DRAFT INTERNATIONAL STANDARD ISO/DIS 10271

ISO/TC **106**/SC **2** Secretariat: **ANSI**

Voting begins on: Voting terminates on:

2019-04-24 2019-07-17

Dentistry — Corrosion test methods for metallic materials

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

ICS: 11.060.10

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Published in Switzerland

Contents						
Fore	eword		v			
Intr	oductio	n	vi			
1		e				
_	Normative references					
2						
3	Term	Terms and definitions				
4	Test	methods	3			
	4.1	Static immersion test				
		4.1.1 Information required				
		4.1.2 Application				
		4.1.3 Reagents				
		4.1.4 Apparatus				
		4.1.5 Solution preparation 4.1.6 Samples				
		4.1.7 Test procedure				
		1 L				
		4.1.8 Elemental analysis 4.1.9 Test report Electrochemical test 4.2.1 Information required 4.2.2 Application 4.2.3 Reagents 4.2.4 Apparatus 4.2.5 Solution preparation 4.2.6 Samples 4.2.7 Test procedure 4.2.8 Test report Sulfide tarnish test (cyclic immersion)	6			
	4.2	Electrochemical test	6			
		4.2.1 Information required	6			
		4.2.2 Application	6			
		4.2.3 Reagents	6			
		4.2.4 Apparatus	7			
		4.2.5 Solution preparation	7			
		4.2.6 Samples				
		4.2.7 Test procedure	9			
	4.3	4.2.8 Test report Sulfide tarnish test (cyclic immersion)	12			
	4.3	4.3.1 Information required				
		4.3.2 Application	13			
		4.3.2 Application 4.3.3 Reagents	13			
		4.3.4 Apparatus	13			
		4.3.5 Solution preparation				
		4.3.6 Samples 4.3.6				
		4.3.7 Test procedure				
		4.3.8 Inspection	15			
		4.3.9 Test report				
	4.4	Sulfide tarnish test (static immersion)				
		4.4.1 Information required				
		4.4.2 Application				
		4.4.3 Reagents				
		4.4.4 Apparatus 4.4.5 Solution preparation				
		4.4.5 Solution preparation 4.4.6 Samples				
		4.4.7 Test procedure				
		4.4.8 Inspection				
		4.4.9 Test report				
	4.5	Static immersion test with periodic analysis				
		4.5.1 Information required				
		4.5.2 Application				
		4.5.3 Reagents				
		4.5.4 Apparatus				
		4.5.5 Solution preparation				
		4.5.6 Samples				
		4.5.7 Test procedure	20			

ISO/DIS 10271:2019(E)

	4.5.8	Elemental analysis	21
	4.5.9	Test report	21
4.6	Dental	Amalgam	21
4.7		e Corrosion	21
	4.7.1	Principle Application	21
	4.7.2	Application	22
	4.7.3	Test medium	22
	4.7.4	Materials	22
	4.7.5	Apparatus	2.2
	4.7.6	Test piece	22
	4.7.7	Procedure	23
	4.7.8	Inspection	23
	4.7.9	Test report	23
Annex A (info	ormative	e) Corrosion test method development	25
Bibliography	31		

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Foreword

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

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

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

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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

This third edition cancels and replaces the second edition (ISO 10271:2011), which has been technically revised, in particular by removing the statement in the Scope about the standard not being applicable to "appliances for orthodontics" and adding a NOTE to the static immersion test method acknowledging that "measuring the total surface area of orthodontic appliances can be difficult" and "this test method may be referenced by other standards with appropriate modifications". Also, since sodium sulfide hydrate (approximately 35% Na₂S) analytical grade is not available in every country, a NOTE was added to the appropriate test methods indicating that sodium sulphide nonahydrate (Na₂S·9H₂O), \leq 98% may be used. Furthermore, this third edition of ISO 10271 was 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.

Introduction

This document was developed from the original Technical Report (ISO/TR 10271) 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 ISO 10271. This test has been used successfully for many years to evaluate the corrosion of silver alloys.

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

The third edition differs from the second by the removal of the statement in the Scope about the standard not being applicable to "appliances for orthodontics". With this 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 "this test method may be referenced by other standards with appropriate modifications".

Besides updating the Normative References and Terms and definitions according to the newest version of the ISO Directives, the third edition of ISO 10271 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:

"Simulate the oxidation procedure and four ceramic firings at the highest temperature allowed for the 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."

Additionally, since sodium sulfide hydrate (approximately 35% Na₂S) analytical grade is not available in every country, this third edition includes a NOTE, which was added to the appropriate test methods, indicating that sodium sulphide nonahydrate (Na₂S·9H2O), \leq 98% may be used.

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 provides 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 instruments and dental amalgam (see ISO TS 17988).

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/ui

IEC Electropedia: available at www.electropedia.org

3.1

breakdown potential

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

3.2

corrosion

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

3.3

corrosion potential

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

ISO/DIS 10271:2019(E)

3.4

corrosion product

substance formed as a result of corrosion

3.5

crevice corrosion

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

3.6

current density

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

3.7

dynamic immersion test

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

3.8

electrode potential

potential difference between the specimen and a reference electrode

3.9

electrolyte

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

3.10

open-circuit potential

 E_{ocp}

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

3.11

pitting corrosion

localized corrosion which results in pits

3.12

potentiodynamic test

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

3.13

potentiostatic test

test in which the electrode potential is maintained constant

3.14

sample

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

3.15

set

subgroup of the specimens of a sample

3.16

specimen

test piece

individual single example of an object for testing

3.17

static immersion test

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

3.18

stress corrosion

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

3.19

synthetic saliva

test medium that approximates the relevant chemistry of natural saliva

3.20

tarnish

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

3.21

zero-current potential

potential at which cathodic and anodic currents are equal

4 Test methods

4.1 Static immersion test

4.1.1 Information required

Composition, including hazardous elements, in accordance with the appropriate ISO standard, 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, C₃H₆O₃), 90 %, analytical grade.
- **4.1.3.2 Sodium chloride** (NaCl), analytical grade.
- **4.1.3.3 Water**, complying with grade 2 of ISO 3696.
- **4.1.3.4 Ethanol** or **methanol** (C₂H₅OH or CH₃OH), analytical grade.
- **4.1.3.5 Compressed air**, oil- and water-free, complying with ISO 7183.

4.1.4 Apparatus

- **4.1.4.1 Containers**, of borosilicate glass, complying with ISO 3585 and with dimensions of approximately 16 mm inner diameter by 160 mm in height.
- **4.1.4.2 pH meter**, calibrated, with a sensitivity of at least ±0,05 pH.
- **4.1.4.3** Chemical analysis instrumentation, capable of measuring ion concentration in μ g/mL (e.g. inductively-coupled plasma atomic emission spectroscopy (ICP-AES), alias 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.

- **Silicon carbide paper**, complying with ISO 6344-1. 4.1.4.5
- **4.1.4.6** Volumetric flasks, of borosilicate glass, 1 000 mL, class A, complying with ISO 1042.

Solution preparation 4.1.5

Prepare an aqueous solution comprising 0,1 mol/L lactic acid and 0,1 mol/L sodium chloride within 2 hours of use. For example, dissolve (10.0 ± 0.1) g 90 % $C_3H_6O_3$ (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 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, etc., shall be tested in the asmanufactured 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).

Sample surface area 4.1.6.3

The total surface area of the sample shall be at least 10 cm² after preparation.

In this latest revision of this document, the Scope has been changed to eliminate the exclusion of appliances for orthodontics. However, it is recognized that measuring the total surface area of orthodontic appliances can be difficult. Therefore, this note acknowledges that this test method may be referenced by other standards with appropriate modifications. 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 $\mu g/7d$ 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 125 µm pure alumina to remove investment.

NOTE Removal should be 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 ceramicfiring schedule has been applied:

Simulate the oxidation procedure and four ceramic firings at the highest temperature allowed for the 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.

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 cm² (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 \pm 0,1 cm² (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 \pm 1) °C for 7 d \pm 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