INTERNATIONAL STANDARD

ISO 6876

Second edition
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Dental root canal sealing materials

Produits dentaires pour le scellement des canaux radiculaires

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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.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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

This second edition cancels and replaces the first edition (ISO 6876:1986), which has been technically revised.

Introduction

This International Standard was first published in 1986 (ISO 6876:1986). There were significant differences between ISO 6876 and the United States specification ANSI/ADA Specification No 57 (1983). In addition test houses had reported difficulties with some of the test procedures in the International Standard. In an attempt to harmonize the ISO and ANSI/ADA Standards and improve the test procedures, a planned programme of revision was commenced in 1991. The changes in this second edition are as follows:

The classification has been removed.

Non-setting materials are no longer covered by this International Standard.

The test procedures for flow, working time and setting time have been altered.

A new test to determine dimensional change following setting has been added.

For dental root canal sealers which require moisture to facilitate setting, a new test procedure to simulate the setting mode of the sealers in the root canal system has been included.

Dental root canal sealing materials

1 Scope

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

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 3665:1996, *Photography — Intra-oral dental radiographic film — Specification*.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

3 Terms and definitions

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

3.1
mixing time
that part of the working time specified or required in order to obtain a satisfactory mix of the components

3.2
working time
period of time, measured from the start of mixing, during which it is possible to manipulate the dental sealer without an adverse effect on its properties

3.3
setting time
period of time measured from the end of mixing until the sealer has set according to the criteria and conditions described in 7.4

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 visually free from extraneous matter when tested according to 7.1.

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 freedom from biological hazards are not included in this International Standard but it is recommended that in assessing possible biological or toxicological hazards reference be made to ISO 10993-1 and ISO 7405.

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 shall 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.

4.3 Physical and mechanical properties

4.3.1 Flow

When determined in accordance with 7.2, each disc shall have a diameter not less than 20 mm.

4.3.2 Working time

The minimum working time of a sealer, when determined by the method described in 7.3, shall be not less than 90 % of the working time stated by the manufacturer.

This test applies only to sealers having a working time claimed by the manufacturer to be less than 30 min.

4.3.3 Setting time

For sealers having a setting time of less than 30 min, the setting time of a sealer, when determined by the method described in 7.4, shall be no greater than 110 % of that stated by the manufacturer.

For sealers having a setting time greater than 30 min, and up to 72 h, for which the manufacturer quotes a time range, the setting time measured shall be within the range stated by the manufacturer.

4.3.4 Film thickness

Sealers shall have a film thickness of not more than 50 μm when tested in accordance with 7.5.

4.3.5 Dimensional change following setting

The mean dimensional change in length of the sealer, measured in accordance with the method set out in 7.6, shall not exceed 1,0 % in shrinkage or 0,1 % in expansion.

4.3.6 Solubility

The solubility of the set sealer, when determined in accordance with 7.7, shall not exceed 3 % mass fraction.

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

4.3.7 Radiopacity

The sealer, when tested in accordance with 7.8, shall have a radiopacity equivalent to not less than 3 mm of aluminium.

5 Sampling

The sample shall consist of 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.

6 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) %. The components shall be conditioned at this temperature and relative humidity for at least 24 h prior to testing.

7 Test methods

7.1 Extraneous matter

When examined under normal visual acuity, the components of the sealer shall show no evidence of any extraneous matter.

7.2 Flow

7.2.1 Apparatus

7.2.1.1 Two glass plates, at least 40 mm × 40 mm and approximately 5 mm thick.

NOTE The mass of one glass plate is approximately 20 g.

7.2.1.2 Weight, of mass approximately 100 g.

7.2.1.3 Graduated syringe, designed to deliver $(0{,}05 \pm 0{,}005)$ ml of mixed sealer.

7.2.2 Procedure

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

Place $(0{,}05 \pm 0{,}005)$ ml of sealer on the centre of one of the glass plates (7.2.1.1) using the graduated syringe (7.2.1.3). At (180 ± 5) s after the commencement of mixing, place the second glass plate centrally on top of the sealer, followed by the weight (7.2.1.2) to make a total mass on the plate of (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 agree to within 1 mm, record the mean of the two diameters. If the two diameters are not within 1 mm, repeat the test.

7.2.3 Treatment of results

7.2.3.1 Carry out three determinations. The result of each determination shall comply with the requirement in 4.3.1.

7.2.3.2 In addition for the purposes of 7.3.3, calculate the mean value of the three specimens to the nearest millimetre.

7.3 Working time

7.3.1 General

This test is applicable only to sealers having a working time claimed by the manufacturer to be less than 30 min.

7.3.2 Apparatus

This is as specified in 7.2.1.

7.3.3 Procedure

Using the working time stated by the manufacturer as a guide, repeat the test procedure as described in 7.2.2 but at increasing intervals of time between the commencement of mixing and the setting time stated by the manufacturer. On each occasion use freshly mixed sealer. When the specimen diameter is 10 % less than the flow value determined in 7.2.3.2, the working time has been determined.

7.3.4 Treatment of results

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

7.4 Setting time

7.4.1 Apparatus

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

7.4.1.2 Indenter, having a mass of $(100 \pm 0{,}5)$ g and a flat end of diameter $(2 \pm 0{,}1)$ mm. The indenter tip shall be cylindrical over a distance of at least 5 mm. The end of the indenter shall be plane and at right angles to the longitudinal axis.

7.4.1.3 Moulds

a) For materials that do not require moisture for setting, a stainless steel ring mould incorporating a cavity of internal diameter $d = 10$ mm and a height $h = 2$ mm.

b) For materials that do require moisture for setting, a dental plaster mould incorporating a cavity of dimensions ($d = 10$ mm, $h = 1$ mm).

NOTE This mould can be made by placing a plastics disc ($d = 10$ mm, $h = 1$ mm) on the bottom of a plastic cup (content 1 ml to 2 ml) and filling the cup with freshly mixed dental plaster. After setting of the dental plaster, the cup and the disc are removed.

7.4.1.4 Metal block, having minimum dimensions of 8 mm × 20 mm × 10 mm conditioned at (37 ± 1) °C in the cabinet (7.4.1.1) for at least 1 h.

7.4.1.5 Flat glass plate, approximately 1 mm thick, e.g. a microscope slide.

7.4.2 Sample preparation

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

a) For materials not requiring moisture for setting, place the mould (7.4.1.3) on the glass plate (7.4.1.5) and fill it to a level surface with mixed sealer. After (120 ± 10) s from the end of mixing, place this assembly on the metal block (7.4.1.4) in the cabinet.

b) For materials that do require moisture for setting, store the plaster of Paris mould at 37 °C and 95 % relative humidity for 24 h. After this time fill the cavity of the preconditioned dental plaster mould with the mixed sealer.

7.4.3 Procedure

When the setting time stated by the manufacturer approaches, carefully lower the indenter (7.4.1.2) vertically on to the horizontal surface of the sealer. Clean the indenter tip and repeat this operation until no indentations can be seen. Record the time, from the end of mixing, at which this occurs.

7.4.4 Treatment of results

Carry out three determinations. Each determination shall comply with 4.3.3.

7.5 Film thickness

7.5.1 Apparatus

7.5.1.1 Two optically flat square or **circular glass plates**, having a minimum uniform thickness of 5 mm and a contact surface area of approximately (200 ± 10) mm^2.

7.5.1.2 Loading device, to apply a load of (150 ± 3) N.

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

7.5.2 Procedure

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

Measure the combined thickness of the two glass plates (7.5.1.1) 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 (7.5.1.2), 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 thickness of the two glass plates and the film of sealer using the micrometer (7.5.1.3).

7.5.3 Treatment 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. Each determination shall comply with 4.3.4.

7.6 Dimensional change following setting

7.6.1 Apparatus and materials

7.6.1.1 Three split cylindrical moulds, having an internal diameter of 6 mm and a height of 12 mm, made of stainless steel or other materials compatible with the samples.

To facilitate the removal of the specimens that require moisture for setting, it is recommended to use a mould-release agent such as 3 % solution of polyvinyl ether wax in hexane.

7.6.1.2 Six flat glass plates, 25 mm × 75 mm × 1 mm thick, e.g. a microscope slide.

7.6.1.3 Plastic sheets impervious to water, e.g. polyethylene sheets (50 ± 30) μm thick.

7.6.1.4 Three 25 mm **C-clamps**.

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

7.6.1.6 Measuring instrument with an accuracy of 1 μm and capable of placing a restraint of no more than 0,1 N, uniformly distributed, over the ends of the specimen.

7.6.2 Sample preparation

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

a) For materials that do not require moisture for setting, mix 2 g of material according to the manufacturer's instructions. Place a mould (7.6.1.1) on a polyethylene sheet (7.6.1.3) backed by a glass plate (7.6.1.2) and fill to slight excess with sealer. Press another glass plate faced with polyethylene sheet on top of the sealer. Hold the mould and the plate together firmly with a C-clamp (7.6.1.4). Five minutes after beginning the mix, transfer the mould with the sealer and clamp to an atmosphere of 95 % to 100 % relative humidity at (37 ± 1) °C. For sealers that have setting times up to 2 h, three times the measured setting time according to 7.4 should be allowed before proceeding to the next step.

b) For materials that do require moisture for setting, place a mould on a polyethylene sheet backed by a glass plate. Mix 2 g of material according to the manufacturer's instructions together with 0,02 ml/0,02 g of water and fill the mould to slight excess. Press another glass plate faced with a sheet of plastic (7.6.1.3) on top of the sealer and continue as described in 7.6.2 a).

For both sample preparation methods, after the specimens have been prepared, grind the ends of the specimen flat by drawing the mould containing the specimen back and forth across fresh 600 grit wet sandpaper. Remove the specimen from the mould, measure the distance between the flat ends to an accuracy of 10 μm and store in distilled water at (37 ± 1) °C until ready for remeasurement. Thirty days after the specimen has been made, remeasure to an accuracy of 10 μm.

7.6.3 Treatment of results

Calculate the change in length as a percentage of the original length. Carry out the test three times and record the mean change in length as the dimensional change. To pass the test, the result shall comply with the requirement in 4.3.5.

7.7 Solubility

7.7.1 Apparatus and materials

7.7.1.1 Two split-ring moulds, having an internal diameter of (20 ± 1) mm and a height of $(1{,}5 \pm 0{,}1)$ mm.

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

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

7.7.1.4 Shallow dish, Petri or of other suitable glass or porcelain having a diameter of approximately 90 mm, with a minimum volume of 70 ml and of known mass to the nearest 0,001 g.

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

7.7.1.6 Water, complying with grade 3 of ISO 3696:1987.

7.7.1.7 Desiccator, containing phosphorus pentoxide or other suitable desiccant.

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

7.7.2 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 (7.7.1.1) on a glass plate (7.7.1.2) and fill to slight excess with sealer mixed in accordance with the manufacturer's instructions. Press another glass plate faced with a sheet of plastic (7.7.1.3) on top of the sealer and carefully remove the glass plate to leave a flat, uniform surface. Place the filled mould in the cabinet (7.7.1.5) 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.

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 together with 0,02 ml/0,02 g of water (7.7.1.6) and fill the mould to slight excess. Press another glass plate faced with a sheet of plastic on top of the sealer and place the mould in the cabinet for 24 h. Remove flash and irregularities carefully from the periphery of the specimen. Determine the mass of the sealer to the nearest 0,001 g.

7.7.3 Procedure

Place two such specimens in the shallow dish (7.7.1.4) so that they 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, and then remove the specimens. Wash the specimens with 2 ml to 3 ml of fresh water, recovering the washings in the shallow dish. Examine the washings in the dish. The presence of particles is evidence of disintegration and such material does not comply with 4.3.6.

Discard the specimens, evaporate the water from the dish without boiling and dry the dish to constant mass at (110 ± 2) °C, cooling the dish in the desiccator (7.7.1.7) to room temperature before each weighing (accurate to the nearest 0,001 g).

7.7.4 Treatment 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 procedure twice and record the mean value as the solubility. To pass the test, the results shall comply with the requirement in 4.3.6.

7.8 Radiopacity

7.8.1 Apparatus

7.8.1.1 Stainless steel ring mould, having an internal diameter of $(10 \pm 0{,}1)$ mm and a height of $(1 \pm 0{,}01)$ mm, together with covers, made of either plastic, paper or other radiolucent material.

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

7.8.1.3 Dental X-ray occlusal film, of speed group D or E as specified in ISO 3665:1996, developing solution and fixer.

7.8.1.4 Radiopacity gauge, consisting of 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, measured to an accuracy of 10 μm.

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

7.8.2 Procedure

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

Place the sealer in the mould (7.8.1.1) 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 (7.8.1.3) adjacent to the step wedge (7.8.1.4). 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 approximately 300 mm until the exposed and processed film under the 1 mm thick section of the step wedge reaches 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 (7.8.1.5). The radiopacity equivalent of the specimens is expressed in millimetres of aluminium.

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

7.8.3 Treatment 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 radiopacity equivalent greater than 3 mm aluminium, and complies with the requirement of 4.3.7.

8 Packaging, marking and information to be supplied by manufacturer

8.1 General

Information additional to that specified in 8.3 and 8.4 may be supplied at the discretion of the manufacturer or as required by legislation.

8.2 Packaging

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

8.3 Marking

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

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

b) name of the product;

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