Designation: G5 – 14

Standard Reference Test Method for Making Potentiodynamic Anodic Polarization Measurements[1]

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_2SO_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.

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[1] This 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.

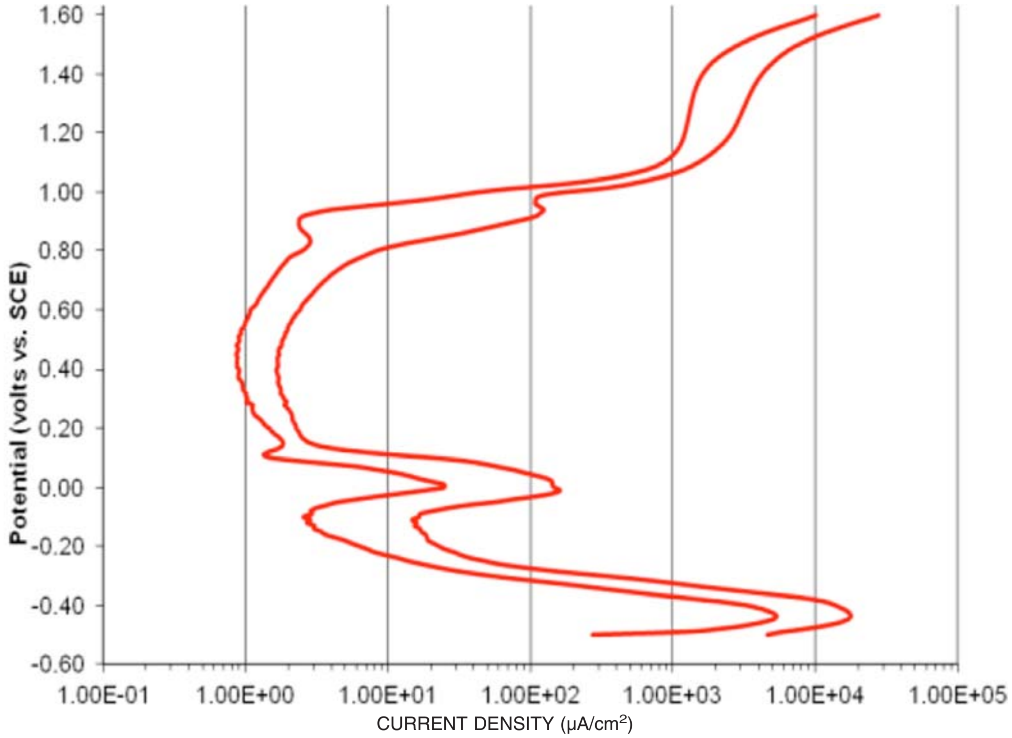

FIG. 1 Typical Standard Potentiodynamic Anodic Polarization Plot

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.

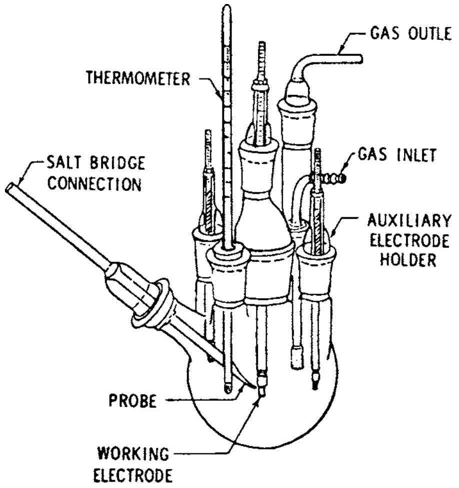

FIG. 2 Schematic Diagram of Polarization Cell (1)

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 µA.

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} Ω 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 µA for a Type 430 stainless steel (UNS S43000) specimen with a surface area of approximately 5 cm^2.

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

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

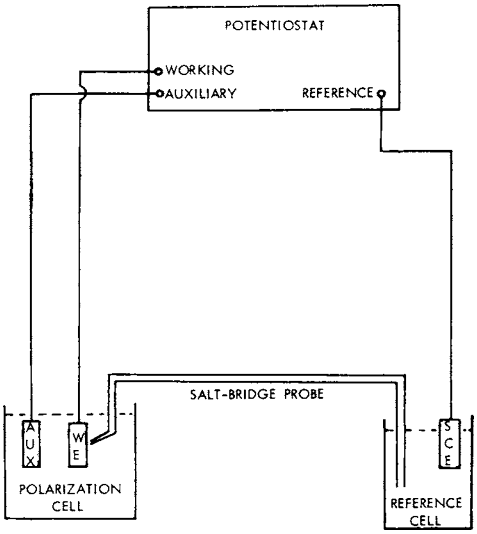

FIG. 3 Schematic Wiring Diagram (2)

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 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.)

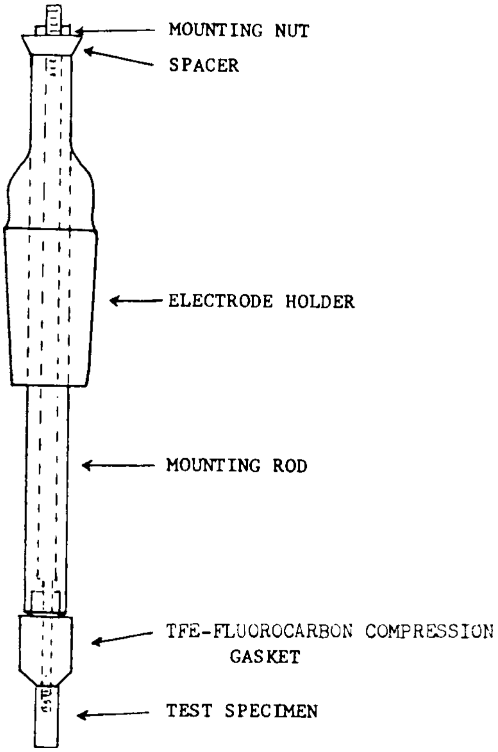

FIG. 4 Specimen Mounted on Electrode Holder

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 ½ 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 HNO_3), 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/cm^2 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 µL/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.

4.7.3.2 Alternatively, a saturated calomel electrode utilizing a semipermeable membrane or porous plug tip may be used. These may require special care.

5. Experimental Procedure

5.1 Prepare 1 L of 1.0 *N* H_2SO_4 from A.C.S. reagent grade acid and distilled water, for example, by using 27.8 mL of 98 % H_2SO_4 /L of solution. Transfer 900 mL of solution to the clean polarization cell.

5.2 Place the platinized auxiliary electrodes, salt-bridge probe, and other components in the test cell and temporarily close the center opening with a glass stopper. Fill the salt bridge with test solution.

NOTE 4—When using a controlled leakage salt bridge, the levels of the solution in the reference and polarization cells should be the same to avoid siphoning. If this is impossible, a closed solution-wet (not greased) stopcock can be used in the salt bridge to eliminate siphoning, or a semipermeable membrane or porous plug tip may be used on the salt bridge.

5.3 Bring the temperature of the solution to 30 ± 1°C by immersing the test cell in a controlled-temperature water bath or by other convenient means.

5.4 Reduce oxygen levels in solution prior to immersion of the test specimen. This may be accomplished by bubbling an oxygen-free gas such as hydrogen, argon, or nitrogen at a rate of 150 cm^3/min for a minimum of ½ h.

5.5 Prepare the working electrode surface within 1 h of the experiment. Wet grind with 240-grit SiC paper, wet polish with 600-grit SiC paper until previous coarse scratches are removed, rinse, and dry. (Drilled and tapped specimens can be threaded onto an electrode holder rod and secured in a lathe or electric drill for this operation.)

5.6 Determine the surface area by measuring all dimensions to the nearest 0.01 mm, subtracting the area under the gasket (usually 0.20 to 0.25 cm^2).

5.7 Mount the specimen on the electrode holder as described in 4.6.1. Tighten the assembly by holding the upper end of the mounting rod in a vise or clamp while tightening the mounting nut until the gasket is properly compressed.

5.8 Degrease the specimen just prior to immersion and then rinse in distilled water.

5.9 Transfer the specimen to the test cell and adjust the salt-bridge probe tip so it is about 2 mm or 2 times the tip diameter, whichever is larger from the specimen electrode.

5.10 Record the open-circuit specimen potential, that is, the corrosion potential, after 55 min immersion. If platinum counter electrodes and hydrogen gas are used, record the platinum potential 50 min after immersion of the specimen.

5.11 *Potential Scan:*

5.11.1 Start the potential scan 1 h after specimen immersion, beginning at the corrosion potential (E_{corr}). Proceed through +1.60 V versus saturated calomel electrode (SCE) (active to noble).

5.11.2 Use a potentiodynamic potential sweep rate of 0.6 V/h (±5 %) recording the current continuously with change in potential from the corrosion potential to +1.6 V SCE.

5.12 Plot anodic polarization data semilogarithmically in accordance with Practice G3, (potential-ordinate, current density-abscissa).

6. Standard Reference Plots and Compliance Limits

6.1 A standard polarization plot prepared from the interlaboratory testing program is shown in Fig. 1. See Research Report RR:G01-1026.[5] The confidence bands were calculated by determining logarithmic average of the current densities at each potential and plotting the current density limits at four logarithmic standard deviations on either side of the logarithmic average. The average corrosion potential was -0.52 V, and the average platinized platinum/hydrogen potential was -0.26 V versus SCE reference electrode.

6.2 To judge compliance with this test method, the current density at four potentials shall be measured and compared to the limits shown in Table 1. The probability that test results would fall outside of these limits while still being in compliance with this method is less than 0.001.

6.3 Typical deviations from the standard plot are shown and discussed in Appendix X1. Reference to this discussion may be helpful in determining the reasons for differences between an experimental curve and the standard plot.

7. Precision and Bias[5]

7.1 The precision of the procedure provided in this reference test method has been determined by an interlaboratory test program. This program initially had eight laboratories participating, but three laboratories were eventually excluded because of problems with their procedures and results. Of the remaining five laboratories, one laboratory did not achieve sufficiently reproducible results at the -0.450 and 0.000 V potentials during one run, so that the results from this run results were also excluded. The interlaboratory program was designed to have each participating laboratory run four replicate tests with the standard Type 430 stainless steel specimens. The current density results at the following potentials were chosen as critical points for evaluation: -0.450, -0.100, 0.000, 0.400 and 1.300 V versus the SCE electrode. The current densities were converted to base 10 logarithm values, and the Practice E691 procedure was used to evaluate the data at these potentials.

NOTE 5—The use of logarithmic conversion assumes that the error

TABLE 1 Compliance Limits for Current Densities (μA/cm^2) at Cited Potentials for Type 430 Stainless Steel in G5 Polarization Tests

Potential Volts (versus SCE)	Min	Max
-0.450	5160	13.860
-0.100	2.16	15.60
0.000	25.8a	134.8
+0.400	0.883	1.669

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:G01-1026. Contact ASTM Customer Service at service@astm.org.

distribution in the measured current densities is best fitted by a log normal distribution.

7.2 Repeatability refers to the agreement that occurs when identical specimens are run sequentially with the same operator using the same procedure and equipment. In this case, two values are reported to characterize the repeatability, the repeatability standard deviation, s_r, and the repeatability, r, which is 2.8 s_r. Reproducibility refers to the agreement that occurs when several laboratories run the procedure using identical specimens with the same procedure. Two values are reported to characterize the reproducibility, the reproducibility standard deviation, s_R, and the reproducibility, R, which is 2.8 s_R. These values, together with the logarithmic average current densities at each potential and their antilogarithmic values are given in Table 2.

7.3 There is no bias in the current densities determined by this reference test method because the potentiodynamic current densities measured at the critical potentials in this method are determined only in terms of this test method.

TABLE 2 Precision Values for Current Densities at the Critical Specimen Potentials

NOTE 1—The logarithmic values are reported in log (A/cm^2). The average current densities are reported in µA/cm^2. All specimen potentials are expressed in V versus the SCE reference electrode. The degrees of freedom for the standard deviations in this table is 14.

Potential	Log i_{ave}	$s_{r(log)}$	$r_{(log)}$	$s_{R(log)}$	$R_{(log)}$	$i_{ave(log)}$
-0.450	-2.0726	0.0256	0.0717	0.0518	0.1450	8461
-0.100	-5.1950	0.0535	0.1500	0.0868	0.2754	6.38
0.000	-4.2298	0.0231	0.0648	0.0868	0.2431	58.9
0.400	-5.9157	0.0219	0.0733	0.0334	0.0934	1.214
1.300	-2.6797	0.0334	0.0935	0.0505	0.1414	2091

8. Keywords

8.1 anodic polarization; electrochemical testing; pitting; potentiodynamic; sulfuric acid; Type 430 stainless steel

APPENDIXES

(Nonmandatory Information)

X1. DEVIATIONS FROM STANDARD POLARIZATION PLOTS

X1.1 High Passive Current Densities (Crevice Effect)

X1.1.1 Examples of passive current densities which are greater than those for a standard potentiostatic plot are shown in Fig. X1.1. This effect is attributable to a crevice between the specimen and mounting material (5). The crevice may be the result of the mounting technique or the material used for mounting.

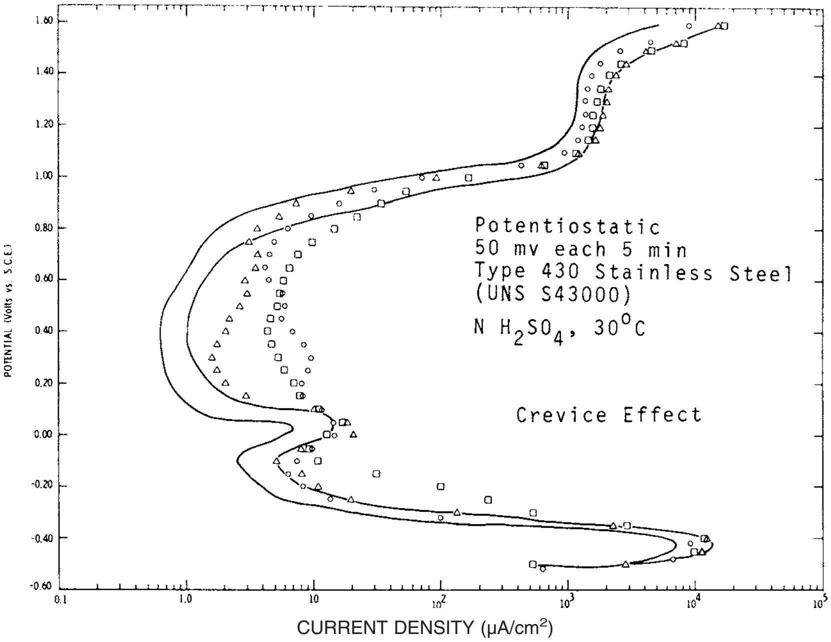

FIG. X1.1 Crevice Effect During Potentiostatic Anodic Polarization

X1.1.2 The potential drop along the narrow path of the electrolyte within the crevice between the specimen and the mounting material prevents this area from passivating. Although the face of the specimen passivates, the high current density associated with the active crevice contributes to an increase in the measured current density. Specimen electrodes for polarization measurements must be mounted without crevice sites to avoid such erroneous passive current densities.

X1.1.3 The curves in this appendix were developed by potentiostatic stepping rather than by potentiodynamic sweep, and were developed on a different lot of material than is currently available as standard specimens, so direct comparison of curves contained in the figures in this appendix with potentiodynamic data generated on current material should not be made. Instead, these figures should be considered as illustrative of trends only.

X1.2 Low Passive Current Densities (Instrumental Effect)

X1.2.1 The low passive current densities shown in Fig. X1.2 are undoubtedly the result of instrumental problems. This effect can be eliminated by calibrating the current over the entire range of interest before conducting an experiment.

X1.3 Cathodic Currents During Anodic Polarization (Oxygen Effect)

X1.3.1 The "negative loop" at potentials between –0.350 V and –0.050 V, shown by dashed lines in Fig. X1.3, occurs when the total cathodic current exceeds the total anodic current. Such results are characteristic of oxygen being present in the solution (**6**). This effect can be anticipated if the recorded platinum potential is considerably more noble than –0.26 V. The gas purge should remove oxygen from the system, but there may be an air leak or the purge gas may be contaminated with oxygen. It is necessary to take extreme care in the design of glassware equipment and to ensure a high order of purity in the gas that is used to avoid oxygen contamination.

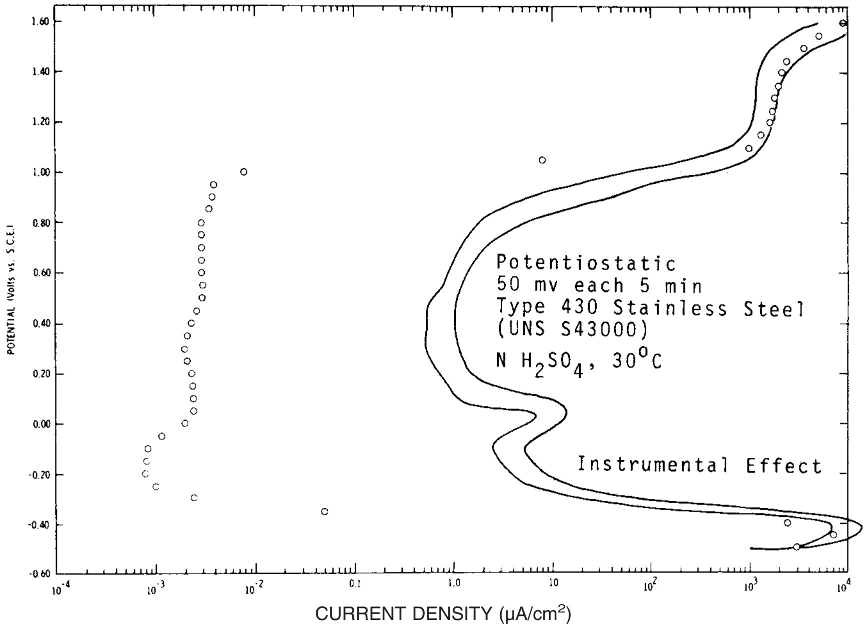

FIG. X1.2 Instrumental Effect During Potentiostatic Anodic Polarization

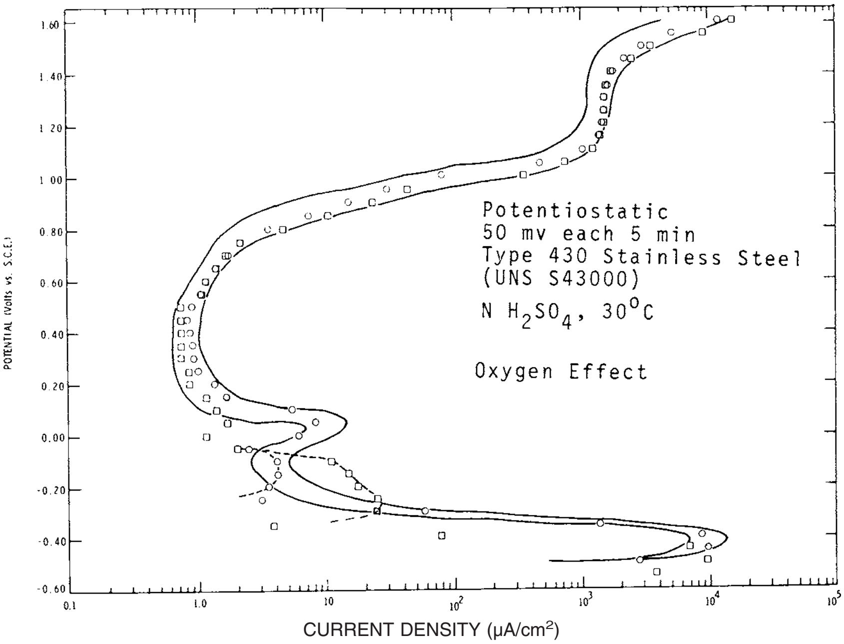

FIG. X1.3 Oxygen Effect During Potentiostatic Anodic Polarization

X2. RECOMMENDED STANDARD DATA FIELDS FOR COMPUTERIZATION OF DATA FROM TEST METHOD G5

X2.1 In order to encourage uniformity in building computerized corrosion databases and facilitate data comparison and data interchange, it is appropriate to provide recommended standard formats for the inclusion of specific types of test data in such databases. This also has the important effect of encouraging the builders of databases to include sufficiently complete information so that comparisons among individual sources may be made with assurance that the similarities or differences, or both, in the test procedures and conditions are covered therein.

X2.2 Table X2.1 is a recommended standard format for the computerization of potentiostatic and potentiodynamic anodic polarization measurements according to Test Method G5. There are three columns of information in Table X2.1.

X2.2.1 *Field Number*—This is a reference number for ease of dealing with the individual fields within this format guideline. It has no permanent value and does not become part of the database itself.

X2.2.2 *Field Name and Description*—This is the complete name of the field, descriptive of the element of information that would be included in this field of the database.

X2.2.3 *Category Sets, Values or Units*—This is a listing of the types of information which would be included in the field, or, in the case of properties or other numeric fields, the units in which the numbers are expressed. Category sets are closed (that is, complete) sets containing all possible (or acceptable) inputs to the field. Values are representative sets, listing sample (but not necessarily all acceptable) inputs to the field.

X2.3 The fields or elements of information included in this format are those recommended to provide sufficiently complete information that users may be confident of their ability to compare sets of data from individual databases and to make the database useful to a relatively broad range of users.

X2.4 It is recognized that many databases are prepared for very specific applications, and individual database builders may elect to omit certain pieces of information considered to be of no value for that specific application. However, there are a certain minimum number of fields considered essential to any database, without which the user will not have sufficient information to reasonably interpret the data. In the recommended standard format, these fields are marked with asterisks.

X2.5 The presentation of this format does not represent a requirement that all of the elements of information included in the recommendation must be included in every database. Rather it is a guide as to those elements that are likely to be useful to at least some users of most databases. It is understood that not all of the elements of information recommended for inclusion will be available for all databases; that fact should not discourage database builders and users from proceeding so long as the minimum basic information is included (the items noted by the asterisks).

X2.6 It is recognized that in some individual cases, additional elements of information of value to users of a database may be available. In those cases, database builders are encouraged to include them as well as the elements in the recommended format. Guidelines for formats for additional elements are given in Guide G107.

X2.7 This format is for potentiostatic and potentiodynamic anodic polarization measurements generated by Test Method

TABLE X2.1 Recommended Standard Data Fields for Computerization of Data from Test Method G5

Field No.	Field Name and Description	Category Sets Values or Units
	Test Identification	
1*	ASTM standard test method	Test Method G5
2	Type of test	anodic polarization
3	Date test started	yyyymmdd
4	Internal laboratory reference number	alphanumeric string
	Test Apparatus	
5*	Cell similar to Fig. 2 in standard	Y/N
6*	If "No" in 5, describe	alphanumeric string
7	Potentiostat potential stability from preset value	mV, ±
8	Potentiostat potential range	V/V
9	Impedance of potential measuring circuit	ohm
10	Accuracy of current measurement	percent of absolute value
11*	Electrode holder similar to Fig. 2	Y/N
12*	If "No" in 11, describe	alphanumeric string
13*	Working electrode	(1) 12.7 long, 9.5 mm rod (2) other
14*	If "Other" in 13, describe	alphanumeric string
15*	Auxiliary electrode	(1) platinum (2) platinized (3) graphite (4) other
16*	If "Other" in 15, describe	alphanumeric string
17*	Reference electrode	(1) saturated calomel (2) Ag/AgCl (3) $Cu/CuSO_4$ (4) other
18*	If "Other" in 17, describe	alphanumeric string
	Test Specimen	
19*	Standard material (UNS S43000)	Y/N
20*	If "No" in 19, give UNS No.	alphanumeric string
21	Surface area	x.xx cm^2
22	Surfaces wet ground and polished (240/600 grit SiC), degreased	Y/N
23	If "No" in 22, describe the alternate	alphanumeric string
	Test Environment	
24*	Standard environment (1 N H_2SO_4, deaerated by bubbling hydrogen, argon, or nitrogen prior to specimen exposure).	Y/N
25*	If "No" in 24, describe	alphanumeric string
26	Standard cell volume (900 mL)	Y/N
27	If "No" in 26, then give volume	mL
37	Low passive current density attributed to instrumental problems	Y/N
38	Negative loop attributed to oxygen in solution	Y/N

* Denotes essential information.

G5. It does not include the recommended material descriptors or the presentation of other specific types of test data (such as mechanical property data). These items are covered in Guide E1338 and by separate formats developed for reporting other material property data.

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(6) Greene, N. D., "Effect of Oxygen on the Active-Passive Behavior of Stainless Steel," *Journal of the Electrochemical Society*, JESOA, Vol 107, 1960, p. 457.

(7) "The Reproducibility of Potentiostatic and Potentiodynamic Anodic Polarization Measurements," ASTM Subcommittee G-1/XI, Section I, Interlaboratory Testing Program, June, 1967. Available from ASTM Headquarters as RR:G01-1000.

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