



# Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion Susceptibility of Iron-, Nickel-, or Cobalt-Based Alloys<sup>1</sup>

This standard is issued under the fixed designation G 61; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers a procedure for conducting cyclic potentiodynamic polarization measurements to determine relative susceptibility to localized corrosion (pitting and crevice corrosion) for iron-, nickel-, or cobalt-based alloys in a chloride environment. This test method also describes an experimental procedure which can be used to check one's experimental technique and instrumentation.

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.*

## 2. Referenced Documents

### 2.1 ASTM Standards:

D 1193 Specification for Reagent Water<sup>2</sup>

G 3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing<sup>3</sup>

G 5 Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements<sup>3</sup>

## 3. Significance and Use

3.1 An indication of the susceptibility to initiation of localized corrosion in this test method is given by the potential at which the anodic current increases rapidly. The more noble this potential, obtained at a fixed scan rate in this test, the less susceptible is the alloy to initiation of localized corrosion. The results of this test are not intended to correlate in a quantitative manner with the rate of propagation that one might observe in service when localized corrosion occurs.

3.2 In general, once initiated, localized corrosion can propagate at some potential more electropositive than that at which the hysteresis loop is completed. In this test method, the potential at which the hysteresis loop is completed is determined at a fixed scan rate. In these cases, the more electropositive the potential at which the hysteresis loop is completed the less likely it is that localized corrosion will occur.

3.3 If followed, this test method will provide cyclic potentiodynamic anodic polarization measurements that will reproduce data developed at other times in other laboratories using this test method for the two specified alloys discussed in 3.4. The procedure is used for iron-, nickel-, or cobalt-based alloys in a chloride environment.

3.4 A standard potentiodynamic polarization plot is included. These reference data are based on the results from five different laboratories that followed the standard procedure, using specific alloys of Type 304 stainless steel, UNS S30400 and Alloy C-276, UNS N10276.<sup>4</sup> Curves are included which have been constructed using statistical analysis to indicate the acceptable range of polarization curves.

3.5 The availability of a standard test method, standard material, and standard plots should make it easy for an investigator to check his techniques to evaluate susceptibility to localized corrosion.

## 4. Apparatus

4.1 The polarization cell should be similar to the one described in Practice G 5. Other polarization cells may be equally suitable.

4.1.1 The cell should have a capacity of about 1 L and should have suitable necks or seals to permit the introduction of electrodes, gas inlet and outlet tubes, and a thermometer. The Luggin probe-salt bridge separates the bulk solution from the saturated calomel reference electrode. The probe tip should be adjustable so that it can be brought into close proximity with the working electrode.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee G01 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.11 on Electrochemical Measurements in Corrosion Testing.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 03.02.

<sup>4</sup> These standard samples are available as a set of one of each type from ASTM Headquarters at a nominal cost Order PCN ADJG0061.

4.2 Specimen Holder:

4.2.1 Specimens should be mounted in a suitable holder designed for flat strip, exposing 1 cm<sup>2</sup> to the test solution (Fig. 1). Such specimen holders have been described in the litera-

10<sup>5</sup> μA should be used. Many commercial units have a build-in instrument with an output as a voltage, which is preferred for recording purposes. For the purpose of the present test a logarithmic output is desirable.

4.6 Anodic Polarization Circuit—A scanning potentiostat is used for potentiodynamic measurements. Potential and current are plotted continuously using an X-Y recorder and a logarithmic converter (contained in the potentiostat or incorporated into the circuit) for the current. Commercially available units are suitable.

4.7 Electrodes:

4.7.1 The standard Type 304 stainless steel (UNS S30400) and Alloy C-276 (UNS N10276) should be machined into flat 0.625-in. (14-mm) diameter disks. The chemical compositions of the alloys used in the round robin are listed in Table 1.

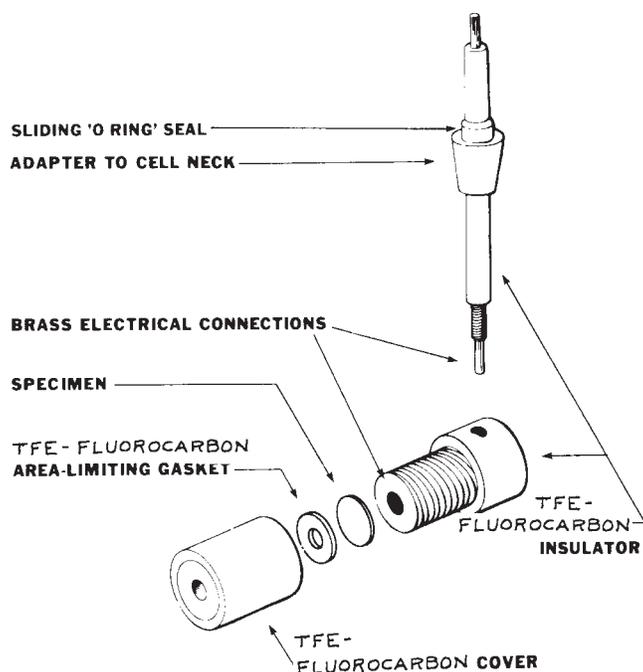


FIG. 1 Schematic Diagram of Specimen Holder<sup>4,5</sup>

ture.<sup>5</sup> It is important that the circular TFE-fluorocarbon gasket be drilled and machined flat in order to minimize crevices.

4.3 Potentiostat (Note 1)—A potentiostat that will maintain an electrode potential within 1 mV of a preset value over a wide range of applied currents should be used. For the type and size of standard specimen supplied, the potentiostat should have a potential range of -1.0 to +1.6 V and an anodic current output range of 1.0 to 10<sup>5</sup> μA. Most commercial potentiostats meet the specific requirements for these types of measurements.

NOTE 1—These instrumental requirements are based upon values typical of the instruments in the five laboratories that have provided the data used in determining the standard polarization plot.

4.4 Potential-Measuring Instruments (Note 1)—The potential-measuring circuit should have a high input impedance on the order of 10<sup>11</sup> to 10<sup>14</sup> Ω to minimize current drawn from the system during measurements. Instruments should have sufficient sensitivity and accuracy to detect a change in potential of ±1 mV, usually included in commercial potentiostats. An output as a voltage is preferred for recording purposes.

4.5 Current-Measuring Instruments (Note 1)—An instrument that is capable of measuring a current accurately to within 1 % of the absolute value over a current range between 1.0 and

TABLE 1 Chemical Composition of Alloys Used in the Round Robin, Weight %

Element	Alloy C-276 (UNS N10276)	Type 304 Stainless Steel (UNS S30400)
Carbon	0.003	0.060
Chromium	15.29	18.46
Cobalt	2.05	...
Columbium	...	0.11
Copper	...	0.17
Iron	5.78	balance
Manganese	0.48	1.43
Molybdenum	16.03	0.17
Nickel	balance	8.74
Phosphorus	0.018	0.029
Silicon	0.05	0.60
Sulfur	0.006	0.014
Vanadium	0.20	...
Tungsten	3.62	...

4.7.2 Counter Electrodes—The counter electrodes may be prepared as described in Practice G 5 or may be prepared from high-purity platinum flat stock and wire. A suitable method would be to seal the platinum wire in glass tubing and introduce the platinum electrode assembly through a sliding seal. Counter electrodes should have an area at least twice as large as the test electrode.

4.7.3 Reference Electrode<sup>6</sup>—A saturated calomel electrode with a controlled rate of leakage (about 3 μL/h) is recommended. This type of electrode is durable, reliable, and commercially available. Precautions should be taken to ensure that it is maintained in the proper condition. The potential of the calomel electrode should be checked at periodic intervals to ensure the accuracy of the electrode.

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 shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,

<sup>5</sup> France, W. D., Jr., *Journal of the Electrochemical Society*, Vol 114, 1967, p. 818; and Myers, J. R., Gruewlar, F. G., and Smulezenski, L. A., *Corrosion*, Vol 24, 1968, p. 352.

<sup>6</sup> Ives, D. J., and Janz, G. J., *Reference Electrodes, Theory and Practice*, Academic Press, New York, NY 1961.

where such specifications are available.<sup>7</sup> 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*—The water shall be distilled or deionized conforming to the purity requirements of Specification D 1193, Type IV reagent water.

5.3 Sodium Chloride (NaCl).

5.4 Samples of Standard Type 304 stainless steel (UNS S30400) and the Alloy C-276 (UNS N10276) used in obtaining the standard reference plot are available for those who wish to check their own test procedure and equipment.

## 6. Procedure

### 6.1 Test Specimen Preparation:

6.1.1 Wet grind with 240-grit SiC paper, wet polish with 600-grit SiC paper until previous coarse scratches are removed, rinse, and dry.

6.1.2 Prior to assembly of the specimen holder, ultrasonically degrease the specimen for 5 min in detergent and water, rinse thoroughly in distilled water, and dry.

6.1.3 Mount the specimen in the electrode holder. Tighten the assembly until the TFE-fluorocarbon gasket is sufficiently compressed to avoid leakage in the gasket.

6.2 Prepare a 3.56 % (by weight) sodium chloride solution by dissolving 34 g of reagent grade NaCl in 920 mL of distilled water.

6.3 Assemble the electrode holder and place in the polarization cell. Transfer 900 mL of test solution to the polarization cell, ensuring that the specimen remains above the solution level.

6.4 Bring the temperature of the solution of  $25 \pm 1^\circ\text{C}$  by immersing the test cell in a controlled-temperature water bath or by other convenient means.

6.5 Place the platinum auxiliary electrodes, salt-bridge probe, and other components in the test cell. Fill the salt bridge with test solution and locate the probe tip approximately 1 mm from the working electrode.

NOTE 2—The levels of the solution in the reference and polarization cells should be the same. If this is impossible, a closed solution-wet (not greased) stopcock can be used in the salt bridge to eliminate siphoning.

6.6 Purge the solution sufficiently with an appropriate gas to remove oxygen before specimen immersion (minimum of 1 h).

6.7 Immerse the specimen for 1 h before initiating polarization. A sliding seal can be used to ensure that an oxygen-free environment is maintained while the specimen is lowered. It is important that all oxygen be removed by purging prior to polarization, otherwise, more noble initial corrosion potential values will be observed.

6.8 Record the platinum potential 50 min after immersion of the specimen. Record the open-circuit specimen potential, that is, the corrosion potential, the instant before beginning polarization.

6.9 *Potential Scan*—Start the potential scan 1 h after specimen immersion, beginning at the corrosion potential ( $E_{\text{corr}}$ ), and scan in the more noble direction at a scan rate of 0.6 V/h ( $\pm 5\%$ ). Record the current continuously with change in potential on an X-Y recorder using semilogarithmic paper.

6.9.1 The onset of localized corrosion is usually marked by a rapid increase of the anodic current at potentials below the oxygen-evolution potential. When the current reaches 5 mA ( $5 \times 10^{-3}$   $\mu\text{A}$ ), reverse the scanning direction (toward more active potentials).

6.9.2 Continue the reverse scan until the hysteresis loop closes or until the corrosion potential is reached.

6.10 Plot anodic polarization data on semilogarithmic paper in accordance with Practice G 3 (potential-ordinate, current density-abscissa). A plot of representative polarization curves generated by the practice is shown in Fig. 2.

## 7. Interpretation of Results

7.1 The polarization curves shown in Fig. 2, Fig. 3, and Fig. 4 indicate that initiation and propagation of localized corrosion occurs at potentials more electronegative than the oxygen evolution potential on Type 304 stainless steel (UNS S30400) in the chloride environment. The curve for Alloy C-276 (UNS N10276) is not a result of localized corrosion but of uniform corrosion in the transpassive or oxygen evolution region. Since the corrosion potentials ( $E_{\text{corr}}$  values) for Alloy C-276 (UNS N10276) and Type 304 stainless steel (UNS S30400) are usually similar, these curves indicate that Alloy C-276 is more resistant to initiation and propagation of localized corrosion than Type 304 stainless steel.

## 8. Precision and Bias

8.1 A standard polarization plot, based on the potentiodynamic data from five different laboratories, has been prepared. The plot has been separated into the forward (Fig. 3) and reverse (Fig. 4) scans for clarity. These plots show the mean values and a range of  $\pm 2$  standard deviations.

8.2 The spread in data obtained from a number of laboratories and used in the preparation of the standard plot (Fig. 3 and Fig. 4) demonstrates the reproducibility that is possible when a standard procedure is followed. An investigator's data should fall within the range of  $\pm 2$  standard deviations since this includes 95 % of all data provided random variations are the only source of error. No information is available on the repeatability when one laboratory conducts several identical tests. Crevice corrosion under gaskets may lead to erroneous results.

8.3 When testing iron-, nickel-, and cobalt-based alloys according to this test method, the repeatability and reproducibility would be expected to be similar to the standard material. However, no data is currently available on other alloys.

8.4 This test method, when conducted in accordance with the procedures described herein, ranks some iron-, nickel-, and cobalt-based alloys relative to their resistance to crevice and pitting corrosion in chloride-containing environments, such as seawater. The test method will not necessarily rank materials properly in environments which are significantly different from

<sup>7</sup> *Reagent Chemicals, American Chemical Society Specifications*, Am. Chemical Soc., 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."

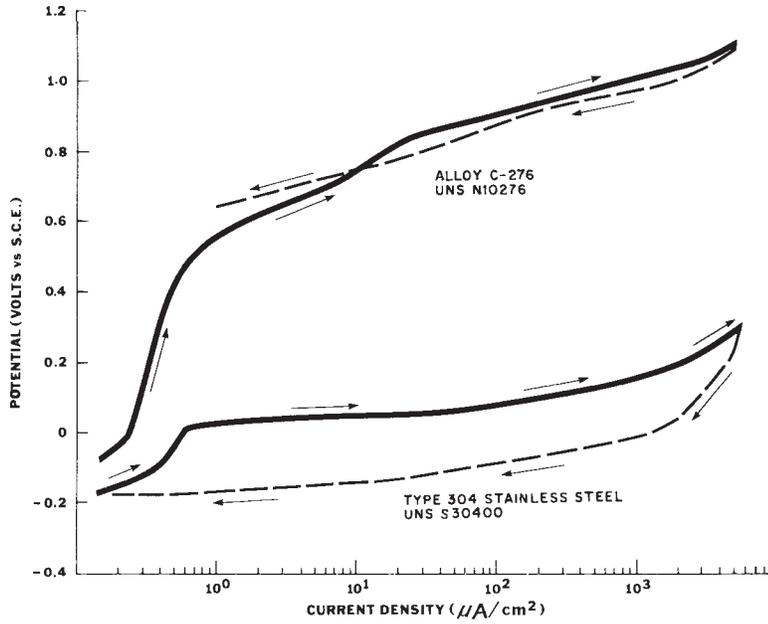


FIG. 2 Representative Cyclic Potentiodynamic Polarization Curves

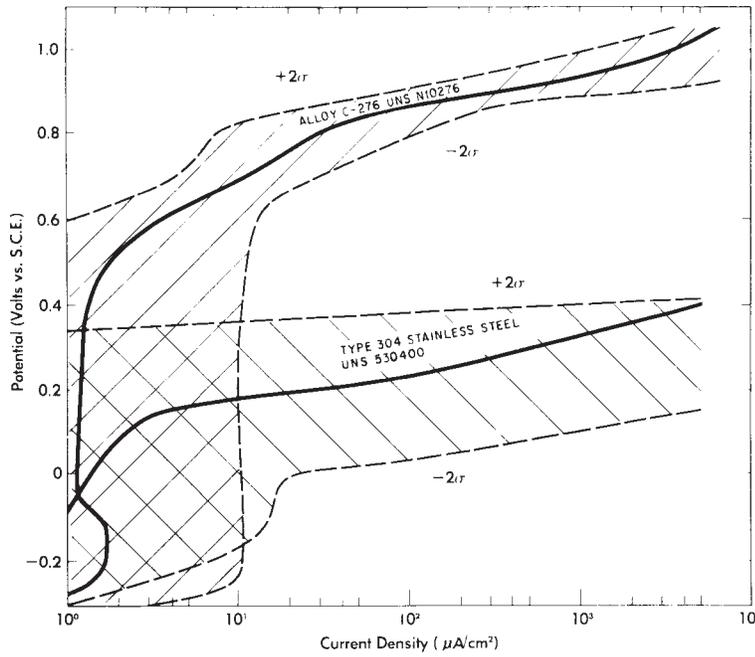


FIG. 3 Standard Potentiodynamic Polarization Plot (Forward Scan)

aqueous, ambient temperature aerated sodium chloride. For other alloys tested in other electrolytes, there is currently no information.

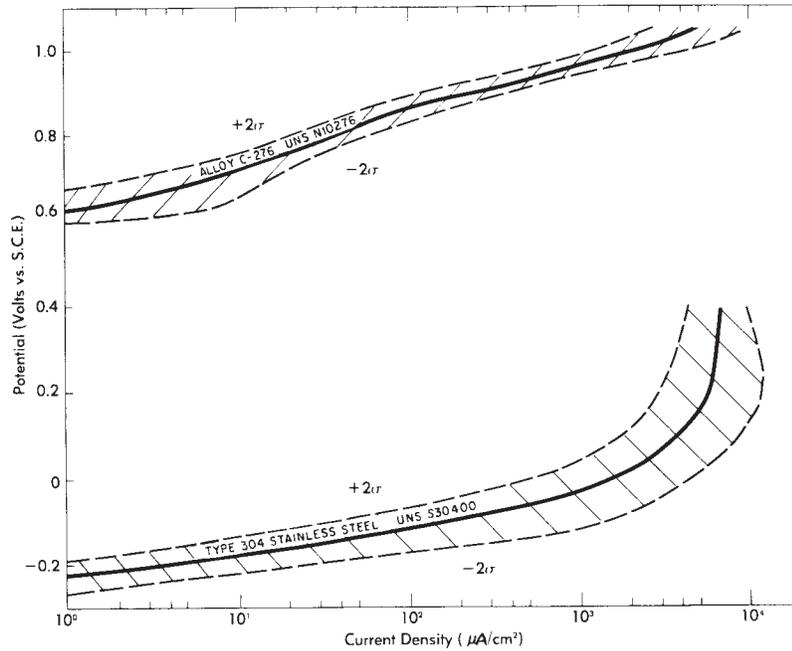


FIG. 4 Standard Potentiodynamic Polarization Plot (Reverse Scan)

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