International Standard 6876

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

Dental root canal sealing materials

Produits dentaires pour le scellement des canaux radiculaires

First edition — 1986-12-01

UDC 615.463 : 616.314 **Ref. No. ISO 6876-1986 (E)**

Descriptors : dental materials, sealing materials, material specifications.

Price based on 4 pages

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.

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 6876 was prepared by Technical Committee ISO/TC 106, *Dentistry*.

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.

Printed in Switzerland

Dental root canal sealing materials

1 Scope and field of application

This International Standard specifies requirements for materials used for permanent obturation of the root canal with or without the aid of obturating points. This International Standard only covers materials intended for orthograde (root filling inserted from coronal aspect) use.

2 References

ISO 3665, *Photography — Intra-oral dental radiographic film — Specification*.

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

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

3 Classification

The root canal sealing materials covered by this International Standard are classified into the following types :

Type 1 : Setting materials.

These are materials which set within 72 h of commencement of mixing.

Type 2 : Non-setting materials.

4 Requirements

4.1 Component

The component of the sealing material shall be free from extraneous matter.

The purity and sterility of the ingredients shall comply with the relevant pharmacopoeia applicable in the country in which the material is marketed, or with such national regulations as are applicable to purity and sterility of pharmaceutical products.

The ingredients shall, when used in accordance with the manufacturer's instructions, form a material which complies with the requirements of this International Standard.

4.2 Freedom from toxicity

The material shall not cause any irreversible damage when in contact with the oral tissues. Reference shall be made to ISO/TR 7405 which gives guidance on the biological evaluation of dental materials.

4.3 Flow

The flow of the material, when determined in accordance with 7.3, shall produce a disc with a diameter of not less than 20 mm.

4.4 Working time

The working time for type 1 materials having a working time of less than 30 min, when determined by the method given in 7.4, shall be within ± 10 % of the working time stated by the manufacturer [see 5e)].

4.5 Setting time

The setting time of type 1 materials, determined by the method given in 7.5, shall be within ± 10 % of the setting time stated by the manufacturer [see 5e)].

For materials having a setting time greater than 30 min for which the manufacturer quotes a range of times, the setting time measured shall be within the specified range.

4.6 Film thickness

The film thickness, for materials intended for use with obturating points, shall not be more than 50 µm, when tested in accordance with 7.6.

4.7 Radio-opacity

The material, when tested in accordance with 7.7, shall have a radio-opacity equivalent of not less than 3 mm of aluminium.

4.8 Solubility and disintegration

The solubility of the material, when determined in accordance with 7.8, shall not exceed 3 % by mass, nor shall the specimen show evidence of disintegration.

1) At present at the stage of draft.

5 Manufacturer's instructions

Instructions shall accompany each package and shall include the following:

a) instructions for use of the material, including, where applicable, the method of mixing and component mixing ratio;

b) the recommended method for sterilization, if possible or necessary;

c) principal components and active ingredients of material(s);

d) recommended conditions of storage;

e) the working time (if less than 30 min) and the setting time of the material — if the material is of the non-setting variety this should be stated; if a precise setting time cannot be determined but it exceeds 30 min, a range may be quoted;

f) indications for clinical use, including whether the material is manufactured for use with obturating points — if the material can cause staining of the tooth, this should be stated together with any precautions necessary to minimize this effect.

6 Sampling

The sample shall consist of one or more retail packages from the same batch, containing sufficient material to carry out the specified tests, plus an allowance for repeats, if necessary.

7 Test methods

7.1 Test conditions

Unless otherwise stated by the manufacturer, all tests shall be carried out at 23 ± 2 °C and at a relative humidity of (50 ± 5) %.

7.2 Preparation of material for testing

The material shall be manipulated in accordance with the manufacturer's instructions.

7.3 Flow

7.3.1 Apparatus

7.3.1.1 Two glass plates, having minimum dimensions of 30 mm × 30 mm, and approximately 5 mm thick.

7.3.1.2 Loading device, to apply a load of 2,5 kg.

7.3.1.3 Graduated glass delivery tube, designed to deliver 0,075 ± 0,005 ml of mixed material.

7.3.2 Procedure

Using the glass delivery tube (7.3.1.3), deposit 0,075 ml of the material, mixed in accordance with the manufacturer's instructions, onto one of the glass plates (7.3.1.1). After 180 ± 5 s from the start of mixing, place the other glass plate centrally on the material and carefully apply, by means of the loading device (7.3.1.2), the 2,5 kg load. After 10 min from the start of mixing, remove the load and measure the major and minor diameters of the disc. Record the mean of the major and minor diameters. If the diameters differ from each other by more than 1 mm, repeat the test.

7.3.3 Calculation and expression of results

Carry out three determinations, calculate the mean result and record it, to the nearest millimetre, as the flow value.

7.4 Working time

NOTE — This test applies only to type 1 materials having a working time of less than 30 min (see 4.4).

7.4.1 Apparatus

The apparatus specified in 7.3.1 shall be used.

7.4.2 Procedure

Using the glass delivery tube (7.3.1.3), deposit 0,075 ml of the material, mixed in accordance with the manufacturer's instructions, onto one of the glass plates (7.3.1.1). After 210 ± 5 s from the start of mixing, place the other glass plate centrally on the material and carefully apply, by means of the loading device (7.3.1.2), the 2,5 kg load. Apply the load for 7 min, then remove it and measure the major and minor diameters of the disc. Record the mean of the major and minor diameters.

Repeat the test with newly mixed material applying the load at increasing intervals of time after the start of mixing until the diameter has decreased by 10 % from the flow value (see 7.3.3).

7.4.3 Calculation and expression of results

Carry out three determinations, calculate the mean result and record it, to the nearest 30 s, as the working time of the material.

7.5 Setting time

NOTE — This test applies only to type 1 materials (see 4.5).

7.5.1 Apparatus

7.5.1.1 Cabinet, capable of being maintained at a temperature of 37 ± 1 °C and a relative humidity of not less than 95 %.

7.5.1.2 Gilmore-type metric indenter, having a mass of 100 ± 0,5 g and a flat end of diameter 2 ± 0,1 mm. The needle tip shall be cylindrical over a distance of at least 5 mm. The end of the needle shall be plane and at right angles to the longitudinal axis and shall be kept clean.

7.5.1.3 Stainless steel ring mould, having an internal diameter of 10 mm and a height of 2 mm.

7.5.1.4 Metal block, having minimum dimensions of 8 mm × 20 mm × 10 mm.

7.5.1.5 Flat glass plate, approximately 1 mm thick.

NOTE — A microscope slide is suitable.

7.5.2 Procedure

Place the mould (7.5.1.3) on the glass plate (7.5.1.5) and fill it to a level surface with material mixed in accordance with the manufacturer's instructions. After 120 ± 10 s from the start of mixing, place this assembly on the metal block (7.5.1.4) maintained at 37 ± 1 °C in the cabinet (7.5.1.1).

When the setting time stated by the manufacturer approaches, carefully lower the Gilmore-type needle (7.5.1.2) vertically onto the horizontal surface of the material. Repeat this operation until indentations cease to be visible and then record the time from the start of mixing.

7.5.3 Calculation and expression of results

Carry out three determinations, calculate the mean result and record it as the setting time.

7.6 Film thickness

7.6.1 Apparatus

7.6.1.1 Two optically flat circular glass plates, having a minimum uniform thickness of 5 mm and a contact surface area of approximately 200 mm^2.

7.6.1.2 Loading device, to apply a load of 15 kg.

7.6.1.3 Micrometer or **similar measuring instrument,** accurate to 1 µm.

7.6.2 Procedure

Measure the combined thickness of the two glass plates (7.6.1.1) in contact to an accuracy of 1 µm. Deposit a portion of material, mixed in accordance with the manufacturer's instructins, onto the centre of one of the glass plates. Place the other glass plate centrally on the material. After 180 ± 10 s from the start of mixing, carefully apply, by means of the loading device (7.6.1.2), a load of 15 kg vertically on the top plate. Ensure that the material completely fills the area between the glass plates. After 10 min from the start of mixing, measure the thickness of the two glass plates and the film of material using the micrometer (7.6.1.3).

7.6.3 Calculation and expression of results

Calculate the thickness of the film by determining the difference in the thickness of the plates with and without the material.

Carry out three determinations, calculate the mean result and record it, to the nearest 5 µm, as the film thickness.

7.7 Radio-opacity

7.7.1 Apparatus

7.7.1.1 Stainless steel ring mould, having an internal diameter of 10 mm and a height of 1 mm, together with covers, made of either plastic, paper or other radiolucent material.

7.7.1.2 Dental X-ray unit, capable of being operated at 65 kV.

7.7.1.3 Dental X-ray occlusal film of speed group D (as specified in ISO 3665), **developing solution** and **fixer.**

7.7.1.4 Aluminium step wedge, 50 mm × 20 mm, having a thickness range from 1 to 10 mm in steps of 1 mm, and made of at least 99,5 % pure aluminium.

7.7.1.5 Photographic densitometer.

7.7.2 Procedure

Place the material, mixed in accordance with manufacturer's instructions, in the mould (7.7.1.1) and press the covers on the top and bottom to make a specimen 1 mm thick. Position the specimen in the centre of the X-ray film (7.7.1.3) adjacent to the aluminium step wedge (7.7.1.4). If a cover is used, place an equivalent cover under the step wedge.

Irradiate the specimen, step wedge and film at a target film distance of approximately 300 mm for such a time that the exposed and processed film under the 1 mm thick section of the step wedge has a photographic density in the region of 1,5 to 2,5, including base and fog.

After developing, fixing and drying the exposed film, measure the photographic density of the radiographic image of the specimen, each of the steps of the step wedge and the totally exposed film, using the densitometer (7.7.1.5).

7.7.3 Expression of results

By comparison with the image of the step wedge, determine the thickness of aluminium equivalent to the specimen. Record this to the nearest 0,1 mm.

Carry out three determinations, calculate the mean result and record it as the radio-opacity value.

7.8 Solubility and disintegration

7.8.1 Apparatus and materials

7.8.1.1 Two split ring moulds, having an internal diameter of 20 mm and a height of 1,5 mm.

7.8.1.2 Four polished flat glass plates, having dimensions larger than the maximum dimensions of the split ring moulds.

7.8.1.3 Sheets of impervious plastic, such as polyethylene.

7.8.1.4 Glass Petri dish, having a diameter of approximately 90 mm, with a minimum volume of 50 ml and of known mass.

7.8.1.5 Cabinet, capable of being maintained at a temperature of 37 ± 1 °C and a relative humidity of not less than 95 %.

7.8.1.6 Water complying with grade 3 of ISO 3696.

7.8.1.7 Desiccator, containing phosphorus pentoxide or other suitable desiccant.

7.8.2 Procedure

Place a mould (7.8.1.1) on a glass plate (7.8.1.2) the net masses of which are known to the nearest 0,001 g and fill to slight excess with mixed (if necessary) material. Press another glass plate faced with a sheet of plastic (7.8.1.3) on top of the material and carefully remove to leave a flat, uniform surface.

If the material is of type 1, place the filled mould in the cabinet (7.8.1.5) for a period of time 50 % longer than the setting time stated by the manufacturer [see 5e)].

Determine the mass of material to the nearest 0,001 g.

Place two such specimens in the Petri dish (7.8.1.4) such that the surfaces do not touch and the material remains undisturbed in the mould. Add 50 ± 1 ml of the water (7.8.1.6) and cover the dish. Store the Petri dish and contents at 37 ± 1 °C for 24 h and then remove the specimens. Wash the specimens with a little of the water allowing the washings to drain back into the Petri dish. Discard the specimens.

Evaporate the water from the dish without boiling and dry the dish to constant mass at 150 °C, cooling the dish in the desiccator (7.8.1.7) to room temperature before each weighing (accurate to the nearest 0,001 g).

7.8.3 Calculation and expression of results

Carry two out tests for each material. Record the mean difference between the original mass of the Petri dish and its final mass, to the nearest 0,001 g, as the amount of material removed from the specimens. Record this difference in mass, calculated as a percentage of the original combined mass of the two specimens reported to the nearest 0,1 %, as the solubility of the material.

8 Packaging

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

9 Marking

Each package and/or container within the package shall be clearly and legibly marked with the following particulars:

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

b) the name of the product;

c) a number or code which shall refer to the manufacturer's record and date of manufacture for the particular batch of the material(s);

d) the minimum mass, in grams, of the powder or pastes and the minimum net volume, in millilitres, of the liquid, if included;

e) recommended conditions of storage and, if applicable, expiry date when stored under those conditions.

This page intentionally left blank

This page intentionally left blank