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Standard Guide for
Laboratory Immersion Corrosion Testing of Metals[1]

This standard is issued under the fixed designation NACE 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. 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 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 standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

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

1.6 *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

E8/E8M 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

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[1] 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.

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[2] 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 *First* Service at firstservice@nace.org.

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

2.2 *NACE/ASTM Standard:*[2]

G193 Terminology and Acronyms Relating to Corrosion

2.3 *NACE International Standard:*[2]

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

2.4 *International Organization for Standardization (ISO) Standards:*[3]

ISO 3651-1 Determination of resistance to intergranular corrosion of stainless steels – Part 1: 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 Anodizing of aluminum and its alloys – Rating system for the evaluation of pitting corrosion – Chart method

ISO 8994 Anodizing of aluminum and its 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 – Guidelines for the 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.

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[3] Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, http://www.iso.org.

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 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 and G110, ASTM Test Methods G28, G34, G66, G67, G108, 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 stress-corrosion 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 mL to 5000 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.

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 samples for test, etc., should be used.
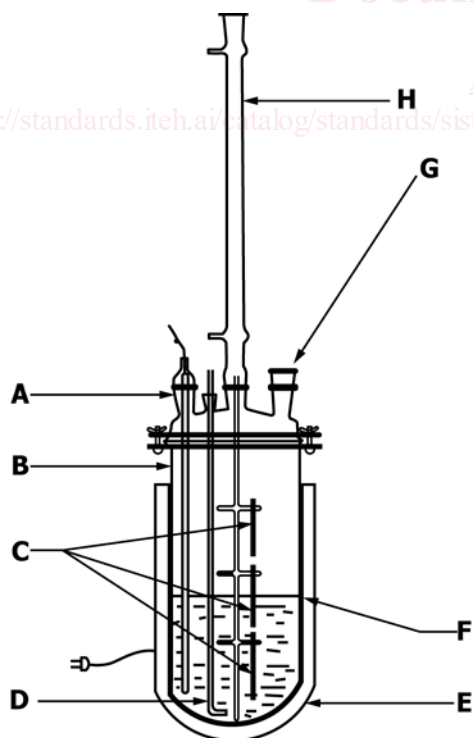
A = thermometer port, B = flask, C = specimens hung on supporting device, D = air inlet, E = heating mantle, F = liquid interface, G = opening in flask for additional apparatus that may be required, and H = reflux condenser.

FIG. 1 Typical Resin Flask

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 ±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/E8M.

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 ±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 mg∕mm^2 to 0.023 mg/mm^2 (5 mg/in.^{2} to 10 mg/in.^{2}) should be removed. (If clad alloy specimens are to be used, special attention shall 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. Materials that form passive films may give different results if exposed after different rest times following polishing. In special cases (for example, for aluminum and certain copper alloys), a minimum of 24 h storage in a desiccator is recommended. The choice of a specific surface treatment shall 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, and so forth, and therefore not be representative of the application.

8.3.1 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 that 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.

8.3.1.1 Specimens of different alloy compositions should never be ground on the same cloth.

8.3.1.2 Wet grinding should be used on alloys that work harden readily, such as austenitic stainless steels.

8.4 Sheared edges should be removed unless the purpose of the test is to study effects of the shearing operation or unless the effect of the deformation resulting from shearing is known to have no effect on corrosion. A sheared edge can be removed before testing by wet grinding to a distance from the sheared edge equal to the thickness of the specimen. It may be desirable to test a surface representative of the material and metallurgical conditions used in practice.

8.5 As-laser-cut edges should be removed unless the purpose of the test is to study effects of the laser-cutting process. The effects of laser cutting can be removed from an edge before testing by sanding or wet grinding to a distance from the cut edge equal to 125 μm (0.005 in.).

8.6 The specimen may be stamped with an appropriate identifying mark. If metallic contamination of the stamped area may influence the corrosion behavior, chemical cleaning shall 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).

8.6.1 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.

8.6.2 Environmentally assisted 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. Additional types of tests should be performed to specifically study the effects of stress.

8.6.3 The relative location of test specimens in the test apparatus should be recorded prior to testing to permit test specimen identification in the event the identification mark is corroded away.

8.7 Test specimens may be scrubbed with a 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 % diethylether), 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 test specimen.

8.7.1 Proper ultrasonic procedures are an acceptable alternate.

8.7.2 The use of towels for drying may introduce an error through contamination of the specimens with grease or lint.

8.7.3 Test specimens should be handled with gloves, tweezers, or tongs to avoid contamination of the surface after cleaning.

8.8 The mass of dried test specimens should be determined on an analytical balance to an accuracy of 1 mg or better. If cleaning deposits (for example, scouring powder) remain or lack of complete dryness is suspected, then recleaning and drying should be performed until a constant mass is attained.

9. Test Conditions

9.1 Selection of the conditions for a laboratory corrosion test shall be determined by the purpose of the test.

9.1.1 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 shall be determined. These factors include oxygen concentration, temperature, rate of flow, pH value, composition, and other important characteristics of the solution.

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

9.3 Test conditions should be controlled throughout the test in order to ensure reproducible results.

9.4 *Composition of Solution:*

9.4.1 Test solutions should be prepared accurately from chemicals conforming to the laboratory-grade standards, such as those of the Committee on Analytical Reagents of the

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American Chemical Society,[4] and using reagent water (ASTM Specification D1193, Type IV or better), except in those cases in which naturally occurring solutions or those taken directly from plant processes are used.

9.4.2 The composition of the test solution should be accurately controlled and should be described as completely and precisely as possible when the results are reported.

9.4.3 Minor constituents should be included because they often affect corrosion rates.

9.4.4 Chemical content should be reported as percentage by mass of the solutions. Molarity and normality are also helpful in defining the concentration of chemicals in some test solutions. Solution density and pH should also be reported.

9.4.5 An environmental sampling plan should be devised. The test solution components of interest should be determined before the test begins to avoid costs associated with measuring components not of interest and to ensure that all components of interest are measured before the test begins. Such a plan may follow the guidance of ASTM Practice E300. When components of interest in the solution are expected to change over time, the composition of the test solution should be checked by analysis before and after testing to determine the extent of change in composition. It may be useful to analyze samples of the test solution during the test as well. In many cases, only certain components of the test solution will be of interest, for example those components that affect corrosiveness such as pH. It may be of interest to measure soluble corrosion products such as metal ion content as a method of determining the amount of corrosion that has occurred.

9.4.6 Evaporation losses may be controlled by a constant level device or by frequent addition of appropriate solution to maintain the original volume within ±1 %. The use of a reflux condenser ordinarily precludes the necessity of adding to the original solution.

9.4.7 In some cases, composition of the test solution may change as a result of catalytic decomposition, by reaction with the test specimen, or through the buildup of corrosion products in the solution. These changes should be determined, if possible. When required, the exhausted constituents should be added or a fresh solution provided during the course of the test.

9.4.8 If several different metals are exposed in the same volume of test solution, the corrosion products from one metal may affect the rate of attack on another metal. For example, copper corrosion products can reduce corrosion of stainless steel and titanium but can accelerate corrosion of aluminum.

9.5 *Temperature of Solution:*

9.5.1 Temperature of the test solution is typically controlled to within ±1 °C or ±2 °F. In tests where the temperature is significantly above ambient (for example, >10 °C or 18 °F) some method of stirring is usually desirable to avoid temperature gradients in the solution. Magnetic stirring or gas sparging is usually sufficient to minimize temperature gradients.

9.5.2 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, should be selected to match the application being investigated.

9.5.3 For tests that are intended to simulate systems that are 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 °C to 45 °C (104 °F to 113 °F) in some areas.

9.5.4 Tests at the boiling point of the solution should be conducted with minimum possible heat input, and inert material boiling chips should be used to minimize excessive bumping, turbulence, and bubble impingement. The use of a reflux condenser is strongly recommended when tests are conducted at the boiling point.

9.6 *Gas Sparging of Solution:*

9.6.1 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 or a controlled gas atmosphere.

9.6.2 If gas sparging or a controlled gas flow is employed, the specimens should not be located in the direct air or gas stream from the sparger. Extraneous effects can be encountered if the air or gas stream impinges on the specimens. Gas spargers that create small bubbles are usually preferred to open tubes.

NOTE 1—Assuming that all other factors are equivalent, the ratio of the gas flow rate to the total solution volume and the average gas bubble diameter may be useful measures of sparging efficiency.

9.6.3 If complete exclusion of dissolved oxygen is necessary, specific techniques are required, such as prior heating of the test solution and sparging with an inert gas (usually nitrogen). A liquid atmospheric seal (that is, vapor lock, similar in function to a sink drain trap) is appropriate for use on the test vessel to prevent further contamination.

9.6.4 If a high oxygen concentration of the test solution is desired, one method of achieving this is by sparging with pure oxygen. For lower concentrations of oxygen in the solution, the solution may be sparged with a mixture of oxygen with air or an inert gas. The saturation concentration of oxygen in a solution is proportional to the partial pressure of oxygen in the gas mixture that is used for sparging. Care should be exercised when sparging with oxygen or oxygen-enriched gas, since many materials have enhanced flammability in gas enriched with oxygen.

9.6.5 Other atmospheres may also be used as required to simulate a specific service. Sparging the gas into the test solution allows the gas composition to approach saturation if the sparging is continued for sufficient time. However, other techniques may also be employed or required to achieve saturation specific concentration.

9.7 *Test Solution Flow:*

9.7.1 The effect of fluid flow is not usually determined in normal laboratory tests, although specific tests have been designed for this purpose. Controlled-flow tests are outside the scope of this guide. ASTM Guide G170, ASTM Practice G184,

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[4] *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.

and ASTM Practice G185 provide useful information regarding testing under flowing conditions.

9.7.2 In tests conducted below the boiling point, thermal convection may be an adequate source of liquid agitation.

9.7.3 In test solutions with viscosities above 100 cp, supplemental controlled stirring with a magnetic stirrer is recommended.

9.7.4 Gas sparging can effectively agitate the test solution and prevent stagnation and the development of unwanted concentration gradients during the test. Gas sparging should not be used in solutions that are prone to foaming.

9.8 *Volume of Test Solution:*

9.8.1 The volume of the test solution should be large enough to avoid any appreciable change in the test solution's corrosiveness through either exhaustion of corrosive constituents or accumulation of corrosion products that might affect further corrosion.

9.8.2 For tests up to 30 days in duration, the preferred minimum ratio of test solution volume to test specimen surface area is 0.20 mL/mm^2 (130 mL/in.^{2}).

9.8.3 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 should also be reproduced as closely as possible to assure that the effects are simulated.

9.9 *Method of Supporting Specimens:*

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

9.9.2 The method of supporting test specimens varies with the test apparatus used but should be designed to hold the specimens securely enough to avoid rubbing against other specimens, to insulate the test specimens from each other electrically, and to insulate the test specimens from any metallic container or supporting device used within the test apparatus. The more tightly a specimen is held the more likely it is to suffer crevice corrosion. Therefore it is usually found that a gentle support with a minimum area in contact with the support device is best. The use of a small-diameter cylindrical hook through a larger-diameter hole in the specimen is most often effective.

9.9.3 The shape and form of the test specimen support should assure free contact of the test specimen with the test solution, the liquid line, or the vapor phase, as shown in Fig. 1. If clad alloys are exposed, special procedures are 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.

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

9.10 *Duration of Test:*

9.10.1 The duration of any test should be determined by the nature and purpose of the test.

9.10.2 Materials that experience severe corrosion generally do not need lengthy tests to obtain accurate corrosion rates.

However, there are cases in which 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 rate decreases considerably so that further corrosion is negligible. This phenomenon of forming a protective film is observed with many corrosion-resistant materials. Short tests on such materials could indicate a high corrosion rate and be misleading.

9.10.3 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. On the other hand, 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.

9.10.4 The planned-interval test is 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. If the environment may change over time, short-duration exposures should be conducted at both the beginning and the end of a longer exposure to ensure that the effects of environmental changes can be separated from the effects of exposure time. Other procedures that require the removal of solid corrosion products between exposure periods do not accurately measure the normal changes of corrosion with time unless this simulates the operating conditions. For example, in laboratory tests simulating flow through systems (for example, vapor condensers), long-term tests that do not involve regular changing of the test solution may seriously underestimate the plant condition corrosiveness by allowing corrosion products to saturate the test solution and create films that do not occur in service. Electrochemical techniques such as polarization resistance, linear polarization, electrochemical impedance spectroscopy, electrical resistance probes, and electrochemical noise measurement often allow determination of the effects of time on corrosion behavior.

9.10.5 If anticipated corrosion rates are moderate or low, Eq 1 gives a suggested test duration:

$$\text{Duration of test (h)} \qquad (1)$$

$$= 50/\text{corrosion rate (mm/y) or } 2000/\text{corrosion rate (mpy)}$$

9.10.5.1 *Example*—When the corrosion rate is 0.25 mm/y (10 mpy), the test should run for at least 200 h.

9.10.5.2 This method of estimating test duration is useful only as an aid in deciding, after a test has been completed, whether it is desirable to repeat the test for a longer period. Common testing periods are 24 h to 240 h (1 day to 10 days).

9.10.5.3 This equation for test duration is acceptable for general corrosion rates only. Localized attack, for example, pitting, environmentally assisted cracking, crevice attack, etc., normally have an initiation period before attack begins.

9.10.6 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