TECHNICAL SPECIFICATION

ISO/TS 17988

First edition
2014-04-01

Dentistry — Corrosion test methods for dental amalgam

Médecine bucco-dentaire — Essais de corrosion des amalgames dentaires

Reference number
ISO/TS 17988:2014(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 106, *Dentistry*, Subcommittee SC 1, *Filling and restorative materials*.

This is the first edition of ISO/TS 17988, *Dentistry — Corrosion test methods for dental amalgam.* Part of the subject matter in this Technical Specification was formerly contained in two annexes to ISO 24234:2004, *Dentistry — Mercury and alloys for dental amalgam.*

Introduction

Dental amalgam alloy and dental mercury are the essential and only components of dental amalgam restorative material. This Technical Specification, of which this is the first edition, 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 are not intended to be used directly for any biocompatibility claims, for which their use is inappropriate.

Should other corrosion test procedures emerge as suitable for application to dental amalgam and use in the comparative evaluations of products, they will be included in future editions of this Technical Specification.

Dentistry — Corrosion test methods for dental amalgam

1 Scope

This Technical Specification gives details of test procedures for evaluating the corrosion resistance of dental amalgam produced from a dental amalgam alloy product.

It is applicable to dental amalgam formed from products that are within the scope of ISO 24234, *Dentistry — Dental amalgam.*

It is not applicable to dental metallic materials that are within the scope of ISO 22674, *Dentistry — Metallic materials for fixed and removable restorations and appliances.*

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

NOTE Dental mercury is at least 99,99 % pure, and as such it is a metallic element of high commercial purity, and not an alloy.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 6344-1, *Coated abrasives — Grain size analysis — Part 1: Grain size distribution test*

ISO 7488, *Dental amalgamators*

ISO 13565-2, *Geometrical Product Specifications (GPS) — Surface texture: Profile method; Surfaces having stratified functional properties — Part 2: Height characterization using the linear material ratio curve*

ISO 13897, *Dentistry — Amalgam 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.

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

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

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.

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.

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, 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.

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

Note 1 to entry: For the present case of dental amalgam, the mercury released as vapour is also relevant.

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, expressed in $C/(cm^2 \cdot d)$

3.8
Hertzian-loading strength-reduction corrosion 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.

4 Sampling

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

For pre-capsulated products, all capsules shall be of the same lot.

For a free-flowing dental amalgam alloy powder in bulk, or dental amalgam alloy tablets, the alloy shall be from a single lot. For use with these, the dental mercury sachets shall be from a single lot that complies with ISO 24234. (Use as many dental mercury sachets as required, according to the mixing ratio recommended by the manufacturer of the dental amalgam alloy product.)

For the immersion corrosion procedure (Clause 6) at least 3 g of dental amalgam alloy is required.

For the potentiostatic corrosion procedure (Clause 7) at least 1 g of dental amalgam alloy is required.

For the Hertzian-loading strength-reduction procedure (Clause 8), at least 35 g of dental amalgam alloy is required.

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 dental mercury should be that recommended by the manufacturer. Use a capsule (with a pestle, if needed) that complies with 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 allowed.

For pre-capsulated products, use as many capsules as needed. Mix the contents of the capsules either simultaneously using the same number of pieces of equipment of the same type, or sequentially on a single piece of equipment. (The latter is allowed, 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 an amalgamator that complies with ISO 7488 and that is recommended for mixing the amalgam alloy product with dental mercury or mixing the pre-capsulated product. Use the amalgamator setting and mixing time that is 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 Mixing

Mix a mass of the dental amalgam 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 diameter and 8 mm in length is approximately 1,2 g.

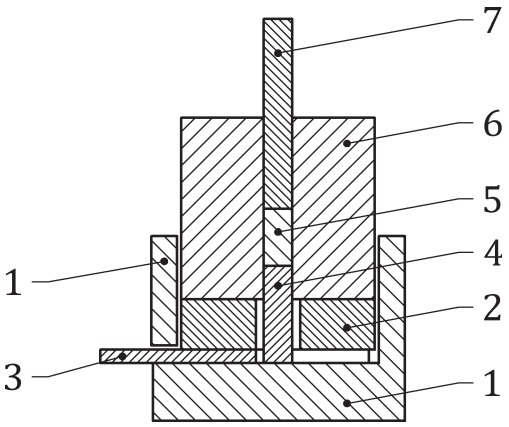
5.2.2 Apparatus for the preparation of dental amalgam cylindrical test-pieces

5.2.2.1 General

Use the apparatus as shown in Figures 1 to 4.

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 core roughness depth (R_k) not greater than 6,3 µm when tested in accordance with ISO 13565-2. Set the limits of clearance between the die and the plungers at F7 and h7, respectively, in accordance with ISO 286-2.



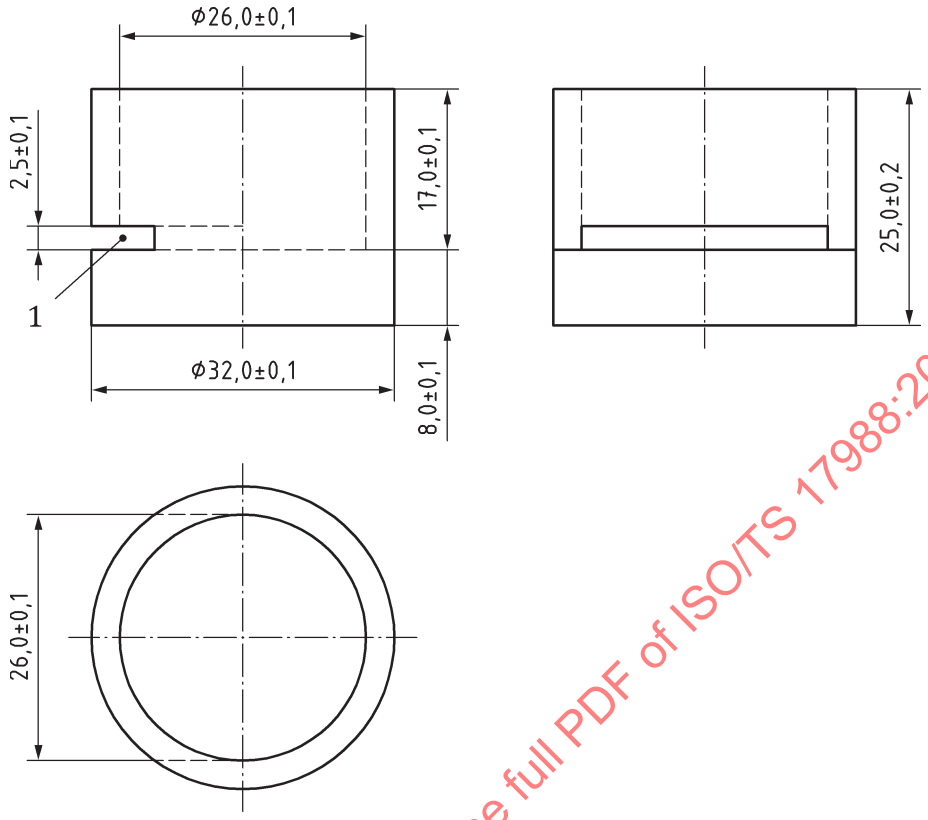
Key

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

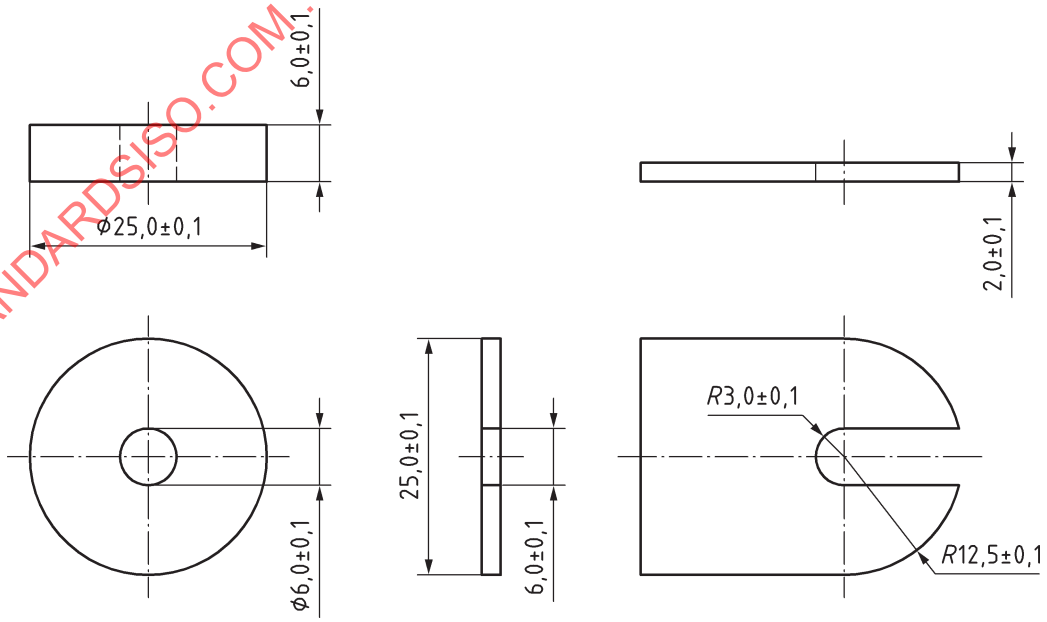
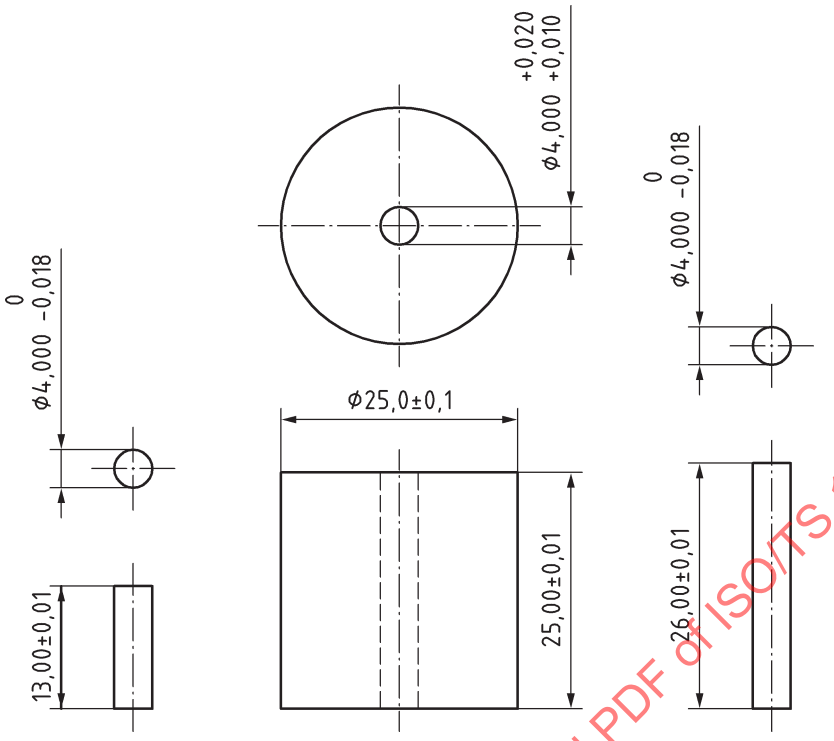


Figure 3 — Spacer no. 1 (left) and spacer no. 2 (right)

Dimensions in millimetres



NOTE 1 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 and 13 mm from one end of plunger no. 1. This end is to be in contact with the dental amalgam. Though such datum lines are not mandatory, their use is recommended.

NOTE 2 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 is between 0 µm and 18 µm less than 4,000 mm. Thus the diameter of the plunger is to be between 3,982 mm and 4,000 mm.

NOTE 3 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 is between 10 µm and 20 µm more than 4,000 mm. Thus the diameter of the hole is to be between 4,010 mm and 4,020 mm.

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

5.2.2.3 Assembly of the apparatus

Assemble the holder, spacers nos. 1 and 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 and 13 mm from the end of the plunger that is in contact with the dental amalgam), the cylinder 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 shall 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 lx and at a distance not exceeding 250 mm. A person making the inspection shall have nominally normal visual acuity. [Corrective (non-magnifying) untinted 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

Time in seconds

Procedure	Time
End of mixing at	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, at	30
Release the force and remove spacer no. 2 at	45
Reapply the force at	50
Re-release the force at	90
Carefully remove excess dental mercury and eject the test-piece at	120

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

5.3.1 Mixing

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

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

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

5.3.2.1 Mould as shown in Figure 5.

5.3.2.2 Flat square scratch-free glazed glass plate with an edge length greater than 30 mm.

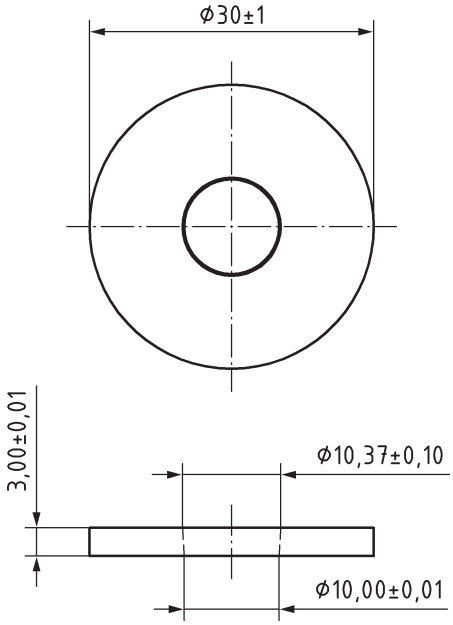
5.3.2.3 Glass microscope slide to provide a straight edge to carve back the dental amalgam.

5.3.2.4 Hand-instrument for dental amalgam packing.

5.3.3 Materials and tolerances for construction of the mould

Make the mould of hardened tool steel or hardened stainless steel. The hole should have a (7 ± 2)° taper to allow the amalgam disc to be ejected without undue force when this is applied to the face that has the smaller diameter for the hole. Hone the surface of the tapered hole to a core roughness depth (R_k) not greater than 6,3 µm when tested in accordance with ISO 13565-2.

Dimensions in millimetres



NOTE The taper is present to enable the test-piece to be ejected with a minimum force. For convenience, to distinguish readily 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.

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

5.3.4 Packing the disc-shaped test-piece

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.

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 min. 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. The surface that had been in contact with the glass plate is termed "the test surface", being the surface at which fracture initiates when the test force is applied. 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) untinted lenses may be worn.] If a defect is detected, reject that test-piece and make a replacement.

NOTE 2 To prevent any damage to the test surface during ejection, placing a thick soft pad, such as 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 Apparatus

6.1.1 Flask, of borosilicate glass (in accordance with ISO 3585), having a round bottom, 250 ml capacity, with 3 parallel necks and ground-glass conical socket joints.

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

6.1.3 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.

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

6.1.4 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 (6.1.2).

6.1.5 Mercury vapour trap (four in number), a commercially-manufactured gold-impregnated silica tubular trap[1)].

6.1.6 Atomic fluorescence mercury vapour analyser that is compatible with the selected mercury vapour trap (6.1.5) [2)].

6.1.7 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 is to fit one of the outer necks of the flask, 6.1.1).

6.1.8 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 (6.1.1) and the socket end of the condenser (6.1.7).

6.1.9 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.

6.1.10 "O" ring, of polychloroprene, with internal diameter < 3,8 mm, to fit the suspension rod (6.1.9).

6.1.11 Thread, nylon, single-ply, sewing.

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

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

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

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

6.1.16 Beakers (6 in number), of borosilicate glass (in accordance with ISO 3585), 250 ml capacity.

6.1.17 Polythene gloves.

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

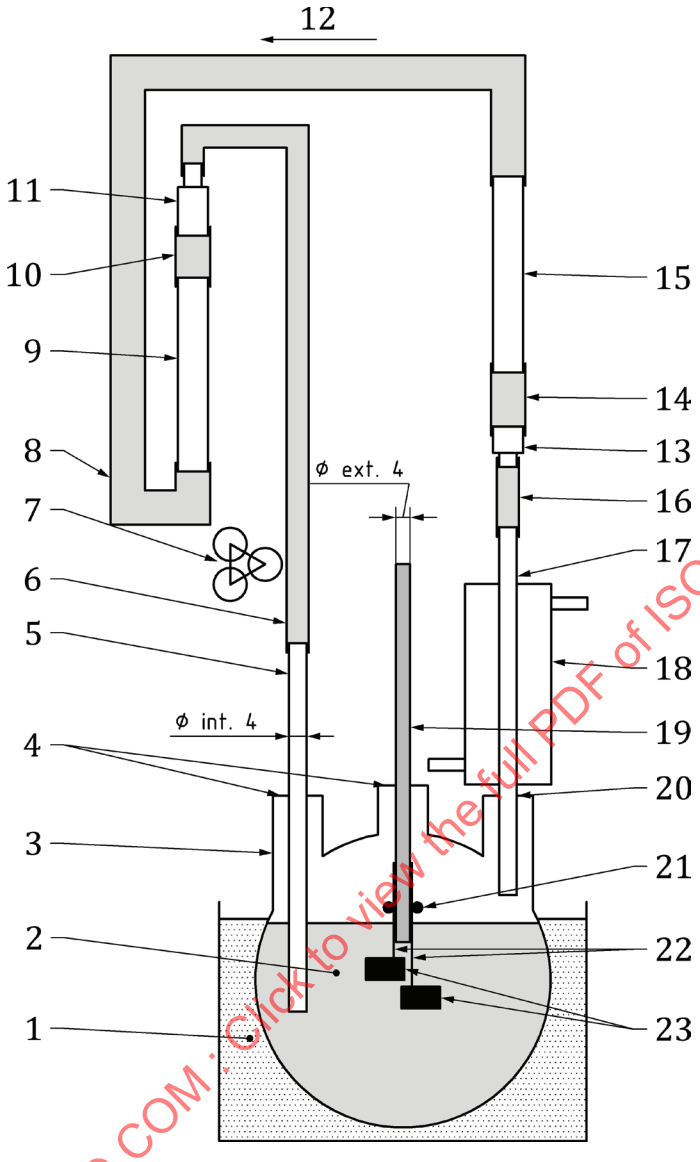
6.1.19 Beaker of borosilicate glass (in accordance with ISO 3585), 500 ml capacity.

---

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

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

Dimensions in millimetres



Key

1	water-bath at (37,0 ± 0,5) °C	13	glass reduction connector
2	0,1 mol/l lactic acid solution	14	PVC tubing (150 ± 50) mm in length
3	round-bottomed flask, 250 ml capacity, 3 parallel necks with ground-glass joints	15	gold-impregnated silica tube mercury vapour trap
4	conical ground-glass joint with a cone screw-thread adapter to create and maintain the seal with the glass rod or tube	16	PVC tubing (150 ± 50) mm in length
5	glass tube (air inlet tube)	17	conical ground-glass joint with a cone adapter to create and maintain the seal with the glass tube
6	PVC tubing (1 000 ± 100) mm in length	18	water-cooled Liebig condenser with ground-glass joints
7	peristaltic pump	19	solid glass rod (suspension rod)
8	PVC tubing (500 ± 50) mm in length	20	conical ground-glass joint
9	variable area air-flow meter	21	polychloroprene O-ring to fit the glass rod
10	PVC tubing (150 ± 50) mm in length	22	nylon threads
11	glass reduction connector	23	test-pieces
12	direction of gas flow		

NOTE 1 The peristaltic pump re-circulates the air during the test.

NOTE 2 In this diagram 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.

Figure 6 — Immersion corrosion test apparatus

6.2 Mercury vapour analyser requirements

Use a tubular gold-impregnated silica mercury vapour trap with a collection capacity of at least 50 mg mercury. To measure the mercury vapour it has collected, use a compatible atomic fluorescence mercury vapour analyser with a 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 It is possible to collect mercury vapour on a mercury vapour trap operating *remote and offline*, after which the trap is sent (to determine of the amount collected) to an analytical laboratory company that possesses an appropriate atomic fluorescence spectrometer and offers a mercury vapour analytical service. If corrosion testing of amalgam is infrequent, this may 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.

6.3 Reagents for the test solution and cleaning the apparatus

6.3.1 Lactic acid solution (approximately 90 %), analytical grade.

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

6.3.3 Nitric acid, spectroscopic grade.

6.3.4 Ethanol, analytical grade.

6.4 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.

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.

6.5 Assembly of the immersion corrosion test apparatus

The components of apparatus required are specified in 6.1. Clean the glassware following the procedure given in 6.4. 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 re-circulated air) in the mercury vapour trap.

6.6 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 (6.9.1) takes a minimum of 10 h to complete. Since the upper time limit for the storage of the second pair of test-pieces is 7d 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 cylinder to an accuracy of 0,02 mm. This is to be done at the ambient room temperature. If handling the test-piece is unavoidable, this should be done wearing previously-unused polythene gloves.

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

Clean the volumetric flask following the procedure given in 6.4.

Following a period of (6,7 ± 0,3) days after the first pair of dental amalgam test-pieces are 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 analytical 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.

6.8 Finishing the dental amalgam test-piece

Clean one of the 250 ml beakers following the procedure given in 6.4.

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 touch neither each other, nor the wall of the beaker, nor 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.

6.9 Test procedure

6.9.1 First determination

6.9.1.1 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.

Following a period of (18,0 ± 0,2) h after the start of the test-piece finishing procedure (6.8) 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 6.5 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 of the 250 ml beakers following the procedure given in 6.4.

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 6.9.1. and 6.9.2), 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 content is 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.

6.9.1.2 Corrosion procedure for the test solution

At the end of their (24,0 ± 0,2) h finishing period (6.8), 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. 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 O-ring. Return the glass rod to its position in the round-

bottomed flask. Suspend the two cylinders in the acid and adjust the lengths of the threads and vertical position of the rod in the adapter, such that the cylinders 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 (key item 4, in Figure 6) until the dental amalgam test-pieces are above the surface of the test solution. N.B. Raising the test-pieces follows the immersion period.

Clean a third 250 ml beaker following the procedure given in 6.4.

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.

6.9.2 Second determination

Make up 1 l of a fresh solution of 0,1 mol/l lactic acid, following the directions given in 6.7.

Finish the two test-pieces that were produced for the second determination (in accordance with 6.6) following the finishing procedure given in 6.8. (Use the fourth 250 ml beaker.)

Disassemble the corrosion test apparatus. Clean the glassware components according to the procedure in 6.4 and reassemble according to 6.5.

Following the procedure that is given in 6.9.1.1, 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 6.4) 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 6.9.1.2. A fresh (the fourth) mercury vapour trap should be used. After the 5 h corrosion period, pour the second test solution into a sixth 250 ml beaker (cleaned using the procedure given in 6.4) 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.

6.10 Analysis

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 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 collection 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.

6.11 Treatment of data and reporting of results

Record both sets of results.

Give values for 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 %, and their sum, in each case expressed per square centimetre of test-piece surface area, as "$\mu g/cm^2$ in 4 h".

Give the value for the release of mercury vapour as "ng/cm^2 in 4 h".

7 Determination of the corrosion by the potentiostatic procedure

7.1 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 (7.6).

Using light pressure, wet-grind the exposed end of the test-piece uniformly on coated abrasives that comply 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.

7.2 Corrosion test cell requirements

7.2.1 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).

7.2.2 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.

7.2.3 Volume of the electrolyte

Use at least 300 ml.

7.3 Reference electrode probe requirements

7.3.1 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 ± 0,002) V (SHE).

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

Table 2 — Potential settings for different reference electrodes and temperatures

Reference electrode type	Reference electrode filling solution	Temperature coefficient V / K	Reference potential V (SHE) when the temperature of the reference electrode is at			Control potential setting V when the temperature of the reference electrode is at		
			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

7.3.2 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.

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

7.3.3 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.

7.4 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.

7.5 Reagents

7.5.1 Sodium chloride, analytical grade.

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

7.6 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.

7.7 Test procedure

Fill the cell with this electrolyte. The cell is to remain open to the atmosphere. However, the cell should 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, e.g. by using a magnetic stirrer and a stirring bar in the corrosion test cell. Stir at a rate no greater than (2 ± 1) Hz. [*i.e.* (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).

7.8 Data acquisition and processing

7.8.1 General

Several options are available and any one may be used.

7.8.2 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 [3)].

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.

7.8.3 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.

7.8.4 Data-logging and integration

If equipment of neither 7.8.2 nor 7.8.3 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.

7.9 Calculation of the total charge transported

7.9.1 Test-pieces embedded by casting without masking

Divide the anodic charge recorded in coulombs in 7.8, by the cross-sectional area of the test-piece, in square centimetres (7.1).

7.9.2 Test-pieces embedded by casting with masking

Divide the anodic charge recorded in coulombs in 7.8, by the area, in square centimetres, of amalgam remaining exposed after the masking has been applied (7.1).

7.10 Reporting of results

Report the open circuit potential (V, SHE), the average rate of charge transfer per unit area for the period of 24 h [C/(cm^2.d)], the type of reference electrode, the reference electrode temperature (°C) and the applied control potential (V, SHE).

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

8.1 Reagents

All reagents are to be analytical grade:

8.1.1 Sodium dihydrogen phosphate.

8.1.2 Potassium chloride.

8.1.3 Sodium chloride.

---

3) A VersaSTAT 3 potentiostat with VersaSTAT300 inbuilt software, 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 Technical Specification and does not constitute an endorsement by ISO of this product.

