

TECHNICAL SPECIFICATION

ISO/TS 17576

First edition
2004-01-15

Dentistry — Corrosion tests for amalgam

Art dentaire — Essais de corrosion des amalgames

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Reference number
ISO/TS 17576:2004(E)

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Published in Switzerland

Foreword

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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.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of normative document:

- an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote.
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An ISO/PAS or ISO/TS is reviewed after three years in order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn. If the ISO/PAS or ISO/TS is confirmed, it is reviewed again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

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/TS 17576 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 1, *Filling and restorative materials*.

Introduction

The development of this Technical Specification is the result of worldwide demand for test methods to determine acceptability of dental amalgams for oral restorations in relation to corrosion and mercury vapour.

The test methods described in ISO 10271 are intended for dental metallic materials and are not suitable for amalgams, due to the risk of formation of precipitates during the test. Furthermore, ISO 10271 does not cover measurement of mercury vapour liberated during corrosion of amalgam.

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Dentistry — Corrosion tests for amalgam

1 Scope

This Technical Specification provides two test methods and concomitant protocols to determine the corrosive behaviour of dental amalgams and their tendency to release mercury vapour during corrosion. The methods measure 1) the integrated current in a potentiostatic corrosion test and 2) the amount of released ions and mercury vapour in a static immersion test.

This Technical Specification is not intended to set limits for the relevant parameters in the two tests described.

2 Normative references

The following referenced documents are indispensable for the application 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 1559:1995, *Dental materials — Alloys for dental amalgam*

ISO 3585:1998, *Borosilicate glass 3.3 — Properties*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 10271:2001, *Dental metallic materials — Corrosion test methods*

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3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

amalgam

alloy of mercury with one or more other metals

3.2

corrosion

deterioration of an amalgam by chemical or electrochemical reaction with its environment

NOTE This deterioration is manifest in the loss in mass of the amalgam, with a release of constituent elements or the formation of an adherent film of reaction products.

3.3

electrolyte

liquid that contains ions whose combination allows the conduction of electricity

3.4

immersion test

test in which the sample is exposed to a corrosive solution without application of variable mechanical stresses to the sample

3.5

potentiostatic test

test in which the electrode potential is kept constant

4 Test methods

4.1 Potentiostatic corrosion test for dental amalgam

4.1.1 Specimen preparation

Prepare an amalgam specimen of the type for the measurement of compression strength, in accordance with ISO 1559. Condition in air at (37 ± 2) °C for $(7,0 \pm 0,1)$ days.

Attach an insulated lead to the specimen for connection to the potentiostat.

Cover the connecting lead junction and all surfaces except one end of the specimen with an insulating material, preferably by casting in epoxy resin. The temperature rise during setting of the resin shall not exceed 15 °C. This material shall not dissolve in or react with the electrolyte.

Wet-grind the exposed end of the specimen uniformly to ASTM 600 or FEPA 1200 using silicon carbide surface abrasive paper. Wash with distilled water.

Eliminate the crevice between the amalgam and the embedding materials by casting or masking.

The testing laboratory may develop its own method of specimen preparation, provided the above procedures are included and the conditions are met.

4.1.2 Reagents

4.1.2.1 Electrolyte, of volume ≥ 300 ml.

Make up a fresh solution of NaCl [$c(\text{NaCl}) = 0,154$ mol/l] by adding $(9,0 \pm 0,1)$ g analytical grade NaCl to 600 ml water (ISO 3696, Grade 2), then make up this solution to $(1000,0 \pm 0,5)$ ml.

4.1.3 Apparatus

4.1.3.1 Corrosion cell.

Use a three-electrode corrosion cell holding the specimen (working electrode), a reference electrode probe, and an inert counter-electrode (platinum or carbon is recommended).

4.1.3.2 Temperature control, consisting of a jacket and temperature control/circulator, or a temperature-controlled bath, capable of maintaining $(37,0 \pm 0,5)$ °C in the cell.

4.1.3.3 Reference electrode and its control.

Use any standard reference electrode with a stable potential of known potential difference from a standard hydrogen electrode (SHE). Control the potential at $(0,000 \pm 0,002)$ V vs. a saturated calomel electrode (SCE) at 25 °C, equivalent to $(0,2415 \pm 0,002)$ V (SHE).

The reference potential values for some other common reference electrodes and corresponding values for the control potential settings are shown in Table 1. Other electrodes can be used based on their known potential difference from SHE.

4.1.3.4 Electronic potentiostat, capable of a current output ≥ 100 mA, a voltage output ≥ 10 V, and a potential control accurate and stable to ± 1 mV.

The hardware/software used shall allow either recording the current for 24 h or integrating the current for 24 h.

4.1.4 Test set-up

4.1.4.1 Temperature of the reference electrode

Measure the temperature of the reference electrode (4.1.3.3). If necessary, adjust the temperature to $(25 \pm 1) ^\circ\text{C}$ by the control potential using the temperature coefficient for the given electrode type.

Temperature coefficients and examples of potential correction are shown in Table 1.

Table 1 — Potential settings for different reference electrodes and temperatures of the reference electrodes

Reference electrode type	Reference electrolyte	Temperature coefficient ^[1] V/K	Reference potential /V (SHE) when the temperature of the reference electrode is			Control potential setting /V when the temperature of the reference electrode is		
			18 °C	25 °C	37 °C	18 °C	25 °C	37 °C
			Saturated calomel (SCE)	Saturated KCl	$-7,50 \times 10^{-4}$	0,2468	0,2415	0,2325
1,0 M calomel	1 mol/l KCl	$-2,40 \times 10^{-4}$	0,2817	0,2800	0,2771	-0,040	-0,039	-0,036
0,1 calomel	0,1 mol/l KCl	$-7,00 \times 10^{-5}$	0,3342	0,3337	0,3329	-0,093	-0,092	-0,091
0,1 silver chloride	0,1 mol/l KCl	$-6,50 \times 10^{-4}$	0,2927	0,2881	0,2803	-0,051	-0,047	-0,039

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4.1.4.2 Positioning of the reference electrode

During the polarization part of the procedure, place the reference electrode probe close to the working electrode (amalgam) surface without touching the surface or shielding substantially the specimen surface. Also, the electrolyte of the reference electrode shall not contaminate the electrolyte in the vicinity of the amalgam. This is commonly achieved by placing the reference electrode in a separate compartment and using a "salt-bridge" between the reference electrode compartment and the main cell. The salt-bridge is a tube filled with the electrolyte and ending in a capillary ("Luggin capillary"), the end of which is placed close to the tested surface. The tip of the capillary should be at a distance from the amalgam surface equal to about two outer diameters of the tip.

4.1.5 Test procedure

Fill the corrosion cell (4.1.3.1) with electrolyte (4.1.2.1). Leave the cell open to the atmosphere. However, the cell should be covered with a lid to prevent excessive evaporation of the electrolyte.

Using the temperature control (4.1.3.2), heat the cell and maintain at a temperature of $(37,0 \pm 0,5) ^\circ\text{C}$.

Insert the specimen, connect the specimen and electrodes to the potentiostat (4.1.3.4) (no potential control) and wait $(10,0 \pm 0,1)$ min. During this potential stabilization it is advisable to stir the solution, e.g. using a magnetic stirrer and a stirring bar in the cell.

Record the potential at the end of the $(10,0 \pm 0,1)$ min exposure period. Stirring also may be used during this open-circuit potential measurement.

Set the potentiostat to the appropriate control potential (see Table 1) and time (24 h). Apply the potential and record or integrate current for $(24,0 \pm 0,2)$ h. During this polarization part of the test, the solution should remain stagnant (no stirring).

4.1.6 Data acquisition and processing

a) Coulometer method

A convenient method of data acquisition is to use an electronic current integrator (coulometer) in the circuit between the potentiostat and the cell. The reading of the coulometer after 24 h of polarization is the anodic charge.

b) Computer-controlled potentiostat method

An equally convenient procedure is to use a computer-controlled potentiostat with a program for potentiostatic control and software which allows post-test integration of the recorded current as displayed on the screen¹⁾.

c) Data-logging and integration method

If neither a) nor b) is available, record the polarization current using any available data acquisition system. The integration then can be performed by averaging all the current data and multiplying the average current, in amperes, by the total exposure time, in seconds (assuming that the time between current measurements was constant), or using any other suitable and accurate integration method.

In a) and b) make sure that the integration procedure uses the true values of current rather than the log of current. For instance, if the current is strip-chart-recorded on a logarithmic scale, or the results have been exported as a text file showing logarithms of the current, the data shall be converted to true values.

4.2 Immersion corrosion test for dental amalgam

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4.2.1 Apparatus

4.2.1.1 **Flask**, of borosilicate glass in accordance with ISO 3585, round bottom, 250 ml with 3 parallel necks and ground glass conical socket joints.

4.2.1.2 **Inlet tube**, of borosilicate glass in accordance with ISO 3585, internal diameter $(4,0 \pm 0,2)$ mm, length approximately 150 mm.

4.2.1.3 **Flowrate tube**, of borosilicate glass in accordance with ISO 3585, with glass float. Measurement range 0 ml/min to 10 ml/min.

4.2.1.4 **Peristaltic pump**, variable speed, to operate up to 20 r/min to provide an air flowrate of $(5,0 \pm 0,3)$ ml/min through the inlet tube.

4.2.1.5 **Gold foil mercury vapour dosimeter**²⁾ with a collection capacity of at least 1 µg mercury.

4.2.1.6 **Mercury vapour analyser**³⁾, compatible with the selected dosimeter, and having a lower limit for detection of 10 ng or less and measurement accuracy of 1 ng.

1) EG&G 252/352 software for the Model 273x, 283 potentiostats, Perkin Elmer Instruments / Princeton Applied Research, Oak Ridge, Tennessee, USA equipment and software combinations are examples of suitable products available commercially. This information is given for the convenience of users of this Technical Specification and does not constitute an endorsement by ISO of these products.

2) The Jerome gold foil mercury vapour dosimeter, Arizona Instruments Inc., Phoenix AZ, USA, is an example of a suitable product available commercially. This information is given for the convenience of users of this Technical Specification and does not constitute an endorsement by ISO of this product.

3) The Jerome mercury vapour analyser, Arizona Instruments Inc., Phoenix AZ, USA is an example of a suitable instrument available commercially. If this instrument is selected its dilution module is required. This information is given for the convenience of users of this Technical Specification and does not constitute an endorsement by ISO of this product.

4.2.1.7 Liebig condenser, of straight water-cooled borosilicate glass in accordance with ISO 3585, at least 20 cm in length with ground glass joints.

The lower cone end of the condenser shall fit one of the outer necks of the flask.

4.2.1.8 Cone/screw-thread adapters, three, of borosilicate glass, in accordance with ISO 3585, to fit outer and centre necks of the flask (4.2.1.1) and the socket end of the condenser (4.2.1.7).

4.2.1.9 Suspension rod, of borosilicate glass in accordance with ISO 3585, diameter $(4,0 \pm 0,2)$ mm, length approximately 150 mm.

4.2.1.10 O-ring, of neoprene, internal diameter $< 3,8$ mm, to fit suspension rod (4.2.1.9).

4.2.1.11 Sewing thread, nylon, single ply.

4.2.1.12 PVC tubing, plasticized, of internal diameters 3,2 mm to 6,4 mm, as required.⁴⁾

4.2.1.13 Reduction connectors, of borosilicate glass in accordance with ISO 3585, of number and size as required.

4.2.1.14 AAS or ICP-OES analytical instrumentation.

4.2.1.15 Waterbath, with temperature control to maintain $(37,0 \pm 0,5)$ °C.

4.2.1.16 Beaker, of borosilicate glass in accordance with ISO 3585, of capacity 250 ml.

4.2.2 Reagents

4.2.2.1 Lactic acid, analytical grade, purity approximately 90 %.

4.2.2.2 Water, in accordance with ISO 3696, grade 2.

4.2.2.3 Nitric acid, spectroscopic grade.

4.2.2.4 Ethanol, analytical grade.

4.2.3 Specimen preparation

Prepare four cylindrical specimens of the type used for compression strength measurement, in accordance with ISO 1559.

Condition the specimens in air for $(7,0 \pm 0,1)$ days at (37 ± 2) °C.

Measure the length and diameter of each cylinder to an accuracy of $\pm 0,02$ mm. If handling the specimens is unavoidable, this should be done wearing previously unused latex surgical gloves.

4.2.4 Lactic acid solution preparation

Twenty-four hours before the start of the 4-h immersion period, make up a 0,1 mol/l solution of lactic acid (4.2.2.1) using water (4.2.2.2). Measure the pH. If the pH value is outside the range $2,3 \pm 0,2$, discard and remake the solution.

4) Tygon® R-3603, Norton Performance Plastics, Akron, Ohio, USA is an example of suitable tubing available commercially. This information is given for the convenience of users of this Technical Specification and does not constitute an endorsement by ISO of this product.