# INTERNATIONAL STANDARD

# ISO 9693

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## Dental ceramic fused to metal restorative materials

*Produits pour restaurations dentaires métallo-céramiques*



Reference number
ISO 9693:1991(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 9693 was prepared by Technical Committee ISO/TC 106, *Dentistry*.





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## Introduction

Dental casting alloys and ceramics are suitable for use in fabrication of metal-ceramic dental restorations.

Specific qualitative and quantitative requirements of freedom from biological hazard are not included in this International Standard but it is recommended that, in assessing possible biological or toxicological hazards, reference should be made to ISO/TR 7405:1984, *Biological evaluation of dental materials*, or any more recent edition.

It is intended to replace the metallo-ceramic bond characterization test with a clinically relevant bond test as soon as it is available in a future revision of this Standard. Requirements and test methods for tarnish and corrosion resistance for the components and for the metallo-ceramic system will also be included in the future as soon as they are available.

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# Dental ceramic fused to metal restorative materials



## 1 Scope

This International Standard specifies requirements and test methods for dental casting alloys and ceramics suitable for use in the fabrication of metallo-ceramic dental restorations together with requirements and test methods for the composite structure.

The requirements of this International Standard apply to the alloys and caramics when used in combination and compliance may not be claimed for either alloys or for ceramics alone.

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

ISO 6872:1984, *Dental ceramic.*

ISO 6892:1984, *Metallic materials — Tensile testing.*

## 3 Definitions

For the purposes of this International Standard, the following definitions apply.

**3.1 alloy:** Casting alloy suitable for use as the substructure of a metallo-ceramic restoration.

**3.2 alloy coatings and bonding agents:** Substances (e.g. electroplated layers, or agents containing ceramic and/or alloy particles) which, when applied to the metal substructure and fired under appropriate time-temperature conditions improve aesthetics and may enhance the adherence of ceramic to the coated alloy surface.

**3.3 alloy conditioning:** Process of conditioning the alloy substructure, either by heat treatment or by other means, designed to enhance the bonding of ceramic to metal.

**3.4 heating rate:** Rate of increase in temperature in degrees Celsius per minute.

**3.5 firing schedule:** Temperature-time cycle stating the initial temperature, the time period at the initial temperature, if any, the heating rate, the final temperature, the time period at the final temperature, if any, and in the case of vacuum firing the temperature of vacuum application and the point of release.

**3.6 opaque bonding dental ceramic:** Ceramic product that, when mixed with distilled water or appropriate modelling liquid, applied to an alloy, and treated according to the firing schedule for the opaque ceramic, will bond to the alloy surface to form a layer that visibly masks the metallic colour.

**3.7 dental dentine ceramic:** Slightly translucent, pigmented dental ceramic used to give the overall shape and basic colour of the ceramic part of a ceramic fused to metal restoration or prosthesis.

**3.8 dental enamel ceramic:** Translucent, lightly-pigmented dental ceramic used on a base (or core) of dentine ceramic to simulate the natural tooth enamel.

## 4 Requirements

### 4.1 Chemical composition

#### 4.1.1 Alloy

The percentage of each of the constituents of the alloy, in excess of 2 % (*m*/*m*), shall be within 0,5 % (*m*/*m*) (noble metal alloys) and within 1 % (*m*/*m*) (base metal alloys) of the values stated on the package label or insert [see 8.2.2 c)]. The percentage of nickel, beryllium and cadmium shall not be greater than the amounts indicated on the package label or insert [see 8.2.2 d)] when analysed according to 6.1.1.

Testing shall be carried out in accordance with 6.1.1.

#### 4.1.2 Ceramic

The ceramic shall meet the requirements of ISO 6872:1984, clauses 5.1 and 5.2 (except for the note in 5.2.2). Fluorescing agents which will increase the radioactivity of the ceramic shall not be added.

### 4.2 Biocompatibility

See the Introduction for guidance on biocompatibility.

### 4.3 Properties

#### 4.3.1 Alloy

The mechanical properties of the alloys shall comply with the requirements of table 1.

The solidus and liquidus temperatures of the alloys shall be within ± 10 °C of the values stated on the package label or insert [see 8.2.2 g)].

The coefficient of linear thermal expansion of the alloys shall be within 0,2 × $10^{-6}$ $K^{-1}$ of the value stated on the package label or insert [see 8.2.2 h)].

The density of the alloy as supplied by the manufacturer shall be within 0,5 g/$cm^3$ of the value stated on the package label or insert [see 8.2.2 i)].

Testing shall be carried out in accordance with 6.1.2, 6.1.3 and 6.3.1 respectively. Standard test procedures shall be used for determining the density.

**Table 1 — Mechanical properties of alloys**

| Proof stress of non-proportional elongation, $R_{p0,2}$<br>MPa<br>min. | Percentage elongation after fracture<br>min. |
|---|---|
| 250 | 2 |

#### 4.3.2 Ceramic

The flexural strength and the chemical solubility of the fired ceramics shall meet the requirements of table 2. The porosity of the fired ceramic shall meet the following requirement: there shall be not more than 16 pores of a diameter greater than 30 µm in any area of 1 mm diameter.

The coefficient of linear thermal expansion of the ceramics shall be within 0,5 × $10^{-6}$ $K^{-1}$ of the value stated by the manufacturer [see 8.2.3 d)].

The glass transition temperature of the ceramics shall be within 10 °C of the value stated by the manufacturer [see 8.2.3 e)].

Testing shall be carried out in accordance with 6.2.1, 6.3.1 and 6.3.2 respectively.

**Table 2 — Properties of ceramics**

| | Flexural strength<br>MPa<br>min. | Chemical solubility, loss in mass<br>%<br>max. |
|---|---|---|
| Opaque | 50 | — |
| Dentine | 50 | 0,05 |
| Enamel | 50 | 0,05 |

#### 4.3.3 Metallo-ceramic system

The alloy and a specified (named) ceramic shall pass the test when assessed according to 6.3.3.4. The ceramic and a specified (named) alloy shall pass the test when assessed according to 6.3.3.4.

Testing shall be carried out in accordance with 6.3.3.

NOTE 1 The measured values for coefficients of linear thermal expansion are compared with the manufacturer's values as a means of quality control, but the values do not themselves provide an assurance that the alloy and ceramic are compatible.

## 5 Sampling

### 5.1 Alloy

All of the alloy procured for testing in accordance with this International Standard shall be unused and obtained from the same batch.

### 5.2 Ceramic

At least 50 g of one shade of each of opaque, dentine, enamel ceramics and the appropriate modelling liquids shall be obtained for testing in accordance with this International Standard.

## 6 Test methods

### 6.1 Alloy

#### 6.1.1 Chemical composition

Standard analytical procedures shall be used for determining the composition.

#### 6.1.2 Mechanical properties

**6.1.2.1 Preparation of test specimens**

Prepare six specimens as shown in figure 1 or figure 2 by the "lost wax" process of investment casting generally employed in a dental laboratory. It is suggested that the specimens should be cast using the sprue system shown in figure 3, but other sprueing is also acceptable. Follow the manufacturer's instructions for processing the alloy and for using the necessary aids and casting equipment. After having carefully separated sprues and any nodules, fins, etc., clean the cast specimens. Discard any specimens with visible shrinkage defects or porosities.

Place the specimens on a tray in a furnace at 950 °C for 10 min and then remove them from the furnace, place them on a refractory plate, and allow them to cool. Use these specimens for the tests in 6.1.2.2 and 6.1.2.3.

**6.1.2.2 Proof stress of non-proportional elongation**

Determine the proof stress of non-proportional elongation in accordance with ISO 6892 on test specimens, cast and conditioned in accordance with 6.1.2.1. Load the test specimens in a tensile tester at a cross-head speed of 1,5 mm/min ± 0,5 mm/min up to the fracture point of the specimens. Determine the values from the resultant stress-strain curves at the 0,2 % offset level and calculate the proof stress on the basis of the original cross-sectional area.

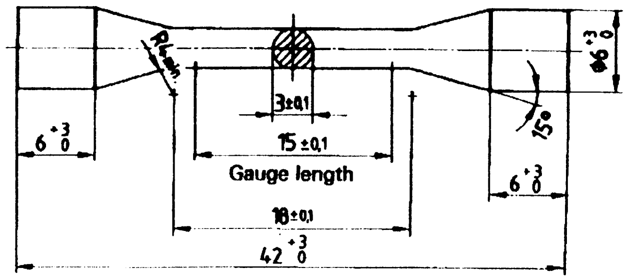
Calculate the value for proof stress as the mean of the values from those four, five or six specimens which are found to comply with the requirements in table 1. If less than four specimens comply with the requirements specified in table 1, reject the alloy.

Report the mean value for the proof stress of non-proportional elongation to the nearest 5 MPa.

**6.1.2.3 Percentage elongation after fracture**

Determine the percentage elongation after fracture in accordance with ISO 6892.

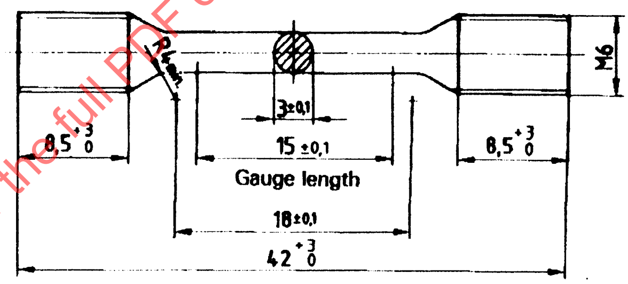
Dimensions in millimetres




NOTE — The cylindrical end length and the presence/absence of threads are given for guidance.

**Figure 1 — Test specimen with cylindrical ends**

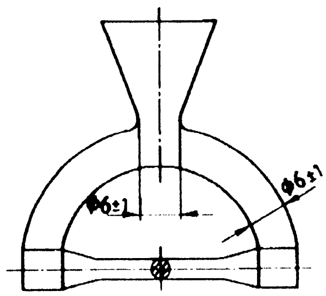
Dimensions in millimetres




NOTE — The cylindrical end length and the presence/absence of threads are given for guidance.

**Figure 2 — Test specimen with threaded ends**

Dimensions in millimetres



NOTE — The sprues can have the shape of a bow or a triangle or a "U".

**Figure 3 — Test specimen with suggested sprues and sprue button**

Calculate the value for elongation as the mean of the value from those four, five or six specimens which are found to comply with the requirements in table 1. If less than four specimens comply with the requirements specified in table 1, reject the alloy.

Report the mean value for the percentage elongation after fracture to the nearest 1 %.

#### 6.1.3 Solidus and liquidus temperature

Determine the solidus and liquidus temperatures by the cooling curve method or other standard procedure of equivalent accuracy.

### 6.2 Ceramic

#### 6.2.1 Preparation of test specimens

Prepare the specimens for testing of flexural strength, chemical solubility and fired porosity according to ISO 6872. When water is required for mixing ceramics, it shall comply with grade 3 of ISO 3696.

#### 6.2.2 Test methods

Test for flexural strength, chemical solubility and fired porosity in accordance with ISO 6872.

#### 6.2.3 Assessment of results

##### 6.2.3.1 Flexural strength

Calculate and record the mean of the 10 results. Results which deviate from the calculated mean by more than ± 25 % should be disregarded and a new mean calculated. If more than three specimens fall below the relevant limit in table 2, prepare a new set of specimens and repeat the test.

##### 6.2.3.2 Chemical solubility

The percentage loss in mass of the specimens (see ISO 6872) shall not exceed the limits stated in table 2.

##### 6.2.3.3 Fired porosity

All three specimens (see ISO 6872) shall meet the requirements for porosity in 4.3.2.

### 6.3 Metallo-ceramic system

#### 6.3.1 Linear thermal expansion

##### 6.3.1.1 Apparatus

a) dental alloy casting equipment;

b) dental ceramic furnace;

c) calibrated dilatometer.

##### 6.3.1.2 Preparation of test specimens

Follow the manufacturer's instructions and prepare two alloy specimens and four opaque, four dentine and four enamel ceramic specimens. Prepare the specimens either as rods or bars of not more than 30 mm$^2$ cross-sectional area. Grind the ends of the specimens flat and parallel.

##### 6.3.1.3 Procedure for alloy

Heat-treat the two cast alloy specimens according to 6.1.2.1. Make dilatometric measurements on each of the two alloy specimens at a heating rate not greater than 5 °C/min up to 550 °C. Determine the coefficient $\alpha_{(25\ °C\ to\ 500\ °C)}$ of linear thermal expansion between 25 °C and 500 °C for each specimen from plotted curves or recorded values of expansion versus temperature.

##### 6.3.1.4 Procedure for ceramic

Prepare the four opaque specimens and the four dentine and enamel specimens by firing two specimens of each type of ceramic once under vacuum and once at atmospheric pressure in air and the other two specimens of each type three times under vacuum and once at atmospheric pressure in air in accordance with the manufacturer's instructions. Make a dilatometric measurement on each of the ceramic specimens at a heating rate of 5 °C/min up to the dilatometric softening point. Determine for each specimen the coefficient of linear thermal expansion between 25 °C and 500 °C (or between 25 °C and the glass transition temperature if that temperature is lower than 500 °C) from plotted curves or recorded values of expansion versus temperature.

##### 6.3.1.5 Assessment of results

Calculate a mean value $\alpha_{(25\ °C\ to\ 500\ °C)}$ for the coefficient of linear thermal expansion from 25 °C to 500 °C for the alloy and from 25 °C to 500 °C (or from 25 °C up to the glass transition temperature, if that temperature is lower than 500 °C) for the ceramic specimens fired twice and four times respectively. Report the mean coefficient of linear thermal expansion in $10^{-6}$ K$^{-1}$, rounded to the nearest 0,1 × $10^{-6}$ K$^{-1}$.

#### 6.3.2 Glass transition temperature

##### 6.3.2.1 Procedure

Determine graphically the glass transition temperature for each ceramic specimen from plotted curves of expansion versus temperature, prepared according to 6.3.1.4.

##### 6.3.2.2 Assessment of results

Calculate the mean glass transition temperatures, in degrees Celsius, for opaque, dentine and enamel ceramics fired twice and four times.

#### 6.3.3 Metallo-ceramic bond characterization

##### 6.3.3.1 Apparatus

a) dental casting equipment;

b) dental ceramic furnace;

c) steel rod of 10 mm diameter;

d) microscope of 10× magnification.

##### 6.3.3.2 Preparation of test specimens

Observing the manufacturer's instructions,

a) cast six alloy specimens 20 mm × 5 mm × 0,4 mm;

b) condition the alloy specimens;

c) apply to one of the 20 mm × 5 mm surfaces of each alloy specimen a thin wash of opaque ceramic and fire it;

d) add opaque ceramic to the previously treated surface of each specimen to a total thickness of approximately 0,2 mm and fire it;

e) add body ceramic to each specimen to form a total ceramic thickness of approximately 1 mm and fire it; and

f) submit each specimen to a glaze firing.

##### 6.3.3.3 Procedure

Bend the fired specimens on the 10 mm diameter rod, with the ceramic located at the opposite side of the contacting area, to a 90° angle of the specimen ends and then straighten them. Before and after straightening of the specimens, remove by prying off any partly detached but retained sections (as determined at 1× magnification) of ceramics and examine the specimens in a light microscope at a maximum magnification of 100×. Quantify the area fraction of the middle third of the specimen, expressed in percent, of retained ceramic by means of a standard quantitative microscopy technique or any other method of equivalent accuracy.

##### 6.3.3.4 Assessment of results

If more than 50 % of the ceramic is retained in the middle third of four or more of the six specimens, the ceramic-alloy system passes the test.

## 7 Manufacturer's instructions

Detailed dated instructions for casting and brazing the alloy and for preparing the surface to obtain satisfactory bonding to specified (named) ceramic materials shall be supplied by the distributor of the alloy.

Evidence has shown that carbon may present problems in bonding porcelain to palladium-based alloys. Specific instructions shall be supplied on how to avoid contamination of the alloy with carbon during casting.

Detailed dated instructions for the application and treatment of ceramic materials, together with the manufacturer's recommendation of at least one (named) casting alloy which can provide satisfactory bonding to the ceramic material considered, shall be supplied by the distributor of the ceramic.

## 8 Packaging and marking

### 8.1 Packaging

The dental ceramic powder shall be supplied in sealed containers that do not contaminate or permit contamination of the contents.

### 8.2 Marking

#### 8.2.1 Alloy and ceramic

Each container shall be marked with:

a) a serial number or combination of letters and numbers which refer to the manufacturer's records for the particular lot or batch of alloy or ceramic;

b) the minimum net mass in grams of the contents.

#### 8.2.2 Alloy

The ingots shall be clearly marked to identify the alloy and the manufacturer or supplier. For alloys supplied as small and/or irregular particles which cannot be marked, this requirement does not apply although the properties and characteristics in sections a) through j) below shall be satisfied.