



Standard Test Method

Testing of Catalyzed Titanium Anodes for Use in Soils or Natural Waters

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NACE International
1440 South Creek Drive
Houston, Texas 77084-4906
+1 281/228-6200

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Foreword

This NACE International standard has been prepared to provide users and manufacturers of cathodic protection (CP) anodes with a test method for evaluating the anode's ability to achieve an expected lifetime. It is applicable for catalyzed titanium anodes intended for use in underground or underwater environments. This test is intended to evaluate whether such an anode complies with required specifications of design life expectancy at rated current output.

This standard is intended for use by consultants, suppliers, and end users of catalyzed titanium CP anodes.

This standard was prepared by NACE Task Group (TG) 017 on Anodes, Catalyzed Titanium: Testing for Use in Soils or Waters. TG 017 is administered by Specific Technology Group (STG) 05 on Cathodic/Anodic Protection. This standard is issued by NACE under the auspices of STG 05.

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NACE International Standard Test Method

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Section 1: General

1.1 Accelerated testing of catalyzed titanium for use as CP anodes in soils or natural waters is intended to provide an indication of the anode's ability to perform satisfactorily for a specific number of years. Because such anodes can fail with little or no weight loss, lifetime expectation cannot be extrapolated from operation at normal operating current density. Testing must instead be conducted at high current density until an acceptable amount of charge has been accumulated. This test is considered appropriate for catalyzed anodes based on film-forming metals, such as tantalum, niobium, and zirconium, when used for applications in soils or natural waters. Catalysts may be mixed-metal oxides (MMO) or precious metal/metal oxide cermets.

1.2 The anode shall survive the accelerated total charge density that is equivalent to the total charge density for the anode design service requirements. The relationship between life and current density is not linear for these anodes, but rather a log-log relationship, as shown in Figure 1. The total charge density for the accelerated test is numerically smaller than the total charge density calculated for the design service requirement.

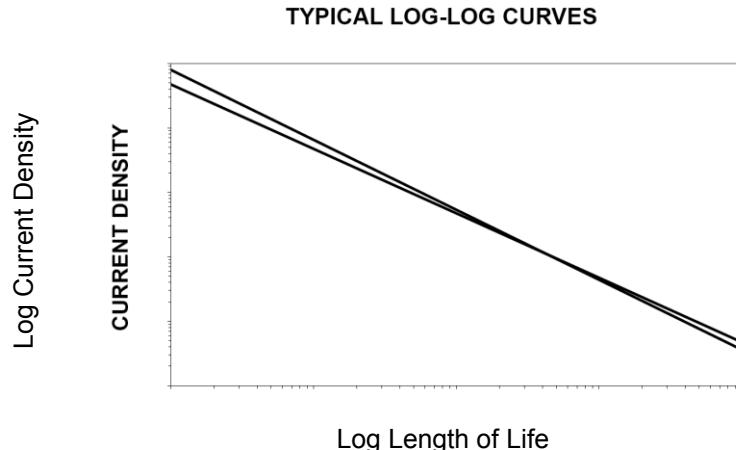


FIGURE 1: Typical Log-Log Curves

1.3 Total charge density is simply the anode life multiplied by the current density. For the accelerated total charge density, it is the current density of the test multiplied by the minimum test duration. For the total design service charge density, it is the current density for the anode in the intended service times the desired service life for the anode.

1.3.1 For example, for an anode intending to be put in service for 20 years at 100 A/m^2 (9.3 A/ft^2), the design service charge density would be $2,000 \text{ A-y/m}^2$ (190 A-y/ft^2) or $17,500,000 \text{ A-h/m}^2$ ($1,630,000 \text{ A-h/ft}^2$). The accelerated total charge density may be determined from the manufacturer's information.

1.4 The electrolyte used for this test shall be an aqueous solution of 1.0 M sodium sulfate. Because this electrolyte is aggressive toward these types of anodes, the results of this test may be considered to be conservative.

1.5 Testing shall be conducted using a full-wave, single-phase, unfiltered power supply with a minimum of 40%

ripple. This condition will simulate the power supplies commonly used for CP in the field.

1.6 An anode specimen with an active surface area of $\sim 500 \text{ mm}^2$ ($\sim 0.78 \text{ in}^2$) should be used for this test, and shall be operated at a test current density so as to verify that the specimen meets the manufacturer's life versus current density curve. If this size or shape of anode specimen is impossible or inappropriate, a different specimen size may be used and current adjusted to provide the current density required.

1.7 At least two specimens of each anode shall be tested in separate cells under identical operating conditions.

1.8 The cell current and voltage, together with anode potential, shall be monitored either automatically or manually during the test period. Anode failure can be detected by a significant rise in cell voltage and anode potential, while operating at the required constant current density.

Section 2: Definitions

Cermet: A composite material or article comprised of a ceramic and a metal, interdistributed in any of various geometrical forms but intimately bonded together.

Charge Density: The product of the current density and time (duration of operation).

Current Density: The current to or from a unit area of an electrode surface.

Luggin Probe: A small tube or capillary filled with electrolyte, terminating close to the metal surface of an electrode under study, which is used to produce an ion-conducting path without diffusion between the electrode under study and a reference electrode.

Ripple: The alternating-current (AC) component in the output of a direct-current (DC) power supply.

Section 3: Test Apparatus

3.1 Test Cell

3.1.1 The test shall be conducted in a tall-form glass 1.0-L beaker that is 180 mm (7.1 in) high and 90 mm (3.5 in) in diameter. Glass beakers of other sizes may be used if the electrodes remain completely immersed for the duration of the test. The top of the beaker shall be fitted with a No. 15 rubber stopper to hold the

thermometer, electrodes, and Luggin probe in place, and to reduce evaporative losses. The gap between the anode and cathode shall be approximately 19 mm (0.75 in). The rubber stopper shall have a hole that is fitted with an extension tube to vent gasses away from the electrical connections. Figure 2 shows a typical setup for a test cell with anode, cathode, and Luggin probe for anode potential measurement.

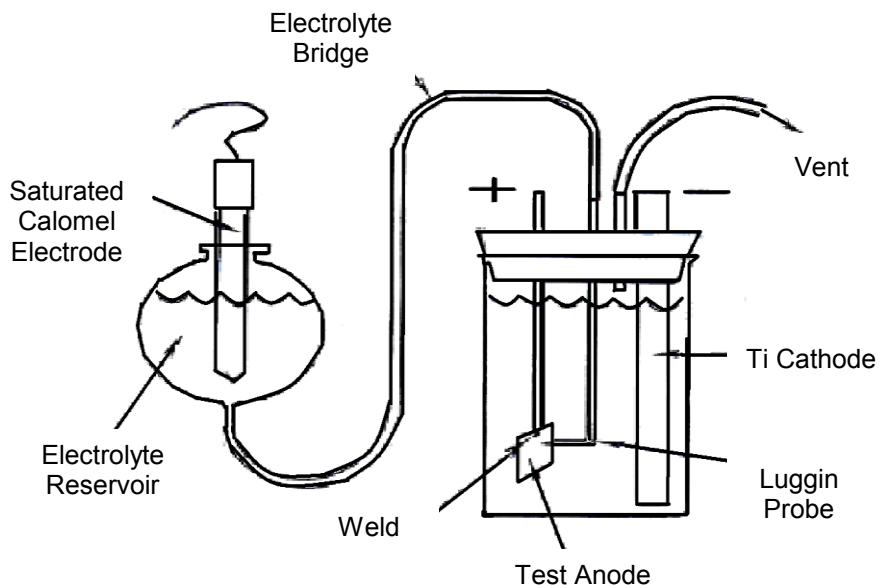


FIGURE 2: Test Cell Setup

3.1.2 A sample of the anode having 500 mm^2 (0.78 in 2) of active anode surface shall be used. If this size or shape of anode specimen is impossible or inappropriate, a different specimen size may be used and the current adjusted to provide the current density as specified in Paragraph 5.5. The active anode surface shall be completely immersed for the duration

of the test. The bottom edge of the anode shall be approximately 10 mm (0.4 in) above the bottom of the beaker. The anode specimen shall be welded to a no smaller than 1.6-mm diameter x 200-mm long (0.063-in diameter x 8-in long) titanium rod as shown in Figure 2. The titanium rod shall be welded to an uncatalyzed area of the test anode so as not to affect the active

area. The titanium rod acts as a current conductor to the anode specimen, and shall not have any catalyst on its surface. External to the cell, the anode current conductor shall be securely connected electrically to an insulated copper wire no smaller than 1 mm² (#16 American Wire Gauge [AWG]). This wire shall in turn be connected to the positive pole of the power supply.

3.1.3 The cathode shall be a 13-mm diameter x 200-mm long (0.50-in diameter x 8-in long) titanium rod. Other inert cathode materials of the same dimensions, such as platinum, niobium, or zirconium, may be used. The cathode shall be fitted through the rubber stopper and extended to approximately 10 mm (0.4 in) from the bottom of the cell. External to the cell, the cathode current conductor shall be securely connected electrically to an insulated copper wire no smaller than 1 mm² (#16 AWG). This wire shall in turn be connected to the negative pole of the power supply.

3.1.4 A Luggin probe shall be installed in the cell with the measuring tip of the probe located about two diameters (of the tip of the probe) away from the center of the anode. The Luggin probe in the cell shall be connected by flexible tubing to an electrolyte reservoir outside the cell. The electrolyte reservoir must be filled with identical solution concentration. The electrolyte reservoir shall be located to maintain an electrolyte level at the same elevation as the electrolyte level in the cell. A suitably stable reference electrode shall be placed in the electrolyte reservoir for the measurement of anode potential. Among acceptable reference electrodes are the saturated calomel electrode and the silver/silver chloride electrode.

3.1.5 A thermometer shall be inserted in each cell. The thermometer shall be either a glass-mercury thermometer, or a glass-encapsulated or polytetrafluoroethylene (PTFE) sheathed thermocouple appropriate for a temperature range of 20 to 100°C (68 to 212°F). Stainless steel or other metallic thermocouples should not be exposed to the electrolyte to avoid corrosion and contamination.

3.2 Power Supply and Electrical Connections.

3.2.1 The power supply shall be a full-wave, single-phase, unfiltered power supply with a minimum of 40% ripple capable of supplying and maintaining a constant current of 5.0 A $\pm 1\%$. This is the typical type of power supply used in the field for CP. Power supply voltage shall be that required to operate the number of cells in electrical series. Necessary voltage may be estimated by multiplying the number of cells in the series by 8.0 V/cell.

3.2.2 Figure 3 illustrates a typical electrical series setup for duplicate evaluations. A separate coulometer or current measuring device with acceptable resolution and tolerance (accurate to $\pm 1\%$) shall be included if not incorporated within the power supply.

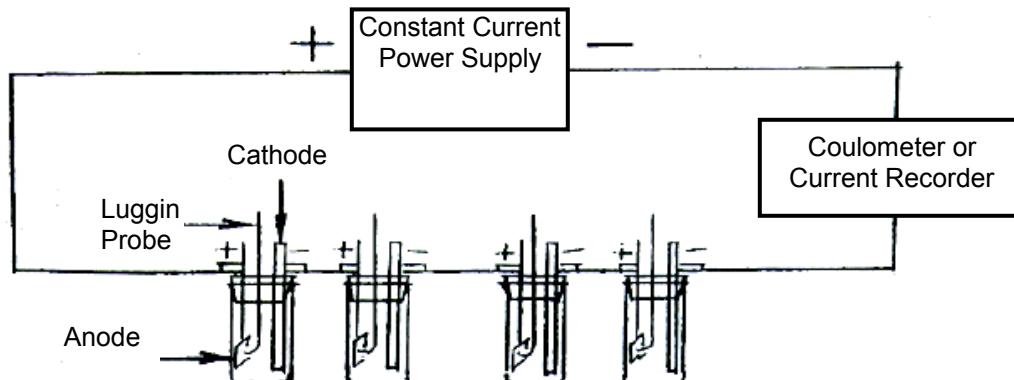


FIGURE 3: Electric Series Setup for Duplicate Evaluations

3.3 The test shall be conducted in a well-ventilated laboratory fume hood. The gas released from the cell is a

potentially explosive mixture of hydrogen and oxygen and must be well ventilated.

Section 4: Test Electrolyte

4.1 A 1-M solution of sodium sulfate (Na_2SO_4) $\pm 5\%$ shall be used.

4.2 Fresh electrolyte shall be used for each anode test.

Section 5: Test Procedure

5.1 The test cell shall be filled with electrolyte to the 900-mL mark. Water lost during the test shall be replaced by distilled or deionized water to maintain an electrolyte level within $\pm 5\%$. The active area of the anode shall remain completely below electrolyte level throughout the test.

5.2 The test sample and apparatus shall be prepared in accordance with Paragraphs 3.1 and 3.2. Care shall be taken that the active area of the anode is completely below the level of the electrolyte and that the anode-cathode gap remains constant throughout the test.

5.3 The Luggin probe and reference electrode electrolyte bridge shall be filled with the same electrolyte used in the cell. Care must be taken during operation that gas bubbles do not collect in the Luggin probe or electrolyte bridge, resulting in a break in the potential measuring circuit.

5.4 The electrical connections shall be made as described in Paragraphs 3.1.2, 3.1.3, and 3.2.2.

5.5 The power supply shall be energized and the current increased to $5.0 \text{ A} \pm 1\%$. This results in a constant current density of 10 kA/m^2 (930 A/ft^2). Current flow shall be confirmed by the production of gas bubbles at both the anode and cathode. Initial current, voltage, and anode potential measurements shall be recorded after about one hour of operation.

5.6 The temperature of the electrolyte shall be maintained at $30 \pm 5^\circ\text{C}$ ($86 \pm 9^\circ\text{F}$) throughout the test. At ambient room temperature, the electrolysis normally heats the electrolyte to approximately $30 \pm 5^\circ\text{C}$ ($86 \pm 9^\circ\text{F}$). As the cell voltage rises toward the end of the life of an anode, cooling by use of a water bath may be required.

5.7 A statistical number of specimens for each anode shall be tested in separate cells under identical test conditions.

5.8 The cell current and voltage, together with anode potential, shall be monitored either automatically or manually in relation to time during the test period. Anode failure can be detected by a significant rise in cell voltage and anode potential, while operating at the required current

density. Failure of one anode in a series of cells may inactivate other cells in the same series. The following data shall be recorded daily:

5.8.1 Cell Voltage: Voltage between the anode and cathode of each single cell. The anode-to-cathode geometry remains constant.

5.8.2 Cell Current: Current flowing through the cell. Current is identical for all the cells in an electrical series.

5.8.3 Anode Potential: Potential measured between the anode of each single cell and the saturated calomel electrode (SCE) reference in close proximity via the Luggin probe. Potential is measured by electrically connecting the positive pole of a high-impedance voltmeter ($10 \text{ M}\Omega$ or greater) to the anode lead of the cell, and the negative pole of the voltmeter to the reference electrode placed in the electrolyte reservoir.

5.8.4 Temperature.

5.8.5 Date and Time.

5.9 Total accumulated charge density shall be measured during the test by an electronic or electrochemical coulometer or continuous current recording device accurate to $\pm 1\%$. If a continuous current recording device is used, the total charge density at the time of anode failure shall be calculated by multiplying cell current by the elapsed time. The total quantity of charge density resulting in the failure of an anode shall be recorded.

5.10 If operation is continued beyond the failure point (as determined by high anode potential), the anode or anode conductor (titanium rod) may dissolve, resulting in an open circuit. The rectifier voltage may be insufficient to maintain the current requirement in the remaining cells. In this case, the cell with the failed anode shall be removed from the circuit and the other cells reactivated. Any time offline for anodes shall be recorded and accounted for when calculating total charge density.

Section 6: Test Completion

6.1 Anode failure is marked by a rapid escalation in both cell voltage and anode potential. It shall be verified that voltage rise is not because of factors other than anode failure. The time of failure shall be recorded when the anode potential increases by 4.0 V above its initial value. Note that there may not be any visual indication at the anode surface of the anode's catalytic coating failure.

6.2 Multiple data points at different charge densities can be plotted on a log-log plot so that the life of the anode can be estimated at current densities other than those tested.

6.3 The use of this test method for various charge density curves and acceptance criteria supplied and published by a reputable anode manufacturer must meet the user's specification and satisfaction. The test method itself provides an acceptable method for testing to these curves and criteria.

Section 7: Reporting Test Results

7.1 Date, time, cell current, cell voltage, anode potential, and temperature shall be reported in tabular form for each anode specimen. If anode failure has been observed, the charge density at the time of anode failure shall be reported.

7.2 Any visible change in anode appearance, such as loss of catalyst, change of color, erosion, or pitting, shall be reported.

7.3 Any deviations from this test method shall be documented in the test report.