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

This standard is issued under the fixed designation 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 practice[2] describes accepted procedures for and factors that influence laboratory immersion corrosion tests, particularly mass loss tests. These factors include specimen preparation, apparatus, test conditions, methods of cleaning specimens, evaluation of results, and calculation and reporting of corrosion rates. This practice also emphasizes the importance of recording all pertinent data and provides a checklist for reporting test data. Other ASTM procedures for laboratory corrosion tests are tabulated in the Appendix. (**Warning—**In many cases the corrosion product on the reactive metals titanium and zirconium is a hard and tightly bonded oxide that defies removal by chemical or ordinary mechanical means. In many such cases, corrosion rates are established by mass gain rather than mass loss.)

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

1.3 *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:*[3]

A262 Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels
E8 Test Methods for Tension Testing of Metallic Materials
G1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens
G4 Guide for Conducting Corrosion Tests in Field Applications
G16 Guide for Applying Statistics to Analysis of Corrosion Data
G46 Guide for Examination and Evaluation of Pitting Corrosion

3. Significance and Use

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

3.2 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 obviously required.

3.3 In designing any corrosion test, consideration must be given to the various factors discussed in this practice, because these factors have been found to affect greatly the results obtained.

4. Interferences

4.1 The methods and procedures described herein represent the best current practices for conducting laboratory corrosion tests as developed by corrosion specialists in the process industries. For proper interpretation of the results obtained, the specific influence of certain variables must be considered. These include:

4.1.1 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) must be employed (**1**).[4]

4.1.2 In laboratory tests, the velocity of the environment relative to the specimens will normally be determined by convection currents or the effects induced by aeration or boiling or both. If the specific effects of high velocity are to be studied, special techniques must be employed to transfer the

[1] This practice is under the jurisdiction of ASTM Committee J01 on Corrosion and is the direct responsibility of Subcommittee J01.01 on Working Group on Laboratory Immersion Tests.

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[2] This practice is based upon NACE Standard TM-01-69, "Test Method-Laboratory Corrosion Testing of Metals for the Process Industries," with modifications to relate more directly to Practices G1 and G1 and Guide G4.

[3] 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.

[4] The boldface numbers in parentheses refer to the list of references at the end of this practice.

environment through tubular specimens or to move it rapidly past the plane face of a corrosion coupon (**2**). Alternatively, the coupon may be rotated through the environment, although it is then difficult to evaluate the velocity quantitatively because of the stirring effects incurred.

4.1.3 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 completely aerated or deaerated in accordance with 8.7.

4.1.4 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.

4.1.5 Corrosion products may have undesirable effects on a chemical product. The amount of possible contamination can be estimated from the loss in mass of the specimen, with proper application of the expected relationships among (*1*) the area of corroding surface, (*2*) the mass of the chemical product handled, and (*3*) the duration of contact of a unit of mass of the chemical product with the corroding surface.

4.1.6 Corrosion products from the coupon 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. Cupric ions may also exhibit a passivating effect upon stainless steel coupons exposed at the same time. In practice, only alloys of the same general type should be exposed in the testing apparatus.

4.1.7 Coupon corrosion testing is predominantly designed to investigate general corrosion. There are a number of other special types of phenomena of which one must be aware in the design and interpretation of corrosion tests.

4.1.7.1 Galvanic corrosion may be investigated by special devices which couple one coupon to another in electrical contact. The behavior of the specimens in this galvanic couple are compared with that of insulated specimens exposed on the same holder and the galvanic effects noted. It should be observed, however, that galvanic corrosion can be greatly affected by the area ratios of the respective metals, the distance between the metals and the resistivity of the electrolyte. The coupling of corrosion coupons then yields only qualitative results, as a particular coupon reflects only the relationship between these two metals at the particular area ratio involved.

4.1.7.2 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.

4.1.7.3 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 coupons after exposure.

4.1.7.4 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.

4.1.7.5 Certain metals and alloys are subject to a highly localized type of attack called pitting corrosion. This cannot be evaluated by mass loss alone. The reporting of nonuniform corrosion is discussed below. It should be appreciated that pitting is a statistical phenomenon and that the incidence of pitting may be directly related to the area of metal exposed. For example, a small coupon 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 contaminated environments.

4.1.7.6 All metals and alloys are subject to stress-corrosion 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 stress-corrosion cracking. It is imperative to note that this usually occurs with no significant loss in mass of the test coupon, although certain refractory metals are an exception to these observations. Generally, if cracking is observed on the coupon, it can be taken as positive indication of susceptibility, whereas failure to effect this phenomenon simply 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 stress corrosion cracking (see Ref. (**3**)).

5. Apparatus

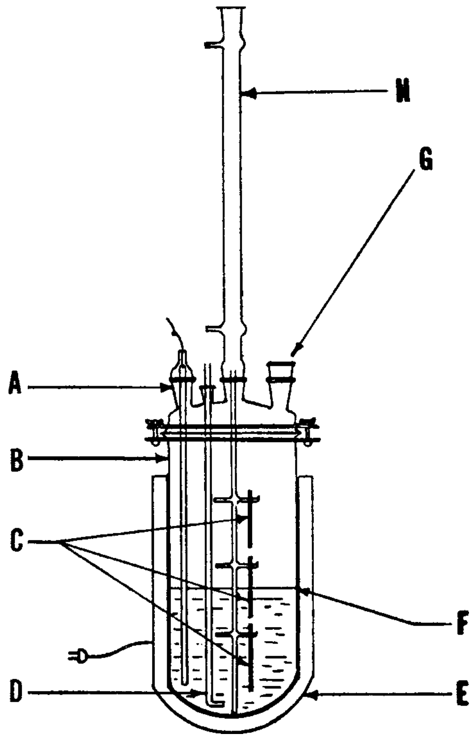
5.1 A versatile and convenient apparatus should be used, consisting of a kettle or flask of suitable size (usually 500 to 5000 mL), a reflux condenser with atmospheric seal, a sparger for controlling atmosphere or aeration, a thermowell and temperature-regulating device, a heating device (mantle, hot plate, or bath), and a 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 resin flask setup for this type test is shown in Fig. 1.

5.2 The suggested components can be modified, simplified, or made more sophisticated to fit the needs of a particular investigation. The suggested apparatus is basic and the apparatus is limited only by the judgment and ingenuity of the investigator.

5.2.1 A glass reaction kettle can be used where the configuration and size of the specimen will permit entry through the narrow kettle neck (for example, 45/50 ground-glass joint). For solutions corrosive to glass, suitable metallic or plastic kettles may be employed.

5.2.2 In some cases a wide-mouth jar with a suitable closure is sufficient when simple immersion tests at ambient temperatures are to be investigated.

5.2.3 Open-beaker tests should not be used because of evaporation and contamination.



NOTE 1—The flask can be used as a versatile and convenient apparatus to conduct simple immersion tests. Configuration of top to flask is such that more sophisticated apparatus can be added as required by the specific test being conducted. *A* = thermowell, *B* = resin 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

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

6. Sampling

6.1 The bulk sampling of products is outside the scope of this practice.

7. Test Specimen

7.1 In laboratory tests, uniform corrosion rates of duplicate specimens are usually within ±10 % under the same test conditions. 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. Therefore, at least duplicate specimens should normally be exposed in each test.

7.2 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 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 Test Methods E8.

7.3 The size and shape of specimens will vary with the purpose of the test, nature of the materials, and apparatus used. A large surface-to-mass ratio and a small ratio of edge area to total area are desirable. These ratios can be achieved through the use of square or circular specimens of minimum thickness. Masking may also be used to achieve the desired area ratios but may cause crevice corrosion problems. Circular specimens should preferably be cut from sheet and not bar stock, to minimize the exposed end grain. Special coupons (for example, sections of welded tubing) may be employed for specific purposes.

7.3.1 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. The total surface area of a circular specimen is given by the following equation:

$$A = \pi/2(D^2 - d^2) + t\pi D + t\pi d \quad (1)$$

where:

t = thickness,
D = diameter of the specimen, and
d = diameter of the mounting hole.

7.3.1.1 If the hole is completely covered by the mounting support, the last term ($t\pi d$) in the equation is omitted.

7.3.2 Strip coupons 50 by 25 by 1.6 or 3 mm (2 by 1 by 1⁄16 or 1⁄8 in.) may be preferred as corrosion specimens, particularly if interface or liquid line effects are to be studied by the laboratory tests (see Fig. 1), but the evaluation of such specific effects are beyond the scope of this practice.

7.3.3 All specimens should be measured carefully to permit accurate calculation of the exposed areas. A geometric area calculation accurate to ±1 % is usually adequate.

7.4 More uniform results may be expected if a substantial 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 (see section 5.7). At least 0.0025 mm (0.0001 in.) or 0.0155 to 0.0233 mg/mm^2 (10 to 15 mg/in.^{2}) should be removed. (If clad alloy specimens are to be used, special attention must 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. 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 treatment must 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.

7.5 Exposure of sheared edges should be avoided unless the purpose of the test is to study effects of the shearing operation. It may be desirable to test a surface representative of the material and metallurgical conditions used in practice.

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

7.6.1 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.

7.6.2 Stress-corrosion 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 (see 4.1.7.6).

7.7 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 which 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.

7.7.1 Coupons of different alloy compositions should never be ground on the same cloth.

7.7.2 Wet grinding should be used on alloys which work harden quickly, such as the austenitic stainless steels.

7.8 The specimens should be finally degreased by scrubbing with 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 % ether), 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 specimen. Proper ultrasonic procedures are an acceptable alternate. The use of towels for drying may introduce an error through contamination of the specimens with grease or lint.

7.9 The dried specimens should be weighed on an analytical balance to an accuracy of at least ±0.5 mg. If cleaning deposits (for example, scouring powder) remain or lack of complete dryness is suspected, then recleaning and drying is performed until a constant mass is attained.

7.10 The method of specimen preparation should be described when reporting test results, to facilitate interpretation of data by other persons.

7.11 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.

7.11.1 The heat-affected zone is also of importance but should be studied separately, because welds on coupons do not faithfully reproduce heat input or size effects of full-size weldments.

7.11.2 Corrosion of a welded coupon is best reported by description and thickness measurements rather than a millimetre per year (mils per year) rate, because the attack is normally localized and not representative of the entire surface.

7.11.3 A complete discussion of corrosion testing of welded coupons or the effect of heat treatment on the corrosion resistance of a metal is not within the scope of this practice.

8. Test Conditions

8.1 Selection of the conditions for a laboratory corrosion test will be determined by the purpose of the test.

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

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

8.3 It is important that test conditions be controlled throughout the test in order to ensure reproducible results.

8.4 The spread in corrosion rate values for duplicate specimens in a given test probably should not exceed ±10 % of the average when the attack is uniform.

8.5 *Composition of Solution*:

8.5.1 Test solutions should be prepared accurately from chemicals conforming to the Specifications of the Committee on Analytical Reagents of the American Chemical Society[5] and distilled water, except in those cases where naturally occurring solutions or those taken directly from some plant process are used.

8.5.2 The composition of the test solutions should be controlled to the fullest extent possible and should be described as completely and as accurately as possible when the results are reported.

8.5.2.1 Minor constituents should not be overlooked because they often affect corrosion rates.

8.5.2.2 Chemical content should be reported as percentage by weight of the solutions. Molarity and normality are also helpful in defining the concentration of chemicals in some test solutions.

8.5.3 If problems are suspected, the composition of the test solutions should be checked by analysis at the end of the test to determine the extent of change in composition, such as might result from evaporation or depletion.

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

8.5.5 In some cases, composition of the test solution may change as a result of catalytic decomposition or by reaction with the test coupons. These changes should be determined if possible. Where required, the exhausted constituents should be added or a fresh solution provided during the course of the test.

8.5.6 When possible, only one type of metal should be exposed in a given test (see 4.1.6).

[5] *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.