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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
Website: www.iso.org

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Foreword

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

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).

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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 ($Na_2S{\cdot}9H_2O$), ≤ 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."

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

