



Designation: A262 – 13

Standard Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels¹

This standard is issued under the fixed designation A262; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

This standard has been approved for use by agencies of the U.S. Department of Defense.

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 E*—Copper–Copper Sulfate–Sulfuric Acid Test for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels (Sections 22 to 31, inclusive), and

1.1.5 *Practice F*—Copper–Copper Sulfate–50 % Sulfuric Acid Test for Detecting Susceptibility to Intergranular Attack in Molybdenum-Bearing Austenitic Stainless Steels (Sections 32 to 38, inclusive).

1.2 The oxalic acid etch test is a rapid method of identifying, by simple etching, those specimens of certain stainless steel grades that 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.” The etch test is applicable only to those grades listed in the individual hot acid tests and classifies the specimens either as “acceptable” or as “suspect.”

1.3 The ferric sulfate–sulfuric acid test, the copper–copper sulfate–50 % sulfuric acid test, and the nitric 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–16 % sulfuric acid test is

based on visual examination of bend specimens and, therefore, classifies the specimens only as acceptable or nonacceptable.

NOTE 1—See [Appendix X1](#) for information regarding test selection.

1.4 The values stated in SI units are to be regarded as standard. The inch-pound equivalents are in parentheses and may be approximate.

1.5 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* (Specific precautionary statements are given in [5.6](#), [11.1.1](#), [11.1.9](#), and [35.1](#).)

2. Referenced Documents

2.1 *ASTM Standards*:²

[A370 Test Methods and Definitions for Mechanical Testing of Steel Products](#)

2.2 *ISO Standard*:³

[ISO 3651-2 Determination of Resistance to Intergranular Corrosion of Stainless Steels—Part 2: Ferritic, Austenitic, and Ferritic-Austenitic \(Duplex\) Stainless Steels—Corrosion Test in Media Containing Sulfuric Acid](#)

PRACTICE A—OXALIC ACID ETCH TEST FOR CLASSIFICATION OF ETCH STRUCTURES OF AUSTENITIC STAINLESS STEELS (1)⁴

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 that are certain to be free of susceptibility to rapid intergranular attack in these other

¹ These practices are under the jurisdiction of ASTM Committee A01 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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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Available from International Organization for Standardization (ISO), 1 rue de Varembe, Case postale 56, CH-1211, Geneva 20, Switzerland, <http://www.iso.ch>.

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

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

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 E—Copper-Copper Sulfate-16 % Sulfuric Acid Test, and Practice F—Copper-Copper Sulfate-50 % 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 suspect performance in that particular test. Specimens having acceptable etch structures need not be subjected to the hot acid test. Specimens having suspect etch structures must be tested in the specified hot acid solution.

3.3 Extra-low-carbon grades, and stabilized grades, such as 304L, 316L, 317L, 321, and 347, are tested after sensitizing heat treatments at 650 to 675°C (1200 to 1250°F), 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 675°C (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 2).

4.3 *Variable Resistance* (Note 2).

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.

4.8 *Electrolyte*—Oxalic acid, (H₂C₂O₄·2H₂O), reagent grade, 10 weight % solution.

NOTE 2—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 25 by 25 mm (1 by 1 in.).

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, 1/2, 1/0, 2/0, and 3/0, 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 cm² 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 (H₂C₂O₄·2H₂O) 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/cm² 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 **Warning**—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 % HNO₃.

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 250x to 500x for wrought steels and at about 250x 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 3):

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.



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

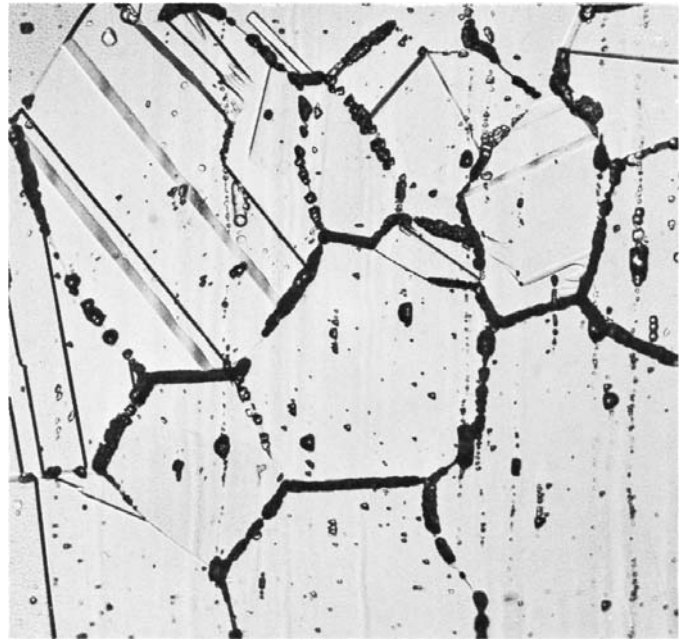


FIG. 2 Dual Structure (250x) (Some ditches at grain boundaries in addition to steps, but no one grain completely surrounded)

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 500x. (Of importance only when nitric acid test is used.)

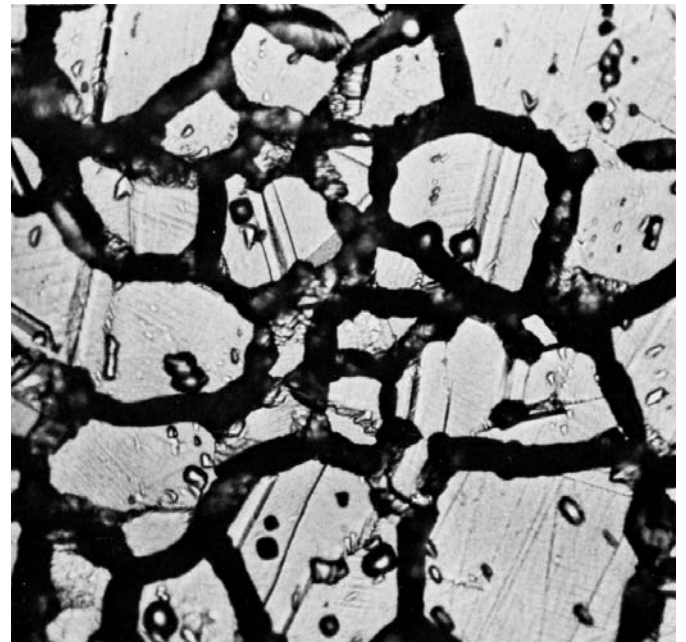


FIG. 3 Ditch Structure (500x) (One or more grains completely surrounded by ditches)

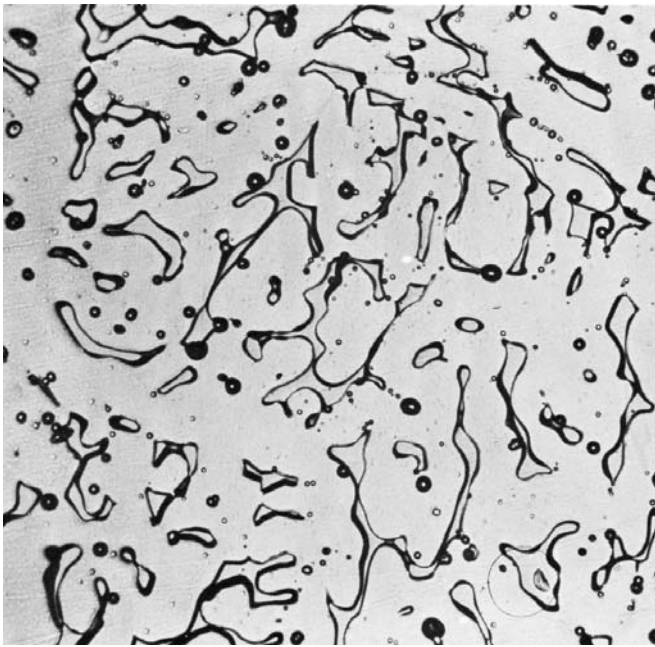
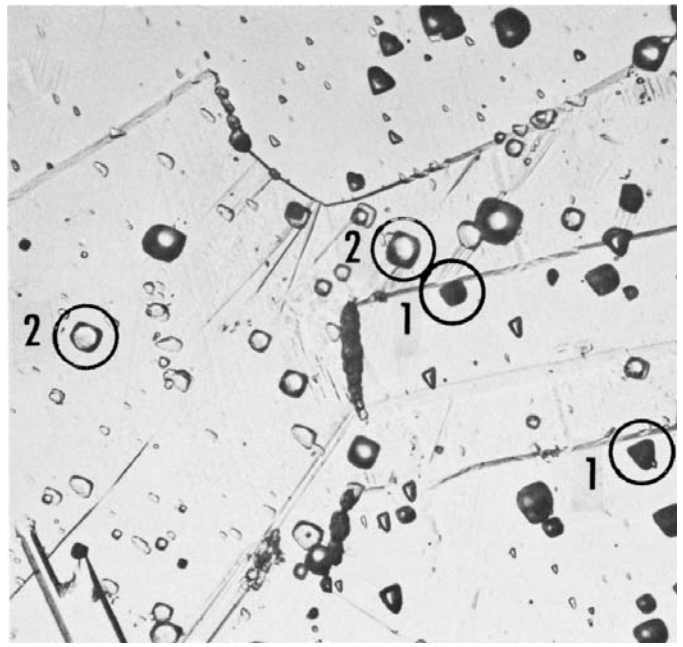


FIG. 4 Isolated Ferrite Pools (250x) (Observed in castings and welds. Steps between austenite matrix and ferrite pools)



To differentiate between the types of pits, use a magnification of 500x 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 (500x) (A few deep end grain pits (see 1 in figure) and shallow etch pits (2))

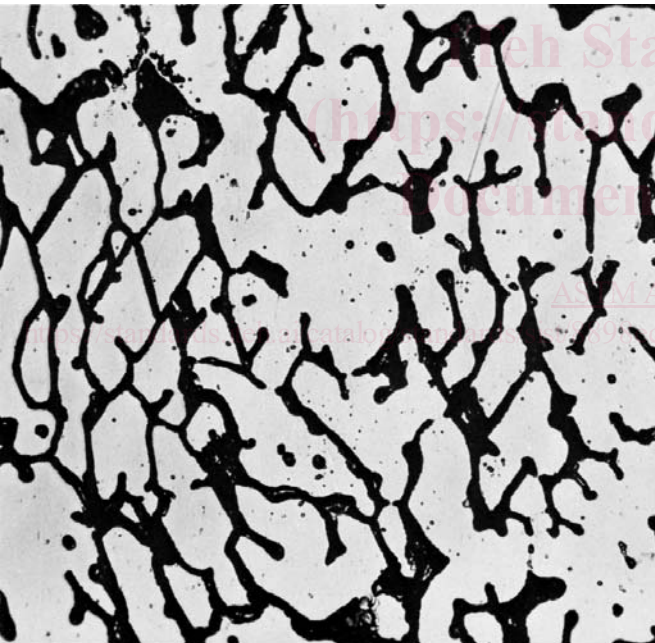
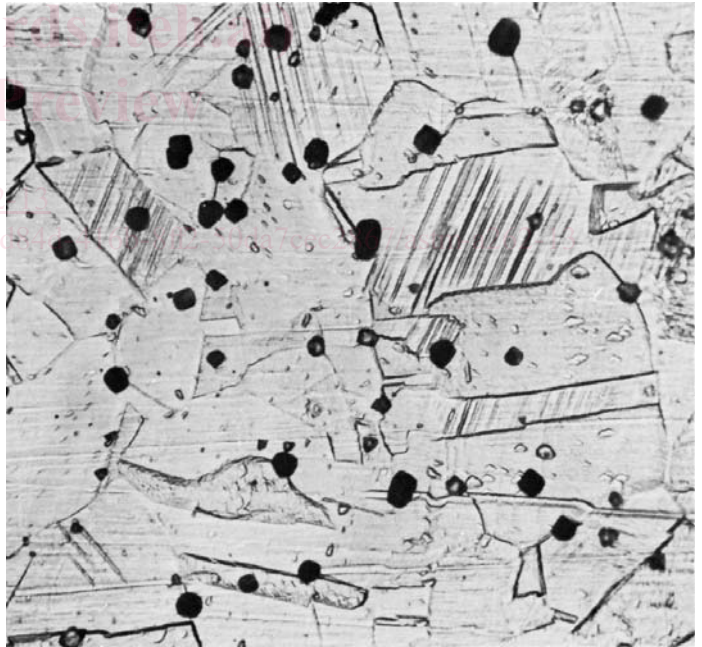


FIG. 5 Interdendritic Ditches (250x) (Observed in castings and welds. Deep interconnected ditches)



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

FIG. 7 End Grain Pitting II (500x)

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

NOTE 3—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 Fig. 1 and Fig. 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.

PRACTICE B—FERRIC SULFATE-SULFURIC ACID TEST FOR DETECTING SUSCEPTIBILITY TO INTERGRANULAR ATTACK IN AUSTENITIC STAINLESS STEELS (3)

8. Scope

8.1 This practice describes the procedure for conducting the boiling 120-h ferric sulfate-50 % sulfuric acid test 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.

8.2 Specimens of extra low carbon and stabilized grades are tested after sensitizing heat treatments at 650 to 675°C (1200 to 1250°F), 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 675°C (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 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. 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 1.

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

TABLE 1 Use of Etch Structure Classifications from the Oxalic Acid Etch Test with Ferric Sulfate-Sulfuric Acid Test

Grade	Acceptable Etch Structures	Suspect Etch Structures ^A
AISI 304	Step, dual, end grain, I & II	Ditch
AISI 304L	Step, dual, end grain, I & II	Ditch
AISI316	Step, dual, end grain, I & II	Ditch
AISI316L	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
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.

specimens are acceptable without testing in the ferric sulfate-sulfuric acid test. All specimens having suspect etch structures must be tested in the ferric sulfate-sulfuric acid test.

10. Apparatus

10.1 The apparatus (Note 5) is illustrated in Fig. 8.

10.1.1 An Allihn or Soxhlet condenser with a minimum of four bulbs and with a 45/50 ground glass joint. Overall length: about 330 mm (13 in.), 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 38 mm (1½ in.) wide.

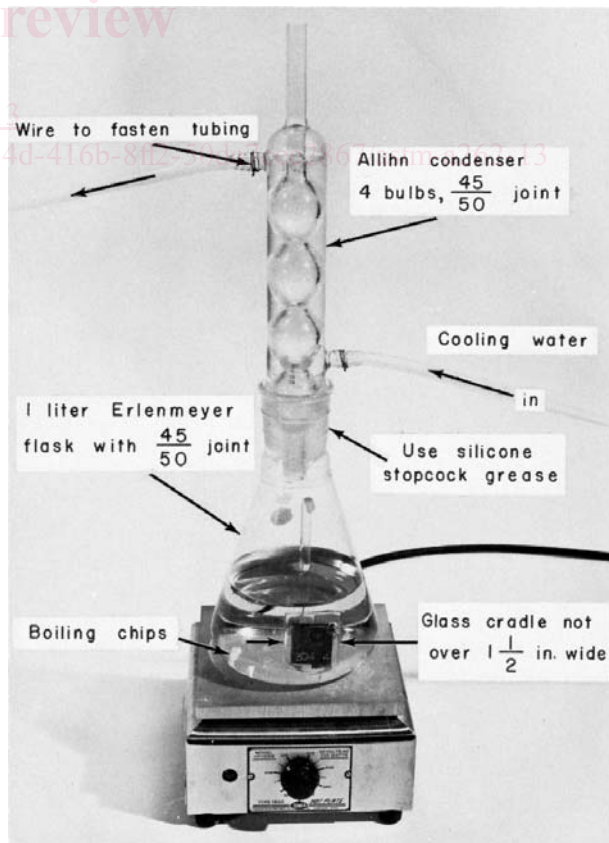


FIG. 8 Apparatus for Ferric Sulfate-Sulfuric Acid Test

10.1.3 The glass cradle (Note 4) 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 38 mm (1½ in.), and the front-to-back distance must be such that the cradle will fit the 34-mm (1⅓-in.) diameter opening. It should have three or four holes to increase circulation of the testing solution around the specimen.

NOTE 4—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 grease⁵ 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 that provides heat for continuous boiling of the solution.

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

NOTE 5—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 **Warning:** 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 that 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 6—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 6).

11.1.9 **Warning:** 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.⁶

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 13-mm (½-in.) 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 surface completely. Such treatment of test specimens is not permissible, except in tests undertaken to demonstrate such surface effects.

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

12.4 Any surfaces of the specimen, including sheared edges, that are to be refinished should be done by 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 that cannot be removed by grinding (for example, in stamped numbers) must be removed by immersing the specimen in concentrated nitric acid at about 93°C (200°F). (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.

⁶ The sole source of supply of the apparatus known to the committee at this time is Amphoteric alundum granules, Hengar Granules, from the Hengar Co., Philadelphia, PA. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

⁵ Dow Corning Stopcock Grease has been found satisfactory for this purpose.