BS EN ISO 6876:2012

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Dentistry — Root canal sealing materials (ISO 6876:2012)

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National foreword

This British Standard is the UK implementation of EN ISO 6876:2012. It supersedes BS EN ISO 6876:2002 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee CH/106/1, Dental restorative and orthodontic materials.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Foreword

This document (EN ISO 6876:2012) has been prepared by Technical Committee ISO/TC 106 "Dentistry" in collaboration with Technical Committee CEN/TC 55 “Dentistry” the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by January 2013, and conflicting national standards shall be withdrawn at the latest by January 2013.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN ISO 6876:2002.

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Foreword

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ISO 6876 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 1, *Filling and restorative materials*.

This third edition cancels and replaces the second edition (ISO 6876:2001), which has been technically revised. The main modifications are the following:

— the test procedures for flow, working time and solubility have been revised and a new limit value has been set;

— the test to determine dimensional change following setting has been removed.

Introduction

Following the publication of the second edition of this International Standard (ISO 6876:2001), test houses reported difficulties with some of the test procedures. In an attempt to improve the test procedures, a planned programme of revision began in 2006. The following should be taken into account when using this International Standard.

— Verification for a claim of sterility is the responsibility of the manufacturer. This International Standard does not specify requirements or test methods for sterility and it is recommended that reference be made to any national requirements that may exist. When no national requirements exist, reference should be made to the United States, European or Japanese Pharmacopoeia.

— If a therapeutic effect is claimed, the purity and sterility of the constituents are expected to comply with the relevant pharmacopoeia applicable in the country in which the sealer is marketed, or with such national regulations as are applicable to purity and sterility of pharmaceutical products.

— Specific qualitative and quantitative test methods for demonstrating freedom from unacceptable biological risks are not included in this International Standard but it is recommended that, for the assessment of such biological risks, reference be made to ISO 10993-1 and ISO 7405.

Dentistry — Root canal sealing materials

1 Scope

This International Standard specifies requirements and test methods for root canal (endodontic) sealing materials which set with or without the assistance of moisture and are used for permanent obturation of the root canal with or without the aid of obturating points/cones. It only covers sealers intended for orthograde use i.e. a root filling placed from the coronal aspect of a tooth.

Specific qualitative and quantitative test methods for demonstrating freedom from unacceptable biological risks are not included in this International Standard but it is recommended that, for the assessment of such biological risks, reference be made to ISO 10993-1 and ISO 7405.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1942, *Dentistry — Vocabulary*

ISO 3665, *Photography — Intra-oral dental radiographic film and film packets — Manufacturer specifications*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 6873, *Dentistry — Gypsum products*

ISO 8601, *Data elements and interchange formats — Information interchange — Representation of dates and times*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1942 and the following apply.

3.1
root canal sealing material
endodontic material intended to permanently seal the root canal filling material into the cavities previously occupied by the removed pulp

3.2
root canal filling material
endodontic material intended to permanently obturate the cavities previously occupied by the pulp

3.3
mixing time
that part of the working time required in order to obtain a satisfactory mix of the components

3.4
working time
period of time, measured from the start of mixing, during which it is possible to manipulate the root canal sealer without any adverse effect on its properties

3.5
setting time
period of time measured from the end of mixing until the sealer has set, according to the criteria and conditions described in 5.4

NOTE For the purposes of this International Standard, the setting time is determined from the end of mixing because of the wide variation in mixing times.

4 Requirements

4.1 Components

The components of the sealer shall be free from extraneous matter when tested according to 5.1.

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

4.2 Microbiological hazard

Specific qualitative and quantitative requirements for the determination of microbiological growth are not included in this International Standard.

4.3 Physical and mechanical properties

4.3.1 Flow

When determined in accordance with 5.2, each disc shall have a diameter of not less than 17 mm.

4.3.2 Working time

For sealers that are claimed by the manufacturer to have a stated working time of up to 30 min, the diameter of the flow test, determined in accordance with 5.2, shall be not less than 17 mm 15 s before the end of the stated working time.

4.3.3 Setting time

For sealers that are claimed by the manufacturer to have a setting time of up to 30 min, the setting time shall be no more than 10 % longer than that claimed by the manufacturer, when determined in accordance with 5.4.

For sealers that are claimed by the manufacturer to have a setting time of more than 30 min and up to 72 h, and for which the manufacturer quotes a time range, the setting time measured shall be within the range stated by the manufacturer, when determined in accordance with 5.4.

4.3.4 Film thickness

Sealers shall have a film thickness of not more than 50 µm when tested in accordance with 5.5.

4.3.5 Solubility and disintegration

The solubility of the set sealer, when determined in accordance with 5.6, shall not exceed 3,0 % by mass.

The specimen shall show no evidence of disintegration when examined visually.

4.3.6 Radio-opacity

The sealer, when tested in accordance with 5.7, shall have a radio-opacity equivalent to not less than 3 mm of aluminium.

4.4 Sampling

Use one or more retail packages from the same batch, containing sufficient sealer to carry out the specified tests, plus an allowance for repeats, if necessary.

4.5 Test conditions

Unless otherwise stated by the manufacturer, carry out all tests at (23 ± 2) °C and at (50 ± 5) % RH. Condition all components at this temperature and relative humidity for at least 24 h prior to testing.

4.6 Preparation of material for testing

Manipulate all the components of the sealer in accordance with the manufacturer's instructions for use.

5 Test methods

5.1 Extraneous matter

Examine under normal visual acuity.

5.2 Flow

5.2.1 Apparatus

5.2.1.1 Two glass plates, at least 40 mm × 40 mm and approximately 5 mm thick, and with a mass of approximately 20 g.

5.2.1.2 A weight with a mass of approximately 100 g.

5.2.1.3 Graduated syringe, designed to deliver (0,05 ± 0,005) ml of mixed sealer.

5.2.2 Procedure

Place (0,05 ± 0,005) ml of sealer on the centre of one of the glass plates using the graduated syringe.

NOTE An alternative method is to use the mass of the sample, having first determined the density, and use the formula:

$$m = V \times d$$

where

m is mass;

d is density;

V is volume.

At (180 ± 5) s after the commencement of mixing, place the second glass plate centrally on top of the sealer and an additional mass on the plate to total (120 ± 2) g. Ten minutes after the commencement of mixing, remove the weight and measure the maximum and minimum diameters of the compressed disc of sealer. If the diameters are within 1 mm of each other, record the mean of the two diameters. If the two diameters are not within 1 mm of each other, repeat the test.

5.2.3 Calculation and expression of results

Carry out three determinations and calculate the mean value. Round the result to the nearest integer in millimetres and record it as the flow value.

5.3 Working time

In order to determine the working time for sealers whose claimed working time is up to 30 min, use the procedure set out in 5.2 with the exception that the load is applied 15 s before the end of the manufacturer's stated working time.

5.4 Setting time

5.4.1 Apparatus

5.4.1.1 Cabinet, capable of being maintained at (37 ± 1) °C and not less than 95 % RH.

5.4.1.2 Gilmore-type metric indenter, having a mass of (100,0 ± 0,5) g and a flat end of diameter (2,0 ± 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.

5.4.1.3 Moulds

a) For materials that do not require moisture for setting, a stainless steel ring mould, having an internal diameter of 10 mm and a height of 2 mm.

b) For materials that do require moisture for setting, a gypsum mould (complying with Type 2 of ISO 6873) incorporating a cavity with a diameter of 10 mm and a height of 1 mm.

NOTE This mould can be made by placing a plastics disc (D = 10 mm, h = 1 mm) on the bottom of a plastics cup (1 ml to 2 ml capacity) and filling the cup with freshly mixed gypsum. After the gypsum has set, the cup and disc are removed.

5.4.1.4 Metal block, with dimensions of at least 8 mm × 20 mm × 10 mm, conditioned at (37 ± 1) °C in the cabinet for at least 1 h.

5.4.1.5 Flat glass plate, approximately 1 mm thick.

NOTE A microscope slide is suitable.

5.4.2 Sample preparation

a) For materials not requiring moisture for setting, place the mould on the glass plate and fill it to a level surface with sealer. After (120 ± 10) s from the end of mixing, place this assembly on the metal block in the cabinet.

b) For materials that do require moisture for setting, store the gypsum mould at (37 ± 1) °C and 95 % RH for 24 h. After this time, fill the cavity in the preconditioned gypsum mould with the mixed sealer and place this assembly in the cabinet.

5.4.3 Procedure

When the setting time stated by the manufacturer approaches, carefully lower the Gilmore-type indenter vertically on to the horizontal surface of the sealer. If an indentation is visible, raise the needle, clean the needle tip and lower the needle to a new position on the surface of the sealer. Repeat this operation until indentations cease to be visible. Record the time, from the end of mixing, at which this occurs.

5.4.4 Calculation and expression of results

Carry out three determinations and calculate the mean value. Record this as the setting time.

5.5 Film thickness

5.5.1 Apparatus

5.5.1.1 Two optically flat square or circular glass plates, having a minimum uniform thickness of 5 mm and a contact surface area of approximately (200 ± 25) mm^2.

5.5.1.2 Loading device, to apply a force of (150 ± 3) N.

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

5.5.2 Procedure

Measure the combined thickness of the two glass plates in contact to an accuracy of 1 µm. Deposit a portion of sealer onto the centre of one of the glass plates. Place the other glass plate centrally on the sealer. After (180 ± 10) s from the start of mixing, carefully apply, by means of the loading device, a load of 150 N vertically on the top plate. Ensure that the sealer completely fills the area between the glass plates. After 10 min from the start of mixing, measure the combined thicknesses of the two glass plates and the film of sealer using the micrometer.

5.5.3 Calculation and expression of results

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

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

5.6 Solubility

5.6.1 Apparatus and materials

5.6.1.1 Two split ring moulds, having an internal diameter of (20 ± 1) mm and a height of (1,5 ± 0,1) mm, made of stainless steel or other materials compatible with the samples (such as polytetrafluoroethylene for resin-based sealers).

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

5.6.1.3 Plastics sheets impervious to water, such as polyethylene plastic, (50 ± 30) µm thick.

5.6.1.4 Two shallow dishes A and B, Petri or other suitable glass or porcelain, having a diameter of approximately 90 mm, with a minimum volume of 90 ml. The mass of dish B shall be known to the nearest 0,001 g.

5.6.1.5 Cabinet, capable of being maintained at (37 ± 1) °C and not less than 95 % RH.

5.6.1.6 Water, complying with grade 3 of ISO 3696.

5.6.1.7 Desiccator, containing phosphorus pentoxide or another suitable desiccant.

5.6.1.8 Heating oven, capable of being maintained at a temperature of (110 ± 2) °C.

5.6.2 Sample preparation

Prepare three specimens in accordance with one of the following methods.

a) For materials that do not require water for setting, place the mould (5.6.1.1) on a glass plate (5.6.1.2) and fill to slight excess with mixed sealer. Press another glass plate faced with a plastics sheet (5.6.1.3) on top

of the sealer and carefully remove the glass plate to leave a flat, uniform surface. Place the filled mould in the cabinet (5.6.1.5) for a period of time 50 % longer than the setting time stated by the manufacturer. Remove the specimen from the mould and determine the mass of sealer to the nearest 0,001 g.

NOTE It is necessary to make sure the specimens are completely set, otherwise removal from the mould can be difficult or impossible. Resin-based sealers need a few days for complete setting at 37 °C.

b) For materials that do require moisture for setting, place the mould on a glass plate. Mix 2 g of material according to the manufacturer's instructions with 0,02 ml or 0,02 g of water (5.6.1.6) and fill the mould to slight excess. Press another glass plate faced with a plastics sheet (5.6.1.3) on top of the sealer and place the mould in the cabinet for 24 h. Remove the specimens carefully from the mould and finish the periphery of the specimen to remove flash and irregularities. Determine the mass of the sealer to the nearest 0,001 g.

5.6.3 Procedure

Place two specimens in the shallow dish A (5.6.1.4) such that the surfaces do not touch and they remain undisturbed in the dish. Add (50 ± 1) ml of water and cover the dish. Place the dish in the cabinet for 24 h. Place a fluted filter into a funnel and place the funnel 20 mm above the bottom of dish B. Pour the water together with the specimens into the fluted filter. Wash the previously used dish A three times with 5 ml of water and pour the water into the fluted filter.

Place dish B, along with the collected water, in an oven at (110 ± 2) °C and evaporate the water to constant mass, cooling the dish in the desiccator (5.6.1.7) to room temperature before each weighing.

5.6.4 Calculation and expression of results

Record the difference between the original mass of the shallow dish and its final mass, to the nearest 0,001 g, as the amount of sealer removed from the specimens. Record this difference in mass, calculated as a percentage of the original combined mass of the two specimens, to the nearest 0,1 %.

Carry out the test twice and record the mean value as the solubility.

5.7 Radio-opacity

5.7.1 Apparatus

5.7.1.1 Stainless steel ring mould, having an internal diameter of 10 mm and a height of (1,00 ± 0,01) mm, with covers, made of either plastics, paper or other radiolucent material.

5.7.1.2 Single-phase dental X-ray unit, capable of operation at (65 ± 5) kV with suitable accessories.

5.7.1.3 Intra-oral X-ray occlusal film, of speed group D or E (as specified in ISO 3665), developing solution and fixer.

5.7.1.4 An aluminium step wedge (purity at least 98 % aluminium with a maximum copper content of 0,1 % and maximum iron content of 1 %), 50 mm long × 20 mm wide, having a thickness from 0,5 mm to 9,0 mm in equally placed steps of 0,5 mm or 1 mm measured to an accuracy of 10 µm.

5.7.1.5 Optical density instrument, capable of measuring in the range of 0,5 to 2,5.

5.7.2 Procedure

Place the sealer in the mould 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, adjacent to the step wedge. Place an equivalent cover under the step wedge.

Irradiate the specimen, step wedge and film with X-rays at (65 ± 5) kV at a target-film distance of 300 mm to 400 mm for sufficient time that the exposed and processed film under the 1 mm thick section of the step wedge has an optical density in the region of 0,5 to 2,5, including base and fog.

After developing, fixing and drying the exposed film, compare the density of the image of the specimen with that of the aluminium step wedge, using the optical density instrument. Express the radio-opacity equivalent of the specimens in millimetres of aluminium.

NOTE The optical densities of X-ray film images decrease with increasing radio-opacity.

5.7.3 Calculation and expression of results

If the numerical value of the optical density of the image of the specimen is less than the density of the 3 mm aluminium step, the sealer has a radio-opacity equivalent greater than 3 mm of aluminium.

6 Packaging, marking and information to be supplied by the manufacturer

6.1 General

Information additional to that specified in 6.2 and 6.3 may be supplied at the discretion of the manufacturer or as required by legislation.

6.2 Packaging

The components shall be supplied in securely sealed containers made of materials which do not react with, nor permit contamination of, the contents.

6.3 Marking

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

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

b) the name of the product;

c) the lot number (batch code) which refers to the manufacturer's record and date of manufacture for the particular batch of components;

d) the expiry date, expressed in accordance with ISO 8601;

e) the minimum net mass, in grams, of the powder or pastes and the minimum net volume, in millilitres, of the liquid, as applicable;

f) the recommended conditions of storage.

6.4 Manufacturer's information and instructions for use

Information and instructions shall accompany each package and shall include the following:

a) the name of the product;

b) the name and address of the manufacturer or the responsible distributor;

c) indications for use and clinical application of the sealer, including whether or not the sealer is suitable for use without obturating points;

d) the method of mixing and the component mixing ratio;

e) the working time and setting times (if claimed);

f) identification of any therapeutically active constituents present and referred to in the material claim for use;

g) the recommended conditions of storage;

h) whether the sealer can cause tooth staining, and any precautions necessary to minimize this effect;

i) special indications or warnings in respect of toxic, hazardous, flammable or tissue-irritant characteristics;

j) if sterility is claimed, appropriate marking on the package.

Bibliography

[1] ISO 7405, *Dentistry — Evaluation of biocompatibility of medical devices used in dentistry*

[2] ISO 10993-1, *Biological evaluation of medical devices — Part 1: Evaluation and testing within a risk management process*

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