

# Standard Guide for Laboratory Immersion Corrosion Testing of Metals<sup>1</sup>

This standard is issued under the fixed designation TM0169/G31; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision.

# 1. Scope

1.1 This guide covers and describes the factors that influence laboratory immersion corrosion tests, particularly mass loss tests. These factors include apparatus, sampling, test specimen, test conditions (test solution composition, temperature, gas sparging, fluid motion, solution volume, method of supporting test specimens, duration of test), methods of cleaning test specimens, interpretation of results, and calculation of corrosion rates. This guide also emphasizes the importance of recording all pertinent data and provides a checklist for reporting test data.

1.2 The specific evaluation of localized attack, environmentally assisted cracking, and effects of solution flow are not within the scope of this guide.

1.3 This guide is intended to be used by those designing laboratory immersion tests who may not be familiar with all of the variables to consider and the pitfalls that could be encountered when designing and conducting this kind of testing. It should be used as a reference to ensure that the test will allow generation of data relevant to the application with the minimum of interferences.

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:<sup>2</sup>

A262 Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels

- D1193 Specification for Reagent Water
- E8 Test Methods for Tension Testing of Metallic Materials E300 Practice for Sampling Industrial Chemicals

- G1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens
- G28 Test Methods for Detecting Susceptibility to Intergranular Corrosion in Wrought, Nickel-Rich, Chromium-Bearing Alloys
- G34 Test Method for Exfoliation Corrosion Susceptibility in 2XXX and 7XXX Series Aluminum Alloys (EXCO Test)
- G46 Guide for Examination and Evaluation of Pitting Corrosion
- G48 Test Methods for Pitting and Crevice Corrosion Resistance of Stainless Steels and Related Alloys by Use of Ferric Chloride Solution
- G66 Test Method for Visual Assessment of Exfoliation Corrosion Susceptibility of 5XXX Series Aluminum Alloys (ASSET Test)
- G67 Test Method for Determining the Susceptibility to Intergranular Corrosion of 5XXX Series Aluminum Alloys by Mass Loss After Exposure to Nitric Acid (NAMLT Test)
- G71 Guide for Conducting and Evaluating Galvanic Corrosion Tests in Electrolytes
- **G78** Guide for Crevice Corrosion Testing of Iron-Base and Nickel-Base Stainless Alloys in Seawater and Other Chloride-Containing Aqueous Environments
- **G82** Guide for Development and Use of a Galvanic Series for Predicting Galvanic Corrosion Performance
- G107 Guide for Formats for Collection and Compilation of Corrosion Data for Metals for Computerized Database Input
- G108 Test Method for Electrochemical Reactivation (EPR) for Detecting Sensitization of AISI Type 304 and 304L Stainless Steels
- G110 Practice for Evaluating Intergranular Corrosion Resistance of Heat Treatable Aluminum Alloys by Immersion in Sodium Chloride + Hydrogen Peroxide Solution
- G112 Guide for Conducting Exfoliation Corrosion Tests in Aluminum Alloys
- G116 Practice for Conducting Wire-on-Bolt Test for Atmospheric Galvanic Corrosion
- G135 Guide for Computerized Exchange of Corrosion Data for Metals
- G170 Guide for Evaluating and Qualifying Oilfield and Refinery Corrosion Inhibitors in the Laboratory
- G184 Practice for Evaluating and Qualifying Oil Field and Refinery Corrosion Inhibitors Using Rotating Cage
- G185 Practice for Evaluating and Qualifying Oil Field and Refinery Corrosion Inhibitors Using the Rotating Cylinder Electrode

<sup>&</sup>lt;sup>1</sup> This guide is under the jurisdiction of NACE/ASTM Committee J01, Joint Committee on Corrosion, and is the direct responsibility of Subcommittee J01.01, Working Group on Laboratory Immersion Tests.

Current edition approved July 1, 2012. Published October 2012. Originally approved in 1972. Last previous ASTM edition approved in 2012 as G31–12. NACE edition originally approved in 1969. Last previous NACE edition approved in 2000 as TM0169-2000. DOI: 10.1520/G0031-12A.

<sup>&</sup>lt;sup>2</sup> For referenced ASTM standards, visit the ASTM Web site, 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 Web site. For NACE standards, visit the NACE Web site, www.nace.org, or contact NACE *FirstService* at firstservice@nace.org.

2.2 NACE/ASTM Standards:<sup>2</sup>

G193 Terminology and Acronyms Relating to Corrosion 2.3 *NACE International Standards:*<sup>2</sup>

SP0690 Standard Format for Collection and Compilation of Data for Computerized Material Corrosion Resistance Database Input

2.4 International Organization for Standardization (ISO) Standards:<sup>3</sup>

- ISO 3651-1 Austenitic Stainless Steels Determination of resistance to intergranular corrosion of stainless steels – Part I: Austenitic and ferritic-austenitic (duplex) stainless steels – Corrosion test in nitric acid medium by measurement of loss in mass (Huey test)
- **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
- **ISO 6509** Corrosion of metals and alloys Determination of dezincification resistance of brass
- **ISO 8407** Corrosion of metals and alloys Removal of corrosion products from corrosion test specimens
- ISO 8993 Anodized aluminum and aluminum alloys Rating system for the evaluation of pitting corrosion – Chart method
- ISO 8994 Anodized aluminum and aluminum alloys Rating system for the evaluation of pitting corrosion – Grid method
- **ISO 9400** Nickel-based alloys Determination of resistance to intergranular corrosion
- **ISO 11463** Corrosion of metals and alloys Evaluation of pitting corrosion
- **ISO 11845** Corrosion of metals and alloys General principles for corrosion testing
- **ISO 11846** Corrosion of metals and alloys Determination of resistance to intergranular corrosion of solution heat-treatable aluminum alloys
- **ISO 11881** Corrosion of metals and alloys Exfoliation corrosion testing of aluminum alloys

# 3. Terminology

3.1 For definitions of terms used in this guide, see NACE/ ASTM Terminology G193.

# 4. Significance and Use

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

4.2 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 required. One purpose for this guide is to promote better correlation of results in the future and the reduction of conflicting reports through a more detailed recording of meaningful factors and conditions.

4.3 In designing any corrosion test, consideration should be given to the various factors discussed in this guide, because these factors have been found to affect the results obtained.

#### 5. Factors Affecting Corrosion Behavior

5.1 The methods and procedures described herein represent the best current practices for conducting laboratory immersion corrosion tests as developed by corrosion specialists in the process industries. For proper interpretation of the results obtained, the specific influence of one or more of the following variables should be considered.

5.1.1 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) shall be employed.

5.1.2 In laboratory immersion tests, the motion of the environment relative to the specimens will normally be provided by convection currents, gas sparging, or boiling. If the specific effects of fluid flow are to be studied, special techniques shall be employed to create and control the relative motion between the environment and the test specimens. This may be accomplished by either moving the environment as through a tube or mechanical stirrer or by moving the specimens as by rotation.

5.1.3 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 air saturated at 1 atm or de-aerated, as appropriate.

5.1.4 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.

5.1.5 Corrosion products may have undesirable effects on a chemical product. The amount of possible contamination can sometimes be estimated from the loss in mass of the specimen or from the changes in the chemical composition of the test environment. This is discussed in more detail in 9.8.3.

5.1.6 Corrosion products from the specimen 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. It may be necessary to expose only alloys of the same general type in the same testing apparatus unless it is known that no interactions will occur.

5.1.7 Specimen corrosion testing is frequently designed to investigate general corrosion only. There are a number of other

<sup>&</sup>lt;sup>3</sup> Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.ch.

forms of corrosion of which one shall be aware in the design and interpretation of corrosion tests.

5.1.7.1 Galvanic corrosion may be investigated by special devices that couple one specimen to another in electrical contact. The behavior of the specimens in this galvanic couple is compared with that of insulated specimens exposed on the same holder. It should be observed, however, that galvanic corrosion can be greatly affected by the area ratios of the respective metals, the separation between the metals, and the conductivity of the electrolyte. The coupling of corrosion specimens then yields only qualitative results, as a particular specimen reflects only the relationship between these two metals at the particular area ratio involved. Galvanic corrosion testing is further discussed in ASTM Guide G71, ASTM Guide G82, and ASTM Practice G116.

5.1.7.2 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. Crevice corrosion testing is further discussed in ASTM Test Methods G48 and ASTM Guide G78.

5.1.7.3 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 specimens after exposure. This type of corrosion may also result in loss of strength or ductility of materials. Such losses can be evaluated by mechanical property determinations before and after exposure to the test environment. Testing for selective corrosion is further discussed in ASTM Practices A262, ASTM Test Methods G28, G34, G66, G67, G108, G110, and ASTM Guide G112 and ISO 3651-1, ISO 3651-2, ISO 9400, ISO 11846, and ISO 11881.

5.1.7.4 Dealloying or "parting" corrosion is a condition in which one constituent is selectively removed from an alloy, as in the dezincification of brass or the graphitization of cast iron. Close attention and a more sophisticated evaluation than a simple mass loss measurement are required to detect this phenomenon. Dealloying testing is further discussed in ISO 6509.

5.1.7.5 Certain metals and alloys are subject to a highly localized type of attack called pitting corrosion. This cannot be evaluated by mass loss alone. Pitting is a statistical phenomenon and the incidence of pitting may be directly related to the area of metal exposed. For example, a small specimen 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-containing environments. Pitting testing is further discussed in ASTM Guide G46, ASTM Test Methods G48, and ISO 8993, ISO 8994, and ISO 11463.

5.1.7.6 Most metals and alloys are subject to environmentally assisted 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 environmentally assisted cracking. This usually occurs with no significant loss in mass of the test specimen, although certain refractory metals are an exception to these observations. Generally, if cracking is observed on the specimen, it can be taken as positive indication of susceptibility, whereas failure to exhibit this phenomenon 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 environmentally assisted cracking. Multiple standards from many different organizations are available to describe stresscorrosion cracking tests.

5.2 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.

5.2.1 The heat-affected zone is also of importance but should be studied separately because welds on test specimens may not adequately reproduce heat input or size effects of full-size vessels.

5.2.2 Corrosion of a welded specimen is normally localized and not representative of the entire surface and therefore separate thickness losses should be determined in the weld metal, heat-affected zone, and base metal.

5.2.3 A complete discussion of corrosion testing of welded specimens or the effect of heat treatment on the corrosion resistance of a metal is not within the scope of this guide. However, important factors to be considered include the welding technique to be used, the filler metal chemistry, and whether the weld will be ground smooth, cleaned, passivated, or left as-welded.

5.3 Cast and wrought alloys considered equivalent often have somewhat different chemical composition and metallurgical structure, resulting in different corrosion resistances in identical service conditions. Therefore, caution should be used in selecting representative test materials.

5.4 Additional discussion of testing considerations is contained in ISO 11845.

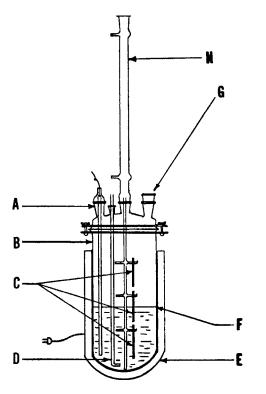
#### 6. Apparatus

6.1 A typical testing apparatus consists of a kettle or flask of suitable size (usually 500 to 5,000 mL), a reflux condenser with or without an atmospheric seal, a sparger for controlling atmosphere or aeration, a thermometer port, a temperature-regulating device, a heating device (mantle, hot plate, or bath), and a test 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 flask setup for this test is shown in Fig. 1.

6.2 These components can be modified to fit the needs of a particular investigation. The chosen apparatus is limited only by the judgment and ingenuity of the investigator.

6.2.1 A glass reaction kettle can be used when configuration and size of test specimens do not permit entry through the narrow neck of a flask. For solutions corrosive to glass, suitable metallic or plastic kettles may be employed.

6.2.2 In some cases, a wide-mouth jar with a suitable closure may be sufficient for simple, ambient-temperature immersion tests.



NOTE 1—A = thermometer port, B = 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

6.2.3 Open-beaker tests should not be used for long-term testing because of evaporation and contamination. If beakers are used, cover plates or watch glasses should be placed over the openings.

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

#### 7. Sampling

7.1 *Statistical Sampling*—Statistical techniques for determining sample size, selecting materials for test, etc., should be used.

7.2 *Corrosion Products*—The bulk sampling of products is outside the scope of this guide.

### 8. Test Specimen

8.1 At least duplicate test specimens should be exposed in each test. In laboratory immersion tests, corrosion rates of duplicate specimens are usually within  $\pm 10\%$  of each other when the attack is uniform. If the rates exceed this variance, retesting should be considered. 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. When large disparities in measured corrosion rates occur, rather than reporting an average corrosion rate, the reason for the disparity should be investigated and reported. If the reason for the disparity cannot be found, retesting should be considered.

8.1.1 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, or at ambient temperature, or at both, 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 ASTM Test Methods E8.

8.2 The size and shape of corrosion test specimens vary with the purpose of the test, nature of the materials, and test apparatus. A rectangular or circular test specimen is preferred for laboratory corrosion testing. Its size and dimensions are typically determined by the test vessel being used and the volume of the test solution available. A ratio of surface area-to-solution mass smaller than in 9.8.2 and a ratio of edge area to total area of less than 20% are desirable. These ratios can be achieved through the use of specimens of minimum thickness, although thin specimens such as shims of some materials produced by heavy machining or cold rolling may have different corrosion rates from material not subjected to these processes. Masking may also be used to achieve the desired area ratios but may cause crevice corrosion problems.

8.2.1 If circular specimens are used, they should be cut from sheet or plate, not bar stock, to minimize the exposed end grain (unless the intent is to test or evaluate bar stock). 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 (5/16 in) or 11 mm (7/16 in) diameter hole for mounting, these specimens will readily pass through a 45/50 ground-glass joint of a distillation kettle. Bar stock may contain long stringers near the center that can lead to corrosion behavior at the center of disk specimens cut from bar, which is not representative of the performance of the bulk alloy. This behavior can cause problems in interpreting performance.

8.2.2 Typically, rectangular test specimens 20 mm by 50 mm (0.75 in by 2.0 in) with a thickness of 1.6 mm to 4.8 mm (0.063 in to 0.19 in), with or without a hole, are preferred. Alternative dimensions may be more suitable for testing of liquid/vapor interface conditions.

8.2.3 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.

8.3 More uniform results can be expected if a uniform 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. Abrasive materials may be picked up in the surface if the metal is soft, and may lead to pitting if not removed. At least 0.0025 mm (0.0001 in) or 0.016 to 0.023 mg/mm<sup>2</sup> (5 to 10 mg/in<sup>2</sup>) should be removed. (If clad alloy specimens are to be used, special