (68 to 86°F), and a relative humidity below 75 %.

9.6 *Duration of Test*—The number of cycles shall be designated by the specification for the material or product being tested or mutually agreed upon by purchaser and seller.

9.7 *Cleaning of Tested Specimens*—At the end of the test period, remove the specimens from the chamber. Before they are examined, allow them to hang freely in a normal indoor atmosphere until any fluid corrosion product

dries. Examine with all corrosion product in position and any cleaning carried out depends on the criteria laid down for evaluation of the test result.

10. Evaluation of Results

10.1 Different criteria for evaluation of the results of the test may be applied to meet particular requirements, for example, mass change, change in appearance, alteration revealed by micrographic examination, or change in mechanical properties. Usually the appropriate criteria will be indicated in the specification for the material or product tested. For most routine applications of the test, only the following need to be considered:

10.1.1 Appearance after drying in air.

10.1.2 Appearance after removing superficial corrosion products by washing with water and a soft bristle brush.

10.1.3 The number and distribution of corrosion defects, for example, pits, cracks, blisters, and other defects may conveniently be assessed by methods such as that described in Method D 1654, and Method D 714, and

10.1.4 The number of cycles elapsing before the appearance of the first sign of corrosion.

11. Report

11.1 The report shall indicate the outcome of the test according to the criteria for evaluation of results prescribed for the test. The result obtained for each specimen tested and, when appropriate, the average result for a group of replicate test specimens shall be reported. The report, may, if required, be accompanied by photographic records of the tested specimens.

11.2 The report shall contain information about all test details. This information may vary according to the purposes of the test and to the directions prescribed for it but a general list of the details likely to be required is as follows:

11.2.1 Chemical composition of the material tested.

11.2.2 Dimensions and shape of the test specimen and the nature and area of the surface tested.

11.2.3 Preparation of the test specimen including any cleaning treatment applied and any protection given to edges or other special areas. Known characteristics of any coating with an indication of the surface finish,

11.2.4 Amount of sulfur dioxide introduced per cycle,

11.2.5 Number of test specimens subjected to test representing each material or product,

11.2.6 Method used to clean test specimens after test with, when appropriate, an indication of the loss of mass resulting from the cleaning operation,

11.2.7 Temperature of test,

11.2.8 Duration of test (number of cycles),

and

11.2.9 Character of any control test panels placed in the cabinet expressly to check the correctness of the operating conditions and the results obtained with them.

12. Precision and Bias

12.1 The precision and bias of results obtained with this method depend on the type of material or specimen used and cannot be generalized.

APPENDIX

(Nonmandatory Information)

X1. MOIST-SO₂ TEST APPARATUS

X1.1 Location

X1.1.1 The chamber shall be located in an area free of unusual pollution, direct sunlight, and drafts. Temperature should be controlled between 20 to 30°C (68 to 86°F) at a relative humidity below 75 %.

X1.2 Chamber

X1.2.1 Standard SO₂ chambers are available from several suppliers, but certain pertinent details are required before they will function according to this practice and provide consistent control for duplication of results.

X1.2.2 The chamber consists of the basic chamber, nonconducting specimen supports, provisions for heating the chamber, and suitable controls for maintaining the desired temperature.

X1.2.3 The chamber shall have an inlet tube through which gas can be introduced into the chamber located at a point no less than 50 mm above the internal floor, a means by which excess pressure can be relieved located in or near the ceiling and a suitable drain in the floor of the chamber.

X1.2.4 The floor and lower parts of the walls of chamber shall be capable of being heated from an external source and must be able to retain without leakage at least 2.5 L of water containing dissolved sulfur dioxide.

X1.2.5 External means of heating the water placed in the base of chamber, sufficient to maintain the temperature inside the chamber at 40 ± 3°C (104 ± 5.4°F) shall be provided.

X1.2.6 A full-opening door made of transparent material such as glass, acrylic, polycarbonate or suitable

plastic material shall be provided and should occupy at least 75 % of the area of the front wall of the chamber.

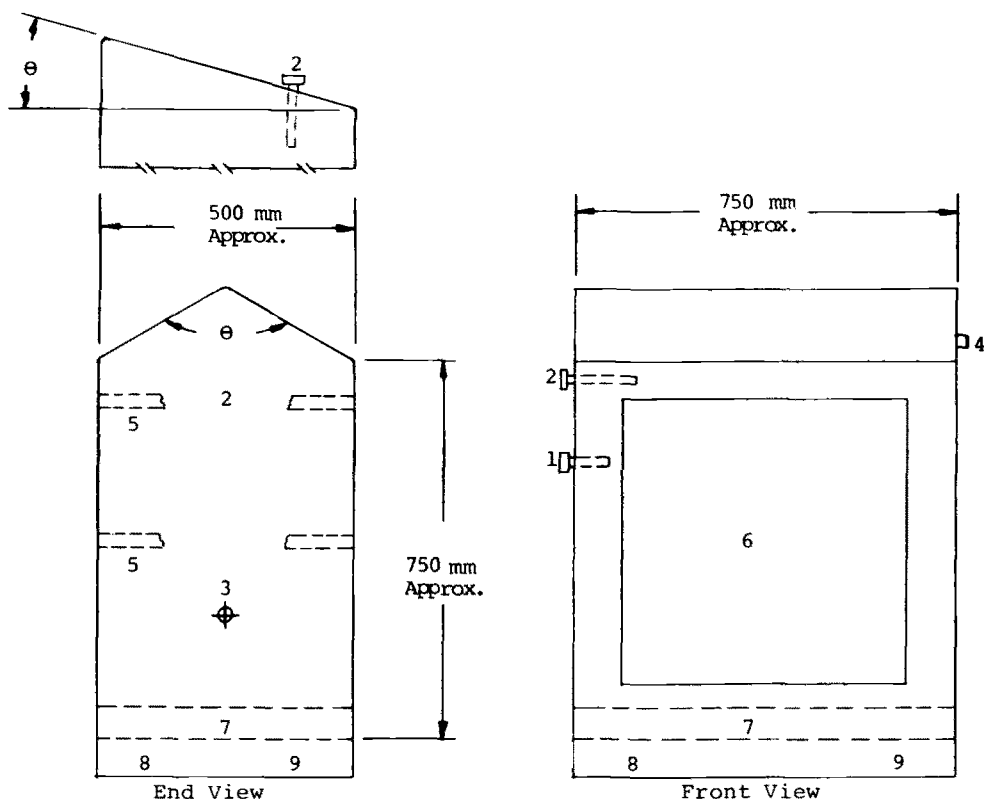
X1.2.7 All materials used in the construction of the chamber must be resistant to the action of moist sulfur dioxide and shall themselves not emit any gases or vapors likely to influence corrosion of test materials. A suitable plastic material, lead or plastic clad steel, or austenitic stainless steel may be used for the basic construction of the chamber and internal fittings.

X1.2.8 Specimen supports shall be of nonconducting material such as glass, plastic, or suitably coated wood or steel. Any material used to suspend the test specimens in the chamber shall be of synthetic fibre or other inert insulating material; metallic materials shall not be used.

X1.2.9 Some variation in the dimensions and shape of the chamber can be tolerated but the total internal area of chamber (including ceiling) should be 300 L (10.6 ft³). The ceiling must be so shaped that moisture condensing on it shall not fall on specimens being tested. An inclination or slope of the ceiling of at least 12° to the horizontal or installation of an internal baffle suspended from the ceiling at that angle provides a suitable safeguard. Suitable dimensions and shape for the chamber are outlined in Fig. X1.

X1.3 Temperature Controls

X1.3.1 A temperature controlling device with its actuating element shall be placed in the upper part of the chamber. A thermometer capable of being read from outside the cabinet shall also be installed in the upper part of the chamber with its bulb located approximately 250 mm from any side wall.



θ Angle of Ceiling

Inclined Type: 120° Minimum (from horizontal)

Gable Type: 120° Minimum (included angle)

- 1 Automatic thermostat for controlling heater
- 2 Thermometer
- 3 Gas inlet
- 4 Excessive pressure vent
- 5 Specimen support
- 6 Door
- 7 Internal reservoir
- 8 Heater
- 9 Air space or water jacket

FIG. X1 Typical Moist SO₂ Test Chamber

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