

Standard Reference Test Method for Making Potentiodynamic Anodic Polarization Measurements¹

This standard is issued under the fixed designation G5; 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 test method covers an experimental procedure for checking experimental technique and instrumentation. If followed, this test method will provide repeatable 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 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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:²

- E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
- E1338 Guide for Identification of Metals and Alloys in Computerized Material Property Databases
- G3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing
- G107 Guide for Formats for Collection and Compilation of Corrosion Data for Metals for Computerized Database Input

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.³

3.3 Standard potentiodynamic polarization plots are shown for a lot of material originally purchased in 1992. This test method is not applicable for standard material purchased before 1992. These reference data are based on the results from different laboratories that followed the standard procedure, using that material in $1.0 N H_2 SO_4$. The four sigma probability bands for current density 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 Fig. 1 is not recommended to evaluate alloys other than Type 430, or lots of Type 430 other than those available through Metal Samples. 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.

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

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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.

³ These standard samples are available from Metal Samples, 152 Metal Samples Rd., Mumford, AL 36268. Generally, one sample can be repolished and reused for many runs. This procedure is suggested to conserve the available material.

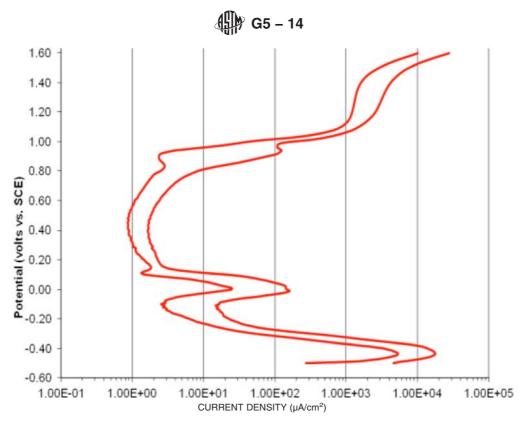
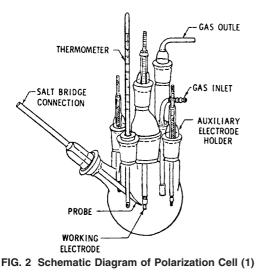


FIG. 1 Typical Standard Potentiodynamic Anodic Polarization Plot

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

4.1.1 A suitable cell is shown in Fig. 2 (1).⁴ A 1-L,



round-bottom 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 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. Potentiostats that scan potential by making frequent potential steps of less than 1.0 mV and those that make continuous analog potential sweeps are both suitable for this test method, providing that they can achieve the required potential scan rate.

4.4 Current-Measuring Instruments (Note 2):

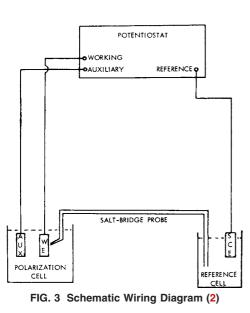
4.4.1 An instrument that is capable of measuring a current accurately to within 1 % of the absolute value over a current range between 1.0 and $10^5 \mu A$ for a Type 430 stainless steel (UNS S43000) specimen with a surface area of approximately 5 cm².

4.5 Anodic Polarization Circuit:

4.5.1 A schematic wiring diagram (2) is illustrated in Fig. 3.

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

⁴ The boldface numbers in parentheses refer to the list of references at the end of this test method.



current is plotted continuously using such instruments as an X-Y recorder and a logarithmic converter incorporated into the circuit shown in Fig. 3. 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 the laboratories that participated in the round robin.

4.6 *Electrode Holder* (1):

4.6.1 The auxiliary and working electrodes are mounted in the type of holder shown in Fig. 4. A longer holder is required

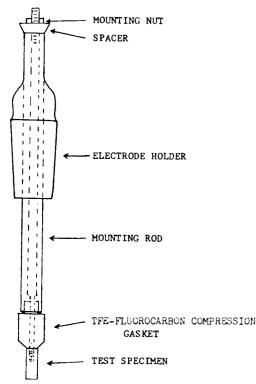


FIG. 4 Specimen Mounted on Electrode Holder

for the working electrode than for the auxiliary electrode. A leakproof 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 length of 9.5-mm 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 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.

4.7.2.2 A platinized surface may be utilized because of the increased surface area. This may be accomplished by cleaning the surface in hot aqua regia (3 parts concentrated HCl and 1 part concentrated HNO₃), washing, and then drying. Both electrodes are platinized by immersing them in a solution of 3 % platinic chloride and 0.02 % lead acetate and electrolyzing at a current density of 40 to 50 mA/cm² for 4 or 5 min (1, 3). The polarity is reversed every minute. Occluded chloride is removed by electrolyzing in a dilute (10 %) sulfuric acid solution for several minutes with a reversal in polarity every minute. Electrodes are rinsed thoroughly and stored in distilled water until ready for use. Since certain ions can poison these electrodes, periodic checks of platinized platinum potentials against a known reference electrode should be made.

4.7.2.3 Alternatively, graphite auxiliary electrodes can be used, but material retained by the graphite may contaminate subsequent experiments. This contamination can be minimized by using high-density graphite or avoided by routinely replacing the graphite electrode.

4.7.3 *Reference Electrode* (4):

4.7.3.1 A saturated calomel electrode with a controlled rate of leakage (about 3 μ L/h) is recommended. This type of electrode is durable, reliable, and commercially available. Precautions shall 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. For other alloy-electrolyte combinations a different reference electrode may be preferred in order to avoid contamination of the reference electrode or the electrolyte.