



Designation: G108 – 23

Standard Test Methods for Electrochemical Reactivation (EPR) for Detecting Sensitization of AISI Type 304 and 304L Stainless Steels¹

This standard is issued under the fixed designation G108; 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 These test methods cover a laboratory procedure for conducting an electrochemical reactivation (EPR) test on AISI Type 304 and 304L (UNS No. S30400 and S30403, respectively) stainless steels. These test methods can provide a nondestructive means of quantifying the degree of sensitization in Type 304 stainless steels (**1, 2, 3**).² These EPR test methods have found wide acceptance in studies of the effects of sensitization on intergranular corrosion and intergranular stress corrosion cracking behavior (see Terminology **G193**). The EPR technique has been successfully used to evaluate other stainless steels and nickel base alloys (**4**), but the test conditions and evaluation criteria used were modified in each case from those cited in the current test methods. This standard test covers two tests, (1) Test Method A or Single Loop, and (2) Test Method B or Double Loop.

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

¹ These test methods are under the jurisdiction of ASTM Committee G01 on Corrosion of Metals and are the direct responsibility of Subcommittee G01.11 on Electrochemical Measurements in Corrosion Testing.

Current edition approved Feb. 15, 2023. Published September 2023. Originally approved in 1992. Last previous edition approved in 2015 as G108 – 94 (2015). DOI: 10.1520/G0108-23.

² The boldface numbers in parentheses refer to a list of references at the end of this standard.

2. Referenced Documents

2.1 *ASTM Standards*:³

- A262 Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels
- D1193 Specification for Reagent Water
- E3 Guide for Preparation of Metallographic Specimens
- E7 Terminology Relating to Metallography
- E112 Test Methods for Determining Average Grain Size
- E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
- G1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens
- G3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing
- G5 Reference Test Method for Making Potentiodynamic Anodic Polarization Measurements
- G28 Test Methods for Detecting Susceptibility to Intergranular Corrosion in Wrought, Nickel-Rich, Chromium-Bearing Alloys
- G61 Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion Susceptibility of Iron-, Nickel-, or Cobalt-Based Alloys
- G193 Terminology and Acronyms Relating to Corrosion
- G215 Guide for Electrode Potential Measurement

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *integrated charge (Q)*, n —charge measured, in coulombs, during reactivation as given by the time integral of current below the reactivation peak of the curve.

3.1.2 *maximum anodic current density (i_p)*, n —current density measured at the peak of the anodic curve during reactivation.

³ 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.

3.1.3 *normalized charge* (P_a), n —integrated current normalized to the specimen size and grain size. P_a represents the charge (in coulombs/cm²) of the grain-boundary area. The method for calculating P_a is given in 9.2.

3.1.4 *reactivation*, n —in the electrochemical reactivation (EPR) test, the potential sweep from the passivation potential returning to the corrosion potential.

3.1.5 *scan rate*, n —rate at which the electrical potential applied to a specimen in a polarization test is changed.

Test Method A—Single Loop

4. Summary of Test Method A—Single Loop

4.1 The EPR test is accomplished by a potentiodynamic sweep from the passive to the active regions of electrochemical potentials in a process referred to as reactivation. The EPR test measures the amount of charge associated with the corrosion of the chromium-depleted regions surrounding chromium carbide precipitated particles. Most of these particles in a sensitized microstructure are located at grain boundaries (see Terminology E7). Discrete particles located within the grain (referred to as intragranular precipitates) will also contribute to the total measured charge. Therefore, it is important to examine the alloy microstructure following an EPR test, to determine the relative proportion of corrosion site associated with intergranular versus intragranular precipitates.

4.2 The chromium-depleted zones around carbide precipitates in sensitized steels are particularly susceptible to corrosion in oxidizing acid solutions. Corrosion at chromium-depleted grain boundary sites causes a rapid rise in the current density when the electrochemical potential is changed from the passive to the active region.

4.3 A sensitized steel produces a curve similar to the active portion of the polarization curve during the reactivation from the passive region back to the rest potential (E_{corr}) as shown in Fig. 1. A nonsensitized (solution annealed) steel polarized under the conditions given in this test method will produce a curve with lower current densities than a sensitized steel.

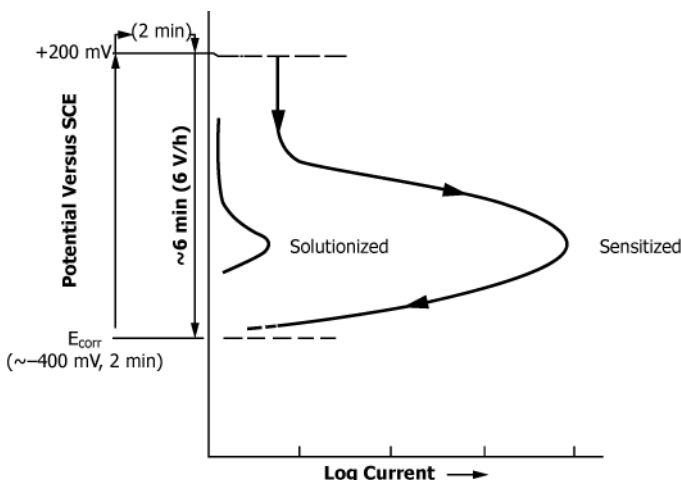


FIG. 1 Schematic EPR Curves for Sensitized and Solutionized AISI Type 304 Stainless Steel for Method A—Single Loop

4.4 The EPR test results are readily reproducible, as long as the electrolyte temperature, electrolyte composition, and scan rate are carefully controlled. The EPR test is significantly affected by the composition, thermomechanical condition and surface finish of the specimen as well as the presence of non-metallic inclusions, that result in pitting of the etched microstructure.

NOTE 1—Various cutting and grinding operations can promote sensitization of Type 304 (5). Superficial carbide precipitation can occur during cutting and grinding or during subsequent low temperature heat treatments, such as 24 h at 500 °C.

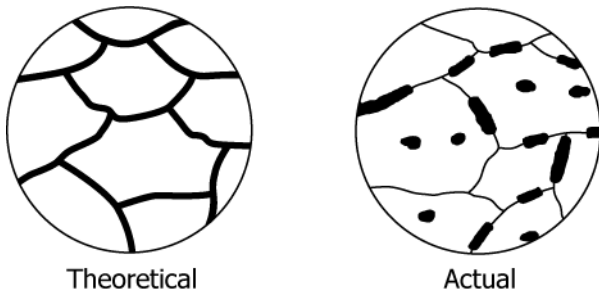
4.5 The criteria used to distinguish between sensitized and solution annealed samples are the activation charge, Q (given by the time integral of current density below the reactivation peak of the curve), or the maximum anodic current density, i_r , in the active state. Sensitized steels are easily activated and show higher Q and i_r values than solution annealed steels, that are not susceptible to intergranular corrosion. The value Q is normalized for both specimen size and grain size. The value normalized in this fashion is called P_a and represents the charge (in units of coulombs) per unit grain-boundary area. This normalization permits direct comparisons of different heats of material that exhibit different Q values solely as a result of differences in grain size.

5. Significance and Use for Test Method A—Single Loop

5.1 This test method describes an EPR test method for quantitatively determining the relative degree of sensitization in AISI Type 304 and 304L stainless steels. The EPR test has found wide use as a means to provide a numerical level of sensitization in studies of the effects of sensitization on intergranular corrosion and intergranular stress corrosion cracking behavior. The results of this test method correlate with other test methods (for example, Practices A262 and Test Methods G28) that are commonly used to assess sensitization in stainless steels.

5.2 The EPR test can also be used for product acceptance, service evaluation, regulatory statutes, and manufacturing controls providing that both the supplier and user have agreed upon appropriate acceptance criteria and a sensitizing treatment. The test is not intended for design purposes since the test conditions accelerate corrosion in a manner that does not simulate any actual service environment.

5.3 The EPR test involves the measurement of the amount of charge resulting from the corrosion of the chromium-depleted regions surrounding the precipitated chromium carbide particles. Most of these particles in a sensitized microstructure are located at the grain boundaries. However, discrete particles located within grains (referred to as intragranular precipitates) will also contribute to the total measured charge. (See Fig. 2.) Therefore, it is important to examine the alloy microstructure following an EPR test to determine the relative proportion of corrosion sites associated with intergranular versus intragranular precipitates. Sites of intergranular attack will appear similar to grain boundary ditching as defined in Practice A of Practices A262.



NOTE 1—The calculation of P_a is based on the assumptions illustrated at left. Mild cases of sensitization usually result in a combination of intergranular attack and pitting as illustrated at right (6).

FIG. 2 Schematic Microstructures After EPR Testing for Method A—Single Loop

6. Apparatus for Test Method A—Single Loop

6.1 The apparatus necessary for obtaining EPR data consists of electronic instruments and a test cell. These instruments may be integrated into one instrument package or may be individual components. Either form of instrumentation can provide acceptable data.

6.2 Typical apparatus, as illustrated in Fig. 3, shall consist of the following: scanning potentiostat (or potentiostat/voltage ramp generator combination), potential measuring instrument, current and current integration measuring instruments, and test cell and specimen holder.

6.2.1 *Scanning Potentiostat*—Requirements shall be in accordance with 4.2 of Reference Test Method G5 with the following refinements: the potentiostat shall control the potential within ± 5 mV accuracy over the range of potential and current density encountered in the EPR measurements. The potentiostat shall be operable in a potential range of -600 mV to $+500$ mV (SCE) and a current density range of $1 \mu\text{A}$ to 100 mA/cm^2 . The applied potential is changed either automatically or manually in the following manners:

6.2.1.1 Shifting the potential from the open circuit potential to a potential in the passive range, and

6.2.1.2 Scanning back to the open circuit potential (re-activation) at a voltage scan rate of 1.67 mV/s (6 V/h).

6.2.2 *Potential Measuring Instruments*—Requirements shall be in accordance with 4.3 of Reference Test Method G5 except that the potential range is as stated above.

6.2.3 *Current Measuring Instruments*—Requirements shall be in accordance with 4.4 of Reference Test Method G5. However, current measurements are essential for passivation assessment and other intermediate checks of system stability. The currents encountered in EPR for a specimen with the dimensions given in 7.3 are in the range of $1 \mu\text{A}$ to 100 mA/cm^2 . For samples of less than 100 mm^2 test area, currents above about 20 mA/cm^2 rarely have been reported.

6.2.4 *Current Integration Measurement Instruments (Optional)*—Current integration, or charge, can be measured by an electronic device incorporated into the potentiostat, or by a separate electronic device, such as a coulometer. If a coulometer is used, it shall be capable of measuring charges from 0.001 coulombs to 2 coulombs. The use of a coulometer shall be considered optional. Charge can also be measured by using a chart recorder, as illustrated in Fig. 3, to record a current versus time trace and then, subsequently, integrating it by various methods. When potentiostat measurements are available in a digitized format, an appropriate computer integration routine can also be used to obtain a value for charge.

6.2.5 *EPR Test Cell*—Requirements shall be in accordance with 4.1 of Reference Test Method G5. A deaeration tube is not required and only one counter electrode is required for EPR testing. A suitable cell and electrode arrangement is shown in Fig. 4.

6.2.6 *Electrode Holder*—Requirements shall be in accordance with 4.6 of Reference Test Method G5 or 4.2.1 of Test Method G61. The requirements for the working electrode (specimen) and counter electrode holders are that the holders

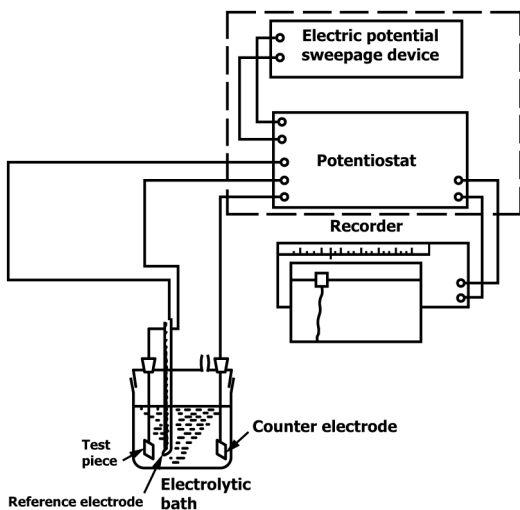
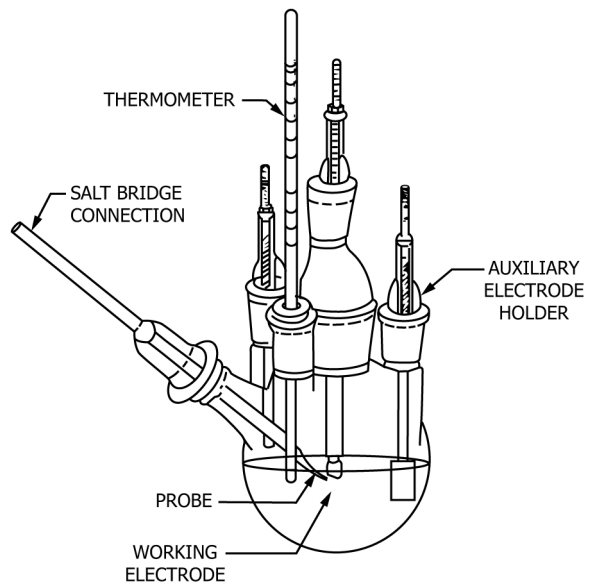


FIG. 3 Schematic Diagram of EPR Test Apparatus for Method A—Single Loop



NOTE 1—The sample face is completely immersed but the connection to the electrode holder is not immersed.

FIG. 4 Schematic Diagram of Electrochemical Cell for EPR Testing

be made of an inert material and any seals must not allow leakage of the electrolyte. When using the Reference Test Method G5-type holder the working electrode can be mounted as shown in Fig. 5 and described in Appendix X1.

6.2.7 *Auxiliary (Counter) Electrodes*—Requirements are in accordance with 4.7.2 of Reference Test Method G5 except that only one counter electrode is necessary for EPR testing. However, two auxiliary electrodes can provide for a more uniform distribution of current. Titanium or high-purity carbon may be used in place of platinum for the counter electrode since it is always the cathode.

6.2.8 *Calomel Reference Electrode*—Requirements are in accordance or equivalent to 4.7.3 of Reference Test Method G5. A KCl saturated silver/silver chloride electrode can also be used for testing (Practice G3 and Guide G215).

NOTE 2—The precision for Method A has not been determined for a KCl saturated silver/silver chloride electrode.

7. Sampling, Test Specimens, and Test Units for Test Method A—Single Loop

7.1 Sampling:

7.1.1 When using this test method to meet product acceptance criteria, the means of sampling of a test specimen shall be decided by agreement between the parties involved; for instance, but not limited to, a user and a supplier.

7.1.2 Specimens removed from a piece of AISI Type 304 or 304L steel by shearing, cutting, burning, and so forth shall have the affected edges removed by grinding or machining.

7.2 *Sensitization of Test Specimens*—Specimens can be given a sensitizing treatment when it is desired to assess the influence of a thermal exposure during fabrication on corrosion resistance.

7.2.1 Specimens may be tested in a condition simulating that of the product as installed in service. Specimens may be welded or heat treated in as nearly the same manner as the product will experience in fabrication or service. The user and supplier must agree to the use and conditions of a sensitization

treatment. The most common sensitizing treatment is 1 h at 675 °C (1250 °F) according to 15.3 of Practices A262.

7.2.2 Heat treatment, particularly carburization, may alter the surface to be tested and may invalidate the EPR test results. Precautions shall be taken to ensure that the specimen surface is representative of the product form in service. Refer to Section 6 of Practice G1 for descaling procedures and 7.3, as well as Section 5 of Practices A262 for guidance in preparing specimens.

7.2.3 Expose specimens to be given a sensitization treatment prior to EPR testing in a furnace at the required temperature and for the required time and then water-quench. Use a thermocouple and a timer to ensure that the entire specimen cross sections are at the specified temperature for the specified amount of time. The number of thermocouples needed to obtain a reliable reading for all specimens exposed in a furnace at a given time is left to the discretion of the user.

7.3 Specimen Preparation:

7.3.1 Test specimens can be any shape but shall be at least 3.2 mm (0.125 in.) in diameter or on a side dimension and of a suitable thickness. Specimens shall not be larger than 130 mm² (0.2 in.²) in area since such specimens will not fit into the recommended mold for mounting (see Appendix X1). A mounted specimen is illustrated in Fig. 5.

7.3.2 Remove any oxides or grease from the specimen as such film may promote loss of adhesion between the mounting compound and the specimen that could cause a crevice to form thereby producing erroneously high current densities during the EPR measurement.

7.3.3 The front surface of the specimen will be evaluated in the EPR test. The back surface of the test specimen is used to establish electrical contact with the specimen (see Note 3).

NOTE 3—A convenient way to make this attachment may be either by spot welding or by using a conducting cement to fasten a stainless steel machine screw (for example, NC4-40 × 0.3 cm (0.75 in.) long) to the back surface of the specimen. This assembly is mounted in a suitable compound that is inert in the EPR electrolyte (see Appendix X1) such that the front surface upon immersion in the EPR electrolyte is fully in contact with the electrolyte.

7.3.4 Measure the surface area of the front surface of the test specimen within 0.1 mm² precision and record on the EPR data record sheet (see Appendix X2).

7.3.5 Specimens can be in any shape that will not be susceptible to crevice corrosion in the solution. Test surface area shall be at least 10 mm² ± 0.1 mm² (0.016 in.²). It is occasionally useful to mask the area to be measured leaving an opening for exposure to the electrolyte. One suitable masking method uses precut pieces of an acid resistant tape. Care must be taken not to introduce undercutting of the tape during the EPR measurement because it will cause erroneously large currents.

8. Procedure for Test Method A—Single Loop

8.1 Metallographic Preparation:

8.1.1 Polish and attach the test specimen, mounted in a suitable inert compound, to the electrode holder following the procedures and cautions described in 8.1.2 to 8.1.6:

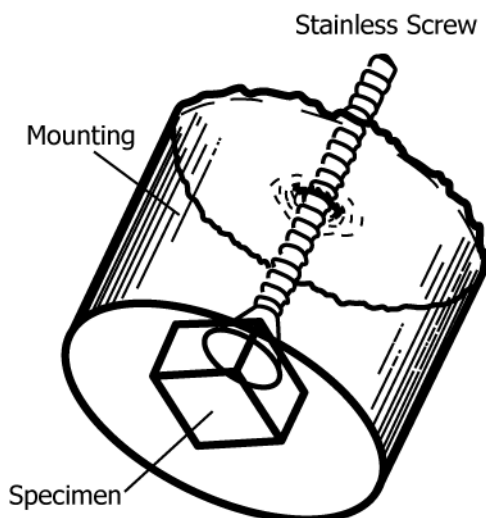


FIG. 5 A Method of Mounting Specimens for EPR Testing (6) for Method A—Single Loop

8.1.2 Exercise care since any crevice between the specimen and the mounting compound could lead to erroneously large current densities.

8.1.3 Prepare the surface within 1 h of the experiment, or store the prepared specimen in a suitable desiccating cabinet. Wet grind with 240 grit and 400 grit silicon carbide papers, and wet polish with 600 grit silicon carbide paper until all coarse scratches are removed. Rinse with water and dry. Polish the specimens in two additional stages with 6 and 1 μm diamond paste on a low speed polishing wheel in accordance with Guide E3.

8.1.4 Polishing specimens on automated, high speed wheels using aluminum oxide slurries is not recommended. Specimens tend to retain an alumina impregnated surface layer that gives erroneous results during the EPR test.

8.1.5 Attach the specimen to the specimen holder as described in either 4.6.1 of Reference Test Method G5 or 4.2.1 of Test Method G61. In the case of the Reference Test Method G5-type holder, tighten the assembly by holding the upper end of the mounting rod in a vise or clamp while turning the mounting nut until the gasket is properly compressed. Similarly for the Test Method G61-type holder, it is important to properly compress the TFE-fluorocarbon gasket to minimize the potential for crevice corrosion.

8.1.6 Clean the specimen just before immersion in the electrolyte by degreasing with a suitable detergent, rinsing in distilled water, then reagent grade methanol, and air drying.

8.2 Test Solution Preparation:

8.2.1 Prepare a mixture of reagent grade sulfuric acid (H_2SO_4) and potassium thiocyanate (KSCN) in reagent water as follows: 1 L of 0.5 M H_2SO_4 + 0.01 M KSCN and Type IV reagent water (in accordance with Specification D1193). The solution can be made up in bulk and stored for one month at room temperature. Transfer approximately 500 mL to 600 mL of solution to a clean test cell.

8.3 Initiating the Test:

8.3.1 Bring the temperature of the solution to $30\text{ }^\circ\text{C} \pm 1\text{ }^\circ\text{C}$ by immersing the cell in a controlled temperature water bath or by other convenient means.

8.3.2 Place the specimen, counter electrodes, salt bridge probe, and other components in the test cell. Ensure the salt bridge is filled with the test solution and contains no air bubbles, particularly in the restricted space at the tip.

8.3.3 Record the open circuit potential (OCP) of the test specimen after 1 min to 2 min of immersion. If the OCP is not consistent with typical values for the given alloy (for example, -350 mV to -450 mV versus SCE for AISI Type 304), cathodically polarize the specimen to -600 mV versus SCE for 0.1 to 1 min and recheck the rest potential. If the rest potential is still abnormal (relative to the usual value around -200 mV for solutionized Type 304 and 304L), the specimen must be removed from the cell and repolished (back to the step for polish with 1 μm or 6 μm diamond paste is usually sufficient).

8.3.4 Passivation is accomplished by applying the potential to $+200\text{ mV}$ versus standard calomel electrode and holding for 2 min. For specimens 1 cm^2 or less in area, a current density of 10 $\mu\text{A}/\text{cm}^2$ or less indicates that the specimen has passivated.

8.4 Reactivation Scan:

8.4.1 Set the current integration to zero and start the current integrator instrument. (Some instruments perform these steps automatically.) Start the potential scan in the active direction at the rate of $1.67\text{ mV/s} \pm 0.08\text{ mV/s}$ (6 V/h). During the reactivation scan, the current density will decay quite rapidly.

8.4.2 Record the reading on current integrator when potential reaches 50 mV above (more positive) the initial E_{corr} . This reading is the integrated current or charge value in coulombs. (Some instruments are capable of ending the experiment automatically.) The test is complete once this reading has been obtained.

8.4.3 Once the test is complete, put all electrochemical polarization equipment on standby. Remove the specimen from the cell and holder, rinse it in water, clean with alcohol or detergent, rinse again, and then air dry.

8.4.4 *Optional E Versus Log I Plot*—The recorder automatically plots the anodic polarization data on semilogarithmic paper in accordance with Practice G3. A strip chart recorder may also be used since potential is linear with time.

8.5 Metallographic Inspection:

8.5.1 Photograph surface of each specimen after testing (without additional preparation or etching) at a suitable magnification to determine grain size and to document the microstructures and extent of grain boundary attack. If the specimen is not sufficiently etched after the EPR test to delineate the microstructure for grain size determination, then the specimen shall be etched by either electrolytic 10 % oxalic acid (in accordance with Practice A of Practices A262), 60 % HNO_3 -40 % H_2O (7), or by other suitable means to delineate the grain boundaries.

8.5.2 Examine the microstructure after the EPR test to ensure that the bulk of the integrated current Q value actually represents attack of the grain boundary areas (that is, “ditching” in terms of Practice A of Practices A262 has occurred). Reactivation of intragranular (matrix) precipitates (principally chromium carbides) that may be present in substantial quantities in some specimens (6, 8) may contribute to the integrated current Q value. Intragranular precipitates are only of concern when the Q value is above an established acceptance criteria. In such cases, the user and supplier may have to agree to higher acceptance criteria values that reflect the contribution of intragranular precipitates to the measured Q value. Examples of correlations of Pa values to the degree of sensitization for AISI Type 304 and 304L stainless steels are offered as a general guide to interpretation of EPR results in Appendix X3.

9. Calculation for Test Method A—Single Loop

9.1 Calculate the surface area (A_s) of the specimen in cm^2 by using the dimensions in 7.3.4.

9.2 Calculate and record the normalized charge (P_a) in units of coulombs/ cm^2 , using the following equation:

$$P_a = Q/X \quad (1)$$

where:

Q = charge measured on current integration measuring instrument (coulombs). Q is normalized for both specimen size and grain size,

$X = A_s [5.1 \times 10^{-3} e^{0.35 G}]$
 A_s = specimen area (cm²), and
 G = grain size at $100 \times$ (in accordance with Test Methods E112).

NOTE 4—Often in the technical literature, the ASTM grain size number is designated as “X” and the grain boundary area is “GBA.”

9.3 In the derivation of the equation in 9.2 it was assumed that the Q value is due to the attack on the specimen surface that is distributed uniformly over the entire grain boundary region of a constant width of $2 \times (5 \times 10^{-5})$ cm. This may not represent the actual physical processes.

10. Report for Test Method A—Single Loop

10.1 Record test parameters as:

- 10.1.1 EPR test number, test operator, and place of test;
- 10.1.2 Specimen number;
- 10.1.3 Material, including vendor alloy certification;
- 10.1.4 Heat;
- 10.1.5 Solution temperature;
- 10.1.6 Reactivation scan rate;
- 10.1.7 Passivation potential/time;
- 10.1.8 Rest potential; and
- 10.1.9 Specimen surface area.

10.2 Use the example data record sheet in Appendix X2 or an equivalent one for recording these data.

11. Precision and Bias⁴ for Test Method A—Single Loop

11.1 *Statement of Precision:*

11.1.1 The precision of the single loop method has been determined by an interlaboratory test program on a set of specimens from a single heat each of Type 304 and Type 304L. Precision in this case has two components repeatability and reproducibility.

11.1.2 Interlaboratory reproducibility of the P_a values decreases with increasing degrees of sensitization. This indicates that P_a values are sensitive indicators of differences in specimens with mild degrees of sensitization, but do not readily distinguish between medium or severely sensitized specimens.

11.1.2.1 Reproducibility refers to the agreement that occurs when samples of a single material are tested by several different laboratories. The results of an interlaboratory test program (4) are shown in Table 1. Samples of Type 304 and 304L, compositions given in Table 2, in four different heat treatment conditions were evaluated in the round robin. Each lab value represents the average of three or more tests.

11.1.2.2 A linear regression (through the origin) analysis of the standard deviation values S_R shown in Table 1 showed that the standard deviations were strongly correlated to the average P_a value of all participating laboratories \bar{P}_a , and could be represented by:

$$s_R = 0.52 \bar{P}_a \quad (2)$$

where S_R is the standard deviation of the average values reported by the participating laboratories.

11.1.2.3 However, further analysis showed that the distribution of S_R values was not normal, but could be adequately represented by Weibull two parameter functions. After fitting each of the data set for each material to a best fit Weibull distribution, a 95 % confidence interval could be calculated. These values are shown in Table 2. Linear regression through the origin for both the upper, UCL, and lower, LCL, confidence limits showed that these values were strongly correlated to the average P_a value and could be adequately represented by the following expressions:

$$\text{UCL} = 2.63 \bar{P}_a \quad (3)$$

$$\text{LCL} = 0.126 \bar{P}_a$$

11.1.3 Repeatability (r) refers to the agreement that occurs when a single laboratory runs sequential tests under identical conditions. Repeatability results are shown in Table 3. The

⁴ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:G01-1010. Contact ASTM Customer Service at service@astm.org.

TABLE 1 EPR Test Interlaboratory Program Reproducibility Study

Lab	P_a (Coulombs/cm ²) ^A Values for Cited Sample Conditions and AISI Types of Stainless Steel							
	As-Received		621 °C for 24 h		621 °C for 1 h		500 °C for 24 h	
	304	304L	304	304L	304	304L	304	304L
1	0.14	0.01	13.6	4.4	0.31	1.10	0.12	0.09
2	0.01	0.00	5.6	2.7	0.20	0.38	0.12	0.16
3	0.00	0.00	5.6	3.6	0.14	0.25	0.07	0.10
4	0.06	0.06	10.3	5.0	0.19	0.11	0.08	0.05
5	0.03	0.01	8.9	2.3	0.15	0.47	0.08	0.10
6	0.00	0.00	1.7	1.8	0.07	0.32	0.05	0.15
7	0.07	0.05	17.9	3.6	0.43	0.95	0.15	0.23
8	0.04	0.02	17.6	3.7	0.47	0.32	0.14	0.08
9	0.002	0.03	17.9	6.6	0.31	0.31	0.03	0.01
10	0.07	0.01	17.6	8.2	0.26	0.50	0.23	0.09
\bar{P}_a	0.0422	0.0190	11.70	4.19	0.253	0.471	0.107	0.106
S_R	0.0445	0.0213	6.13	1.97	0.128	0.314	0.058	0.061
LCL ^B	8.62×10^{-4}	0.0022	1.26	1.12	0.050	0.067	0.020	0.008
UCL ^B	0.264	0.0800	3.20	7.85	0.536	1.11	0.230	0.335

^A Each P_a value is the average of three or more tests.

^B Lower and upper 95 % confidence limits based on Weibull analysis of data distributions.

TABLE 2 Chemical Compositions of the Alloys, in Weight Percent, Used in the Round Robin

	T-304	T-304L
C	0.049	0.021
Mn	1.30	1.38
P	0.033	0.027
S	0.015	0.009
Si	0.65	0.59
Cr	18.67	18.83
Ni	8.78	10.27
Mo	0.38	0.58
Cu	0.12	0.11
N	0.056	0.020

variation in repeatability, as measured by the standard deviation S_R is correlated to the average P_a value. The following expression was determined by a linear regression through the origin of the data listed in Table 3.

$$s_R = 0.19 \bar{P}_a \quad (4)$$

where \bar{P}_a is the average of three sequential tests.

11.1.4 The 95 % confidence interval, r , is 2.8 s_r , or:

$$r = \pm(0.53) \bar{P}_a \quad (5)$$

$$\text{UCL} = 1.53 \bar{P}_a \quad 00$$

$$\text{LCL} = 0.47 \bar{P}_a$$

where UCL and LCL are the upper and lower 95 % confidence interval limits.

11.2 Statement of Bias:

11.2.1 The results of the interlaboratory test program for the single loop EPR test are available from ASTM Headquarters by requesting Research Report RR:G01-1010.⁴ Variation in electrolyte temperature, electrolyte composition, scan rate, specimen composition, specimen thermomechanical condition, and specimen surface finish affect the measured Q value and will constitute a source of bias.

11.2.2 The EPR results are reproducible using different polarization instrumentation and correlate well to the degree of intergranular carbide precipitation observed metallographically in Practice A of Practices A262 (6).

11.2.3 As discussed in Section 5, pitting caused by the dissolution of non-metallic inclusions can increase the P_a value. In such cases, it is recommended to examine the microstructure after the test to identify the source of the elevated P_a value.

Test Method B—Double Loop

12. Summary of Test Method for Test Method B—Double-loop (DL) Test

12.1 As noted in Section 4, the results from the single-loop test could be highly sensitive to test variables such as the surface finish of the test specimen. A double-loop test has been shown to minimize this sensitivity on specimen preparation and area measurement (9, 11). In the Method B or double-loop test, the potential is scanned in the positive direction from the corrosion potential to the passive region and then from the passive region back to the corrosion potential (called the

reverse scan) (Fig. 6). Two current peaks may be observed in the double-loop test. In the forward scan, the peak is called Ia, and in the reverse scan, the peak (if present) is called Ir. The degree of sensitization (DOS) is measured by the ratio, Ir/Ia. The higher this ratio, the higher the degree of sensitization of the tested specimen. If a peak is not present in the reverse scan, the DOS ratio is practically 0 (nil) (9, 10). If the DOS ratio is below 0.05, the material can be considered nonsensitized.

13. Specimens, Apparatus, and Electrolytes for Test Method B—Double-loop (DL) Test

13.1 For Method B, the surface preparation and surface area are not as critical as for the Method A. A recommended surface finish for Method B 304 SS is a wet grinding with 320 grit paper. Clean the specimen just before immersion in the electrolyte by degreasing with a suitable detergent, rinsing in reagent water, then reagent-grade alcohol, and air drying. The area of the specimen is not important except for setting the maximum allowed current in the potentiostat (see Note 6). For specimen preparation, test cell and equipment setup follow recommendations from Reference Test Method G5. To perform the Method B electrochemical testing, use the electrolyte 0.5 M H₂SO₄ + 0.01 M KSCN, naturally aerated, at ambient temperature. To prepare 1 L of this solution, dissolve 28 cm³ of concentrated sulfuric (~96 %) acid and 0.97 g of KSCN salt with reagent water (Specification D1193) to a final volume of 1 L.

14. Procedure for Test Method B—Double-loop (DL) Test

14.1 Immerse the 304 SS test specimen (working electrode) into the test solution and monitor the corrosion potential (using a reference electrode) for 30 min. Record the value of E_{corr} . The corrosion potential value should be in the vicinity of -400 mV SCE or -355 mV SSC (see Note 5). Perform a cyclic potentiodynamic test (described in Test Method G61) starting at the corrosion potential at a forward positive scan rate of 1.67 mV/s. When the potential reaches +300 mV SCE or +345 mV SSC, reverse the direction of the potential scan at a scan rate of 1.67 mV/s and end the scan at the original E_{corr} (Fig. 6). Record the current and potential during the scan (Fig. 6 and Test Method G61).

NOTE 5—SCE saturated calomel electrode, SSC silver/silver chloride (sat KCl) electrode.

NOTE 6—The peak current density i_a in the forward scan could be in the order of 35 mA/cm², therefore, the potentiostat should be properly set up (for example, to be able to read a maximum current density of 100 mA/cm²).

15. Calculations and Report for Test Method B—Double-loop (DL) Test

15.1 From the cyclic polarization test in Section 14, record the values of I_a and I_r (Fig. 6) and calculate the degree of sensitization $\text{DOS} = (I_r/I_a)$. If the DOS value is below 0.05, the material is considered nonsensitized (9, 10). If the tested specimen does not show an I_r peak, the I_r peak value is taken as the current value in the reverse scan at the potential of the I_a peak (10). The report should contain information on place of testing, characteristics of testing materials such as alloy certification documentation and metallurgical condition of the