

Designation: G 5 – 94 (Reapproved 1999)^{∈1}

Standard Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements¹

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 (ϵ) indicates an editorial change since the last revision or reapproval.

 ϵ^1 Note—Footnote 4 was updated editorially August 2002.

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 Documents

2.1 ASTM Standards:

E 1338 Guide for the Identification of Metals and Alloys in Computerized Material Property Databases²

- G 3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing³
- G 107 Guide for Formats for Collection and Compilation of Corrosion Data for Metals for Computerized Database Input³

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.⁴

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 H₂SO₄. 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. 1 and 2 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.

 $\ensuremath{\operatorname{NOTE}}$ 1—Borosilicate glass and TFE-fluorocarbon have been found suitable.

4.1.1 A suitable cell is shown in Fig. 3 (1).⁵ A 1-L, roundbottom flask has been modified by the addition of various

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¹ 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.

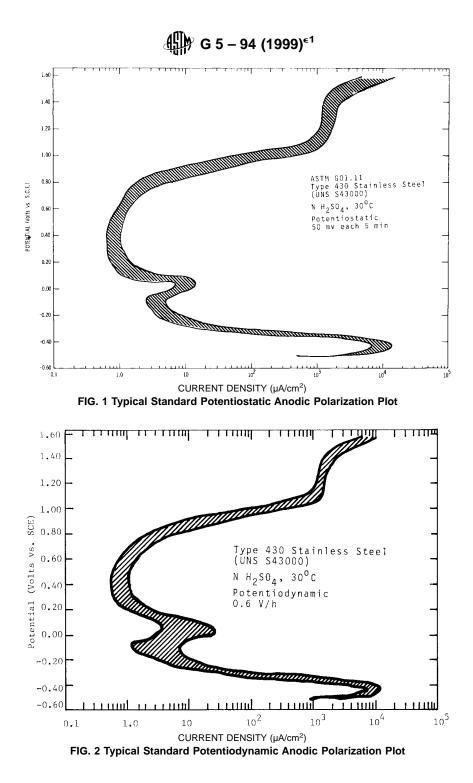
Current edition approved March 15, 1994. Published May 1994. Originally published as G 5 - 69. Last previous edition G 5 - 87.

² Annual Book of ASTM Standards, Vol 14.01.

³ Annual Book of ASTM Standards, Vol 03.02.

⁴ These standard samples are available from Metal Samples, P.O. Box 8, Mumford, AL 36268. 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.

⁵ The boldface numbers in parentheses refer to the list of references at the end of this test method.



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.

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

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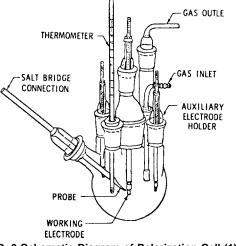


FIG. 3 Schematic Diagram of Polarization Cell (1)

(UNS S43000) specimen with a surface area of approximately 5 cm^2 .

4.5 Anodic Polarization Circuit:

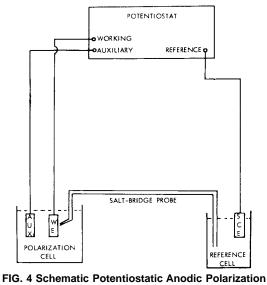
4.5.1 A schematic potentiostatic anodic polarization wiring diagram (2) is illustrated in Fig. 4.

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. 4. 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. 5. A longer holder is required



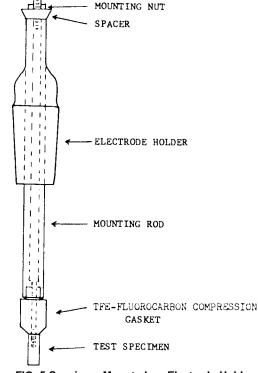


FIG. 5 Specimen Mounted on Electrode Holder

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 break-age 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.

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

FIG. 4 Schematic Potentiostatic Anodic Polarization Wiring Diagram (2)

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.

4.7.3.2 Alternatively, a saturated calomel electrode utilizing a semi-permeable 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 semi-permeable membrane or porous plug tip may be used on the salt bridge.

5.3 Bring the temperature of the solution to $30 \pm 1^{\circ}$ 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 cm³/min for a minimum of $\frac{1}{2}$ 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 or step 1 h after specimen immersion, beginning at the corrosion potential ($E_{\rm corr}$) for potentiodynamic measurements and the nearest 50-mV increment above $E_{\rm corr}$ for the potentiostatic measurements. Proceed through + 1.60 V versus saturated calomel electrode (SCE) (active to noble).

5.11.2 In the potentiostatic method, use a potentiostatic potential step rate of 50 mV every 5 min, recording the current at the end of each 5-min period at potential. These steps are repeated until a potential of + 1.6 V SCE is reached.

5.11.3 In the potentiodynamic method, 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 on semilogarithmic paper in accordance with Practice G 3, (potential-ordinate, current density-abscissa). If a potentiostat with a logarithmic converter is used, this plot can be produced directly during the measurement.

6. Standard Reference Plots

6.1 Standard polarization plots prepared from data obtained by following the standard procedure discussed in this test method are supplied with the purchase of reference material. Typical data are shown in Fig. 1 and Fig. 2 (5). The plots show a range of acceptable current density values at each potential. The average corrosion potential is -0.52 V, and the average platinized platinum potential is -0.26 V.

NOTE 5—The plots in Fig. 1 and Fig. 2 correspond to a lot of Type 430 stainless steel that is no longer available from ASTM (after July 1992). Figs. 1 and 2 are presented primarily for the discussion of precision and bias in Sections 6, 7, and Appendix X1. The scatter bands presented in Appendix X2 were developed from a round robin testing program on the lot of Type 430 stainless steel that is currently available from ASTM.

6.2 Typical deviations from the standard potentiostatic 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 plots.

6.3 The potentiodynamic standard curve shows good agreement with the potentiostatic standard curve determined at an equivalent overall polarization rate.

6.4 Differences in the size and placement of the scatter bands presented in Figs. 1 and 2 versus those in Appendix X2 are attributed to minor differences in the two heats of Type 430 stainless steel that were evaluated in separate round robins.

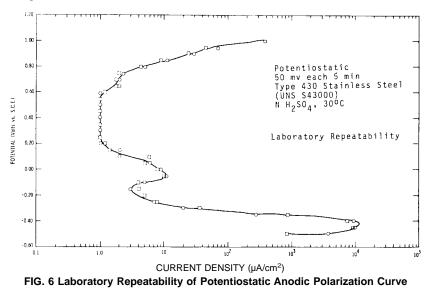
7. Precision and Bias

7.1 The repeatability of this test method is being developed. However, the repeatability on a previous interlaboratory test in which one material was run twice by one laboratory is shown in Fig. 6.

7.2 The reproducibility of this test method is being developed by interlaboratory testing. 7.3 There is no bias in this test method because the potentiodynamic curve is defined only in terms of this test method.

8. Keywords

8.1 anodic polarization; electrochemical testing; pitting; potentiodynamic; potentiostatic; 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 (6). The crevice may be the result of the mounting technique or the material used for mounting.

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.

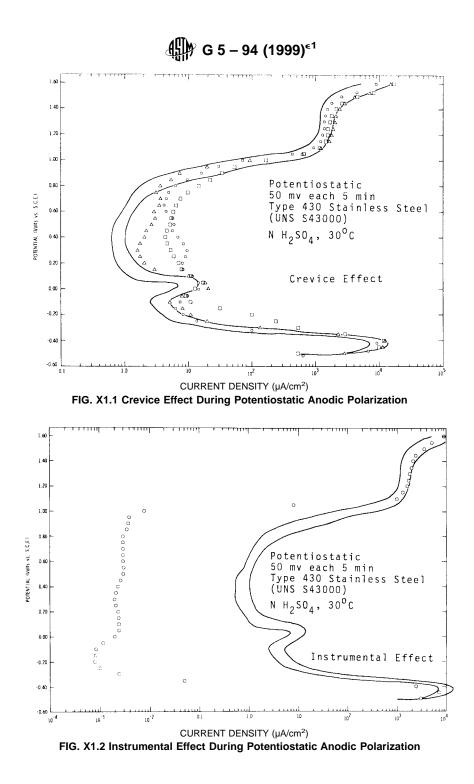
X1.2 Low Passive Current Densities (Instrumental Effect)

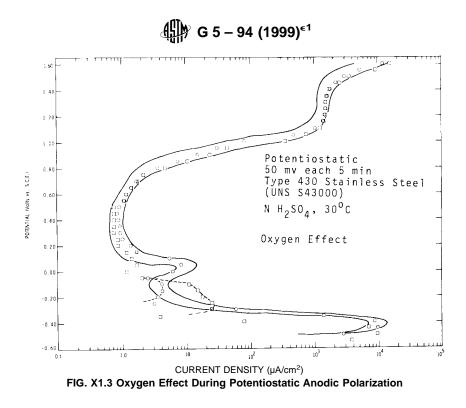
X1.2.1 The low passive current densities shown in Fig. X1.2 are undoubtedly the result of instrumental problems. This

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 (7). 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.





X2. STANDARD REFERENCE PLOTS FROM ROUND ROBIN TESTS OF THE LOT OF TYPE 430 STAINLESS STEEL

X2.1 Standard polarization plots prepared from data obtained by following the standard procedure discussed in this test method are supplied with the purchase of reference samples.⁶ All of the material available at any given time is from a lot taken from a single heat of Type 430 stainless steel bar stock. Whenever the available supply is exhausted, a new heat must be melted and the new samples qualified in a new round robin test program.

X2.2 The plots in Figs. 1 and 2 correspond to a lot of Type 430 stainless steel that is no longer available from ASTM (after

July 1992). The scatter bands presented in Fig. X2.1 and Fig. X2.2 and Table X2.1 were developed from a round robin testing program on the lot of Type 430 stainless that is currently available from ASTM.

X2.3 Fig. X2.1 and Table X2.1 summarize the round robin potentiodynamic and potentiostatic test results and define scatter bands for the new lot of Type 430 stainless steel. The plots and table show the range of current density values at each potential obtained by the laboratories that participated in the round robin to qualify the new lot of Type 430 stainless steel. Fig. X2.2 compares the scatter bands for the old and new lots of Type 430 stainless steel. The two lots are distinguished by the year of introduction, either 1987 or 1992.

⁶ Available from ASTM Headquarters. Order PCN 12-700050-00.

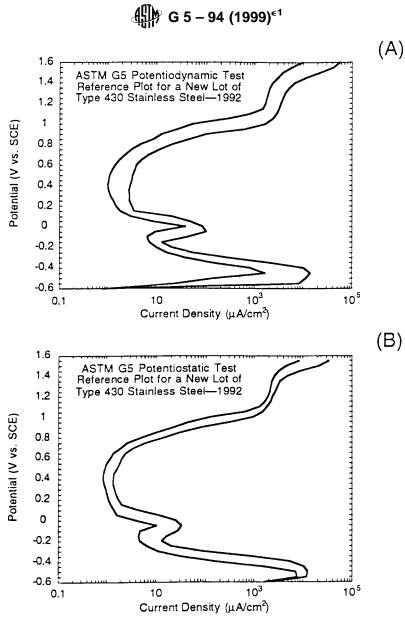


FIG. X2.1 Standard Potentiodynamic (A) and Potentiostatic (B) Polarization Plots for New Type 430 Stainless Steel Standard Introduced in 1992

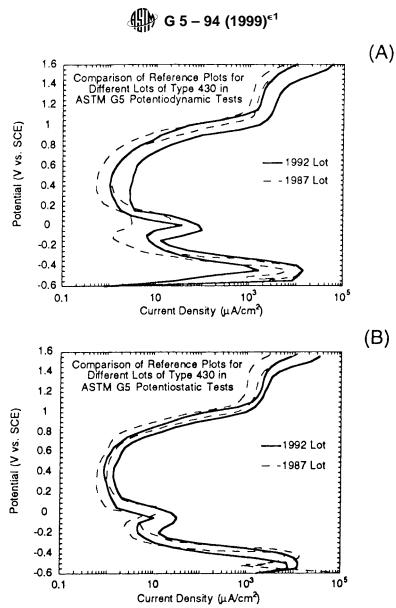


FIG. X2.2 Comparison of the Standard Reference Potentiodynamic (A) and Potentiostatic (B) Polarization Plots for the 1992 and the 1987 Lots of Type 430 Stainless Steel

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TABLE X2.1 Range of Current Densities (μA/cm²) at Cited Polarization Potentials for the 1992 Lot of Type 430 Stainless Steel in G5 Polarization Tests

Potential	Potentiodynamic		Potentiostatic	
Volts (v. SCE)	Min	Max	Min	Max
-0.600	0.76	0.76	1632	2247
-0.500	147	11451	714	12595
-0.400	810	10418	161	5453
-0.300	37.68	282	11.29	87.17
-0.200	10.02	20.23	4.77	13.09
-0.100	6.41	41.44	4.89	27.40
0.000	37.18	84.77	3.92	24.83
0.100	3.00	19.93	1.43	4.35
0.200	1.49	3.13	1.11	1.78
0.300	1.15	2.85	0.98	1.46
0.400	1.02	2.93	0.85	1.39
0.500	1.12	3.30	0.94	1.65
0.600	1.46	3.90	1.20	2.07
0.700	2.27	6.97	1.96	3.51
0.800	4.73	20.59	5.10	14.55
0.900	12.61	99.25	22.0	80.48
1.000	53.40	1628	126	954
1.100	781	2872	1300	1846
1.200	1692	3530	1923	2413
1.300	1925	4283	2176	3026
1.400	2397	6813	2619	4762
1.500	3814	22366	4570	21883
1.600	9775	57341		

X3. RECOMMENDED STANDARD DATA FIELDS FOR COMPUTERIZATION OF DATA FROM TEST METHOD G5

X3.1 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.

X3.2 Table X3.1 is a recommended standard format for the computerization of potentiostatic and potentiodynamic anodic polarization measurements according to Test Method G 5. There are three columns of information in Table X3.1.

X3.2.1 *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.

X3.2.2 *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.

X3.2.3 *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.

X3.3 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.

X3.4 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.

X3.5 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).

X3.6 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 G 107.

X3.7 This format is for potentiostatic and potentiodynamic anodic polarization measurements generated by Test Method

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TABLE X3.1 Recommended Standard Data Fields for Computerization of Data from Test Method G 5

Field Name and Description	Category Sets Values or Units			
Test Identification				
ASTM standard test method	Test Method G 5			
Type of test	anodic polarization			
Date test started	yyyymmdd			
Internal laboratory reference number	alphanumeric string			
Test Apparatus				
Cell similar to Fig. 1 in standard	Y/N			
If "No" in 5, describe	alphanumeric string			
Potentiostat potential stability from preset value	mV, ±			
Potentiostat potential range	V/V			
Impedance of potential measuring circuit	ohm			
Accuracy of current measurement	percent of absolute value			
Electrode holder similar to Fig. 3 in standard	Y/N			
If "No" in 11, describe	alphanumeric string			
Working electrode	(1) 12.7 long, 9.5 mm rod			
	(2) other			
	alphanumeric string			
Auxiliary electrode	(1) platinum			
	(2) platinized			
	(3) graphite			
If "Other" in A.C. describe	(4) other			
	alphanumeric string (1) saturated calomel			
Reference electrode				
	(2) Ag/AgCl (3) Cu/CuSO₄			
	(4) other			
If "Other" in 17 describe	alphanumeric string			
	Y/N			
	alphanumeric string x.xx cm ²			
	Y/N			
	f/IN			
	alphanumeric string			
	alphanumenc sung			
	N/A1			
deaerated by bubbling hydrogen, argon, or nitrogen prior to specimen	Y/N			
	alphanumeric string			
	Y/N			
	mL			
Low passive current density attributed	Y/N			
Negative loop attributed to oxygen in solution	Y/N			
	Test Identification ASTM standard test method Type of test Date test started Internal laboratory reference number Test Apparatus Cell similar to Fig. 1 in standard If "No" in 5, describe Potentiostat potential stability from preset value Potentiostat potential range Impedance of potential measuring circuit Accuracy of current measurement Electrode holder similar to Fig. 3 in standard If "No" in 11, describe Working electrode If "Other" in 13, describe Auxiliary electrode If "Other" in 17, describe Reference electrode If "Other" in 17, describe Standard material (UNS \$43000) If "No" in 19, give UNS No. Surface area Surfaces wet ground and polished (240/600 grit SiC), degreased If "No" in 22, describe the alternate Test Environment Standard environment (1 N H ₂ SO ₄ , deaerated by bubbling hydrogen, argon, or nitrogen prior to specimen exposure).<			

* Denotes essential information.

G 5. 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 E 1338 and by separate formats developed for reporting other material property data.

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