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# Accelerated life test method of mixed metal oxide anodes for cathodic protection —

## Part 2: Application in soils and natural waters

*Méthode d'essai accéléré de durabilité des anodes à oxydes métalliques mixtes pour la protection cathodique —*

*Partie 2: Application dans les sols et aux naturelles*

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# Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

A list of all the parts in the ISO 19097 series can be found on the ISO website.

# Introduction

Impressed current cathodic protection (ICCP) is an effective method to control corrosion of metallic structures immersed in seawater, brackish water, and fresh water or buried in soil. ICCP is also widely applied to prevent steel reinforcement in concrete from corrosion in marine or other chloride contaminated environment.

Mixed metal oxide (MMO) anodes have been widely taken as impressed current anodes in the ICCP system due to their good electrocatalytic activity, low consumption rate, long service life, light weight, malleability, high ratio of performance to cost, and wide suitability for different electrolytes.

Durability is one of the most important properties of MMO anodes. ISO 19097 (all parts) examines the accelerated life test method of MMO anodes for cathodic protection to evaluate the anode's ability to achieve an expected lifetime. The accelerated lifetime can also be used to compare the stability of different MMO anodes. This document is applicable to MMO anodes intended for use in underground or underwater environments. This method is not intended to give the exact service life of the anodes in the practical applications, but to provide users and manufacturers of MMO anodes a way to evaluate whether the designed life expectancy can be achieved.

Large portions of this document were derived from NACE International Standard TM0108-2012[1].

# Accelerated life test method of mixed metal oxide anodes for cathodic protection —

# Part 2:
# Application in soils and natural waters

## 1 Scope

This document specifies accelerated life test method of mixed metal oxide anodes for impressed current cathodic protection used in soil or natural waters. The accelerated life test results can be used to compare the durability of the anodes and to evaluate whether the anodes can comply with required specifications of design life expectancy at rated current output.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 8044, *Corrosion of metals and alloys — Basic terms and definitions*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 8044 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

**3.1**
**mixed metal oxide anode**
impressed current anode for cathodic protection consisting of conductive coating of mixed metal oxides formed on titanium substrate

Note 1 to entry: The most common mixture used for cathodic protection is iridium oxide and tantalum oxide. Exact compositions may vary.

**3.2**
**accelerated life**
lifetime of *mixed metal oxide anode* (3.1) under accelerated testing condition, usually in the specific electrolyte applied with large current density

Note 1 to entry: The total period of testing until the deactivation of the mixed metal oxide anode is taken as the accelerated life.

**3.3**
**cell voltage**
voltage between anode and cathode in a single cell

# 4 Test method

## 4.1 Principle

The accelerated life testing of mixed metal oxide anode is conducted in specific simulated environments at a much higher current density than experienced in usual working conditions. This can shorten the period of time to deactivation of the anode significantly.

## 4.2 Test solution

**4.2.1** The composition of the electrolyte used for the test shall be suitable to force the reaction to be the electrolysis of water to produce oxygen at the anode and hydrogen at the cathode. The ionic concentrations shall be suitable to provide sufficient conductivity in the solution to avoid excessive voltage requirements for the power supply.

**4.2.2** Some suitable electrolytes are as follows:

— 1 M sulfuric acid ($H_2SO_4$);

— 1 M sodium sulfate ($Na_2SO_4$);

— 180 g/l sodium sulfate with 0,1 N sulfuric acid to maintain pH at 1.

**4.2.3** Suitable electrolytes should not contain chlorides. The presence of chlorides results in chlorine gas being generated in lieu of oxygen at the anode. Chlorine gas poses handling and safety concerns during testing. Catalysed titanium anodes are much more severely tested for lifetime by oxygen generation than chlorine generation.

**4.2.4** Fresh electrolyte shall be used for each test.

**4.2.5** The concentration shall be maintained at ±5 % of the specified target throughout the test.

**4.2.6** The temperature of the electrolyte shall be maintained at 30 °C ± 5 °C.

## 4.3 Test apparatus

**4.3.1** The test equipment comprises an anode (test sample), a cathode, a thermometer, a glass vessel, electrolyte agitation device (e.g. magnetic stirrer), rubber stopper for tall glass beaker, a funnel for deionized water addition during the test, vent tubing and power supply.

**4.3.2** The glass vessel shall be sized to minimize the fluctuation in level resulting from the electrolysis of water during the test and evaporative losses. A 1 l beaker may be used.

**4.3.3** A means of securing the relative positions of the anode sample, the cathode and the thermometer should be used. This apparatus can be a rubber stopper with holes for the various pieces of equipment and vents or a series of stands and clamps. The gap between the anode and cathode shall be fixed. The anode top and bottom edges shall be at least 10 mm from the liquid level and the bottom of the beaker, respectively.

**4.3.4** The anode-to-cathode gap should be approximately 20 mm.

**4.3.5** The cathode shall be zirconium, titanium, niobium or platinum. The size of the cathode shall be sufficient to extend from the bottom of the beaker to well above the top of the beaker. External to the cell, the cathode current conductor shall be securely connected to an insulated copper wire no smaller than 1 $mm^2$. This wire shall be connected to the negative pole of the power supply.

**4.3.6** A thermometer shall be inserted in the cell. If more than one cell is used, each cell shall have a thermometer installed. The exact type of thermometer used shall be in accordance with the general safety practices of the laboratory and shall not have any materials exposed to the electrolyte that could corrode and contaminate the solution. Alternately, a thermocouple may be used. The temperature range of the thermometer shall be 20 °C to 100 °C. Stainless steel or other metallic thermocouples shall not be exposed to the electrolyte to avoid corrosion and contamination.

**4.3.7** The power supply shall be a constant current control, typical for laboratory use. The required amperage shall be determined from the sample size and the current density for the particular accelerated test point. For example, 5 A is appropriate for a 500 mm$^2$ sample being run at 10 kA/m$^2$. One power supply can also energize several electrolytic cells connected in series at the same time. The required voltage depends on the number of test stations in series that may be used. Typically, 8 V per cell times the number of cells in series is sufficient.

**4.3.8** Use data acquisition device or voltmeter to monitor the voltage of each cell continuously until the failure of the anode. The voltage measuring device shall have a high input impedance of 10 MΩ or greater, and be capable of measuring cell voltage accurate to ±1 %.

**4.3.9** Temperature control equipment may be necessary to maintain the electrolyte within the required range depending on laboratory conditions. Essentially, a fluid may be used to input or extract heat from the electrolyte indirectly. Examples of such equipment are jacketed beakers and water bath for beakers.

NOTE A schematic diagram of test apparatus is shown in Annex A.

## 4.4 Test specimens

**4.4.1** The number and type of test specimens shall be selected according to the specifications for the anode material or product being tested. To ensure the accuracy of the data, usually three, but at least two, duplicate specimens of each anode shall be tested in the separate cells under identical operating conditions.

**4.4.2** The test specimens shall be carefully cleaned prior to testing so as to remove those traces (dirt, oil or other foreign matter) that could influence the result.

Care shall be taken that specimens are not contaminated after cleaning by careless handling.

## 4.5 Test procedure

**4.5.1** 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 shall be well ventilated.

**4.5.2** The test cell shall be filled with electrolyte, making sure that the anode sample is completely immersed in the solution, and agitated with a magnetic stirrer or similar means of ensuring good mixing.

**4.5.3** Water lost during the test shall be replaced by distilled or deionized water to maintain the electrolyte level ±5 %. At no time shall the level be less than that required to maintain the test equipment setup.

**4.5.4** The power supply shall be energized, the current increased to the test setting, and the cell allowed to stabilize for 3 h. Current flow shall be confirmed by the production of gas bubbles at both the anode and the cathode.

If the current changes more than 1 % from the set point, then the test shall be stopped and the cause investigated. The test may be continued after appropriate corrective action.

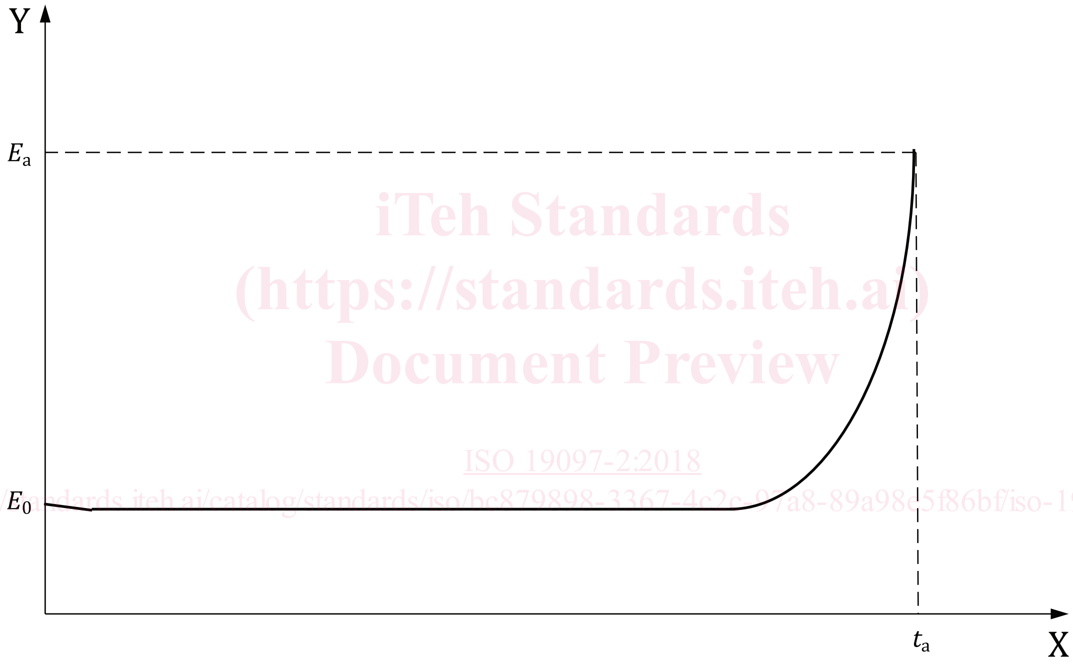
If the cell voltage exceeds the target voltage, then the test shall be paused to allow the removal of the cell from the test station. After removal of the failed cell from the circuit, the test may be started again so that the remaining samples can be tested to completion.

**4.5.5** The temperature of the test solution shall be kept at 30 °C ± 5 °C through the testing period.

**4.5.6** The cell current and voltage shall be monitored and recorded during the test hourly. The test duration should include the 3 h for assuring stabilization of cell operation before recording of voltage.

**4.5.7** A typical curve of cell voltage with electrolysis time is shown in Figure 1. Anode failure is marked by a rapid escalation in cell voltage. The specific cell voltage is dependent on the selected current density and test conditions. The test is stopped when the testing anode sample is failed.

**4.5.8** When the cell voltage $E_a$ is 1,5 V higher than the starting cell voltage $E_0$ (starting voltage is the cell voltage 3 h after test), the test anode can be taken as failed. The period from the beginning of the test to the failure of the anode is the accelerated life time $t_a$ (see Figure 1).



**Key**

| | |
|---|---|
| X | electrolysis time |
| Y | cell voltage |

**Figure 1 — Typical curve of cell voltage against electrolysis time**

**4.5.9** It should be verified that the voltage rise is not the result of any other factor than anode failure, e.g. loose electrical connection.

**4.5.10** A reliable power supply shall be used to make sure that current fluctuations and power outages are avoided during the test.

**4.5.11** If the test is to be compared to a correlation curve (see Annex B), then the test may be stopped after sufficient time has passed to demonstrate that the product is satisfactory for the intended design.