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# Standard Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements<sup>1</sup>

This standard is issued under the fixed designation G 5; 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 describes an experimental procedure for checking experimental technique and instrumentation. If followed, this test method will provide repeatable potentiostatic and potentiodynamic anodic polarization measurements that will reproduce data determined by others at other times and in other laboratories provided all laboratories are testing reference samples from the same lot of Type 430 stainless steel.

1.2 Values stated in SI units are to be regarded as the standard. Inch-pound units 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 Document

2.1 ASTM Standard:

G 3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing<sup>2</sup>

### 3. Significance and Use

3.1 The availability of a standard procedure, standard material, and a standard plot should make it easy for an investigator to check his techniques. This should lead to polarization curves in the literature which can be compared with confidence.

3.2 Samples of a standard ferritic Type 430 stainless steel (UNS S43000) used in obtaining standard reference plot are available for those who wish to check their own test procedure and equipment.<sup>3</sup>

3.3 Standard potentiostatic and potentiodynamic polarization plots are supplied with the purchase of the reference material. These reference data are based on the results from different laboratories that followed the standard procedure, using that material in 1.0 N H<sub>2</sub>SO<sub>4</sub>. Maximum and minimum current values are shown at each potential to indicate the acceptable range of values. 3.4 This test method may not be appropriate for polarization testing of all materials or in all environments.

3.5 This test method is intended for use in evaluating the accuracy of a given electrochemical test apparatus, not for use in evaluating materials performance. Therefore, the use of the plots in Figs. 4 and 5 or Appendix X2 is not recommended to evaluate alloys other than Type 430, or lots of Type 430 other than those available through ASTM. The use of the data in this test method in this manner is beyond the scope and intended use of this test method. Users of this test method are advised to evaluate test results relative to the scatter bands corresponding to the particular lot of Type 430 stainless steel that was tested.

#### 4. Apparatus

4.1 The test cell should be constructed to allow the following items to be inserted into the solution chamber: the test electrode, two auxiliary electrodes, a Luggin capillary with salt-bridge connection to the reference electrode, inlet and outlet for an inert gas, and a thermometer. The test cell shall be constructed of materials that will not corrode, deteriorate, or otherwise contaminate the test solution.

Note 1—Borosilicate glass and TFE-fluorocarbon have been found suitable.

-4.1.1 A suitable cell is shown in Fig. 1 (1).<sup>4</sup> A 1-L, roundbottom flask has been modified by the addition of various necks 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, and the probe tip can be easily adjusted to bring it in close proximity with the working electrode.

4.2 Potentiostat (Note 2):

4.2.1 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 from -0.6 to 1.6 V and an anodic current output range from 1.0 to  $10^5 \,\mu$ A.

4.3 Potential-Measuring Instruments (Note 2):

4.3.1 The potential-measuring circuit should have a high input impedance on the order of  $10^{11}$  to  $10^{14} \Omega$  to minimize current drawn from the system during measurements. Such circuits are provided with most potentiostats. Instruments should have sufficient sensitivity and accuracy to detect a change of 1.0 mV over a potential range between -0.6 and 1.6 V.

<sup>&</sup>lt;sup>4</sup> The boldface numbers in parentheses refer to the list of references at the end of this test method.



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

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<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 03.02.

<sup>&</sup>lt;sup>3</sup> These standard samples are available from ASTM Headquarters. Generally, one sample can be repolished and reused for many runs. This procedure is suggested to conserve the available material. Order PCN 12-700050-00.



FIG. 1 Schematic Diagram of Polarization Cell (1)

#### 4.4 Current-Measuring Instruments (Note 2):

4.4.1 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  $10^5 \ \mu A$  for a Type 430 stainless steel (UNS S43000) specimen with a surface area of approximately 5 cm<sup>2</sup>.

4.5 Anodic Polarization Circuit:

4.5.1 A schematic potentiostatic anodic polarization wiring diagram (2) is illustrated in Fig. 2.

4.5.2 A scanning potentiostat is used for potentiodynamic measurements. For such measurements the potentiostat shall be capable of automatically varying the potential at a constant rate between two preset potentials. A record of the potential and current is plotted continuously using such instruments as an X-Y recorder and a logarithmic converter incorporated into the circuit shown in Fig. 2. Some potentiostats have an output of the logarithm of the current as a voltage, which allows direct plotting of the potential log current curve using an X-Y recorder.

NOTE 2—The instrumental requirements are based upon values typical of the instruments in 15 laboratories.

# 4.6 Electrode Holder (1):

4.6.1 The auxiliary and working electrodes are mounted in the type of holder shown in Fig. 3. A longer holder is required for the working electrode than for the auxiliary electrode. A leak-proof assembly is obtained by the proper compression fit between the electrode and a TFE-fluorocarbon gasket. (Too much pressure may cause shielding of the electrode or breakage of the glass holder, and too little pressure may cause leakage and subsequently crevice corrosion which may affect the test results.)

4.7 Electrodes:

4.7.1 Working Electrode, prepared from a 12.7-mm  $(\frac{1}{2}-in.)$  length of 9.5-mm  $(\frac{3}{8}-in.)$  diameter rod stock. Each electrode is drilled, tapped, and mounted in the manner discussed in 4.6.1.

Note: 3—If specimen forms are used other than those called for by this test method, for example, flat sheet specimen, care should be taken since it was shown that crevices may be introduced which can lead to erroneous results (see Fig. X1.1).

4.7.1.1 The standard AISI Type 430 stainless steel (UNS

S43000) should be used if one wishes to reproduce a standard reference plot. This material is prepared from a single heat of metal that is mill-annealed for  $\frac{1}{2}$  h at 815°C (1500°F) and air cooled. The chemical composition of the standard stainless steel is supplied with the purchase of reference material.

4.7.2 Auxiliary Electrodes:

4.7.2.1 Two platinum auxiliary electrodes are prepared from high-purity rod stock. Each electrode is drilled, tapped, and mounted with a TFE-fluorocarbon gasket in the same manner as the working electrode. A large platinum sheet sealed into a glass holder is also acceptable.



FIG. 2 Schematic Potentiostatic Anodic Polarization Wiring Diagram (2)



FIG. 3 Specimen Mounted on Electrode Holder