



Designation: G1 – 03

Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens¹

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1. Scope

1.1 This practice covers suggested procedures for preparing bare, solid metal specimens for tests, for removing corrosion products after the test has been completed, and for evaluating the corrosion damage that has occurred. Emphasis is placed on procedures related to the evaluation of corrosion by mass loss and pitting measurements. (**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 *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.* For specific precautionary statements, see 1 and 7.2.

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
- D1384 Test Method for Corrosion Test for Engine Coolants in Glassware
- D2776 Test Methods for Corrosivity of Water in the Absence of Heat Transfer (Electrical Methods)
- G15 Terminology Relating to Corrosion and Corrosion Testing
- G16 Guide for Applying Statistics to Analysis of Corrosion Data

¹ This practice is under the jurisdiction of ASTM Committee G01 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.

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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.

- G31 Practice for Laboratory Immersion Corrosion Testing of Metals
- G33 Practice for Recording Data from Atmospheric Corrosion Tests of Metallic-Coated Steel Specimens
- G46 Guide for Examination and Evaluation of Pitting Corrosion
- G50 Practice for Conducting Atmospheric Corrosion Tests on Metals
- G78 Guide for Crevice Corrosion Testing of Iron-Base and Nickel-Base Stainless Alloys in Seawater and Other Chloride-Containing Aqueous Environments

3. Terminology

3.1 See Terminology G15 for terms used in this practice.

4. Significance and Use

4.1 The procedures given are designed to remove corrosion products without significant removal of base metal. This allows an accurate determination of the mass loss of the metal or alloy that occurred during exposure to the corrosive environment.

4.2 These procedures, in some cases, may apply to metal coatings. However, possible effects from the substrate must be considered.

5. Reagents and Materials

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.³ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV of Specification D1193.

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.

6. Methods for Preparing Specimens for Test

6.1 For laboratory corrosion tests that simulate exposure to service environments, a commercial surface, closely resembling the one that would be used in service, will yield the most meaningful results.

6.2 It is desirable to mark specimens used in corrosion tests with a unique designation during preparation. Several techniques may be used depending on the type of specimen and test.

6.2.1 *Stencil or Stamp*—Most metallic specimens may be marked by stenciling, that is, imprinting the designation code into the metal surface using hardened steel stencil stamps hit with a hammer. The resulting imprint will be visible even after substantial corrosion has occurred. However, this procedure introduces localized strained regions and the possibility of superficial iron contamination in the marked area.

6.2.2 Electric engraving by means of a vibratory marking tool may be used when the extent of corrosion damage is known to be small. However, this approach to marking is much more susceptible to having the marks lost as a result of corrosion damage during testing.

6.2.3 Edge notching is especially applicable when extensive corrosion and accumulation of corrosion products is anticipated. Long term atmospheric tests and sea water immersion tests on steel alloys are examples where this approach is applicable. It is necessary to develop a code system when using edge notches.

6.2.4 Drilled holes may also be used to identify specimens when extensive metal loss, accumulation of corrosion products, or heavy scaling is anticipated. Drilled holes may be simpler and less costly than edge notching. A code system must be developed when using drilled holes. Punched holes should not be used as they introduce residual strain.

6.2.5 When it is undesirable to deform the surface of specimens after preparation procedures, for example, when testing coated surfaces, tags may be used for specimen identification. A metal or plastic wire can be used to attach the tag to the specimen and the specimen identification can be stamped on the tag. It is important to ensure that neither the tag nor the wire will corrode or degrade in the test environment. It is also important to be sure that there are no galvanic interactions between the tag, wire, and specimen.

6.3 For more searching tests of either the metal or the environment, standard surface finishes may be preferred. A suitable procedure might be:

6.3.1 Degrease in an organic solvent or hot alkaline cleaner. (See also Practice [G31](#).)

NOTE 1—Hot alkalis and chlorinated solvents may attack some metals.

NOTE 2—Ultrasonic cleaning may be beneficial in both pre-test and post-test cleaning procedures.

6.3.2 Pickle in an appropriate solution if oxides or tarnish are present. In some cases the chemical cleaners described in Section [6](#) will suffice.

NOTE 3—Pickling may cause localized corrosion on some materials.

6.3.3 Abrade with a slurry of an appropriate abrasive or with an abrasive paper (see Practices [A262](#) and Test Method

[D1384](#)). The edges as well as the faces of the specimens should be abraded to remove burrs.

6.3.4 Rinse thoroughly, hot air dry, and store in desiccator.

6.4 When specimen preparation changes the metallurgical condition of the metal, other methods should be chosen or the metallurgical condition must be corrected by subsequent treatment. For example, shearing a specimen to size will cold work and may possibly fracture the edges. Edges should be machined.

6.5 The clean, dry specimens should be measured and weighed. Dimensions determined to the third significant figure and mass determined to the fifth significant figure are suggested. When more significant figures are available on the measuring instruments, they should be recorded.

7. Methods for Cleaning After Testing

7.1 Corrosion product removal procedures can be divided into three general categories: mechanical, chemical, and electrolytic.

7.1.1 An ideal procedure should remove only corrosion products and not result in removal of any base metal. To determine the mass loss of the base metal when removing corrosion products, replicate uncorroded control specimens should be cleaned by the same procedure being used on the test specimen. By weighing the control specimen before and after cleaning, the extent of metal loss resulting from cleaning can be utilized to correct the corrosion mass loss.

NOTE 4—It is desirable to scrape samples of corrosion products before using any chemical techniques to remove them. These scrapings can then be subjected to various forms of analyses, including perhaps X-ray diffraction to determine crystal forms as well as chemical analyses to look for specific corrodants, such as chlorides. All of the chemical techniques that are discussed in Section [7](#) tend to destroy the corrosion products and thereby lose the information contained in these corrosion products. Care may be required so that uncorroded metal is not removed with the corrosion products.

7.1.2 The procedure given in [7.1.1](#) may not be reliable when heavily corroded specimens are to be cleaned. The application of replicate cleaning procedures to specimens with corroded surfaces will often, even in the absence of corrosion products, result in continuing mass losses. This is because a corroded surface, particularly of a multiphase alloy, is often more susceptible than a freshly machined or polished surface to corrosion by the cleaning procedure. In such cases, the following method of determining the mass loss due to the cleaning procedure is preferred.

7.1.2.1 The cleaning procedure should be repeated on specimens several times. The mass loss should be determined after each cleaning by weighing the specimen.

7.1.2.2 The mass loss should be graphed as a function of the number of equal cleaning cycles as shown in [Fig. 1](#). Two lines will be obtained: AB and BC. The latter will correspond to corrosion of the metal after removal of corrosion products. The mass loss due to corrosion will correspond approximately to point B.

7.1.2.3 To minimize uncertainty associated with corrosion of the metal by the cleaning method, a method should be chosen to provide the lowest slope (near to horizontal) of line BC.

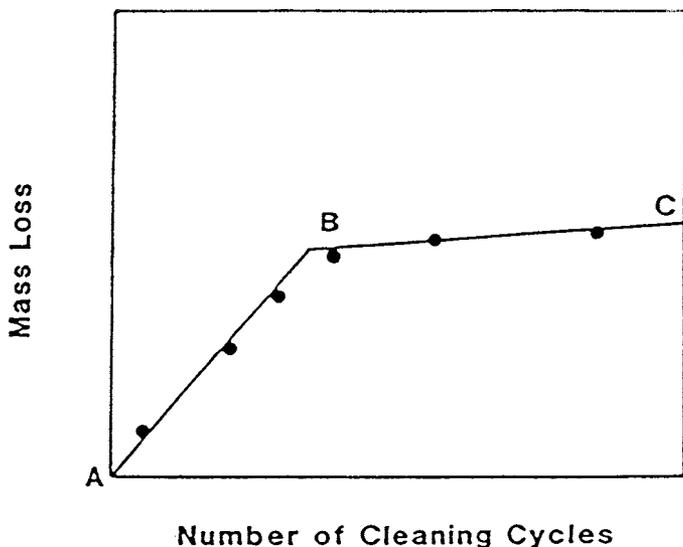


FIG. 1 Mass Loss of Corroded Specimens Resulting from Repetitive Cleaning Cycles

7.1.3 Repeated treatment may be required for complete removal of corrosion products. Removal can often be confirmed by examination with a low power microscope (for example, 7× to 30×). This is particularly useful with pitted surfaces when corrosion products may accumulate in pits. This repeated treatment may also be necessary because of the requirements of 7.1.2.1. Following the final treatment, the specimens should be thoroughly rinsed and immediately dried.

7.1.4 All cleaning solutions shall be prepared with water and reagent grade chemicals.

7.2 Chemical procedures involve immersion of the corrosion test specimen in a specific solution that is designed to remove the corrosion products with minimal dissolution of any base metal. Several procedures are listed in Table A1.1. The choice of chemical procedure to be used is partly a matter of trial and error to establish the most effective method for a specific metal and type of corrosion product scale. (**Warning**—These methods may be hazardous to personnel).

7.2.1 Chemical cleaning is often preceded by light brushing (non metallic bristle) or ultrasonic cleaning of the test specimen to remove loose, bulky corrosion products.

7.2.2 Intermittent removal of specimens from the cleaning solution for light brushing or ultrasonic cleaning can often facilitate the removal of tightly adherent corrosion products.

7.2.3 Chemical cleaning is often followed by light brushing or ultrasonic cleaning in reagent water to remove loose products.

7.3 Electrolytic cleaning can also be utilized for removal of corrosion products. Several useful methods for corrosion test specimens of iron, cast iron, or steel are given in Table A2.1.

7.3.1 Electrolytic cleaning should be preceded by brushing or ultrasonic cleaning of the test specimen to remove loose, bulky corrosion products. Brushing or ultrasonic cleaning should also follow the electrolytic cleaning to remove any loose slime or deposits. This will help to minimize any redeposition of metal from reducible corrosion products that would reduce the apparent mass loss.

7.4 Mechanical procedures can include scraping, scrubbing, brushing, ultrasonic cleaning, mechanical shocking, and impact blasting (for example, grit blasting, water-jet blasting, and so forth). These methods are often utilized to remove heavily encrusted corrosion products. Scrubbing with a nonmetallic bristle brush and a mild abrasive-distilled water slurry can also be used to remove corrosion products.

7.4.1 Vigorous mechanical cleaning may result in the removal of some base metal; therefore, care should be exercised. These should be used only when other methods fail to provide adequate removal of corrosion products. As with other methods, correction for metal loss due to the cleaning method is recommended. The mechanical forces used in cleaning should be held as nearly constant as possible.

8. Assessment of Corrosion Damage

8.1 The initial total surface area of the specimen (making corrections for the areas associated with mounting holes) and the mass lost during the test are determined. The average corrosion rate may then be obtained as follows:

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

where:

- K = a constant (see 8.1.2),
- T = time of exposure in hours,
- A = area in cm^2 ,
- W = mass loss in grams, and
- D = density in g/cm^3 (see Appendix X1).

8.1.1 Corrosion rates are not necessarily constant with time of exposure. See Practice G31 for further guidance.

8.1.2 Many different units are used to express corrosion rates. Using the units in 7.1 for T , A , W , and D , the corrosion rate can be calculated in a variety of units with the following appropriate value of K :

Corrosion Rate Units Desired	Constant (K) in Corrosion Rate Equation
mils per year (mpy)	3.45×10^6
inches per year (ipy)	3.45×10^3
inches per month (ipm)	2.87×10^2
millimetres per year (mm/y)	8.76×10^4
micrometres per year ($\mu\text{m}/\text{y}$)	8.76×10^7
picometres per second (pm/s)	2.78×10^6
grams per square meter per hour ($\text{g}/\text{m}^2\cdot\text{h}$)	$1.00 \times 10^4 \times D$
milligrams per square decimeter per day (mdd)	$2.40 \times 10^6 \times D$
micrograms per square meter per second ($\mu\text{g}/\text{m}^2\cdot\text{s}$)	$2.78 \times 10^6 \times D$

NOTE 5—If desired, these constants may also be used to convert corrosion rates from one set of units to another. To convert a corrosion rate in units X to a rate in units Y , multiply by K_Y/K_X ; for example:

$$15 \text{ mpy} = 15 \times (2.78 \times 10^6)/(3.45 \times 10^6) \text{ pm/s} \quad (2)$$

8.1.3 In the case of sacrificial alloy coatings for which there is preferential corrosion of a component whose density differs from that of the alloy, it is preferable to use the density of the corroded component (instead of the initial alloy density) for calculating average thickness loss rate by use of Eq 1. This is done as follows: (1) cleaning to remove corrosion products only and determine the mass loss of the corroded component; (2) stripping the remaining coating to determine the mass of the uncorroded component; (3) chemical analysis of the stripping solution to determine the composition of the uncorroded

component; (4) performing a mass balance to calculate the composition of the corroded component; (5) using the mass and density of the corroded component to calculate the average thickness loss rate by use of Eq 1. An example of this procedure is given in [Appendix X2](#).

The procedure described above gives an average penetration rate of the coating, but the maximum penetration for a multiphase alloy may be larger when the corroded phase is not uniformly distributed across the surface. In such cases, it is generally considered good practice to obtain a cross section through the corroded surface for microscopic examination. This examination will reveal the extent of selective corrosion of particular phases in the coating, and help in understanding the mechanism of attack.

8.2 Corrosion rates calculated from mass losses can be misleading when deterioration is highly localized, as in pitting or crevice corrosion. If corrosion is in the form of pitting, it may be measured with a depth gage or micrometer calipers with pointed anvils (see [Guide G46](#)). Microscopical methods will determine pit depth by focusing from top to bottom of the pit when it is viewed from above (using a calibrated focusing knob) or by examining a section that has been mounted and metallographically polished. The pitting factor is the ratio of the deepest metal penetration to the average metal penetration (as measured by mass loss).

NOTE 6—See [Guide G46](#) for guidance in evaluating depths of pitting.

NOTE 7—See [Guide G78](#) for guidance in evaluating crevice corrosion.

8.3 Other methods of assessing corrosion damage are:

8.3.1 *Appearance*—The degradation of appearance by rusting, tarnishing, or oxidation. (See [Practice G33](#).)

8.3.2 *Mechanical Properties*—An apparent loss in tensile strength will result if the cross-sectional area of the specimen (measured before exposure to the corrosive environment) is reduced by corrosion. (See [Practice G50](#).) Loss in tensile strength will result if a compositional change, such as dealloying taking place. Loss in tensile strength and elongation will result from localized attack, such as cracking or intergranular corrosion.

8.3.3 *Electrical Properties*—Loss in electrical conductivity can be measured when metal loss results from uniform corrosion. (See [Test Methods D2776](#).)

8.3.4 *Microscopical Examination*—Dealloying, exfoliation, cracking, or intergranular attack may be detected by metallographic examination of suitably prepared sections.

9. Report

9.1 The report should include the compositions and sizes of specimens, their metallurgical conditions, surface preparations, and cleaning methods as well as measures of corrosion damage, such as corrosion rates (calculated from mass losses), maximum depths of pitting, or losses in mechanical properties.

10. Precision and Bias

10.1 The factors that can produce errors in mass loss measurement include improper balance calibration and standardization. Generally, modern analytical balances can determine mass values to ± 0.2 mg with ease and balances are available that can obtain mass values to ± 0.02 mg. In general, mass measurements are not the limiting factor. However, inadequate corrosion product removal or overcleaning will affect precision.

10.2 The determination of specimen area is usually the least precise step in corrosion rate determinations. The precision of calipers and other length measuring devices can vary widely. However, it generally is not necessary to achieve better than ± 1 % for area measurements for corrosion rate purposes.

10.3 The exposure time can usually be controlled to better than ± 1 % in most laboratory procedures. However, in field exposures, corrosive conditions can vary significantly and the estimation of how long corrosive conditions existed can present significant opportunities for error. Furthermore, corrosion processes are not necessarily linear with time, so that rate values may not be predictive of the future deterioration, but only are indications of the past exposure.

10.4 Regression analysis on results, as are shown in [Fig. 1](#), can be used to obtain specific information on precision. See [Guide G16](#) for more information on statistical analysis.

10.5 Bias can result from inadequate corrosion product removal or metal removal caused by overcleaning. The use of repetitive cleaning steps, as shown in [Fig. 1](#), can minimize both of these errors.

10.5.1 Corrosion penetration estimations based on mass loss can seriously underestimate the corrosion penetration caused by localized processes, such as pitting, cracking, crevice corrosion, and so forth.

11. Keywords

11.1 cleaning; corrosion product removal; evaluation; mass loss; metals; preparation; specimens