

# Standard Test Method for Electrochemical Critical Pitting Temperature Testing of Stainless Steels<sup>1</sup>

This standard is issued under the fixed designation G150; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

# 1. Scope

1.1 This test method covers a procedure for the evaluation of the resistance of stainless steel and related alloys to pitting corrosion based on the concept of the determination of a potential independent critical pitting temperature (CPT).

1.2 This test methods applies to wrought and cast products including but not restricted to plate, sheet, tubing, bar, forgings, and welds, (see Note 1).

Note 1—Examples of CPT measurements on sheet, plate, tubing, and welded specimens for various stainless steels can be found in Ref (1).<sup>2</sup> See the research reports (Section 14).

1.3 The standard parameters recommended in this test method are suitable for characterizing the CPT of austenitic stainless steels and other related alloys with a corrosion resistance ranging from that corresponding to solution annealed UNS S31600 (Type 316 stainless steel) to solution annealed UNS S31254 (6 % Mo stainless steel).

1.4 This test method may be extended to stainless steels and other alloys related to stainless steel that have a CPT outside the measurement range given by the standard parameters described in this test method. Appropriate test potential and solution must then be determined.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 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:<sup>3</sup>
- D1193 Specification for Reagent Water
- D1293 Test Methods for pH of Water
- E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
- 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
- G46 Guide for Examination and Evaluation of Pitting Corrosion
- G107 Guide for Formats for Collection and Compilation of Corrosion Data for Metals for Computerized Database Input
- G193 Terminology and Acronyms Relating to Corrosion

# 3. Terminology

3.1 Definitions:

3.1.1 *critical pitting temperature (CPT)*—the lowest temperature on the test surface at which stable propagating pitting occurs under specified test conditions indicated by a rapid increase beyond a set limit of the measured anodic current density of the specimen.

3.1.2 *pitting potential range*—the range of measured potentials where pitting is initiated. This potential range only exists above the minimum critical pitting temperature; see also Appendix X1.

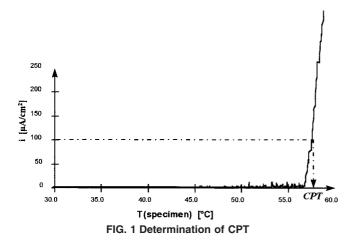
3.1.3 *potential dependent CPT*—the CPT determined at a potential within the pitting potential range of the tested material; see also Appendix X1.

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

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 $<sup>^{2}</sup>$  The boldface numbers in parenthesis refer to the list of references at the end of this standard.

<sup>&</sup>lt;sup>3</sup> 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.



3.1.4 *potential independent CPT*— the CPT determined at a potential above the pitting potential range, but below the transpassive potential; see also Appendix X1.

3.1.5 *temperature ramp*—the rate (°C/min) at which the test temperature is increased during the test.

3.2 *sign conventions*—the sign conventions used in this procedure are in agreement with Practice G3.

3.3 Unless otherwise stated, this test method uses the general terminology relating to corrosion and corrosion testing as defined in Terminology G193.

#### 4. Summary of Test Method

4.1 The test method determines the potential independent critical pitting temperature (CPT) by way of a potentiostatic technique using a temperature scan and a specimen holder that is designed to eliminate the occurrence of crevice corrosion (see Fig. 1). The specimen is exposed, either entirely or in part, depending on test cell configuration to a 1M NaCl solution, initially at 0°C. After an initial temperature stabilization period, the solution is heated at a rate of 1°C/min. About 60 s before the temperature scan is commenced, the specimen is anodically polarized to a potential above the pitting potential range. This potential is held constant during the whole temperature scan. A potential of 700 mV versus SCE (25°C) has been found suitable for most stainless steels. The current is monitored during the temperature scan, and the CPT is defined as the temperature at which the current increases rapidly, which for practical reasons is defined as the temperature at which the current density exceeds 100 µA/cm<sup>2</sup> for 60 s. Pitting on the specimen is confirmed visually after the test.

### 5. Significance and Use

5.1 This test method provides a prediction of the resistance to stable propagating pitting corrosion of stainless steels and related alloys in a standard medium (see Note 1). The CPT test can be used for product acceptance, alloy development studies, and manufacturing control. In the case of product acceptance, the supplier and user must agree upon the preconditioning of the specimen with regard to surface finish. 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.

5.2 Another method to determine the potential independent CPT with an electrochemical technique has been discussed in the literature (1-4). This test method involves a potentiodynamic (potential sweep) procedure performed on specimens at different temperatures. A comparison (2) of the test method described in this test method and the potentiodynamic technique has indicated no difference in the test result obtained.

#### 6. Apparatus

6.1 The apparatus necessary for determining the CPT consists of instruments for measuring electronic signals, a temperature controlling apparatus, a specimen holder, and a test cell. The instruments for measuring electronic signals may be integrated into one instrument package or may be individual components. Either form of instrumentation can provide acceptable data. Typical test equipment consists of the following: (*I*) potentiostat (2) potential measuring instrument (3) current measuring instrument (4) temperature controller (5) temperature measuring instrument (6) test cell (7) specimen holder, and (8) electrodes.

6.2 *Potentiostat*—The potentiostat shall be able to apply the constant potential to within 1 mV at a current density of 10 mA/cm<sup>2</sup>. The applied potential is changed either automatically or manually by shifting the potential from the open circuit potential to another more noble potential.

6.3 *Potential Measuring Instrument*—Requirements shall be in accordance with the section on Potential Measuring Instruments in Test Method G5.

6.4 *Current Measuring Instruments*—An instrument that is capable of measuring a current accurately to within 5 % of the actual value. The typical current densities encountered during the CPT test are in the range of 1  $\mu$ A/cm<sup>2</sup> to 10 mA/cm<sup>2</sup>.

6.5 Temperature Controller:

6.5.1 Thermostat equipment is required that can provide cooling and heating of the test solution in the temperature range from 0°C to approximately 100°C. Further, the temperature controller is used to provide controlled heating, which gives the test solution temperature a temperature increase rate of 1°C/min in the range from 0°C to approximately 100°C.

6.5.2 Above 10°C, the average rate of temperature change of the test solution shall be  $1.0 \pm 0.3$ °C/min, where the average is calculated over a temperature range of 10°C.

6.6 *Temperature Measurement Instrumentation*, shall be capable of measuring the temperature of the test solution with an accuracy of  $\pm 0.4^{\circ}$ C.

#### 6.7 Test Cell:

6.7.1 *Option 1, G5 Type*—The test cell should be similar to the one described in Test Method G5. Other similar polarization cells may be equally suitable. The gas purger should distribute the gas in numerous small bubbles.

6.7.2 Option 2, Flushed-port Cell—This cell design is based on that published by R. Qvarfort (**3**) and includes the specimen holder in the design. The advantages of this cell design are that the specimen edges and back do not need to be machined, the specimen does not have to be mounted inside the cell, and crevice corrosion at the contact area of the cell port is completely eliminated, even at elevated test temperatures. See Appendix X2 for a description of this cell. The gas purger should distribute the gas in numerous small bubbles.

6.7.3 The test cell shall be able to contain a test solution volume of minimum 100 mL per square centimetre test area. A maximum dilution of 15 % of the test solution during the test period is allowed in case a flushed port cell or similar arrangement is used.

### 6.8 Specimen Holder:

6.8.1 Any part of the specimen holder coming in contact with the test solution during testing shall be made of an inert material, and any seal shall not allow leakage of electrolyte.

6.8.2 The specimen holder shall have a design that ensures no occurrence of crevice corrosion at the contact area between specimen holder and specimen.

6.8.3 Two examples of specimen holder designs in accordance with this standard are shown in Appendix X2 and Appendix X3. The major difference between the specimen holder designs lies in the allowable specimen geometry and the number of surfaces on the specimen that are being tested simultaneously.

# 6.9 Electrodes:

6.9.1 *Auxiliary (Counter) Electrode*—Requirements shall be in accordance with the section Auxiliary Electrodes in Test Method G5 with the exception that only one counter electrode is necessary for CPT testing. The electrode material shall be of a type which can be considered inert under the test conditions.

6.9.2 *Reference Electrode*—The reference electrode shall be kept at room temperature outside the actual test cell. The reference electrode shall be capable of ensuring a constant reference potential within  $\pm 5$  mV during the entire test procedure (see Note 2). Electrical contact to the test solution shall be provided by the use of a luggin capillary placed in the test solution. Requirements shall otherwise be in accordance with the section on Reference Electrode in Test Method G5.

Note 2—It may be difficult to ensure a fully constant reference potential due to the large variations in temperature of the test solution; therefore, the allowable is  $\pm 5$  mV. This does, however, not affect the measured potential independent CPT (1).

#### 7. Test Specimens

7.1 *Finish*—Any geometry and surface finish (see Note 3) compatible with the chosen specimen holder as specified in 6.8 may be used.

NOTE 3—The state of the surface may be dependent on the time and location of storage between the final mechanical or chemical surface treatment and testing. The time and location of storage may, therefore, in some situations be considered an integral part of the surface finish.

7.2 *Sampling*—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.

7.3 Test Area—A minimum test area of 1 cm<sup>2</sup> shall be used.

7.4 Specimens removed from a work piece or component by shearing, cutting, burning, and so forth shall have the affected

edges removed by grinding or machining, unless it is explicitly intended to study the effects of these edge factors.

# 8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.<sup>4</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to purified water shall be understood to mean reagent water as defined by Type IV of Specification D1193.

8.3 *Standard Test Solution*—To prepare 1 L of 1 M sodium chloride (NaCl) solution, dissolve 58.45 g sodium chloride (NaCl) in purified water to a total solution volume of 1 L. The solution can be made up in bulk and stored for one month at room temperature.

8.4 *Purging Gas*—Nitrogen gas of minimum 99.99 % purity should be used.

# 9. Applied Potential

9.1 *Standard Potential*—An anodic potential of 700 mV versus SCE (25°C) is used. This has been found appropriate for most stainless steels (1).

9.2 Alternative Potential:

9.2.1 If uncertainty exists concerning whether the standard potential is sufficiently high to obtain the potential independent CPT, a test at 800 mV versus SCE (25°C) may be performed. A significant deviation between the CPT obtained at 700 mV and 800 mV will indicate a need for a reevaluation and new choice of potential.

Note 4—Using a lower potential than the standard potential of 700 mV versus SCE (25°C) is fully acceptable, provided the determined CPT still is potential independent. To change the measurement range provided by the standard test conditions, a new test solution composition will have to be chosen. Following the choice of test solution, a test potential that ensures the determination of a potential independent CPT will have to be determined.

9.2.2 Evaluation of differences in obtained CPT at the two potentials should take into account the repeatability of the test method. The homogeneity of the material used for the two different potentials shall also be considered before an alternative potential is used.

# 10. Procedure

10.1 Sample Mounting, Cleaning and Placement:

10.1.1 The recommendations given in Practice G1 are to be followed, where applicable, unless otherwise stated in this procedure.

<sup>&</sup>lt;sup>4</sup> Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analar Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.