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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Dental zinc oxide/eugenol cements and zinc oxide non-eugenol cements

Ciments dentaires à base d'oxyde de zinc-eugénol et ciments dentaires à base d'oxyde de zinc sans eugénol

Reference number
ISO 3107 : 1988 (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 approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 3107 was prepared by Technical Committee ISO/TC 106, *Dentistry*.

This second edition cancels and replaces the first editions of ISO 3106 : 1974 and ISO 3107 : 1974.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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0 Introduction

Specific qualitative and quantitative requirements for 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.

1 Scope

This International Standard specifies requirements and test methods for zinc oxide/eugenol or zinc oxide/non-eugenol cements supplied as two separate components which may be either powder/liquid or paste/paste and which are suitable for use in the oral cavity. These non-aqueous cements may contain eugenol or an aromatic oil, compounds capable of reacting with zinc oxide such as accelerators, and gums, resins and inert inorganic fillers.

2 Field of application

This International Standard covers commercially manufactured zinc oxide/eugenol and modified zinc oxide/eugenol cements suitable for use in restorative dentistry for temporary cementation, for permanent cementation, such as temporary restorations and bases, and as cavity liners. This International Standard also covers non-eugenol cements containing zinc oxide and aromatic oils suitable for temporary cementation.

3 References

ISO 2590, *General method for the determination of arsenic — Silver diethyldithiocarbamate photometric method.*

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

4 Classification

For the purposes of this International Standard, zinc oxide/eugenol cements are classified, according to their intended use in restorative dentistry, into the following types.

Type I: For temporary cementation — setting and non-setting

Class 1: Powder and liquid

Class 2A: Setting paste and paste containing eugenol

Class 2B: Setting paste and paste not containing eugenol

Class 3: Non-setting paste and paste

Type II: For permanent cementation

Class 1: Powder and liquid

Type III: For temporary restorations and bases

Class 1: Powder and liquid

Class 2: Paste and paste

Type IV: For cavity liners

Class 1: Powder and liquid

Class 2: Setting paste and paste

Zinc oxide/non-eugenol cements covered by this International Standard are indicated as such.

5 Requirements

5.1 Material

The components of the material, when mixed in accordance with the manufacturer's instructions, shall produce a material with characteristics suitable for its intended use within a given time.

5.2 Components

5.2.1 Liquid

The liquid shall be clear, colourless or have only a slight amber tinge, and shall be free from suspended matter or deposits.

5.2.2 Powder

The powder shall be free of extraneous materials. When coloured, the pigment shall be uniformly dispersed throughout the powder.

5.2.3 Pastes

The unit package of pastes shall consist of two collapsible tubes or other containers, one containing the zinc oxide paste with or without modifiers, and the other containing eugenol or non-eugenol paste with or without modifiers. These pastes shall be homogeneous and free from extraneous matter.

5.3 Performance requirements

When tested in accordance with the appropriate test methods specified in clause 7, cements shall comply with the performance requirements specified in the table.

5.4 Biocompatibility

See clause 0 for use of ISO/TR 7405.

The total arsenic content of the cement shall not exceed the limit specified in the table, when tested in accordance with 7.6.

5.5 Manufacturer's instructions

Instructions for guidance of the user in proportioning, mixing and manipulation shall accompany each unit package. The following details shall be included:

a) the recommended temperature and humidity for mixing, and condition and type of mixing surface;

b) the component ratio recommended for each specific application;

c) the rate of incorporation of the components;

d) the time of mixing;

e) the working time after the end of mixing;

f) the setting time, where appropriate.

6 Sampling and inspection

6.1 Procurement

The method of procurement shall be the subject of an agreement between the manufacturer and test authority, and shall be recorded.

6.2 Sampling

A sample drawn from one batch shall provide sufficient powder and liquid or the appropriate pastes to complete all the specified tests.

6.3 Inspection

Compliance with the requirements specified in 5.2.1, 5.2.2, 5.2.3, 5.5 and clause 8 shall be determined by visual inspection.

7 Test methods

7.1 Preparation of test specimens

7.1.1 Ambient conditions

Carry out all mixing of the cement for the preparation of test specimens at a temperature of 23 °C ± 1 °C and a relative humidity of 50 % ± 2 %.

Table — Performance requirements

Type and class	Setting time at 37 °C, min		Compressive strength at 24 h, MPa		Disintegration after 24 h, % (*m*/*m*)	Film thickness, µm	Acid-soluble arsenic content, mg/kg (ppm)
	min.	max.	min.	max.	max.	max.	max.
Type I-class 1	4	10		35	2,5	25	2
Type I-class 2A	4	10		35	2,5	25	2
Type I-class 2B	4	10		35	2,5	25	2
Type I-class 3	Penetration at 1 h		NA*	NA*	NA*	25	2
Type II-class 1	4	10	35		1,5	25	2
Type III-class 1	3	10	25		1,5	NA*	2
Type III-class 2	3	10	25		1,5	NA*	2
Type IV-class 1	4	10	5		1,5	NA*	2
Type IV-class 2	4	10	5		1,5	NA*	2

* NA = not applicable

7.1.2 Apparatus for mixing

7.1.2.1 Smooth glass slab, approximately 150 mm × 75 mm × 20 mm. If a mixing pad is supplied by the manufacturer, it may be used.

7.1.2.2 Rigid spatula, inert to the cement.

All apparatus used for mixing and testing shall be kept clean, dry and free from particles of hardened cement.

7.1.3 Conditioning

Before the start of mixing, condition the test samples and apparatus at the ambient conditions specified in 7.1.1, for at least 1 h, except where otherwise stated by the manufacturer.

7.1.4 Procedure for mixing

Place the components on the mixing surface in the ratio specified by the manufacturer.

If the material is supplied as a paste/paste system, use a component ratio in grams per gram or in measured lengths, in accordance with the manufacturer's instructions, producing a minimum of 0,75 ml of mixed material.

Completely mix the components in accordance with the manufacturer's instructions.

7.2 Determination of setting time

7.2.1 Apparatus

7.2.1.1 Oven or **cabinet,** capable of being maintained at a temperature of 37 °C ± 1 °C and a relative humidity from 95 % to 100 %.

7.2.1.2 Indentor needle, of mass 400 g ± 2 g and having a flat end of diameter 1,0 mm ± 0,1 mm. The needle tip shall be cylindrical for a distance of approximately 5,0 mm. The needle end shall be plane and at right angles to the axis of the rod. This indentor needle shall be used for testing type I-class 1, type II-class 1, type III-class 1 and type IV-class 1.

A similar indentor needle of mass 100 g ± 0,5 g and having a flat end of diameter 2,0 mm ± 0,1 mm shall be used for type I-classes 2A and 2B, and type IV-class 2.

7.2.1.3 Mould, made of non-corrodible metal, consisting of a rectangular plate with a circular hole conforming to the dimensions given in figure 1.

7.2.1.4 Metal block, of minimum dimensions 8 mm × 20 mm × 10 mm, either as part of 7.2.1.1 or 7.2.1.2 or as a separate item.

7.2.1.5 Flat glass plate, approximately 1 mm thick (for example, a microscope slide).

7.2.2 Procedure

Condition the metal block (7.2.1.4) and indentor needle (7.2.1.2) in the oven (7.2.1.1) at 37 °C ± 1 °C.

Place the metal mould (7.2.1.3), conditioned at 23 °C ± 1 °C, on the flat glass plate (7.2.1.5) and fill to a level surface with the cement mixed in accordance with the manufacturer's instructions.

After 120 s ± 10 s for type III-class 1, or 180 s ± 10 s for type I-classes 1, 2A and 2B, type II-class 1 and type IV-classes 1 and 2 from the start of mixing, transfer the specimen to the oven for testing.

As soon as possible after placing the specimens in the oven, carefully lower the indentor needle vertically onto the surface of the cement. Make indentations at 15 s intervals until the time of setting has been reached. Maintain the needle in a clean condition between indentations.

Record the setting time as the period of time which elapses from the start of mixing to the time when the needle fails to penetrate completely the 2 mm depth of cement. This penetration can be confirmed by holding the specimen up to the light and examining visually. Repeat this test once.

NOTE — Type I-class 3 is non-setting. To verify this property, use the 100 g ± 0,5 g indentor needle and test every 15 min for 1 h. Complete penetration shall be obtained for each trial.

7.2.3 Expression of results

Calculate the average of two determinations and record the result to the nearest 15 s.

7.3 Determination of compressive strength

7.3.1 Apparatus

7.3.1.1 Oven or **cabinet,** as specified in 7.2.1.1.

7.3.1.2 Five split moulds and **plates,** such as shown in figure 2, 6 mm high and with an internal diameter of 4 mm, made of stainless steel or other material which is not attacked or corroded by the cement.

7.3.1.3 Five individual screw clamps, such as those shown in figure 3.

7.3.1.4 Compressive strength-testing apparatus, having a cross-head speed of 1,00 mm/min ± 0,25 mm/min.

7.3.2 Preparation of test specimens

Prepare at least five specimens.

Condition the moulds (7.3.1.2), screw clamps (7.3.1.3) and top and bottom plates (7.3.1.2) at 23 °C ± 1 °C.

After mixing in accordance with the manufacturer's instructions, pack the cement, to a slight excess, into the split moulds within 1 min of the completion of mixing.

NOTE — In order to consolidate the cement and to avoid trapping air, it is advisable to convey the largest convenient portions of mixed cement to the mould and apply to one side with a suitable instrument.

Fill the mould to excess in this manner and then place on the bottom plate with some pressure.

Remove any bulk extruded cement, place the top metal plate in position and squeeze together. Put the mould and plates in the clamp (7.3.1.3) and screw tightly together. Not later than 2 min after completion of mixing, transfer the whole assembly to the oven (7.3.1.1), maintained at 37 °C ± 1 °C.

One hour after completion of mixing, remove the plates, and prepare the surface of the ends of the specimen plane, at right angles to its long axis, using a small amount of 45 µm silicon carbide powder or similar abrasive, mixed with water on a flat glass plate. Alternatively, use an equivalent grade of abrasive coated paper and water. Keep both ends of the specimen wet during grinding and rotate the specimen by one-quarter turn every few strokes.

Remove the specimen from the mould immediately after surfacing and examine for air-voids or chipped edges. Discard any specimens with these defects.

NOTE — To facilitate the removal of the hardened cement specimen, the internal surface of the mould may be evenly coated, prior to filling, with a 3 % solution of micro-crystalline or paraffin wax in pure toluene. Alternatively, a thin film of silicone grease or polytetrafluoroethylene (PTFE) dry film lubricant may be used.

Immerse each acceptable specimen in distilled or deionized water and maintain at 37 °C ± 1 °C for 24 h, after which, place in distilled or deionized water at 23 °C ± 1 °C for at least 15 min prior to testing.

7.3.3 Procedure

Test at least five specimens.

Twenty-four hours after completion of mixing, determine the compressive strength of the test specimens using the compressive strength-testing apparatus (7.3.1.4).

Place the specimen with the flat ends between the platens of the apparatus so that the load is applied in the long axis of the specimen.

7.3.4 Expression of results

Record the maximum load applied when the specimen fractures, and calculate the compressive strength, κ in megapascals, using the following formula

$$\kappa = \frac{4F}{\pi d^2}$$

where

F is the maximum applied load, in newtons;

d is the diameter of the test specimen, in millimetres.

7.3.5 Compliance

If at least four of the five results obtained are below the minimum strength specified in the table, the material shall be deemed to have failed to meet the requirements of the table. If at least four of the five results are above the minimum strength specified in the table, the material shall be deemed to have met the requirements of the table. In other cases, prepare a further 10 specimens and calculate the median result for all 15 specimens. Round this value to two significant figures and record as the compressive strength.

7.4 Determination of film thickness

7.4.1 Apparatus

7.4.1.1 Two optically flat, circular glass plates, of minimum uniform thickness 5 mm and having a contact surface area of approximately 200 mm² ± 10 mm².

7.4.1.2 Loading device, of the type shown in figure 4, or an equivalent means whereby a force of 147 N (15 kg mass) may be applied vertically onto the cement. The bottom surface of the rod carrying the load shall be horizontal and parallel to the base and large enough to cover one of the glass plates. The loading device shall be capable of applying the load smoothly in such a manner that no rotational motion occurs. Each glass plate shall be attached to the loading device by guides to prevent movement when the load is applied.

7.4.1.3 Micrometer or similar measuring instrument, accurate to 1 µm.

7.4.2 Procedure

Measure accurately the thickness of the two optically flat glass plates (7.4.1.1) stacked in contact (reading A).

Place a small quantity of cement, mixed in accordance with the manufacturer's instructions, on the centre of one of the glass plates and place the plate in the guides. Place the second glass plate centrally on the cement.

At the working time specified in the manufacturer's instructions, carefully apply, by means of the loading device (7.4.1.2), a force of 147 N vertically on the top and leave for 8 min. Ensure that the cement completely fills the space between the two glass plates.

Measure the thickness of the two glass plates and cement film (reading B).

7.4.3 Expression of results

Calculate the difference in thickness of the plate with and without the cement film (reading B − reading A), and record this as the thickness of the film.

Record the mean result of three such tests to the nearest 1 µm.

7.5 Determination of disintegration

7.5.1 Apparatus

7.5.1.1 Oven or **cabinet,** as specified in 7.2.1.1.

7.5.1.2 Mould, consisting of a split stainless steel ring of height 1,5 mm and of internal diameter 20 mm, contained in a former or retaining plate similar to that shown in figure 5.

The former or retaining plate shall ensure that excess cement does not expand the split ring beyond a diameter of 20 mm.

7.5.1.3 Two pieces of wire, made of stainless steel or other non-corrodible material, of diameter approximately 0,25 mm and of length approximately 50 mm, each weighed to the nearest 0,001 g.

7.5.1.4 Two wide-mouth weighing bottles, having a capacity of at least 50 ml, as shown in figure 6.

7.5.1.5 Single or multiple spring clamp, as shown in figure 7.

Condition the spring clamp by placing in the oven for at least 5 min before preparing the test specimen. Do not remove until required.

7.5.1.6 Desiccator, containing thoroughly dry anhydrous calcium sulfate or silica gel freshly dried at 130 °C.

7.5.2 Preparation of test specimens

Prepare two specimens for each determination.

Place the mould (7.5.1.2) on a thin polyethylene or cellulose acetate sheet backed by a flat glass plate.

Insert a convenient tared length of wire (7.5.1.3) through the split ring so that at least 10 mm projects into the ring. Fill the split ring with a slight excess of the cement mixed in accordance with the manufacturer's instructions. Cover with another flat glass plate faced with a sheet of polyethylene or cellulose acetate and press firmly together.

Three minutes after the start of mixing, place the mould and plates into the spring clamp (7.5.1.5) which is in the oven (7.5.1.1) maintained at a temperature of 37 °C ± 1 °C.

After 1 h, remove the plates containing the specimen from the clamp and carefully separate the cement disc and attached wire from the split ring.

NOTE — Due to the comparatively brittle nature of some cements at this early hardening stage, it is essential to clean any excess cement from the surface of the split ring mould before attempting removal of the specimens. A suitable release agent should be used to facilitate removal of the specimen from the mould; polytetrafluoroethylene (PTFE) dry film lubricant is suggested.

Remove any surplus cement from the edge of the specimen disc and lightly brush any loose material from the surface.

7.5.3 Procedure

Place the two test specimen discs in the wide-mouth bottle (7.5.1.4) and record the net mass of cement to the nearest 0,001 g (mass m_1).

Immediately submerge the two discs by pouring 50 ml of distilled water into the bottle, then store for 24 h at 37 °C ± 1 °C. Suspend the specimens by the wire (see figure 6) so they touch neither each other nor the sides of the bottle and close the lid as tightly as possible.

After immersing the discs for 24 h, remove the specimens from the water. Rinse their surfaces with a small amount of distilled water. Blot the surface dry with clean absorbent paper. Store the specimens in the desiccator (7.5.1.6) for 24 h and reweigh to the nearest 0,001 g. Repeat until a constant mass (± 0,001 g) is reached. Record the final mass (mass m_2).

7.5.4 Expression of results

Express the disintegration, D, as a percentage by mass, using the following formula:

$$D = \frac{m_1 - m_2}{m_1} \times 100$$

Record the average of duplicate tests (two bottles containing two specimens each) to the nearest 0,01 %.

7.6 Determination of acid-soluble arsenic content

7.6.1 Preparation of test sample

Powder the set cement and sieve throught a 75 µm (200 mesh) sieve. Disperse 2 g of the sieved powder in 30 ml of water and add 10 ml of hydrochloric acid, 36 % (m/m) (ϱ = 1,18 g/ml). Maintain the mixture at 37 °C ± 1 °C for 1 h, then filter the solution and use it in the test for total arsenic content.

7.6.2 Procedure

Determine the total arsenic content by the method described in ISO 2590 or any other analytical method of equivalent sensitivity.

8 Packaging and marking

8.1 Packaging

The components shall be supplied in securely sealed containers, made from materials which do not react with or permit contamination of the contents.

NOTE — For the purposes of this International Standard, the container is considered to be the immediate wrapping of the component.

8.2 Instructions for use

Instructions detailed in 5.5 shall accompany each unit package.

8.3 Marking of containers

Each container shall be clearly marked with the following particulars:

a) the name and/or trade-mark of the manufacturer;

b) the type and class of cement;

c) the minimum net mass, in grams, of the powder or paste, and the minimum net volume, in millilitres, of the liquid;

d) a serial number or code number to identify each batch or lot, together with the actual date of manufacture and the estimated shelf life.

Dimensions in millimetres

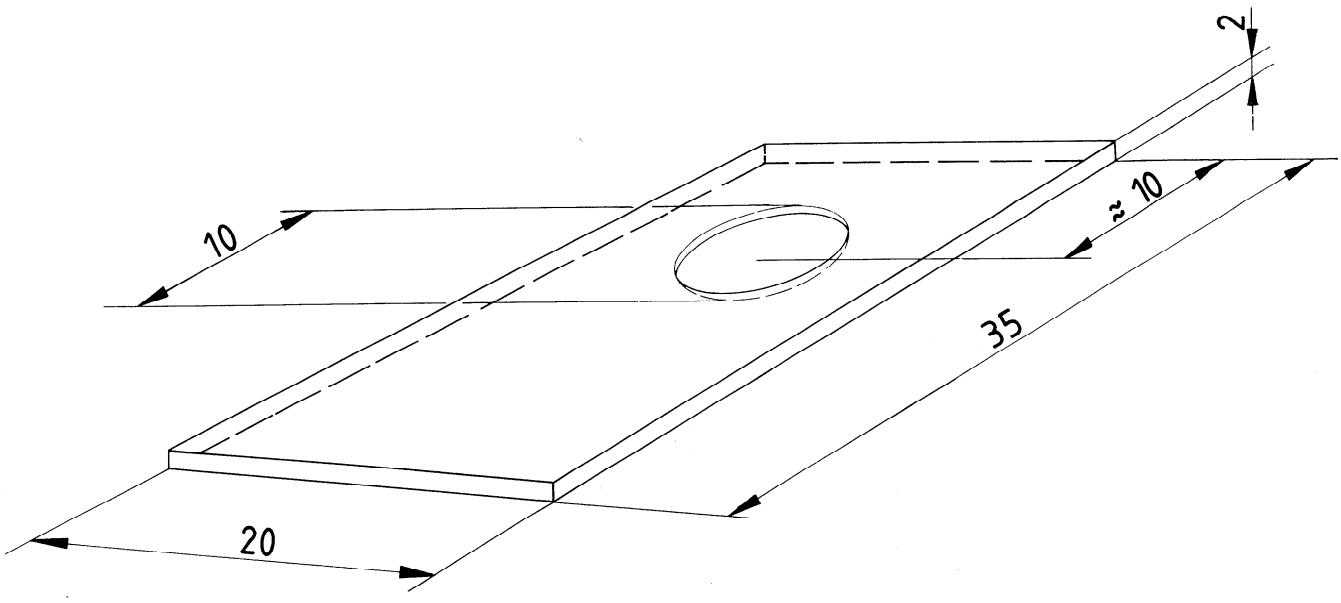

Figure 1 — Mould for use in determination of setting time

Dimensions in millimetres

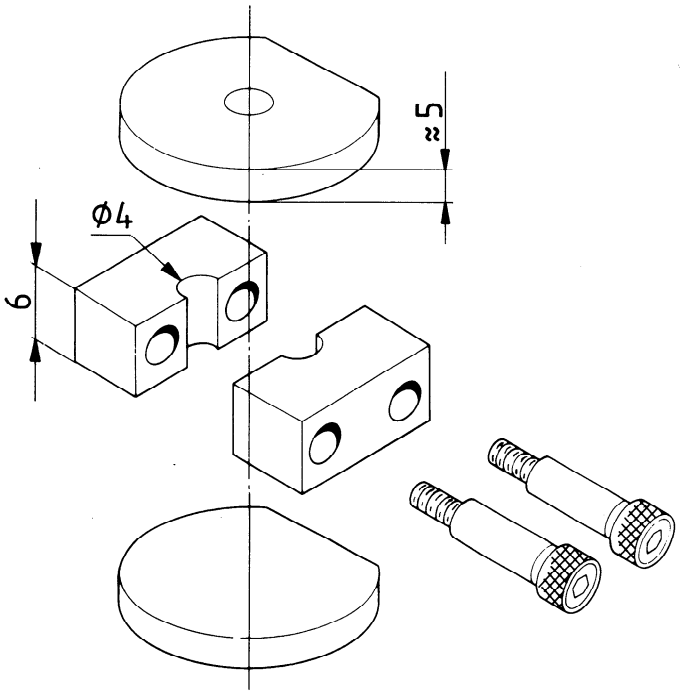

Figure 2 — Mould for preparation of compressive strength test specimens

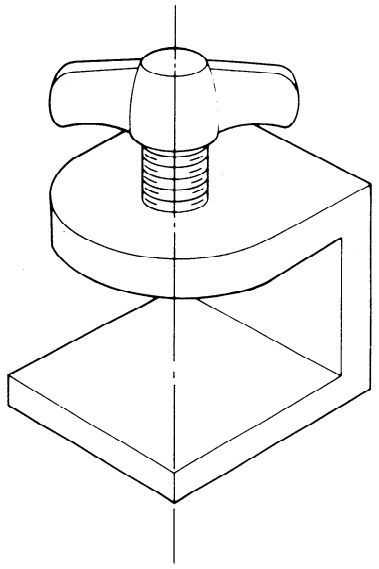

Figure 3 — Clamp for preparation of compressive strength test specimens