106

TECHNICAL REPORT

ISO TR 10271

First edition
1993-06-01

Dentistry — Determination of tarnish and corrosion of metals and alloys

Art dentaire — Détermination du ternissement et de la corrosion des métaux et alliages

Reference number
ISO/TR 10271:1993(E)

Foreword

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The main task of technical committees is to prepare International Standards, but in exceptional circumstances a technical committee may propose the publication of a Technical Report of one of the following types:

— type 1, when the required support cannot be obtained for the publication of an International Standard, despite repeated efforts;

— type 2, when the subject is still under technical development or where for any other reason there is the future but not immediate possibility of an agreement on an International Standard;

— type 3, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example).

Technical Reports of types 1 and 2 are subject to review within three years of publication, to decide whether they can be transformed into International Standards. Technical Reports of type 3 do not necessarily have to be reviewed until the data they provide are considered to be no longer valid or useful.

ISO/TR 10271, which is a Technical Report of type 2, was prepared by Technical Committee ISO/TC 106, *Dentistry*, Sub-Committee SC 2, *Prosthodontic materials*.

Annex A of this Technical Report is for information only.

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Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Dentistry — Determination of tarnish and corrosion of metals and alloys

1 Scope

This Technical Report indicates currently available test methods for the tarnish and corrosion behaviour of metals and alloys used in dentistry. It applies to all metals and alloys introduced to the oral cavity such as:

— direct filling metals and alloys;

— cast metals and alloys (including implants);

— wrought metals and alloys (including implants);

— solder/braze filler metals and alloys;

— deposited metals and alloys;

— combinations of the above.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Technical Report. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this Technical Report are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1559:1986, *Dentistry — Alloys for dental amalgam.*

ISO/TR 7405:1984, *Biological evaluation of dental materials.*

3 Definitions

For the purposes of this Technical Report, the following definitions apply.

3.1 corrosion: Physico-chemical interaction between a metal or an alloy and its environment resulting in a partial or total destruction of the metal or in a change of its properties.

3.2 tarnish: Type of corrosion as (surface change) characterized by surface discoloration.

3.3 loss of substance: Reduction of mass of the specimen by corrosion.

3.4 pitting corrosion: Corrosion resulting in pits; i.e. cavities extending from the surface into the metal.

3.5 crevice corrosion: Corrosion associated with, and taking place in, or immediately around, a narrow aperture or clearance.

3.6 corrosion product: Substance formed as a result of corrosion.

3.7 accelerated test: Corrosion test carried out under more severe conditions to yield results in a shorter time than in service.

3.8 *in vitro* test: Test which predicts service or clinical performance using physical, chemical and/or electrical systems in the test laboratory.

3.9 extraction product: Product soluble in an extraction vehicle, obtained from a material.

3.10 extraction vehicle: Liquid selected specifically to obtain extraction products from a material, and simulating human body fluids.

3.11 animal test: Test which relates to clinical performance preliminary to use in humans.

3.12 clinical trial: Test designed for and used in the human oral cavity.

3.13 artificial saliva: Test medium which simulates natural saliva for the performance of a specific test.

4 Requirements for tests

4.1 Test specimens

The materials shall be processed according to the manufacturer's instructions.

4.1.1 General test specimens

Test specimens applicable to the tarnish test, loss of substance, pitting corrosion and crevice corrosion are described in the relevant tests. See table 1.

4.1.2 Test specimens for filling material

The test specimens for filling materials shall be prepared in accordance with ISO 1559, table 1 or applicable national standards.

4.1.3 Specimens for tensile testing

Tensile specimens shall be prepared as shown in the applicable standard with reference to the test method.

4.2 Tests

4.2.1 Static immersion test

This test requires placement in a test solution for a specified period of time.

4.2.2 Rotating immersion test

The apparatus usually consists of a vertical wheel, driven by an electric motor at a specified speed. The wheel consists of a material that neither is attacked by the test solution nor can interfere with the test result in any other way. The wheel shall be provided with fittings so that the specimens can be attached to it. The test solution is placed in such a manner that the specimens are immersed in the solution for each revolution of the wheel.

4.2.3 Loss of substance test

Immersion, electrochemical, nuclear and photometric tests are described in table 1.

4.2.4 Potentiodynamic polarization test

See table 1.

4.2.5 Potentiostatic polarization

See table 1.

4.2.6 Pitting corrosion test

See table 1.

4.2.7 Crevice corrosion test

See table 1.

4.2.8 Corrosion cracking test

See table 1.

4.2.9 Tarnish test

See table 1.

5 Deleterious effects

The deleterious effects of tarnish and corrosion may be defined in three areas.

5.1 Safety: generation of harmful corrosion products

Corrosion and tarnish denote the formation of metal compounds, whether formed by simple oxidation-type reactions or by the release of metal or metal ions. The harmful effects of these metals or metal compounds, such as oxides, sulfides, chlorides, metal organics or other species need evaluation for their effect on the soft and hard tissues and other parts of the body, in accordance with ISO/TR 7405.

Harmful effects may include, for instance, inflammatory, allergenic, cariogenic, mutagenic, carcinogenic, toxic, cytotoxic, teratogenic, or distasteful effects.

Electrical currents and pain can occur due to the use of dissimilar metals and alloys, and galvanic action.

5.2 Efficacy

5.2.1 Loss of substance

If the loss is sufficient to reduce section thickness significantly, it is conceivable that enough mechanical strength will be lost that failure will occur by bending or fracture. Where the loss of material occurs by pitting or roughening, sites can be formed for plaque deposition and growth. Pitting may initiate fatigue fracture.

5.2.2 Metal/non-metal interface

Where corrosion occurs at the interface of metal with ceramic, plastic, cement, etc., the bond or adhesion between the two materials may be weakened or destroyed, or a crevice can be created or enlarged. The result may be loss of the non-metal, discoloration of the non-metal or increased corrosion within the crevice. At the interface between metal and hard or soft tissue, corrosion is more likely because of oxygen depletion.

5.2.3 Stress corrosion cracking

Failure may occur with the combined action of corrosion and stress.

5.2.4 Corrosion fatigue

Service life may be reduced from repeated or fluctuating load when occurring in a corrosive environment.

5.3 Aesthetics

Corrosion may result in loss of lustre since reflected light is diffused, giving the appearance of a discoloration. Actual discoloration results from coloured reaction products which are tenacious enough to remain on the surface. Such coloured or uncoloured reaction products might even protect against further reaction. Well known coloured reaction products are silver and copper sulfides. With many other elements being tested for use in dental alloys, other coloured compounds are possibilities in future alloys. Discoloration in itself, where the coloured film is not a part of another corrosion reaction, does not affect safety or efficacy. Therefore this aspect can be ignored in this context and the market will be the determining factor as to whether a discolouring metal is acceptable.

6 Proposal for future testing

It is well established that no single test can give a complete indication of the reaction between metal or alloy, and the oral environment. This Report therefore, suggests only types of tests and the type of information to be gathered which will lead to an International Standard.

6.1 Testing regarding safety

a) immersion tests (including depleted oxygen tests);

b) electrochemical tests (including galvanic coupling);

c) identify and quantify corrosion products:

   — identify harmful corrosion products and rate of release,

   — establish acceptable limits of concentration of harmful products based on release ratio in the oral cavity.

d) correlate *in vitro* with *in vivo*. From identification of corrosion products, it may be inferred that such products will be produced *in vivo*. Such products should be evaluated for biocompatibility. The simple conclusion that harmful species can be formed and that injury to the patient can be expected may be the basis for rejection of a metal or alloy.

6.2 Testing regarding efficacy

6.2.1 From 6.1 a) to c), determine rate of loss of substance *in vitro*.

Test for loss of substance *in vivo* (providing that reaction products are not found harmful).

Translate this loss of thickness to millimetres per year. Compare with average lifespan of restoration and with normal thicknesses found in restorations of the type for which the metal or alloy would be used.

6.2.2 Pitting corrosion is tested as follows.

Determine density and dimension of pits on samples tested in 6.1 a) to c).

Check *in vivo* results on samples from 6.2.1.

6.2.3 Interface corrosion (including crevice and reduced oxygen corrosion) is tested on specially designed specimens *in vitro* for corrosion in an experimental crevice.

Repeat *in vivo* tests and correlate between *in vitro* and *in vivo* values. Relate the rate of penetration to normal restorations and estimate lifespan.

6.2.4 Stress corrosion cracking is assessed by formation and propagation of cracks when subject to a corrosive environment under stress and dynamic load.

6.2.5 Alloy combinations are assessed by similar tests to 6.2.1, 6.2.2 and 6.2.3, run on soldered samples where a solder is recommended for a particular alloy.

6.3 Testing regarding aesthetics

6.3.1 Test by the methods of 6.1 a) and b).

The acceptance of discoloration may vary considerably depending on the standards required by peoples of differing social origin. The specification shall be set in such a manner that it can be varied according to local circumstances and in a voluntary form so acceptable alloys will not be ruled out.

6.3.2 Tenacity of film is assessed by a toothbrush test as prepared by several researchers (see annex A).

Table 1 — Available corrosion tests for dental materials — Amalgam alloys

No.	Test type/source	Test apparatus	Test medium	Test conditions	Specimen design	Specimen preparation	Test requirements
1	Immersion NIOM	Vessel with 15 ml solution for each specimen	50 g Na_2S, 1 000 ml distilled H_2O changed each week	6 specimens 3 months	Immersion specimens ISO 1559	Embedded polish, 1 µm diamond paste	Specimens shall exhibit 95 % compressive strength of non-corroded specimen
2	Potentio-dynamic polarization NIOM	Electrochemical closed, stirred system	Artificial saliva: 0,4 NaCl, 0,4 KCl, 0,795 g of $CaCl.2H_2O$, 0,78 g of $NaH_2PO_4.2H_2O$, 0,005 g of $Na_2S.2H_2O$, 1 g urea, 1 000 ml distilled H_2O	37 °C 2 h O_2 depleted by N_2 bubbling 7 days	Immersion specimens ISO 1559	Embedded polish, 1 µm diamond paste	7 day specimens rupture potential not less than E corr + 250 mV. 1) No crevice or stress corrosion
3	Extraction AFNOR	Autoclave oven, teflon-lined borosilicate glass	Artificial saliva NF 30-S91	Incubation at 37 °C for 120 h	12 × 3 mm disc	600 to 1 200 emery paper	Quantitative analysis of extract
4	Microphotometric Hong Kong	Vessel containing artificial saliva Microscope with photometer	Artificial saliva 3 stock solution (100 ml) NaH_2PO_4 56 NaCl 150 NH_4Cl 22 Na_3 citrate.$2H_2$ 2,2 Lactic acid 7,0 + dist H_2O to 1 000 ml Urea 20 Uric acid 1,5 NaOH 0,4 + dist H_2O to 2 000 ml $NaHCO_3$ 60 NaSCN 20 + dist H_2O to 1 000 ml	4 samples embedded in each specimen 37 °C 22 °C 50 °C	Pellets embedded in polyester, thick	Amalgam triturated *in vacuo* to 10^{-1} torr,	Reproducible screening test using discoloration
5	Electrochemical after immersion NIOM	Neutron irradiation gamma ray spectrophotometry SEM X-ray diffraction	Artificial saliva see 2 above	Thermal neutron flux for 1 h 60 °C to 100 °C immersion 2 h to 30 days	5 × 10 × 0,3 mm	1 000 grit paper grind	Release of Cu, Hg, Zn
6	Electrochemical polarization curves and *in vivo* Australia	Epoxy resin cylinder three electrode system, button in denture	Artificial saliva *in vivo*		4 mm hemispherical buttons	Polished with diamond paste	Comparison of *in vitro* and *in vivo* results
7	Linear polarization and *in vivo* USA	Linear polarization Primates	Artificial saliva	37 °C 45 days, baboon teeth 1 080 h	4 mm^2 to 14 mm^2 samples	Polished with fine emery discs	Anodic and cathodic tafel slopes

1) Start at E corr, make an anodic sweep to + 300 mV, reverse direction to E corr – 1 mV/s. Express in $\mu A/cm^2$

Table 2 — Available corrosion tests for dental materials — Cast metals and alloys

No.	Test type/source	Test apparatus	Test medium	Test conditions	Specimen design	Specimen preparation	Test requirements
8	Immersion DIN	Vertical wheel at a speed of 1 r/min	Na_2S 0,1 mol/l Na_2S 7,8 g/l Na_2S 22,2 g/l Na_2S 7 to 9 H_2O distilled H_2O	Specimens immersed 10 s to 15 s per revolution for 72 h	Embedded specimens	Polish with 1 µm diamond paste	Darkening, loss of lustre compared to untreated specimen
9	Loss of substance, pitting corrosion DIN	Vessel with test medium	Lactic acid 0,1 mol/l NaCl 0,1 mol/l pH 2 distilled H_2O	37 °C weigh specimens to ± 0,1 mg hang vertical in aerated solution 7 days	2 specimens	Polish with 1 µm diamond paste	Visual inspection
10	Crevice corrosion DIN	Vessel with test medium	Lactic acid 0,1 mol/l NaCl 0,1 mol/l pH 2 distilled H_2O	37 °C weigh specimens to ± 0,1 mg on plate glass and solution, aerated 7 days	2 specimens	Polish with 1 µm diamond paste	Average decrease in mass mg/cm^2 compared with above test
11	Stress corrosion DIN	Vessel with test medium	Lactic acid 0,1 mol/l NaCl 0,1 mol/l pH 2 distilled H_2O	37 °C specimens stored in aerated solution 7 days	2 rolled sheets 70 × 8 × 0,5 mm as needed, and bent around a 10 mm diameter cylinder	Rolled around cylinder	Visual inspection for cracks
12	Tarnish USA	Vertically rotating wheel with eight 1-in holes for specimens	0,5 % Na_2S	1 r/min rotation; 15 s immerse mould; 45 s withdraw; mech. wiped	2 × 1/4 × 4 in 1,5 g pcs 0,02 in	Mounted in acrylic grind 600 grit polished 0,3 µm Al_2O_3	Microscopic inspection
13	Linear polarization and *in vivo* USA	Linear polarization Primates	Artificial saliva	37 °C 45 days baboon teeth 1 080 h	4 mm^2 to 14 mm^2 samples	Polished with fine emery discs	Anodic and cathodic tafel slopes
14	Static immersion DIN 13906-2	Vessel with test medium	Lactic acid 0,1 mol/l NaCl 0,1 mol/l pH 2,3	7 days immersion 37 °C	6 specimens 32 × 10 × 1,5 mm	Pickling, blasting, grinding to ASTM 600 grit ≙ FEPA 1 200 SiC paper	Analytical determination of metal ions e.g. AAS or ICP
15	Tarnish Germany	Vertically rotating wheel with eight 1-in holes for specimens	0,1 mol % Na_2S	Immersed 10 s to 15 s per min, 4 320 dippings over 3 days	10 × 10 × 1 mm castings	Pickling, blasting, grinding, polishing, 1 µm diamond paste	Naked eye or microscope do not reveal darkening or loss of lustre
16	Nuclear tracer NIOM	Glass filter frits, neutron flutes	100 ml modified Fusayama solution	15 h to 1 week gamma ray spectrometry	Rectangular foils 1 cm^2 of Au alloy cylindrical discs 5 × 2 mm titanium	Ground 1 000 grit paper	Not available
17	Immersion Japan	Vessel with test medium	0,1 % Na_2S	37 °C ± 2 °C 3 days	15 × 20 × 1 mm Au 58 % Au Pd Ag Ag Pd	Ground to 800 grit	Discoloration limit to 7,5 year 8 + /C6 − to 10 year 7 + /C6 −
25[1)]	Potentiostatic polarization AFNOR	Electrochemical closed system	Artificial saliva	37 °C 2 to 4 h	12 × 3 mm disc dipping contact	Polishing 1 µm diamond paste	Polarization potential E corr + 100 mV E corr + 200 mV
25[1)]	Potentiostatic France	Electrochemical closed system, magnetic agitator Pt counter electrode	Isotonic solution Artificial plasma NaCl 9 g pH 7,4 deionized water NF 97141	37 °C Air or argon 10 mV/s, argon bubbling	Section 0,2 cm^2	SiC 1 200 1 µm diamond paste	Measure intensity µA potential mV/s rest potential after 15 min and 24 h

1) Time and polarization potential depend on concentration of extracted products.

Table 3 — Available corrosion tests for dental materials — Wrought metals and alloys

No.	Test type/source	Test apparatus	Test medium	Test conditions	Specimen design	Specimen preparation	Test requirements
18	Immersion Japan	Vessel with test solution	0,1 % Na_2S 50 ml	37 °C ± 2 °C 3 days	Various	Not available	Discoloration 10 year 7 + /6 –

Table 4 — Available corrosion tests for dental materials — Solders/brazing materials

No.	Test type/source	Test apparatus	Test medium	Test conditions	Specimen design	Specimen preparation	Test requirements
14	Static immersion DIN 13906-2	Vessel with test medium	Lactic acid 0,1 mol/l NaCl 0,1 mol/l pH 2,3	7 days immersion 37 °C	6 specimens 32 × 10 × 1,5 mm	Pickling, blasting, grinding to ASTM 600 grit = FEPA 1 200 SiC paper	Analytical determination of metal ions e.g. AAS or ICP
15	Tarnish Germany	Vertically rotating wheel with eight 1-in holes for specimens	0,1 mol % Na_2S	Immersed 10 s to 15 s per min, 4 320 dippings over 3 days	10 × 10 × 1 mm castings	Pickling, blasting, grinding, polishing, 1 µm diamond paste	Naked eye or microscope do not reveal darkening or loss of lustre
19	Immersion Japan	Vessel with test solution	0,1 % Na_2S 50 ml	37 °C ± 2 °C 3 days	Au Pd Ag Au 15 Au Ag 30 +	Not available	Discoloration 10 year 7 + /6 –
20	Electrochemical NIOM	Electronics with reference electrode and Pt counter electrode	Artificial saliva (see 2)	37 °C – three specimens shall be subjected to potential of E corr + 300 mV	6 tensile specimens	Specimens having a solder joint, 3 specimens untreated	Treated and untreated specimens subjected to tensile test with cross head speed of 0,5 mm/min

Table 5 — Available corrosion tests for dental materials — Deposited metals and alloys

No.	Test type/source	Test apparatus	Test medium	Test conditions	Specimen design	Specimen preparation	Test requirements
21	Immersion NIOM	Vessel with 15 ml solution for each specimen	50 g Na_2S 1 000 ml distilled H_2O, changed each week	6 specimens 3 months	Immersion specimens ISO 1559	Embedded polish, 1 µm diamond paste	Specimens shall exhibit 95 % compressive strength of non-corroded specimen

Table 6 — Available corrosion tests for dental materials — Dental instruments

No.	Test type/source	Test apparatus	Test medium	Test conditions	Specimen design	Specimen preparation	Test requirements
22	Not available						

Table 7 — Available corrosion tests for dental materials — Dental burs

No.	Test type/source	Test apparatus	Test medium	Test conditions	Specimen design	Specimen preparation	Test requirements
23	Not available						

Table 8 — Available corrosion tests for dental materials — Combinations

No.	Test type/source	Test apparatus	Test medium	Test conditions	Specimen design	Specimen preparation	Test requirements
14	Static immersion DIN 13906-2	Vessel with test medium	Lactic acid 0,1 mol/l NaCl 0,1 mol/l pH 2,3	7 days immersion 37 °C	6 specimens 32 × 10 × 1,5 mm	Pickling, blasting, grinding to ASTM 600 grit = FEPA 1 200 SiC paper	Analytical determination of metal ions e.g. AAS or ICP
15	Tarnish Germany	Vertically rotating wheel with eight 1-in holes for specimens	0,1 mol % Na_2S	Immersed 10 s to 15 s per min, 4 320 dippings over 3 days	10 × 10 × 1 mm castings	Pickling, blasting, grinding, polishing, 1 µm diamond paste	Naked eye or microscope do not reveal darkening or loss of lustre
24	Clinical ADA USA	Oral cavity clinical evaluators crowns embedded specimens	Oral cavity	Specimen surface evaluated by clinician every 3 months for 1 year	1. Cast crowns or fixed prostheses 2. Specimens embedded in RPD or full denture	Varies	Specimens *in vivo* compared to control which have not been subjected to oral environment for tarnish. Crowns subjected to margin adaptation test

Annex A
(informative)

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