International Standard 6872

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Dental ceramic

Céramique dentaire

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Descriptors : dentistry, dental materials, ceramics definitions, material specifications, physical properties, chemical properties, test specimen conditioning, sampling, inspections, test equipment, marking.

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 6872 was developed by Technical Committee ISO/TC 106, *Dentistry*, and was circulated to the member bodies in April 1982.

It has been approved by the member bodies of the following countries :

Australia	India	Sweden
Belgium	Ireland	Switzerland
Canada	Netherlands	United Kingdom
Egypt, Arab Rep. of	Norway	USA
France	Romania	USSR
Germany, F.R.	South Africa, Rep. of	

No member body expressed disapproval of the document.

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Dental ceramic

1 Scope and field of application

This International Standard specifies the requirements and the corresponding methods of test for dental ceramics to be used for ceramic jacket-crown and inlay restorations.

2 References

ISO 3696, *Water for laboratory use — Specifications.*[1)]

ISO/TR 7405, *Biological testing of dental materials.*

WHO, *Specifications for the quality control of pharmaceutical preparations (second edition of the International Pharmacopoeia).*

3 Definitions

For the purposes of this International Standard the following definitions apply :

3.1 air-firing ceramic : A dental ceramic to be fired under ambient atmospheric pressure.

3.2 aluminous core ceramic : A dental opaque ceramic, the composition of which is significantly enriched with crystalline alpha-alumina in order to increase its strength.

3.3 clinical crown : That portion of a tooth not covered by the supporting tissues.

3.4 condensation : Any process by which an unfired dental ceramic is compacted.

3.5 core ceramic : An opaque dental ceramic which provides a mechanically strong and suitably coloured base for dentine ceramic.

3.6 dental ceramic : A ceramic material used for dental prosthesis and restorations.

3.7 dentine/ceramic : A slightly translucent, pigmented dental ceramic used to give the overall shape and basic colour of a ceramic restoration or prosthesis.

3.8 degree of fusion :

a) **opaque (core)** : The fusion attained when firing shrinkage is just complete.

b) **medium glaze** : The fusion attained when the glaze is clinically and aesthetically acceptable.

3.9 enamel ceramic : A translucent, lightly-pigmented dental ceramic used on a base of dentine ceramic to simulate the natural tooth enamel.

3.10 fusion range :

a) Low-fusing range < 1 050 °C

b) Medium-fusing range 1 050 to 1 200 °C

c) High-fusing range > 1 200 °C

3.11 lot : Dental ceramic powder of the same class and type, but not necessarily of the same shade [see 9.2 a)], from one manufacturer and submitted at any one time for inspection and testing.

3.12 modelling fluid : A liquid (other than water) with which a dental ceramic may be mixed prior to condensation.

3.13 pyroplasticity; (melt viscosity) : Plastic deformation by gravitational and/or surface tension forces at elevated temperatures.

3.14 type : The intended use of a dental ceramic powder.

3.15 vacuum-firing ceramic : A dental ceramic which is specially formulated for firing at much less than atmospheric pressure.

1) At present at the stage of draft.

4 Types and their identification

Dental ceramics shall be divided into types according to their intended use and, if a colour coding system is used to identify the type of a powder, the appropriate colour given in table 1 below shall be used :

Table 1 — Colours for dental ceramics

Type	Colour
Core ceramic	Yellow or none
Dentine/body ceramics	Pink
Enamel ceramics	Blue
Neck material	Green
Transparent material	None
Concentrate material	None
Add-on material	None
Glaze material	None

5 Requirements

5.1 Uniformity

The inorganic pigment(s) used to produce the fired colour of a dental ceramic and any dye(s) shall be uniformly dispersed throughout the dental ceramic powder, and no segregation of the pigment(s) shall take place when the powder is mixed as in 8.1.4.

5.2 Freedom from extraneous materials, irritants and toxic ingredients

5.2.1 Dental ceramic powders shall be free from extraneous materials.

5.2.2 When a dental ceramic is used in accordance with the manufacturer's instructions, the concentration in the fired ceramic of any irritant or toxic ingredient shall not be high enough to cause prolonged irritation or a toxic reaction, when tested according to ISO/TR 7405.

NOTE — It is envisaged that a future revision of this International Standard (ISO 6872) will disallow the addition of radioactive material to the powders.

5.3 Mixing and condensation properties

When mixed as in 8.1.4 with water or a suitable modelling fluid, a dental ceramic powder shall not form lumps or granules.

The paste so formed shall be suitable for the hand-building (by conventional dental laboratory techniques) of a jacket-crown or inlay restoration, and when the paste is condensed as in 8.1.5 it shall not crack or crumble during the drying cycle recommended by the manufacturer.

5.4 Physical and chemical properties

5.4.1 The physical and chemical properties of ceramic test specimens, prepared from the powder and tested in accordance with the relevant methods detailed in clause 8, shall comply with the requirements specified in table 2.

5.4.2 Fired porosity : there shall not be more than 16 pores of a diameter greater than 30 µm in any area of 1 mm diameter, and no more than 6 of those pores shall be between 40 and 150 µm in diameter. There shall be no pores of diameter greater than 150 µm.

5.5 Manufacturer's instructions

When so required by the purchaser, the dental ceramic powder shall be accompanied by instructions (supplied by the manufacturer) that include the following information :

a) a time-temperature cycle for drying the condensed ceramic;

b) a time-temperature cycle for the firing schedule (including the final temperature, the time it should be held, and the rate of heating) and, in the case of a vacuum-fired ceramic, the level, duration and time of application of vacuum;

Table 2 — Physical and chemical properties

Property	Requirement		
	Type		
	Core	Dentine/body	Enamel
Volumetric firing shrinkage, %, max.	40	40	40
Linear firing shrinkage, %, max.	16	16	16
Flexural strength, N/mm^2, min.	100	55	50
Resistance to pyroplastic flow :			
a) change in height after 2 min, %	−4 to 0	2 to 8	2 to 8
b) change in height after 16 min, %	−4 to 1	10 to 19	10 to 19
Chemical solubility : loss in mass, %, max.	0,5	0,05	0,05
Resistance to staining : visible stains on any surface	Nil	Nil	Nil

c) the glazing temperature;

d) powder/liquid ratio;

e) linear firing shrinkage, expressed as a percentage;

f) a warning regarding the potential health hazards presented by prolonged exposure to dusts containing a high concentration of quartz, cristobalite or other siliceous material(s), or radioactive material;

g) if the powder contains radioactive material, a warning to the effect that the powder must not be swallowed or come into contact with the mouth.

6 Sampling

The following samples shall be obtained :

a) Where there is more than one shade in a type of dental ceramic, take one 50 g sample of each shade.

b) Where there is only one shade of a type of dental ceramic take three 50 g samples.

Sufficient quantities of essential modelling fluids should be obtained, if their use is recommended by the manufacturers. The quantities shall be those recommended by the manufacturer concerned. If the shades of a type of ceramic comply with the requirements of clause 4, sub-clauses 5.1 and 5.2, form a pool of powder of that type by taking samples of equal mass from each shade using the grid type sample divider principle described in the annex. The total mass of the pool of powder shall be 100 g.

7 Inspection

Use visual inspection to assess compliance of each sample, taken in accordance with clause 6, with the requirements of clause 4, sub-clauses 5.1 and 5.2.1, that are not checked during testing (see 8.1.4 and 8.1.5).

8 Test methods

8.1 Preparation of test specimens

NOTE — Unless otherwise stated or inconsistent with the text, the apparatus detailed in 8.1.3 and 8.1.5.1 and the conditions for mixing, condensation and firing apply to all test methods.

8.1.1 Ambient conditions

All mixing of the ceramic pastes during the preparation of test specimens and all testing shall be carried out at 23 ± 2 °C.

NOTE — No special precautions need to be taken to control the humidity.

8.1.2 Components of test specimens

8.1.2.1 The liquid used in the preparation of test specimens shall be water that complies with the relevant requirements for Grade 5 water, ISO 3696 or, when applicable, the modelling fluid recommended by the manufacturer of the dental ceramic powder.

8.1.2.2 The required amount of powder shall be taken from the appropriate sample by means of the grid-type sample divider (see figure 5 in the annex).

8.1.3 Apparatus for mixing

All apparatus used for mixing shall be clean and dry.

The following apparatus shall be used for mixing dental porcelain powder with water or a modelling fluid :

8.1.3.1 Glass slab or **mixing palette.**

8.1.3.2 Spatula, made from a material (other than a metal) that is not readily abraded by the dental porcelain powder (glass is recommended).

8.1.4 Method of mixing

Combine the mixing liquid and the ceramic powder in the proportions recommended by the manufacturer. Avoid vigorous mixing which will tend to incorporate air bubbles with the paste and, both during and after mixing, examine for compliance with 5.1 and 5.2.1.

8.1.5 Condensation

8.1.5.1 Apparatus

8.1.5.1.1 Open multipart mould from which the condensed specimen may be removed without distortion.

8.1.5.1.2 Vibration system

A suitable system may consist of

a) a 50 to 60 Hz sine-wave generator with a variable output amplitude;

b) a transducer (see figure 1); and

c) a mould-carrier (see figure 1) that is pivoted at one end and is so spring-loaded that the mould is subjected to an impact in each cycle of vibration.

8.1.5.2 Procedure

Attach the mould (8.1.5.1.1) firmly to the mould carrier [8.1.5.1.2 c)], over-fill it with dental ceramic paste, and vibrate. When excess liquid appears at the free surface of the specimen, place a paper tissue (or similar absorbent material) on the surface of the specimen, and remove the excess liquid continually

by replacing the tissue as soon as it becomes saturated with liquid. Continue vibration and absorption until no further liquid can be removed, and then level the free surface of the condensed specimen by means of a suitable instrument (a bevelled glass microscope slide is ideal for this purpose). After removing the specimen from the mould, place it on a firing tray, dry it in accordance with the manufacturer's instructions [see 5.5 a)], and check for compliance with 5.3. Clean and dry the mould before reassembling it for further use.

8.1.6 Firing

Position the specimens in the furnace so that they will be uniformly fired, and on a substrate to which they will not adhere and from which there will be no material pick-up. Unless otherwise specified in this International Standard, fire the ceramic specimens according to the manufacturer's instructions for the first build-up firing of the material (i.e. without glaze firing).

8.2 Firing shrinkage

8.2.1 Apparatus

8.2.1.1 Mould as shown in figure 2 and having a cavity of nominal size 25,0 mm × 6,0 mm × 3,0 mm.

8.2.1.2 Hand micrometer accurate to 0,01 mm.

8.2.1.3 Balance (accurate to 0,1 mg) together with a balance straddle, a 250 ml glass beaker, and fine (0,10 mm diameter) corrosion-resistant wire.

8.2.2 Preparation of test specimens

Use 10 g of the pool of powder (see clause 6) and the mould (8.2.1.1) and prepare 10 specimens as described in 8.1, and fire each specimen according to 8.1.6.

8.2.3 Procedure

Determine, to an accuracy of ± 0,01 mm, the dimensions of the mould cavity and the length (L_s) of each fired specimen.

Determine the mass of each fired specimen suspended

a) in air (m_1); and

b) in water (m_2) that is in equilibrium with the ambient temperature (i.e. 23 ± 2 °C).

Retain the specimens for the test described in 8.4.

8.2.4 Calculation and expression of results

Calculate, using the relevant formula,

a) the volume, V_m, of the mould cavity as follows :

$$V_m = L_m \times m_m \times D_m$$

where

L_m is the length of the cavity, in millimetres;

m_m is the width of the cavity, in millimetres;

D_m is the depth of the cavity, in millimetres.

b) the volume, V_s, of each fired specimen as follows :

$$V_s = \frac{(m_1 - m_2)}{\varrho}$$

where

ϱ is the density of water at the temperature of the test;
m_1 and m_2 are as defined in 8.2.3.

NOTE — Tables for density of water are given in ISO 649/2.

c) the volumetric firing shrinkage, VS, of each specimen, expressed as a percentage, as

$$VS = \frac{(V_m - V_s)}{V_m} \times 100$$

d) the average volumetric firing shrinkage, i.e. the mean of the individual results obtained in c) above. Discard any individual value that differs by more than 10 % from the mean, and ensure that the final test result is the mean of the results on at least eight specimens by, when necessary, testing additional specimens.

e) the linear firing shrinkage, LS, of each specimen, expressed as a percentage, as follows :

$$LS = \frac{(L_m - L_s)}{L_m} \times 100$$

f) the average linear firing shrinkage, i.e. the mean of the individual results obtained in e) above.

8.3 Fired porosity

8.3.1 Apparatus

8.3.1.1 Equipment for the production, by grinding, of flat, parallel-sided specimens.

8.3.1.2 Mould as shown in figure 3 and having a circular cavity of diameter 16,0 ± 0,2 mm and depth 1,6 ± 0,1 mm.

8.3.1.3 Optical microscope.

8.3.1.4 Equipment for the preparation of polished sections.

8.3.2 Preparation of test specimens

Take a 5 g sample from the pool of powder (see clause 6). Using the mould (8.3.1.2), prepare three specimens fired as described in 8.1.6.

Prepare a polished section of each specimen, the section being approximately parallel to, and approximately midway between the flat faces of the specimen.

8.3.3 Procedure

View under the microscope (8.3.1.3) the three specimens. Count on a surface of approximately 1 mm in diameter the pores between 30 and 40 µm in cross-section and the pores larger than 40 µm in cross-section. For example a photomicrograph with a final enlargement of X 100 can be used and the counted pores marked.

8.4 Flexural strength

8.4.1 Apparatus

8.4.1.1 Equipment for the production, by grinding, of flat parallel-sided specimens.

8.4.1.2 Flexural strength testing machine, having a span between bearers of 12 to 15 mm, bearing surfaces of diameter 1,6 mm, and capable of a rate of application of force not exceeding 0,5 N/s.

8.4.2 Preparation of test specimens

Dry the 10 specimens prepared according to 8.2.2, and grind each specimen so as to produce a rectangular test piece of width 5,0 ± 0,25 mm, thickness 1,0 ± 0,05 mm and length at least 20 mm. Then grind the surfaces of the test piece on loose, wet 320 grit silicon carbide powder on a glass plate. Ensure that opposing faces of the test pieces are flat and parallel to within 0,01 mm.

Thoroughly clean the test pieces, ensuring that all traces of grinding debris are removed, and then fire them to a medium glaze degree of fusion. In the case of a core type ceramic, fire the ground test pieces to the manufacturer's recommended firing schedule for dentine ceramics as in 8.1.6, before submitting them to the glaze firing.

8.4.3 Procedure

Measure the cross-section dimensions of each test piece to the nearest 0,01 mm. Then place a test piece centrally on the bearers of the testing machine so that the load is applied to a 5 mm wide face along a line perpendicular to the long axis of the test piece, and determine, to the nearest 0,1 N, the load required to break the test piece. Repeat the procedure with the remaining test pieces retaining one of the broken pieces for the test described in 8.7.

8.4.4 Calculation and expression of results

Calculate the flexural strength, M, in newtons per square millimetre[1], of each test piece from the formula :

$$M = \frac{3Wl}{2bd^2}$$

where

W is the breaking load in newtons;

l is the test span (centre-to-centre distance between bearers), in millimetres;

b is the width of the specimen, i.e. the dimension of the side at right angles to the direction of the applied load, in millimetres;

d is the thickness of the specimen, i.e. the dimension of the side parallel to the direction of the applied load, in millimetres.

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 fail below the relevant limit in table 2, prepare a new set of specimens and repeat the test.

8.5 Pyroplasticity

8.5.1 Apparatus

8.5.1.1 Mould, as shown in figure 4 and having a circular cavity of diameter 6,0 ± 0,1 mm and depth 4,0 ± 0,1 mm.

8.5.1.2 Pure platinum foil, of thickness 0,025 mm.

8.5.1.3 Hand micrometer, accurate to 0,01 mm.

8.5.2 Preparation of test specimens

Using 5 g of the pool of powder (see clause 6) and the mould (8.5.1.1), prepare six specimens as described in 8.1, firing them to the degree specified in 8.1.6. Check visually that the specimens are uniformly fired, that the end faces are flat and parallel, that the cylindrical faces are plane and that no edge is chipped. Replace any specimen that does not comply with these requirements.

Using the hand micrometer (8.5.1.3), measure the height and diameter of each specimen. Replace any specimen whose aspect ratio (i.e. diameter/height) is outside the range 1,45 to 1,55.

8.5.3 Procedure

8.5.3.1 Place the six fired specimens on a flat sheet of the platinum foil (8.5.1.2), leaving a gap of at least 3 mm between specimens. Fire the specimens to the ceramic manufacturer's recommended final firing temperature, using the recommended rate of rise of temperature. Maintain the final temperature for 2 min, then allow the specimens to cool to room temperature, and measure and record the height of each specimen.

1) 1 N/mm^2 = 1 MPa

8.5.3.2 Refire the specimens as in (8.5.3.1) but maintain the final firing temperature for 14 min. Allow to cool and again measure and record the height of each specimen.

NOTE — If the specimens adhere to the platinum foil, measure their heights *in situ* (i.e. including the platinum foil), and obtain the actual specimen heights by subtracting the thickness of the foil.

8.5.4 Calculation and expression of results

Calculate, for each heating period, the percentage change in height of each specimen and the average of each set of six results.

8.6 Chemical solubility

8.6.1 Reagent

Acetic acid (analytical grade), 4 % (*V*/*V*) solution in distilled or demineralized water.

8.6.2 Apparatus

8.6.2.1 Mould, as specified in 8.3.1.2.

8.6.2.2 Reflux-condenser-type extraction apparatus, consisting of :

a) a 50 ml flask;

b) a 200 mm Allihn or 140 mm Davies double-surface condenser;

c) a 20 ml extractor;

d) **clean, dry sintered glass thimble** (porosity grade 2) 20 mm × 55 mm.

8.6.2.3 Balance accurate to 0,1 mg.

8.6.2.4 Drying oven, capable of being controlled at 150 ± 3 °C.

8.6.3 Preparation of test specimens

Using 10 g of the pool of powder (see clause 6) and the mould (8.6.2.1), prepare specimens (with an aggregate mass of at least 5 g) as described in 8.1, firing them according to the manufacturers instructions for the first build-up firing and the glaze firing of the material.

8.6.4 Procedure

Place the specimens in the sintered glass-bottomed thimble [8.6.2.2 d)] and weigh them to the nearest 0,1 mg. Place the thimble in the extraction apparatus (8.6.2.2), and extract the specimens with the 4 % acetic acid solution (8.6.1) by refluxing for 16 h. (Reflux rate 15 to 20 min cycles.)

Wash the specimens (in the thimble) with distilled or demineralized water, dry them at 150 ± 3 °C for 4 h, and reweigh the specimens and thimble.

8.6.5 Calculation and expression of results

Calculate the percentage loss in mass of the specimens.

8.7 Resistance to staining

8.7.1 Staining solution

A saturated solution of methylene blue in ethanol [ethanol 95 % (*V*/*V*) (see WHO specification)].

8.7.2 Apparatus

Small vial of length approximately 20 mm and diameter approximately 10 mm.

8.7.3 Preparation of test specimens

Using loose wet 320 grit silicon carbide powder on a glass plate, grind the glaze off one large face of the piece of broken flexural strength specimen retained in terms of 8.4.3.

8.7.4 Procedure

Immerse the specimen [in the vial (8.7.2)] in the staining solution (8.7.1) for 24 h. Then wash it thoroughly by scrubbing with a toothbrush or wash it in an ultrasonic cleaner in clean methylated spirit for 15 s.

Examine all the outer surfaces for staining (without magnification).

9 Packaging, marking, and labelling

9.1 Packaging

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

9.2 Marking and labelling

The following information shall be clearly marked on each container or on a label securely attached to the container :

a) the shade (as identified in the manufacturer's shade guide);

b) the manufacturer's batch identification;

c) the minimum net mass, in grams, of the contained powder;

d) the manufacturer's name or brand name.

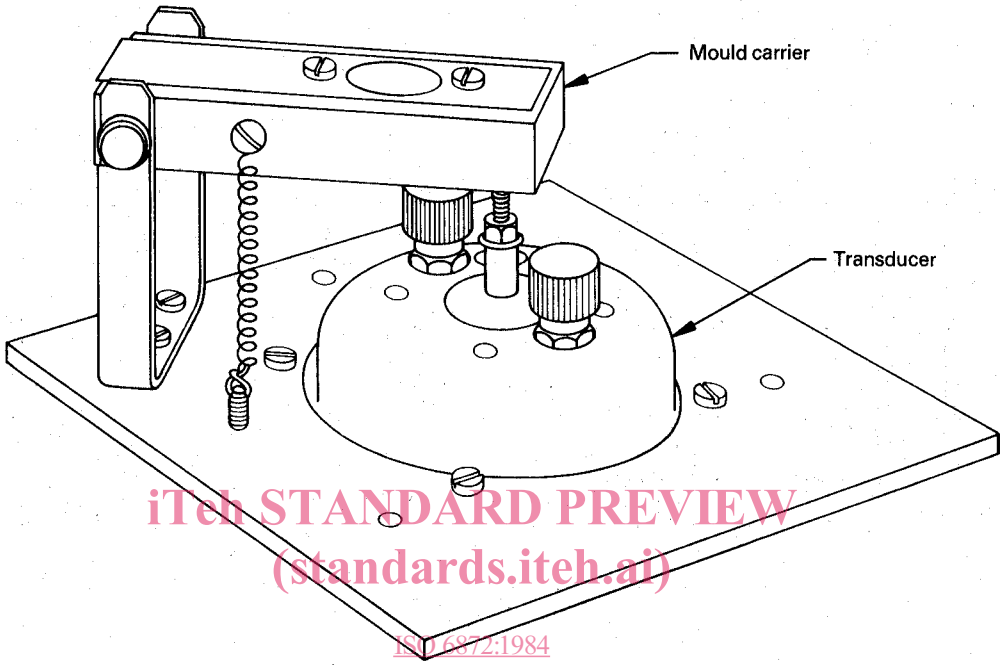

Figure 1 — Mould vibrator assembly