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## Dentistry — Polymer-based luting materials containing adhesive components

*Médecine bucco-dentaire — Produits de scellement à base de  
polymères contenant des composants adhésifs*

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html).

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

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This corrected version of ISO TS 16506:2017 incorporates the following corrections.

- [Figure A.4](#) b) has been replaced.
- Additional minor editorial changes have been made.

## Introduction

This document provides test methods and information of performances for polymer-based restorative materials for luting which contain adhesive components. Test methods specified in this document used for a group of materials with varying compositions has proved difficult to set performance limits. Evidence is needed from using this document to develop it into an International Standard.

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

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# Dentistry — Polymer-based luting materials containing adhesive components

## 1 Scope

This document specifies test methods and information of bond strength to dentine and physical and chemical performances of dental polymer-based luting materials containing adhesive components. The materials are supplied in a form suitable for mechanical mixing or hand-mixing, including using auto-mixing tips, for self-curing and/or external energy activation, or non-mixing for external energy activation.

The polymer-based luting materials covered by this document are intended to be used for the cementation or fixation of restorations and appliances such as inlays, onlays, veneers, posts, crowns and bridges.

This document does not cover the following polymer-based luting materials:

- a) those which do not have an adhesive component within the structure of the material (see ISO 4049);
- b) those intended for veneering sub-frames (see ISO 10477).

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## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 6344-1, *Coated abrasives — Grain size analysis — Part 1: Grain size distribution test*

ISO 7491, *Dental materials — Determination of colour stability*

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

ISO 13116, *Dentistry — Test method for determining radio-opacity of materials*

## 3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

### 3.1 adhere

to be in a state of adherence

[SOURCE: ISO/TS 11405:2015, 3.1]

**3.2  
adherend**

body that is held or is intended to be held to another body by an *adhesive* (3.3)

[SOURCE: ISO/TS 11405:2015, 3.3]

**3.3  
adhesive**

substance capable of holding materials together by interfacial forces

**3.4  
bond strength**

force per unit area required to break a bonded assembly with failure occurring in or near the *adhesive* (3.3)/*adherend* (3.2) interface

[SOURCE: ISO/TS 11405:2015, 3.6]

**3.5  
opaque luting material**

intensely pigmented polymer-based luting material intended to mask underlying materials and tooth structure

[SOURCE: ISO 4049:2009, 3.1]

**3.6  
substrate**

material upon the surface of which an *adhesive* (3.3) is spread for any purpose, such as bonding or coating

[SOURCE: ISO/TS 11405:2015, 3.8]

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**4 Classification** [ISO/TS 16506:2017  
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**4.1 Class 1:** materials whose setting is effected by mixing an initiator and activator (self-curing materials).

**4.2 Class 2:** materials whose setting is effected by the application of energy from an external source, such as visible light [external-energy-activated materials, see also 8.3 d)].

**4.3 Class 3:** materials whose setting is effected by the application of external energy and which also have a self-curing mechanism present (dual-cure materials).

**5 Performance issues**

**5.1 Biocompatibility**

See the Introduction for guidance on biocompatibility. Further information is available in ISO 10993-1 and ISO 7405.

**5.2 Bond strength and physical and chemical performances**

This document does not specify any limit values of bond strength and physical and chemical performances. When such properties are tested, refer to [Annex C](#).



## 6 Sampling

The test sample shall consist of packages prepared for retail sale from the same batch or lot containing enough material to carry out the specified tests, plus an allowance for repeat tests, if necessary. 50 g should be sufficient.

## 7 Test methods

### 7.1 General

#### a) Reagent — Water

For the tests, use water prepared in accordance with ISO 3696 Grade 2.

#### b) Equipment

Validate all test equipment prior to use.

### 7.2 Test conditions

Unless specified otherwise, prepare and test all specimens at  $(23 \pm 2)$  °C. Control the relative humidity to ensure that it remains  $(50 \pm 20)$  % at all times. If the material was refrigerated, e.g. for storage, allow it to attain  $(23 \pm 2)$  °C.

For Class 3 materials, perform the tests for film thickness (see 7.5), working time (see 7.6) and setting time (see 7.7) in the absence of activating radiation.

Ambient light, both natural and artificial, is capable of activating Class 2 and Class 3 materials. For good control, the test should be performed in a darkened room with any artificial light filtered by a yellow filter.<sup>1)</sup>

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### 7.3 Inspection

Inspect visually to check that requirements specified in [Clause 8](#) have been met.

### 7.4 Preparation of test specimens

For the preparation of Class 2 and Class 3 materials, refer to the manufacturer's instructions for use [see 8.3 d)] that states the external energy source or sources recommended for the materials to be tested. Ensure that the source is in a satisfactory operating condition.

NOTE ISO 10650 gives guidance on this.

Mix or otherwise prepare the material in accordance with the manufacturer's instructions for use and the test conditions specified in 7.2.

When fully cured specimens are required for testing (7.10 to 7.12), ensure that the specimens are homogeneous after removal from the mould. Discard any specimens containing clefs, voids, discontinuities or air inclusions when inspected visually without magnification.

1) Polyester filter 101, Lee Filters, Andover, Hants, UK is an example of a suitable product available commercially. This information is given for the convenience of the users of this document and does not constitute an endorsement of this product by ISO.

## 7.5 Film thickness

### 7.5.1 Apparatus

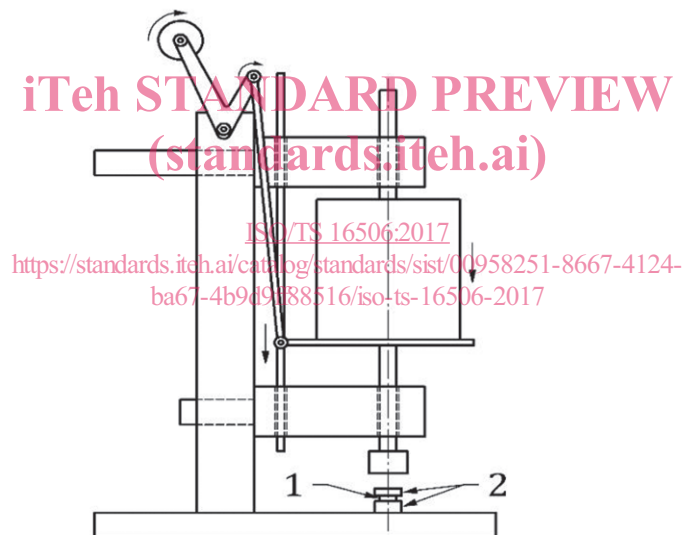
**7.5.1.1 Two glass plates**, optically flat, square or circular, each having a contact surface area of  $(200 \pm 25) \text{ mm}^2$  and a uniform thickness not less than 5 mm.

**7.5.1.2 Loading device**, of the type illustrated in [Figure 1](#) or an equivalent means, whereby a force of  $(150 \pm 2) \text{ N}$  can be applied vertically to the specimen via the upper glass plate. In [Figure 1](#), the anvil that is attached to the bottom of the rod is horizontal and parallel to the base, such that the load can be applied smoothly and without rotation of the specimen.

**NOTE** A holder can be used to assist in the positioning of the plates. Such a device consists of a base-plate with three vertical pins to align circular plates or four pins to align square plates.

**7.5.1.3 External energy source** (for Class 2 and Class 3 materials), as recommended by the manufacturer for use with the test material.

**7.5.1.4 Micrometer**, accurate to at least  $0,5 \mu\text{m}$ .



#### Key

- 1 specimen
- 2 glass plates ([7.5.1.1](#))

**Figure 1 — Loading device for use in the film thickness test**

### 7.5.2 Test procedure

#### 7.5.2.1 Preliminary steps

Measure with the micrometer ([7.5.1.4](#)), to an accuracy of  $1,0 \mu\text{m}$ , the combined thickness of the two optically flat glass plates ([7.5.1.1](#)) stacked in contact (reading A). Remove the upper plate and place between  $0,02 \text{ ml}$  and  $0,10 \text{ ml}$  of the test material prepared in accordance with the manufacturer's instructions for use in the centre of the lower plate and centre the plate below the loading device ([7.5.1.2](#)) on its lower plate. Centre the second glass plate on the test specimen in the same orientation as in the original measurement.

### 7.5.2.2 Class 1 materials

At  $(60 \pm 2)$  s after the completion of mixing Class 1 materials, apply a force of  $(150 \pm 2)$  N vertically and centrally to the specimen via the top plate smoothly and in such a manner that no rotation occurs for  $(180 \pm 10)$  s. Ensure that the cement has completely filled the space between the glass plates. At least 10 min after the commencement of mixing, remove the plates from the loading device and measure the combined thickness of the two glass plates and the specimen film, again taking the reading in the centre of the plates (reading B).

Record the difference between reading A and reading B, to the nearest micrometre, as the film thickness of the material.

Carry out five determinations.

### 7.5.2.3 Class 2 and Class 3 materials

Immediately after dispensing Class 2 materials or at  $(60 \pm 2)$  s after the completion of mixing Class 3 materials, apply a force of  $(150 \pm 2)$  N vertically and centrally to the specimen via the top plate smoothly and in such a manner that no rotation occurs for  $(180 \pm 10)$  s. Ensure that the specimen has completely filled the space between the glass plates. After  $(180 \pm 10)$  s, release the load and irradiate the specimen through the centre of the upper glass plate for twice the exposure time recommended by the manufacturer.

NOTE This irradiation is not intended to cure the material totally, but to stabilize the specimen for measurement.

After the irradiation of Class 2 and Class 3 materials, remove the plates from the loading device and measure the combined thickness of the two glass plates and the specimen film, again taking the reading in the centre of the plates (reading B).

Record the difference between reading A and reading B, to the nearest micrometre, as the film thickness of the material.

Carry out five determinations.

Record the film thickness and report the values.

## 7.6 Working time

### 7.6.1 Apparatus

#### 7.6.1.1 Two glass microscope slides.

#### 7.6.1.2 Timer, accurate to 1 s.

### 7.6.2 Procedure

This test is required only for Class 1 and Class 3 materials.

At  $(60 \pm 2)$  s after the completion of mixing, place a spheroidal mass of approximately 30 mg of material on a glass microscope slide (7.6.1.1) and immediately press the second microscope slide against the material using a shearing action to produce a thin layer.

Visually inspect the material to see whether it is recognizably homogeneous.

NOTE During this test, if the material has begun to set, clefs and voids will appear in the specimen when the thin layer is being produced. Alternatively, with rapid setting materials, there will be an increase in viscosity that will prevent the layer being produced.

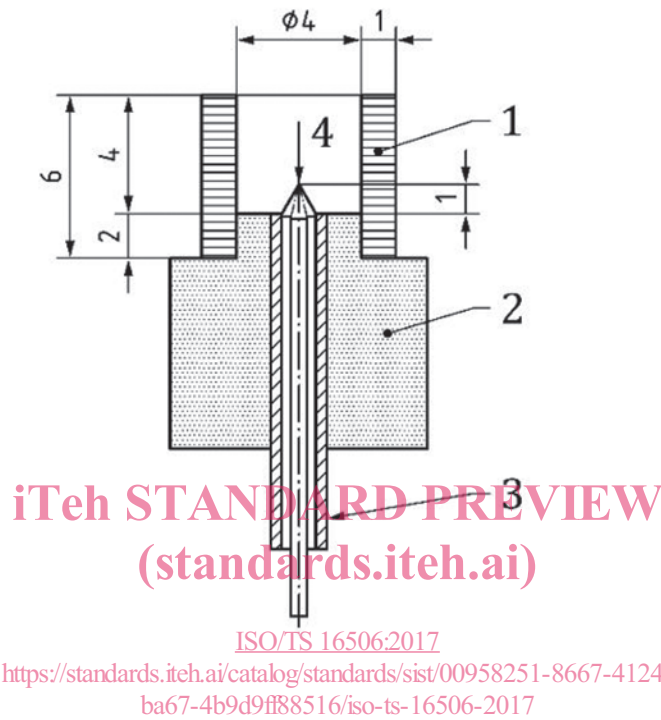
Repeat the entire procedure twice, using a new sample for each test.

Record the results of all three tests and report the results.

## 7.7 Setting time

### 7.7.1 Apparatus

7.7.1.1 Thermocouple apparatus, as shown in [Figure 2](#).



#### Key

- 1 polyethylene tubing
- 2 polyamide block
- 3 stainless steel tube
- 4 thermocouple with a cone of solder

**Figure 2 — Apparatus for determination of setting time (7.7)**

The apparatus consists of a piece of high density polyethylene (or similar material) tubing (key 1), located on a block of polyamide or similar material (key 2) having a hole into which is inserted a stainless steel tube (key 3) containing a stabilized thermocouple (key 4).

The tubing is 6 mm in length and 4 mm in internal diameter and has a wall thickness of 1 mm. The locating part of the polyamide block is 4 mm in diameter and 2 mm in height. When assembled, the two components form a specimen well 4 mm in height and 4 mm in diameter. In order to facilitate removal of the specimen after testing, the thermocouple has a conical tip which protrudes 1 mm into the base of the specimen well.

The tolerances on the above-mentioned dimensions are  $\pm 0,1$  mm.

The thermocouple consists of wires ( $0,20 \pm 0,05$ ) 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.

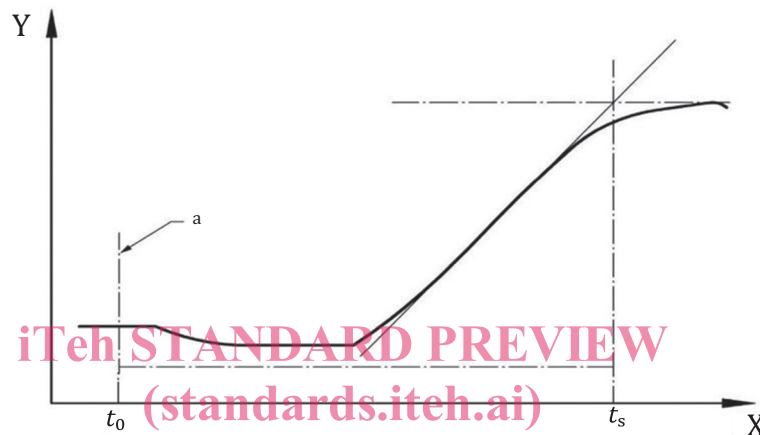
### 7.7.2 Procedure

This test is required only for Class 1 and Class 3 materials.

Prepare the test material in accordance with the manufacturer's instructions for use (see 8.3) and start timing from the moment mixing is begun, time  $t_0$ . Maintain the mould at  $(37 \pm 1)^\circ\text{C}$ , and immediately after the completion of mixing, place the mixed material in the mould and record the temperature of the material. Maintain the thermocouple apparatus (7.7.1.1) at  $(37 \pm 1)^\circ\text{C}$  and continuously record the temperature of the material until the maximum temperature has plateaued.

Extend the plateau backwards to meet an extension of the straight line of temperature increase. Record the time at the intersection of the two lines as  $t_s$  (see Figure 3).

Perform the test five times.



#### Key

- X time  
Y temperature  
a Start of mixing.

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NOTE  $t_s$  is determined by extending the plateau backwards to meet an extension of the straight line of temperature increase. This provides a distinct datum point.

**Figure 3 — Method for determining setting time**

Calculate the setting time,  $ST$ , using Formula (1):

$$ST = t_s - t_0 \quad (1)$$

where

$t_s$  is the time at the intersection of the two lines determined above;

$t_0$  is the starting time of mixing.

Record the setting times and report the results.