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## Dentistry — Corrosion test methods for metallic materials

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

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ISO copyright office  
CP 401 • Ch. de Blandonnet 8  
CH-1214 Vernier, Geneva  
Phone: +41 22 749 01 11  
Email: [copyright@iso.org](mailto:copyright@iso.org)  
Website: [www.iso.org](http://www.iso.org)

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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](http://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](http://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](http://www.iso.org/iso/foreword.html).

This document was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 2, *Prosthetic 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  $\mu\text{g}$  in seven days for a specified number of orthodontic brackets”;
- since sodium sulfide hydrate (approximately 35 %  $\text{Na}_2\text{S}$ ) analytical grade is not available in every country, text was added to the appropriate test methods indicating that sodium sulfidenonahydrate ( $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ ),  $\leq 98\%$  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](http://www.iso.org/members.html).

## Introduction

This document was developed from the original Technical Report (ISO/TR 10271<sup>1)</sup>) 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  $\mu\text{g}$  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<sub>2</sub>S) 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<sub>2</sub>S·9H<sub>2</sub>O), ≤ 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.

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# 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,  $C_3H_6O_3$ ),  $\geq 85\%$ , 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  $\pm 0,05$  pH.

**4.1.4.3 Chemical analysis instrumentation**, capable of measuring ion concentration in  $\mu\text{g/ml}$ , 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  $(10,0 \pm 0,1)$  g  $\geq 85\%$   $\text{C}_3\text{H}_6\text{O}_3$  (4.1.3.1) and  $(5,850 \pm 0,005)$  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 the mark. The pH shall be  $2,3 \pm 0,1$ . 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<sup>2</sup> 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  $\mu\text{g}$  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  $\mu\text{m}$  to 250  $\mu\text{m}$  pure alumina to remove investment.

For precious metals, it is recommended to blast all surfaces with 110  $\mu\text{m}$  pure alumina. For non-precious metals, it is recommended to blast with 250  $\mu\text{m}$  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.