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Dentistry — Corrosion tests for amalgam

Art dentaire — Essais de corrosion des amalgames

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

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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*

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

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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 ± 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(NaCl = 0,154 mol/l)] by adding (9,0 ± 0,1) g analytical grade NaCl to 600 ml water (ISO 3696, Grade 2), then make up this solution to (1000,0 ± 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 ± 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 ± 0,002) V vs. a saturated calomel electrode (SCE) at 25 °C, equivalent to (0,2415 ± 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.

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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 ± 1) °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	−0,005	0,000	0,009
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

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 ± 0,5) °C.

Insert the specimen, connect the specimen and electrodes to the potentiostat (4.1.3.4) (no potential control) and wait (10,0 ± 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 ± 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 ± 0,2) h. During this polarization part of the test, the solution should remain stagnant (no stirring).

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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 screen[1].

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

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 ± 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 ± 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.

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[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.

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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 ± 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 ± 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 ± 0,1) days at (37 ± 2) °C.

Measure the length and diameter of each cylinder to an accuracy of ± 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 ± 0,2, discard and remake the solution.

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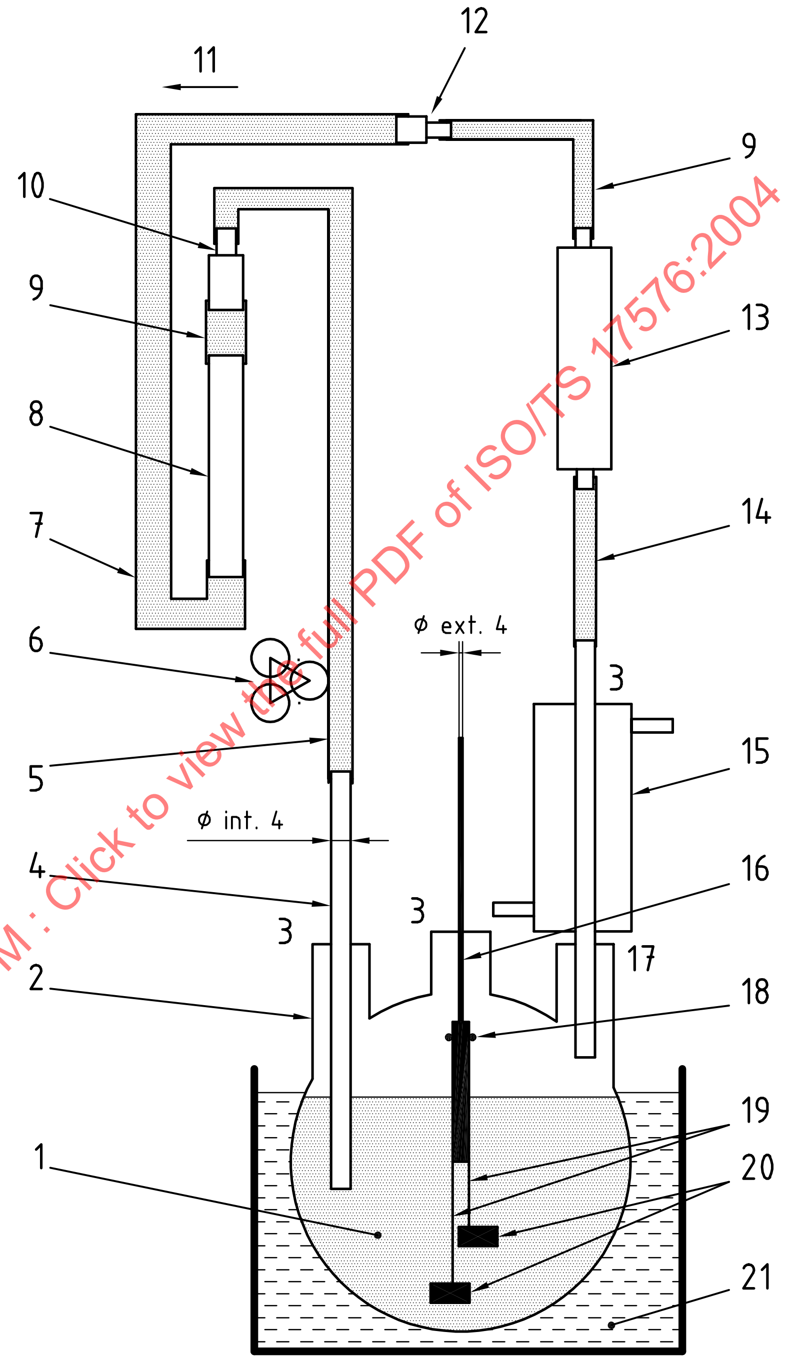
4) Tygon® R-3603, Norton Performance Plastics, Akron, Ohio, USA is an example of suitable tubing available commercially. This information is given for the convenience of users of this Technical Specification and does not constitute an endorsement by ISO of this product.

4.2.5 Corrosion test cell set-up

Using the apparatus specified in 4.2.1, assemble the corrosion test cell as shown Figure 1.

Dimensions in millimetres



Key

1	solution
2	round bottom flask. 250 ml, 3 parallel necks, ground glass joint
3	conical ground glass joint with a cone/screw-thread adapter to create and maintain the seal with the glass rod or tube
4	glass tube (inlet tube)
5	PVC tubing (1 000 ± 100) mm length
6	peristaltic pump
7	PVC tubing (250 ± 50) mm length
8	gas flow-tube
9	PVC tubing (150 ± 50) mm length
10	glass reduction connector
11	direction of gas flow
12	glass reduction connector
13	dosimeter
14	PVC tubing (500 ± 50) mm length
15	water-cooled Liebig condenser with ground glass joints
16	solid glass rod (suspension rod)
17	conical ground glass joint
18	neoprene O-ring to fit the glass rod
19	nylon threads
20	test specimens
21	waterbath at (37,0 ± 0,5) °C

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In this diagram the use and positioning of reduction connectors is intended to be schematic. Use connectors in appropriate numbers and sizes as required to attach tubing with appropriate diameters to the components of the apparatus.

Figure 1 — Example of corrosion test apparatus

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Before assembly wash all inner surfaces of the glassware with water (4.2.2.2), then with 20 % nitric acid (4.2.2.3). Rinse with water (4.2.2.2) and shake off residual moisture. Rinse with analytical grade ethanol. Allow to dry in air.

When assembling the apparatus, use new PVC tubing to connect the condenser to dosimeter, the dosimeter to the flow-tube and the flow-tube to the glass inlet tube. Use tubing with an internal diameter which will produce a tight fit when it is pushed over the ends of these components. It may be necessary to use a range of tube diameters and glass reduction connectors. At this stage, do not add the solution or the amalgam specimens.

The peristaltic pump recirculates the air during the test. The temperature of the water flowing through the condenser should be sufficiently low to prevent condensation of water (from vapour in the recirculated air) in the dosimeter.

The mercury vapour is collected on a gold foil dosimeter (4.2.1.5) and analysed by a gold foil mercury vapour analyser (4.2.1.6). Instrumentation other than the gold foil dosimeter/analyser may be used if it has similar or better accuracy and specificity, and permits the collection of mercury vapour from air as it is recirculated at a flowrate of 5 ml/min.

4.2.6 Test procedure

4.2.6.1 Pre-corrosion procedure

Suspend two cylinders by nylon thread (4.2.1.11) in a clean 250 ml glass beaker (4.2.1.16) containing (200,0 ± 0,1) ml of the lactic acid solution (4.2.4). Immerse the specimens and adjust the length of the nylon suspension threads such that the specimens do not touch each other, nor the wall nor the base of the container. Cover the beaker to prevent evaporation. Pre-corrode these specimens for (24,0 ± 0,2) h at (37,0 ± 0,5) °C. Discard this solution when the specimens are removed. (see 4.2.7.1)

NOTE This procedure is carried out because the rate of release of the mercury vapour decreases substantially during the first 24 h period, and the rate of release after this initial burst is considered to be more representative of long-term release.

4.2.6.2 Preparation of a reference solution

In order to measure the metal ion content of the solution, it is necessary to establish the background level due to impurities in the solution and contamination from the glassware.

At (18,0 ± 0,2) h after the start of the pre-corrosion procedure, add (200,0 ± 0,1) ml of lactic acid solution (4.2.4) to the flask (4.2.1.1). Set the glass inlet tube vertically and adjust so that the end is at a depth of (20 ± 2) mm. Place the glass rod (without specimens attached) in the central neck of the flask. Raise a waterbath [maintained at (37,0 ± 0,5) °C] around the flask until the surface of the lactic acid is at the same height as the surface of the water in the bath. (10 ± 2) min after this action, turn on the peristaltic pump and adjust the speed to give a flowrate of (5,0 ± 0,3) ml/min.

The bubbling of gas through the lactic acid solution can produce fluctuations in the flowrate. The average value should be calculated by reading the flow-tube at 10 s intervals over a period of 1 min.

After (5,0 ± 0,1) h, lower the waterbath. Disconnect the dosimeter and measure the mercury vapour it has collected by using the gold foil mercury vapour analyser (4.2.1.6). Using a recognized procedure with adequate sensitivity (i.e. $0,1 \times 10^{-6}$ or better), analyse the solution for the ions Ag, Sn, Cu, Zn, Hg. (For this, ICP or AAS are acceptable procedures).

Pour out and discard any acid solution remaining in the flask.

NOTE It is convenient to analyse the reference solutions (obtained in 4.2.6.2 and 4.2.7.2) and the specimen solutions (obtained by 4.2.7.1 and 4.2.7.2) at the same time. Therefore, the reference solutions may be stored in clean, covered borosilicate glass containers while the specimen solutions are being produced.

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