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Dentistry — Corrosion test methods for dental amalgam

Médecine bucco-dentaire — Essais de corrosion des amalgames dentaires

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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.

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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 1, *Filling and restorative materials*.

This second edition cancels and replaces the first edition (ISO/TS 17988:2014), which has been technically revised. The main changes to the previous edition are as follows:

— The scope has been extended to include products that are within the scope of ISO 20749.

— Clause 3 includes additional terms and definitions.

— Clause 4: quantities required for the production of test-pieces for each of the three test procedures are given now as the mass of dental amalgam alloy per test-piece, in place of the total mass of dental amalgam alloy for the complete test (i.e. the estimated quantity for all test-pieces including permitted replacements).

— 5.2.2.2 and 5.3.2: the parameter R_a has replaced R_k to specify surface roughness on steel moulds.

— 5.3.2: the surface roughness of the tapered hole in the Hertzian-indentation strength-reduction test-piece mould has been revised.

— 8.3.1.1 and 8.3.1.2: two additional items have been added to the list of apparatus.

— 8.3.1.4: blood dilution vials without protuberances on the interior base surface might not be available. A means by which the required flat surface can be created has been added.

— 8.3.4 and 8.4.2.2: a technical addition has been made to the procedure. Instructions are given for replacing test-pieces from which invalid results had been produced. Also, advice is given to make the maximum number of permitted replacements at the time that the actual test-pieces are made (to avoid a possible 31-day delay should a result be invalid and a replacement test-piece be required).

— 8.4.2.2: instructions are given to inspect the substrate disc and to replace it if damage is observed.

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 gives the practical details of three test methods for the measurement of the resistance to corrosion of dental amalgam. These corrosion test methods are laboratory procedures for evaluating the relative performances of dental amalgam alloy products. They are designed to produce a measurable effect (and differences between products) within a relatively short time period, a time period appropriate for a comparative laboratory evaluation.

The results of these tests should not be used for any biocompatibility claims, for which their use is inappropriate.

Should other corrosion test procedures emerge in time as suitable for application in comparative evaluations of dental amalgam products, they will be included in future editions of this document.

Dentistry — Corrosion test methods for dental amalgam

1 Scope

This document gives the details of test procedures for evaluating the corrosion resistance of dental amalgam formed from products that are within the scopes of ISO 24234 and ISO 20749.

This document is not applicable to other metallic materials in which an alloy powder reacts with a liquid alloy to produce a solid metallic material intended for dental restoration.

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 286-2, *Geometrical product specifications (GPS) — ISO code system for tolerances on linear sizes — Part 2: Tables of standard tolerance classes and limit deviations for holes and shafts*

ISO 1942, *Dentistry — Vocabulary*

ISO 3585, *Borosilicate glass 3.3 — Properties*

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

ISO 4287, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

ISO 6344-1, *Coated abrasives — Grain size analysis — Part 1: Grain size distribution test*

ISO 7488, *Dentistry — Mixing machines for dental amalgam*

ISO 13897, *Dentistry — Dental amalgam reusable mixing-capsules*

ISO 24234, *Dentistry — 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
dental amalgam alloy
alloy in fine particles, composed mainly of silver, tin and copper, which when mixed with dental mercury produces a dental amalgam for dental restoration

[SOURCE: ISO 20749:2017, 3.1]

3.2
dental mercury
mercury supplied for use in the preparation of dental amalgam

[SOURCE: ISO 20749:2017, 3.2]

3.3
pre-capsulated product
product supplied in a sealed capsule that contains measured amounts of dental amalgam alloy powder and dental mercury with masses that are appropriate for the production of a mass of dental amalgam that is considered to be suitable for a single small or medium size restoration in a single tooth

Note 1 to entry: The dental amalgam alloy powder and dental mercury are separated by a barrier that is broken immediately prior to mixing, allowing their contact. The capsule remains sealed until mixing has been completed.

[SOURCE: ISO 20749:2017, 3.3]

3.4
dental amalgam alloy tablet
quantity of dental amalgam alloy powder that has been compressed to form a single entity for the purpose of providing a pre-dosed quantity of the alloy that, when mixed with an appropriate mass of dental mercury, produces a mass of dental amalgam that is considered to be suitable for a single small or medium size restoration in a single tooth

Note 1 to entry: During mixing the tablet is intended to break apart, forming a fine powder.

[SOURCE: ISO/TS 20746:2016, 3.4]

3.5
dental mercury sachet
measured quantity of dental mercury supplied in a sachet (for use in a reusable mixing capsule) in a mass that, when mixed with an appropriate mass of dental amalgam alloy powder, produces a mass of dental amalgam that is considered to be suitable for a single small or medium size restoration in a single tooth

Note 1 to entry: The sachet is intended to rupture during mixing to allow the dental mercury to come into contact with the dental amalgam alloy powder.

Note 2 to entry: The dental mercury sachet is also known as a dental mercury pillow.

[SOURCE: ISO/TS 20746:2016, 3.5, modified — Note 2 to entry added.]

3.6
immersion corrosion test
test in which a test-piece of known surface area is immersed in a specified solution (at a specified temperature) for a defined period of time to determine quantitatively the elemental release into the solution and thereby allow a comparison of the corrosion resistance between this and other products of a similar type

3.7
potentiostatic corrosion test
test in which a test-piece of known surface area is immersed in a specified electrolyte (at a specified temperature) with a set potential applied for a defined period of time during which the corrosion current is recorded, integrated and then normalized by the anodic surface area and time to produce the total charge transported per unit of area in a unit of time [units $C/(cm^2.d)$]

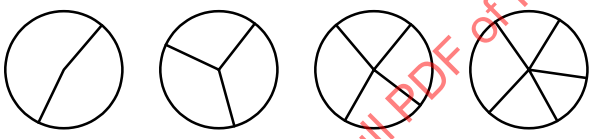
3.8
Hertzian-loading strength-reduction test
test in which a test-piece is immersed for a defined period of time in a specified solution (at a specified temperature) in a way that creates crevice corrosion conditions on one surface, after which it is removed from the solution and fractured with the force to do this then compared with the force to fracture an identical test-piece subjected to ageing in air at the same temperature

Note 1 to entry: Fracture is initiated from the surface subjected to crevice corrosion conditions and proceeds by radial crack growth.

3.9
radial cracking
fracture pattern of a Hertzian-loaded test-piece in which (more or less) planar cracks form along radii, normal to the face of the disc shaped test-piece, thus dissecting it into two or more sectors

Note 1 to entry: Such radial cracks initiate on the test surface of the test-piece and propagate through the disc to produce approximately equiangular dissection in most cases.

EXAMPLE Some radial fracture patterns in disc shaped test-pieces are illustrated here.



[SOURCE: ISO/TS 20746:2016, 3.8]

3.10
top surface
surface of the disc shaped test-piece that has been produced by carving back unset amalgam that is above the level of the mould until the surface of the test-piece is flat and level with that mould surface

[SOURCE: ISO/TS 20746:2016, 3.6]

3.11
test surface
surface of the disc shaped test-piece that has been produced by contact with the polished glass plate when the mixed amalgam is packed into the mould

[SOURCE: ISO/TS 20746:2016, 3.7]

3.12
mixing machine for dental amalgam
DEPRECATED: amalgamator
electrically powered mixing machine that operates using an oscillating action for mixing dental amalgam alloy and dental mercury (in a capsule) to produce a dental amalgam

4 Sampling

Products shall be procured in packages that have been produced for retail.

For pre-capsulated dental amalgam products, procure a sufficient number of capsules from a single lot.

For dental amalgam alloy in the form of a powder supplied in bulk or as dental amalgam alloy tablets, procure sufficient dental amalgam alloy and a sufficient number of dental mercury sachets from single lots. The dental mercury sachets shall conform to ISO 24234.

NOTE In this context, “sufficient” is deemed to be the quantity to make the required number of test-pieces and the maximum number of test-pieces allowed to replace any that are rejected.

For the immersion corrosion procedure (see Clause 6), at least 1,5 g of dental amalgam alloy is required per test-piece.

For the potentiostatic corrosion procedure (see Clause 7), at least 1,5 g of dental amalgam alloy is required per test-piece.

For the Hertzian-loading strength-reduction procedure (see Clause 8), at least 3,5 g of dental amalgam alloy is required per test-piece.

5 Preparation of dental amalgam test-pieces

5.1 General

5.1.1 Temperature

Prepare test-pieces at (23 ± 2) °C.

5.1.2 Mixing

For a dental amalgam alloy product supplied either as tablets or as a free-flowing powder in bulk, the ratio by mass of the dental amalgam alloy to the mass of dental mercury should be that recommended by the manufacturer. Use a reusable mixing-capsule (with a pestle, if needed) that conforms to ISO 13897. Use any other mixing accessory that is required, as recommended by the manufacturer. If more than one mix is required to make the test-piece, produce these mixes simultaneously using equipment of the same type for each mix. However, if the last mix can be produced within the working time of the first mix, mixing these masses sequentially on a single piece of equipment is permitted.

For pre-capsulated products, use as many capsules as are needed. Mix the contents of the capsules either simultaneously using the same number of mixing machines for dental amalgam of the same type, or sequentially on a single mixing machine for dental amalgam. (The latter is permitted, provided the mixing of the last capsule is completed before the end of the working time of the first.) If necessary, use only a portion of the dental amalgam mix from one of these capsules.

Use a mixing machine for dental amalgam that conforms to ISO 7488 and that is recommended for mixing the dental amalgam alloy product with dental mercury or mixing the pre-capsulated product. Use the mixing machine settings and mixing time that are recommended by the manufacturer of the dental amalgam alloy or pre-capsulated product (for the mass of dental amalgam alloy that is being mixed).

5.2 Cylindrical test-pieces for use in the immersion and potentiostatic corrosion test procedures

5.2.1 Mass of dental amalgam to be mixed

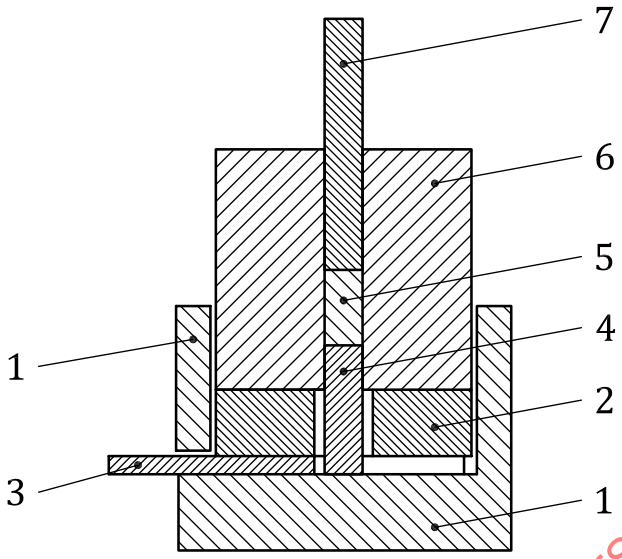
Mix a mass of the dental amalgam at least sufficient to make a cylindrical test-piece (8 ± 1) mm in length after packing into the die shown in Figure 1.

NOTE The mass of a dental amalgam cylinder that is 4 mm in diameter and 8 mm in length is approximately 1,2 g.

5.2.2 Apparatus for the preparation of dental amalgam cylindrical test-pieces

5.2.2.1 General

Use the apparatus as shown in Figures 1 to 4.



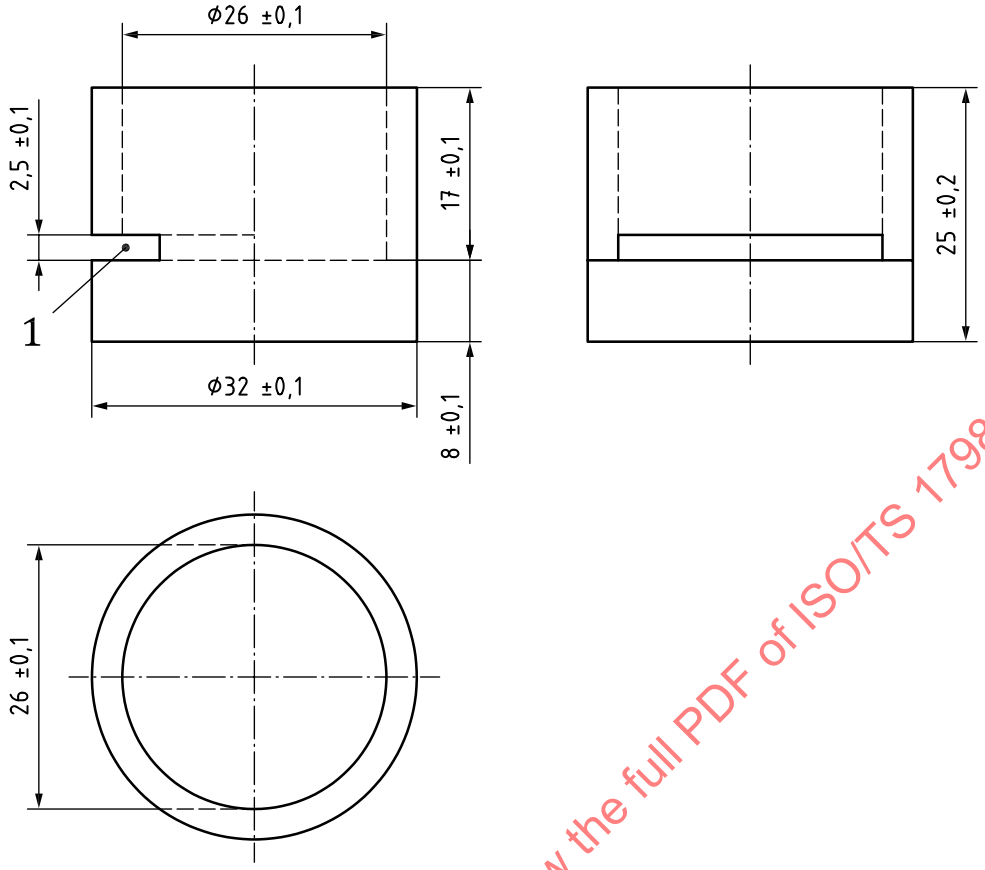
Key

1 holder
2 spacer no. 1
3 spacer no. 2
4 plunger no. 2
5 test-piece
6 die
7 plunger no. 1

NOTE The dimensions for each of the components are given in the figures that follow.

Figure 1 — Vertical section through the apparatus for making dental amalgam cylindrical test-pieces, showing the assembled apparatus with a test-piece in place

Dimensions in millimetres



Key

1 slot

Figure 2 — The holder

Dimensions in millimetres

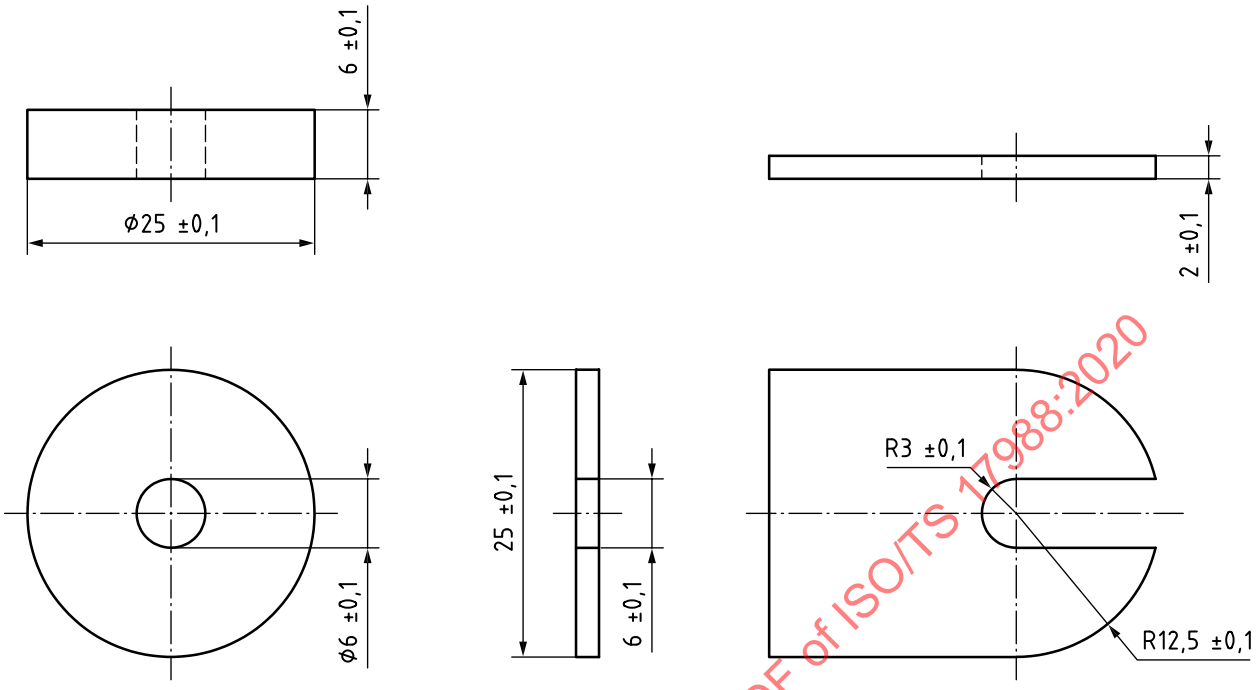


Figure 3 — Spacer no. 1 (left) and spacer no. 2 (right)

Dimensions in millimetres

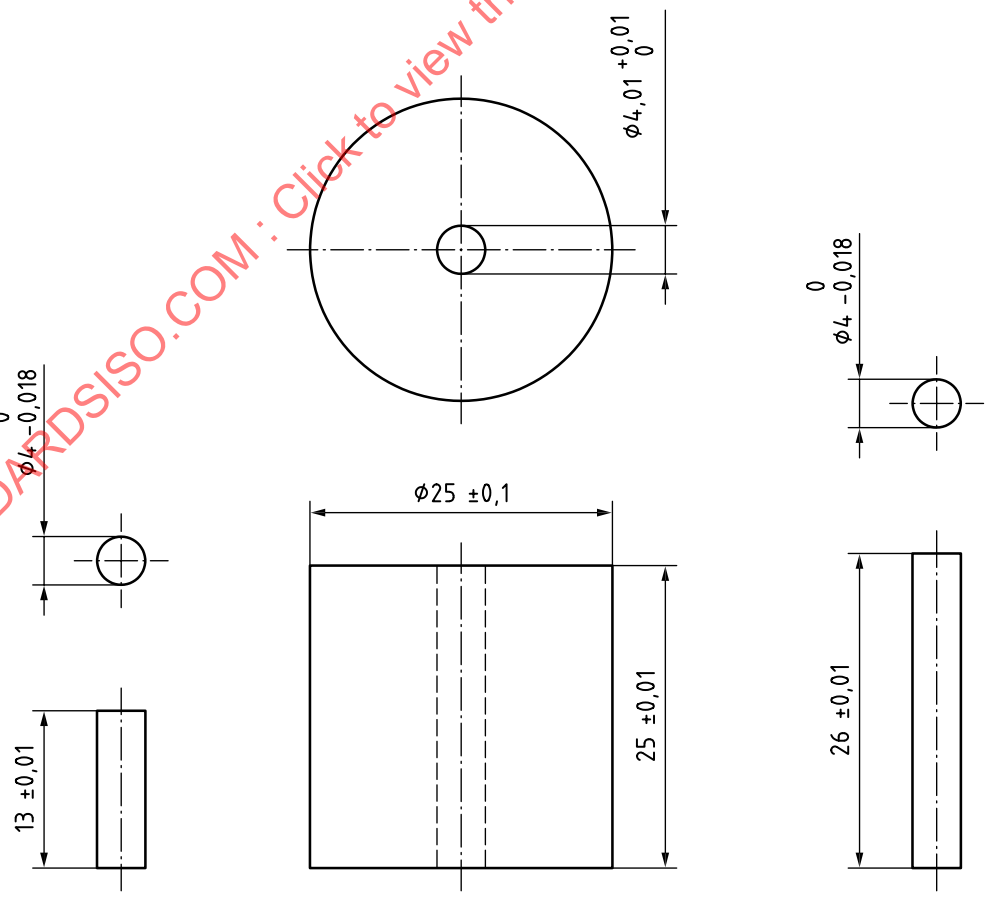


Figure 4 — Plunger no. 2 (left), the die (centre) and plunger no. 1 (right)

To assist the operator in judging whether the correct quantity of dental amalgam has been inserted into the die, for the test-piece to be within the permitted range for length [i.e. (8 ± 1) mm], circumferential datum lines may be scribed at 11 mm and 13 mm from one end of plunger no. 1. This end shall be in contact with the dental amalgam. Though such datum lines are not mandatory, their use is recommended.

The diameters of the plungers are subject to a shaft (or in this case a plunger) clearance (with a tolerance) of h7 according to ISO 286-2. For a plunger that is nominally 4,000 mm in diameter, its diameter shall be between 0 µm and 18 µm less than 4,000 mm. Thus, the diameter of the plunger shall be between 3,982 mm and 4,000 mm.

The diameter of the hole in the die is subject to a clearance (with a tolerance) of F7 according to ISO 286-2. For a hole that is nominally 4,000 mm in diameter, its diameter shall be between 10 µm and 20 µm more than 4,000 mm. Thus, the diameter of the hole shall be between 4,010 mm and 4,020 mm.

5.2.2.2 Materials and tolerances for construction of the apparatus to make test-pieces

Make the holder and the spacers of cold-rolled or stainless steel. Make the die and the plungers of hardened tool steel or hardened stainless steel. Hone the working surfaces of the die and the plungers to a roughness, R_a, not greater than 6,3 µm when tested in accordance with ISO 4287. Set the limits of clearance between the die and the plungers at F7 and h7, respectively, in accordance with ISO 286-2.

5.2.2.3 Assembly of the apparatus

Assemble the holder, spacers no. 1 and no. 2, the die and plunger no. 2 as shown in Figure 1.

5.2.3 Packing

Place the coherent mass of mixed dental amalgam on top of the die cavity and insert immediately with several thrusts of a hand instrument for dental amalgam packing that is slightly less than 4 mm in diameter. Do not express dental mercury during this process. Then insert plunger no. 1 into the die cavity and proceed, following the schedule given in Table 1.

If plunger no. 1 has circumferential datum lines scribed on its cylindrical surface (at 11 mm and 13 mm from the end of the plunger that is in contact with the dental amalgam), the test-piece will be within the permitted (8 ± 1) mm range for length if the 13 mm datum line can be seen and the 11 mm datum line cannot.

After ejection from the mould, the test-piece should not be trimmed.

Inspect the surfaces of the test-piece for any defects. Use visual inspection without magnification. Carry out this inspection at an illuminance of at least 1 000 lux and at a distance not exceeding 250 mm. A person making the inspection shall have nominally normal visual acuity. [Corrective (non-magnifying) non-tinted lenses may be worn.] If the test-piece is defective, replace it.

Table 1 — Schedule for the production of a dental amalgam cylindrical test-piece

Procedure	Time s
End of mixing	0
Insert the mixed mass into the die cavity, then plunger no. 1 and apply a force of (176 ± 13) N to produce a pressure of (14 ± 1) MPa	30
Release the force and remove spacer no. 2	45
Reapply the force	50
Re-release the force	90
Carefully remove excess dental mercury and eject the test-piece	120

5.3 Disc-shaped test-pieces for use in the Hertzian-loading strength-reduction test

5.3.1 Apparatus for the preparation of dental amalgam disc-shaped test-pieces

5.3.1.1 Mould as shown in Figure 5.

5.3.1.2 Flat glass plate, with a polished scratch-free surface and square with an edge length greater than 30 mm.

5.3.1.3 Microscope slide, glass, to provide a straight edge to carve back the dental amalgam.

5.3.1.4 Hand instrument for dental amalgam packing.

5.3.2 Materials and tolerances for construction of the mould

The mould shall be made of hardened tool steel or hardened stainless steel. The upper and lower surfaces shall be flat and parallel, and have an arithmetic mean roughness value, R_a, not greater than 6,3 µm when tested in accordance with ISO 4287. The hole shall have a taper of (7 ± 2) ° to allow the amalgam disc to be ejected without undue force when this is applied to the face that has the smaller diameter. The tapered surface shall be smooth enough not to impede the ejection of the test-piece. For example, it may be honed to an arithmetic mean roughness value, R_a, of 6,3 µm (when tested in accordance with ISO 4287).

NOTE 1 For convenience, to distinguish between the two surfaces during test-piece production, a small engraved mark (set away from the hole) can be made on one of the mould faces.

NOTE 2 The angle of the taper, (7 ± 2) °, is the included angle. The wall of the mould is at an angle of (3,5 ± 1,0) ° with the centre line.

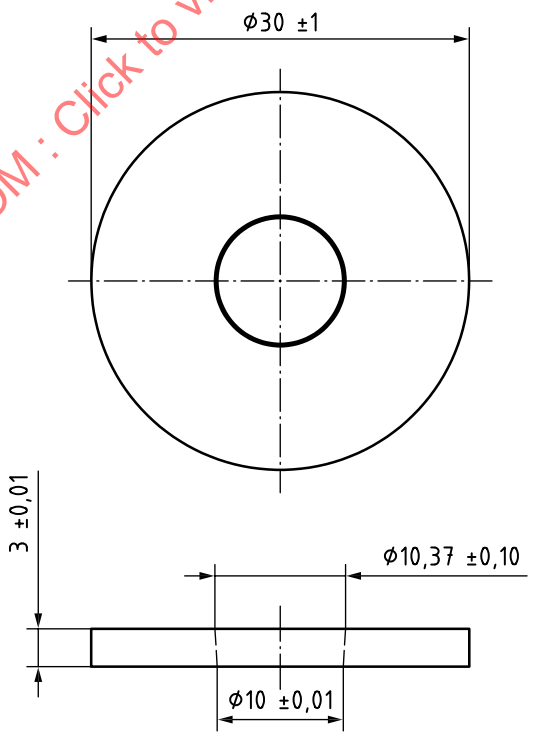


Figure 5 — The mould to produce disc-shaped test-pieces for the Hertzian-loading strength-reduction test

5.3.3 Packing the mould, removal of test-piece and inspection for surface defects

Place the steel mould on the glass plate with the side that has the greater diameter for the tapered hole in contact with the plate.

NOTE 1 The surface of the glass plate acts as a matrix for the test surface of the test-piece.

Mix a mass of the dental amalgam sufficient to make a disc-shaped test-piece that is 10 mm in diameter and 3 mm high after packing into the die shown in Figure 5.

NOTE 2 The mass of a 10 mm diameter dental amalgam disc 3 mm in height is approximately 3,0 g.

Pack the dental amalgam by hand, overfilling slightly. Carve back using the edge of the microscope slide to produce a flat surface (on the dental amalgam) that is level with that of the mould.

Allow the dental amalgam to set for 10 minutes. Carefully eject the test-piece from the mould by applying light finger-pressure to the surface of the test-piece that had been carved back (the "top" surface), while holding the mould in the other hand. Check visually that the test surface is defect-free everywhere, other than possibly at the margin. Use visual inspection without magnification. Carry out this inspection at an illuminance of at least 1 000 lux and at a distance not exceeding 250 mm. A person making the inspection shall have nominally normal visual acuity. [Corrective (non-magnifying) non-tinted lenses may be worn.] If a defect is detected, reject that test-piece and make a replacement.

To prevent any damage to the test surface during ejection, placing a thick soft pad, such as a number of dental napkins, under the mould to "catch" the ejected test-piece is recommended.

After ejection do not grind or polish the surfaces of the test-piece.

6 Determination of the resistance to corrosion by the immersion procedure

6.1 Principle

Static immersion corrosion tests, of which this is one, are used extensively in the metals industry to assess the corrosion resistance of different alloys to a specific potentially corrosive environment. In its simplest form, the weight loss from a test-piece (immersed for a specified time at a defined temperature) is measured. The composition of the test solution is relevant to the intended application but modified to yield a measurable effect in a period of time very much less than the expected service life. (It is an accelerated test.) It is reliable as a screening test for uniform surface corrosion resistance under conditions that are static. A static immersion corrosion test does not measure the effect of pitting corrosion, crevice corrosion or other local corrosion processes.

The test procedure given here was developed specifically for dental amalgam from the static immersion test used for dental metallic materials (specified in ISO 10271) in which the release of metal ions into the electrolyte is measured quantitatively by using spectroscopic analysis. (This replaces measurement of weight loss.) For dental amalgam, the apparatus is also modified to collect any mercury vapour that might be released. Because dental amalgam has a lower corrosion resistance than, for example, dental gold alloys, the electrolyte composition is less aggressive.

The data collected is processed to give the release of metallic elements in micrograms per unit of exposed surface area of the test-pieces and in nanograms of mercury vapour released per unit of exposed surface area.

6.2 Reagents for the test solution and cleaning the apparatus

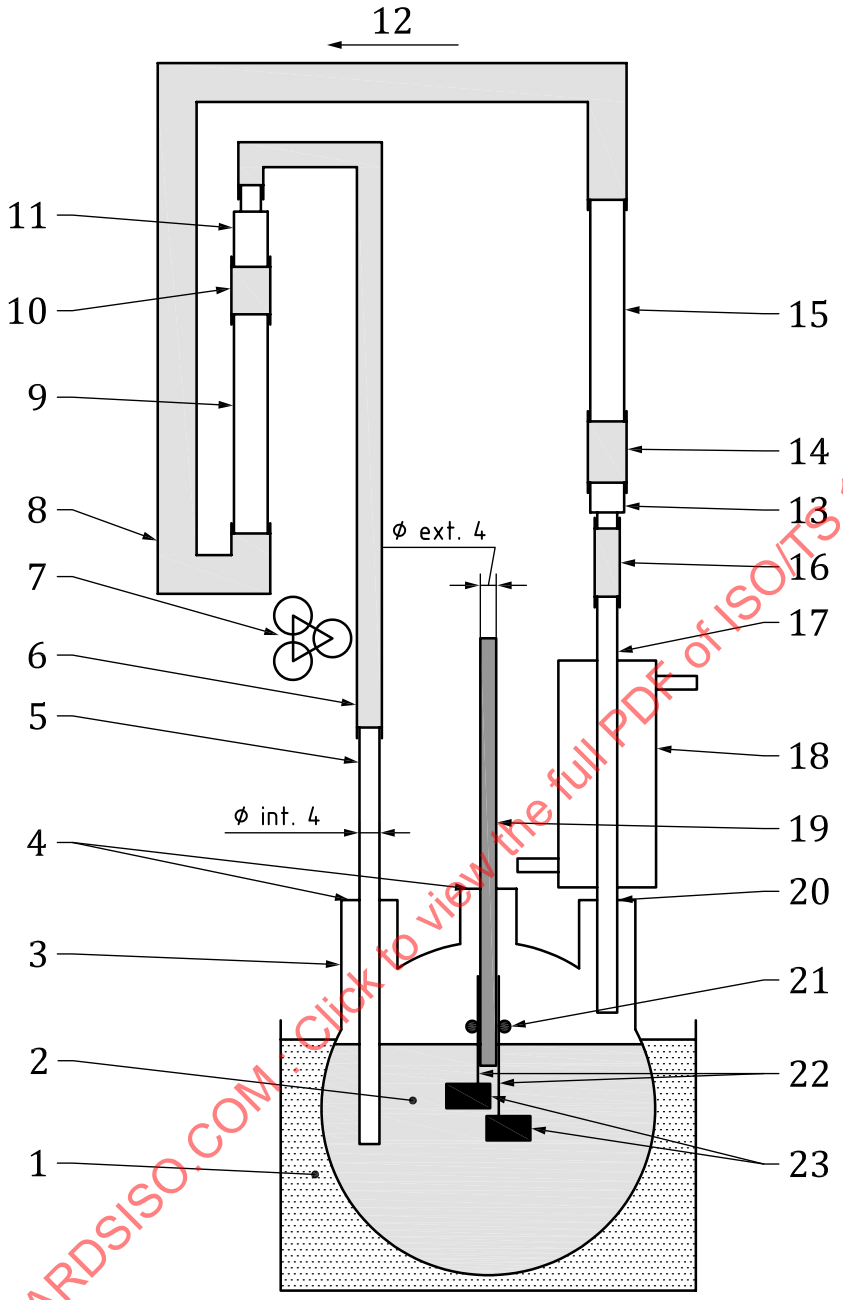
6.2.1 Lactic acid solution ≥ 85 % grade.

6.2.2 Water, to Grade 2 as specified in ISO 3696.

6.2.3 **Nitric acid**, spectroscopic grade.

6.2.4 **Ethanol**, analytical grade.

Dimensions in millimetres



Key

1 water-bath at (37,0 ± 0,5) °C
2 0,1 mol/l lactic acid solution
3 round-bottomed flask, 250 ml capacity, three parallel necks with ground-glass joints
4 conical ground-glass joint with a cone screw-thread adapter to create and maintain the seal with the glass rod or tube
5 glass tube (air inlet tube)
6 PVC tubing (1 000 ± 100) mm in length
7 peristaltic pump
8 PVC tubing (500 ± 50) mm in length
9 variable area air-flow meter
10 PVC tubing (150 ± 50) mm in length
11 glass reduction connector

12 direction of gas flow
13 glass reduction connector
14 PVC tubing (150 ± 50) mm in length
15 gold-impregnated silica tube mercury vapour trap
16 PVC tubing (150 ± 50) mm in length
17 conical ground-glass joint with a cone adapter to create and maintain the seal with the glass tube
18 water-cooled Liebig condenser with ground-glass joints
19 solid glass rod (suspension rod)
20 conical ground-glass joint
21 polychloroprene[a] O-ring to fit the glass rod
22 nylon threads
23 test-pieces

[a] Neoprene® is the trade name of a product supplied by Dupont Performance Elastomers L.L.C., Dupont de Nemours Inc., Wilmington DE, USA. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

NOTE The peristaltic pump recirculates the air during the test.

Figure 6 — Immersion corrosion test apparatus

In Figure 6 the use and positioning of reduction connectors is intended to be schematic. Use such connectors in such numbers and with sizes as required to attach tubing with appropriate diameters to the components of the apparatus.

6.3 Apparatus

6.3.1 Flask, of borosilicate glass (in accordance with ISO 3585), having a round bottom, 250 ml capacity, with three parallel necks and ground-glass conical socket joints.

6.3.2 Inlet tube, of borosilicate glass (in accordance with ISO 3585), with an internal diameter of (4,0 ± 0,2) mm and approximate length of 150 mm.

6.3.3 Variable area air-flow meter, of borosilicate glass (in accordance with ISO 3585), with a glass float and a measurement range of 0 ml/min to 10 ml/min.

Other flow measuring instrumentation may be used if it can measure air-flow within the same range.

6.3.4 Peristaltic pump (variable speed), to operate at up to 20 r/min, to provide an air-flow rate of (5,0 ± 0,3) ml/min through the inlet tube (6.3.2).

6.3.5 Atomic fluorescence mercury vapour analyser that is compatible with the selected mercury vapour trap (6.3.6)[1)].

6.3.6 Mercury vapour trap (four in number), a commercially manufactured gold-impregnated silica tubular trap[2)].

1) PSA 10.525 Sir Galahad mercury vapour detector by PS Analytical, Orpington, Kent, UK is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

2) Quartz Amasil 30 mg filled by PS Analytical, Orpington, Kent, UK is an example of a suitable mercury vapour trap available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

6.3.7 Liebig condenser, straight, water-cooled, of borosilicate glass (in accordance with ISO 3585), and at least 20 cm in length, with ground-glass joints. (The lower cone end shall fit one of the outer necks of the flask, 6.3.1).

6.3.8 Cone screw-thread adapter (three in number), of borosilicate glass (in accordance with ISO 3585) to fit the outer and centre necks of the flask (6.3.1) and the socket end of the condenser (6.3.7).

6.3.9 Solid suspension rod, of borosilicate glass (in accordance with ISO 3585) with a diameter of (4,0 ± 0,2) mm and approximately 150 mm length.

6.3.10 "O" ring, of polychloroprene[3)], with internal diameter < 3,8 mm, to fit the suspension rod (6.3.9).

6.3.11 Thread, nylon, single-ply, sewing.

6.3.12 PVC tubing, clear plasticized, with internal diameters (3,2 mm to 7,8 mm) and lengths, both as required.

6.3.13 Reduction connector, of borosilicate glass (in accordance with ISO 3585). Number and size of these as required.

6.3.14 Analytical facility, AAS, ICP-OES or ICP-MS.

6.3.15 Water bath, with a temperature control to maintain (37,0 ± 0,5) °C.

6.3.16 Beakers (six in number), of borosilicate glass (in accordance with ISO 3585), 250 ml capacity.

6.3.17 Polyethylene gloves.

6.3.18 Volumetric flask with stopper, of borosilicate glass (in accordance with ISO 3585), 1 l capacity.

6.3.19 Beaker of borosilicate glass (in accordance with ISO 3585), 500 ml capacity.

6.4 Mercury vapour analyser requirements

Use a tubular gold-impregnated silica mercury vapour trap with a collection capacity of at least 50 mg of mercury. To measure the mercury vapour it has collected, use a compatible atomic fluorescence mercury vapour analyser with a lower detection limit no greater than 1 ng and accuracy no worse than ± 1 ng. Before the trap is used all mercury from a previous recording must be discharged in accordance with the manufacturer's instructions.

NOTE For the convenience of the user of this document, instrumentation manufactured by PS Analytical is given as suitable. It is possible to collect mercury vapour on such a mercury vapour trap operating *remote and offline*, after which the trap is sent to an analytical laboratory company that possesses an appropriate atomic fluorescence spectrometer and offers a mercury vapour analytical service, to determine the amount collected. If corrosion testing of amalgam is infrequent, this could offer an economic alternative to the purchase of the spectrometer. If this option is taken, a minimum of four such mercury vapour traps are required. This information is for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

3) Neoprene® is the trade name of a product supplied by Dupont Performance Elastomers L.L.C., Dupont de Nemours Inc., Wilmington DE, USA. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

6.5 Cleaning the glassware

For cleaning the glassware, make up a solution of nitric acid with a concentration of 3 mol/l [using the spectroscopic grade nitric acid and water (to Grade 2, as specified in ISO 3696)] in the 500 ml beaker.

Rinse all the items of glassware with water (to Grade 2, as specified in ISO 3696), then acid-wash them with the 3 mol/l nitric acid solution. The surfaces that are to be acid-washed are those that are within the corrosion apparatus. Follow this by rinsing with water (to Grade 2, as specified in ISO 3696) and shake off any residual water droplets. Then rinse with analytical grade ethanol. Dry by evaporation in air.

If acid washing the variable area air-flow meter dissolves painted-on graduation marks, exclude this component from acid washing.

WARNING — Care should be exercised when cleaning glassware to ensure that rinsing with water takes place between washing in acid and rinsing using ethanol. Ethanol–nitric acid mixtures can be highly unstable and explosive. Keep the two chemicals well separated during the cleaning process.

6.6 Assembly of the immersion corrosion test apparatus

The components of the apparatus required are specified in 6.3. Clean the glassware following the procedure given in 6.5. Assemble the apparatus in accordance with Figure 6. However, Figure 6 shows the immersion corrosion test apparatus with the two test-pieces in place and the solution present. At this (assembly) stage do not add the solution or the amalgam test-pieces to the apparatus.

Use new PVC tubing to connect the condenser to the mercury vapour trap, the mercury vapour trap to the variable area air-flow meter and the variable area air-flow meter to the glass inlet tube. Use tubing with an internal diameter that 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.)

The temperature of the cooling water flowing through the Liebig condenser shall be sufficiently low to prevent condensation of water (from vapour in the recirculated air) in the mercury vapour trap.

6.7 Test-piece production

Measurement of the resistance to corrosion is made by two determinations. Produce two cylindrical test-pieces 4 mm in diameter and (8 ± 1) mm in length, according to 5.1 and 5.2, for the first determination. The actual diameter of these test-pieces is greater than 4,00 mm as a consequence of the F7 clearance for the hole in the die that is used and any setting expansion that takes place after the removal of the test-piece from the die.

NOTE 1 The 4 mm diameter cylindrical hole in the die has a nominal diameter of 4,000 mm which is given a clearance (with a tolerance) for a shaft (i.e. plunger) of F7, in accordance with ISO 286-2. As a consequence, the test-piece diameter will not be less than 4,010 mm.

At a minimum time of 10 h after the start of production of the first two test-pieces, start to produce two more of these test-pieces to be used for the second determination.

NOTE 2 The production of the reference and test solutions for the first determination (6.10.1) takes a minimum of 10 h to complete. Since the upper time limit for the storage of the second pair of test-pieces is 7 d 7 h and the second determination (with the production of its reference solution) cannot start until the test solution for the first determination is removed from the round-bottomed flask (which is then to be cleaned), a delay in the start of the production of the second pair of test-pieces is necessary.

Store all test-pieces in air at (37 ± 2) °C for (7,0 ± 0,3) days.

After storage, measure the length and the diameter of each test-piece to an accuracy of 0,02 mm. This shall be done at the ambient room temperature. If handling the test-piece is unavoidable, this shall be done wearing previously unused polyethylene gloves.

6.8 Preparation of the 0,1 mol/l lactic acid solution

Clean the volumetric flask following the procedure given in 6.5.

(6,7 ± 0,3) days after the first pair of dental amalgam test-pieces have been removed from the production mould, make up 1 l of lactic acid solution ($c_{\text{lactic acid}}$ = 0,1 mol/l) in the graduated flask, using the ≥ 85 % grade lactic acid and water (to Grade 2, as specified in ISO 3696). Measure the pH of the solution. If the pH value is outside the range 2,3 ± 0,2, discard and remake the solution. If the problem persists, obtain and use a new bottle of lactic acid.

6.9 Finishing the dental amalgam test-piece

Clean one of the 250 ml beakers following the procedure given in 6.5.

Add (200,0 ± 0,1) ml of the 0,1 mol/l lactic acid solution, as a test-piece finishing solution, to the cleaned beaker. Immediately after measuring the dimensions of the first pair test-pieces, suspend them by nylon threads in this aliquant of the acid solution. Adjust the length of the threads to immerse the test-pieces such that they do not touch each other, the wall of the beaker or its base. Cover the beaker to prevent evaporation. Hold the test-pieces in this finishing solution at (37,0 ± 0,5) °C for (24,0 ± 0,2) h.

Discard this finishing solution when the test-pieces are removed.

6.10 Test procedure

6.10.1 First determination

6.10.1.1 Production of the reference solution

It is necessary to produce a reference solution and measure the content for relevant elements to establish background values for these elements that result from impurities in the solution and contamination from the glassware. Because mercury vapour (potentially to be released during corrosion of amalgam) is relevant, it is necessary to establish a background value for it. This is the mercury collected in the mercury vapour trap during production of the reference solution.

(18,0 ± 0,2) h after the start of the test-piece finishing procedure (6.9) for the first determination, add (200,0 ± 0,1) ml of the 0,1 mol/l lactic acid solution to the round-bottomed flask of the immersion corrosion test apparatus.

Set the glass air inlet tube in its adaptor and place the latter in position in the appropriate neck of the flask. Adjust the tube vertically, to place the end at a depth of (20 ± 2) mm below the surface of the solution in the flask, then tighten the screw-thread adaptor.

Place the glass rod (without test-pieces attached) in its adaptor and place the latter in the centre neck of the flask.

Add the condenser to its neck. (All other connections were completed in 6.6 and now the corrosion apparatus has a closed environment.)

Position the flask in the water-bath [which is maintained at (37,0 ± 0,5) °C] such that the surface of the lactic acid is at the same height as the surface of the water in the bath. (10 ± 2) min later, turn on the peristaltic pump and adjust its rotational speed control to give an air-flow rate of (5,0 ± 0,3) ml/min. The bubbling of gas through the lactic acid solution may produce a slight fluctuation in the flow rate. The average value should be calculated by reading the flow rate on the variable area air-flow meter at 10 s intervals over a period of 1 min.

Clean a second 250 ml beaker following the procedure given in 6.5.

At (5,0 ± 0,1) h turn off the peristaltic pump. However, maintain the water-bath temperature at (37,0 ± 0,5) °C and keep the cooling water flowing through the condenser. Disconnect the round-

bottomed flask and remove it from the water-bath. Pour the lactic acid solution that it contains into the cleaned beaker. This sample of solution is the reference solution. Cover it until it is analysed.

Disconnect the mercury vapour trap. Place it in its transit container and cap the ends of the container. Retain for analysis.

It is convenient to analyse all of the samples (the reference solutions, the test solutions and the mercury vapour traps obtained by 6.10.1 and 6.10.2) consecutively. Therefore, the liquid samples are retained in separate, clean and covered borosilicate glass beakers until all four samples have been produced before the analysis is done. Also, the mercury vapour traps are retained until the fourth (and last) collection is made before their mercury contents are measured.

Pour out and discard any of the reference solution that remains in the flask. Return the round-bottomed flask to its position in the corrosion apparatus. Replace the used mercury vapour trap with a fresh (i.e. the second) mercury vapour trap. Reconnect all tubes.

6.10.1.2 Corrosion procedure to obtain the test solution

At the end of their (24,0 ± 0,2) h finishing period (6.9), remove the two dental amalgam test-pieces from the solution and rinse them with water (to Grade 2, as specified in ISO 3696). Do not touch the test-pieces with bare or gloved hands. Tie a length of nylon thread securely around each test-piece at mid-length. Add (200,0 ± 0,1) ml of the 0,1 mol/l lactic acid solution to the round bottomed flask. Remove the glass rod from the immersion corrosion test apparatus and attach the test-pieces to it using the nylon threads and polychloroprene[4)] O-ring. Return the glass rod to its position in the round-bottomed flask. Suspend the two test-pieces in the acid and adjust the lengths of the threads and vertical position of the rod in the adapter, such that the test-pieces are completely immersed and touch neither each other nor the surface of the flask. Recheck that the vertical position of the flask in the water-bath is such that the surface of the lactic acid is at the same height as the water in the bath. (10 ± 2) min later turn on the peristaltic pump and adjust the speed to give a flow rate of (5,0 ± 0,3) ml/min.

The position of the polychloroprene O-ring on the glass rod should allow the rod to be raised by sliding the rod through its screw-thread adapter (Figure 6, key reference 4) until the dental amalgam test-pieces are above the surface of the test solution.

NOTE Raising the test-pieces follows the immersion period.

Clean a third 250 ml beaker following the procedure given in 6.5.

At (4,0 ± 0,1) h after the start of the immersion, remove the test-pieces from the solution (without opening the flask to the atmosphere) by loosening slightly the screw-thread adapter and raising the glass rod. Retighten the screw-thread adapter when the test-pieces are above the solution. Continue to circulate the air for a further (1,0 ± 0,1) h to flush mercury vapour from the airspace above the solution.

At (5,0 ± 0,1) h disconnect the mercury vapour trap. Place it in its transit container and cap the ends of the container. Retain for analysis.

Disconnect the round-bottomed flask and remove it from the water-bath. Pour the test solution into the cleaned beaker and cover until analysed.

6.10.2 Second determination

Make up 1 l of a fresh solution of 0,1 mol/l lactic acid, following the directions given in 6.8.

Finish the two test-pieces that were produced for the second determination (in accordance with 6.7) following the finishing procedure given in 6.9. (Use the fourth 250 ml beaker.)

4) Neoprene® is the trade name of a product supplied by Dupont Performance Elastomers L.L.C., Dupont de Nemours Inc., Wilmington DE, USA. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

Disassemble the corrosion test apparatus. Clean the glassware components according to the procedure in 6.5 and reassemble according to 6.6.

Following the procedure that is given in 6.10.1.1, produce a (second) reference solution. During production of this reference solution collect any mercury vapour present on a fresh (i.e. the third) mercury vapour trap. This reference solution should be retained in the fifth 250 ml beaker (cleaned using the procedure given in 6.5) and covered until it is analysed.

Disconnect and remove the mercury vapour trap from the corrosion apparatus. Place the mercury vapour trap in its transit container and cap the ends of the container. This mercury vapour trap should be retained until the second corrosion test procedure has been completed before its mercury content is determined.

On completion of the finishing procedure, subject the second pair of test-pieces to the corrosion test procedure that is set out in 6.10.1.2. A fresh (the fourth) mercury vapour trap should be used. After the 5 h period during which air was bubbled through the solution, pour the second test solution into the sixth 250 ml beaker (cleaned using the procedure given in 6.5) and cover until it is analysed. Disconnect and remove the mercury vapour trap from the corrosion apparatus. Place the mercury vapour trap in its transit container and cap the ends of the container. Retain for analysis.

6.11 Analysis to determine the metal ion and mercury vapour release

Using a recognized analytical procedure (e.g. ICP) with adequate sensitivity (i.e. 10^{-7} mass fraction, or better), analyse both corrosion test solutions and their respective reference solutions for Ag, Sn, Cu, Zn and Hg. To this list add any other element that is declared by the product manufacturer to be present in the dental amalgam alloy in a mass fraction greater than 0,5 %.

Measure the amount of mercury vapour that has been collected on each of the four mercury vapour traps by using the atomic fluorescence mercury vapour analyser.

Subtract the background reading, i.e. the reference solution values, for the specified elements from the respective values obtained from analysis of the corrosion test solution to obtain the elemental release produced by corrosion. Do this for both determinations. Using the test solution volume, 200 ml, convert concentrations to masses.

Subtract the background value for mercury vapour from the corrosion test value for mercury vapour collected to obtain the mercury vapour released by corrosion.

For both determinations, calculate the mass for each element that has been released from the two dental amalgam test-pieces per square centimetre of their combined surface area.

Record both sets of results. Calculate and record the mean value for the mass of the release of each element (per square centimetre of test-piece surface area).

6.12 Test report

A test report shall be prepared. At least the following information shall be included:

a) name of the dental amalgam alloy product and its lot number;

b) a reference to this document (i.e. ISO/TS 17988:2020);

c) name and address of the manufacturer;

d) the mean values of the two determinations for the release of each of the elements Ag, Sn, Cu, Zn, Hg and any other element declared by the product manufacturer to be present in the dental amalgam alloy in a mass fraction greater than 0,5 %, in each case expressed per square centimetre of test-piece surface area, "µg/cm^2 in 4 h";

e) the mean values of the two determinations for the sum of the release of all elements released measured in "µg/cm^2 in 4 h";

f) the value for the release of mercury vapour in "ng/cm^2 in 4 h" – this shall be the mean value for the two determinations;

g) name and address of the organization responsible for the testing (e.g. test house, university, department of manufacturer);

h) any deviations from the procedure;

i) any unusual features observed;

j) date of testing.

7 Determination of the corrosion by the potentiostatic procedure

7.1 Principle

Because metallic corrosion is an electrochemical process in which there is a flow of electrons, the potential on a metallic material in a corrosion cell can be controlled electronically and the current measured. This is the basis of the potentiostatic experimental procedure. Here, it is used to characterize the corrosion properties of dental amalgam immersed in an electrolyte relevant to the oral condition. The "open circuit potential" (which gives the thermodynamic tendency of the dental amalgam to participate in electrochemical corrosion in its surrounding medium) is obtained. Imposing a potential and then continuously measuring the consequent current over a specified time yields the rate of charge transfer and gives the susceptibility to corrosion. Advocates of such testing point to it being a rapid procedure, in general.

7.2 Test-piece preparation

Produce one cylindrical dental amalgam test-piece 4 mm in diameter and (8 ± 1) mm in length, in accordance with 5.1 and 5.2. The actual diameter of this test-piece is greater than 4,00 mm as a consequence of the F7 clearance for the hole in the die that is used and any setting expansion that takes place after the removal of the test-piece from the die.

NOTE The 4 mm diameter cylindrical hole in the die has a nominal diameter of 4,000 mm which is given clearance (with a tolerance) for a shaft (i.e. plunger) of F7, in accordance with ISO 286-2. As a consequence, the test-piece diameter will not be less than 4,010 mm.

Store this test-piece in air at (37 ± 2) °C for (7,0 ± 0,3) days.

Measure the diameter of the test-piece to an accuracy of 0,05 mm. Calculate the cross-sectional area and express this in square centimetres.

Attach an insulated lead to the test-piece for connection to the potentiostat.

Cover the connecting lead junction and all surfaces except one end of the test-piece with an insulating material, preferably by casting in epoxy resin.

The temperature rise of the test-piece during setting of the resin should not exceed 15 °C. This embedding material should not dissolve in, nor react with, the electrolyte solution (7.7).

Using light pressure, wet-grind the exposed end of the test-piece uniformly on coated abrasive that conforms with micro-grit size P1200 (in accordance with ISO 6344-1) to produce a smooth surface across the dental amalgam and casting polymer. Wash with water (to Grade 2, as specified in ISO 3696). Examine the surface, to determine whether a crevice is present at the interface between the dental amalgam and the casting resin (having been created possibly by the casting process).

If a crevice is seen it can be eliminated by masking. If masking is considered essential and applied, determine the area of the amalgam that is left exposed, to an accuracy of 0,01 mm^2. This area should be expressed in square centimetres.

The testing laboratory may develop its own way of preparing the embedded test-piece, provided the above procedures are included and the conditions are met.

7.3 Corrosion test cell requirements

7.3.1 Corrosion cell

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

7.3.2 Temperature control

Use a jacket and temperature control with circulator, or a temperature-controlled bath, capable of maintaining (37,0 ± 0,5) °C in the cell.

7.3.3 Volume of the electrolyte

Use at least 300 ml.

7.4 Reference electrode probe requirements

7.4.1 Reference electrode and its control

Use any standard reference electrode with a stable potential of known potential difference from a standard hydrogen electrode (SHE). Adjust the control potential to (0,000 ± 0,002) V vs. saturated calomel electrode (SCE) at 25 °C, equivalent to (0,2415 ± 0,0020) V (SHE).

Other electrodes can be used, based on their known potential difference from SHE.

7.4.2 Temperature of the reference electrode

Measure the temperature of the reference electrode. If the temperature differs from 25 °C by more than 1 °C, adjust the control potential using the temperature coefficient for the given electrode type.

Temperature coefficients and examples of potential correction are shown in Table 2.

Table 2 — Potential settings for different reference electrodes and temperatures

Reference electrode type	Reference electrode filling solution	Temperature coefficient V/K	Reference potential by temperature of the reference electrode V (SHE)			Control potential setting by temperature of the reference electrode V		
			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 M 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 M silver chloride	0,1 mol/l KCl	$-6{,}50 \times 10^{-4}$	0,2927	0,2881	0,2803	−0,051	−0,047	−0,039

7.4.3 Positioning of the reference electrode

During the polarization part of the procedure, place the reference electrode probe close to the working electrode (dental amalgam) surface without touching that surface or shielding it substantially. Also, incorporate features in the apparatus to prevent the filling solution of the reference electrode contaminating the electrolyte in the vicinity of the dental amalgam.

If a salt bridge is used to prevent contamination, place the end of the salt bridge capillary a distance from the dental amalgam surface equal to about two outer diameters of the tip.

NOTE Prevention of contamination 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 solution defined for the reference electrode and ending in a capillary ("Luggin capillary"), the end of which is placed close to the test surface.

7.5 Potentiostat requirements

Any electronic potentiostat capable of a current output ≥ 100 mA, voltage output ≥ 10 V, and a potential control accurate and stable to 1 mV can be used. Select hardware and software that allows either recording the current for 24 h or integrating the current for 24 h.

7.6 Reagents

7.6.1 Sodium chloride, analytical grade.

7.6.2 Water to Grade 2, as specified in ISO 3696.

7.7 Preparation of the electrolyte

Make up a fresh solution of 0,154 mol/l sodium chloride by adding (9,0 ± 0,1) g analytical grade sodium chloride to 600 ml water (Grade 2, as specified in ISO 3696) then make up with water (Grade 2, as specified in ISO 3696) to (1 000,0 ± 0,5) ml.

7.8 Test procedure

Fill the cell with this electrolyte. The cell shall remain open to the atmosphere. However, the cell shall be covered with a lid to prevent excessive evaporation from the electrolyte.

Raise the temperature of the corrosion test cell to (37,0 ± 0,5) °C and maintain it at this temperature.

Insert the test-piece, connect the test-piece and electrodes to the potentiostat (no potential control) and wait (10,0 ± 0,1) min. During potential stabilization it is advisable to stir the solution, for example by using a magnetic stirrer and a stirring bar in the corrosion test cell. Stir at a rate no greater than (2 ± 1) Hz. [(120 ± 60) rpm.]

Record the potential at the end of the (10,0 ± 0,1) min exposure period. After making this measurement, allow (5 ± 1) min for the motion of the electrolyte to become insignificant before starting the polarization part of the test.

Set the potentiostat to the appropriate control potential (see Table 2) and time (24 h). Apply the potential and record or integrate the current for (24,0 ± 0,2) h. During the polarization part of the test, the electrolyte shall be stagnant (no stirring).

7.9 Data acquisition and processing

7.9.1 General

Several options are available and any one may be used.

7.9.2 Computer-controlled potentiostat

A 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[5)].

Ensure that the integration procedure uses the true values of current rather than their logarithms. If the recording is logarithmic, convert the data to true values.

7.9.3 Coulometer

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

7.9.4 Data-logging and integration

If equipment of neither 7.9.2 nor 7.9.3 is available, record the polarization current using any available data acquisition system. The integration can be performed then 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 is constant, or by using any other suitable and sufficiently accurate integration method.

7.10 Calculation of the total charge transported

7.10.1 Test-pieces embedded by casting without masking

Divide the anodic charge recorded in coulombs in 7.9, by the cross-sectional area of the test-piece, in square centimetres (7.2).

7.10.2 Test-pieces embedded by casting with masking

Divide the anodic charge recorded in coulombs in 7.9, by the area, in square centimetres, of amalgam remaining exposed after the masking has been applied (7.2).

7.11 Test report

A test report shall be prepared. At least the following information shall be included:

a) name of the dental amalgam alloy;

b) a reference to this document (i.e. ISO/TS 17988:2020);

c) name and address of the manufacturer;

d) open circuit potential in V, SHE;

e) the average rate of charge transfer per unit area in the period of 24 h in C/(cm^2.d);

f) reference electrode;

g) temperature of reference electrode in °C;

h) applied potential in V, SHE;

i) name and address of organization responsible for the test (e.g. test house, university, department of manufacturer);

5) A VersaSTAT 3 potentiostat with VersaSTAT300 inbuilt software by Princeton Applied Research, Oak Ridge, TN, USA, is an example of a suitable equipment and software combination available commercially. This information is for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

j) any deviations from the procedure;

k) any unusual features observed;

l) date of the test.

8 Determination of the resistance to corrosion by the Hertzian-loading strength-reduction test

8.1 Principle

For dental amalgam, localized enhanced corrosion can occur in thin crevices filled with an electrolyte (i.e. saliva). This phenomenon exists for many other alloy systems and is recognized to be a cause of failure even when uniform surface corrosion is slight. When comparing alloys, their relative performance in crevices should not be taken as similar (though enhanced) to that for uniform surface corrosion. To do this ignores the susceptibility of a particular alloy to crevice corrosion. Resistance to crevice corrosion should be determined experimentally.

In this experimental procedure, crevice corrosion conditions are created between a flat disc-shaped test piece and the base of a container. Over time a significant loss of metal may take place from this surface and lead to a reduction in the force required to fracture the test-piece when it is subjected to Hertzian indentation loading. This reduction can be determined by measuring the force to fracture an identical test-piece that has been held in dry air. Because the fracture force for different dental amalgam products is not the same, the resistance to corrosion under crevice conditions can be compared by calculating the percentage reduction.

8.2 Test solution (artificial saliva)

8.2.1 Reagents

All reagents shall be analytical grade, with the exception of lactic acid which shall be ≥ 85 %.

8.2.1.1 Sodium dihydrogen phosphate.

8.2.1.2 Potassium chloride.

8.2.1.3 Sodium chloride.

8.2.1.4 Ammonium chloride.

8.2.1.5 Trisodium citrate dihydrate.

8.2.1.6 Lactic acid solution.

8.2.1.7 Urea.

8.2.1.8 Uric acid.

8.2.1.9 Sodium hydroxide.

8.2.1.10 Potassium thiocyanate.

8.2.1.11 Hydrochloric acid.

8.2.1.12 Water to Grade 2, as specified in ISO 3696.

8.2.2 Stock solutions

Make the following three stock solutions using water to Grade 2, as specified in ISO 3696. Store these separately in a refrigerator at (4 ± 2) °C.

8.2.2.1 Stock solution A

8.2.2.1.1 Sodium dihydrogen phosphate 28,0 g/l.

8.2.2.1.2 Potassium chloride 86,8 g/l.

8.2.2.1.3 Sodium chloride 7,2 g/l.

8.2.2.1.4 Ammonium chloride 11,0 g/l.

8.2.2.1.5 Trisodium citrate dihydrate 1,1 g/l.

8.2.2.1.6 Lactic acid solution 3,5 g/l.

8.2.2.2 Stock solution B

8.2.2.2.1 Urea 5,0 g/l.

8.2.2.2.2 Uric acid 0,375 g/l.

8.2.2.2.3 Sodium hydroxide 0,1 g/l.

8.2.2.3 Stock solution C

8.2.2.3.1 Potassium thiocyanate 12,0 g/l.

8.2.3 Test solution (artificial saliva)

Immediately before making the dental amalgam test-pieces, make the test solution by placing 500 ml of water (to Grade 2, as specified in ISO 3696) into a 1 l volumetric flask. To this add, in turn, 20 ml of solution A, 40 ml of solution B and 20 ml of solution C, followed by a further addition of water (to Grade 2, as specified in ISO 3696) to make up the volume to within 20 ml of the mark. If necessary, adjust to pH 6,2 by using sodium hydroxide or hydrochloric acid, as required. Then finally, make up to the mark with water (to Grade 2, as specified in ISO 3696).

8.3 Test-piece production and procedure for test-piece conditioning

8.3.1 Apparatus

8.3.1.1 Air oven or incubator maintained at (37 ± 2) °C.

8.3.1.2 Measuring cylinder with a minimum measuring capacity of 25 ml and accurate to 1 ml (or similar volume-measuring piece of equipment).

8.3.1.3 Micrometer screw gauge or similar measuring instrument, to measure test-piece thickness to an accuracy of 0,01 mm.

