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Dental restorations — Phosphate-bonded refractory die materials

Restaurations dentaires — Produits pour modèles réfractaires à liant phosphate

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Contents

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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 3.

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 11245 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 2, *Prosthodontic materials.*

Annex A forms a normative part of this International Standard.

Introduction

During preparation of this International Standard for phosphate-bonded refractory die material, consideration was made to address issues regarding the linear setting expansion requirement. The importance of the setting expansion measurement relative to the performance of a phosphate-bonded refractory die material is recognized. However, measurement of the linear setting expansion with existing apparatus, method and techniques may provide results with a high degree of variation. It is therefore recommended that the test remain in this International Standard as a normative annex until suitable means are available to achieve consistent results in the measurement of linear setting expansion.

Dental restorations — Phosphate-bonded refractory die materials

1 Scope

This International Standard is applicable to phosphate bonded refractory die materials used in the production of dental restorations by a sintering technique.

It specifies requirements for the essential physical properties of the refractory die material and the test methods to be used to determine these properties. It also includes a requirement for adequate instructions to accompany each package.

2 Terms and definitions

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

2.1
refractory die material
powder mixture of a refractory filler system and a binding system specially designed so that when mixed with a liquid it forms a hardened die suitable for the production of dental ceramic restorations using the sintering technique

NOTE The refractory filler system usually consists of refractory oxides such as silica. The binding system usually consists mainly of an acidic phosphate such as ammonium dihydrogenphosphate together with a basic oxide such as magnesium oxide. When the mixture is mixed with a suitable liquid it forms a paste that hardens at room temperature to form a refractory die. The suitable liquid may be the special liquid (2.2), the special liquid diluted with water, or it can be water alone.

2.2
special liquid
liquid made available by the manufacturer or supplier for mixing with the powder mixture

NOTE The special liquid usually consists mainly of a suspension of colloidal silica particles in water.

3 Requirements

3.1 Quality

The powder (2.1) shall be uniform and free from foreign matter and lumps when examined visually. If a special liquid (2.2) is required, it shall be free of sediment.

3.2 Fluidity

The diameter of the base of the set mass shall be at least 70 mm. Test in accordance with 5.1.

3.3 Setting time

The setting time shall not differ by more than 30 % from the time stated by the manufacturer. If the manufacturer gives a range of setting time, then the setting time shall not differ from the midpoint of this range by more than 30 %. Test in accordance with 5.2 and report in accordance with 6 h).

3.4 Compressive strength

The compressive strength of the set refractory die material shall be not less than 13 MPa. Test in accordance with 5.3 and report in accordance with 6 j).

3.5 Linear setting expansion

For the reasons stated in Introduction, the linear setting expansion requirement shall be as specified in clause A.1 of annex A.

3.6 Linear thermal dimensional change

The linear dimensional change after firing (degassing) and the linear thermal expansion shall not differ by more than 15 % from the values stated by the manufacturer. If the manufacturer gives a range of linear thermal change after firing and a range of linear thermal expansion, then the values shall not differ from the midpoint of these ranges by more than 15 %. Test in accordance with 5.5 and report according to 6 k).

4 Sampling, test conditions and mixing

4.1 Sampling

The date when material is tested shall not be later than the expiry date [see 7.1 f)] stated on the package. Sufficient retail packages of the material of one batch shall be obtained to provide at least 3 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.

If a special liquid is recommended by the manufacturer [6 d)], an adequate supply shall be obtained.

4.2 Test conditions

All test specimen preparation shall be carried out in an environment at (23 ± 1) °C and (50 ± 10) % relative humidity. All specimens should remain in this environment until they are ready to be tested.

Testing according to 5.1, 5.2, 5.3, 5.4 and 5.5 shall be carried out at (23 ± 1) °C and (50 ± 10) % relative humidity. All other testing of the die material shall be carried out in a room shielded from obvious draughts and at (23 ± 2) °C and (50 ± 10) % relative humidity.

All test equipment shall be clean, dry and at test temperature. Before testing begins, material shall be held for at least 16 h at the test conditions of temperature and humidity.

4.3 Mixing

4.3.1 Apparatus

4.3.1.1 Clean apparatus for mechanical mixing in vacuum as recommended by the manufacturer and used exclusively for phosphate-bonded materials.

4.3.1.2 Timing device, such as a stopwatch.

4.3.2 Test procedure

Measure, to within ± 1 %, the required mass of powder and the required volume of liquid in the mixture ratio given by the manufacturer [6 c)]. If the manufacturer specifies a range of concentrations or volumes for the liquid, use the mean concentration or volume. Pour the liquid into the mixing bowl and sift the powder into the liquid within 10 s, minimizing entrapment of air. Begin timing from the moment the powder and liquid first make contact. Hand spatulate for (15 ± 1) s. Mechanically mix for the time specified by the manufacturer and then transfer the mix to the test moulds or form within 15 s.

5 Test methods

5.1 Fluidity

5.1.1 Apparatus

5.1.1.1 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.

5.1.1.2 Mould-release agent such as dry silicone spray.

5.1.1.3 Flat, square **glass plate,** of dimensions at least 150 mm by 150 mm.

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

5.1.1.5 Scale graduated in millimetres to measure the major and minor diameters of the slumped mix.

5.1.2 Test procedure

Coat the inside surface of the mould with mould-release agent. Mix the material as described in 4.3 using 200 g of powder. Start the timing device when the powder and the liquid first make contact. Centre the mould base on the glass plate and place it on the dental vibrator platform. Pour the mix into the mould until it is slightly overfilled. After filling, vibrate for 5 s. Level the mix flush with the top of the mould. At (120 ± 2) s from the start of mixing, lift the mould vertically from the plate at a rate of approximately 10 mm/s, allowing the mix to slump on the plate. As soon as the material has set, measure the largest and the smallest diameters of the base, and report the average value.

5.1.3 Evaluation

Test two specimens as described in 5.1.2. If both results meet the requirement of 3.2, the material meets the requirement of 3.2. If neither result meets the requirement, the material fails the requirement of 3.2. If only one result meets the requirement, test three more specimens. If all three results meet the requirement, the material meets the requirement of 3.2. Otherwise the material fails.

5.2 Setting time

5.2.1 Apparatus

5.2.1.1 Vicat needle apparatus; an example of which is shown in Figure 1, meeting the following requirements:

a) Vicat needle (C), 50 mm long, of circular cross-section and a diameter of (1,0 ± 0,05) mm, having a plane end perpendicular to the long axis;

b) rod (B), of approximate dimensions 270 mm long and 10 mm in diameter, with an additional weight (A);

c) total mass of the rod and needle (A, B and C) shall be (300 ± 1) g;

d) scale (D), graduated in millimetres;

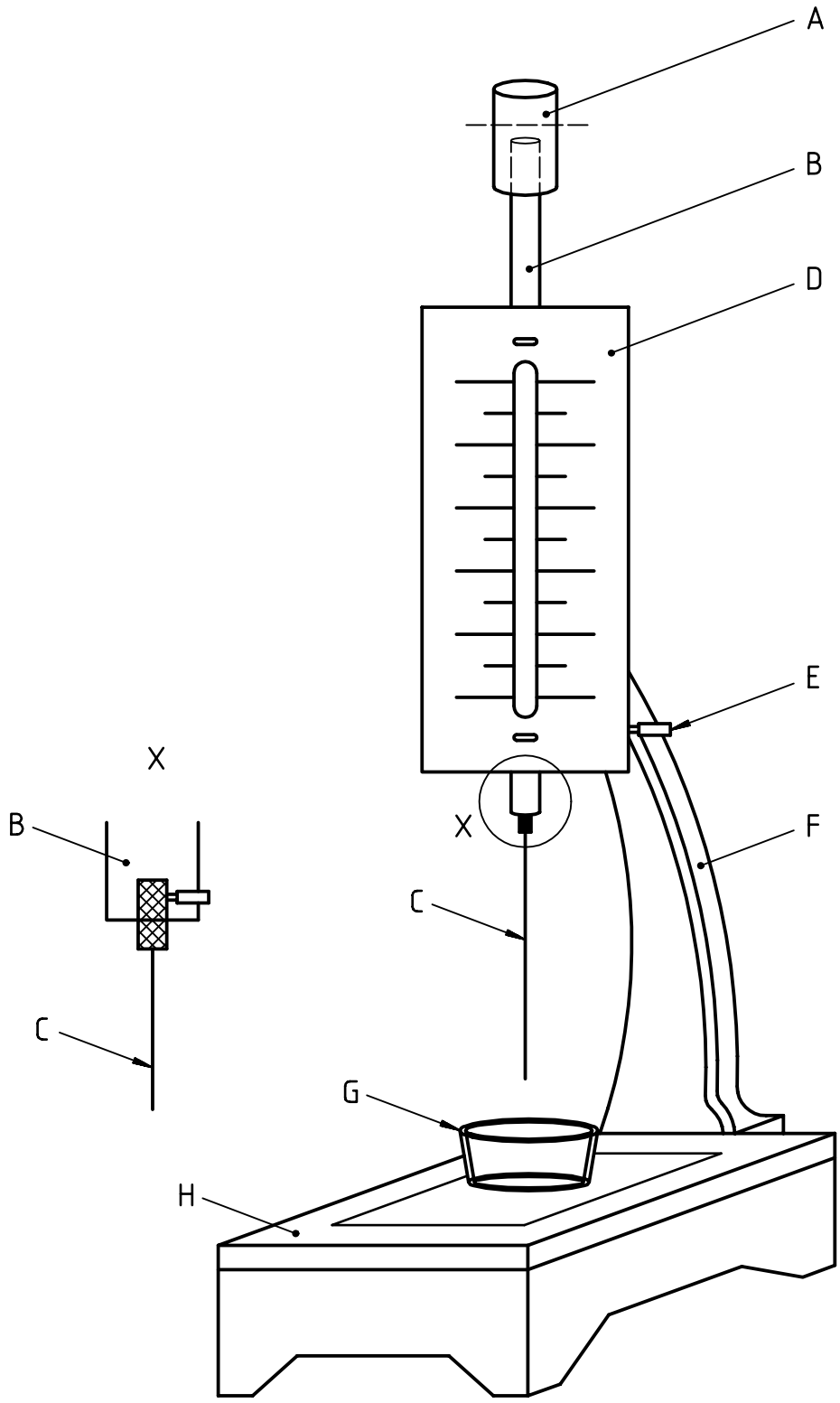
e) clean dry conical mould, constructed from a corrosion-resistant nonabsorbent and nonconductive material, of inside diameter (70 ± 2) mm at the top and (60 ± 2) mm at the base, and of height (40 ± 2) mm.

5.2.1.2 Mould-release agent, such as dry silicone spray or silicone grease.

5.2.1.3 Graduated cylinder, accurate to ± 0,5 ml.

5.2.1.4 Calibrated thermometer or digital thermocouple.

5.2.1.5 Timing device, accurate to 1 s.



Key

A Additional weight
B Rod
C Vicat needle
D Scale
E Lock screw
F Support bracket
G Conical ring mould
H Baseplate

Figure 1 — Typical Vicat needle apparatus (see 5.2.1.1**)**

5.2.2 Test procedure

Prepare a mix according to 4.3, using 200 g of powder, accurately weighed, and an amount of special liquid measured in the graduated cylinder to give the manufacturer's recommended liquid/powder ratio. Pour the mix into the mould prelubricated with the mould-release agent, and level the top surface. At 2 min after the start of mixing, the temperature of the mix shall be (26 ± 1) °C.

When the glossy surface of the mix has disappeared, lower the Vicat 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 10 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 the beginning of mixing until the needle first fails to penetrate to within 1 mm of the base.

5.2.3 Evaluation

Test two specimens as described in 5.2.2. If both results meet the requirement of 3.3, the material meets the requirement of 3.3. If neither result meets the requirement, the material fails the requirement of 3.3. If only one result meets the requirement, test three more specimens. If all three results meet the requirement, the material meets the requirement of 3.3. Otherwise the material fails.

5.3 Compressive strength

5.3.1 Apparatus

5.3.1.1 Sectional or split moulds, sufficient to produce cylindrical specimens with a diameter of (20,0 ± 0,2) mm and a length of (40,0 ± 0,4) mm, constructed from a corrosion-resistant material. Ends of the mould shall be parallel to within 0,05 mm.

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

5.3.1.3 Dental vibrator.

5.3.1.4 Compression-testing machine, adjusted to an average rate of loading of (5 ± 2) kN/min.

NOTE When using a testing machine with a constant cross-head rate, this rate is 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 are run to determine the appropriate cross-head speed.

5.3.1.5 Mould-release agent, such as dry silicone spray.

5.3.1.6 Micrometer or vernier calliper, graduated in divisions of 0,1 mm or less.

5.3.2 Test procedure

Make a mix according to 4.3 using 200 g of powder. Place the mould on a glass plate and slightly overfill it with the 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 60 min after the start of mixing. (Use at least two mixes of die material to prepare five specimens). Specimens are then allowed to set in air for 60 min.

Commence testing each specimen (120 ± 5) min from the beginning of mixing.

Position each specimen between the loading platens of the testing machine so that the specimen will be loaded in an axial direction. Do not use packing between specimen and platen. Using the compression testing machine (5.3.1.4), apply compressive force until fracture occurs and record the compressive force (F) at which fracture occurs.

5.3.3 Evaluation

For each specimen tested in accordance with 5.3.2, calculate the maximum stress (S), in megapascals, using the recorded maximum load, (F), in newtons, as follows:

$$S = F/314$$

If four or five of the results meet the requirement of 3.4, the material meets the requirement of 3.4. If three of the five results meet the requirement, test five more specimens. If all five of these results meet the requirement, the material meets the requirement of 3.4. Otherwise the material fails.

5.4 Linear setting expansion

Test method and evaluation for the linear setting expansion shall be in accordance with clause A.2 of annex A.

5.5 Linear thermal dimensional change

5.5.1 Apparatus

5.5.1.1 Vitreous silica dilatometer, including a linear inductive transducer instrument or other measuring instrument which exerts a measuring force to produce a stress no greater than 10 kPa, which is no greater than 0,8 N. The equipment shall be capable of measuring the change in length to the nearest 0,01 mm and of heating at a rate of (5 ± 1) °C/min, over the range of 23 °C to 700 °C.

5.5.1.2 Mould, constructed from a corrosion-resistant material or from a silicone-type material and capable of producing a specimen 20 mm to 50 mm long ,to an accuracy of 0,02 %, and of uniform cross-section ranging from 30 mm^2 to 170 mm^2.

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

5.5.1.4 Micrometer, accurate to 0,01 mm.

5.5.1.5 Sandpaper, 300 to 400 mesh, and a supporting block.

5.5.1.6 Mould-release agent, such as dry silicone spray.

5.5.2 Test procedure

5.5.2.1 Specimen preparation

A flexible mould constructed from silicone-type material does not have to be lubricated. If a rigid mould constructed from corrosion-resistant material is used, lubricate its inside surface with the mould-release agent.

The specimen to be used may be made from the same mix as prepared for the setting expansion measurement. Alternatively, prepare another mix using 200 g of powder at the manufacturer's recommended liquid-to-powder ratio in accordance with 4.3. Pour the mix to fill the mould completely. Scrape the top surface of the specimen to be flush with the surface of the mould. Remove the set specimen from the mould after 100 min to 110 min, so that subsequent preheating for degassing of the specimen can be started at (120 ± 5) min from the beginning of mixing. Scrape to level both ends of the specimen parallel to each other, and remove warpage, if any, using the sandpaper and the supporting block to make the specimen flat and square. Measure the dimensions of the specimen with the micrometer to a precision of at least 0,1 mm.

5.5.2.2 Linear firing shrinkage

Fire the specimen according to the manufacture's instruction at (120 ± 5) min from the beginning of mixing. Cool the specimen to room temperature. Measure the length of the fired specimen with the micrometer to a precision of at least 0,01 mm. Calculate the change in length (firing shrinkage) as a percentage of the initial length, to the nearest 0,02 %.

5.5.2.3 Linear thermal expansion

The specimen used for determining the linear firing shrinkage (5.5.2.2) can be utilized for measurement of the linear thermal expansion. As an alternative, a new specimen can be prepared, followed by firing and measurement (5.5.2.1 and 5.5.2.2).

Place the fired specimen in the dilatometer (5.5.1.1). Raise the temperature to 600 °C at a rate of (5 ± 1) °C/min. Maintain at (600 ± 10) °C for 15 min. Record the change in length at 600 °C, to the nearest 0,01 mm. Calculate the change in length, as a percentage of the length obtained after firing, to the nearest 0,02 %.

5.5.3 Evaluation

Test two specimens as described in 5.5.2.2 and 5.5.2.3. If both results meet the requirements of 3.6, the material meets the requirements for linear thermal dimensional changes (shrinkage during firing and expansion during thermal cycle). If neither result meets the requirements of 3.6, the material fails. If only one result meets the requirements of 3.6, test three more specimens. If all three results meet the requirements of 3.6, the material complies with the requirements for linear thermal dimensional change (3.6) of this International Standard. Otherwise, the material fails.

6 Instructions

The material shall be accompanied by instructions which contain at least the following information:

a) thermal expansion curve, determined in accordance with 5.5.2.3.

b) description of restorative materials (by brand name or specifications) which can be processed with this refractory die material;

c) recommended liquid-to-powder ratio, expressed as volume-to-mass;

d) if a special liquid is recommended, instructions for its use, storage and dilution;

e) recommended mixing procedure, including mixer type and mixing time;

f) description of duplicating materials and procedure, if applicable;

g) instructions for preheating or initial firing prior to the sintering process;

h) setting time;

i) linear setting expansion;

j) compressive strength;

k) linear thermal dimensional change after firing, and linear thermal expansion.

7 Marking

7.1 Containers for 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 refractory die material;

c) batch or lot number;

d) net mass of the powder, in grams or kilograms, net volume of special liquid;

e) recommended storage conditions;

f) expiry date (year and month), under recommended storage conditions.

g) warning, if the powder contains free silica which can cause lung damage when inhaled.

7.2 Individual packet

If the powder is packed in individual packets, each individual packet shall show at least the following information:

a) name or trademark of manufacturer and/or supplier;

b) batch or lot number;

c) name of the refractory die material;

d) minimum net mass of content, in grams or kilograms.

7.3 Container of liquid

Each container shall be marked with the following information:

a) name or trademark of manufacturer and/or supplier;

b) net volume, in millilitres or litres.

Annex A
(normative)

Linear setting expansion

A.1 Requirement

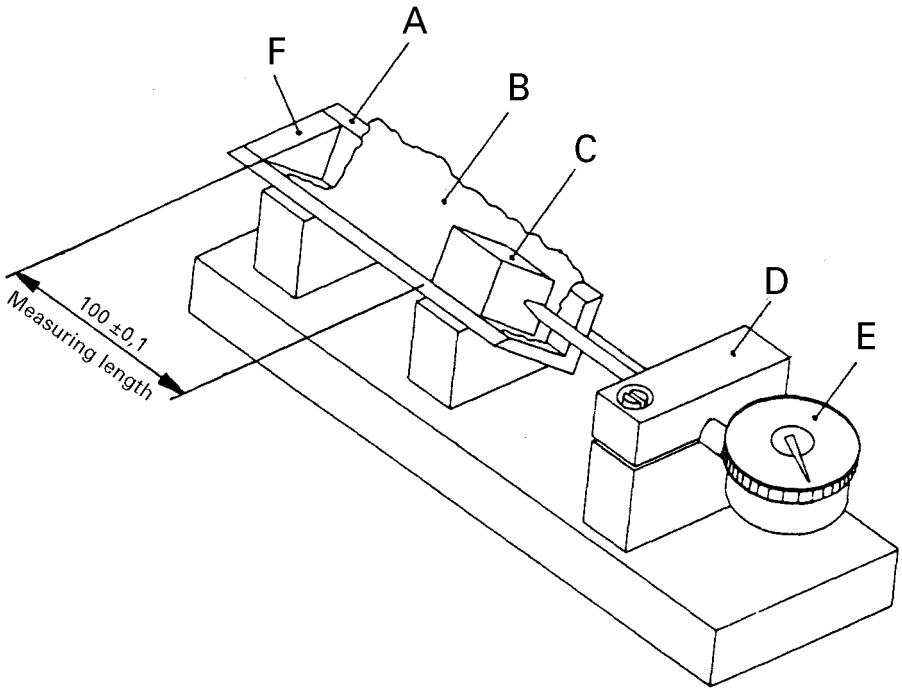
The linear setting expansion shall not differ by more than 35 % from the value stated by the manufacturer. If the manufacturer gives a range of linear setting expansion, then the linear setting expansion shall not differ by more than 35 % from the mid-point of the stated range. Test in accordance with clause A.2 and report in accordance with 6 i).

A.2 Test methods

A.2.1 Apparatus and materials

A.2.1.1 Extensometer, as shown in Figure A.1, capable of producing a specimen with a length of (100 ± 1) mm. The apparatus is fitted with a device or a gauge 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 with internal side lengths of (30 ± 1) mm. One end of the trough is blocked with an immovable endpiece and the other with a movable endpiece having a mass of (200 ± 10) g.

Dimensions in millimetres



Key

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

Figure A.1 — Example of suitable extensometer

A.2.1.2 Polytetrafluoroethylene (PTFE) film 0,1 mm to 0,2 mm thick, or a **dental rubber dam sheet** 0,15 mm to 0,35 mm thick.

A.2.1.3 Mould-release agent, such as dry silicone spray.

A.2.1.4 Timer, accurate to 1 s.

A.2.2 Test procedure

Prior to each measurement, apply the mould-release agent to the ends of the stop plates that contact the material being tested, and then line the trough of the extensometer with a prefolded PTFE or dental rubber dam sheet.

Set the movable endpiece in the trough position so that the specimen is (100 ± 1) mm long and the dial gauge reads zero (0).

Accurately weigh 200 g of powder and measure the recommended amount of special liquid in the graduated cylinder. Prepare the mix in accordance with 4.3. Pour the mix into the trough of the extensometer. The trough should not be overfilled. Record the initial length of the specimen at 1 min prior to the setting time as determined in accordance with 5.2. Do not cover the top surface of the specimen, to avoid unnatural expansion.

Take the final reading at (120 ± 1) min from the start 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 %.

A.3 Evaluation

Perform two tests. If both measurements meet the requirement for linear setting expansion (clause A.1), then the material complies with the requirement for linear setting expansion of this International Standard. If one measurement meets the requirement and the other fails, carry out three more tests. If all three measurements meet the requirement for linear setting expansion (clause A.1), then the material complies with the requirement for linear setting expansion of this International Standard. Otherwise, it fails to comply.

Bibliography

[1] ISO 3696, *Water for analytical laboratory use — Specification and test methods.*

[2] ISO 8601, *Data elements and interchange formats — Information exchange — Representation of dates and times.*

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