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

**Part 1:
Application in concrete**

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

Partie 1: Application dans le béton

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 concrete. 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 TM0294-2016[1].

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

Part 1: Application in concrete

1 Scope

This document specifies accelerated life test method of mixed metal oxide anodes for impressed current cathodic protection used in concrete. 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.

This document is also applicable to other anode systems that are used as impressed current anodes embedded in concrete with suitably modified apparatus to hold anodes of different geometry.

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 679, *Cement — Test methods — Determination of strength*

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 conditions, 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

3.4

charge density

product of applied current density multiplied by operating time

3.5

ripple

alternating current (AC) component in the output of a direct current (DC) power supply, arising within the power supply from incomplete filtering or from commutator action in a DC generator

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.

This document includes two parts of a test method for evaluating the anode material.

Test Method Part A is designed to evaluate whether the mixed metal oxide anode material can meet an expected lifetime criterion. It is possible that an anode may suffer from current reversal caused by erroneously connecting as a cathode or other reasons during the initial stage in practice. Therefore, current reversal testing should be conducted for a period of time before the later testing to ensure the ability of the anode to survive a brief current reversal.

Test Method Part B is a quicker test to ensure that a sample from a particular batch of anode material is suitable. It shall only be conducted on samples of a product that has passed Test Method Part A for the required design life.

4.2 Test solutions

4.2.1 General

Accelerated life testing cannot be conducted in concrete because testing at high-current levels results in premature failure of the concrete as the test electrolyte. Accelerated life testing shall therefore be conducted in an aqueous solution. The following clauses give instructions for the preparation and use of the simulated concrete electrolyte in Test Method Part A, and test solutions in Test Method Part B. All the chemicals used are reagent grade and water is distilled or deionized. All the solutions shall be freshly prepared for each test.

4.2.2 Sodium chloride (NaCl) solution

A mass of $30,0 \text{ g} \pm 0,1 \text{ g}$ sodium chloride shall be added to a 1,0 l volumetric flask, then about 500 ml of distilled or deionized water added to the above. The solution shall be swirled in the flask until the NaCl crystals are totally dissolved. More distilled or deionized water shall be added to the level of the 1,0 l mark in the flask to produce a concentration of 30 g/l NaCl solution. The solution shall be thoroughly mixed.

In the cathodic protection for reinforced concrete structures like bridge piers and pilings in marine or chloride contaminated environment, the mixed metal oxide anode is exposed to the solution containing chloride ions. So sodium chloride solution is used to simulate these environments and test the ability of the mixed metal oxide anodes to tolerate chlorine evolution reaction.

4.2.3 Sodium hydroxide (NaOH) solution

An amount of $40,0\text{ g} \pm 0,1\text{ g}$ sodium hydroxide shall be slowly added to a 1,0 l volumetric flask containing approximately 500 ml of distilled water or deionized water. The solution shall be swirled until the NaOH is totally dissolved. This reaction is exothermic and generates heat. More distilled water or deionized water shall be filled in the flask just under the 1,0 l level and the solution shall be left to cool to room temperature. Finally, the flask shall be filled to the 1,0 l mark with distilled water or deionized water to form the solution of 40 g/l NaOH. The solution shall be thoroughly mixed.

In new reinforced concrete, the pH of electrolyte solution is high with little contamination of chloride in the environment. This solution is used to simulate actual environment in new concrete and to measure the ability of the anode to tolerate oxygen evolution reaction, which is more favoured at low level of chloride contamination experienced with fresh overlays.

4.2.4 Simulated pore water in sand

A mass of $26,3\text{ g} \pm 0,1\text{ g}$ sodium hydroxide, $10,74\text{ g} \pm 0,1\text{ g}$ potassium hydroxide, $34,35\text{ g} \pm 0,1\text{ g}$ potassium chloride and $2,15\text{ g} \pm 0,01\text{ g}$ calcium hydroxide shall be dissolved in 1,0 l of distilled water or deionized water one by one in a flask, stirred until no solid left to form the simulated concrete pore solution. The composition by mass of the simulated pore water used shall be as follows:

- a) 0,20 % $\text{Ca}(\text{OH})_2$;
- b) 3,20 % KCl;
- c) 1,00 % KOH;
- d) 2,45 % NaOH;
- e) 93,15 % distilled or deionized water.

Fine natural silica sand (270 μm to 380 μm) shall be obtained in accordance with ISO 679. The test cell shall first be filled with enough sand to cover the anode completely after the electrodes and Luggin probe are in place. The simulated pore water shall then be added to displace any air and fill the remainder of the cell.

The electrolyte available to the embedded mixed metal oxide anode in a cured concrete structure is pore water. This solution tests the ability of the anode to tolerate the actual concentrations of the pore water components and any possible synergistic effects imposed by these components. The use of fine sand to encompass the electrode, eliminating convective mixing, tests the ability of the anode to tolerate the situation most closely simulating its operation in cured concrete.

4.2.5 Other testing solutions

The composition of the electrolyte used for Test Method Part B shall be suitable to force the reaction of oxygen evolution at the anode. The ionic concentrations shall be suitable to provide sufficient conductivity in the solution to avoid excessive voltage requirements for the power supply.

Some suitable solutions 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.

4.3 Test apparatus

NOTE A schematic diagram of test apparatus is shown in [Annex A](#).

4.3.1 The test cell shall be a tall-form glass 1,0 l beaker fitted with a rubber stopper at the top to hold the electrodes and reduce air contact. Glass beakers of other sizes may be used as long as electrodes remain immersed for the duration of the test. The gap between the anode and cathode shall be approximately 50 mm. The rubber stopper shall have a hole located midway between the electrodes that is fitted with an extension tube to vent gases away from the electrical connections. This hole may be used to locate the current reverse supplemental anode and Luggin probe. There shall be an additional hole large enough to measure pH of the test solution. A typical setup for a test cell taking a reference electrode with Luggin probe for anode potential measurement is shown in [Annex A](#).

4.3.2 A sample of the anode having 2 000 mm² of anode surface area shall be used. The anode surface area shall be calculated by including all the active surfaces that will be in contact with concrete when embedded. The anode sample shall be welded to a 1,6 mm diameter x 203 mm long Ti rod in two spots as shown in [Figure A.1](#). The Ti rod acts as the current carrier. For other types of anodes, an appropriate anode connection shall be made in accordance with the anode manufacturer's recommendations. The anode shall be connected to the positive lead (the negative lead during the current reversal test) of the power supply external to the cell using an insulated 1 mm diameter copper wire with a copper spring-loaded clip with jaws.

4.3.3 The cathode shall be a 12,7 mm diameter × 200 mm long Ti rod. Other shape of cathode like Ti plate with enough surface area may also be applicable. Alternatively, relatively inert cathodes such as platinum or niobium or zirconium may also be used. The cathode shall be fitted through the rubber stopper and extended to approximately 10 mm from the bottom of the cell. The cathode shall be connected to the negative lead of the power supply (not connected during the current reversal test) external to the cell using an insulated 1 mm diameter copper wire with a copper spring-loaded clip with jaws.

4.3.4 A supplemental anode is required for the current reversal portion of the testing. This anode shall be identical to the one being life tested and shall be referred to as the current reversal (CR) anode. The CR anode should be installed midway between the cathode rod and the anode to be life tested. During the current reversal test, the CR anode shall be connected to the positive lead of the power supply external to the cell using an insulated 1 mm diameter copper wire with a copper spring-loaded clip with jaws or similar type fastener. The CR anode shall be removed from the cell during normal testing.

4.3.5 The DC power supply shall be a constant current control type filtered with a maximum ripple of 5 %. One power supply can also energize several electrolytic cells connected in series at the same time. The required voltage depends on the testing current and the number of test stations in series that may be used. Typically, 8 volts per cell times the number of cells in series is sufficient.

4.3.6 [Figure A.2](#) illustrates a typical setup for a series electrical hookup for duplicate evaluations.

4.3.7 A voltmeter shall be used to measure the anode potential and the voltage of each cell at the specified intervals. The voltmeter shall have a high input impedance of 10 MΩ or greater and shall be capable of measuring cell voltage with an accuracy of ±1 %. A data logging voltmeter may also be used.

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, 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), which could influence the result. Care shall be taken that specimens are not contaminated after cleaning by careless handling.

4.5 Test conditions

4.5.1 The test conditions used in Test Method Part A and Test Method Part B are shown in [Table 1](#).

Table 1 — Test conditions for accelerated life test of mixed metal oxide anode

Test method	Test electrolytes	Test conditions
Test Method Part A	Simulated concrete electrolytes (see 4.2.2 , 4.2.3 , 4.2.4)	Anode shape: mesh, ribbon or plate. Anode surface area: 2 000 mm ² ; Cathode shape: rod or plate. Typical cathode size: φ12,7 mm × 200 mm; The gap between the anode and cathode: 50 mm; Electrolyte temperature: 20 °C ± 5 °C; Current density: for the accelerated life tests, the anode sample is first operated at reversed (cathodic) current density of 8,9 A/m ² for 8 h, and then continues to be tested to the end under positive polarity at 8,9 A/m ² (see 4.5.2.3).
Test Method Part B	Some suitable electrolytes are (see 4.2.5): a) 1 M sulfuric acid (H ₂ SO ₄); b) 1 M sodium sulfate (Na ₂ SO ₄); c) 80 g/l sodium sulfate with 0,1 N sulfuric acid to maintain pH at 1.	Anode shape: mesh, ribbon or plate. Anode surface area: 2 000 mm ² ; Cathode shape: rod or plate, Typical cathode size: φ12,7 mm × 200 mm; The gap between the anode and cathode: 50 mm; Electrolyte temperature: 30 °C ± 5 °C; Current density: 1,45 kA/m ² .

4.5.2 Test procedure

4.5.2.1 The test shall be conducted in a well-ventilated laboratory fume hood. The gas released from the cell shall be well ventilated. A small volume of air shall be injected into the cell to dilute the hydrogen/oxygen gas mixture in Test Method Part B due to high current density.

4.5.2.2 The test cell shall be filled with fresh electrolyte, making sure that the anode sample is completely immersed in the solution. The test solution shall be kept at the required temperature through the testing period. Water evaporated 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.

4.5.2.3 Procedure for Test Method Part A

- The anode material shall be tested in the three different simulated concrete electrolytes described in [4.2.2](#), [4.2.3](#) and [4.2.4](#).
- The current reversal test shall be conducted first. The anode to be life tested shall be tested under reverse, or cathodic, polarity for 8 h at 17,8 mA (with a current density of 8,9 A/m²). This results in a total charge density of 71 A h/m² of anode surface (or the equivalent of about one month of operation at an anode current density of 108 mA/m², which is usually the maximum design current density of MMO anode for reinforcement cathodic protection in practice).

- c) During the current reversal portion of the test, cell voltage and cell current shall be measured at 1 min, 1 h and 8 h. The cell voltage measurements shall be obtained by attaching the leads from a voltmeter to the test electrode and the CR anode.
- d) After completing the current reversal test, the power supply leads shall be changed so that the positive lead is connected to the anode being life tested and the negative lead is connected to the cathode rod. The CR anode shall be removed from the cell. The Luggin probe shall be installed for anode potential measurements with a reference electrode. The tip of the Luggin probe shall be two diameters of the probe tip away from the surface of the anode. The cells requiring sand shall be completely emptied and refilled with fresh sand and test electrolyte in order to accomplish all tasks.

NOTE Although potentials are quoted on the SCE (saturated calomel electrode) scale, the SCE is no longer recommended for use due to it containing mercury. Silver/silver chloride/potassium chloride electrodes are widely used but their potentials vary with the concentration of potassium chloride used and so are calibrated against a suitable standard reference electrode.

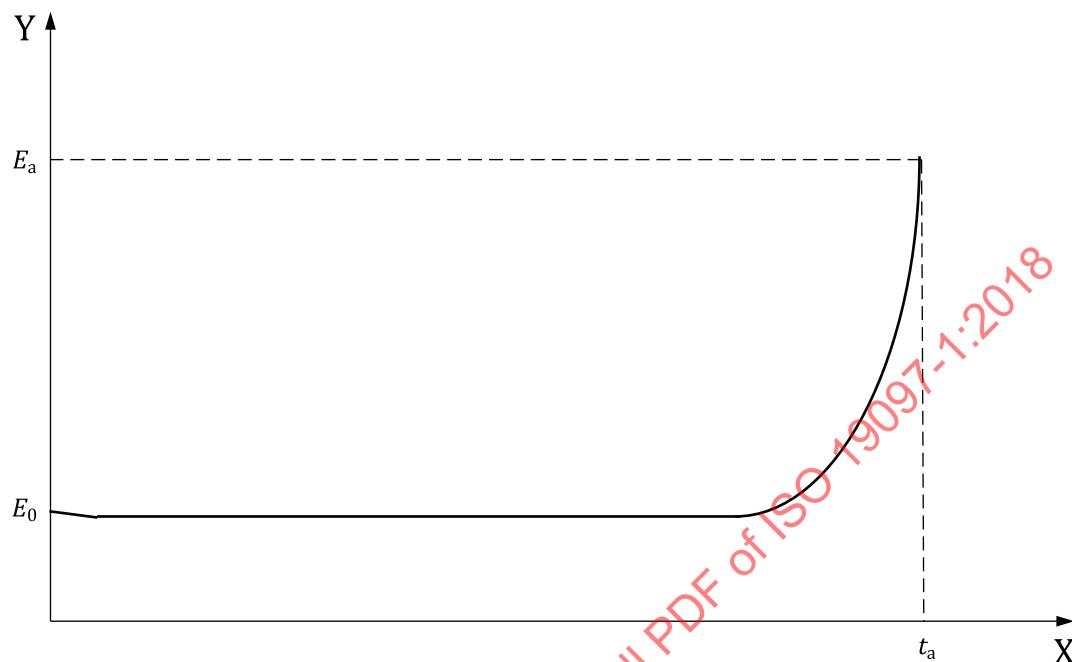
- e) The test anode should then be operated under normal, or positive, polarity at 17,8 mA (with a current density of 8,9 A/m²).
- f) The following parameters shall be measured during the normal, or positive, polarity portion of the test: cell voltage, cell current, anode potential shall be measured at 1 h, 24 h, 7 d, 14 d, 28 d, 42 d, 56 d, 70 d, 84 d, 98 d, 112 d, 126 d, 140 d, 154 d, 168 d and 180 d. The electrolyte pH shall be recorded at the beginning of the test, and then measured periodically (for example, approximately every 100 h).
- g) The total quantity of charge passed during the test shall be measured to an accuracy of $\pm 1\%$. The total charge at the end of the test shall confirm the minimum total charge density of 38,500 A h/m² of anode surface (the equivalent of 40 years of operation at an anode current density of 108 mA/m²), or greater if required by the user. If the anode has failed (see 4.5.2.6), both measured and calculated charges shall be recorded.
- h) The anodic portion of life testing shall be conducted over a period of at least 180 d. If the required life testing period exceeds 180 d, it should be run in consecutive cycles, each one conducted over a period of at least 180 d. At the beginning of each additional cycle, the aqueous solutions shall be refreshed. After the first cycle is performed, a new reversal current test is not required.

4.5.2.4 Procedure for Test Method Part B

- a) Test Method Part B shall only be conducted on samples of a product that have passed Test Method Part A for the required design life.
- b) The suitable electrolytes described in 4.2.5 should be selected for the Part B testing.
- c) The anode shall be tested under normal or positive polarity continuously without any current reversal. The test anode current density is 1,45 kA/m².
- d) Cell voltages or anode potential shall be recorded every 30 min by computer data acquisition. The test shall be terminated either having reached the required test time or at the time of anode failure with a sharp increase in cell voltage or anode potential.
- e) The minimum test period is 65 h for anodes with an expected design life of 100 years, or 78 h for anodes with an expected design life of 120 years at a maximum current density of 108 mA/m².

4.5.2.5 For multiple cells in series, if the cell voltage exceeds the target voltage with the testing anode failed, 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.

4.5.2.6 A typical curve of cell voltage with electrolysis time is shown in [Figure 1](#). Anode failure is marked by a rapid escalation in both cell voltage and anode potential. The time of failure should be recorded.



Key

X electrolysis time

Y cell voltage

Figure 1 — Typical curve of cell voltage against electrolysis time

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

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

4.5.2.10 The solutions shall be properly neutralized and disposed of after completion of the test.

4.6 Continuity of tests

Tests shall preferably be continued without interruption throughout the test duration. When it is necessary to interrupt the operation for the removal of specimens for inspection, the interruption time shall be minimized.

If a longer period of interruption is unavoidable, the test specimens shall be taken out of the electrolyte and dried as soon as the test is interrupted. The test specimens should be kept in desiccator until the test restarts.

4.7 Treatment of specimens after test

At the end of the test period, the test specimens shall be removed from the electrolyte, washed with distilled or deionized water and dried.

5 Reporting test results

Applied current, cell voltage and other parameters shall be reported in tabular form for each anode sample, examples are shown in [Annex B](#). The interruption and replacement of solution shall be remarked. The variation of cell voltage or anode potential with electrolysis time can also be presented in a graph (see [Figure 1](#)).

Any other relevant information, such as observer(s), date, change of electrolyte colour, photographic records or descriptions of the appearance of tested specimens before and after testing, shall also be recorded. The total charge density of each anode sample at the time of fail or at the end of test shall be reported.

The shape and dimensions of the anode specimens shall be described. The material, shape and dimensions of the cathode should also be reported. Any deviation from this standard testing method shall be documented in the report.

6 Application of results

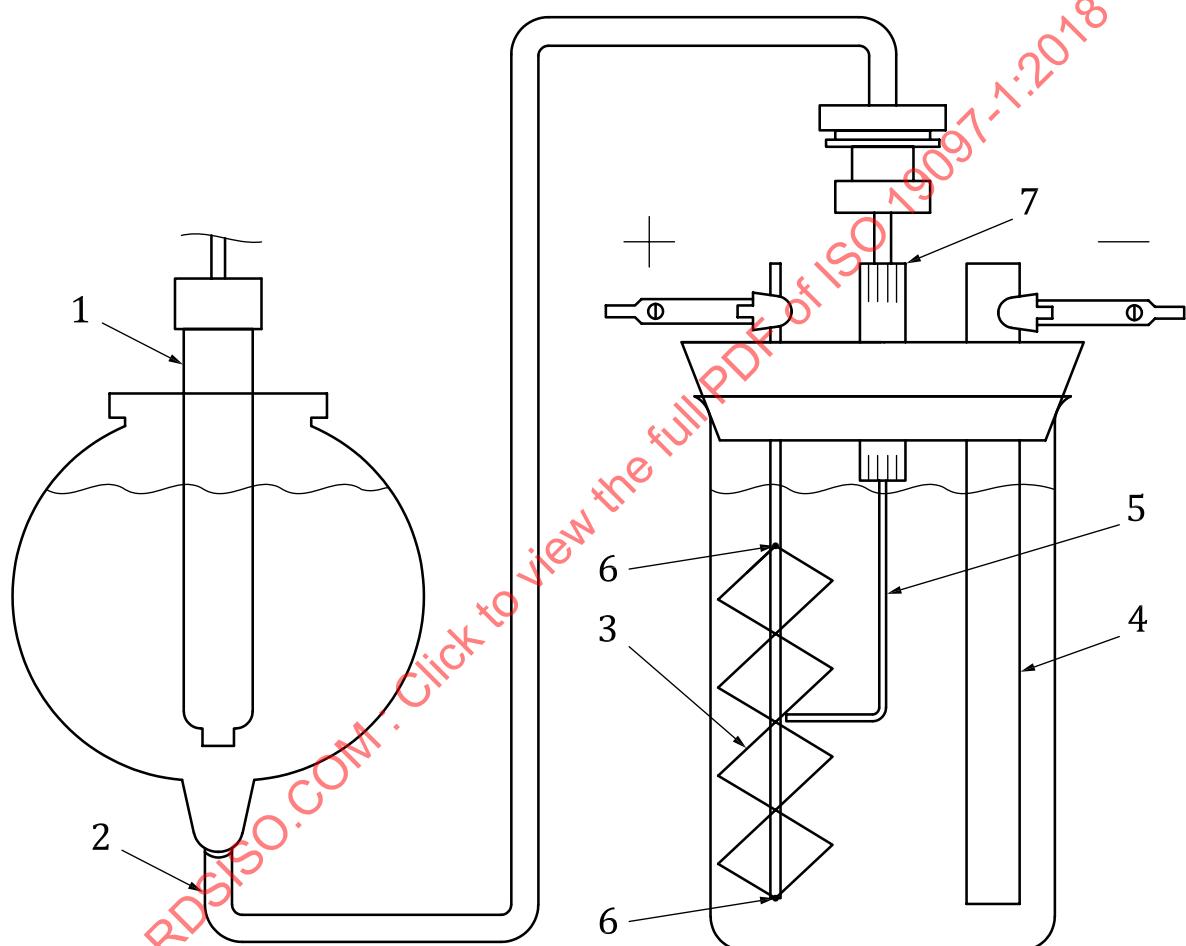
The test results can be used for comparison of anode stability between different anodes under the same specified test conditions. The accelerated life test results can also be used to evaluate if the anode can meet the design requirements of life expectancy at the specified current density. The accelerated life test produces a conservative result due to the more severe stressing on the anode than that at normal application.

Annex A

(informative)

Typical apparatus for accelerated life test

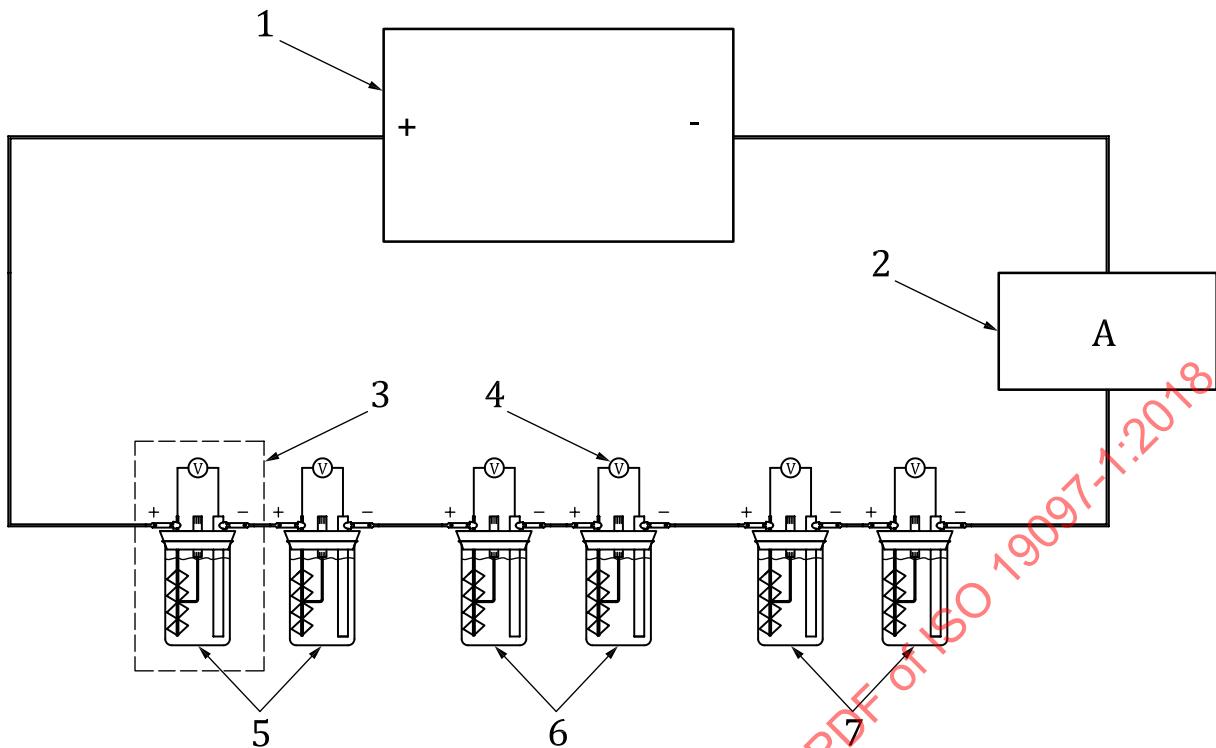
The typical apparatus for the accelerated life test of MMO anodes used in concrete is shown in [Figures A.1](#) and [A.2](#).



Key

- 1 reference electrode
- 2 electrolyte bridge
- 3 test anode
- 4 cathode
- 5 luggin probe
- 6 weld
- 7 vent

Figure A.1 — Test cell with Luggin probe anode potential measurement setup



Key

- 1 constant current power supply
- 2 current recorder
- 3 single testing cell
- 4 cell voltage recorder
- 5 30 g/l NaCl
- 6 40g/l NaOH
- 7 simulated pore water with sand

Figure A.2 — Series electrical hookup for duplicate evaluations

Annex B

(informative)

The typical testing results of mixed metal oxide anode

The typical testing results of MMO anode samples in tabular form are shown in [Tables B.1, B.2](#) and [B.3](#).

Table B.1 — The typical results of MMO anode sample tested in 30 g/l NaCl solution

Test	Time	Cell current (mA)	Cell voltage (V)	Anode potential vs.SCE(V)	Solution pH
Reverse current	1 min	17,8	1,91		
	1 h	17,8	2,40		
	8 h	17,8	2,23		
Normal current	1 h	17,8	2,53	1,181	6,4
	24 h	17,8	2,49	1,177	7,0
	7 d	17,8	2,28	1,187	7,6
	14 d	17,8	2,45	1,333	7,9
	28 d	17,8	2,61	1,456	8,2
	42 d	17,8	2,62	1,484	8,0
	56 d	17,8	2,61	1,480	8,1
	70 d	17,8	2,64	1,508	8,7
	84 d	17,8	2,65	1,520	8,0
	98 d	17,8	2,80	1,534	8,0
	112 d	17,8	2,72	1,551	8,4
	126 d	17,8	2,67	1,536	8,0
	140 d	17,8	2,74	1,583	7,8
	154 d	17,8	2,71	1,550	8,2
	168 d	17,8	2,76	1,591	8,0
	180 d	17,8	2,71	1,579	7,8