INTERNATIONAL STANDARD

ISO 1559

Third edition
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Dental materials — Alloys for dental amalgam

Produits dentaires — Alliages pour amalgame dentaire

Reference number
ISO 1559:1995(E)

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.

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.

International Standard ISO 1559 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 1, *Filling and restorative materials*.

This third edition cancels and replaces the second edition (ISO 1559:1986), of which it constitutes a technical revision. (See the Introduction.)

International Organization for Standardization
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Introduction

This International Standard was first published in 1978 and was based on International Dental Federation (FDI) Specification No. 1. It was then the subject of a planned programme of revision, and a second edition was published in 1986. The more significant changes in this third edition are the following.

— The requirement for chemical composition has been changed.

— The procedures for amalgamation have been specified in more detail.

— A pass rate rather than an average value has been incorporated in the test requirements.

— The requirements for marking of the packages have been extended.

— The requirement for dimensional change has been revised.

— A requirement to limit foreign material in the alloy powder has been added.

— The precaution for zinc-containing alloys has been revised.

It is proposed to continue the efforts to include a corrosion test requirement for dental amalgam at the earliest possible date.

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Dental materials — Alloys for dental amalgam

1 Scope

This International Standard specifies requirements and test methods for alloys composed mainly of silver, tin and copper, complying with the composition requirements (see 4.1). The alloy may be in either powder or tablet form, or in capsules with portions of alloy and mercury predosed by the manifacturer, suitable for the preparation of dental amalgam.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 78-4:1983, *Layouts for standards — Part 4: Standard for atomic absorption spectrometric analysis.*

ISO 286-2:1988, *ISO system of limits and fits — Part 2: Tables of standard tolerance grades and limit deviations for holes and shafts.*

ISO 468:1982, *Surface roughness — Parameters, their values and general rules for specifying requirements.*

ISO 1560:1985, *Dental mercury.*

ISO 3310-1:1990, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth.*

ISO 7488:1991, *Dental amalgamators.*

3 Definitions

For the purposes of this International Standard, the following 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 capsulated alloy and mercury: Capsules predosed with alloy and mercury by the manufacturer.

4 Requirements

4.1 Chemical composition

4.1.1 General

The chemical composition of the alloy shall comply with table 1.

The total contamination by other elements shall not exceed 0,1 % (m/m).

Table 1 — Chemical composition requirements

Metal	Content % (m/m)
Silver	40 min.
Tin	32 max.
Copper	30 max.
Indium	5 max.
Palladium	1 max.
Platinum	1 max.
Zinc	2 max.
Mercury	3 max.

If atomic absorption spectrometry is used for chemical analysis, it shall be carried out in accordance with ISO 78-4.

4.1.2 Deviations in chemical composition

Compositions other than those specified in 4.1.1 may be permissible subject to approval by regulatory authorities. The silver content shall nevertheless not be less than 40 % (m/m).

4.2 Physical properties

When tested in accordance with 6.2 to 6.4, the material shall comply with the requirements given in table 2.

Table 2 — Physical properties

Creep	Dimensional change	Compressive strength	
		after 1 h	after 24 h
%	%	MPa	MPa
max.		min.	min.
3	− 0,15 to + 0,20	50	300

4.3 Mass

The coefficient of variation of the mass of alloy tablets shall not exceed 1,5 % (m/m) and the arithmetic mean of the mass of alloy tablets shall be within ± 2 % (m/m) of the manufacturer's stated mass when tested in accordance with 6.5.

For capsules containing preproportioned alloy and mercury, the coefficients of variation of the masses of the alloy and the mercury shall each not exceed 1,5 % (m/m) and the arithmetic means of the masses of both the alloy and the mercury shall each be within ± 2 % (m/m) of the manufacturer's stated masses when tested in accordance with 6.5.

4.4 Loss of mercury

Capsules predosed with alloy and mercury by the manufacturer shall meet the following requirements:

— the mercury in capsules shall comply with ISO 1560;

— the loss in mass for each capsule during amalgamation shall not exceed 0,5 mg when tested according to 6.6.

4.5 Foreign material

When tested in accordance with 6.7, the alloy shall contain no more than five particles of foreign material.

5 Sampling

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

6 Test methods

6.1 Preparation of test specimens

6.1.1 Temperature

Prepare all test specimens at (23 ± 2) °C.

6.1.2 Amalgamation

The mass of the amalgam mix shall be sufficient to make a test cylinder (8 ± 1) mm high after condensation with a diameter according to figure 1. If necessary, adjust the height of the cylinder by condensing only a portion of the amalgam mix into the mould. For alloy supplied as tablets or powder, the alloy mass and the mercury mass to be mixed, the capsule (if needed), the pestle (if needed), and any other mixing accessories required shall conform to those recommended by the manufacturer in accordance with 7.3.1 and 7.3.2. For capsulated alloy and mercury, simultaneously amalgamate as many capsules as needed. An amalgamator that complies with ISO 7488 shall be used. The amalgamator's frequency (or machine setting) and the amalgamation time shall be as specified by the manufacturer in accordance with 7.3.2.

Dimensions in millimetres

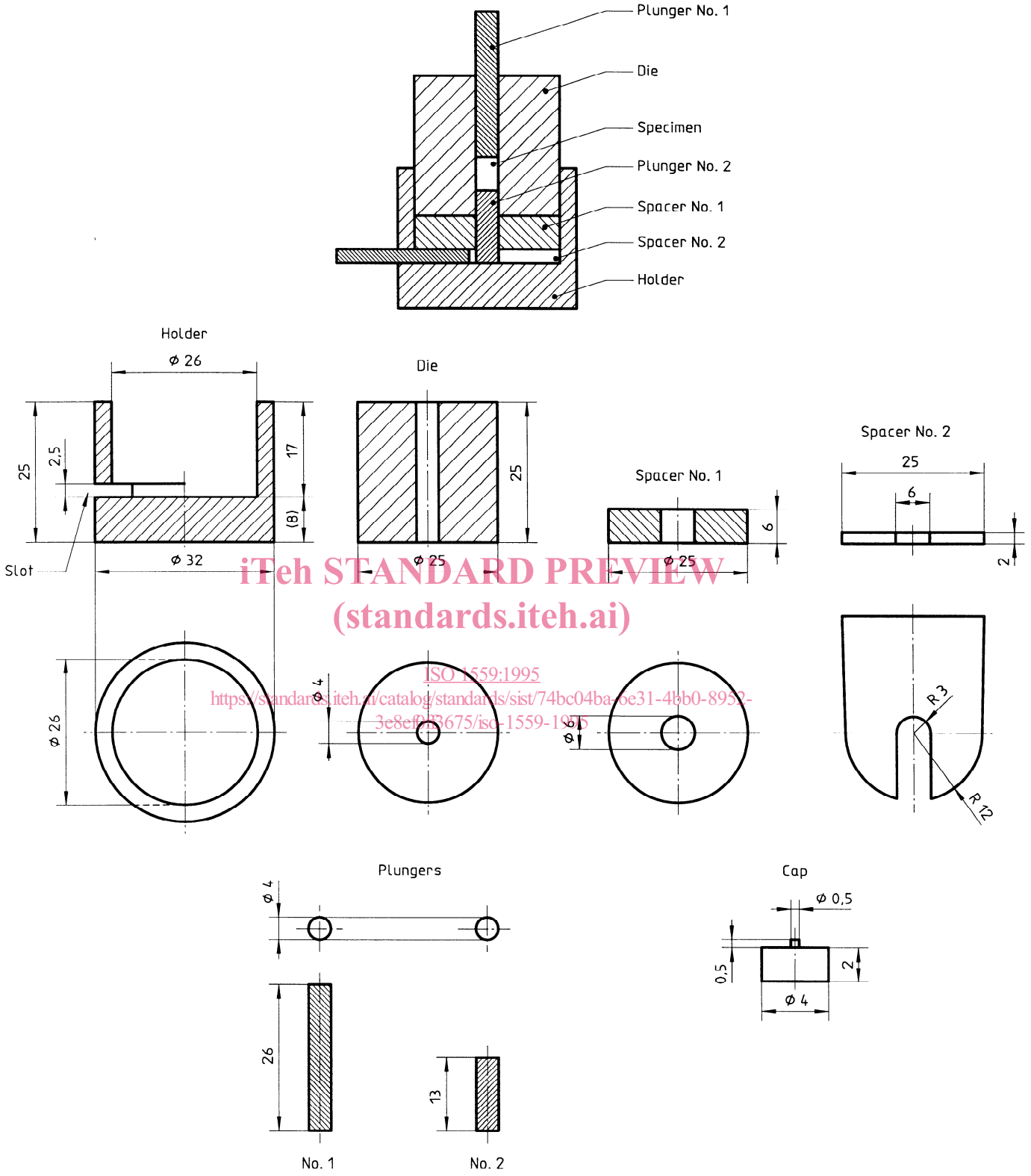

Figure 1 — Apparatus for making amalgam test specimens, showing assembled apparatus and dimensions of each component

6.1.3 Apparatus for preparation of test specimens for determination of creep, dimensional change and compressive strength

Use an all-mechanical apparatus as shown in figure 1.

The holder, spacers and cap need not be hardened; they shall be made of cold-rolled or stainless steel. Make the die and plungers of hardened tool steel or hardened stainless steel. Hone the working surfaces of the die and plungers to a surface roughness value (r_a) of not greater than 6,3 µm when tested in accordance with ISO 468. Limits of clearance between the die and plungers shall be F7/h7 in accordance with ISO 286-2.

Assemble the holder, spacers Nos. 1 and 2, the die and plunger No. 2 as shown in figure 1. Position the cap shown in figure 1 on top of plunger No. 2 when dimensional change specimens are being made. A glass plate may be used instead of a cap.

6.1.4 Condensation

Empty the coherent mass of amalgamated material on top of the cavity of the die and immediately insert the amalgam into the mould with several thrusts of an amalgam condenser slightly less than 4 mm in diameter. Do not express mercury during insertion. When preparing specimens for dimensional change tests, insert the other cap (shown in figure 1) with the flat surface up. Insert plunger No. 1 and follow the time schedule in table 3. The specimen shall not be trimmed. After ejection, transfer the specimen to an environment maintained at (37 ± 1) °C.

Table 3 — Schedule for preparation of test specimens

Procedure	Time s
End of amalgamation	—
Place amalgamated mass in the mould and apply a load to produce a pressure of (14 ± 1) MPa	30
Release load and remove No. 2 spacer at	45
Replace load at	50
Release load at	90
Carefully remove excess mercury and eject specimen at	120

6.2 Determination of creep

6.2.1 Preparation of test specimens

Take three specimens prepared in accordance with 6.1), using plungers Nos. 1 and 2 (see figure 1). Store the specimens at (37 ± 1) °C for 7 days. Prior to testing, prepare the surface of both ends of each specimen plane and perpendicular to the axis with wet ASTM 600 grit size or FEPA P1200 silicon carbide papers. Measure the length of each specimen and record it to the nearest 10 µm as the original length.

6.2.2 Procedure

Axially apply a stress of (36 ± 0,2) MPa continuously to the specimen for 4 h at (37 ± 0,5) °C. Record the change in length between the 1 h and 4 h readings and calculate the creep to the nearest 0,1 %, as follows:

$$\text{Creep} = \frac{\text{change in length between 1 h and 4 h}}{\text{original length}} \times 100$$

If one out of three fails, make two more. Three out of three or four out of five shall meet the requirement given in table 2.

6.3 Determination of dimensional change during hardening

6.3.1 Preparation of test specimens

Five specimens shall be prepared in accordance with 6.1.

6.3.2 Procedure

Place each specimen in the measuring instrument; do not subject specimens to a restraint greater than 0,02 N during the test. Make the initial measurement 5 min after the start of mixing and the final measurement after 24 h. During the test, maintain the specimen at a temperature of (37 ± 1) °C. Measure the dimensional change with an accuracy of 0,5 µm and calculate the change to the nearest 0,01 % by dividing the height change by the height at 24 h. Four out of five specimens shall meet the requirement given in table 2.

6.4 Determination of compressive strength

6.4.1 Preparation of test specimens

Ten specimens shall be prepared in accordance with 6.1.

6.4.2 Procedure

Determine the compressive strength of five specimens (60 ± 2) min after amalgamation and the other five specimens (24 ± 1) h after amalgamation by

means of a suitable test machine. The load shall be applied parallel to the cylinder axis at a crosshead speed of (0,5 ± 0,01) mm/min. For each specimen, record the compressive strength to the nearest 1 MPa. Four out of five specimens shall meet the requirement given in table 2.

6.5 Determination of mass

Twenty-five tablets and, in the case of capsulated alloy and mercury, both the alloy and mercury in 25 capsules, shall be individually weighed to the nearest 1 mg. To measure the mercury and alloy in the capsules, first remove the mercury and weigh it. Next, weigh the capsule then, after removing the alloy powder using a brush or vacuum, reweigh the capsule. The mass of the alloy shall be calculated by subtracting the capsule's mass after removing the powder from its mass before removing the powder. The arithmetic mean and standard deviation shall be determined and the coefficient of variation v in percent shall be calculated as follows:

$$v = \frac{s}{x} \times 100$$

where

s is the standard deviation;

x is the arithmetic mean.

6.6 Determination of loss in mass during amalgamation of capsulated alloy and mercury

Weigh each of five capsules to the nearest 0,1 mg after removing any material from the surface and before amalgamating in accordance with 6.1.2. After amalgamating, allow each capsule to cool to room temperature for 1 h and then reweigh it.

The loss in mass shall be calculated as the capsule mass before amalgamation minus the capsule mass after amalgamation. If one out of five fails, test 10 more capsules. Five out of five or 14 out of 15 shall meet the requirement given in 4.4.

6.7 Foreign material

Powdered alloy is required for this test. For alloy supplied as tablets, place two tablets in a capsule. Break the tablets in the capsule into powder using a mechanical amalgamator set at one-half the machine setting and the time specified by the alloy manufacturer for mixing alloy and mercury in accordance with 7.3.2.

Place 10 g of powdered alloy on a 150 µm sieve 76 mm in diameter. The sieve shall conform to ISO 3310-1. Hold the sieve assembly, consisting of pan, sieve and cover, in one hand and tap it gently against the other hand at a rate of approximately 100 times a minute for 120 s. Inspect the residue remaining on the sieve for foreign material at a magnification of × 10.

7 Marking, labelling and packaging

7.1 Packaging

The alloy shall be packaged so as to prevent spillage or contamination of the alloy or, in the case of capsulated alloy and mercury, of the mercury.

7.2 Marking

7.2.1 Information

The marking of each outer pack containing bulk alloy powder, tubes of tablets or capsulated alloy and mercury shall include

a) the alloy's brand- or trade-name;

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

c) the type of material and its application;

d) the manufacturer's batch reference;

e) the mass and/or number of units in each package of bulk alloy, tablets or predosed capsules;

f) a list of those elements present in the alloy in concentrations greater than 0,1 % (m/m);

g) a description of the particle shape(s).

7.2.2 Safety precautions

If the outer pack contains capsulated alloy and mercury, both the outer pack and the manufacturer's instructions shall be marked with

a) a hazard warning symbol complying with requirements of ISO 1560 and a reference to that International Standard;

b) a recommendation to store at temperatures no higher than 25 °C.