
**Dentistry — Corrosion test methods for
metallic materials**

*Médecine bucco-dentaire — Méthodes d'essai de corrosion des
matériaux métalliques*

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 10271 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 2, *Prosthetic materials*.

This second edition cancels and replaces the first edition (ISO 10271:2001), which has been technically revised, in particular by the inclusion of two additional test methods. It also incorporates Technical Corrigendum ISO 10271:2001/Cor.1:2005.

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Introduction

This International Standard was developed from the original Technical Report (ISO/TR 10271) as a result of worldwide demand for standard test methods to determine acceptability of metallic materials for oral restorations in relation to corrosion.

Specific qualitative and quantitative requirements for freedom from biological hazard are not included in this International Standard, but it is recommended that reference be made to ISO 10993-1 and ISO 7405 for assessing possible biological or toxicological hazards.

The testing of the corrosion behavior of metallic materials in dentistry is complicated by the diversity of the materials themselves, their applications and the environment to which they are exposed. Variation occurs between devices and within the same device during the exposure time. The type of corrosion behavior or effect can also vary with exposure time. Accordingly, it is not possible to specify a single test capable of covering all situations, nor is it a practical proposition to define a test for each situation. This International Standard, therefore, gives detailed procedures for test methods that have been found to be of merit as evidenced by considerable use.

This second edition differs from the first edition by the addition of two new test methods. To supplement the existing static immersion test, a static immersion test with periodic analysis has been added. A major reason for the addition of this test is that the rate of corrosion of most dental metallic materials varies over time. Thus, the aim of this supplementary test is to provide information on this variation in the corrosion of a dental metallic material. A classification scheme to interpret the rate of corrosion of a tested material with time (i.e. steady, decreasing, increasing) was not included as part of the static immersion test with periodic analysis. It is intended to monitor the use of the test through appropriate working groups of ISO/TC 106 to ascertain whether a classification scheme is needed in a future revision of this International Standard.

To supplement the sulfide tarnish test (cyclic immersion), a sulfide tarnish test (static immersion) has also been added to this second edition of ISO 10271. This test has been used successfully for many years to evaluate the corrosion of silver alloys.

In addition, an informative annex (Annex A) is provided that sets out a procedure for each element of the test system such that a consistent approach can be taken for the development of further test methods. Equally, it is recognized that any element can represent only the current recommendation, but changes in the future are unlikely to change the framework.

It is not the purpose of this International Standard to propose corrosion test methods for specific applications or to set limits as precise as those in the standard relating to the type of product and its application.

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Dentistry — Corrosion test methods for metallic materials

1 Scope

This International Standard provides test methods and procedures to determine the corrosion behavior of metallic materials used in the oral cavity. It is intended that the test methods and procedures in this International Standard be referred to in the individual International Standards specifying such metallic materials.

This International Standard is not applicable to instruments and dental amalgam and appliances for orthodontics.

2 Normative references

The following referenced documents are indispensable for the application 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 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 1942, *Dentistry — Vocabulary*

ISO 3585, *Borosilicate glass 3.3 — Properties*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 6344-1, *Coated abrasives — Grain size analysis — Part 1: Grain size distribution test*

ISO 7183, *Compressed-air dryers — Specifications and testing*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1942 and the following apply.

3.1

breakdown potential

E_p

least noble potential at which pitting or crevice corrosion, or both, initiates and propagates

3.2

corrosion

physicochemical interaction between a metallic material and its environment that results in a partial or total destruction of the material or in a change of its properties

**3.3
corrosion potential**

E_{corr}

open-circuit potential measured under either service conditions, or laboratory conditions that closely approximate service conditions

**3.4
corrosion product**

substance formed as a result of corrosion

**3.5
crevice corrosion**

corrosion associated with and taking place in or near a narrow aperture or crevice

**3.6
current density**

value of electric current per unit surface area flowing through a conductor

**3.7
dynamic immersion test**

test in which the sample is exposed to a corrosive solution under conditions of relative motion between sample and solution

**3.8
electrode potential**

potential difference between the sample and a reference electrode

**3.9
electrolyte**

solution or liquid that conducts an electrical current by means of ions

**3.10
open-circuit potential**

E_{ocp}

potential of an electrode measured with respect to a reference electrode or another electrode when no current flows

**3.11
pitting corrosion**

localized corrosion which results in pits

**3.12
potentiodynamic test**

test in which the electrode potential is varied at a preprogrammed rate and the relationship between current density and electrode potential is recorded

**3.13
potentiostatic test**

test in which the electrode potential is maintained constant

**3.14
sample**

totality of material for one type being tested, the group of all such specimens

**3.15
set**

subgroup of the specimens of a sample

3.16**specimen**

individual single example of an object for testing

3.17**static immersion test**

test in which the sample is exposed to a corrosive solution under conditions of effectively no relative motion between sample and solution

3.18**stress corrosion**

corrosion resulting from the combined action of static tensile stress and an electrolyte

3.19**synthetic saliva**

test medium that simulates the chemistry of natural saliva

3.20**tarnish**

surface discoloration due to the chemical reaction between a metallic material and its environment

3.21**zero-current potential**

potential at which cathodic and anodic currents are equal

4 Test methods**4.1 Static immersion test****4.1.1 Information required**

Composition, including hazardous elements, in accordance with the appropriate ISO standard is required.

4.1.2 Application

This is an accelerated test that is intended to provide quantitative data on the metallic ion released from metallic materials under *in vitro* conditions relevant to those expected in the oral cavity.

4.1.3 Reagents

4.1.3.1 Lactic acid ($C_3H_6O_3$), 90 %, analytical grade.

4.1.3.2 Sodium chloride (NaCl), analytical grade.

4.1.3.3 Water, complying with grade 2 of ISO 3696.

4.1.3.4 Ethanol or methanol (C_2H_5OH or CH_3OH), analytical grade.

4.1.3.5 Compressed air, oil- and water-free, complying with ISO 7183.

4.1.4 Apparatus

4.1.4.1 Containers, of borosilicate glass, complying with ISO 3585 and with dimensions of approximately 16 mm inner diameter by 160 mm in height.

4.1.4.2 pH meter, with a sensitivity of at least $\pm 0,05$ pH units.

4.1.4.3 Chemical analysis instrumentation, capable of measuring ion concentration in $\mu\text{g/ml}$ (e.g. ICP and AAS).

4.1.4.4 Micrometer gauge, accurate to 0,01 mm.

4.1.4.5 Silicon carbide paper, complying with ISO 6344-1.

4.1.4.6 Volumetric flasks, of borosilicate glass, 1 000 ml, class A, complying with ISO 1042.

4.1.5 Solution preparation

Prepare an aqueous solution comprising 0,1 mol/l lactic acid and 0,1 mol/l sodium chloride immediately before use. For example, dissolve $(10,0 \pm 0,1)$ g 90 % $\text{C}_3\text{H}_6\text{O}_3$ (4.1.3.1) and $(5,85 \pm 0,005)$ g NaCl (4.1.3.2) in approximately 300 ml of water (4.1.3.3). Transfer into a 1 000 ml volumetric flask (4.1.4.6) and fill to mark. The pH shall be $2,3 \pm 0,1$. If not, the solution shall be discarded and reagents checked.

4.1.6 Samples

4.1.6.1 Fabrication

4.1.6.1.1 Cast

Specimens shall be cast in accordance with the manufacturer's recommendations.

4.1.6.1.2 Prefabricated

Prefabricated parts/devices shall be used in the as-received condition.

4.1.6.1.3 Other

Specimens prepared by other methods, e.g. machined, sintered, eroded, etc., shall be tested in the as-manufactured condition after suitable cleaning.

4.1.6.2 Sampling

The number of specimens shall be sufficient to provide at least two parallel sets. (The number of specimens in a set may vary.)

4.1.6.3 Sample surface area

The total surface area of the sample shall be at least 10 cm^2 after preparation.

4.1.6.4 Preparation

4.1.6.4.1 Cast samples

Remove any sprues, runners or other projections from surface. Blast surfaces with $125 \mu\text{m}$ pure alumina to remove investment.

If recommended, heat-treat according to the manufacturer's instructions.

In the case of metal-ceramic materials, heat-treat for 10 min at the highest firing temperature recommended by the metallic material manufacturer and bench cool.

Remove at least 0,1 mm, measured using a measuring instrument [e.g. micrometer gauge (4.1.4.4)], from each surface using standard metallographic procedures unless specimens are being tested in the as-received

condition. Use fresh abrasive paper for each metallic material. Finish with P1200 wet silicon carbide paper (4.1.4.5). If the described procedure is not applicable, treat the surfaces according to the manufacturer's instructions for clinical use.

Determine each sample surface area to the nearest 1 %.

Clean surfaces ultrasonically for 2 min in ethanol or methanol (4.1.3.4). Rinse with water (4.1.3.3). Dry with oil- and water-free compressed air (4.1.3.5).

If a specimen contains any porosity visible on the surfaces being exposed to the solution, the specimen shall be rejected and replaced with a new one.

4.1.6.4.2 Machined, sintered, eroded or electroformed samples

Heat-treat the specimens if recommended.

Remove at least 0,1 mm, measured using a measuring instrument [e.g. micrometer gauge (4.1.4.4)], from each surface using standard metallographic procedures unless specimens are being tested in the as-received condition. Use fresh abrasive paper for each metallic material. Finish with P1200 wet silicon carbide paper (4.1.4.5).

Determine each sample surface area to within $\pm 0,1$ cm².

Clean surfaces ultrasonically for 2 min in ethanol or methanol (4.1.3.4).

Rinse with water (4.1.3.3). Dry with oil- and water-free compressed air (4.1.3.5).

4.1.6.4.3 Prefabricated parts/devices

Treat the surfaces according to the manufacturer's instructions for clinical use.

Determine each sample surface area to within $\pm 0,1$ cm².

Clean the surfaces ultrasonically for 2 min in ethanol or methanol (4.1.3.4).

Rinse with water (4.1.3.3). Dry with oil- and water-free compressed air (4.1.3.5).

4.1.7 Test procedure

Parallel specimen sets shall be treated in identical fashion. If a set consists of one specimen, it shall be placed in a container (4.1.4.1) such that it does not touch the container surface except in a minimum support line or point. If a set consists of two or more specimens, they may be placed in the same or a number of separate containers, but if more than one is placed in a container they shall not touch.

Record the pH of the solution. Add the solution to each container sufficient to produce a ratio of 1 ml of solution per 1 cm² of sample surface area. The specimens shall be covered completely by the solution. Record the volume of solution to an accuracy of 0,1 ml. Close the container to prevent evaporation of the solution. Maintain at (37 ± 1) °C for $7 \text{ d} \pm 1 \text{ h}$. Remove the specimens and record the pH of the residual solution.

Use an additional container (4.1.4.1) to hold a reference solution to be maintained in parallel with the solutions containing the specimens. The reference solution shall be used to establish the impurity level for each element of interest in the solution. Add approximately the same volume of solution as used for the solutions containing the specimens and record the volume to an accuracy of 0,1 ml. Close the container to prevent evaporation of the solution and maintain at (37 ± 1) °C for the same time period as the solutions containing the specimens.

4.1.8 Elemental analysis

Use chemical analysis instrumentation (4.1.4.3) of adequate sensitivity. Analyze the solution qualitatively and quantitatively. Emphasis shall be on those elements indicated in 4.1.1, but if impurities are found in a concentration greater than 0,1 %, they shall also be reported. For each element of interest, subtract the value obtained for the element in the reference solution from the value obtained in the test solution. The elements boron, carbon, and nitrogen shall be disregarded.

4.1.9 Test report

The test report shall contain the following information.

- a) Report the method of analysis and detection limits of all the analyzed elements.
- b) Report and justify any deviations from preparation of specimens (see 4.1.6.4) or test procedure (see 4.1.7).
- c) Report the number of specimens making up a set and the number of sets tested (see 4.1.6.2).
- d) From the elemental analysis of the corrosion solution (see 4.1.8), calculate and report the ion release for each element of each set (see 4.1.6.2) separately in $\mu\text{g}/\text{cm}^2/7 \text{ d}$. The elements indicated in 4.1.1 shall be reported as well as any others found. From this information, sum the ion release values for all the elements of each set to obtain the total metal ion release for the dental material. Average the results by dividing by the number of sets tested and report as the average total ion release for the dental material in $\mu\text{g}/\text{cm}^2/7 \text{ d}$.
- e) Report undissolved corrosion product like precipitates and discoloration of the specimen surface.

4.2 Electrochemical test

4.2.1 Information required

Composition, including hazardous elements, in accordance with the appropriate material standard is required.

4.2.2 Application

This test is intended to assess the corrosion susceptibility of metallic materials used in the oral cavity using potentiodynamic polarization.

4.2.3 Reagents

- 4.2.3.1 **Lactic acid** ($\text{C}_3\text{H}_6\text{O}_3$), 90 %, chemically pure.
- 4.2.3.2 **Sodium chloride** (NaCl), analytical grade.
- 4.2.3.3 **Sodium hydroxide** (NaOH), analytical grade.
- 4.2.3.4 **Water**, complying with grade 2 of ISO 3696.
- 4.2.3.5 **Argon or nitrogen gas**, with a minimum purity of 99,99 %.
- 4.2.3.6 **Ethanol or methanol** ($\text{C}_2\text{H}_5\text{OH}$ or CH_3OH), analytical grade.
- 4.2.3.7 **Acetone** ($\text{C}_3\text{H}_6\text{O}$), analytical grade.

4.2.4 Apparatus

4.2.4.1 Test cell, temperature-controlled, of borosilicate glass, complying with ISO 3585 [a double-walled cell is unnecessary if the test is carried out at $(23 \pm 2) ^\circ\text{C}$].

4.2.4.2 Scanning potentiostat, potential range $\pm 1\ 600$ mV, current output range 10^{-9} A to 10^{-1} A.

4.2.4.3 Potential measuring instrument, with input impedance $> 10^{11} \Omega$ and sensitivity/accuracy able to detect a change of 1 mV over a potential change of $\pm 1\ 600$ mV.

4.2.4.4 Current-measuring instrument, capable of measuring a current to within 1 % of the absolute value over a current range between 10^{-9} A and 10^{-1} A.

4.2.4.5 Working electrode (specimen holder).

4.2.4.6 Counter electrode(s), composed of high-purity vitreous carbon or platinum.

4.2.4.7 Reference electrode, either saturated calomel electrode (SCE) or Ag/AgCl electrode [saturated silver chloride electrode (SSE)].

4.2.4.8 pH meter, with a sensitivity of $\pm 0,05$ pH units.

4.2.4.9 Silicon carbide paper, complying with ISO 6344-1.

4.2.4.10 Diamond paste, 1 μm .

4.2.4.11 Micrometer gauge, accurate to 0,01 mm.

4.2.4.12 Light microscope, minimum magnification of $\times 50$.

4.2.5 Solution preparation

Dissolve 9,0 g NaCl (4.2.3.2) in approximately 950 ml water (4.2.3.4). Adjust to pH $7,4 \pm 0,1$ using 1 % $\text{C}_3\text{H}_6\text{O}_3$ (4.2.3.1) or 4 % NaOH (4.2.3.3). Dilute with water to 1 000 ml.

4.2.6 Samples

4.2.6.1 Fabrication

4.2.6.1.1 Cast

Specimens shall be cast in accordance with the manufacturer's recommendations.

4.2.6.1.2 Prefabricated

Prefabricated parts/devices shall be used in the as-received condition.

4.2.6.1.3 Other

Specimens prepared by other methods, e.g. machined, sintered, eroded, etc., shall be tested in the as-manufactured condition after suitable cleaning.

4.2.6.2 Sampling

At least four specimens shall be tested.

4.2.6.3 Sample surface area

The surface area of each specimen shall be not less than 0,1 cm² after preparation.

4.2.6.4 Preparation

Remove any sprues, runners or other projections from surface. Blast surfaces with 125 µm pure alumina to remove investment.

If recommended, heat-treat according to the manufacturer's instructions.

In the case of metal-ceramic materials, heat-treat for 10 min at the highest firing temperature recommended by the metallic material manufacturer and bench cool.

Specimens shall be prepared with one flat surface exposed. A suitable contact shall be provided for connection to the electrochemical apparatus.

The working electrode shall be prepared in a way that ensures the absence of crevices. The recommended procedure is that of embedding in epoxy resin that does not exhibit loss of electrical insulation during immersion. Instead of embedding, a specimen holder (4.2.4.5) may be used if the absence of crevices is confirmed by specimen examination after the test.

Remove at least 0,1 mm, measured using a measuring instrument [e.g. micrometer gauge (4.2.4.11)], from each free surface using standard metallographic procedures unless specimens are being tested in the as-received condition. Use fresh abrasive paper for each metallic material. Finish with P1200 wet silicon carbide paper (4.2.4.9) and end with 1 µm diamond paste (4.2.4.10). If the described procedure is not applicable, treat the surfaces according to the manufacturer's instructions for clinical use.

Examine with a light microscope at ×50 (4.2.4.12) for cracks or crevices at the specimen/resin interface. Replace the specimen if any cracks or crevices are found.

Determine the area exposed to solution of each specimen to within 0,01 cm².

Manually clean surface with acetone (4.2.3.7). Clean surfaces ultrasonically in the following sequence: alcohol (4.2.3.6) and water (4.2.3.4) for 2 min each. Store in water (4.2.3.4) until transferred to test cell.

4.2.7 Test procedure

4.2.7.1 Test set-up

An example of an electrochemical measuring circuit is shown in Figure 1.

An example of an electrolytic cell is shown in Figure 2.

Fill test cell with electrolyte.

Test at room temperature (23 ± 2) °C. If there is a phase transition between room temperature and 37 °C, then a test temperature of (37 ± 1) °C shall be used.

Place counter electrode(s) in the test cell followed by the reference electrode. Then place the working electrode in the test cell without immersion. Activate the magnetic stirrer. Bubble oxygen-free nitrogen or argon at a rate of about 100 cm³/min through the electrolyte for at least 30 min. Immerse the working electrode in the electrolyte. Adjust the reference electrode. Adjust gas flow-rate to give a slight bubbling. Begin measurement procedure.

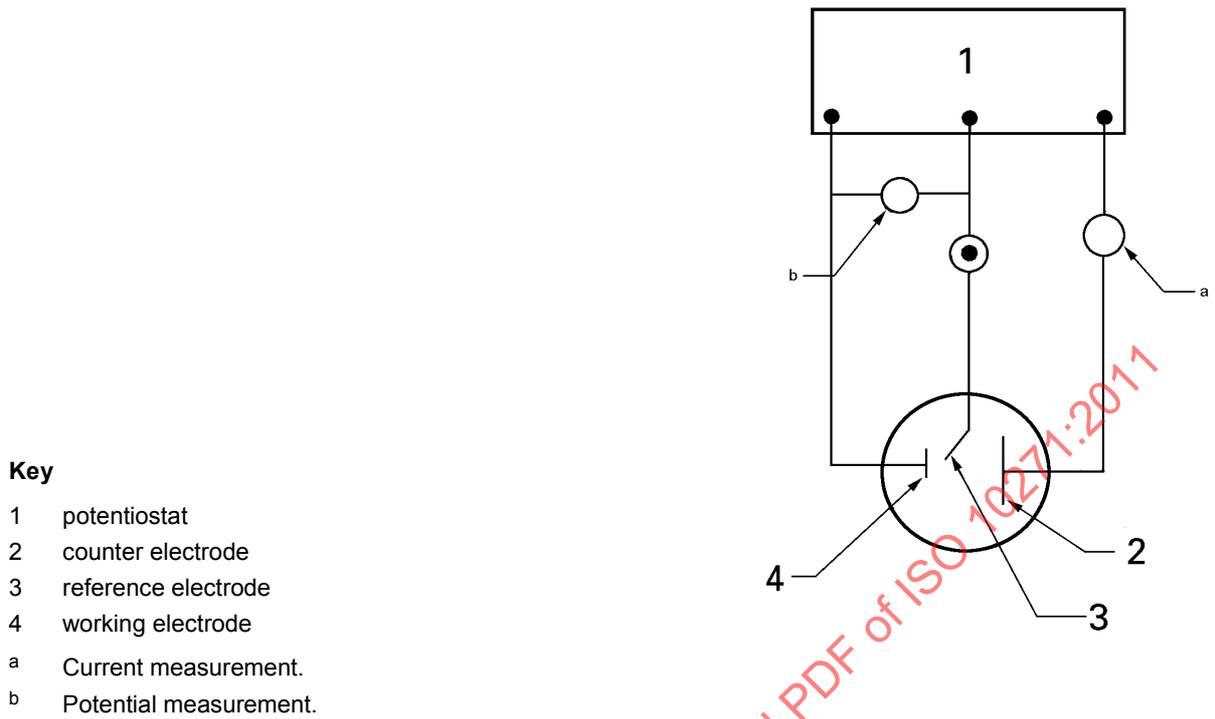


Figure 1 — Schematic diagram of measuring circuit

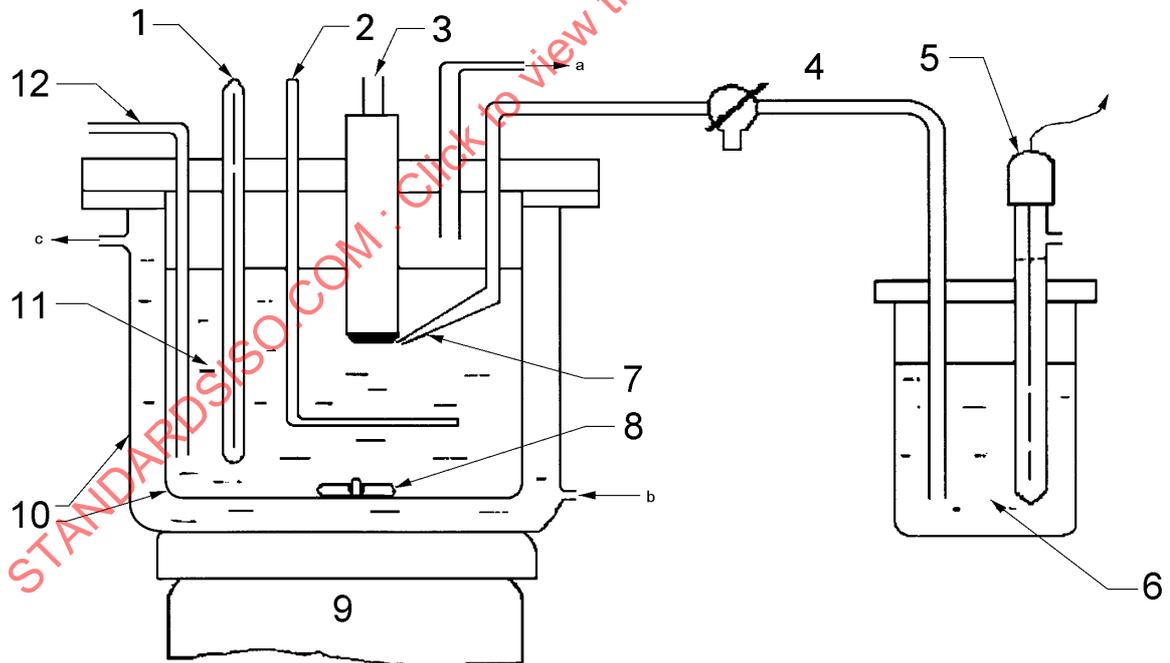


Figure 2 — Schematic diagram of electrolytic cell

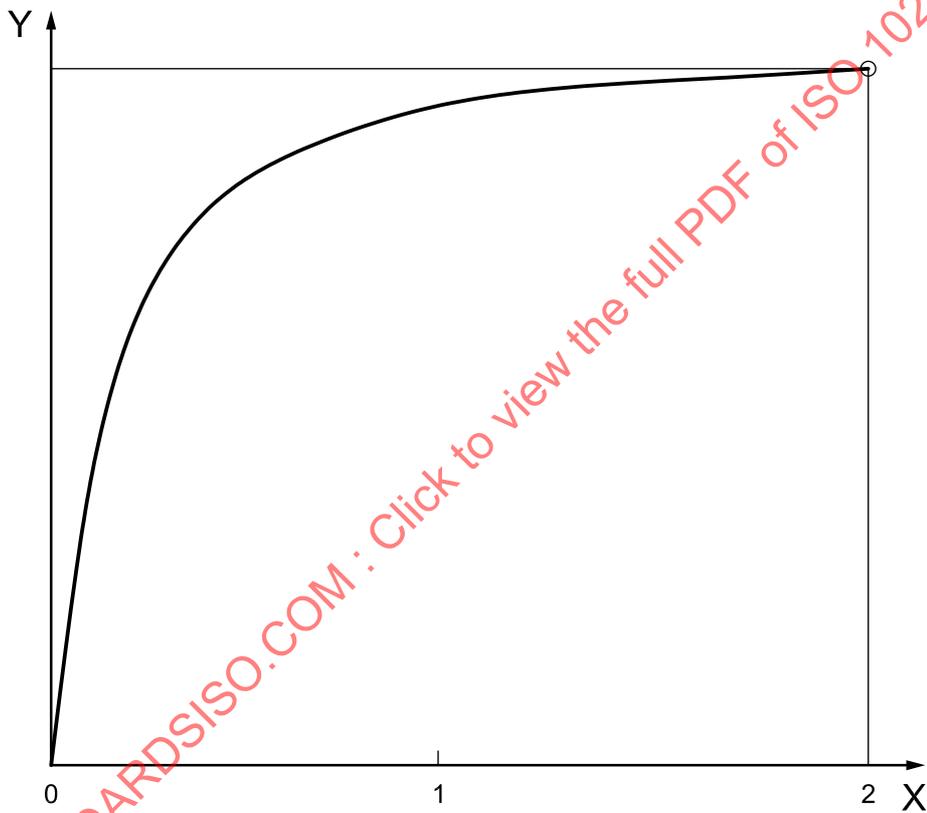
4.2.7.2 Open-circuit potential measurement

Record the open-circuit potential versus time curve for 2 h. Determine the open-circuit potential (E_{ocp}) in mV (SCE) after immersion for $2\text{ h} \pm 6\text{ min}$. An example of a potential versus time curve is shown in Figure 3.

4.2.7.3 Potential measurements (anodic polarization)

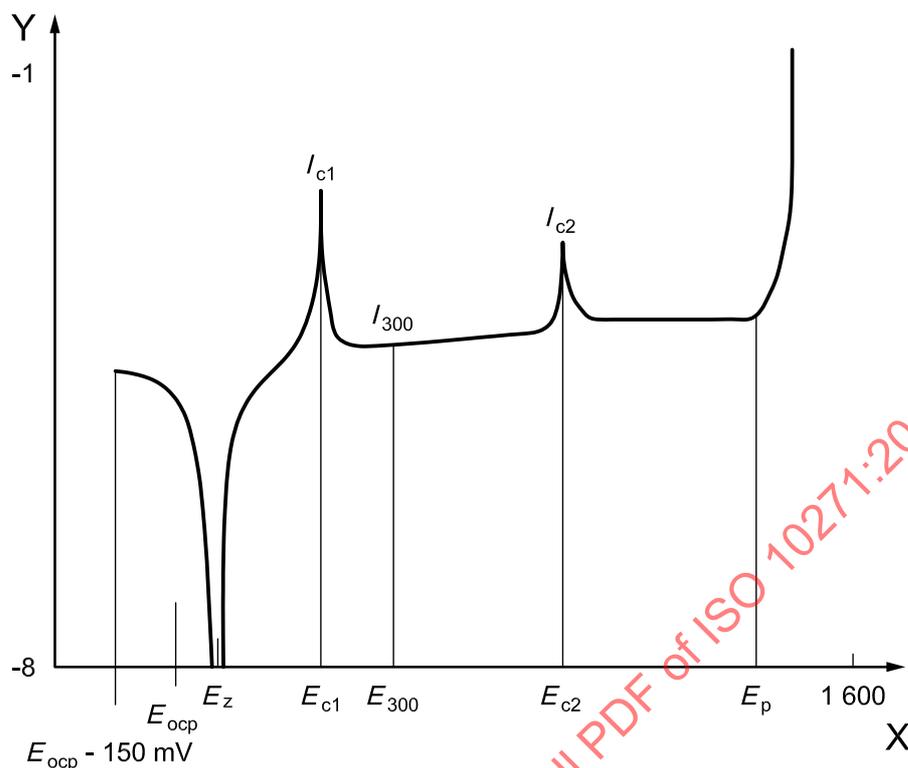
Start the potentiodynamic scan 5 min after finishing the open-circuit potential measurement at E_{ocp} minus 150 mV.

The potentiodynamic sweep rate should be 1 mV/s up to a current density threshold two decades greater than the current density recorded at breakdown or a potential of +1 500 mV (SCE) [or breakdown potential (E_p) + 300 mV]. Record the curve of potential versus logarithm of current density. A reverse scan back to the original potential may be used to obtain information on pitting corrosion. An example of a potential versus log current density curve is shown in Figure 4.



Key
 X time, in hours
 Y potential, E_{ocp} , in millivolts (SCE)

Figure 3 — Open-circuit potential versus time



Key

X	potential, in millivolts (SCE)
Y	log current, expressed in amperes per square centimetre
SCE	saturated calomel electrode
E_{ocp}	open-circuit potential, expressed in millivolts (SCE)
$E_{ocp} - 150$ mV	potential at the open-circuit potential minus 150 mV, expressed in millivolts (SCE)
E_z	zero-current potential, expressed in millivolts (SCE)
E_{c1}	first active peak potential, expressed in millivolts (SCE)
E_{300}	potential at the open-circuit potential plus 300 mV, expressed in millivolts (SCE)
E_{c2}	second active peak potential, expressed in millivolts (SCE)
E_p	breakdown potential, expressed in millivolts (SCE)
I_{c1}	current density, expressed in amperes per square centimetre that corresponds to E_{c1}
I_{300}	current density, expressed in amperes per square centimetre that corresponds to E_{300}
I_{c2}	current density, expressed in amperes per square centimetre that corresponds to E_{c2}

Figure 4 — Log current density versus potential

4.2.8 Test report

The test report shall contain the following information:

- identity of the test metallic material;
- details of heat treatment, if applicable;
- test temperature (23 ± 2) °C; if 37 °C is used, the reason for this choice;
- description of any deviations from preparation of specimens or test procedure;

- e) open-circuit potential (E_{ocp}), expressed in millivolts (SCE);
- f) potential versus log current density curve or potential versus current density curve;
- g) zero-current potential, E_z , expressed in millivolts (SCE);
- h) breakdown potential, E_p , expressed in millivolts (SCE), with the corresponding current density, I_p , expressed in amperes per square centimetre;
- i) active peak potentials, E_c , expressed in millivolts (SCE) between E_z and E_p with the corresponding current density, I_c , expressed in amperes per square centimetre;
- j) current density, I_{300} , expressed in amperes per square centimetre at potential of $(E_z + 300)$ mV (SCE);
- k) description of any significant changes of the electrolyte or the metallic material surface.

If a reference electrode other than a saturated calomel electrode (SCE) is used, the reported potential values shall be converted to millivolts (SCE).

4.3 Sulfide tarnish test (cyclic immersion)

4.3.1 Information required

Composition, including hazardous elements, in accordance with the appropriate ISO material standard is required.

4.3.2 Application

This test method is suitable for dental metallic materials that are susceptible to sulfide tarnish, such as those containing silver.

4.3.3 Reagents

4.3.3.1 Sodium sulfide hydrate (approximately 35 % Na_2S), analytical grade.

4.3.3.2 Water, complying with grade 2 of ISO 3696.

4.3.3.3 Ethanol or methanol ($\text{C}_2\text{H}_5\text{OH}$ or CH_3OH), analytical grade.

4.3.3.4 Acetone (C_3H_6), analytical grade.

4.3.3.5 Compressed air, oil- and water-free, complying with ISO 7183.

4.3.4 Apparatus

4.3.4.1 Dipping device, that dips each specimen in the test solution for 10 s to 15 s every minute with the temperature controlled at (23 ± 2) °C.

4.3.4.2 Silicon carbide paper, complying with ISO 6344-1.

4.3.4.3 Micrometer gauge, accurate to 0,01 mm.

4.3.5 Solution preparation

Prepare a fresh solution for each test. Dissolve 22,3 g sodium sulfide hydrate (4.3.3.1) in water (4.3.3.2). Dilute to 1 000 ml with water.

4.3.6 Samples

4.3.6.1 Fabrication

Specimens shall be fabricated in accordance with the manufacturer's recommendations.

4.3.6.2 Sampling

At least two specimens shall be tested.

4.3.6.3 Sample surface area

The surface area of each specimen shall be approximately 1 cm².

4.3.6.4 Preparation

Remove any sprues, runners or other projections from surface. Blast surfaces with 125 µm pure alumina to remove investment.

If recommended, heat-treat according to the manufacturer's instructions.

In the case of metal-ceramic materials, heat-treat for 10 min at the highest firing temperature recommended by the metallic material manufacturer and bench cool.

Cold-mount each specimen. The recommended procedure is that of embedding in epoxy resin.

Remove at least 0,1 mm, measured using a measuring instrument [e.g. micrometer gauge (4.3.4.3)], from each free surface using standard metallographic procedures unless specimens are being tested in the as-received condition. Use fresh abrasive paper for each metallic material. Finish with P800 wet silicon carbide paper (4.3.4.2). If the described procedure is not applicable, treat the surfaces according to the manufacturer's instructions for clinical use.

Manually clean surface with acetone (4.3.3.4). Clean surfaces ultrasonically in the following sequence: alcohol (4.3.3.3) and water (4.3.3.2) for 2 min each. Dry with oil- and water-free compressed air (4.3.3.5).

4.3.7 Test procedure

Tests shall be carried out under a properly operating hood with air extraction. Place one mounted specimen in the dipping device (4.3.4.1) with a freshly made test solution. The test solution shall be replaced every (24 ± 1) h. Remove specimen after (72 ± 1) h. Rinse with water (4.3.3.2). Dip specimen in ethanol or methanol (4.3.3.3). Dry with oil- and water-free compressed air (4.3.3.5).

4.3.8 Inspection

Compare treated and untreated surfaces visually without magnification for any surface deterioration (see A.7.3.1).

4.3.9 Test report

The test report shall contain the following information.

- a) Report and justify any deviations from specimen preparation and/or test procedure.
- b) Record details of heat treatment if applicable.
- c) Report all visible differences from untreated specimen with regard to color and reflectivity.

4.4 Sulfide tarnish test (static immersion)

4.4.1 Information required

Composition, including hazardous elements, in accordance with the appropriate ISO material standard is required.

4.4.2 Application

This test method is suitable for dental metallic materials that are susceptible to sulfide tarnish, such as those containing silver.

4.4.3 Reagents

4.4.3.1 **Sodium sulfide nonahydrate** ($\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$), $\geq 98\%$.

4.4.3.2 **Water**, complying with grade 2 of ISO 3696.

4.4.3.3 **Ethanol or methanol** ($\text{C}_2\text{H}_5\text{OH}$ or CH_3OH), analytical grade.

4.4.3.4 **Compressed air**, oil- and water-free, complying with ISO 7183.

4.4.4 Apparatus

4.4.4.1 **Static immersion test apparatus**, consisting of a **borosilicate glass container** (4.1.4.1 or 4.5.4.1), complying with ISO 3585, and a thin **glass rod** with a hook on the end (or a corrosion resistant string). See Figure 5.

4.4.4.2 **Silicon carbide paper**, complying with ISO 6344-1.

4.4.4.3 **Micrometer gauge**, accurate to 0,01 mm.

4.4.5 Solution preparation

Prepare a fresh solution for each test. Dissolve $(3,1 \pm 0,05)$ g sodium sulfide nonahydrate (4.4.3.1) in water (4.4.3.2). Dilute to $(1\ 000 \pm 10)$ ml with water. Be aware that the solution shall be made no longer than 24 h before the start of the test.

4.4.6 Samples

4.4.6.1 Fabrication

Specimens shall be fabricated in accordance with the manufacturer's instructions.

4.4.6.2 Sampling

At least two specimens shall be tested.

4.4.6.3 Sample size

The size of each specimen shall be $(15 \pm 0,5)$ mm in width \times $(20 \pm 0,5)$ mm in length \times $(1 \pm 0,5)$ mm in thickness.

4.4.6.4 Preparation

Remove any sprues, runners or other projections from surface. Blast surfaces with 125 µm pure alumina to remove investment.

If recommended, heat-treat according to the manufacturer's instructions.

In the case of metal-ceramic materials, heat-treat for 10 min at the highest firing temperature recommended by the metallic material manufacturer and bench cool.

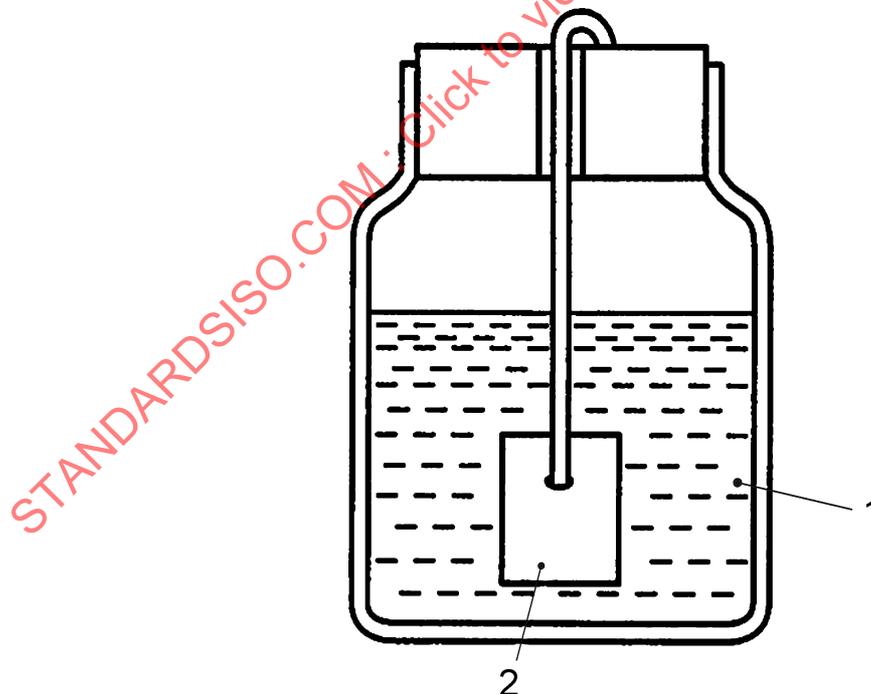
Remove at least 0,1 mm, measured using a measuring instrument [e.g. micrometer gauge (4.4.4.3)], from each free surface using standard metallographic procedures unless specimens are being tested in the as-received condition. Use fresh abrasive paper for each metallic material. Finish with P800 wet silicon carbide paper (4.4.4.2). If the described procedure is not applicable, treat the surfaces according to the manufacturer's instructions for clinical use.

Clean surfaces ultrasonically in the following sequence: alcohol (4.4.3.3) and water (4.4.3.2) for 2 min each. Dry with oil- and water-free compressed air (4.4.3.4).

4.4.7 Test procedure

Immerse one specimen in 50 ml of freshly prepared test solution in the static immersion test apparatus (4.4.4.1) and maintain at $(37 \pm 2) ^\circ\text{C}$ for (72 ± 1) h. Remove the specimen and rinse it with water (4.4.3.2). Dip specimen in ethanol or methanol (4.4.3.3). Dry with oil- and water-free compressed air (4.4.3.4).

An example of a static immersion test apparatus with a test specimen immersed in test solution is shown in Figure 5.



Key

- 1 test solution
- 2 specimen

Figure 5 — An example of static immersion test apparatus

4.4.8 Inspection

Compare treated and untreated surfaces visually without magnification for any surface deterioration and/or discoloration. See paragraphs 1 and 2 of A.7.3.1.

4.4.9 Test report

The test report shall contain the following information.

- a) Report and justify any deviations from specimen preparation and/or test procedure.
- b) Record details of heat treatment if applicable.
- c) Report all visible differences from untreated specimen with regard to color and reflectivity.

4.5 Static immersion test with periodic analysis

4.5.1 Information required

Composition, including hazardous elements, in accordance with the appropriate ISO material standard is required.

4.5.2 Application

In order to evaluate the corrosion rate over time, it is necessary to obtain corrosion information over an extended time period with analysis at intermediate times.

Based on the static immersion test in 4.1, which at the date of this publication has been successfully performed on all major dental metallic materials including prefabricated parts, the static immersion test with periodic analysis provides information on any change in corrosion rate over the time period of the test. Combined with the corrosion level given by the static immersion test, a more complete evaluation of the corrosion behavior of a metallic material for dental applications can be obtained.

4.5.3 Reagents

- 4.5.3.1 **Lactic acid** ($C_3H_6O_3$), 90 %, analytical grade.
- 4.5.3.2 **Sodium chloride** (NaCl), analytical grade.
- 4.5.3.3 **Water**, complying with grade 2 of ISO 3696.
- 4.5.3.4 **Ethanol or methanol** (C_2H_5OH or CH_3OH), analytical grade.
- 4.5.3.5 **Oil- and water-free compressed air**, complying with ISO 7183.

4.5.4 Apparatus

- 4.5.4.1 **Containers**, of borosilicate glass, complying with ISO 3585, with dimensions of approximately 16 mm inner diameter by 160 mm in height.
- 4.5.4.2 **pH meter**, with a sensitivity of at least $\pm 0,05$ pH units.
- 4.5.4.3 **Chemical analysis instrumentation**, capable of measuring ion concentration in $\mu\text{g/ml}$ (e.g. ICP and AAS).
- 4.5.4.4 **Micrometer gauge**, accurate to 0,01 mm.
- 4.5.4.5 **Silicon carbide paper**, complying with ISO 6344-1.
- 4.5.4.6 **Volumetric flasks**, of borosilicate glass, 1 000 ml, class A, complying with ISO 1042.

4.5.5 Solution preparation

Prepare an aqueous solution comprising 0,1 mol/l lactic acid and 0,1 mol/l sodium chloride immediately before use. For example, dissolve $(10,0 \pm 0,1)$ g 90 % $C_3H_6O_3$ (4.5.3.1) and $(5,85 \pm 0,005)$ g NaCl (4.5.3.2) in approximately 300 ml of water (4.5.3.3). Transfer into a 1 000 ml volumetric flask (4.5.4.6) and fill to mark. The pH shall be $2,3 \pm 0,1$. If not, the solution shall be discarded and the reagents checked.

4.5.6 Samples

4.5.6.1 Fabrication

4.5.6.1.1 Cast

Specimens shall be cast in accordance with the manufacturer's recommendations.

4.5.6.1.2 Prefabricated

Prefabricated parts/devices shall be used in the as-received condition.

4.5.6.1.3 Other

Specimens prepared by other methods, e.g. machined, sintered, eroded, etc., shall be tested in the as-manufactured condition after suitable cleaning.

4.5.6.2 Sampling

The number of specimens shall be sufficient to provide at least two parallel sets. (The number of specimens in a set may vary.)

If statistical analysis of the results is required, a minimum of five parallel sets shall be tested.

4.5.6.3 Sample surface area

The total surface area of the sample shall be at least 10 cm^2 after preparation.

4.5.6.4 Preparation

4.5.6.4.1 Cast samples

Remove any sprues, runners, or other projections from surface. Blast surfaces with $125 \mu\text{m}$ pure alumina to remove investment.

If recommended, heat-treat according to the manufacturer's instructions.

In the case of metal-ceramic materials, heat-treat for 10 min at the highest firing temperature recommended by the metallic material manufacturer and bench cool.

Remove at least 0,1 mm, measured using a measuring instrument [e.g. micrometer gauge (4.5.4.4)], from each surface using standard metallographic procedures unless specimens are to be tested in the as-received condition. Use fresh abrasive paper for each metallic material. Finish with P1200 wet silicon carbide paper (4.5.4.5). If the described procedure is not applicable, treat the surfaces according to the manufacturer's instructions for clinical use.

Determine each sample area to the nearest 1 %.

Clean surfaces ultrasonically for 2 min in ethanol or methanol (4.5.3.4). Rinse with water (4.5.3.3). Dry with oil- and water-free compressed air (4.5.3.5).

If a specimen contains any porosity visible on the surfaces being exposed to the solution, the specimen shall be rejected and replaced with a new one.

4.5.6.4.2 Machined, sintered, eroded or electroformed samples

Heat-treat the specimens if recommended.

Remove at least 0,1 mm, measured using a measuring instrument [e.g. micrometer gauge (4.5.4.4)], from each surface using standard metallographic procedures unless specimens are to be tested in the as-received condition. Use fresh abrasive paper for each metallic material. Finish with P1200 wet silicon carbide paper (4.5.4.5).

Determine each sample surface area to within 0,1 cm².

Clean surfaces ultrasonically for 2 min in ethanol or methanol (4.5.3.4).

Rinse with water (4.5.3.3). Dry with oil- and water-free compressed air (4.5.3.5).

4.5.6.4.3 Prefabricated parts/devices

Treat the surfaces according to the manufacturer's instructions for clinical use.

Determine each sample surface area to within $\pm 0,1$ cm².

Clean the surfaces ultrasonically for 2 min in ethanol or methanol (4.5.3.4).

Rinse with water (4.5.3.3). Dry with oil- and water-free compressed air (4.5.3.5).

4.5.7 Test procedure

Parallel specimen sets shall be treated in identical fashion. If a set consists of one specimen, it shall be placed in a container (4.5.4.1) such that it does not touch the container surface except in a minimum support line or point. If a set consists of two or more specimens, they may be placed in the same number of containers, but if more than one is placed in a container they shall not touch.

Record the pH of the solution. Add the solution to each container sufficient to produce a ratio of 1 ml of solution per 1 cm² of sample surface area. The specimens shall be covered completely by the solution. Record the volume of solution to an accuracy of 0,1 ml. Close the container to prevent evaporation of the solution. Maintain at (37 ± 1) °C for the time schedule specified in Table 1.

After each time period, take out the set of specimens from the individual container(s) with a metal-free tool, rinse with distilled water, and place the set of specimens into a new container(s) with fresh solution as described above.

Analyze the solutions for released ions according to 4.5.1 at the times marked "mandatory" in Table 1. If the static immersion test has been performed, then the results from this test may be used to estimate a value for the seventh day of elapsed time, as noted in Table 1. Analysis of the solution at the "recommended" time periods specified in Table 1 increases the information obtained from the test.

Use an additional container (4.5.4.1) to hold a reference solution which is maintained in parallel with the solutions containing the specimens. The reference solution shall be used to establish the impurity level for each element of interest in the solution. Add approximately the same volume of solution as used for the solutions containing the specimens and record the volume to an accuracy of 0,1 ml. Close the container to prevent evaporation of the solution and maintain at (37 ± 1) °C for 42 d.

Table 1 — Time schedule for determination of corrosion rate

Time schedule	Elapsed time d	Analysis
1 d ± 1 h	1	Mandatory
3 d ± 1 h	4	Mandatory
3 d ± 1 h	7	Mandatory if static immersion test has not been performed Otherwise the analysis is recommended
7 d ± 1 h	14	Recommended
7 d ± 1 h	21	Recommended
7 d ± 1 h	28	Recommended
7 d ± 1 h	35	Recommended
7 d ± 1 h	42	Mandatory

4.5.8 Elemental analysis

Use chemical analysis instrumentation (4.5.4.3) of adequate sensitivity. Analyze the solution qualitatively and quantitatively. Emphasis shall be on those elements as indicated in 4.5.1, but if impurities are found in a concentration greater than 0,1 %, they shall be reported. For each element of interest, subtract the value obtained for the element in the reference solution from the value obtained in the test solution. The elements boron, carbon, and nitrogen shall be disregarded.

4.5.9 Test report

The test report shall contain the following information.

- a) Report the method of analysis and detection limits of all the analyzed elements.
- b) Report and justify any deviations from preparation of specimens (see 4.5.6.4) or test procedure (see 4.5.7).
- c) Report the number of specimens making up a set and the number of sets tested (see 4.5.6.2).
- d) From the elemental analysis of the corrosion solution (see 4.5.8), calculate and report the ion release for each element of each set (see 4.5.6.2) separately in micrograms per square centimetre per time period at the times marked "mandatory" in Table 1. Each element indicated in 4.5.1 shall be reported as well as any others found. From this information, calculate and report the total ion release over the total elapsed time of the test for each element of each set separately. For an individual element in a set, this is accomplished by calculating the sum of the ion release values at the times marked "mandatory" in Table 1 to yield a total amount of ion release in micrograms per square centimetre for that element over the total elapsed time of the test. From this information, calculate and report the ion release rate in $\mu\text{g}/\text{cm}^2$ per day for each element of each set separately. For an individual element in a set, this is accomplished by taking the total amount of ion release over the total elapsed time of the test calculated above and dividing this sum by the total number of days of the test to yield an ion release rate in micrograms per square centimetre per day for that element. From this information, sum the ion release rate values for all the elements of each set to obtain the total ion release rate for the dental material. Average the results by dividing by the number of sets tested and report as the average total ion release rate for the dental material in micrograms per square centimetre per day.

NOTE If analysis of the solution at the "recommended" time periods specified in Table 1 is performed, this increases the information obtained from the testing and can be included in the calculations in item d).

- e) Report undissolved corrosion product like precipitates and discoloration of the specimen surface.

Annex A (informative)

Corrosion test method development

A.1 Method development

A.1.1 Principle

Test methods should be chosen according to the provisions of A.2. The provisions of A.3 to A.6 should be fulfilled to ensure acceptable corrosion rates with respect to safety and efficacy.

A.1.2 Safety — Generation of harmful corrosion products

Corrosion and tarnish indicate the formation of metal compounds, whether formed by simple oxidation-type reactions or by the release of metal or metal ions. It is necessary to evaluate the harmful effects of these metals or metal compounds, such as oxides, sulfides, chlorides, metal organics or other species, on the soft and hard tissues and other parts of the body as described in ISO 7405 and the relevant parts of ISO 10993.

A.1.3 Efficacy — Loss of substance

If the loss of substance is sufficient to significantly reduce sample thickness, it is conceivable that enough mechanical strength has been lost so that failure occurs by bending or fracture. Where the loss of material occurs by pitting or other roughening corrosion, sites can be formed for plaque deposition and growth. Pitting can initiate fatigue fracture.

A.1.4 Aesthetics — Change in appearance

Corrosion can result in loss of lustre since reflected light can be diffused, giving the appearance of a discoloration. Actual discoloration results from colored reaction products that are tenacious enough to remain on the surface. Well known colored reaction products include silver and copper sulfides. With many other elements coming into use for dental metallic materials, other colored compounds can be possible. Discoloration in itself might not affect safety or efficacy.

A.2 Procedure

Selection of the requisite test conditions should be made by reference to A.3 to A.6, which describe preferred pathways for the four basic components: test type, sample type, medium and other conditions.

The assessment method should be chosen from A.7. More than one assessment method may be used, as appropriate.

A.3 Test type

A.3.1 General conditions

In all cases where a condition is not specified explicitly in this International Standard, the appropriate requirements should be stated in the material (application) standard in order that the test conditions be sufficient and complete.

All tests and all solution storage and transport should be in vessels of, and with closures made from, borosilicate glass or unfilled non-contaminating polymers, e.g. polystyrene, high-density polyethylene, polypropylene or polytetrafluoroethylene.

All tests should be conducted at least in duplicate and additional samples and tests are recommended.

A.3.2 Sampling

A.3.2.1 Embedded samples

Exposure solutions should be replenished at intervals of not more than 7 d and, if required for analysis of dissolved metallic elements or loss of material, such aliquots of solution should be stored after use in separate containers and analyzed separately.

A.3.2.2 Duration

Exposure conditions intended to represent oral condition simulation in accordance with A.3.2.1 and A.5.3 should be used for not less than 28 d.

Other specific conditions representing short duration challenges should be used for not less than 24 h or 20 complete treatment cycles, if the treatment process is otherwise defined.

A.3.3 Exposure

A.3.3.1 Oral simulation

The following apply.

- a) Immersion: The sample should be immersed in a medium selected from those listed in A.5.
- b) Static immersion: This is used to simulate the condition of plaque build-up in interproximal areas. No relative motion between sample and medium is permitted.
- c) Dynamic immersion: This is used to simulate the general oral cavity condition. Motion of the sample and/or agitation of the medium is prescribed.
- d) Cyclic immersion: This is used to simulate opening and closing the mouth. A cyclic exposure to the medium, followed by exposure to ambient air, is prescribed.

A.3.3.2 Voltametric

Two types of voltametric exposure are possible:

- a) potentiostatic;
- b) potentiodynamic.

A.4 Sample type

A.4.1 Embedded sample

Test pieces may be prepared from the as-supplied material or after preparation or fabrication (e.g. casting) in accordance with the manufacturer's instructions and normal good laboratory practice.

The sample should consist of a piece or pieces of one material, one batch, embedded after cleaning and degreasing in an auto-curing resin such that the sample temperature does not rise above 50 °C. If the sample is more than one piece, the pieces should not touch each other. Reduced pressure should be applied while