# Standard Practice for
# Exposure of Metals and Alloys by Alternate Immersion in Neutral 3.5 % Sodium Chloride Solution[1]

This standard is issued under the fixed designation G44; 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 Department of Defense.*

## 1. Scope

1.1 This practice covers procedures for making alternate immersion stress corrosion tests in 3.5 % sodium chloride (NaCl) (Note 1). It is primarily for tests of aluminum alloys (Test Method G47) and ferrous alloys, but may be used for other metals exhibiting susceptibility to chloride ions. It sets forth the environmental conditions of the test and the means for controlling them.

NOTE 1—Alternate immersion stress corrosion exposures are sometimes made in substitute ocean water (without heavy metals) prepared in accordance with Specification D1141. The general requirements of this present practice are also applicable to such exposures except that the reagents used, the solution concentration, and the solution pH should be as specified in Specification D1141.

1.2 This practice can be used for both stressed and unstressed corrosion specimens. Historically, it has been used for stress-corrosion cracking testing, but is often used for other forms of corrosion, such as uniform, pitting, intergranular, and galvanic.

1.3 This practice is intended for alloy development and for applications where the alternate immersion test is to serve as a control test on the quality of successive lots of the same material. Therefore, strict test conditions are stipulated for maximum assurance that variations in results are attributable to variations in the material being tested.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1141 Practice for the Preparation of Substitute Ocean Water
D1193 Specification for Reagent Water
E3 Guide for Preparation of Metallographic Specimens
G1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens
G16 Guide for Applying Statistics to Analysis of Corrosion Data
G47 Test Method for Determining Susceptibility to Stress-Corrosion Cracking of 2XXX and 7XXX Aluminum Alloy Products

## 3. Summary of Practice

3.1 The alternate immersion test utilizes a 1-h cycle that includes a 10-min period in an aqueous solution of 3.5 % sodium chloride (NaCl) followed by a 50-min period out of the solution, during which the specimens are allowed to dry. This 1-h cycle is continued 24 h/day for the total number of days recommended for the particular alloy being tested. Typically, aluminum and ferrous alloys are exposed from 20 to 90 days or longer, depending upon the resistance of the alloy to corrosion by saltwater.

## 4. Significance and Use

4.1 The 3.5 % NaCl alternate immersion procedure is a general, all-purpose procedure that produces valid comparisons for most metals, particularly when specimens are exposed at high levels of applied stress or stress intensity.

4.2 While the alternate immersion test is an accelerated test and is considered to be representative of certain natural conditions, it is not intended to predict performance in specialized chemical environments in which a different mode of cracking may be operative. For example, it does not predict the performance of aluminum alloys in highly acidic environments

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[1] This practice is under the jurisdiction of ASTM Committee G01 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.06 on Environmentally Assisted Cracking.

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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.

such as heated inhibited red fuming nitric acid (IRFNA). For such cases, the results of the alternate immersion test are of doubtful significance until a relationship has been established between it and anticipated service environments.

4.3 While this practice is applicable in some degree to all metals, it is not equally discriminative of all alloys, even within the same metal system. Consequently, information should be established to allow comparisons of performances of the alloy of interest in the alternate immersion test and in natural environments.

NOTE 2—The alternate immersion concept can be useful for exposure of corrosion specimens in other solutions because the procedure and apparatus provide a controlled set of conditions. Details of this are beyond the scope of this practice.

## 5. Interferences

5.1 A disadvantage of the 3.5 % NaCl alternate immersion test for stress-corrosion cracking tests of certain high-strength aluminum alloys is the severe pitting that develops in the specimens. Such pitting can interfere with the initiation of stress-corrosion cracks and may cause mechanical failures that complicate the interpretation of the stress-corrosion test results. This is particularly a problem with copper-bearing aluminum alloys when tested with specimens of small cross section. Thorough metallographic examination of the specimens is necessary for proper diagnosis of the cause of failure and separation of stress corrosion failures from those caused by mechanical overload.

5.2 An advantage of the substitute ocean water (Note 1) is that it causes less pitting corrosion of aluminum alloys than the 3.5 % NaCl solution.

## 6. Apparatus

6.1 *Method of Cycling*—Any suitable mechanism may be used to accomplish the immersion portion of the cycle provided that: *(1)* it achieves the specified rate of immersion and removal, and *(2)* the apparatus is constructed of suitable inert materials. The usual methods of immersion are:

6.1.1 Specimens placed on a movable rack that is periodically lowered into a stationary tank containing the solution.

6.1.2 Specimens placed on a hexagonal Ferris wheel arrangement which rotates every 10 min through 60° and, thereby, passes the specimens through a stationary tank of solution. Use of a Ferris wheel continuously rotating at a rate of 1 revolution per hour is not recommended for very large specimens for which the rate of immersion would be slower than that specified in 6.2.

6.1.3 Specimens placed in a stationary tray open to the atmosphere and having the solution moved by air pressure, nonmetallic pump, or gravity drain from a reservoir to the tray.

6.2 *Rate of Immersion*—The rate of immersion and removal of the specimens from the solution should be as rapid as possible without jarring them. For purposes of standardization, an arbitrary limit shall be adopted such that no more than 2 min elapse from the time the first portion of any specimen is covered (or uncovered) until it is fully covered (or uncovered) by solution.

6.3 *Materials of Construction:*

6.3.1 Materials of construction that come in contact with the salt solution shall be such that they are not affected by the corrodent to an extent that they can cause contamination of the solution and change its corrosiveness.

6.3.2 Use of inert plastics or glass is recommended where feasible.

6.3.3 Metallic materials of construction should be selected from alloys that are recommended for marine use and of the same general type as the metals being tested. Preferably, all metal parts should be protected with a suitable corrosion-resistant coating that also satisfies paragraph 6.3.1.

6.4 *Specimen Holders:*

6.4.1 Specimen holders should be designed to electrically insulate the specimens from each other and from any other bare metal. When this is not possible, as in the case of certain stressing bolts or jigs, the bare metal contacting the specimen should be isolated from the corrodent by a suitable coating. Protective coatings should be of a type that will not leach inhibiting or accelerating ions or protective oils over the noncoated portions of the specimen. Coatings containing chromates are to be particularly avoided.

NOTE 3—Coatings that have been satisfactorily used by several laboratories are described in Appendix X1.

6.4.2 The shape and form of specimen supports and holders should be such that:

6.4.2.1 They avoid, as much as possible, any interference of free contact of the specimen with the salt solution;

6.4.2.2 They do not obstruct air flow over the specimen, thereby retarding the drying rate;

6.4.2.3 They do not retain a pool of solution in contact with the specimen after the immersion period; and

6.4.2.4 Drainage from one specimen does not directly contact any other specimen.

## 7. Reagents

7.1 Reagent grade sodium chloride (NaCl) shall be used conforming to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are applicable (see Note 1).[3]

7.2 The solution shall be prepared using distilled or deionized water conforming to the purity requirements of Specification D1193, Type IV reagent water except that for this practice the values for chloride and sodium shall be disregarded.

## 8. Solution Conditions

8.1 *Concentration*—The salt solution shall be prepared by dissolving 3.5 ± 0.1 parts by weight of NaCl in 96.5 parts of water.

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[3] *Reagent Chemicals, American Chemical Society Specifications,* American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

8.2 *Solution pH:*

8.2.1 The pH of the salt solution, when freshly prepared, shall be within the range from 6.4 to 7.2. Only diluted, reagent grade hydrochloric acid (HCl) or reagent grade sodium hydroxide (NaOH) shall be used to adjust the pH.

8.2.2 Experience has shown that periodic adjustment of pH is not necessary when the minimum volume of solution is met and the solution is replaced at the specified interval. For a new testing facility, however, it is recommended that daily pH measurements be made for one week to verify stable operation and adjustments made if required. If the solution is used longer than the recommended interval (8.6), a pH measurement and any necessary adjustment should be made at least weekly.

8.3 *Temperature*—A freshly prepared solution should be allowed to come to within 3°C of the specified room temperature before being used (9.1.1). Thereafter, no control is required on the solution temperature per se. Instead, the room air temperature is controlled and the solution is allowed to reach temperature equilibrium.

8.4 *Minimum Volume*—The volume of the test solution should be large enough to avoid any appreciable change in its corrosiveness either through exhaustion of corrosive constituents, or the accumulation of corrosion products or other constituents that might significantly affect further corrosion. An arbitrary minimum ratio between the volume of test solution and area of specimen (including any uncoated accessories) of 32 mL/cm$^2$ (200 mL/in.$^2$) of specimen area is recommended.

8.5 *Replenishment of Water Lost by Evaporation*—Evaporation losses should be made up by frequent, at least daily, additions of water of the required purity (7.2). Evaporation losses must not be replenished with the salt solution. The simplest and recommended procedure is to initially fill the solution to a liquid level line and refill to that line daily. Automatic constant liquid level devices may be used, but are not required. An alternative method is to check the solution with a hydrometer and add the necessary amount of water to bring the salt concentration to 3.5 %.

8.6 *Replacement of Solution*—Fresh solution shall be prepared weekly. At such time, the portions of the apparatus that contact the solution should be cleansed by flushing with water. More frequent replacement of solution may be required for certain steels if severe rusting occurs.

## 9. Air Conditions

NOTE 4—Other than temperature and humidity, the gaseous and particulate makeup of the laboratory atmosphere is beyond the scope of this recommended practice. However, testers are cautioned not to expose specimens in the vicinity of obvious fumes or airborne contamination, and to consider the possibility of such contamination if unusual results are obtained.

9.1 *Temperature:*

9.1.1 The air temperature shall be maintained at 27 ± 1°C (80 ± 2°F) throughout the entire test cycle.

NOTE 5—When a large Ferris wheel apparatus is used such that the specimens travel through several feet of height, care must be taken to ensure that the air temperatures at all height levels are within the prescribed limits.

9.2 *Relative Humidity*—The percent relative humidity of the air shall be controlled at 45 ± 10 % throughout the entire test cycle.

NOTE 6—Care should be taken to avoid overnight and weekend changes in the operation of the laboratory heating, ventilating, and air conditioning equipment which could result in systematic excursions outside the temperature and humidity control ranges. Occasional excursions related to power outage, mechanical failure, or weather conditions shall be recorded.

9.3 *Air Circulation:*

9.3.1 Air circulation is recognized to be an important consideration because it affects both the rate at which specimens dry and the loss of water by evaporation. Optimum conditions for air circulation have not been established, but the recommendations described in 9.3.2 should be followed.

9.3.2 The most important consideration is to achieve the moderate specimen drying conditions stipulated in 11.2. Because various testing facilities use different immersion apparatus and room sizes, individual experimentation is required to achieve adequate circulation. A mild circulation of air is recommended with two precautionary considerations:

9.3.2.1 Drying by forced air blasts on the specimens is not recommended because of difficulty in maintaining uniform drying of large groups of specimens.

9.3.2.2 Stagnant air conditions should be avoided.

## 10. Calibration and Standardization

10.1 When a new test facility is established, calibration stress corrosion tests should be conducted to determine how the results obtained compare with published data on well established alloys. Such tests are best made on products of simple geometry with uniform grain structure, such as rolled bar stock or plate.

10.2 It is recommended that each testing facility maintain a supply of a control lot of stress corrosion-susceptible material that can be exposed periodically to demonstrate the reproducibility of its test procedure. Preferably this should be material for which a history of performance in natural environments is available.

## 11. Procedure

11.1 *Period of Cycle*—Totally immerse specimens in the salt solution for 10 min of each hour and then remove from the solution and allow to dry for 50 min. Continue cycle 24 h/day for the specified period or until failure occurs, with interruptions only for changing solution or examining specimens.

NOTE 7—To some extent this is an arbitrary cycle, but it has considerable historical usage and correlation with exposures to natural atmospheres.[4]

11.2 *Specimen Drying*—As with air circulation (9.3), no fixed procedure has been established, and probably cannot be unless a standardized immersion apparatus and test chamber are adopted. The objective, however, is to ensure that all specimens dry slowly during the 50-min period. Because they drain differently, specimens with different accrued corrosion

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[4] Romans, H. B., “Stress Corrosion Test Environments and Test Durations,” *Symposium on Stress Corrosion Testing, ASTM STP 425,* Am. Soc. Testing Mats., 1967, pp. 182–208.

films will dry at different rates. New specimens with little accumulated corrosion products become dry in about 15 min, while other specimens with an accumulation of corrosion product and salt should be allowed about 40 min to dry.

NOTE 8—Use of heated air to promote drying is not permitted because elevated temperatures can have effects other than acceleration of the drying process.

11.3 *Concurrent Exposure of Various Alloys*—Do not expose specimens of different base metals, aluminum, copper, iron, magnesium, and so forth, concurrently in the same salt solution. For maximum assurance of reproducibility, do not expose specimens of low alloy content to the same solution used for specimens of high alloy content if the alloying element differs appreciably from the base metal in the galvanic series in salt water. For example, specimens of copper-free aluminum alloys should not be exposed with specimens from alloys containing copper greater than about 0.5 %.

11.4 *Test Duration:*

11.4.1 The duration of test shall be determined by the inherent resistance to corrosion of the alloy, the configuration of the test specimen, and the object of the test. Appropriate exposure periods are determined by correlation with service environments with caution to avoid interference effects described in Section 4.

11.4.2 When the purpose of the test is control of the quality of production lots, the test duration shall be as required in pertinent specifications or standards.

11.5 *Cleaning Specimens*—After exposure, specimens should be rinsed with water and then cleaned as soon as possible; otherwise, they will continue to corrode because the accumulated salt and corrosion products are hygroscopic. It is important that the specimens be cleaned as thoroughly as possible by recommended methods of cleaning, such as Practice G1.

NOTE 9—Certain post-test appraisals, such as fractographic examination at high magnification, may require special cleaning methods.

## 12. Records and Reports

12.1 Record temperature, humidity, and pH data.

12.2 Report any excursions from specified conditions, such as those discussed in Note 6.

## 13. Keywords

13.1 accelerated corrosion test; alternate immersion test; aluminum alloys; ferrous alloys; quality control test; sodium chloride solution; stress corrosion cracking

## APPENDIX

### (Nonmandatory Information)

### X1. SATISFACTORY PROTECTIVE COATINGS FOR USE IN ALTERNATE IMMERSION TESTS

X1.1 The following list of coatings is merely a suggested guide and is not intended to be mandatory or exclusive:

X1.1.1 *Maskcoat No. 2.*[5]This is a cellulose acetate butyrate base with plasticizers added. It is obtained in bricks which must be melted, 177 to 188°C (350 to 370°F). The portions to be coated are dipped in the melt and allowed to air cool, usually less than 5 min required. Use of *Maskcoat Primer,* by the same company, is recommended for better adhesion at the edges of the coating. The coating is tough, resilient, and transparent and can be stripped without solvents. It may darken when heated for long periods, so it is advisable not to heat longer than necessary. If darkening has not occurred and the coating is clean, it can be recycled. Care should be taken during application to avoid contact with the skin, as severe burns can result.

X1.1.2 *Gaco N-700A*[6](black). This is a solvent base, liquid neoprene. Thinned with *Gaco Neoprene Thinner N-450-11,* it can be applied by any conventional paint technique and air dries in a few hours or overnight depending on the film thickness applied. It is removed by dissolving in the above thinner or other suitable solvent, such as acetone.

X1.1.3 *Turco Form Mask 544.*[7]Good stripping characteristics.

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[5] The sole source of supply of the apparatus known to the committee at this time is manufactured by Western Coating Co., Box 598, Oakridge Station, Royal Oak, MI 48073. 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.

[6] The sole source of supply of the apparatus known to the committee at this time is produced by Gates Engineering Div. of Glidden Co., Wilmington, DE 19898. 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.

[7] The sole source of supply of the apparatus known to the committee at this time is produced by Turco Products, 24600 South Main St., Wilmington, CA 90745. 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.

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