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Dental brazing investments

Revêtements dentaires pour le brasage

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Foreword

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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 11244 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 2, *Prosthetic materials*.

Annex A of this International Standard is for information only.

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Dental brazing investments

1 Scope

This International Standard establishes a classification of and specifies requirements for dental brazing investments. It specifies test methods to be used to determine compliance with these requirements. It lists information which shall be included in the manufacturer's instructions and also gives requirements for labelling.

2 Definitions

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

2.1

brazing

brazing process

process of joining generally applied to metals in which, during or after heating, molten filler metal is drawn into or retained in the space between closely adjacent surfaces of the parts to be joined by capillary attraction

NOTE In general the melting range of the filler metal is above 450 °C, but always below the melting temperature of the parent metal.

2.2

brazing investment

blend of refractory fillers and binding system specially designed to allow the formation of a model on which dental restoration components are held in place while they are being joined by brazing

2.3

gypsum-bonded dental brazing investment

refractory filler system and binding system consisting essentially of calcium sulfate hemihydrate, specially designed for use when brazing dental alloy restorations

2.4

phosphate-bonded dental brazing investment

refractory filler system and binding system consisting essentially of an acidic phosphate (such as monoammonium phosphate) and a basic oxide (such as magnesium oxide) specially designed for use when brazing dental alloy restorations

2.5

special liquid

liquid, consisting mainly of a suspension of colloidal silica particles in water, made available by the manufacturer or supplier for mixing with the brazing investment powder for the purpose of increasing thermal expansion

3 Classification

Dental brazing investments are classified into two types according to their composition.

- **Type 1:** Gypsum-bonded dental brazing investments.
- **Type 2:** Phosphate-bonded dental brazing investments.

Unless the manufacturer states otherwise, Type 1 shall be used with brazing alloys having brazing temperatures below 1000 °C and Type 2 with those having brazing temperatures above 1000 °C.

4 Requirements

4.1 Quality

The powder shall be a uniform and dry mixture, free from foreign matter and lumps. Test in accordance with 6.1.

4.2 Fluidity

The average diameter of the base of the set investment shall be not more than 100 mm. Test in accordance with 6.2.

4.3 Setting time

The setting time shall not differ by more than 30 % from the setting time stated by the manufacturer. If the manufacturer gives a range of setting time, then the measured setting time shall not differ from the midpoint of this range by more than 30 %. Test in accordance with 6.3.

4.4 Compressive strength

The compressive strength shall fall within the range of 2,0 MPa to 10,0 MPa. Test in accordance with 6.4.

4.5 Linear thermal expansion

The linear thermal expansion shall not differ by more than 15 % from the value stated by the manufacturer. If the manufacturer gives a range of linear thermal expansion, then the linear thermal expansion shall not differ from the midpoint of this range by more than 15 %. Test in accordance with 6.5.

4.6 Linear setting expansion

For Type 1 materials, the linear setting expansion shall be within 15 % of the value stated by the manufacturer. If the manufacturer gives a range of linear setting expansion, then the linear setting expansion shall not differ from the midpoint of this range by more than 15 %. Test in accordance with 6.6.

5 Sampling, test conditions and mixing

5.1 Sampling

Sufficient retail packages of the material of one lot shall be obtained to provide at least 5 kg of material. Any packages that are not sealed shall be discarded.

When the powder is supplied in bulk, it shall be thoroughly blended and stored in a moisture-proof container.

All specimens shall be prepared using the same lot of powder.

5.2 Test conditions

All mixing and testing shall be carried out at $(23 \pm 2) ^\circ\text{C}$ and $(50 \pm 10) \%$ relative humidity in a room shielded from obvious drafts. Prior to mixing, all test materials shall be stored for at least 16 h under these conditions.

All equipment shall be clean and at the test temperature. The bowls used for mixing shall be moistened and then shaken free of any excess water before being used. All other equipment shall be dry.

5.3 Mixing

5.3.1 Apparatus

It is recommended that separate sets of mixing bowls, tools and material contacting parts be used for Type 1 and Type 2 dental brazing investments. If the manufacturer recommends an apparatus for mechanical mixing, it shall be used.

5.3.2 Procedure

Measure to an accuracy of $\pm 1\%$ the required amount of powder and the required volume of water or special liquid in the mixture ratio recommended by the manufacturer. The water should meet the requirements of ISO 3696 (grade 3). If the manufacturer specifies a range of volumes, use the mid-point value. Pour the water or special liquid into the mixing bowl and sift the investment powder into the water or liquid within 10 s, minimizing entrapment of air. Begin timing from the moment the investment powder and the water or liquid first make contact. Hand spatulate with a speed of approximately two turns per second for 15 s. Mechanically mix according to the manufacturer's recommended mixing procedure [item 7 d)] for the time specified by the manufacturer and then transfer the mixed investment to the test moulds or form within 15 s.

6 Test methods

6.1 Visual inspection

The material shall be examined by eyesight with normal acuity and shall meet the quality requirement in 4.1.

6.2 Fluidity

6.2.1 Apparatus

6.2.1.1 Clean, dry cylindrical mould, having a length of (50 ± 1) mm and an inside diameter of (35 ± 1) mm constructed from a corrosion-resistant, non-absorbent material.

6.2.1.2 Flat, square glass plate of dimensions at least 150 mm \times 150 mm.

6.2.1.3 Dental vibrator operating on 50 Hz or 60 Hz power supply.

6.2.1.4 Measuring scale, graduated in millimetres.

6.2.1.5 Mould release agent, such as silicone spray or silicone grease.

6.2.2 Procedure

Coat the inside surface of the mould with mould release agent. Mix the investment as described in 5.3 using 200 g of powder and the recommended amount of liquid. Centre the mould base on the glass plate and place it on the dental vibrator platform. Vibrate the investment mix into the mould until it is slightly overfilled. Do not vibrate for more than 20 s. Level the mix flush with the top of the mould. At 20 s after the end of mixing, lift the mould from the plate vertically at a rate of approximately 10 mm/s, allowing the mix to slump on the plate. As soon as the mixed investment has set, measure with the scale the largest and smallest horizontal dimensions of the base of the set investment, and report the average value.

6.2.3 Evaluation

Make two such tests as described in 6.2.2. If the results of both tests meet the requirement (4.2), the product complies with this requirement of this International Standard. If neither meets the requirement, then the product fails to comply. If one test result meets the requirement and one fails the requirement, then the test shall be repeated three more times. If all three test results meet the requirement (4.2), then the product complies with this requirement of this International Standard. Otherwise it fails to comply.

6.3 Setting time

6.3.1 Apparatus

6.3.1.1 Needle apparatus, as shown in figure 1, meeting the following requirements.

- Total mass of the rod and needle (A, B and C in figure 1) shall be (300 ± 1) g.
- Scale (D), graduated in millimetres.
- Baseplate (H) of plate glass, measuring about $100 \text{ mm} \times 100 \text{ mm}$.
- Vicat needle (C) of circular cross-section and with a diameter of $(1,00 \pm 0,05)$ mm and a flat end.

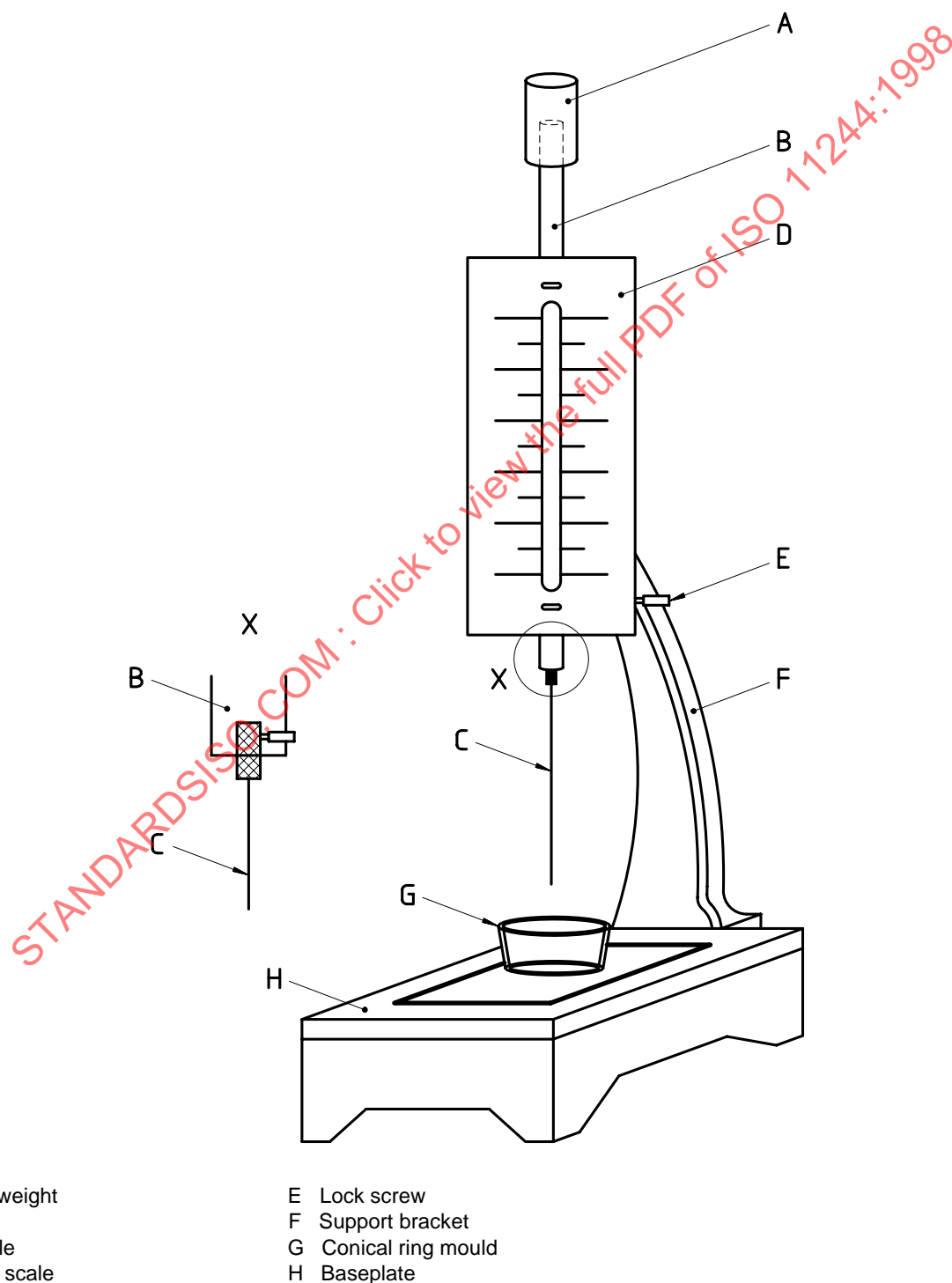


Figure 1 — Apparatus for Vicat needle (see 6.3.1.1)

6.3.1.2 Clean, dry conical mould, constructed from a corrosion-resistant, non-absorbent material, having an inside diameter 70 mm at the top and 60 mm at the base and a height of 40 mm.

6.3.1.3 Mould release agent, such as silicone spray or silicone grease.

6.3.2 Procedure

Adjust the scale of the needle apparatus to read zero (0) when the needle is in contact with the baseplate. Make an investment mix in accordance with 5.3, using 300 g of investment powder and the recommended amount of liquid. Overfill the ring mould with the mix and then level the surface. When the glossy surface of the mix has completely disappeared, lower the needle until it touches the surface and then release it gently, allowing it to sink into the mix under its own mass. Repeat this procedure at 15-s intervals, wiping the needle clean after each penetration and moving the sample at least 5 mm so that the needle does not enter the same place twice. Avoid making any penetration of the needle closer than 5 mm to the mould walls. Record the setting time as the time from beginning mixing until the needle first fails to penetrate to within 5 mm of the mould bottom.

6.3.3 Evaluation

Make two such tests as described in 6.3.2. If the results of both tests meet the requirement (4.3), then the product complies with this requirement of this International Standard. If neither meets the requirement, then the product fails to comply with the requirements of this International Standard. If one test meets the requirement and one fails the requirement, then the test shall be repeated three more times. If all three tests meet the requirement (4.3), then the product complies with this requirement of this International Standard. If any of the three fails to meet the requirement, then the product fails to comply with the requirements of this International Standard.

6.4 Compressive strength

6.4.1 Apparatus

6.4.1.1 One or more moulds, sufficient to produce cylindrical specimens with a diameter of $(20,0 \pm 0,2)$ mm and a length of $(40,0 \pm 0,4)$ mm, constructed from a corrosion-resistant material. Ends of the mould shall be parallel within 0,05 mm.

6.4.1.2 Flat glass plates, sufficient in size and quantity to cover the ends of all moulds.

6.4.1.3 Dental vibrator.

6.4.1.4 Compression-testing machine, adjusted to a rate of loading of (5 ± 2) kN/min.

NOTE When using a testing machine with a constant crosshead rate, this rate should be adjusted so that the average rate of loading between the initial application of the load and the failure of the specimen is (5 ± 2) kN/min. Trial specimens should be run to determine the appropriate crosshead speed.

6.4.1.5 Mould release agent.

6.4.2 Procedure

Lubricate the inside surface of the mould with the mould release agent. Place the mould on the glass plate. Make an investment mix in accordance with 5.3, using 200 g of powder and the recommended amount of liquid. Slightly overfill the mould with the investment mix, applying slight vibration. Before the glossy surface has completely disappeared from the mix, put the second glass plate on the mould and press it down until the glass contacts the mould. Remove the specimen from the mould 30 min after the start of mixing and store it at (23 ± 2) °C and (50 ± 10) % relative humidity until (120 ± 5) min from the start of mixing. Prepare and test five specimens of investment. It may be necessary to use more than one mix to produce five specimens if the fluidity or setting rate of the mix complicates the preparation of specimens. Prior to testing, measure the diameter of each specimen.

Position each specimen between the loading platens of the test machine so that the specimen will be loaded in an axial direction. Do not use packing between specimen and platen. Using the machine (6.4.1.4), commence the application of force at (120 ± 5) min from the beginning of mixing. Apply an increasing force until fracture occurs. Record the fracture force.

6.4.3 Evaluation

If the compressive strengths of at least four of the five specimens are within the range permitted (4.4), the material complies with the requirement of this International Standard. If three of the five specimens are within the permitted range, then a further set of five specimens shall be tested. If all five of these specimens are within the permitted range, then the material complies with this requirement of this International Standard, otherwise it fails to comply.

6.5 Linear thermal expansion

6.5.1 Apparatus

6.5.1.1 Vitreous silica dilatometer, a measuring instrument which exerts a force on the specimen of no more than 5 kPa and which can measure the change in length with a precision of 0,1 %.

6.5.1.2 Mould constructed from a corrosion-resistant material and capable of producing a specimen which is suitable for testing in the dilatometer (6.5.1.1).

6.5.1.3 Recording equipment, such as an X - Y recorder, to permit a record of the thermal expansion curve to be obtained.

6.5.1.4 Mould release agent.

6.5.2 Procedure

Lubricate the inside surface of the mould with the mould release agent, and slightly overfill it using the investment mix. At (58 ± 2) min from the start of mixing, scrape the specimen level with the top of the mould. At (60 ± 1) min from the start of mixing remove the specimen from the mould. At (120 ± 1) min, measure the length of the specimen with an accuracy of 0,01 mm and place the specimen in the dilatometer. Raise the dilatometer temperature at a rate of (5 ± 1) °C/min from room temperature to 750 °C for Type 1 materials and to 900 °C for Type 2 materials. Maintain the maximum temperature for 15 min. Determine the change in length to the nearest 0,01 mm. Calculate the thermal expansion as a percentage of the initial length, to the nearest 0,02 %.

6.5.3 Evaluation

Carry out two tests as described in 6.5.2. If the results of both tests meet the requirement (4.5), then the product complies with this requirement of this International Standard. If neither meets the requirement, then the product fails to comply. If one test result meets the requirement and one fails the requirement, then the test shall be repeated three more times. If all three test results meet the requirement (4.5), then the product complies with this requirement of this International Standard. Otherwise it fails to comply.

6.6 Linear setting expansion

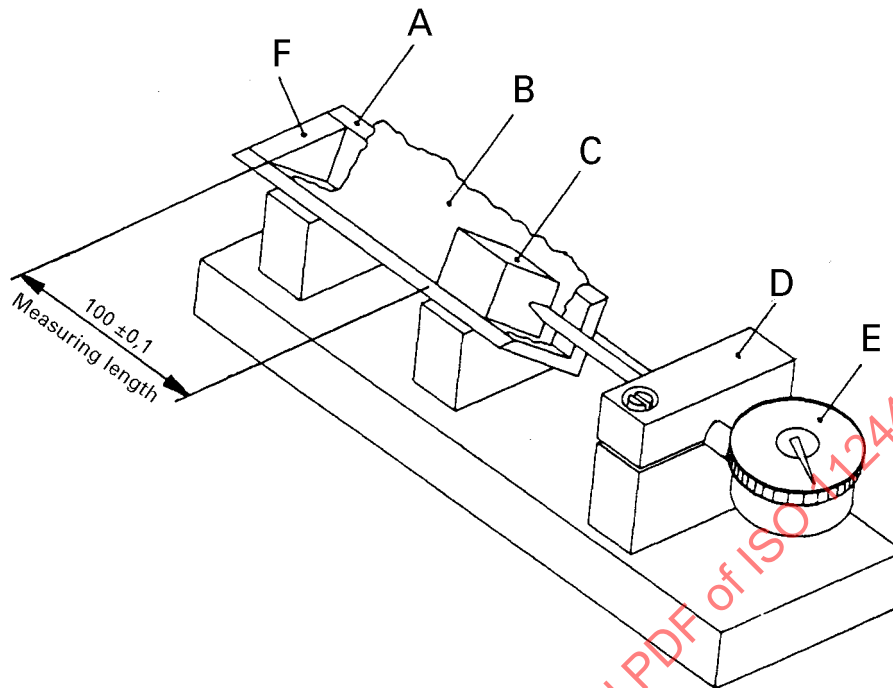
6.6.1 Apparatus and materials

6.6.1.1 Extensometer, as shown in figure 2, producing a specimen with a length of 100 mm.

The apparatus is fitted with a device which measures change in length to within 0,01 mm and exerts a measuring force which is no greater than 0,8 N. The internal cross-section of the trough shall be an isosceles triangle having an angle of 90° and internal sides of length (30 ± 1) mm. One end of the trough shall be blocked with a fixed end-piece and the other by a movable end-piece having a mass of (200 ± 10) g.

On the inside of this trough a horizontal line shall be scribed so as to define a triangle with included sides of length (25 ± 1) mm.

Dimensions in millimetres

**Key**

- A Trough
- B PTFE sheet
- C Movable endpiece
- D Gauge support
- E Dial gauge or equivalent
- F Immovable endpiece

Figure 2 — Example of suitable extensometer

6.6.1.2 Polytetrafluoroethylene (PTFE) sheet, of thickness 0,1 mm to 0,2 mm.

6.6.1.3 Mould release agent.

6.6.2 Procedure

Line the trough with the PTFE sheet (6.6.1.2).

Reset the equipment so that the specimen length will be $(100 \pm 0,5)$ mm.

Apply the mould release agent to the ends of the stop plates that contact the material being tested. Pour the mix (prepared as described in 5.3) into the trough until the investment is level with the scribed line in the trough. Measure the initial length of the specimen. Take the initial reading 1 min prior to the setting time as determined in 6.3. Take the final reading at (120 ± 5) min from the beginning of mixing and determine the change in length to the nearest 0,01 mm.

Calculate the setting expansion as a percentage of the original length, to the nearest 0,01 %.

6.6.3 Evaluation

Perform two tests. If the results of both measurements meet the requirement for linear setting expansion (4.6), then the material complies with the requirement of this International Standard. If neither meets the requirement (4.6), then the material fails to comply. If one measurement meets the requirement and the other one fails to meet the

requirement, carry out three more tests. If the results of all three of these measurements meet the requirement (4.6), then the material complies with this requirement of this International Standard, otherwise it fails to comply.

7 Manufacturer's instructions

The manufacturer's or supplier's instructions accompanying the product shall contain at least the following information:

- a) the type of brazing investment according to clause 3;
- b) recommended liquid-to-powder ratio, expressed as volume in millilitres to mass in grams;
- c) if a special liquid is provided, instructions for its use, storage and dilution;
- d) recommended mixing procedure, including mixer type and mixing time if applicable;
- e) recommended investing technique including, if applicable, duplicating material, time for trimming and burnout procedure.

Information on the following physical properties determined according to this International Standard shall be included:

- a) the specific liquid/powder ratio at which these properties were measured;
- b) setting time;
- c) compressive strength;
- d) linear thermal expansion value;
- e) linear thermal expansion curve;
- f) linear setting expansion value for Type 1 materials.

8 Packaging

The powder shall be packed in tight, moisture-resistant containers which, if appropriate, shall be capable of being resealed.

9 Marking

9.1 Powder

Each container shall be marked with at least the following information:

- a) name or trademark of the manufacturer and/or supplier, and address;
- b) name of the investment material;
- c) description of the investment type, in accordance with clause 3;
- d) lot number;
- e) minimum net mass of the powder, expressed in grams or kilograms; minimum net volume of special liquid;
- f) recommended storage conditions;