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Third edition
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Dentistry — Corrosion test methods for metallic materials

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

Reference number
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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 of 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 2, *Prosthodontic materials*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 55, *Dentistry*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 10271:2011), which has been technically revised. The main changes compared with the previous edition are as follows:

- in the Scope, the statement about this document not being applicable to "appliances for orthodontics" and "dental amalgam" has been removed;
- in 4.1.6.3, a NOTE has been added to the static immersion test method acknowledging that "measuring the total surface area of orthodontic appliances can be difficult" and, therefore, if required in the appropriate standard, "it is acceptable for the ion release for each element of a set of orthodontic brackets to be reported in terms of µg in seven days for a specified number of orthodontic brackets";
- since sodium sulfide hydrate (approximately 35 % Na_2S) analytical grade is not available in every country, text was added to the appropriate test methods indicating that sodium sulfidenonahydrate ($Na_2S·9H_2O$), ≤ 98 % may be used;
- this document has been harmonized with ISO 22674:2016 by making changes in the preparation sections of the various test methods that reflect changes that were made for the preparation of metals supplied for metal-ceramic restorations in ISO 22674:2016;
- subclause 4.6 "Dental amalgam" has been added, which refers the user to ISO/TS 17988 when testing the corrosion behaviour of dental amalgam;
- subclause 4.7 "Crevice corrosion" has been added, which provides a test method to evaluate the susceptibility of a dental metallic material to crevice corrosion.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document was developed from the original Technical Report (ISO/TR 10271[1]) as a result of worldwide demand for standard test methods to determine the 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 document, 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 behaviour 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 behaviour 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 document, therefore, gives detailed procedures for test methods that have been found to be of merit as evidenced by considerable use.

In the second edition, two new test methods were added. To supplement the existing static immersion test, a static immersion test with periodic analysis was 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 document. In this third edition, a classification scheme is still not included.

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

In addition, the second edition added Annex A, which 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.

The third edition differs from the second by the removal of the statement in the Scope about the document not being applicable to "appliances for orthodontics" and "dental amalgam". With the appliances for orthodontics change in mind, a NOTE was added to the static immersion test acknowledging that "measuring the total surface area of orthodontic appliances can be difficult" and stating that, if required in the appropriate standard, "it is acceptable for the ion release for each element of a set of orthodontic brackets to be reported in terms of µg in seven days for a specified number of orthodontic brackets". Also, with reference to dental amalgam, a subclause on dental amalgam (see 4.6) has been added, which refers the user to ISO/TS 17988 when testing the corrosion behaviour of dental amalgam. Additionally, there is a clarification statement that the test methods given in 4.1 to 4.5 are still not applicable to the evaluation of dental amalgam.

The third edition was harmonized with ISO 22674:2016 by adding to the preparation sections of the various test methods the following change concerning metals supplied for metal-ceramic restorations:

— "Following the manufacturer's instructions, simulate the oxidation procedure and four ceramic firings at the highest temperature recommended for fusing ceramic to the metallic material. Remove and place the specimens on a ceramic plate (which is at room temperature) to cool to room temperature after the oxidation and ceramic firing simulation."

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[1] Withdrawn document.

Additionally, since sodium sulfide hydrate (approximately 35 % Na_2S) analytical grade is not available in every country, this third edition includes a statement, which was added to the appropriate test methods, indicating that sodium sulfide nonahydrate ($Na_2S·9H2O$), ≤ 98 % may be used.

Also of importance, a test method to evaluate the susceptibility of a dental metallic material to crevice corrosion was added as 4.7.

It is not the purpose of this document to propose corrosion test methods for specific applications or to set limits as precise as those that may be required in a standard relating to a type of product and its application.

Dentistry — Corrosion test methods for metallic materials

1 Scope

This document specifies test methods and procedures to determine the corrosion behaviour of metallic materials used in the oral cavity. It is intended that these test methods and procedures be referred to in individual International Standards specifying such metallic materials.

This document is not applicable to dental instruments.

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 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*

ISO/TS 17988, *Dentistry — Corrosion test methods for dental amalgam*

3 Terms and definitions

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

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

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at http://www.electropedia.org/

3.1
breakdown potential
E_p
least noble potential at which pitting or *crevice corrosion* (3.4), 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 product
substance formed as a result of *corrosion* (3.2)

3.4
crevice corrosion
corrosion (3.2) associated with and taking place in or near a narrow aperture or crevice

3.5
current density
value of electric current per unit surface area flowing through a conductor

3.6
dynamic immersion test
test in which the *specimen* (3.15) is exposed to a corrosive solution under conditions of relative motion between specimen and solution

3.7
electrode potential
potential difference between the *specimen* (3.15) and a reference electrode

3.8
electrolyte
solution or liquid that conducts an electrical current by means of ions

3.9
open-circuit potential
E_{ocp}
potential of an electrode measured with respect to a reference electrode or another electrode when no current flows

3.10
pitting corrosion
localized *corrosion* (3.2) that results in pits

3.11
potentiodynamic test
test in which the *electrode potential* (3.7) is varied at a predetermined rate and the relationship between *current density* (3.5) and electrode potential is recorded

3.12
potentiostatic test
test in which the *electrode potential* (3.7) is maintained constant

3.13
sample
totality of material for one type being tested, the group of all such *specimens* (3.15)

3.14
set
subgroup of the *specimens* (3.15) of a *sample* (3.13)

3.15
specimen
test piece
individual single example of an object for testing

3.16
static immersion test
test in which the *specimen* (3.15) is exposed to a corrosive solution under conditions of effectively no relative motion between specimen and solution

3.17
stress corrosion
corrosion (3.2) resulting from the combined action of static tensile stress and an *electrolyte* (3.8)

3.18
synthetic saliva
test medium that approximates the relevant chemistry of natural saliva

3.19
tarnish
surface discoloration due to the chemical reaction between a metallic material and its environment

3.20
zero-current potential
E_z
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 International Standard, shall be provided.

4.1.2 Application

This is an accelerated test that is intended to provide quantitative data on the metal ions 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 (2-hydroxypropanoic acid, $C_3H_6O_3$), ≥ 85 %, analytical grade.

4.1.3.2 Sodium chloride (NaCl), analytical grade.

4.1.3.3 Water, in accordance 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, in accordance with ISO 7183.

4.1.4 Apparatus

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

4.1.4.2 pH meter, calibrated, with a sensitivity of at least ±0,05 pH.

4.1.4.3 Chemical analysis instrumentation, capable of measuring ion concentration in µg/ml, e.g. inductively coupled plasma atomic emission spectroscopy (ICP-AES), inductively coupled plasma optical emission spectrometry (ICP-OES) or atomic absorption spectroscopy (AAS).

4.1.4.4 Micrometer screw gauge, accurate and reading to 0,01 mm.

4.1.4.5 Silicon carbide paper, in accordance with ISO 6344-1.

4.1.4.6 **Volumetric flasks**, of borosilicate glass, 1 000 ml, class A, in accordance 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 within a few hours of use. For example, dissolve (10,0 ± 0,1) g ≥ 85 % $C_3H_6O_3$ (4.1.3.1) and (5,850 ± 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 the mark. The pH shall be 2,3 ± 0,1. If it is not, the solution shall be discarded and the 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 or 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, 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.

NOTE It is recognized that measuring the total surface area of orthodontic appliances can be difficult. Therefore, for prefabricated appliances, such as orthodontic brackets, it is acceptable to report the corrosion rate in terms of ion release per sample, where the sample consists of sets of items that represent the appliance in clinical use. As an example, it is acceptable for the ion release for each element of a set of orthodontic brackets to be reported in terms of µg in seven days for a specified number of orthodontic brackets, as required in the appropriate orthodontic brackets' standard.

4.1.6.4 Preparation

4.1.6.4.1 Cast samples

Remove any sprues, runners or other projections from the surface. Blast all surfaces with 110 µm to 250 µm pure alumina to remove investment.

For precious metals, it is recommended to blast all surfaces with 110 µm pure alumina. For non-precious metals, it is recommended to blast with 250 µm pure alumina.

It is advised that the removal of sprues, runners and other projections is done cold (i.e. under running water) to prevent transformations.

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

In the case of metals supplied for metal-ceramic restorations, test after the following simulated ceramic-firing schedule has been applied.

- Following the manufacturer's instructions, simulate the oxidation procedure and four ceramic firings at the highest temperature recommended for fusing ceramic to the metallic material. Remove and place the specimens on a ceramic plate (which is at room temperature) to cool to room temperature after the oxidation and ceramic firing simulation.
- Remove at least 0,1 mm, as determined using a measuring instrument [e.g. micrometer screw 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 % (see NOTE in 4.1.6.3).
- 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 has any porosity visible on any surface intended to be exposed to the test 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 this is recommended.

Remove at least 0,1 mm, as determined using a measuring instrument [e.g. micrometer screw 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 ±0,1 cm^2 (see NOTE in 4.1.6.3).

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 ±0,1 cm^2 (see NOTE in 4.1.6.3).

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 single 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 cm^2 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 d ± 1 h. Remove the specimen(s) and record the pH of the solution.

Use an additional container (4.1.4.1) to hold a reference ("blank") solution to be maintained in parallel with the solutions containing the specimens. The reference solution shall be used to establish the impurity concentration for each element of interest in the test 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. Analyse the solution qualitatively and quantitatively. Emphasis shall be on those elements indicated in 4.1.1, but if impurities are found to have a concentration greater than 0,1 % by mass in the original metal, 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) the method of analysis and detection limits of all the analysed elements;

b) any deviations from the preparation of specimens (see 4.1.6.4) or test procedure (see 4.1.7) and their justification;

c) the number of specimens making up a set and the number of sets tested (see 4.1.6.2);

d) the calculated ion release for each element of each set (see 4.1.6.2) separately in µg/cm^2 in seven days from the elemental analysis of the corrosion solution (see 4.1.8) (see NOTE in 4.1.6.3), for elements indicated in 4.1.1, as well as any others found; the sum of the ion release values for all the elements of each set (i.e. the total metal ion release for the dental material); and the average total ion release for the dental material in µg/cm^2 in seven days (i.e. that sum divided by the number of sets tested);

e) the presence of undissolved corrosion products, such as precipitates, and any discoloration of the specimen surface;

f) the International Standard used (including its year of publication);

g) the method used (in this case, “static immersion test”);

h) the date of the test.

4.2 Electrochemical test

4.2.1 Information required

Composition, including hazardous elements, in accordance with the appropriate International Standard, shall be provided.

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 (2-hydroxypropanoic acid, $C_3H_6O_3$), ≥ 85 %, 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, in accordance 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** (C_2H_5OH or CH_3OH), analytical grade.

4.2.3.7 Acetone [dimethyl ketone, $(CH_3)_2CO$], analytical grade.

4.2.4 Apparatus

4.2.4.1 Test cell, temperature-controlled, of borosilicate glass, in accordance with ISO 3585.

NOTE A double-walled cell is unnecessary if the test is carried out at (23 ± 2) °C.

4.2.4.2 Scanning potentiostat, potential range ±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} Ω and both the sensitivity and accuracy to be able to detect a change of 1 mV over a potential change of ±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)]. If a reference electrode other than a SCE is used, all reported potential values shall be converted to millivolts (SCE) by reference to standard values with respect to the standard hydrogen electrode. For example, E(SCE) = E(SSE) – 44 mV.

4.2.4.8 pH meter, calibrated, with a sensitivity of ±0,05 pH.

4.2.4.9 Silicon carbide paper, in accordance with ISO 6344-1.

4.2.4.10 Diamond paste,1 µm.

4.2.4.11 Micrometer screw gauge, accurate and reading to 0,01 mm.

4.2.4.12 Light microscope, minimum magnification of 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 ±0,1 using 1 % $C_3H_6O_3$ (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 or 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, 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 cm^2 after preparation.

4.2.6.4 Preparation

Remove any sprues, runners or other projections from the surface. Blast all surfaces with 110 µm to 250 µm pure alumina to remove investment.

For precious metals, it is recommended to blast all surfaces with 110 µm pure alumina. For non-precious metals, it is recommended to blast with 250 µm pure alumina.

It is advised that the removal of sprues, runners and other projections is done cold (i.e. under running water) to prevent transformations.

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

In the case of metals supplied for metal-ceramic restorations, test after the following simulated ceramic-firing schedule has been applied.

- Following the manufacturer's instructions, simulate the oxidation procedure and four ceramic firings at the highest temperature recommended for fusing ceramic to the metallic material. Remove and place the specimens on a ceramic plate (which is at room temperature) to cool to room temperature after the oxidation and ceramic firing simulation.
- 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 such 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, as determined using a measuring instrument [e.g. micrometer screw 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 cm^2.
- Manually clean surface with acetone (4.2.3.7). Clean surfaces ultrasonically in the following sequence: alcohol (4.2.3.6), then 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

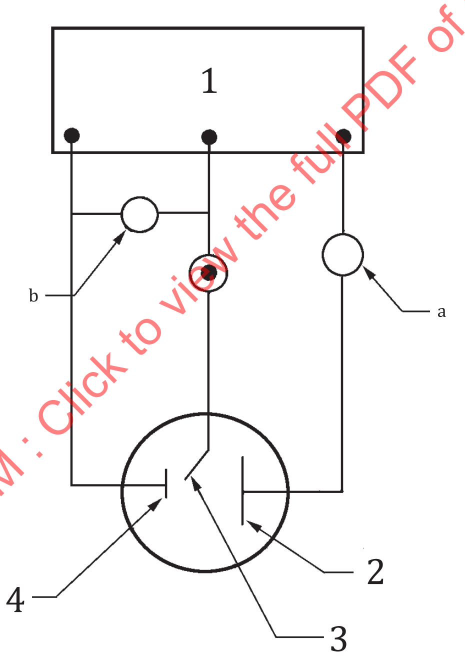
An example of an electrochemical measuring circuit is shown in Figure 1.

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

Fill a 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.

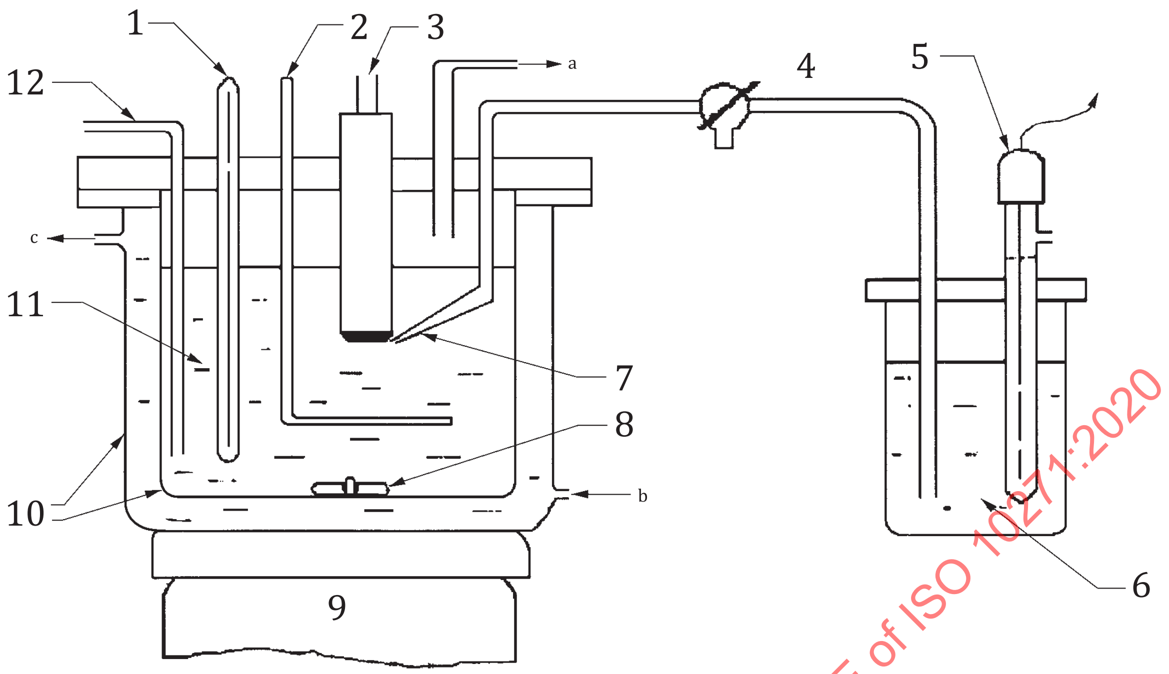
Place the counter electrode(s) in the test cell followed by the reference electrode. Then place the working electrode in the test cell without immersion. Activate the magnetic stirrer. Bubble oxygen-free nitrogen or argon at a rate of about 100 cm^3/min through the electrolyte for at least 30 min. Immerse the working electrode in the electrolyte. Adjust the reference electrode. Reduce the gas flow rate to give a slight bubbling. Begin the measurement procedure.



Key

1 potentiostat
2 counter electrode
3 reference electrode
4 working electrode
[a] Current measurement.
[b] Potential measurement.

Figure 1 — Schematic diagram of measuring circuit



Key

1	thermometer	9	magnetic agitator (motor)
2	counter electrode	10	double-walled vessel
3	working electrode	11	electrolyte
4	electrolytic bridge	12	bubbler (nitrogen or argon)
5	reference electrode	a	Gas outlet.
6	saturated solution of KCl	b	Water inlet.
7	Luggin capillary	c	Water outlet.
8	magnetic stirrer (PTFE-coated)		

Figure 2 — Schematic diagram of electrolytic cell

4.2.7.2 Open-circuit potential measurement

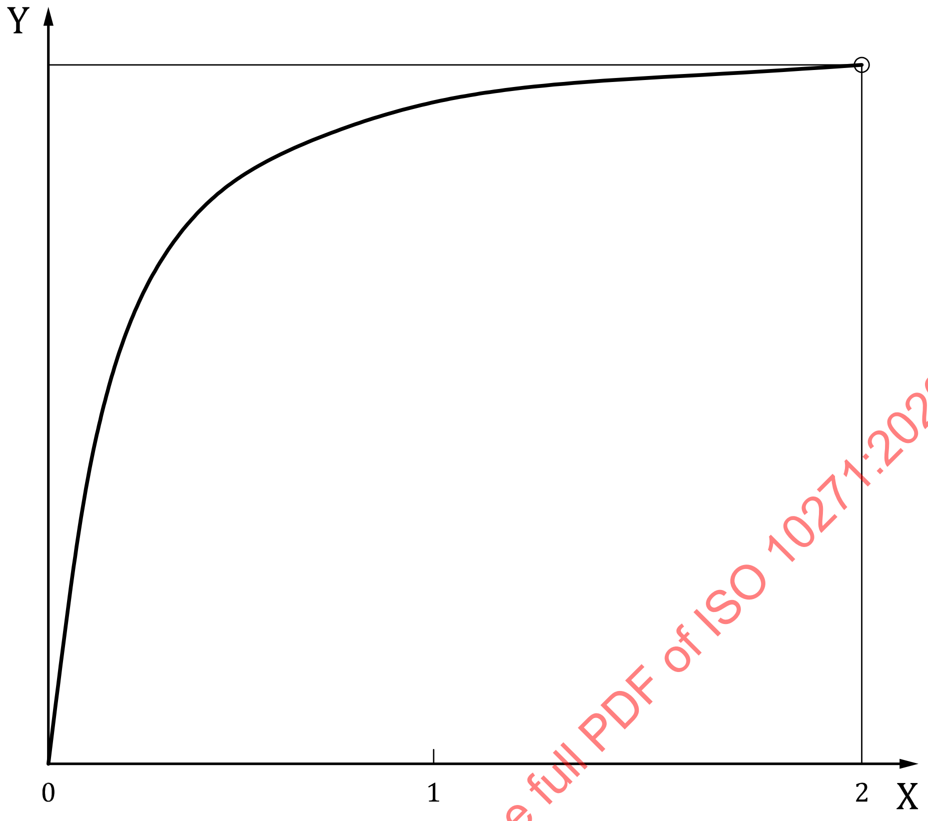
Record the open-circuit potential versus time for 2 h. Determine the open-circuit potential (E_{ocp}) in mV (SCE) after immersion for 2 h ± 6 min. An example of a plot of potential versus time is shown in Figure 3.

If a reference electrode other than a SCE is used, all reported potential values shall be converted to millivolts (SCE) by reference to standard values with respect to the standard hydrogen electrode. For example, E(SCE) = E(SSE) – 44 mV.

4.2.7.3 Potential measurements (anodic polarization)

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

Use a potentiodynamic sweep rate of no more than 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 potential versus the 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 plot of potential versus log(current density) is shown in Figure 4.

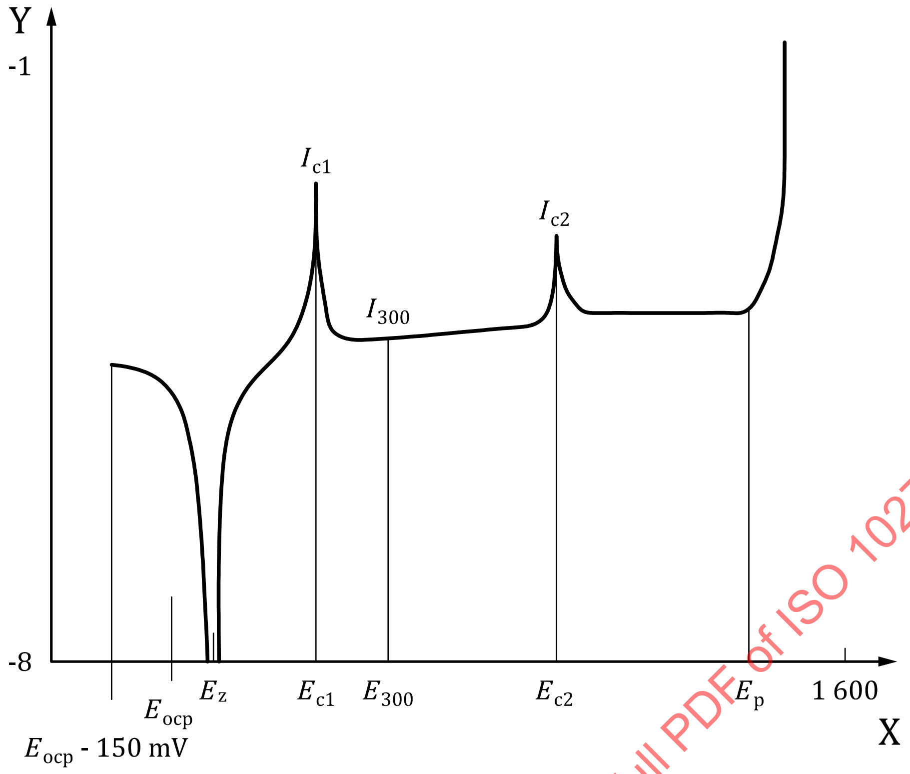


Key

X time, in hours

Y potential, E_{ocp}, in millivolts (SCE)

Figure 3 — Open-circuit potential versus time



Key

X	potential, in mV (SCE)
Y	log(current density), expressed as log(A/cm^2)
I_{c1}	current density at E_{c1}
I_{300}	current density at E_{300}
I_{c2}	current density at E_{c2}
E_{ocp} − 150 mV	potential at the open-circuit potential minus 150 mV
E_{ocp}	open-circuit potential
E_z	zero-current potential
E_{c1}	first active peak potential
E_{300}	potential at the open-circuit potential plus 300 mV
E_{c2}	second active peak potential
E_p	breakdown potential

Figure 4 — Log (current density) versus potential

4.2.8 Test report

The test report shall contain the following information:

a) the identity of the test metallic material;

b) details of the heat treatment, if applicable;

c) the test temperature and, if 37 °C is used, the reason for this choice;

d) a description of any deviations from the preparation of specimens or test procedure;

e) the open-circuit potential, E_{ocp}, expressed in millivolts (SCE);

f) the potential versus log current density curve or potential versus current density curve;

g) the zero-current potential, E_z, expressed in millivolts (SCE);

h) the breakdown potential, E_p, expressed in millivolts (SCE), with the corresponding current density, I_p, expressed in amperes per square centimetre;

i) the 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) the current density, I_{300}, expressed in amperes per square centimetre at potential of (E_z + 300) mV (SCE);

k) a description of any significant changes of the electrolyte or the metallic material surface;

l) the International Standard used (including its year of publication);

m) the method used (in this case, “electrochemical test”);

n) the date of the test.

If a reference electrode other than SCE is used, all reported potential values shall be converted to millivolts (SCE) by reference to standard values with respect to the standard hydrogen electrode. For example, E(SCE) = E(SSE) − 44 mV.

4.3 Sulfide tarnish test (cyclic immersion)

4.3.1 Information required

Composition, including hazardous elements, in accordance with the appropriate International Standard, shall be provided.

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.

Sodium sulfide, nonahydrate ($Na_2S·9H_2O$), ≥ 98 % may also be used.

NOTE Suitable sodium sulfide is found in various hydrated forms. If the proper chemical analysis is provided, an alternative sodium sulfide is acceptable (see 4.3.5).

4.3.3.2 Water, in accordance with grade 2 of ISO 3696.

4.3.3.3 Ethanol or **methanol** (C_2H_5OH or CH_3OH), analytical grade.

4.3.3.4 Acetone [dimethyl ketone, $(CH_3)_2CO$], analytical grade.

4.3.3.5 Compressed air, oil- and water-free, in accordance 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, in accordance with ISO 6344-1.

4.3.4.3 Micrometer screw gauge, accurate and reading to 0,01 mm.

4.3.5 Solution preparation

Prepare 1 000 ml of test solution by dissolving enough sodium sulfide hydrate (4.3.3.1) in water (4.3.3.2) to reach a concentration of Na_2S of (0,100 ± 0,005) mol/l. Prepare a fresh solution for each test, no longer than 24 h before the start of the test.

NOTE When sodium sulfide nonahydrate is used, dissolve 24 g in 1 000 ml water (4.3.3.2).

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 cm^2.

4.3.6.4 Preparation

Remove any sprues, runners or other projections from the surface. Blast all surfaces with 110 µm to 250 µm pure alumina to remove investment.

For precious metals, it is recommended to blast all surfaces with 110 µm pure alumina. For non-precious metals, it is recommended to blast with 250 µm pure alumina.

It is advised that the removal of sprues, runners and other projections is done cold (i.e. under running water) to prevent transformations.

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

In the case of metals supplied for metal-ceramic restorations, test after the following simulated ceramic-firing schedule has been applied.

- Simulate the oxidation procedure and four ceramic firings at the highest temperature allowed for ceramics recommended for fusing to the metallic material in accordance with the manufacturer’s instructions. Remove and place the specimens on a ceramic plate (which is at room temperature) to cool to room temperature after the oxidation and ceramic firing simulation.
- Cold-mount each specimen. The recommended procedure is that of embedding in epoxy resin.
- Remove at least 0,1 mm, as determined using a measuring instrument [e.g. micrometer screw 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 the surface with acetone (4.3.3.4). Clean the surface ultrasonically in the following sequence: alcohol (4.3.3.3), then 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 fume 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) any deviations from the specimen preparation and/or test procedure and their justification;

b) details of the heat treatment if applicable;

c) any visible differences from the untreated specimen with regard to colour and reflectivity;

d) the International Standard used (including its year of publication);

e) the method used [in this case, “sulfide tarnish test (cyclic immersion)”];

f) the date of the test.

4.4 Sulfide tarnish test (static immersion)

4.4.1 Information required

Composition, including hazardous elements, in accordance with the appropriate International Standard shall be provided.

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, hydrate (approximately 35 % Na_2S), analytical grade.

Sodium sulfide, nonahydrate ($Na_2S{\cdot}9H_2O$), ≥ 98 % may also be used.

NOTE Suitable sodium sulfide is found in various hydrated forms. If the proper chemical analysis is provided, an alternative sodium sulfide is acceptable (see 4.4.5).

4.4.3.2 Water, in accordance with grade 2 of ISO 3696.

4.4.3.3 Ethanol or **methanol** (C_2H_5OH or CH_3OH), analytical grade.

4.4.3.4 Compressed air, oil- and water-free, in accordance 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), in accordance 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, in accordance with ISO 6344-1.

4.4.4.3 Micrometer screw gauge, accurate and reading to 0,01 mm.

4.4.5 Solution preparation

Prepare 1 000 ml of test solution by dissolving enough sodium sulfide (4.4.3.1) in water (4.4.3.2) to reach a concentration of Na_2S of (0,012 9 ± 0,000 2) mol/l. Prepare a fresh solution for each test, no longer than 24 h before the start of the test.

NOTE When sodium sulfide nonahydrate is used, dissolve (3,10 ± 0,05) g in 1 000 ml water (4.4.3.2).

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, and at least one further reference specimen shall be prepared for comparison (see 4.4.8).

4.4.6.3 Sample size

The size of each specimen shall be (15,0 ± 0,5) mm in width × (20,0 ± 0,5) mm in length × (1,0 ± 0,5) mm in thickness.

4.4.6.4 Preparation

Remove any sprues, runners or other projections from surface. Blast all surfaces with 110 µm to 250 µm pure alumina to remove investment.

For precious metals, it is recommended to blast all surfaces with 110 µm pure alumina. For non-precious metals, it is recommended to blast with 250 µm pure alumina.

It is advised that the removal of sprues, runners and other projections is done cold (i.e. under running water) to prevent transformations.

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

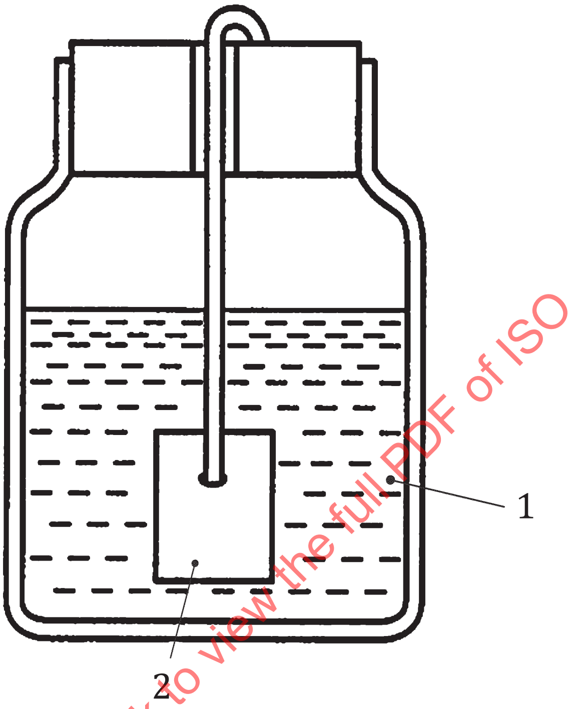
In the case of metals supplied for metal-ceramic restorations, test after the following simulated ceramic-firing schedule has been applied.

- Following the manufacturer's instructions, simulate the oxidation procedure and four ceramic firings at the highest temperature recommended for fusing ceramic to the metallic material. Remove and place the specimens on a ceramic plate (which is at room temperature) to cool to room temperature after the oxidation and ceramic firing simulation.
- Remove at least 0,1 mm, as determined using a measuring instrument [e.g. micrometer screw 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), then water (4.4.3.2), for 2 min each. Dry with oil- and water-free compressed air (4.4.3.4).
- The reference specimen(s) shall be stored in air over a desiccant at ambient temperature, (23 ± 2) °C.

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 ± 2) °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 — Example of static immersion test apparatus

4.4.8 Inspection

Compare treated and reference (untreated) surfaces visually without magnification for any surface deterioration 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) any deviations from the specimen preparation or test procedure and their justification;

b) details of the heat treatment if applicable;

c) any visible differences from the untreated reference specimen(s) with regard to colour and reflectivity;

d) the International Standard used (including its year of publication);

e) the method used [in this case, “sulfide tarnish test (static immersion)”];

f) the date of the test.

4.5 Static immersion test with periodic analysis

4.5.1 Information required

Composition, including hazardous elements, in accordance with the appropriate International Standard shall be provided.

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, 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 behaviour of a metallic material for dental applications can be obtained.

4.5.3 Reagents

4.5.3.1 Lactic acid (2-hydroxypropanoic acid, $C_3H_6O_3$), ≥ 85 %, analytical grade.

4.5.3.2 Sodium chloride (NaCl), analytical grade.

4.5.3.3 Water, in accordance 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 Compressed air, oil- and water-free, in accordance with ISO 7183.

4.5.4 Apparatus

4.5.4.1 Containers, of borosilicate glass, in accordance with ISO 3585, with dimensions of approximately 16 mm inner diameter by 160 mm in height.

4.5.4.2 pH meter, calibrated, with a sensitivity of ±0,05 pH.

4.5.4.3 Chemical analysis instrumentation, capable of measuring ion concentration in µg/ml (e.g. ICP and AAS).

4.5.4.4 Micrometer screw gauge, accurate and reading to 0,01 mm.

4.5.4.5 Silicon carbide paper, in accordance with ISO 6344-1.

4.5.4.6 Volumetric flasks, of borosilicate glass, 1 000 ml, class A, in accordance 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 within a few hours of use. For example, dissolve (10,0 ± 0,1) g ≥ 85 % $C_3H_6O_3$ (4.5.3.1) and (5,850 ± 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 the mark. The pH shall be 2,3 ± 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 or 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, 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 the surface. Blast all surfaces with 110 µm to 250 µm pure alumina to remove investment.

For precious metals, it is recommended to blast all surfaces with 110 µm pure alumina. For non-precious metals, it is recommended to blast with 250 µm pure alumina.

It is advised that the removal of sprues, runners and other projections is done cold (i.e. under running water) to prevent transformations.

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

In the case of metals supplied for metal-ceramic restorations, test after the following simulated ceramic-firing schedule has been applied.

- Following the manufacturer’s instructions, simulate the oxidation procedure and four ceramic firings at the highest temperature recommended for fusing ceramic to the metallic material. Remove and place the specimens on a ceramic plate (which is at room temperature) to cool to room temperature after the oxidation and ceramic firing simulation.
- Remove at least 0,1 mm, as determined using a measuring instrument [e.g. micrometer screw 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 specimens 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 has any porosity visible on any surface intended to be exposed to the test 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, according to the manufacturer's instructions.

Remove at least 0,1 mm, as determined using a measuring instrument [e.g. micrometer screw 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 cm^2.

Clean specimens 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 or devices

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

Determine each sample surface area to within ±0,1 cm^2.

Clean specimens 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 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 cm^2 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 water (4.5.3.3), and place the set of specimens into a new container(s) with fresh solution as described above.

Analyse 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 (the "blank") shall be used to establish the impurity concentration 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 interval	Total 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. Analyse 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) the method of analysis and detection limits of all the analysed elements;

b) any deviations from the preparation of specimens (see 4.5.6.4) or test procedure (see 4.5.7) and their justification;

c) the number of specimens making up a set and the number of sets tested (see 4.5.6.2);

d) the calculated 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 for each element indicated in 4.5.1 as well as any others found; the calculated total ion release over the total elapsed time of the test for each element of each set separately, which for an individual element in a set can be 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; the calculated ion release rate in µg/cm^2 per day for each element of each set separately, which for an individual element in a set can be 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; the sum of the ion release rate values for all the elements of each set (i.e. the total ion release rate for the dental material); and the average total ion release rate for the dental material in micrograms per square centimetre per day (i.e. the total ion release rate divided by the number of sets tested);

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) the presence of undissolved corrosion products, such as precipitates, and any discoloration of the specimen surface;

f) the International Standard used (including its year of publication);

g) the method used (in this case, “static immersion test with periodic analysis”);

h) the date of the test.

4.6 Dental amalgam

See ISO/TS 17988.

ISO/TS 17988 shall be considered when testing the corrosion behaviour of dental amalgam. Test methods 4.1 to 4.5 are not applicable to the evaluation of dental amalgam.

4.7 Crevice corrosion

4.7.1 Principle

Spontaneous crevice corrosion occurs with many metals when the rate of diffusion of oxygen into the deepest part of the space is less than the rate of its reduction, leading to a localized reduction in pH. Such a crevice is then said to be anodically activated, the resulting polarization tending to protect the free surfaces cathodically, and the rate of loss of metal in the crevice may be many times that from the free surfaces.

4.7.2 Application

This test method is suitable for all dental metallic materials in that all applications involve an interface with a second body, whether or not intended to be retained in place by a third body (a cement or adhesive material). The second body may be metallic or otherwise, and if metallic then similar or otherwise. However, galvanic corrosion is not of concern here, merely the effect of a crevice on the first (test) body.

4.7.3 Test medium

Artificial saliva (see A.5.2) at (37 ± 1) °C is the medium of choice for all materials intended to be exposed to the intraoral environment. Artificial plaque fluid (see A.5.3) would be appropriate for materials used in sites susceptible to plaque accumulation, while an artificial body fluid would be appropriate for implanted materials.

4.7.4 Materials

4.7.4.1 Water, in accordance with grade 2 of ISO 3696.

4.7.4.2 Ethanol or **methanol** (C_2H_5OH or CH_3OH), analytical grade.

4.7.4.3 Compressed air, oil- and water-free, in accordance with ISO 7183.

4.7.5 Apparatus

4.7.5.1 Container, volume approximately 25 ml, of non-metallic, non-contaminating, optically transparent material such as medical use-grade polystyrene, preferably with a flat internal bottom surface 20 mm across. A non-metallic closure (e.g. snap-on cap) shall be used.

NOTE A suitable container is a blood dilution vial (V130[2]).

4.7.5.2 Microscope slide cover slip, or equivalent optically flat object (see 4.7.6.2), capable of being laid on the bottom of the container (4.7.5.1) when that container does not have a flat bottom.

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[2] V130 is the trade name of a product supplied by Simport® Scientific, Beloeil, QC, Canada. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

4.7.6 Specimen

4.7.6.1 Size

The test specimen shall be (10 ± 1) mm across and may be square or circular.

NOTE Thickness of the specimen is not critical.

4.7.6.2 Preparation

One surface shall be prepared to be optically flat (which, for the purposes of this document, is defined as having a deviation in flatness of no more than λ/2 over the entire surface, with λ being a reference wavelength of 632,8 nm), or as close to this as metallographic techniques permit; this is the test surface. The remaining (free) surface(s) may be in any convenient form, but shall be finished according to the manufacturer's instructions, if any, or according to conventional dental practice for the material's application.

NOTE For the purposes of this document, optical flatness can be verified by using a certified optical flat, with a maximum peak to valley deviation of λ/2, in combination with a monochromatic light source.

For metallographic preparation of the test surface, care should be taken to use a napless cloth as the abrasive carrier, and to further consider that significant rounding of the edges of the specimen have been noted when not supported by a medium at least as hard as the specimen material.

For cast specimens, remove any sprues, runners or other projections from the surface. Blast all surfaces with 110 µm to 250 µm pure alumina to remove investment.

For precious metals, it is recommended to blast all surfaces with 110 µm pure alumina. For non-precious metals, it is recommended to blast with 250 µm pure alumina.

It is advised that the removal of sprues, runners and other projections is done cold (i.e. under running water) to prevent transformations.

For prefabricated parts or devices, as large a flat area as is conveniently possible should be ground using standard metallographic techniques. Subsequently, treat all surfaces according to the manufacturer's instructions for clinical use, if any.

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

In the case of metals supplied for metal-ceramic restorations, test after the following simulated ceramic-firing schedule has been applied.

- Following the manufacturer's instructions, simulate the oxidation procedure and four ceramic firings at the highest temperature recommended for fusing ceramic to the metallic material. Remove and place the specimens on a ceramic plate (which is at room temperature) to cool to room temperature after the oxidation and ceramic firing simulation.
- The test surface shall be finished according to the relevant requirements appropriate to the context. Thus, a cementation surface shall be treated according to manufacturer's instructions or conventional dental practice, and similarly surfaces intended to carry a ceramic coating shall be subject to the usual treatment(s). Test specimens for other contexts shall be prepared on the same principle. A given material may have two or more such contexts where crevice corrosion is a possibility. All should be tested.
- Clean specimens ultrasonically for 2 min in ethanol or methanol (4.7.4.2). Rinse with water (4.7.4.1).
- A reference specimen shall be prepared in identical fashion, then dried with a jet of oil-free air (4.7.4.3).
- If a test specimen has any porosity visible on any free surface, the specimen shall be rejected and replaced with a new one. Such defects on the test surface are not disqualifying as they are then

considered inherent in the material. Any defects on any surface of a reference specimen are not disqualifying.

NOTE A photographic record of such defects on a test surface is helpful for later comparison.

4.7.7 Procedure

Into the container (4.7.5.1) shall be placed (20,0 ± 0,1) ml of the test medium. If needed, the microscope slide cover slip (4.7.5.2) shall then be inserted to lie on the bottom of the container (4.7.5.1) without trapping any bubble (to avoid the possibility of subsequent movement). The test specimen shall then be placed, using non-contaminating tools as required, with the test surface resting on the bottom of the container (4.7.5.1) or the cover slip (4.7.5.2), ensuring that no bubble is trapped between the two. The container shall then be closed and placed in an incubator at (37 ± 1) °C where it shall remain completely undisturbed for the duration of the test. No less than one month is an appropriate test exposure.

The reference specimen shall be stored in air over a desiccant at (37 ± 1) °C.

At the completion of the test period, the test specimen shall be retrieved without making contact with the test surface, rinsed with water (4.7.4.1) and dried with a jet of oil-free air (4.7.4.3).

4.7.8 Inspection

Compare test and reference surfaces visually without magnification for any corrosion products, surface degradation, discoloration or other change in appearance. See paragraphs 1 and 2 of A.7.3.1.

Loosely adherent corrosion products, if any, may then be removed from the test surface by gentle brushing with a very soft-bristle brush or pad of non-contaminating polymeric or natural bristles or fabric, and then the test surface re-inspected.

Removed corrosion products may be retained for subsequent analysis, including any found loose in the container.

4.7.9 Test report

The test report shall contain the following information:

a) the methods of preparation used for both the test and free surfaces, in the context of the test;

b) the test medium;

c) any deviations from the required preparation process for specimens or the test procedure and their justification;

d) the number of specimens tested;

e) the number of discarded specimens and the reasons for that;

f) whether any defects were apparent on the test surface of any specimen;

g) fully on all visible changes to and on the test surface, including but not limited to undissolved corrosion products in situ, and their colours, and any pitting or discoloration of the test surface; if more than one test specimen was used, on the uniformity and consistency or otherwise of any changes; and if defects were noted on the test surface before testing, any changes associated with those defects;

h) fully on all visible changes to and on the free surfaces, including but not limited to undissolved corrosion products in situ, and any pitting or discoloration; and if more than one test specimen was used, on the uniformity and consistency or otherwise of any changes;

i) the International Standard used (including its year of publication);

j) the method used (in this case "crevice corrosion");

k) the date of the test.

NOTE Annex A provides guidance on corrosion test method development.