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Dental resin-based pit and fissure sealants

Produits dentaires à base de résine pour comblement des puits et fissures

Reference number
ISO 6874 : 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 stndardization.

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 6874 was prepared by Technical Committee ISO/TC 106, *Dentistry.*

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Introduction

The efficacy of pit and fissure sealant materials for the prevention of dental caries is now widely accepted. The resin-based materials harden by free radical polymerization and protect teeth from the formation of caries in the pits and fissures.

Specific qualitative and quantitative requirements for freedom from biological hazard are not included in this International Standard, but it is recommended that reference should be made to ISO/TR 7405 : 1984, *Biological evaluation of dental materials* when assessing possible biological or toxicological hazards.

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Dental resin-based pit and fissure sealants

1 Scope

This International Standard specifies requirements and test methods for resin-based materials suitable for sealing pits and fissures in teeth.

This International Standard covers both chemically cured and external-energy-cured materials.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 2014 : 1976, *Writing of calendar dates in all-numeric form.*

ISO 7491 : 1985, *Dental materials — Determination of colour stability of dental polymeric materials.*

3 Classification

For the purposes of this International Standard, resin-based pit and fissure sealants are classified, according to the method of curing, as follows:

Type 1: Chemically cured

Type 2: External-energy-cured

4 Requirements

4.1 General

Sealants shall consist of one or more liquids, or of a combination of liquids and powders or pastes that, when mixed or activated in accordance with the manufacturer's instructions, fulfil the requirements laid down in 4.2, 4.3 and 4.4.

4.2 Condition of components

The components, when handled in accordance with the manufacturer's instructions [see 8.3c)], shall be of uniform consistency and colour, and shall be free from extraneous matter when inspected (see 7.1).

4.3 Set sealant

4.3.1 Appearance

The appearance of the set sealant shall be as described by the manufacturer [see 8.3a)] when inspected (see 7.1).

4.4 Physical properties

4.4.1 Working time, type 1 sealant

The working time for type 1 sealants, determined in accordance with 7.2, shall not be less than that stated by the manufacturer [see 8.3d)] or 45 s, whichever is the greater.

4.4.2 Setting time, type 1 sealant

The setting time for type 1 sealants, determined in accordance with 7.3, shall not differ by more than 30 s from that stated by the manufacturer [see 8.3 d)]; in no case shall it exceed 5 min.

4.4.3 Sensitivity to ambient light, type 2 sealant

The time at which the onset of polymerization is detectable for type 2 sealants, determined in accordance with 7.4, shall be not less than 25 s.

4.4.4 Curing time, type 2 sealant

The curing time (i.e. the exposure time required for application of the external source to cause complete setting) for type 2 sealants, determined in accordance with 7.5, shall not exceed that stated by the manufacturer or 60 s, whichever is the lesser.

4.4.5 Depth of cure, type 2 sealant

The depth of cure for type 2 sealants, determined in accordance with 7.6, shall be not less than 1,5 mm.

4.4.6 Uncured film thickness

The uncured film thickness, determined in accordance with 7.7, shall be not more than 0,1 mm.

4.4.7 Biocompatibility

See the Introduction for guidance on biocompatibility.

5 Sampling

The test sample shall consist of a retail package, or packages, from the same batch containing sufficient material (a minimum of 30 g) to carry out the specified tests and repeat tests, if necessary.

6 Preparation of test specimens

6.1 Ambient conditions

All test specimens shall be prepared at (23 ± 1) °C and at a relative humidity of (50 ± 5)%.

6.2 Procedure

Prepare the sealant in accordance with the manufacturer's instructions [see 8.3b)].

7 Test methods

7.1 Inspection

Visually inspect the test specimens to determine compliance with requirements laid down in 4.2, 4.3.1 and clause 8.

7.2 Working time, type 1 sealant

7.2.1 Apparatus

Thermocouple apparatus, as shown in figure 1.

The apparatus consists of a piece of polyethylene tubing (A) located on a polyamide block (B) which has a hole into which is inserted a stainless steel tube (C) containing a silver brazed thermocouple (D).

The tube (A) is 6 mm long, 4 mm in internal diameter and has a wall thickness of 1 mm. The locating part of block (B) is 4 mm diameter and 2 mm high. When assembled the two components form a specimen well 4 mm deep × 4 mm in diameter.

The tube (C) is 2 mm in diameter and has a wall thickness of 0,25 mm. The thermocouple wires are led to the surface through a small hole in the conical part of the tube and silver soldered in a minor joint. The conical-shaped tip (which facilitates removal of the specimen after testing) protrudes 1 mm into the base of the specimen well. The tolerance for the wall thickness of the tube is ± 0,05 mm: the tolerance for the other dimensions is ± 0,2 mm.

The thermocouple consists of wires (0,25 ± 0,05) mm in diameter, made of a material (e.g. copper/constantan) capable of registering temperature changes in a specimen of setting material to an accuracy of 0,1 °C. The thermocouple is connected to an instrument (e.g. voltmeter or chart recorder) capable of recording the temperature to that accuracy.

7.2.2 Procedure

Prepare the sealant in accordance with the manufacturer's instructions (see 8.3) and start timing from the moment mixing is begun. Maintain the mould at (23 ± 1) °C; 30 s after the start of mixing, place the mixed sealant in the mould and record the temperature, T_1, of the sealant. Maintain the apparatus at (23 ± 1) °C and continuously record the temperature of the sealant until the peak temperature is reached.

NOTE — A typical recording trace is shown in figure 2. As soon as the sealant is inserted into the mould, the temperature falls slightly until it becomes steady at T_0 and then starts to increase. The point at which the temperature begins to increase denotes the start of the setting reaction and, therefore, the end of the working time. The results are extremely temperature-dependent and slight variations within the permitted temperature range will cause variations of several seconds.

Record the time, t_w, from the start of mixing until the temperature starts to increase.

Carry out five determinations and calculate the mean time to the nearest 10 s as the working time of the sealant.

7.3 Setting time, type 1 sealant

7.3.1 Apparatus

Thermocouple apparatus, as specified in 7.2.1.

7.3.2 Procedure

Repeat the procedure described in 7.2.2, but maintain the apparatus at (37 ± 1) °C in air.

Record the time elapsed between the start of mixing and the peak temperature, T_2, as the setting time, t_s.

7.4 Sensitivity to ambient light, type 2 sealant

7.4.1 Apparatus

a) **Lamp,** as specified in ISO 7491 and having an illuminance of (8 000 ± 500) lx.

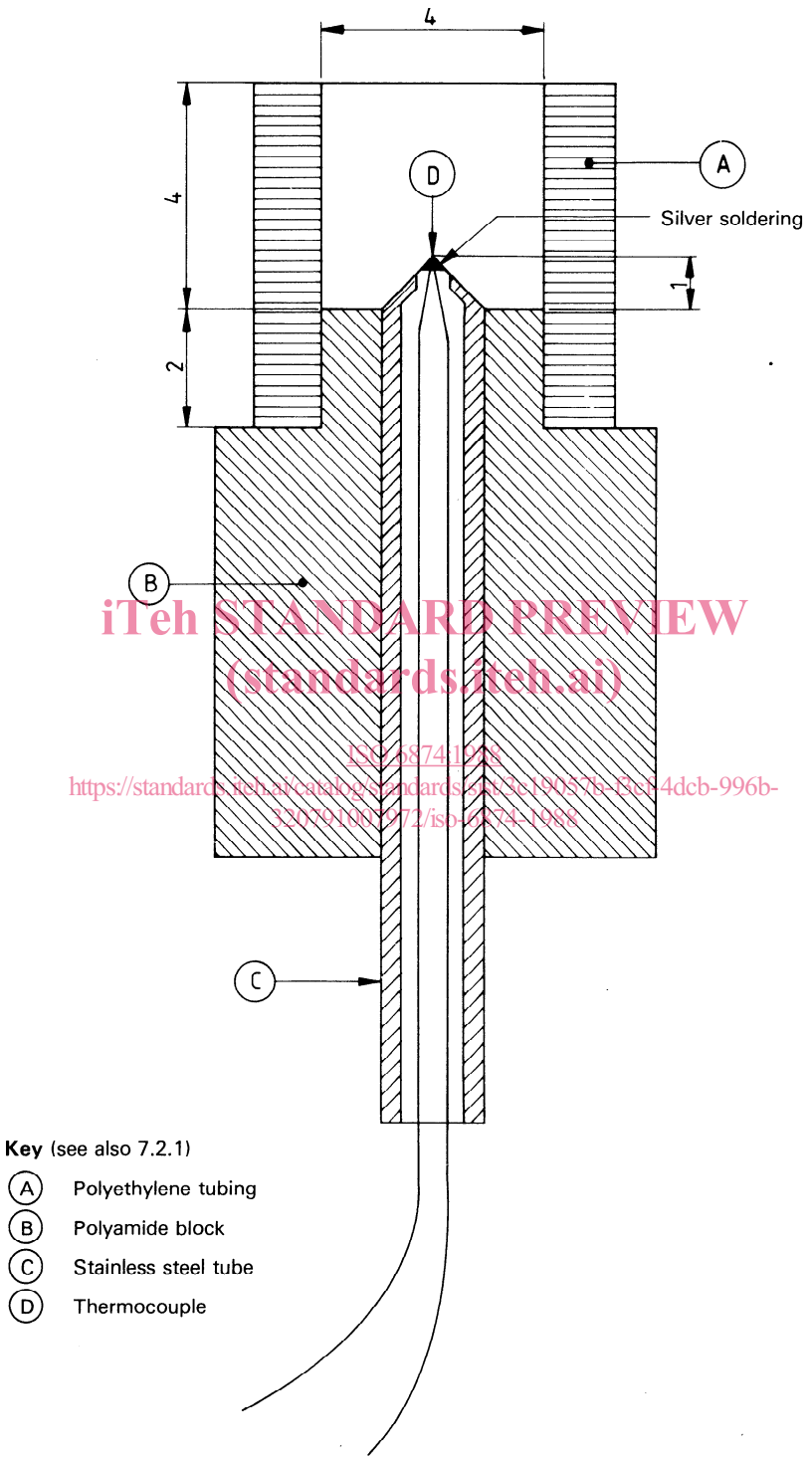
b) **Luxmeter,** having a photometric accuracy of at least ± 5 %.

c) **Thermocouple apparatus,** as specified in 7.2.1, except that the depth of the mould is 2 mm.

7.4.2 Procedure

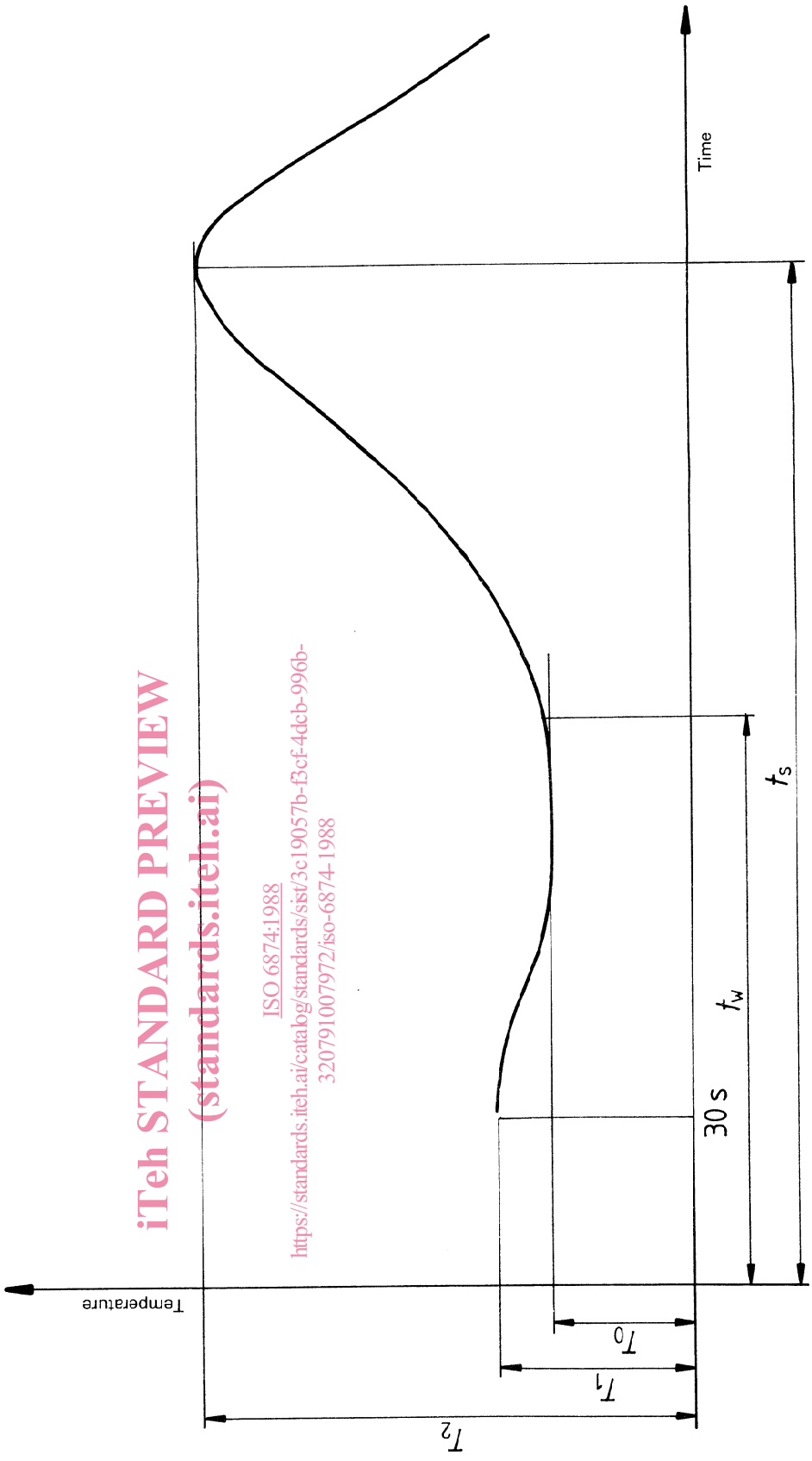
In a dark room, position the light source (7.4.1.1) perpendicular to the opening of the mould and arrange it so that the illumination level at the opening of the mould is (8 000 ± 500) lx. Place

Dimensions in millimetres



NOTE — Dimensional tolerances shall be ± 0,2 mm, except for the wall thickness of the tube (see 7.2.1).

Figure 1 — Apparatus for determination of working and setting times



NOTE — The typical recording trace illustrated shows the temperature at the time of insertion, T_1, the temperature after a slight drop immediately after insertion, T_0, and the peak temperature, T_2. The initial time of temperature increase, t_w, denotes the start of the setting reaction and, therefore, the end of working time. The time of peak temperature, t_s, denotes the setting time.

Figure 2 — Typical recording trace showing temperature changes in time for determination of working and setting times

the sealant in the mould at (23 ± 1) °C and place the mould under the light source. Record the time from the beginning of illumination to the start of deviation from a linear increase in temperature.

Repeat this procedure four more times.

If the time recorded for at least four of the five specimens is not less than 25 s, the material complies with the requirement laid down in 4.4.3.

7.5 Curing time, type 2 sealant

7.5.1 Apparatus

a) **Thermocouple apparatus,** as specified in 7.2.1.

b) **External energy source,** as specified by the manufacturer [see 8.3e)].

c) **Film,** which is transparent to the curing radiation (e.g. polyester).

d) **Microscope slides,** of sufficient area to cover one end of the mould.

7.5.2 Procedure

Place the sealant, prepared in accordance with the manufacturer's instructions (see 8.3), in the mould and maintain the apparatus at (37 ± 1) °C. Take care to exude air bubbles and slightly overfill the mould. Press the mould and a strip of the film (7.5.1.3) under a microscope slide (7.5.1.4) to exude excess material. Remove the microscope slide, leaving the film in place, and gently place the exit window of the energy source (7.5.1.2) against the film. Irradiate the sealant for the time recommended by the manufacturer [see 8.3e)]. Record the period from the time when the energy source is turned on to the time when the peak temperature occurs. Repeat this procedure four more times and calculate the mean of the five determinations as the curing time.

7.6 Depth of cure, type 2 sealant

7.6.1 Apparatus

a) **Apparatus,** as specified in 7.5.1.

b) **Micrometer,** accurate to 0,1 mm.

7.6.2 Procedure

After completing the procedure described in 7.5.2, remove the sealant from the mould. Remove the uncured surface film from the top and bottom of each specimen by wiping with a tissue. Using the micrometer (7.6.1.2), measure the height of the test specimen. Record this height as the depth of cure.

Carry out determinations on five test specimens.

If more than two specimens have a depth of cure less than 1,5 mm, the sealant fails to comply with the requirement laid down in 4.4.5.

7.7 Uncured film thickness

7.7.1 Principle

The refractive indices of cured and uncured monomers are different; a sharp line between the two phases can be detected by light microscopy.

7.7.2 Apparatus

a) **Light microscope,** having a minimum magnification of X50 and fitted with a micrometer eyepiece.

b) **External energy source,** as specified by the manufacturer for type 2 sealants [see 8.3e)].

c) **Microscope slide(s),** clean and dry.

d) **Glass coverslip(s).**

7.7.3 Procedure

Position a microscope slide (7.7.2.3) horizontally on the stage of the light microscope (7.7.2.1). 30 s after the start of mixing for type 1 sealants, but immediately for type 2 sealants, place a drop of sealant on the microscope slide and carefully place a glass coverslip (7.7.2.4) on it before the start of polymerization. The sealant disc should be circular or nearly circular; if it is not, discard it.

Expose type 2 sealants to the energy source (7.7.2.2) for the manufacturer's stated curing time [see 8.3e)].

After a further 5 min, inspect the periphery of the sealant disc and measure the distance, to an accuracy of 5 µm, from the dividing line between cured and uncured sealant and the outside of the sealant disc. Make four measurements at equidistant points around the sealant disc and record the mean value to the nearest 5 µm.

Carry out the procedure on five specimens and calculate the mean value to the nearest 10 µm.

If four of the five specimens show an uncured film thickness of not more than 0,1 mm, the material complies with the requirements laid down in 4.4.6.

8 Packaging, marking and instructions and information to be supplied by manufacturer

8.1 Packaging

The sealant materials shall be supplied in packaging that prevents contamination and deterioration of the contents.