Designation: ~~G28 – 02 (Reapproved 2015)~~ G28 – 22

Standard Test Methods for Detecting Susceptibility to Intergranular Corrosion in Wrought, Nickel-Rich, Chromium-Bearing Alloys[1]

This standard is issued under the fixed designation G28; 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 standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 These test methods cover two tests as follows:

1.1.1 *Method A, Ferric Sulfate-Sulfuric Acid Test* (Sections 3 – 10, inclusive)—This test method describes the procedure for conducting the boiling ferric sulfate—50 % sulfuric acid test which measures the susceptibility of certain nickel-rich, chromium-bearing alloys to intergranular corrosion (see Terminology ~~G15~~G193), which may be encountered in certain service environments. The uniform corrosion rate obtained by this test method, which is a function of minor variations in alloy composition, may easily mask the intergranular corrosion components of the overall corrosion rate on alloys N10276, N06022, N06059, and N06455.

1.1.2 *Method B, Mixed Acid-Oxidizing Salt Test* (Sections 11 – 18, inclusive)—This test method describes the procedure for conducting a boiling 23 % sulfuric + 1.2 % hydrochloric + 1 % ferric chloride + 1 % cupric chloride test which measures the susceptibility of certain nickel-rich, chromium-bearing alloys to display a step function increase in corrosion rate when there are high levels of grain boundary precipitation.

1.2 The purpose of these two test methods is to detect susceptibility to intergranular corrosion as influenced by variations in processing or composition, or both. Materials shown to be susceptible may or may not be intergranularly *corroded* in other environments. This must be established independently by specific tests or by service experience.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3.1 *Exception*—Some desired corrosion rate units in 8.1.1 are given in inch-pound units.

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

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1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate ~~safety~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.* Warning statements are given in 5.1.1, 5.1.3, 5.1.9, 13.1.1, and 13.1.11.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

A262 Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels
D1193 Specification for Reagent Water
~~G15~~G193 Terminology and Acronyms Relating to Corrosion ~~and Corrosion Testing~~ ~~(Withdrawn 2010)~~

METHOD A—Ferric Sulfate—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
~~N06007~~	~~120~~
N06007	120
N06022	24
N06030	120
N06059	24
N06200	24
N06455	24
N06600	24
N06625	120
N06686	24
N06985	120
N08020	120
N08367	24
Alloy	Testing Time, h
N08800	120
N08825^A	120
N10276	24

^{A} While the ferric sulfate-sulfuric acid test does detect susceptibility to inter- granular corrosion in Alloy N08825, the boiling 65 % nitric acid test, Practices A262, Practice C, for detecting susceptibility to intergranular corrosion in stainless steels is more sensitive and should be used if the intended service is nitric acid.

3.2 This test 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 test method may be applied to wrought and weldments of products. The test method is not applicable to cast products.

4. Apparatus

4.1 The apparatus (Note 1) is illustrated in Fig. 1.

4.1.1 *Allihn or Soxhlet Condenser,* 4-bulb,[3] with a 45/50 ground-glass joint, overall length about 330 mm, condensing section about 240 mm.

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

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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~~standard's Document Summary page on the ASTM website.

[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, has been found suitable for this use.

FIG. 1 Apparatus for Ferric Sulfate-Sulfuric Acid Test

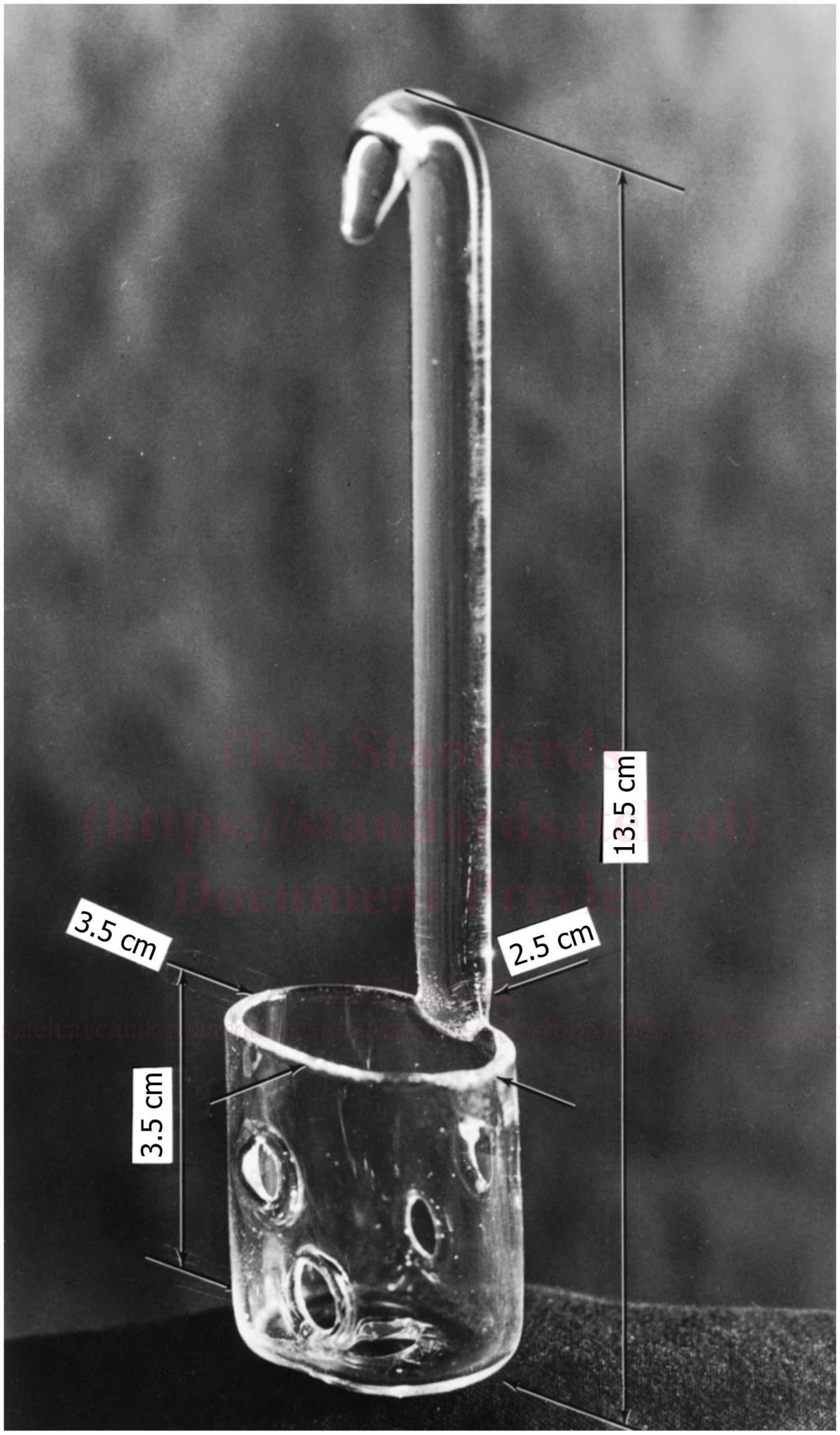

FIG. 2 Glass Cradle

4.1.3 *Glass Cradle* (Fig. 2)—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~~40 mm diameter opening. It should have three or four holes to increase circulation of the test solution around the specimen (Note 2).

NOTE 1—Substitution for this equipment may not be used. The cold-finger type of standard Erlenmeyer flask may not be used.

NOTE 2—Other equivalent means of specimen support, such as glass hooks or stirrups, may also be used.

4.1.4 *Boiling Chips,*[4] ~~or~~ or some other boiling aids must be used to prevent bumping.

4.1.5 *Silicone Grease,* (for example, stopcock grease) is recommended for the ground-glass joint.

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

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

5. Test Solution

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

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

5.1.2 First, measure 400 mL of Type IV reagent water (Specification D1193) in a ~~500-mL~~500 mL graduate and pour into the flask.

5.1.3 Then measure 236 mL of reagent-grade sulfuric acid (H_2SO_4) of a concentration which must be in the range from ~~95.0~~ 95.0 weight percent to 98.0 weight percent in a ~~250-mL~~250 mL graduate. Add the acid slowly to the water in the flask to avoid boiling by the heat evolved (Note 3). Externally cooling the flask with water during the mixing will also reduce overheating.

NOTE 3—Loss of vapor results in concentration of the acid.

5.1.4 Weigh 25 g of reagent grade ferric sulfate (contains about 75 % $Fe_2(SO_4)_3$ (Note 4)) and add to the H_2SO_4 solution. A trip balance may be used.

NOTE 4—Ferritic sulfate is a specific additive that establishes and controls the corrosion potential. Substitutions are not permitted.

5.1.5 Add boiling chips.

5.1.6 Lubricate the ground glass of the condenser 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 ferric sulfate is dissolved.

5.1.9 **Warning—**It has been reported that violent boiling can occur resulting in acid spills. It is important to ensure that the concentration of acid does not increase and that an adequate number of boiling chips (which are resistant to attack by the test solution) are present.[4]

6. Test Specimens

6.1 A specimen having a total surface area of ~~5~~5 cm^2 to ~~20 cm~~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

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[4] The sole source of supply of the apparatus known to the committee at this time is amphoteric alundum Hengar Boiling Granules, available from Hengar Company, a division of Henry Troemner, LLC, 201 Wolf Drive, Thorofare, NJ 08086. 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.

cut to represent the grain flow direction that will see service, for example, specimens should not contain 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 maintained 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 must be removed by machining or grinding to a depth equal to the thickness of the specimen to remove cold worked metal.

6.4 All surfaces of the specimen, including edges, should be finished using wet No. ~~80-grit~~80 grit or dry No. ~~120-grit~~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 has been observed to cause spurious specimen 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 prior to stamping identification codes.

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 using suitable nonchlorinated agents such as soap and acetone, dried, and then weighed to the nearest ~~0.001 g.~~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~~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 3, 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 Intermediate weighing is not 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 Replacement of acid is not 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 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 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 mat.

$$\text{Corrosion Rate} = (K \times W)/(A \times T \times D) \quad (1)$$

~~*where:*~~
where:

K = a constant (see 8.1.1),
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 8.1.2).

8.1.1 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[A]
~~mils per year (mpy)~~	~~3.45 × 10^6~~
mils per year (mpy)	3.45 × 10^6
~~inches per year (ipy)~~	~~3.45 × 10^3~~
inches per year (ipy)	3.45 × 10^3
~~inches per month (ipm)~~	~~2.87 × 10^2~~
inches per month (ipm)	2.87 × 10^2
~~millimetres per year (mm/Y)~~	~~8.76 × 10^4~~
millimetres per year (mm/Y)	8.76 × 10^4
~~micrometres per year (µm/y)~~	~~8.76 × 10^7~~
micrometres per year (µm/y)	8.76 × 10^7
~~picometres per second (pm/s)~~	~~2.78 × 10^6~~
picometres per second (pm/s)	2.78 × 10^6
~~grams per square metre-hour (g/m^2-h)~~	~~1.00 × 10^4 × D[B]~~
grams per square metre-hour (g/m^2 h)	1.00 × 10^4 × D[B]
~~milligrams per square decimetre-day (mdd)~~	~~2.40 × 10^6 × D[B]~~
milligrams per square decimetre-day (mdd)	2.40 × 10^6 × D[B]
~~micrograms per square metre-second (µg/m^2-s)~~	~~2.78 × 10^6 × D[B]~~
micrograms per square metre-second (µg/m^2-s)	2.78 × 10^6 × 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.

8.1.2

UNS Designation	Density, g/cm^3
N06007	8.31
N06022	8.69
N06030	8.22
~~N06059~~	~~8.80~~
N06059	8.60
N06200	8.50
N06455	8.64
N06600	8.41
N06625	8.44
N06686	8.73
N06985	8.31
N08020	8.05
N08367	8.06
N08800	8.03
N08825	8.14
N10276	8.87