



## Designation: **G150–99 (Reapproved 2010) G150 – 13**

# 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 ( $\epsilon$ ) 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

~~**G15** Terminology Relating to Corrosion and Corrosion Testing (Withdrawn 2010)~~<sup>4</sup>

**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:*

<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>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.

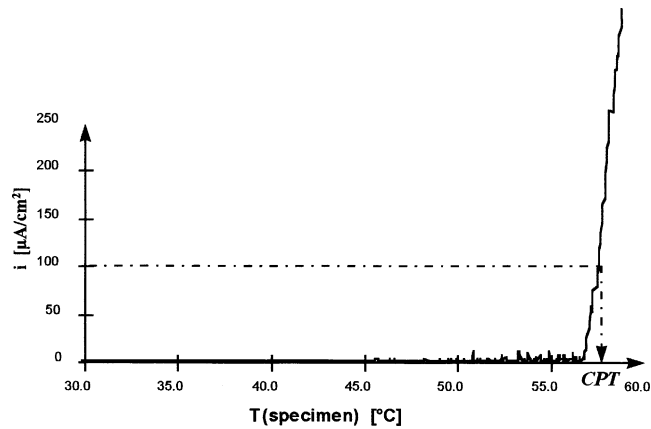


FIG. 1 Determination of CPT

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](#).

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 ( $^{\circ}\text{C}/\text{min}$ ) 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 [G15G193](#).

## 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  $0^{\circ}\text{C}$ . After an initial temperature stabilization period, the solution is heated at a rate of  $1^{\circ}\text{C}/\text{min}$ . 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 ( $25^{\circ}\text{C}$ ) 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  $100 \mu\text{A}/\text{cm}^2$  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: (1) 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 μA/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 ± 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 ±0.4°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 ±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 ±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.

10.1.2 Clean the specimen just before immersion in the electrolyte by degreasing with a suitable detergent, rinsing in purified water, followed by ethanol or similar solvent, and air drying. After degreasing, handle the specimen with clean gloves, soft clean tongs, or equivalent preventive measures, to avoid surface contamination.

10.2 *Test Solution Preparation:*

10.2.1 Prepare the test solution according to Section **8**.

10.2.2 Bring the solution to an initial temperature at or below 3°C.

10.3 Mount the specimen in the specimen holder.

10.4 Place the specimen, counter electrode, salt bridge probe, and other components in the empty test cell.

10.5 Fill the cell with cold ( $\leq 3^\circ\text{C}$ ) solution.

10.6 Ensure that the salt bridge is filled with the test solution and is free of air bubbles, particularly in the restricted space at the tip. A lugging probe should contain a wick, or equivalent device, to ensure electric contact even when small gas bubbles are formed during the test.

10.7 Commence bubbling nitrogen gas through the solution.

NOTE 5—The purpose of the purging gas is to enhance the stirring of the test solution. No reduction in the oxygen level of the test solution is intended. Presence or absence of oxygen has been shown not to affect the test result **(1)**.

10.8 Connect the electrodes to the potentiostat and data recording device and the connections for temperature measurement and control. Let the system cool and stabilize at  $0 \pm 1^\circ\text{C}$  for a minimum of 10 min.

<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.

10.9 Record the open circuit potential (OCP) of the test specimen shortly before the test is begun.

10.10 Set the data acquisition for electrode current and solution temperature. The sampling rate shall correspond to a minimum of two measurements every minute to follow solution temperature and the variation of the current on the working electrode.

10.11 Apply the constant anodic potential to the working electrode.

10.12 The potential shall be applied for  $60 \pm 5$  s before the temperature increase.

10.13 The temperature is then ramped at  $1^\circ\text{C}/\text{min}$ .

10.14 Continue measuring the temperature until the CPT has been determined or the maximum required temperature is reached. The CPT is determined when the current density reaches  $100 \mu\text{A}/\text{cm}^2$  and remains above this level for a minimum of 60 s. Terminate the test and data acquisition after either of these limits has been reached.

10.15 *Completion of Test*—Dismount the specimen as soon as possible after test completion. Inspect the specimen to locate pits (see [Note 6](#)). Rinse the specimen in water, clean with ethanol (95 % is suggested) or detergent, rinse again with water, and then air dry.

**NOTE 6**—Pits may be difficult to locate if the test is stopped shortly after pitting initiation. Pits may be located based on leaking rust or by using a needle to uncover pits hidden below a thin metal or oxide film. The lack of visible pitting may indicate that general corrosion has occurred, for example, transpassive corrosion. However, only a more thorough examination of the tested specimen can give a possible explanation.

## 11. Visual Examination of Test Electrode

11.1 Confirm the existence of pits and the absence of crevice corrosion using a microscope at  $20\times$  magnification. Note the location of pits relative to the test geometry (in the center, on edges, at the bottom center, at the bottom edge, and so forth).

11.2 Any crevice corrosion observed on the specimen after testing means that the test results are invalid and must be discarded.

11.3 If required, a more thorough examination of the electrode can involve measurement of pit density and pit depths on specimen electrodes, according to [Guide G46](#), using a microscope at  $20\times$  magnification.

## 12. Data Analysis

12.1 Measured current as a function of time shall be converted to current density values. The data may be presented as in [Fig. 1](#), which shows an example of a current density versus temperature plot.

12.2 *Evaluation of the CPT:*

12.2.1 *Standard Evaluation*—The critical pitting temperature is taken as the temperature at which the current increases above  $100 \mu\text{A}/\text{cm}^2$  (see [Note 7](#)) and stays above this critical current density for a minimum of 60 s; see [Fig. 1](#).

**NOTE 7**—The CPT is defined as the lowest temperature at which stable propagating pitting occurs. For practical reasons, this is being translated to the temperature at which the current density increases above a certain level ( $100 \mu\text{A}/\text{cm}^2$  has been chosen as a standard in this test method). A 60 s delay is introduced in order to ensure that the observed current increase originates from stable propagating pitting and not short lived current peaks originating from metastable pitting.

12.2.2 *Alternative Evaluation:*

12.2.2.1 For materials that generally exhibit a very high passive current density (for example some, but not all, stainless steel welds) or low pitting propagation rate (some, but not all, nickel-base alloys) a different critical current density may be chosen, but generally this should be avoided. Any change in the evaluation criteria shall be noted specifically in the report.

12.2.2.2 Comparison of CPTs obtained with different critical current densities is very difficult and should generally not be attempted.

12.2.3 The registered temperature in the solution will not be exactly the same as the temperature of the specimen, because the solution temperature is continuously changed during most of the test. A conversion of the increasing solution temperature to specimen temperature should be performed and the CPT should be defined relative to the temperature of the specimen.

12.2.4 The conversion between solution and specimen temperature may be done either by direct measurement of the specimen surface temperature during the test or by using a suitable calibration formula based on an earlier parallel measurement of specimen temperature and solution temperature. A detailed guideline of how to obtain a suitable calibration formula is given in [Annex A1](#).

## 13. Report

13.1 Report the following *mandatory* information:

13.1.1 Test identification number and date of test.

13.1.2 Critical pitting temperature (CPT). CPTs below  $10^\circ\text{C}$  shall only be reported as below  $10^\circ\text{C}$  or  $<10^\circ$ .

13.1.3 Formula for conversion of test solution temperature to specimen temperature shall be reported. If no conversion of the solution temperature to specimen temperature has been done, this shall be stated specifically in the test report.

13.1.4 Location of pits on the tested surface.

13.1.5 Test area.

13.1.6 Material identification data.

**TABLE 1 Precision**

Material, UNS No.	Average CPT, °C	Repeatability Standard Deviation, $s_r$	Reproducibility Standard Deviation, $s_R$	95 % Repeatability Limit, $r$	95 % Reproducibility Limit, $R$
S31600	19.5	1.9	2.8	5.4	7.9
S31803	51.9	1.7	2.4	4.9	6.7
N08904	54.1	3.7	3.7	11.3	11.3
S31254	82.7	3.4	6.6	9.4	18.5 <sup>A</sup>

<sup>A</sup> See Note 8.

13.1.7 Type of test cell and test solution volume.

13.1.8 Surface finish and approximate geometry of the tested specimen including the approximate storage time between final surface finish preparation and testing.

13.1.9 If no pits were observed despite an observed rapid current increase, this lack of visual pitting identification shall be noted explicitly. The evaluation of such deviations should lead to a more thorough examination of the specimen.

13.1.10 If parameters deviating from the standard values in this test method have been used, then all deviations shall be reported.

13.2 *Optional Reporting*—If required, a more elaborate report can, additional to the mandatory report, contain one or more of the following information:

13.2.1 Test identification number; specimen number; material; heat number; product form; solution temperature at CPT; open circuit potential; solution pH before start and after completion of test.

13.2.2 Together with the basic test results, it is recommended that the data from the CPT test should be presented graphically, as shown in Fig. 1.

13.2.3 In addition the following data are useful to report, (a) the scatter or deviation in the CPT values based on multiple runs if available, (b) pit geometry, number of pits formed and their depth in accordance with Guide G46.

13.3 The example data record sheet in Appendix X4, or equivalent, may be used for reporting.

## 14. Precision and Bias<sup>5</sup>

14.1 *Interlaboratory Test Program*—An interlaboratory study was run in which the critical pitting temperatures were determined for four grades of stainless steels with laboratory ground surface. Ten laboratories participated in the study. Each laboratory tested three to five test specimens of each of the four materials. Practice E691 was followed for the design and analysis of the data.

14.2 *Precision*—See Table 1. The terms, repeatability limit and reproducibility limit, are used as specified in Practice E177. The repeatability and reproducibility limits were obtained by multiplying the respective standard deviations by 2.8.

NOTE 8—The high reproducibility limit for material UNS S31254 is believed to be caused by problems with temperature calibration, which is most critical for materials with high CPTs. The variations and reported procedures in the round robin results were, however, judged to be insufficient to exclude the values from two laboratories (out of ten laboratories), which otherwise would have resulted in that the repeatability limit would have been  $\pm 7.4^\circ\text{C}$  and the reproducibility limit would have been  $\pm 12.5^\circ\text{C}$ .

14.3 *Bias*—This test method has no bias, because the electrochemically (potentiostatic) determined potential independent CPT is defined by this test method and no accepted reference standard exists.

## 15. Keywords

15.1 critical pitting temperature; electrochemical test; pitting corrosion; stainless steel

<sup>5</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Reports RR:G01-1017 and RR:G01-1020.