# INTERNATIONAL STANDARD

# ISO 24234

First edition
2004-10-15

# Dentistry — Mercury and alloys for dental amalgam

*Art dentaire — Mercure et alliages pour amalgame dentaire*

Reference number
ISO 24234:2004(E)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 24234 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 1, *Filling and restorative materials*.

This International Standard cancels and replaces ISO 1559:1995, ISO 1559:1995/Cor.1:1997 and ISO 1560:1985.

A number of technical revisions have been made as improvements or as a consequence of combining the International Standards that have been replaced.

- The scope of this International Standard applies to alloys for dental amalgam and dental mercury, whether provided individually or together.
- The clause permitting a deviation in the composition of alloys for amalgam has been removed.
- Guidance on biocompatibility assessment has been introduced.
- A limit on the presence of large alloy particles has been introduced.
- The requirement for loss of mercury from predosed capsules has been removed, since it is a requirement in ISO 13897.
- The values for the requirements on creep, dimensional change and compressive strength at 1 h have been revised.
- The criterion for compliance with the compressive strength requirements has been revised.
- Provisions for packaging and marking have been revised.
- Markings required for mercury safety warnings and precautions have been revised to conform to ISO requirements and the United Nations *Globally Harmonized System of Classification and Labelling of Chemicals* (GHS). They are no longer dependent upon national or regional requirements.
- Procedures for corrosion testing have been added as normative annexes.

# Introduction

Dental amalgam alloy and mercury are the essential and only components of dental amalgam restorative material. This International Standard combines the requirements and the test methods for the alloy with those for the mercury in a single standard, of which this is the first edition. Formerly, these were contained in two separate standards.

Specific qualitative and quantitative requirements for freedom from biological hazard are not included in this International Standard, but it is recommended that, in assessing possible biological hazards, reference be made to ISO 10993-1 and ISO 7405.

To enhance the safety of dentists and support staff, it would have been preferred to limit the scope solely to the use of predosed capsules of alloy and mercury. It is, however, recognised and accepted that both amalgam alloy and mercury are supplied in bulk form in some parts of the world where, for economic reasons, this is necessary for the provision of dental treatment. Therefore requirements for these products are included in this International Standard. Safety precautions relating to marking, labelling and packaging have been strengthened in this revision.

Inclusion of a requirement for corrosion resistance was considered, using the procedures for corrosion testing given in ISO/TS 17576. However it was decided that the data available were insufficient to justify a corrosion requirement in this International Standard, and as a consequence the test methods alone are given, as normative annexes. A requirement for the corrosion resistance will be set and incorporated at the earliest possible date.

# Dentistry — Mercury and alloys for dental amalgam

**WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and to determine the applicability or regulatory limitations prior to use.**

## 1 Scope

This International Standard specifies the requirements and test methods for alloys and for mercury suitable for the preparation of dental amalgam, together with the requirements and test methods for that amalgam and the requirements for packaging and marking.

It is applicable to alloys supplied in the form of either a powder in bulk, or a powder compressed to form a tablet, or a powder in predosed capsules.

It is applicable to dental mercury supplied either in bulk quantities, or in predosed sachets, or in predosed capsules.

This International Standard does not exclude the supply of alloy or mercury separately.

This International Standard is not applicable to alloys intended for use with liquid metals that are not mercury, nor is it applicable to liquid metal alloys.

## 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 286-2, *ISO system of limits and fits — Part 2: Table of standard tolerance grades and limit deviations for holes and shafts*

ISO 3310-1, *Test sieves — Technical requirements for testing — Part 1: Test sieves of metal wire cloth*

ISO 3585, *Borosilicate glass 3.3 — Properties*

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

ISO 3864-2, *Graphical symbols — Safety colours and safety signs — Part 2: Design principles for product safety labels*

ISO 4793:1980, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation*

ISO 6344-1:1998, *Coated abrasives — Grain size analysis — Part 1: Grain size distribution test*

ISO 7488, *Dental amalgamators*

ISO 8282, *Dental equipment — Mercury and alloy mixers and dispensers*

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*

*Globally Harmonized System of Classification and Labelling of Chemicals (GHS)*. United Nations, New York and Geneva, 2003, ISBN 92-1-116840-6

## 3 Terms and definitions

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

**3.1**
**alloy for dental amalgam**
alloy in fine particles, composed mainly of silver, tin and copper, which when mixed with mercury produces a dental amalgam

**3.2**
**predosed capsule**
capsule, as-supplied, containing measured amounts of alloy powder and mercury for dental amalgam, separated in such a way that premature combination is prevented

NOTE The separating barrier is broken immediately prior to mixing or breaks during mixing, allowing the alloy and mercury to come into contact.

**3.3**
**amalgam alloy tablet**
quantity of dental amalgam alloy powder that has been compressed to form a single entity for the purpose of providing a predosed quantity of the alloy.

NOTE During mixing, the tablet is intended to break apart, forming a fine powder.

**3.4**
**tailing**
phenomenon which occurs when mercury that contains impurities moves over a clean surface, tending to leave behind a portion of the liquid, forming an elongated tail as if it were sticking to that surface

**3.5**
**primary container**
container or package that is in direct contact with the material

**3.6**
**dental mercury sachet**
measured quantity of dental mercury supplied in a sachet that is suitable for a reusable mixing capsule

NOTE The sachet is broken immediately prior to mixing, or breaks during mixing, allowing the mercury to come into contact with the alloy

# 4 Requirements

## 4.1 General

**4.1.1** If the alloy is supplied as a powder in bulk, the product shall comply with

- requirements 4.2, 4.3, 4.4, 4.8, 4.9, and
- packaging requirement 7.1, and
- marking requirements 7.2.1 a) to f), h) to j) and 7.3.

**4.1.2** If the alloy is supplied as tablets, the product shall comply with

- requirements 4.2, 4.3, 4.4, 4.7, 4.8, 4.9, and
- packaging requirement 7.1, and
- marking requirements 7.2.1 a) to f), h) to j) and 7.3.

**4.1.3** If the alloy and the dental mercury are supplied together in predosed capsules, the product shall comply with

- all requirements.

**4.1.4** If the dental mercury is supplied as mercury in bulk, the product shall comply with

- requirements 4.3, 4.5, 4.6, and
- packaging requirement, 7.1 and
- marking requirements 7.2.1 a) to d), f) and g), 7.2.2 and 7.3.2.

**4.1.5** If the dental mercury is supplied in dental mercury sachets, the product shall comply with

- requirements 4.3, 4.5, 4.6 and 4.7, and
- packaging requirement 7.1, and
- marking requirements 7.2.1 a) to d), f) and g), 7.2.2 and 7.3.2.

## 4.2 Chemical composition of the alloy

The manufacturer shall declare every element that is present in a concentration greater than or equal to 0,1 % (mass fraction). All alloying elements in concentrations greater than 0,5 % (mass fraction) shall be given by name, with values rounded to the nearest percentage (mass fraction). Alloying elements that are present in concentrations between 0,1 % and 0,5 % (mass fraction) shall be named without a percentage value.

Test in accordance with 6.1.

The chemical composition shall comply with Table 1.

The total concentration of other elements shall not exceed 0,1 % (mass fraction).

**Table 1 — Requirements for chemical composition of the alloy**

| Element | Mass fraction<br>% |
|---|---|
| silver | ⩾ 40 |
| tin | ⩽ 32 |
| copper | ⩽ 30 |
| indium | ⩽ 5 |
| palladium | ⩽ 1 |
| platinum | ⩽ 1 |
| zinc | ⩽ 2 |
| mercury | ⩽ 3 |

## 4.3 Biocompatibility

See the Introduction for guidance on biocompatibility.

## 4.4 Foreign material and large particles in the alloy powder

When tested in accordance with 6.2, no more than five particles of foreign material shall be found on the sieve.

The mass of alloy particles that remain on the sieve shall not exceed 0,1 % (mass fraction) of the sample used for this test.

## 4.5 Contamination of the mercury by oil, water and foreign material

When the procedure in 6.3.1 and 6.3.2 is followed, the dental mercury shall be visibly free from contamination by oil, water or foreign material and shall have a bright mirror-like surface that does not form a scum in air within the specified period.

If the dental mercury is supplied in bulk form and does not have a bright mirror-like surface, then the procedures in 6.3.3 and 6.3.4 shall be carried out. After this procedure has been completed, the dental mercury shall have a bright, mirror-like surface that does not form a scum in air within the specified period.

## 4.6 Purity of mercury — Free pouring

When tested according to the procedure in 6.4, the mercury shall pour freely and completely without tailing.

Small droplets not coalesced with the bulk after shaking shall not be construed as evidence of non-compliance with this subclause.

## 4.7 Variability of preproportioned masses

### 4.7.1 For products supplied as predosed capsules

The coefficients of variation of the masses of the dental alloy and the dental mercury in the capsules shall not exceed 1,5 %, when tested in accordance with 6.5.1.

The arithmetic means of the masses of both alloy and mercury shall be within ± 2,0 % (mass fraction) of the manufacturer’s stated masses, when tested in accordance with 6.5.1.

### 4.7.2 For products supplied as tablets

The coefficient of variation of the mass of the dental alloy tablets shall not exceed 1,5 %, when tested in accordance with 6.5.2.

The arithmetic mean of the mass of the alloy tablet shall be within ± 2,0 % (mass fraction) of the manufacturer's stated mass, when tested in accordance with 6.5.2.

### 4.7.3 For products supplied as dental mercury sachets

The coefficient of variation of the mass of the dental mercury in the sachet shall not exceed 1,5 %, when tested in accordance with 6.5.3.

The arithmetic mean of the mass of the dental mercury in the sachet shall be within ± 2,0 % (mass fraction) of the manufacturer's stated mass, when tested in accordance with 6.5.3.

## 4.8 Properties of the amalgam

### 4.8.1 General

When tested in accordance with 6.7 to 6.9, the material shall comply with the requirements given in Table 2.

**Table 2 — Properties of amalgam**

| **Maximum creep** | **Permitted dimensional change during hardening** | **Minimum compressive strength at 1 h** | **Minimum compressive strength at 24 h** |
|---|---|---|---|
| % | % | MPa | MPa |
| 2,0 | –0,10 to +0,20 | 80 | 300 |

### 4.8.2 Creep

When tested in accordance with 6.7, either three out of three, or four out of five specimens shall meet the requirement in Table 2.

### 4.8.3 Dimensional changes during hardening

When tested in accordance with 6.8, at least four out of five specimens shall meet the requirement in Table 2.

### 4.8.4 Compressive strength at 1 h

When tested in accordance with 6.9, at least four out of five specimens, or eight out of ten specimens shall meet the requirement in Table 2.

### 4.8.5 Compressive strength at 24 h

When tested in accordance with 6.9, at least four out of five specimens, or eight out of ten specimens shall meet the requirement in Table 2.

## 4.9 Appearance of the mixed amalgam before setting

When the alloy and mercury are mixed according to the manufacturer's instructions and tested in accordance with 6.10, it shall form a coherent plastic mass, with a shiny surface.

## 5 Sampling

At least 50 g of alloy of the same lot in packages produced for retail shall be procured.

At least 50 g of mercury of the same lot in packages produced for retail shall be procured.

## 6 Test methods

### 6.1 Chemical composition of the alloy

Use a recognized, instrumented analytical procedure that has adequate sensitivity to determine the composition of the alloy for the elements declared by the manufacturer in compliance with 4.2. Inductively coupled plasma spectroscopy (ICP) is a suitable example.

For other elements that are detected at levels greater than 0,01 % (mass fraction), but are not alloying elements, sum their masses and report the sum as the “mass fraction of other elements”.

### 6.2 Foreign material and large particles in the alloy powder

For alloy supplied as a powder in bulk, weigh a (10,0 ± 0,1) g sample to an accuracy of ± 0,001 g and record ($m_s$).

For alloy supplied in predosed capsules, select and open a sufficient number of capsules to obtain a (10,0 ± 0,1) g sample of alloy. Weigh this sample to an accuracy of 0,001 g and record ($m_s$).

For alloy supplied as tablets, place a tablet in a reusable capsule that complies with ISO 13897. Break the tablet in the capsule to its constituent powder particles using a mechanical amalgamator that complies with ISO 7488, set at the machine setting and at one-half the time recommended by the alloy manufacturer for mixing the alloy and dental mercury in accordance with 7.3.1. If the manufacturer’s recommendations include any other action to break-up the tablet (e.g. use of a pestle), incorporate this at the appropriate point. Repeat this using a sufficient number of tablets to obtain (10,0 ± 0,1) g of powder. Weigh this sample to an accuracy of 0,001 g and record ($m_s$).

Place the powder sample on a sieve of mesh size 150 µm and diameter 76 mm. The sieve shall conform to ISO 3310-1. Hold the sieve assembly (consisting of collecting pan, sieve and cover) in one hand and tap it gently against the other hand at a rate of approximately twice a second for 120 s. Inspect the sieve at a magnification of × 10 for any remaining foreign material and alloy particles. Record the number of foreign material particles.

Remove any foreign material and then transfer the remaining alloy particles to a balance. Weigh to an accuracy of 0,001 g and record ($m_r$). Calculate the percentage (mass fraction) of alloy that occurs in particles that have a size greater than 150 µm, as follows:

$$\frac{m_r}{m_s} \times 100 = w\,(\%) \tag{1}$$

where

$m_r$ is the mass of alloy particles remaining on the sieve;

$m_s$ is the mass of the powder sample;

$w$ is the percentage (mass fraction) of alloy particles greater than 150 µm in diameter.

## 6.3 Contamination of the mercury by oil, water or foreign material

### 6.3.1 Preparation of the sample

#### 6.3.1.1 Dental mercury supplied in bulk form

Take a clean small flask made of borosilicate or similar glass (100 ml is a suitable capacity). Rinse with water (in accordance with ISO 3696:1987, grade 2) then with 3 mol/l nitric acid (analytical grade). Once more, rinse thoroughly with water (in accordance with ISO 3696:1987, grade 2), dry at 100 ºC and allow to cool to room temperature.

Open a new bottle of mercury and pour approximately 5 g of mercury into the flask.

#### 6.3.1.2 Dental mercury supplied in predosed capsules

Take a clean small flask made of borosilicate or similar glass (100 ml is a suitable capacity). Rinse with water (in accordance with ISO 3696:1987, grade 2) then with 3 mol/l nitric acid (analytical grade). Once more, rinse thoroughly with water (in accordance with ISO 3696:1987, grade 2), dry at 100 ºC and allow to cool to room temperature.

Select a number of capsules sufficient to obtain 5 g of mercury. Remove the mercury from the capsules, using a technique that prevents contact between the mercury and the alloy. (For this test, even trace amounts of powder dissolving in the mercury will act as a contaminant). Empty the mercury into the flask.

#### 6.3.1.3 Dental mercury in dental mercury sachets

Take a clean small flask made of borosilicate or similar glass (100 ml is a suitable capacity). Rinse with water (in accordance with ISO 3696:1987, grade 2) then with 3 mol/l nitric acid (analytical grade). Once more, rinse thoroughly with water (in accordance with ISO 3696:1987, grade 2), dry at 100 ºC and allow to cool to room temperature.

Select a number of dental mercury sachets sufficient to obtain 5 g of mercury. Open the sachets and empty the mercury into the flask.

### 6.3.2 Initial assessment

Conduct this evaluation immediately after opening the bottle, or exposing the mercury from predosed capsules or sachets. Use visual inspection, without magnification, to determine whether the dental mercury is free from contamination, has the required appearance and that the surface remains scum-free for 20 min. Carry out the inspection at an illuminance of at least 1 000 lx and at a distance not exceeding 250 mm.

Persons making the inspection shall have nominally normal visual acuity. Corrective (non-magnifying) untinted lenses may be worn.

### 6.3.3 Preparation of the mercury supplied in bulk form

This preparation procedure shall be applied only to dental mercury that is supplied in bulk form if the appearance seen at the initial assessment, 6.3.2, does not meet the requirement.

Filter approximately 5 g of the mercury through a sintered glass filter that has a pore size in the range 16 µm to 40 µm (porosity grade P 40 as defined in ISO 4793:1980) into a borosilicate (or similar) glass collection flask that has been cleaned as described in 6.3.1.1. Apply a slight vacuum if necessary.

Take a small flask, made of borosilicate or similar glass, that has an accurately fitting ground-glass stopper or a polytetrafluoroethylene-lined screw cap (100 ml is a suitable capacity). Rinse with water (in accordance with ISO 3696:1987, grade 2) then with 3 mol/l nitric acid (analytical grade). Once more, rinse thoroughly with water (in accordance with ISO 3696:1987, grade 2), dry at 100 ºC and allow to cool to room temperature.

Transfer the filtered sample of mercury to the flask. Place the stopper or cap securely (do not use grease) and shake the flask vigorously for 5 s.

### 6.3.4 Second assessment of the mercury supplied in bulk form

Examine the mercury prepared following 6.3.3. Use visual inspection, without magnification, to determine whether the dental mercury is free from contamination, has the required appearance and that the surface remains scum-free for 20 min. Carry out the inspection using conditions of lighting and visual acuity given in 6.3.2.

## 6.4 Free-pouring of mercury

Take the mercury sample produced to determine compliance with requirement 4.6. Pour the mercury. Use visual inspection to observe whether the mercury pours freely and completely without tailing. Carry out the inspection using conditions of lighting and visual acuity given in 6.3.2.

## 6.5 Determination of the variability of preproportioned masses

### 6.5.1 Predosed capsules

Weigh the alloy and the mercury separately, each to the nearest 1 mg. Determine the masses of the alloy and mercury in 25 predosed capsules. Record each value.

In order to separate the alloy powder from the mercury and prevent premature combination, several different capsule designs have been developed. Use a technique appropriate to the capsule being tested that removes all material from the capsule and avoids contact between mercury and alloy. It may be necessary to measure the mass of mercury in one set of 25 capsules, and the mass of alloy in a different set of 25 capsules.

Determine the arithmetic mean and the standard deviation for the mass of mercury, and then those for the alloy.

Calculate coefficient of variation $C_\mathrm{V}$ as follows:

$$\frac{s}{\bar{x}} \times 100 = C_\mathrm{V} \ (\%) \qquad (2)$$

where

$s$ is the standard deviation;

$\bar{x}$ is the arithmetic mean;

$C_\mathrm{V}$ is the coefficient of variation.

### 6.5.2 Amalgam alloy tablets

Weigh 25 tablets individually to the nearest 1 mg.

Determine the arithmetic mean and standard deviation for the mass of the tablet.

Calculate the coefficient of variation according to Equation (2).

### 6.5.3 Dental mercury sachets

Determine the mass of the mercury in 25 sachets.

Remove all mercury from the sachets and weigh the contents individually to the nearest 1 mg.

Determine the arithmetic mean and standard deviation.

Calculate the coefficient of variation according to Equation (2).

## 6.6 Preparation of test specimens to determine compliance with the requirements for creep, dimensional change and compressive strength

### 6.6.1 Temperature

Prepare specimens at (23 ± 2) ºC, unless specified otherwise.

### 6.6.2 Mixing

Mix a mass of the amalgam sufficient to make a test cylinder (8 ± 1) mm high after condensation into the die shown in Figure 1. If necessary, adjust the height of the cylinder by condensing only a portion of the amalgam mix into the die.

For alloy supplied either as tablets or as a powder in bulk, the alloy mass and the mercury mass to be mixed, the capsule, the pestle (if needed), and any other mixing accessory required shall conform to those recommended by the manufacturer in accordance with 7.3.2 and 7.2.1 h). Use dental mercury that complies with requirements 4.5 and 4.6 of this International Standard.

For products supplied in predosed capsule form, use as many capsules as needed. If more than one capsule has to be used, mix them simultaneously using equipment of the same type.

Use an amalgamator that complies with ISO 7488. The manufacturer of the amalgam product, in accordance with 7.3.1 b), shall specify the amalgamator's setting and the mixing time.

### 6.6.3 Apparatus for the preparation of test specimens for determining creep, dimensional change and compressive strength

#### 6.6.3.1 General

Use the apparatus as shown in Figures 1 to 5.

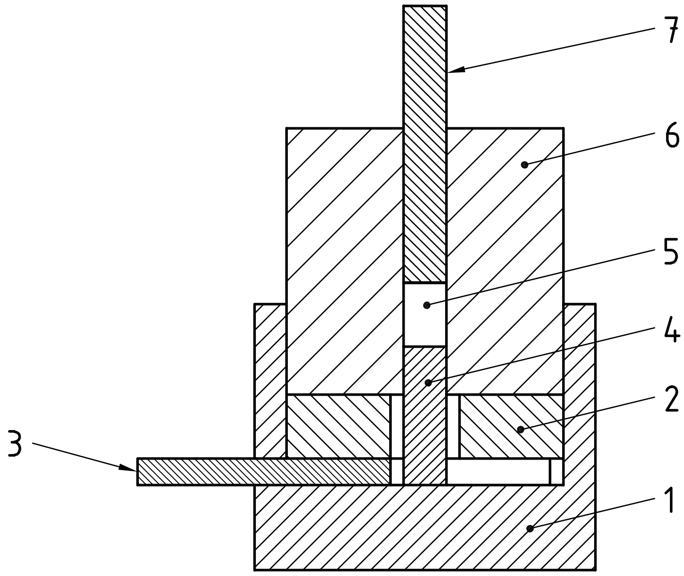
#### 6.6.3.2 Materials and tolerances for construction of the apparatus

Make the holder, the spacers and the cap 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. Limits of clearance between the die and the plungers shall be F7h7 in accordance with ISO 286-2.

#### 6.6.3.3 Assembly of the apparatus

For production of creep and compressive strength specimens, assemble the holder, spacers Nos. 1 and 2, the die and plunger No. 2 as shown in Figure 1.

Particular measuring instruments used in the dimensional-change test (e.g. interferometers) may require an impression on the end surface of the specimen that is produced by the cap that is shown in Figure 5. For production of dimensional-change specimens, include the cap in the assembly if this is appropriate for the measuring instrument that is to be used. In which case, position the cap on top of the plunger No. 2.



**Key**

1 holder

2 spacer No. 1

3 spacer No. 2

4 plunger No. 2

5 test specimen

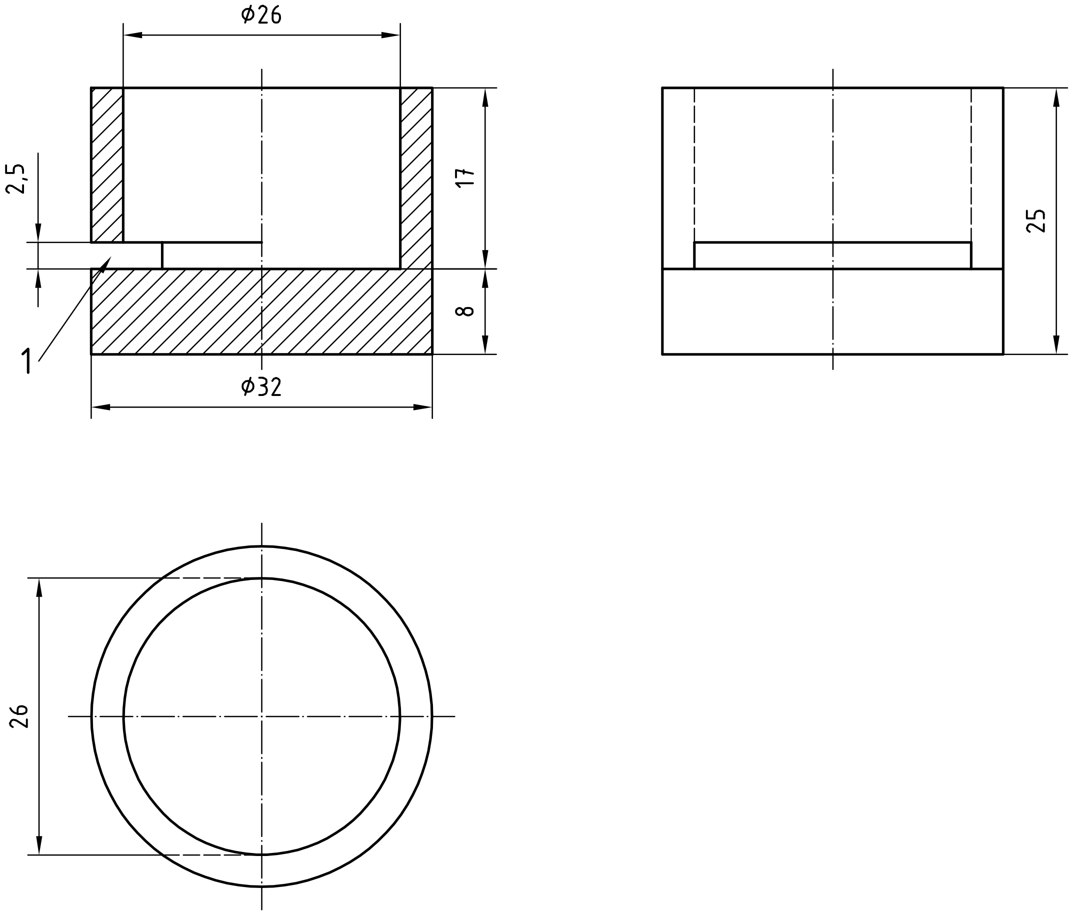
6 die

7 plunger No. 1

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

**Figure 1 — Vertical section through the apparatus for making amalgam test specimens, showing the assembled apparatus with a specimen in place**

Dimensions in millimetres

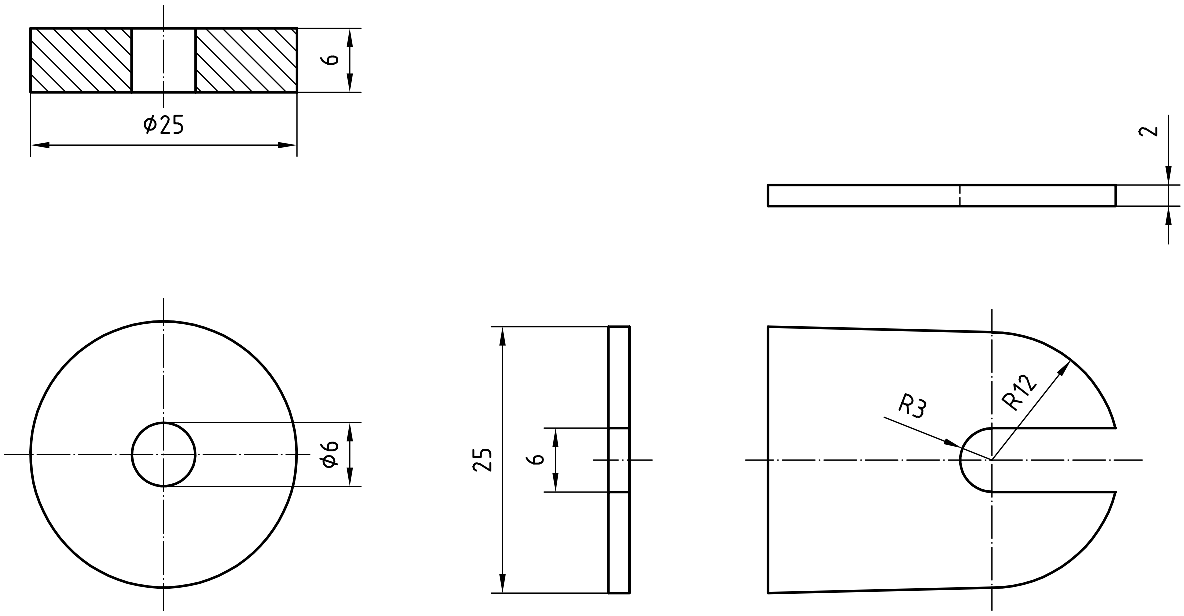


**Key**

1 slot

Figure 2 — The holder

Dimensions in millimetres

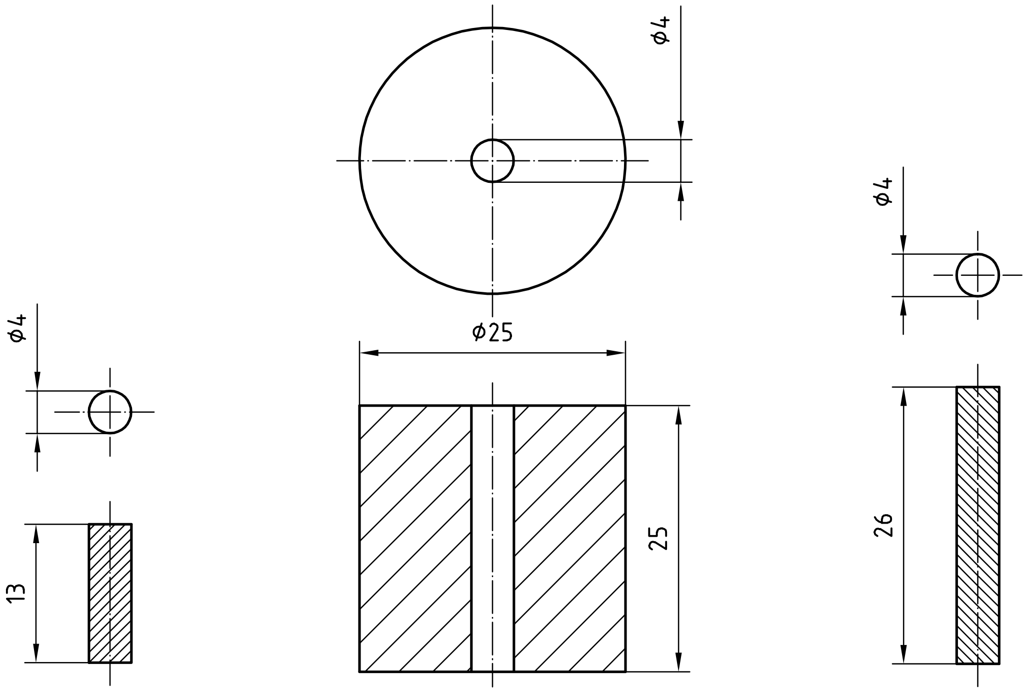


a) **Spacer No. 1**

b) **Spacer No. 2**

**Figure 3 — Spacer No. 1 and Spacer No. 2**

Dimensions in millimetres



a) **Plunger No. 2**

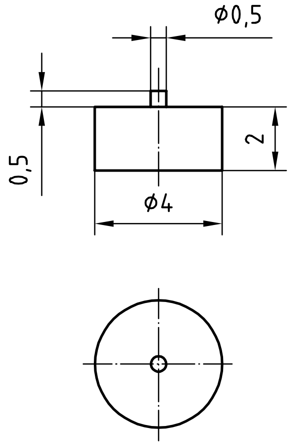
b) **The die**

c) **Plunger No. 1**

To assist the operator to judge whether the correct quantity of amalgam has been inserted, to give a specimen that is within the permitted range for length, i.e. (8 ± 1) mm, three circumferential datum lines should be scribed at 9 mm, 11 mm and 13 mm from one end of plunger No. 1. This end shall be in contact with the amalgam. Though such datum lines are not mandatory, their use is recommended.

**Figure 4 — Plunger No. 2, The die and Plunger No. 1**

Dimensions in millimetres



This cap shall be used in conjunction with the apparatus shown in Figure 1 if the measuring instrument requires a small indentation at one end of the specimen.

**Figure 5 — Cap used for the production of dimensional-change specimens**

### 6.6.4 Condensation

**Table 3 — Schedule for the preparation of test specimens**

Time in seconds

| Procedure | Time |
|---|---|
| End of mixing | 0 |
| Place mixed mass into the die, insert plunger No. 1 and apply a load to produce a pressure of (14 ± 1) MPa | 30 |
| Release load and remove spacer No. 2 at | 45 |
| Replace load at | 50 |
| Release load at | 90 |
| Carefully remove excess mercury and eject specimen at | 120 |

Empty the coherent mass of the mixed amalgam on top of the die cavity and insert immediately, using several thrusts of an amalgam condenser slightly less than 4 mm in diameter. Do not express mercury during insertion. Insert plunger No. 1 and follow the schedule in Table 3. The specimen shall not be trimmed. After ejection, transfer the specimen to air maintained at (37 ± 1) °C.

NOTE If plunger No. 1 has circumferential datum lines scribed on its cylindrical surface, they will provide reference points to determine whether the amount of amalgam inserted will give a specimen that is within the permitted range for length. The lines are at 9 mm, 11 mm and 13 mm from the end of the plunger that is in contact with the amalgam.

If the cap (Figure 5) is not present in the assembled apparatus and the 13 mm datum line alone can be seen, the specimen will have a length that is (8 ± 1) mm.

If the cap (Figure 5) is present in the assembled apparatus and both 11 mm and 13 mm datum lines can be seen but the 9 mm line cannot, the specimen will have a length that is (8 ± 1) mm.

## 6.7 Determination of creep

### 6.7.1 Preparation of test specimens

Prepare the specimens in accordance with 6.6. Make five specimens. Store these at (37 ± 1) ºC for (7,0 ± 0,2) days. Prior to testing, grind the surface of both ends of each specimen plane and perpendicular to the axis using wet, coated abrasives that comply with microgrit size P1200, according to ISO 6344-1:1998. Measure the length of each specimen and record it, to the nearest 0,01 mm, as the original length.

### 6.7.2 Procedure

Apply a stress of (36,0 ± 0,2) MPa normally and uniformly over the cylinder ends, continuously for 4 h at a temperature of (37,0 ± 0,5) ºC. Record the change in specimen length between (1,00 ± 0,05) h and (4,0 ± 0,1) h to an accuracy of 0,01 mm.

Calculate the creep strain, to the nearest 0,1 %, as follows:

$$\frac{\Delta l}{l_\text{o}} \times 100 = \varepsilon_\text{C} \qquad (3)$$

where

$\Delta l$ is the change in length occurring between 1 h and 4 h;

$l_\text{o}$ is the original length;

$\varepsilon_\text{C}$ is the creep strain, as a percentage.

Test three specimens. If all three results meet the requirement in Table 2, it is not necessary to test the other two specimens.

If one of the three specimens fails to meet the requirement in Table 2, test two more specimens.

Test no more than five specimens.

## 6.8 Determination of dimensional change during hardening

### 6.8.1 Preparation of test specimens

Prepare the specimens in accordance with 6.6.

### 6.8.2 Procedure

Place the specimen in the measuring instrument. Do not subject the specimen to a restraint greater than 0,02 N during the test. Maintain the specimen at a temperature of (37 ± 1) ºC during the test. Measure the dimensional change that occurs between (5,0 ± 0,1) min and (24,0 ± 0,1) h from the end of mixing, to an accuracy of ± 0,5 µm. At (24,0 ± 0,1) h measure the specimen length to an accuracy of ± 0,01 mm.

Calculate the dimensional change, to the nearest 0,01 %, as follows:

$$\frac{\Delta l_{\mathrm{d}}}{l_{\mathrm{d}}} \times 100 = \varepsilon_{\mathrm{d}}(\%) \tag{4}$$

where

$\Delta l_{\mathrm{d}}$ is the dimensional change occurring between 5 min and 24 h;

$l_{\mathrm{d}}$ is the length at 24 h;

$\varepsilon_{\mathrm{d}}$ is the dimensional change as a percentage.

## 6.9 Determination of compressive strength

### 6.9.1 Preparation of test specimens

Prepare the specimens in accordance with 6.6.

### 6.9.2 Procedure

Determine the compressive strength by means of a suitable mechanical testing machine. During the test, maintain the specimen at a temperature of (23 ± 2) °C. Apply the load normally and uniformly over the cylinder ends at a crosshead speed of (0,5 ± 0,1) mm/min. For each specimen, record the compressive strength to the nearest 5 MPa.

### 6.9.3 Compressive strength at 1 h

Determine the compressive strength of five specimens at (60 ± 2) min after mixing.

If only three specimens meet the requirement for compressive strength at 1 h in Table 2, determine the compressive strength of five more specimens.

Test no more than ten specimens.

### 6.9.4 Compressive strength at 24 h

Determine the compressive strength of five specimens at (24 ± 1) h after mixing.

If only three specimens meet the requirement for the compressive strength at 24 h in Table 2, determine the compressive strength of five more specimens.

Test no more than ten specimens.

## 6.10 Appearance of the mixed amalgam before setting

### 6.10.1 Apparatus

**6.10.1.1 Glass plate,** with an area of at least 50 mm × 50 mm, a thickness of at least 5 mm and having glazed surfaces.

**6.10.1.2 Mould and ejection components,** comprising spacer No. 1, die and plunger No. 2 of the apparatus for making amalgam test specimens that are specified in Figures 3 a), 4 a) and 4 b).

### 6.10.2 Procedure and assessment

Place spacer No. 1 on the glass plate. Upend plunger No. 1 in the centre hole of the spacer. Place the die over the protruding end of plunger No. 1, thereby creating a cavity 5 mm deep into which the amalgam will be condensed.

Mix following the procedure in 6.6.1 and 6.6.2.

Use visual inspection to determine the appearance of the surface of the mixed amalgam and whether a coherent mass exists initially.

30 s after the end of mixing, place a piece of mixed amalgam on top of the die, over the cavity. The amount should be sufficient to fill the cavity to the extent that, after condensation, approximately 1 mm of the cavity above the amalgam surface remains unfilled. Condense immediately, with 10 thrusts within a period of 10 s, using a hand condenser that has a diameter of approximately 4 mm. Eject the specimen at 120 s by removing the spacer, then pressing down the die so that the plunger forces out the specimen.

Use visual inspection to determine whether a coherent mass has been maintained during condensation.

Carry out both inspections under conditions of lighting and visual acuity given in 6.3.2.

# 7 Marking, labelling and packaging

## 7.1 Packaging

Dental amalgam alloy and dental mercury shall be packaged in primary containers that (under normal conditions of storage and use in a dental clinic) prevent spillage or contamination.

Dental mercury that is supplied in bulk shall be packaged in a primary container that, when filled with the mass of mercury supplied, will remain intact without splitting or cracking if dropped 0,8 m onto a smooth ceramic floor. Dental mercury that is supplied in bulk shall be packaged in a primary container that has a top or lid that seals the container to prevent the release of mercury vapour during storage.

For shipment, these primary containers shall be packaged in outer containers that (under normal conditions of shipment) prevent spillage or contamination.

The packaging of predosed capsules shall comply with the requirements in ISO 13897.

## 7.2 Marking

### 7.2.1 Information

The following information shall be included in the package or accompanying printed literature:

a) the type of material and its application;

b) the product’s brand- or trade-name;

c) the name and address of the manufacturer or agent in the country of sale;

d) the lot number;

e) the mass of alloy in each primary container of bulk powder, or in each tablet, or in each predosed capsule;

f) the number of containers of bulk powder, or number of capsules, or number tablets, or the number of dental mercury sachets, or the number of containers of bulk mercury in each package;

g) the mass of mercury in each primary container of mercury, or in each predosed capsule, or in each dental mercury sachet;

h) the nominal recommended alloy-to-mercury mass ratio;

i) a list of those elements present in the alloy in concentrations greater than 0,1 % (mass fraction). If an element is present in a concentration greater than 0,5 % (mass fraction), its proportion shall also be listed, to the nearest percentage point;

j) a general description of the alloy particle shape(s);

k) in addition, if the product is supplied in predosed capsules, the labelling shall be in accordance with the requirements of ISO 13897.

#### 7.2.2 Safety precautions — Packs that contain dental mercury

The outer pack and the manufacturer's instructions shall be marked with the warning labelling given in a) below and the storage recommendation given in b) below.

The primary container for dental mercury supplied in bulk form shall carry this warning labelling on the outside surface of each primary container, in addition to those on the outer pack and the manufacturer's instructions.

a) a warning symbol, the general warning symbol (with the signal word WARNING and supplementary information texts) and the mandatory action symbol (and supplementary text). All shall comply with the requirements of ISO 3864-2. These shall be presented as follows (see Figure 6):

   - The general warning symbol (an exclamation mark) and warning triangle shall be printed in black on an orange triangular background.
   - The mandatory action circle shall be printed in blue on a white circular background. The general warning symbol (an exclamation mark) shall be printed in white.
   - The signal word (WARNING) and supplementary information shall be printed in black.
   - The outlines of the boxes containing these warnings shall be printed in black.

b) a recommendation to store at a temperature no higher than 25 ºC.

The preceding instructions are intended for the safety of the user. For shipment, the containers of multiple packs shall be marked in accordance with the *Globally Harmonized System of Classification and Labelling of Chemicals* (United Nations). Symbols, signal words and hazard statements shall be applied that are appropriate for mercury (UN substance number 2809).

### 7.3 Manufacturer's instructions

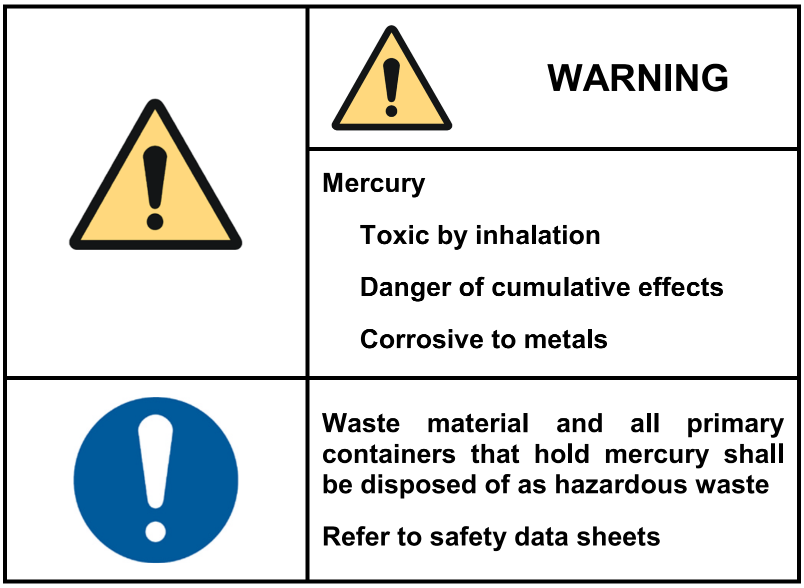
#### 7.3.1 General instructions

Printed instructions shall accompany each package and shall include at least the following information.

a) Masses of alloy and mercury

   For alloy supplied as powder in bulk, instructions shall include the setting of at least one specified dispenser that conforms with ISO 8282 and the recommended mass of dental mercury and mass of alloy for each of several masses of amalgam mix.

   For alloy supplied as tablets, instructions shall include the recommended mass of mercury for mixes made with one, two and three tablets.



**WARNING**

**Mercury**

**Toxic by inhalation**

**Danger of cumulative effects**

**Corrosive to metals**

**Waste material and all primary containers that hold mercury shall be disposed of as hazardous waste**

**Refer to safety data sheets**

**Figure 6 — Warning labelling**

b) Mixing

The instructions shall specify the brand and model number or name of an amalgamator complying with ISO 7488 that is recommended for mixing the alloy with mercury. The machine setting and the time required for mixing each of the mixes specified in accordance with 7.3.1 a) or the mix in the predosed capsule specified in accordance with 7.2.1 e) and 7.2.1 g) shall be given. In addition, the mixing accessories (e.g. capsules or other mixing containers and pestles) that are needed shall be specified. If such mixing accessories are not included with the package, information needed for ordering the mixing accessories shall be given.

The instructions shall also provide a description of the initial appearance of correctly mixed amalgam, such as the initial appearance (reflectivity), texture and coherence.

### 7.3.2 Precautionary notes

The manufacturer's printed instructions shall contain the following precautionary notes:

a) Spillages

"Mercury presents a health hazard if incorrectly handled. Spillages of mercury should be removed immediately, including from places which are difficult to access. Use a plastic syringe to draw it up. Smaller quantities can be covered by sulfur powder and removed. Avoid inhalation of the vapour."

b) Moisture contamination

"If moisture is introduced into the amalgam before it has set, properties such as strength and corrosion resistance may be affected adversely. If the alloy contains zinc, such contamination may result in an excessive expansion (delayed expansion). Whenever it is possible, use a dry field."

# Annex A
(normative)

# Determination of immersion corrosion for dental amalgam

## A.1 Apparatus

**A.1.1 Flask**, of borosilicate glass in accordance with ISO 3585, round bottom, 250 ml with 3 parallel necks and ground-glass conical socket joints.

**A.1.2 Inlet tube**, of borosilicate glass in accordance with ISO 3585, with internal diameter (4,0 ± 0,2) mm and approximate length 150 mm.

**A.1.3 Flow-tube**, of borosilicate glass in accordance with ISO 3585, with glass float and a measurement range 0 ml/min to10 ml/min.

**A.1.4 Peristaltic pump**, variable speed, capable of operating up to 20 r/min, to provide an air flow rate of (5,0 ± 0,3) ml/min through the inlet tube (see A.1.2).

**A.1.5 Gold foil mercury vapour dosimeter**1).

**A.1.6 Mercury vapour analyser**, compatible with the selected dosimeter2).

**A.1.7 Liebig condenser**, straight water-cooled borosilicate glass (in accordance with ISO 3585) at least 20 cm in length, with ground glass joints (lower cone end to fit one of the outer necks of the flask).

**A.1.8 Three cone/screwthread adapters,** of borosilicate glass, in accordance with ISO 3585, to fit outer and centre necks of the flask (A.1.1) and the socket end of the condenser (A.1.7).

**A.1.9 Suspension rod**, of borosilicate glass in accordance with ISO 3585, with diameter (4,0 ± 0,2) mm and approximate length 150 mm.

**A.1.10 O-ring**, of neoprene, internal diameter < 3,8 mm to fit suspension rod (A.1.9).

**A.1.11 Thread**, nylon, single ply, sewing.

**A.1.12 PVC tubing**, clear, plasticized, with internal diameters 3,2 mm to 6,4 mm, as required3).

**A.1.13 Reduction connectors**, of borosilicate glass in accordance with ISO 3585, of number and size as required.

**A.1.14 AAS, ICP-OES or ICP-MS analytical facility**.

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

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

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

**A.1.15 Water bath**, with temperature control to maintain (37,0 ± 0,5) ºC.

**A.1.16 Beaker**, of borosilicate glass in accordance with ISO 3585, of capacity 250 ml.

## A.2 Reagents

**A.2.1 Lactic acid**, (approximately 90 %) analytical grade.

**A.2.2 Water**, grade 2 in accordance with ISO 3696:1987.

**A.2.3 Nitric acid**, spectroscopic grade.

**A.2.4 Ethanol**, analytical grade.

## A.3 Specimen preparation

Prepare four cylindrical specimens of the type used for compressive strength measurement, according to 6.6.

Store the specimens in air for (7,0 ± 0,3) days at (37 ± 2) ºC.

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

## A.4 Preparation of lactic acid solution

24 h before the start of the 4 h immersion period, make up a solution of lactic acid ($c$ = 0,1 mol/l) using lactic acid (A.2.1) and water (A.2.2). Measure its pH. If the pH value is outside the range 2,3 ± 0,2 discard and prepare a fresh solution.

## A.5 Corrosion test cell

The apparatus required is specified in A.1 and its assembly shown Figure A.1.

Before assembly, wash all inner surfaces of the glassware with water (A.2.2) then with 3 mol/l nitric acid (A.2.3). Rinse with water (A.2.2) and shake off residual water. Rinse with analytical grade ethanol (A.2.4). Allow to dry in air.

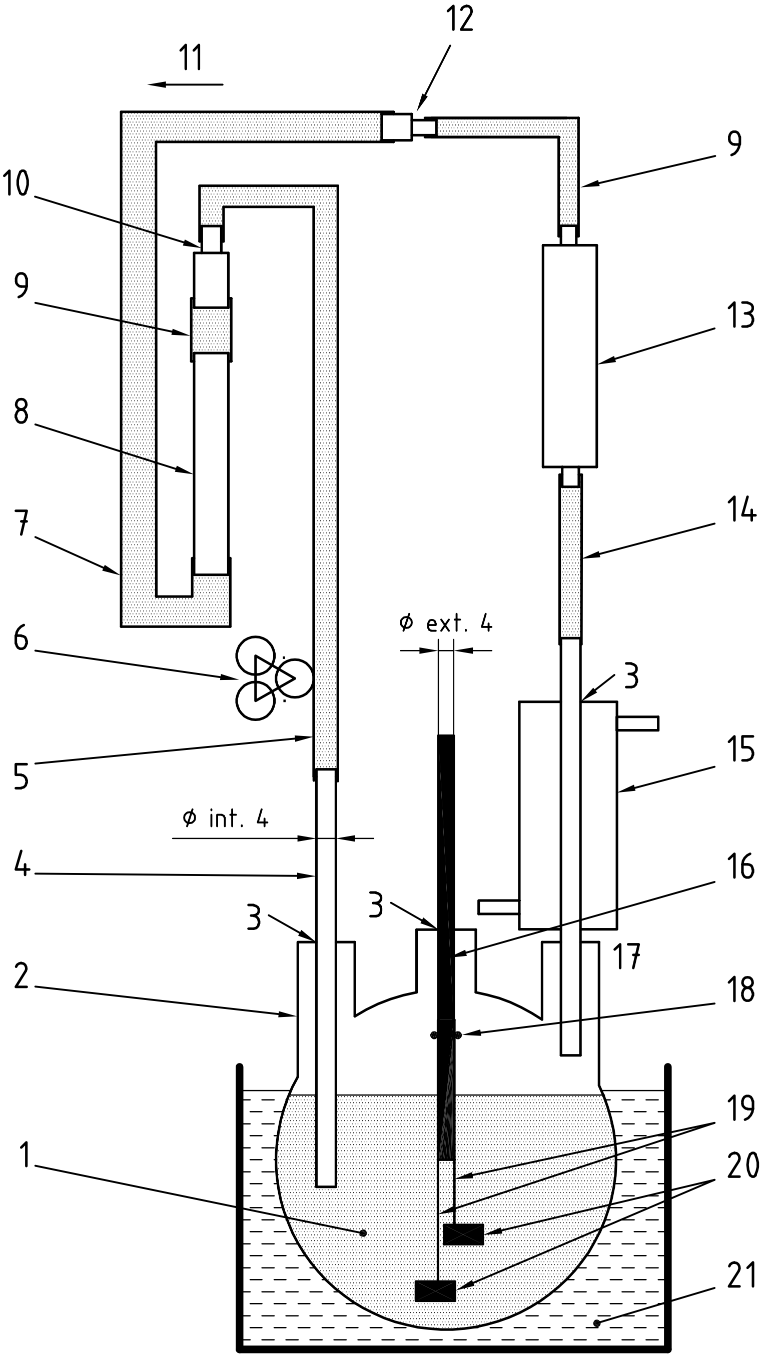
Assemble the apparatus. Use new PVC tubing (A.1.12) to connect the condenser (A.1.7) to the dosimeter (A.1.5), the dosimeter to the flow-tube (A.1.3) and the flow-tube to the glass inlet tube (A.1.2). Use tubing with an internal diameter that will produce a tight fit when it is pushed over the ends of these components. (It may be necessary to use a range of tube diameters and glass reduction connectors.) At this stage do not add the solution or the amalgam specimens.

The peristaltic pump (A.1.4) recirculates the air during the test. The temperature of the water flowing through the condenser should be low enough to prevent condensation of water (from vapour in the recirculated air) in the dosimeter.

Dimensions in millimetres

**Key**

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



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

Figure A.1 — Corrosion test apparatus

## A.6 Mercury vapour analyser specification

The mercury vapour is collected on a gold foil dosimeter and analysed by a gold foil mercury vapour analyser. The dosimeter shall have a collection capacity of at least 1 µg mercury. The analyser shall have a lower limit for detection of 10 ng or less and measurements shall be accurate to 1 ng.

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

## A.7 Test procedure

### A.7.1 Pre-corrosion

Suspend two cylinders by nylon threads (A.1.11) in a clean 250 ml glass beaker (A.1.16) containing (200,0 ± 0,1) ml of the 0,1 mol/l lactic acid solution (A.4). Immerse the specimens and adjust the length of the nylon suspension threads such that the specimens do not touch each other, nor the wall nor the base of the container. Cover the beaker to prevent evaporation. Pre-corrode these specimens for (24,0 ± 0,2) h at (37,0 ± 0,5) °C. Discard this solution when the specimens are removed (see A.7.3.1).

### A.7.2 Preparation of blank solution

It is necessary to measure the metal ion content of the solution to establish the background value that results from impurities in the solution and contamination from the glassware.

At (18,0 ± 0,2) h after the start of pre-corrosion, add (200,0 ± 0,1) ml of the 0,1 mol/l lactic acid solution (A.4) to the flask. Set the glass inlet tube vertically and adjust, to place the end at a depth of (20 ± 2) mm. Place the glass rod (without specimens attached) in the centre neck of the flask. Raise a waterbath (maintained at (37,0 ± 0,5) °C) around the flask until the surface of the lactic acid is at the same height as the surface of the water in the bath. (10 ± 2) min after this action, turn on the peristaltic pump and adjust the speed to give a flow rate of (5,0 ± 0,3) ml/min.

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

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

Pour out and discard any acid solution that remains in the flask.

NOTE It is convenient to analyse the blank solution and the test specimen solutions (obtained by A.7.3.1 and A.7.3.2) at the same time. Therefore, it is permitted to hold these in a clean and covered borosilicate glass container while the remainder are being prepared.

### A.7.3 Corrosion procedure and analysis

#### A.7.3.1 First determination

At the end of the (24,0 ± 0,2) h pre-corrosion period, remove the two specimens from the solution and rinse them with water (A.2.2). Do not touch the specimens with bare or gloved hands. Add (200,0 ± 0,1) ml of the 0,1 mol/l lactic acid solution (A.4) to the flask. Attach the specimens to the glass rod using the nylon threads and neoprene O-ring (A.1.10). Suspend the two cylinders in the acid and adjust the lengths of the threads such that the cylinders are completely immersed and touch neither each other nor the surface of the flask. Raise the waterbath until 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. Maintain the flask containing the two specimens and the lactic acid at (37,0 ± 0,5) °C for the duration of the test.

At $(4,0 \pm 0,1)$ h after immersion, remove the specimens from the solution by loosening the screwthread adapter and raising the glass rod. Tighten the screwthread adapter when the specimens are above the solution. Continue to circulate the air for a further $(1,0 \pm 0,1)$ h to flush mercury vapour from the airspace above the solution.

Disconnect the dosimeter and measure the mercury vapour it has collected using the gold foil mercury vapour analyser. The range of the instrument can be extended by using a dilution module that has been set at a factor of approximately $\times 10$. Calculate the amount of released mercury vapour per square centimetre of specimen surface.

Using a recognized procedure (for example, ICP) with adequate sensitivity (i.e. $0,1 \times 10^{-6}$ or better), analyse the solution for ions of the elements that are present in the alloy in concentrations greater than 0,5 % (mass fraction) according to 7.2.1 i), and for mercury which has leached out into solution. Calculate the amounts released for each element per square centimetre of specimen surface.

### A.7.3.2 Second determination

Disassemble the apparatus. Wash the glassware. Rinse in water (A.2.2), then 3 mol/l nitric acid (A.2.3). Rinse again with water (A.2.2) and finally rinse with ethanol (A.2.4). Allow to dry. Reassemble with new PVC tubing (A.1.12).

Make up a fresh solution of 0,1 mol/l lactic acid, following directions given in A.4.

Pre-corrode the other two specimens, following directions given in A.7.1.

Repeat the corrosion test and analysis, following directions given in A.7.3.1.

### A.7.3.3 Reporting results

Report both sets of results. Subtract the background values obtained in A.7.2. Give values for each of the elements that are present in the alloy in concentrations (mass fractions) greater than 0,5 % in accordance with 7.2.1 i) and for mercury, and their sum per square centimetre of specimen surface, as “μg/cm$^2$ in 4 h”. Give the value for the release of mercury vapour as “ng/cm$^2$ in 4 h”.

# Annex B
(normative)

# Potentiostatic determination of corrosion for dental amalgam

## B.1 Specimen preparation

Prepare an amalgam specimen of the type used for the measurement of compression strength, in accordance with 6.6. Store in air at (37 ± 2) ºC for (7,0 ± 0,3) days.

Measure the diameter of the specimen to an accuracy of 0,01 mm. Then calculate the cross-sectional area (in square centimetres).

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

Cover the connecting lead junction and all surfaces except one end of the specimen with an insulating material, preferably by casting in epoxy. The temperature rise during setting of the resin should not exceed 15 ºC. This material should not dissolve in, nor react with the electrolyte.

Uniformly wet-grind the exposed end of the specimen on coated abrasives that comply with microgrit size P1200 in accordance with ISO 6344-1. Wash with water (ISO 3696, grade 2).

Eliminate the crevice between the amalgam and the embedding materials by casting or masking. If masking is considered essential and applied, the area of the amalgam that is left exposed shall be determined to an accuracy of 0,000 1 cm$^2$. This area shall be recorded in units of square centimetres.

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

## B.2 Requirements for corrosion test cell

### B.2.1 Corrosion cell

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

### B.2.2 Temperature control

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

### B.2.3 Volume of the electrolyte

Use a volume $\geqslant$ 300 ml.

## B.3 Reference electrode/probe requirements

### B.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). Control the potential at (0,000 ± 0,002) V vs. saturated calomel electrode (SCE) at 25 ºC, equivalent to (0,241 5 ± 0,002) V (SHE). The reference potential values for some other common reference electrodes, and corresponding values for the control potential settings are shown in Table B.1. Other electrodes can be used based on their known potential difference from SHE.

### B.3.2 Temperature of the reference electrode

Measure the temperature of the reference electrode. If the temperature differs from 25 ºC by more than 1 ºC, the control potential shall be adjusted using the temperature coefficient for the given electrode type. Temperature coefficients and examples of potential correction are shown in Table B.1.

### B.3.3 Positioning of the reference electrode

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

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

## B.5 Test procedure

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

Fill the cell. The cell remains open to the atmosphere, however, it should be covered with a lid to prevent excessive evaporation of the electrolyte.

Raise the temperature of the cell to (37,0 ± 0,5) ºC and maintain at this temperature.

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

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

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

## B.6 Data acquisition and processing

### B.6.1 General

The following options are available for data acquisition and processing.

### B.6.2 Coulometer

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

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

### B.6.3 Computer-controlled potentiostat

An equally convenient procedure is to use a computer-controlled potentiostat with a program for potentiostatic control and software which allows post-test integration of the recorded current4) .

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

### B.6.4 Data-logging and integration

If neither the B.6.2 nor B.6.3 instrument is available, record the polarization current using any available data-acquisition system. The integration then can be performed by averaging all the current data and multiplying the average current, in amperes, by the total exposure time, in seconds (assuming that the time between current measurements is constant), or using any other suitable and sufficiently accurate integration method.

## B.7 Calculation of the surface density of charge

### B.7.1 Specimens embedded by casting

Divide the anodic charge, recorded in coulombs in B.6, by the cross-sectional area of the specimen, in square centimetres (B.1).

### B.7.2 Specimens embedded by casting with masking

Divide the anodic charge, recorded in coulombs in B.6, by the area, in square centimetres, of amalgam remaining exposed after the masking has been applied (B.1).

## B.8 Reporting results

Report the open-circuit potential (V, SHE), the anodic surface density of charge (C/cm$^2$), the type of reference electrode, the reference electrode temperature (°C) and the applied control potential (V, SHE).

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4) EG&G 252/352 software for the Model 273x, 283 potentiostats, Perkin Elmer Instruments / Princeton Applied Research, Oak Ridge, TN, USA are examples of suitable equipment and software combinations available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product. If this instrument is selected its dilution module is required.

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

| **Reference electrode type** | **Electrolyte** | **Temperature coefficient**[5] | **Reference potential**<br>V(SHE)<br>when the temperature of the reference electrode is | | | **Control potential setting**<br>V<br>when the temperature of the reference electrode is | | |
|---|---|---|---|---|---|---|---|---|
| | | V/K | 18 °C | 25 °C | 37 °C | 18 °C | 25 °C | 37 °C |
| Saturated calomel (SCE) | saturated KCl | $-7,50 \times 10^{-4}$ | 0,246 8 | 0,241 5 | 0,232 5 | –0,005 | 0,000 | 0,009 |
| 1,0 M calomel | 1 mol/l KCl | $-2,40 \times 10^{-4}$ | 0,281 7 | 0,280 0 | 0,277 1 | –0,040 | –0,039 | –0,036 |
| 0,1 M calomel | 0,1 mol/l KCl | $-7,00 \times 10^{-5}$ | 0,334 2 | 0,333 7 | 0,332 9 | –0,093 | –0,092 | –0,091 |
| 0,1 M silver chloride | 0,1 mol/l KCl | $-6,50 \times 10^{-4}$ | 0,292 7 | 0,288 1 | 0,280 3 | –0,051 | –0,047 | –0,039 |

# Bibliography

[1] ISO 7405, *Dentistry — Preclinical evaluation of biocompatibility of medical devices used in dentistry — Test methods for dental materials*

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