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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

Reference number
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Foreword

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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).

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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.

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

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.

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).

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]

4 Classification

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 ± 2) °C. Control the relative humidity to ensure that it remains (50 ± 20) % at all times. If the material was refrigerated, e.g. for storage, allow it to attain (23 ± 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.[1)]

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 clefts, 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

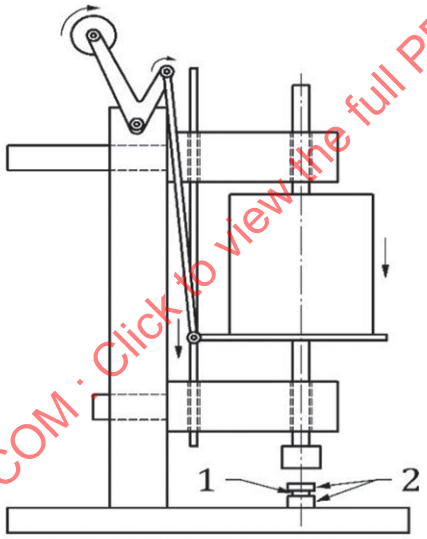
7.5.1.1 Two glass plates, optically flat, square or circular, each having a contact surface area of (200 ± 25) mm^2 and a uniform thickness not less than 5 mm.

7.5.1.2 Loading device, of the type illustrated in Figure 1 or an equivalent means, whereby a force of (150 ± 2) 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 µm.



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 µm, 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 ml and 0,10 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 ± 2) s after the completion of mixing Class 1 materials, apply a force of (150 ± 2) N vertically and centrally to the specimen via the top plate smoothly and in such a manner that no rotation occurs for (180 ± 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 ± 2) s after the completion of mixing Class 3 materials, apply a force of (150 ± 2) N vertically and centrally to the specimen via the top plate smoothly and in such a manner that no rotation occurs for (180 ± 10) s. Ensure that the specimen has completely filled the space between the glass plates. After (180 ± 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 ± 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, clefts 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.

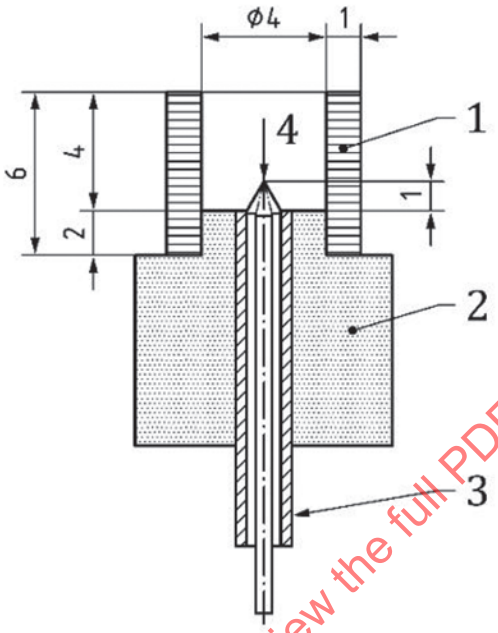
Repeat the entire procedure twice, using a new sample for each test.

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 ±0,1 mm.

The thermocouple consists of wires (0,20 ± 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.

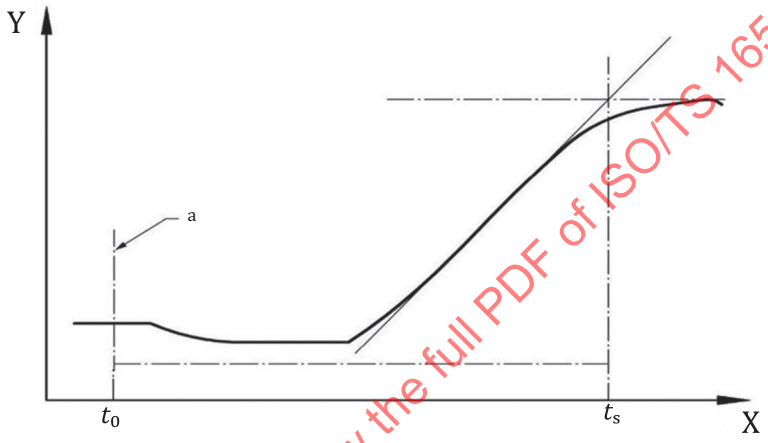
7.7.2 Procedure

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 ± 1) °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 ± 1) °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.

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.

7.8 Sensitivity to ambient light

7.8.1 Apparatus

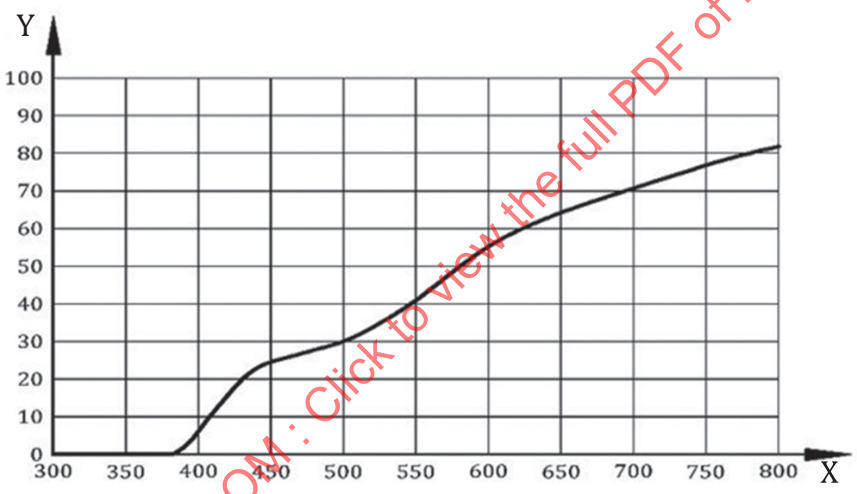
7.8.1.1 Xenon lamp, or radiation source of equivalent performance (a suitable apparatus is described in ISO 7491) with colour conversion and ultraviolet filters inserted.

The colour conversion filter has an internal transmittance that is within ±10 % of that shown in Figure 4.

NOTE A suitable conversion filter for photography is commercially available.

The ultraviolet filter is made of borosilicate glass with a transmittance less than 1 % below 300 nm and more than 90 % above 370 nm.

The purpose of the filter is to convert the spectrum of the incandescent light source (e.g. xenon radiation or equivalent), to that approximating a dental operating light. The filters and the output of the light should be checked periodically to ensure that the colour temperature at the luxmeter cell is 3 600 K to 6 500 K. Preferably, when the operating light is adjusted to the maximum illuminance level, the correlated colour temperature should be between 4 500 K and 6 400 K.



Key

X wavelength, in nanometres

Y internal transmission, T_i

Figure 4 — Internal transmittance for colour conversion filter

7.8.1.2 Two glass microscope slides/plates.

7.8.1.3 Illuminance measuring device, e.g. luxmeter, capable of measuring illuminance of (8 000 ± 1 000) lx.

7.8.1.4 Adjustable table, capable of adjusting a height of the light-accepting cell of the illuminance-measuring device (7.8.1.3).

7.8.1.5 Matt black cover, for the luxmeter cell.

NOTE The cover is used to prevent reflection from the cell interfering with the observation of the specimen.

7.8.1.6 Timer, accurate to 1 s.

7.8.2 Procedure

This test is required only for Class 2 and Class 3 materials.

In a dark room, position the illuminance-measuring device cell (7.8.1.3) under the xenon lamp (7.8.1.1) with colour conversion and ultraviolet filters inserted at such a height as to provide an illuminance of (8 000 ± 1 000) lx, using the adjustable table (7.8.1.4).

Cover the cell with the matte black cover (7.8.1.5). Place a spheroidal mass of approximately 30 mg of material on a glass microscope slide (7.8.1.2), position the slide on top of the cell and expose it to the light for (60 ± 5) s. Remove the slide with the sample from the irradiated area 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, discontinuities and voids will appear in the specimen when the thin layer is being produced. It might aid the inspection to compare the test specimen with one that has been produced in the absence of light.

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

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

7.9 Depth of cure

7.9.1 Apparatus

7.9.1.1 Mould, made of stainless steel or other opaque material which is verified that the equivalent results as stainless steel are obtained, for the preparation of a cylindrical specimen, 6 mm in length and 4 mm in diameter.

NOTE When the luting materials bond to the mould (7.9.1.1), a mould release agent which does not interfere with the setting reaction (for example, a 3 % solution of polyvinyl ether wax in hexane) can be used to facilitate removal of the specimen.

7.9.1.2 Two glass slides/plates, each of sufficient area to cover one side of the mould.

NOTE Standard glass microscope slides can be used.

7.9.1.3 White filter paper.

7.9.1.4 Film, transparent to the activating radiation, (50 ± 30) µm thick, e.g. polyester.

7.9.1.5 External energy source, as recommended by the manufacturer for use with the test material [see 8.3 d)].

7.9.1.6 Micrometer, accurate to 0,01 mm.

7.9.1.7 Plastic spatula.

7.9.2 Procedure

This test is required only for Class 2 materials.

Place the mould (7.9.1.1) on a strip of the transparent film (7.9.1.4) on a glass slide (7.9.1.2). Fill the mould with the test material, prepared in accordance with the manufacturer's instructions for use, taking care to exclude air bubbles. Slightly overfill the mould and put a second strip of the transparent film on top, followed by the second glass slide. Press the mould and strips of film between the glass slides

(7.9.1.2) to displace excess material. Place the mould on the filter paper (7.9.1.3), remove the glass slide covering the upper strip of film and gently place the exit window of the external energy source (7.9.1.5) against the strip of film. Irradiate the material for the time which the manufacturer claims is required to achieve a depth of cure of at least 0,5 mm for opaque materials or 1,5 mm for other materials.

Immediately after completion of irradiation, remove the specimen from the mould and remove the uncured material with the plastic spatula (7.9.1.7). Measure the maximum height of the cylinder of cured material using the micrometer (7.9.1.6) to an accuracy of ±0,1 mm and divide the value by two.

Record this value as the depth of cure.

Repeat the test twice.

Report the values.

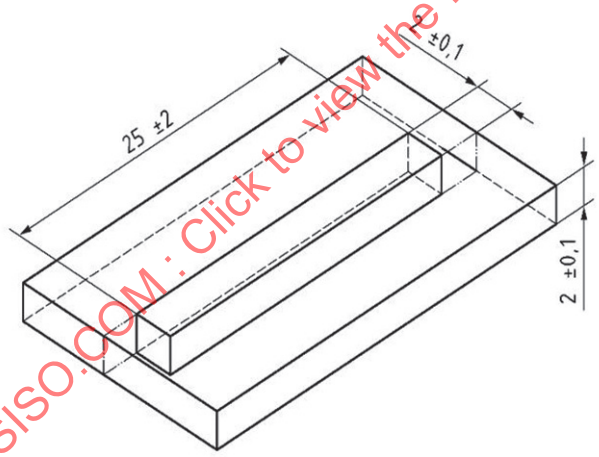
7.10 Flexural strength

7.10.1 Apparatus

7.10.1.1 Mould, made of stainless steel or other material which is verified that the equivalent results as stainless steel are obtained, for the preparation of a test specimen (25 ± 2) mm × (2,0 ± 0,1) mm × (2,0 ± 0,1) mm. A suitable mould is illustrated in Figure 5.

NOTE A mould made of modified poly-tetrafluoro-ethylene (m-PTFE) by injection moulding can be used.

Dimensions in millimetres



NOTE When the luting material has ability to bond to the mould (7.10.1.1), a mould release agent (see 7.9.1.1, note) can be used.

Figure 5 — Mould for flexural strength test specimens

7.10.1.2 Two metal plates, each of sufficient area to cover the mould (7.10.1.1).

7.10.1.3 Glass microscope slide, for use during polymerization of Class 2 and Class 3 materials.

7.10.1.4 Small screw clamp capable of exerting pressure on the metal plates (7.10.1.2) during specimen preparation.

7.10.1.5 Film, transparent to the activating radiation, (50 ± 30) µm thick, made of, for example, polyester.

7.10.1.6 White filter paper.

7.10.1.7 Water bath, capable of the water being maintained at (37 ± 2) °C.

7.10.1.8 External energy source(s), (for Class 2 and Class 3 materials) as recommended by the manufacturer for use with the test material [see 8.3 d)].

7.10.1.9 Micrometer, accurate to at least 0,005 mm.

7.10.1.10 Flexural strength test apparatus, appropriately calibrated, to provide a constant cross-head speed of (0,75 ± 0,25) mm/min or a rate of loading of (50 ± 16) N/min.

The apparatus consists essentially of two rods (2 mm in diameter), mounted parallel with (20 ± 0,1) mm between centres, and a third rod (2 mm in diameter) centred between, and parallel to the other two, so that the three rods in combination can be used to give a three-point loading to the specimen.

7.10.2 Preparation of test specimens

7.10.2.1 Class 1 and Class 3 materials

Cover one of the metal plates (7.10.1.2) with the filter paper (7.10.1.6) followed by the film (7.10.1.5) and position the mould (7.10.1.1) upon it. Prepare the material in accordance with the manufacturer's instructions for use and immediately place it as evenly as possible without bubbles or voids in the mould with a slight excess. Place a second piece of film on the material in the mould and cover this with the second metal plate.

Apply pressure to displace the excess material by means of the clamp (7.10.1.4) for 1 min. After the manufacturer's recommended curing time, place the assembly in water in the water bath (7.10.1.7), at (37 ± 2) °C.

After 60 min from the start of mixing, separate the mould and remove the specimen carefully. Inspect the specimen visually for any bubbles, voids or other defects. If there are any such defects, discard the specimen and make a new one. Remove any flash by gentle abrasion with 320-grit abrasive paper. Store the specimen in water [7.1 a)] at (37 ± 2) °C until the start of testing (see 7.10.3).

Prepare five specimens.

7.10.2.2 Class 2 and Class 3 materials

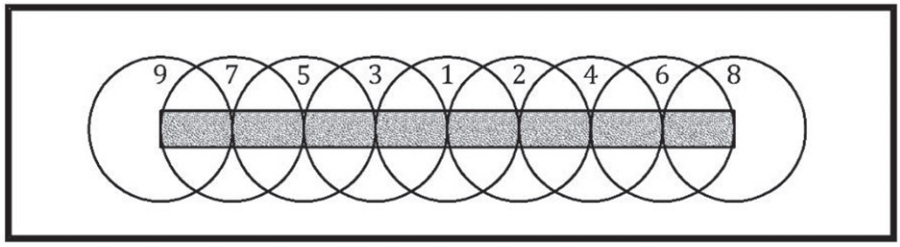
Prepare the material in accordance with the manufacturer's instructions for use and fill the mould with the material, as described in 7.10.2.1. Replace one of the metal plates with a glass slide (7.10.1.3) and place the exit window of the external energy source (7.10.1.8) at the centre of the specimen and against the glass plate.

Irradiate that section of the specimen for the recommended exposure time. Move the exit window to the section next to the centre, overlapping the previous section by half the diameter of the exit window and irradiate for the appropriate time. Irradiate the section on the other side of the centre in the same way. Continue this procedure until the entire length of the specimen has been irradiated (see Figure 6).

Turn the mould over, remove the other metal plate and replace it with a glass slide. Repeat the irradiation procedure on this side of the specimen. Place the assembly in water in the water bath at (37 ± 2) °C for 15 min.

Remove the specimen from the mould and carefully remove any flash by gently abrading it with 320-grit abrasive paper. Store the specimen in water [7.1 a)] at (37 ± 2) °C until the start of testing (see 7.10.3).

Prepare five specimens.



NOTE In the example, the diameter of the irradiation window of the external energy source is 7 mm.

Figure 6 — Schematic diagram of overlapping irradiation zones for the preparation of the flexural strength specimens

7.10.3 Procedure

Measure the dimensions of the specimen at its centre to an accuracy of 0,01 mm. Transfer the specimen to the flexural strength test apparatus (7.10.1.10).

At 24 h after the start of mixing (Class 1 materials) or irradiation (Class 2 and Class 3 materials), apply a load to the specimen at a cross-head speed of (0,75 ± 0,25) mm/min or at a rate of loading (50 ± 16) N/min until either the specimen reaches the yield point or, if there is no yield point, fractures.

If the manufacturer of Class 3 material indicates in the instructions for use or states on the package that the material can be used without irradiation of external energy, perform the test using the specimen cured only by the self-curing process (7.10.2.1).

Record the maximum load exerted on the specimen either at the yield point or at the point of fracture. Repeat the test on the four other specimens to obtain five data.

7.10.4 Treatment of results

Calculate the flexural strength, σ, in megapascals (MPa), from Formula (2):

$$\sigma = \frac{3Fl}{2bh^2} \tag{2}$$

where

F is the maximum load, in newtons, exerted on the specimen;

l is the distance, in millimetres, between the supports, accurate to 0,01 mm;

b is the width, in millimetres, at the centre of the specimen measured immediately prior to testing;

h is the height, in millimetres, at the centre of the specimen measured immediately prior to testing.

Report the values obtained.

7.11 Water sorption

7.11.1 Apparatus

7.11.1.1 Mould, of internal dimensions (15,0 ± 0,1) mm in diameter and (1,0 ± 0,1) mm in depth, for the preparation of specimen discs.

NOTE 1 A split ring or "washer" mould can be suitable.

NOTE 2 A mould release agent that does not interfere with the setting reaction (for example, a 3 % solution of polyvinyl ether wax in hexane) can be used to facilitate removal of the specimen.

7.11.1.2 Film, transparent to the activating radiation, e.g. made of polyester, (50 ± 30) µm thick.

7.11.1.3 Two metal plates, of sufficient area to cover the mould (7.11.1.1).

7.11.1.4 Glass microscope slide, for use during polymerization of Class 2 and Class 3 materials

7.11.1.5 Two desiccators, containing silica gel freshly dried for 5 h at 130 °C.

7.11.1.6 External energy source(s), (for Class 2 and Class 3 materials), as recommended by the manufacturer for use with the material to be tested [see 8.3 d)].

7.11.1.7 Oven, capable of the air being maintained at (37 ± 2) °C.

7.11.1.8 Analytical balance, accurate to 0,05 mg in the measuring range required in the test.

7.11.1.9 Micrometer, accurate to at least 0,005 mm.

7.11.1.10 Clamps.

7.11.1.11 Plastics tweezers.

NOTE To avoid contamination, handle the specimens at all times using tweezers.

7.11.1.12 Hand dust blower or source of oil-free compressed air, with micro-jet nozzle.

7.11.2 Preparation of test specimens

7.11.2.1 Class 1 materials

Place a piece of film (7.11.1.2) on one of the metal plates (7.11.1.3) and place the mould (7.11.1.1) upon it. Slightly overfill the mould with the material prepared in accordance with the manufacturer's instructions for use. Place a second piece of film on the material in the mould and cover this with the second metal plate, thus displacing excess material.

Clamp the mould together and transfer the assembly immediately to the oven (7.11.1.7) at (37 ± 2) °C. At 60 min from the start of mixing, remove the specimen from the mould, taking care to avoid surface contamination. Finish the periphery of the specimen to remove flash and irregularities. Hold the periphery of the specimen against 1 000 grit abrasive paper on a non-rotating grinding table and rotate the specimen so that the periphery is abraded. Visually inspect the specimen periphery to ensure it is smooth. Blow debris away with the compressed air jet or dust blower (7.11.1.12). Ensure the diameter of the finished specimen is not less than 14,8 mm.

Prepare five specimen discs in this way.

7.11.2.2 Class 2 and Class 3 materials

Prepare the material in accordance with the manufacturer's instructions for use and fill the mould with the material as described in 7.11.2.1 and, having displaced excess material, remove the metal plate, leaving the film in place, and replace it with the glass plate. For Class 2 and Class 3 materials, place the exit window of the external energy source (7.11.1.6) against the glass plate (See 7.11.1.4).

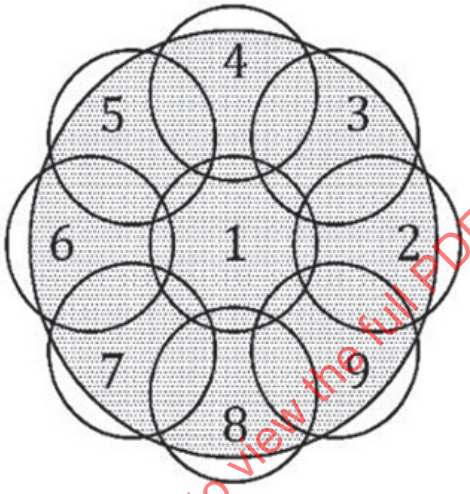
Irradiate that section of the specimen for the recommended exposure time. Move the exit window and irradiate a section of the specimen overlapping the previous section of the specimen. Continue this procedure until the whole specimen has been irradiated.

Turn the mould over, remove the other metal plate and replace it with a glass slide. Irradiate the second side of the specimen in the same way as the first side.

NOTE A template is useful in order to perform this overlapping irradiation efficiently and the exact number of exposures is dependent on the diameter of the exit window. Figure 7 shows an example of such a template.

Immediately after irradiation, transfer the mould to the oven (7.11.1.7) at (37 ± 2) °C. At 15 min after the commencement of irradiation, remove the specimen from the mould and finish the periphery as described in 7.11.2.1.

Prepare five specimen discs in this way.



NOTE For example, the diameter of the irradiation window of the external energy source is 7 mm.

Figure 7 — Schematic diagram of overlapping irradiation zones for the preparation of the water sorption specimens

7.11.3 Procedure

7.11.3.1 Pre-conditioning in dry state

a) Transfer the specimens to one of the two desiccators (7.11.1.5) at (37 ± 2) °C.

b) After 22 h, remove the specimens, store them in the second desiccator at (23 ± 1) °C for 2 h and weigh them to an accuracy of 0,1 mg. Replace the silica gel with freshly dried gel after each weighing sequence.

c) Repeat this cycle [a) and b)] until a constant mass, m_1, is obtained, i.e. until the mass loss of each specimen is not more than 0,1 mg over any 24 h period.

NOTE Approximately two to three weeks might be necessary to achieve constant mass.

7.11.3.2 Volume measurement

a) After final drying, take two measurements of the diameter of the specimen, at right angles to each other, to an accuracy of 0,01 mm and calculate the mean diameter, d.

b) Measure the thickness of the specimen to an accuracy of 0,01 mm at its centre and at four equally spaced points on the circumference and calculate the mean thickness, t.

c) Calculate the volume, V, in cubic millimetres, from the mean diameter and the mean thickness, using Formula (3):

$$V = \pi t \left(\frac{d}{2} \right)^2 \quad (3)$$

where

V is the volume of the disc, in cubic millimetres;

t is the mean thickness, in millimetres;

d is the mean diameter, in millimetres.

7.11.3.3 Immersion in water

a) Immerse the specimens in water [7.1 a)] at (37 ± 2) °C for 7 d such that they are vertical and have a minimum of 3 mm separation between them.

A rack should be used to do this effectively.

Ensure that the volume of water for immersion of the specimens is at least 10 ml per specimen.

b) After 7 d, remove the specimens, rinse with water [(7.1 a)], blot away surface water until free from visible moisture, wave in the air for 15 s, and weigh 1 min after removal from the water. Record this mass as m_2.

7.11.3.4 Treatment of results

Calculate W_{sp} from Formula (4):

$$W_{sp} = \frac{m_2 - m_1}{V} \quad (4)$$

where

m_1 is the mass of the conditioned specimen, in micrograms (see 7.11.3.1);

m_2 is the mass of the specimen, in micrograms, after immersion in water for 7 d (see 7.11.3.3);

V is the volume of the specimen, in cubic millimetres (see 7.11.3.2).

Report the results.

7.12 Colour stability

7.12.1 General

The test is intended to demonstrate the colour stability of a material after xenon irradiation and water sorption, by comparing an irradiated specimen and a non-irradiated, water-immersed specimen with a dry reference specimen.

7.12.2 Apparatus

7.12.2.1 Oven, capable of the air being maintained at (37 ± 2) °C.

7.12.2.2 Radiation source, **water bath and other apparatus**, as described in ISO 7491.

7.12.3 Preparation of test specimens

Prepare three disc specimens as described in 7.11.2.1 for Class 1 materials or 7.11.2.2 for Class 2 and Class 3 materials but omitting the precise finishing of the periphery.

7.12.4 Procedure

— **Specimen 1**: after removal from the mould, store one specimen dry in the dark in the oven (7.12.2.1) at (37 ± 2) °C for 7 d; this is the reference specimen.

— **Specimen 2**: after removal from the mould, store one specimen in the dark in the oven (7.12.2.1) in water (7.1) at (37 ± 2) °C for 7 d; this specimen will demonstrate any deterioration in colour due to water sorption alone.

— **Specimen 3**: after removal from the mould, store one specimen dry in the dark in the oven (7.12.2.1) at (37 ± 2) °C for (24 ± 2) h. After this time, remove the specimen from the oven and blank off half of it with aluminium or tin foil. Place this specimen in the radiation chamber (7.12.2.2), immerse it in water at (37 ± 5) °C and expose it to the radiation for 24 h. Ensure that the water level is (10 ± 3) mm above the specimen. After exposure, remove the metal foil and transfer the specimen back to the oven at (37 ± 2) °C and store it dry in the dark for 5 d.

7.12.5 Colour comparison

After 7 d, remove specimen 1 and specimen 3 from the oven. Compare the colour of water-absorbed specimen 2 (See 7.12.4) with the reference specimen 1.

Compare the colour of both halves of specimen 3 with each other and with the reference specimen 1.

Carry out the colour comparison in accordance with ISO 7491.

Report the results obtained.

7.13 Radio-opacity

7.13.1 Preparation of test specimen

Prepare one disc specimen with its thickness in the range (1,0 ± 0,1) mm as described in 7.11.2.1 for Class 1 materials or 7.11.2.2 for Class 2 and Class 3 materials, omitting the precise finishing of the periphery.

7.13.2 Test procedure

Determine the radio-opacity of the material in accordance with ISO 13116 using the specimens prepared in accordance with 7.13.1.

Report the values obtained.

8 Packaging, marking, instructions and information to be supplied

NOTE Additional information can be included at the discretion of the manufacturer. Regulations might also require additional information to be supplied.

8.1 Packaging

The materials shall be supplied in containers or capsules that afford adequate protection and have no adverse effect on the quality of the contents. For the purposes of this document, the container or capsule shall be considered to be the immediate wrapping of the material.

An outer pack may also be used to present the containers or capsules as a single unit that provides protection and/or marking for them.

8.2 Marking

NOTE Requirements for marking are summarized in Table 2.

8.2.1 Capsule or single-dose container

NOTE A single-dose container is, for example, a small syringe containing no more than 0,5 ml of material.

a) Each capsule or single-dose container shall be marked in order to identify the product.

b) If the product is supplied in pre-coloured shades, each capsule or single-dose container shall be marked or colour coded so that the shade of its contents can be identified.

c) Each capsule or single-dose container shall be identified with the lot or batch, consisting of a serial number or a combination of letters and numbers, that refers to the manufacturer's records for that particular lot or batch of material.

d) The expiry date expressed in accordance with ISO 8601 for the material, when stored under the manufacturer's recommended conditions, shall be marked on each capsule or single-dose container or, if not applicable, on the outer pack.

8.2.2 Multi-dose container

NOTE Multi-dose container is, for example, a dispensing syringe containing 2 g of the material.

The following information shall be clearly visible on each multi-dose container:

a) the trade name of the material;

b) identification of the shade, if the product is supplied in pre-coloured shades;

c) lot or batch identification, consisting of a serial number or a combination of letters and numbers, that refers to the manufacturer's records for that particular lot or batch of material;

d) the net mass, in grams, or net volume, in millilitres;

e) the expiry date, expressed in accordance with ISO 8601, for the material if stored under the manufacturer's recommended conditions.

8.2.3 Outermost package

The outermost package shall have the following information clearly visible:

a) the manufacturer's name and address or agent responsible for the country of sale;

b) the trade name of the material;

c) identification of the shade, if the product is supplied in pre-coloured shades;

d) recommended conditions of storage;

e) the net mass, in grams, or net volume, in millilitres and number of containers;

f) the expiry date for the material, expressed in accordance with ISO 8601, if stored under the manufacturer's recommended conditions [see 8.3, i)];

g) presence of hazardous substances indicated by text or symbol;

h) the legend “polymer-based dental adhesive luting material” or “resin-based dental adhesive luting material”.

Furthermore, the following information shall be clearly visible either on the outermost package or in the manufacturer’s instructions for use (see 8.3) or both:

i) the word “radio-opaque” if the material complies with the recommendation of C.2.2.9; if the manufacturer wishes to claim a specific value for radio-opacity, this shall be stated in the manufacturer’s instructions for use [see 8.3 m)] and shall be tested in accordance with 7.13 (which referrers ISO 13116);

j) a statement indicating whether the material is chemically activated, activated by external energy or dual cure;

k) a statement indicating whether the manufacturer claims colour stability;

l) manufacturer's lot or batch identification.

8.3 Manufacturer’s instructions and information for the user

The following information, together with a description of the product, shall accompany each individual pack of material:

a) the principal organic component of the polymer base;

b) the indications for clinical use;

c) instructions for the preparation and proportioning of components and mixing, including any necessary precautions regarding the manipulation of the material and, if appropriate, the maximum proportion of tints and blenders that may be used without detriment to the physical properties (the ambient conditions under which this should be carried out may be included);

d) recommended external energy source(s) and exposure/processing times for all versions of Class 2 and Class 3 materials and, for Class 3 material only, add a statement whether activation by external energy is mandatory or optional;

e) working time and setting time for Class 1 and Class 3 materials, and an indication of the time when a protective material, e.g. a matrix band, used to prevent an excess luting material from sticking to surrounding objects, may be removed if this differs from the setting time;

f) information on the use of a base or liner, if recommended, or other recommended protective measures if the material is likely to cause pulpal irritation, and information on whether any base or liner is known to be incompatible with the material (e.g. eugenol-containing materials);

g) recommended instructions for finishing;

h) recommended storage conditions (e.g. need for refrigeration) and the shelf life under those conditions of storage beyond which the material should not be used, making reference to the expiry date [see 8.2.1 d), 8.2.2 e), and 8.2.3 f)];

i) any pharmacologically-active ingredients, when present and referred to in the material claim for use;

j) special indications or warnings, when necessary, in respect of such properties as toxicity, hazard, flammability or tissue irritation, for both patient and operator;

k) whether colour stability is claimed by the manufacturer;

l) whether the material has the ability to bond to a metal;

m) whether radio-opacity is claimed by the manufacturer. If a specific value for radio-opacity is stated, an explanation of the radio-opacity value shall be included.

EXAMPLE Aluminium has a radio-opacity equivalent to that of dentine, thus a 1 mm thickness of material having a radio-opacity equivalent to a 1 mm thickness of aluminium has a radio-opacity equivalent to that of dentine.

Table 2 — Marking

Item No.	Information	Capsule or single dose container	Multi-dose container	Outermost package
1	Identification of the product	M	—	—
2	Trade name of the product	—	M	M
3	Identification of the shade, if the product is supplied in pre-coloured shades	M	M	M
4	The manufacturer's name and address or agent responsible for the country of sale	—	—	M
5	Recommended conditions of storage	—	—	M
6	Manufacturer's lot or batch identification	M	M	M
7	The expiry date, expressed in accordance with ISO 8601, for the material if stored under the manufacturer's recommended conditions	M[a]	M	M
8	The net mass, in grams, or net volume, in millilitres	—	M	M
9	The legend "polymer-based dental adhesive luting material" or "resin-based dental adhesive luting material"	—	—	M
10	Presence of hazardous substances indicated by text or symbol	—	—	M
11	The word "radio-opaque" if the material complies with the recommendations of C.2.2.9	—	—	OPT
12	A statement indicating whether the manufacturer claims colour stability	—	—	OPT

M Mandatory information.

OPT Optional information.

"—" Not-mandatory or not-optional information.

[a] Outer pack may be used for marking of expiry date.

Annex A
(informative)

Test methods to determine shear bond strength to dentine

A.1 General

This annex specifies test methods to determine shear bond strength between dental luting materials and tooth structure, dentine or enamel, for evaluation of the luting materials containing adhesive components and claimed by the manufacturer as the materials adhere to tooth structure.

The methods include tooth substrate selection, storage and preparation of tooth specimen, as well as test procedures for determining bond strength of the luting material to tooth structure for indirect restoration.

NOTE Bond strength obtained using enamel is assumed to be greater than that obtained using dentine. Therefore, the test method is described only for dentine in this annex (see Note 1 in C.1).

A.2 Test methods

A.2.1 Material and apparatus

A.2.1.1 Tooth, extracted human erupted permanent molar or incisor, or bovine incisor.

Use caries-free, unrestored and not root-filled (endodontically treated) tooth. In case of human tooth, wash the tooth thoroughly in running water and remove all blood and adherent tissue as soon as possible after extraction.

Store teeth under wet conditions at below 30 °C for not more than three days after extraction. Preserve the tooth in water of Grade 3 of ISO 3696 or in sterilizing solution, and store in a refrigerator or freezer for not more than six months after extraction.

If the tooth is stored in a refrigerator, replace the storage medium periodically to minimize deterioration.

A.2.1.2 Mould, tube or cup for embedding, made of metal or rigid plastics, having one or more cylindrical cavities, the inner diameter of each being capable of holding a tooth.

Prepare the ends of the mould, tube or cup plane and parallel to each other.

NOTE Use the mould or tube with its top and bottom open. Suggested internal dimensions of the mould tube or cup are around 25 mm in diameter and between 10 mm and 25 mm in height.

A.2.1.3 Potting material, gypsum, or self-curing polymer-based material which does not reach a high curing temperature (e.g. more than 37 °C) and which does not impregnate tooth substance.

A.2.1.4 Silicon carbide abrasive paper, water-proof and grit sizes of silicon carbide of ISO P120, P200, P400 and P600 according to ISO 6344-1.

A.2.1.5 Cylindrical rod made of a rigid material such as cured dental resin composite, rigid plastics or metal. The planes of both ends of the rod are prepared so as to be perpendicular to the longitudinal axis of the rod, and if necessary, the end surface of the rod to be bonded to the tooth structure is pre-treated in accordance with the instructions for use provided by the manufacturer of the luting material.

NOTE 1 This pre-treatment ensures that the rod does not debond at the interface between the rod and the layer of the cured adhesive luting material.

The cylindrical rod may be prepared by cutting or lathe turning a prefabricated rod made of the rigid materials, or by moulding and curing the polymer-based filling material specified in A.2.1.7.

A.2.1.6 Fixing jig with weight, capable of fixing and statically loading (5,0 N) the cylindrical rod (A.2.1.5) to the ground surface of the tooth (A.2.1.1) perpendicularly. See Figure A.2 and Figure A.3.

A.2.1.7 Polymer-based filling material, as recommended by the manufacturer in the instructions for use of the adhesive luting material; otherwise, a dental resin composite of high flexural modulus and strength.

A.2.1.8 Mould for preparation of cylindrical rods with polymer-based filling material, capable of fabricating the cylindrical rod (A.2.1.5). See Figure A.1.

A.2.1.9 Device for measuring dimensions, accurate to 0,005 mm.

A.2.1.10 Oxygen barrier material, capable of preventing the penetration of oxygen from the air and which does not contain any additive(s) that accelerate or restrain the polymerization reaction of the luting material significantly. The barrier material should be flowable at (23 ± 2) °C (e.g. flowable gel). Alternatively, the material specified in the manufacturer's instructions for use may be used.

Latent heat of evaporation of the material should be investigated before applying the material to the specimen.

A.2.1.11 Incubator or air oven, the air being maintained at (37 ± 2) °C.

A.2.1.12 Shearing blade, made of metal or rigid plastics with circular hole(s) or a half circular arch of slightly larger diameter than the cylindrical rod (A.2.1.5). The edge of the wall of the opening of the circular hole(s) or the half circular arch at the end of the blade is prepared as perpendicular to the longitudinal axis of the blade.

A.2.1.13 Shear loading jig, Type A or Type B, capable of thrusting the shearing blade (A.2.1.12) parallel to the adhered plane of the tooth (A.2.1.1).

NOTE See Figure A.4 a) for Type A (for the shearing blade with a circular hole) and Figure A.4 b) for Type B (for the shearing blade with a half circular arch).

A.2.1.14 Bond strength test apparatus, appropriately calibrated, to provide a constant cross-head speed of (0,75 ± 0,30) mm/min or a rate of loading of (50 ± 2) N/min, and free from bending or torsion force.

A.2.2 Procedure

A.2.2.1 General

Except otherwise specified, perform the procedure at (23 ± 2) °C and not less than 30 % relative humidity.

A.2.2.2 Preparation of tooth specimen

A.2.2.2.1 Initial preparation of tooth

If the tooth has been stored frozen, wash in running water, remove excess water and store in water of Grade 3 of ISO 3696. For a bovine tooth, cut off the root at the cemento-enamel junction (CEJ) and remove the pulp. Prior to potting the tooth, fill the pulp chamber with dental utility wax or dental cement.

A.2.2.2.2 Potting of tooth

a) Place the mould or tube (A.2.1.2) for the tooth (A.2.1.1) on a horizontal and plane working surface. Place the tooth in the mould or tube in its intended position and orientation for bonding, i.e. the surface to be bonded is placed down. For bovine teeth, place the labial surface down.

NOTE The cup (A.2.1.2) cannot be used for this procedure. Pressure-sensitive adhesive tape can be used to secure the tooth in the correct place and orientation during the potting procedure. A transparent adhesive tape for packaging can be placed under the mould or tube with the adhesive side upward. It is also useful for easy removal of the potted tooth and clean-up of the mould or tube.

b) Pour the mixed potting material (A.2.1.3) into the mould or tube through the top opening of the mould or tube.

c) After the curing time stated in the manufacturer's instructions for use, ensure that the resin is sufficiently cured and remove the cured cylindrical block holding the tooth as soon as possible from the mould or tube.

d) Immediately after the removal from the mould or tube, store the cured cylindrical block in water (Grade 3 of ISO 3696) or in 100 % relative humidity (RH) at 4 °C, and use it within 7 d.

A.2.2.2.3 Surface preparation

a) General

Prepare a standard, reproducible and flat surface. Keep tooth surfaces wet at all times.

NOTE Exposure of a tooth surface to the air for several minutes can cause irreversible changes in bonding character. Dentine is especially sensitive to dehydration.

For dentine, use the superficial dentine (i.e. as close to enamel as possible) in order to reduce the variations of the results of the test.

b) Procedure

1) Fix the silicon carbide abrasive paper of P120 (A.2.1.4) to a hard and flat surface. Grind the surface of the tooth to be bonded under running water to expose the bonding surface until a bonding area sufficient for the cylindrical rod (A.2.1.5) is exposed.

NOTE For dentine, it is important to stop grinding when superficial dentine is exposed. When ground to excess into the dentine, the bond strength can decrease.

2) Change the silicon carbide abrasive paper to P400 (A.2.1.4) and grind under running water until the surface is even and smooth when visually inspected without magnification. Discard teeth that have any perforations into the pulp chamber. Align the ground surface of the tooth as parallel to the plane of the bottom of the cured cylindrical block which is holding the tooth.

3) Rinse the ground tooth well with water to remove any foreign matter (e.g. residual grit of the silicon carbide and ground tooth particles).

The grinding may be performed in an automatic grinding machine with rotating abrasive discs and running water.

Prepare five tooth specimens in this way.

A.2.2.2.4 Storage of ground tooth specimen

After grinding to expose the surface for bonding, store the ground tooth embedded in the cured cylindrical block in water of Grade 3 of ISO 3696 at (37 ± 2) °C sealed in an appropriate package in the incubator or air oven (A.2.1.11) and use it on the same day on which it was ground.

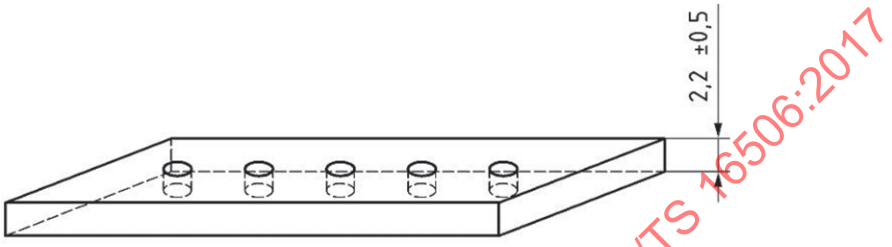
A.2.2.3 Fabrication procedure of cylindrical rod as adherend

A.2.2.3.1 General

Select the most appropriate type of the cylindrical rod (A.2.1.5) as adherend in accordance with the purpose of the test. When the adherend is made of a polymer-based filling material, follow A.2.2.3.2.

A.2.2.3.2 Fabrication of cylindrical rod (A.2.1.5) made of polymer-based filling material

Dimensions in millimetres



The overall length and width of the mould should be not less than 10 times the diameter of the hole. There should be several holes in the mould (e.g. five), and the distance between the centres of each hole should be not less than the diameter of the irradiation window of the photo-irradiation device to be used.

Figure A.1 — Mould for preparation of cylindrical rods with polymer-based filling material (Informative)

a) Place the mould (A.2.1.8) on a transparent film and fill the holes in the mould to slight excess with the paste of the light-cure polymer-based filling material (see A.2.1.7, hereinafter, "CR" or composite resin) without bubbles or voids. Place another transparent film on the mould and a flat plate on both sides of the mould. Apply pressure to exclude the excess material.

   In order to facilitate removal of the CR from the mould, a very thin layer of a separating agent, which does not interfere the curing of the CR, may be applied to the inner surface of the hole; for example, a 3 % solution of polyvinyl ether wax in hexane (see 7.9.1.1, note). The difficulty in removing the cured CR depends on the material of which the mould is made, the microstructure of the inner surface of the hole or the characteristics of the CR.

b) Remove both plates and irradiate the CR in accordance with the CR manufacturer's instructions for use, through the transparent film, the side of the hole with the extruded excess of CR first, followed by the other side.

c) Leave the cured cylinders of CR (hereinafter, the CR rod) in the mould, remove the excess material from both surfaces of the mould using the waterproof abrasive-paper of grade ISO P200 or finer, and clean and dry the surfaces. Identify the end of each CR rod which was cured second.

d) Polish the end of each CR rod which was irradiated first using the waterproof abrasive paper of grade ISO P600 (A.2.1.4) so that the surface is plane and perpendicular to the principal axis of the CR rod.

   NOTE The purpose of this test is to determine the bond strength between the luting material and the tooth structure. Thus, unless debonding between the luting material and the CR rod occurs, any grade of abrasive paper is acceptable.

e) Clean the CR rods, while still in the mould, in water using ultrasonication for (60 ± 10) s, and then push the CR rod out from the hole of the mould by pressing the marked surface using an appropriate instrument. Clean the CR rod again in water using ultrasonication for (60 ± 10) s.

f) Using a measuring device (A.2.1.9) such as a micrometer, measure the diameter of the CR rods at the first-irradiated surface to an accuracy of 0,01 mm and record it as *d* (see A.3.2).

Prepare five CR rods.

Store the rods in clean and dry environment at room temperature and use them within 4 d.

A.2.2.4 Bonding

A.2.2.4.1 General

This bonding procedure consists of:

a) pre-treatment of the ground tooth specimen;

b) preparation and application of the luting material to the cylindrical rod;

c) attaching the cylindrical rod with the luting material to the tooth surface and loading the weight on the cylindrical rod;

d) removal of the excess luting material outside the intended bonding area;

e) curing by irradiation of outer-energy at (23 ± 2) °C and/or self-curing of the luting material at (37 ± 2) °C;

f) storage of the bonded specimen in water at (37 ± 2) °C.

A.2.2.4.2 Pre-treatment of the cylindrical rod and the ground tooth specimen

a) Condition the fixing jig and the weight (see Figure A.2 and Figure A.3) in the incubator or air oven (A.2.1.11) at (37 ± 2) °C for at least 1 h.

b) Apply any necessary pre-treatment and/or priming to the end surface of the rod to be bonded to the tooth structure, in accordance with the instructions for use provided by the manufacturer(s) of the luting material and the material of which the rod is made.

   When the rod made of the polymer-based filling material (A.2.1.7) has high content of glass filler, an appropriate primer for coupling the luting material to glass components should be applied prior to application of the luting material to the rod.

c) Rinse the tooth surface with running water of Grade 3 of ISO 3696 at (37 ± 2) °C. Thoroughly remove any water from all the surfaces except the area of the ground tooth by wiping with a thick, clean and dry water-absorbable paper(s). On the area of the ground tooth, gently place another clean and dry water-absorbable paper to remove any visible layer of water.

d) Perform any pre-treatment(s) to the ground tooth surface indicated by the manufacturer's instructions for use.

e) Using a small syringe, place water of Grade 3 of ISO 3696 on the pre-treated surface of the ground tooth such that all of the area to be bonded is covered with a plane and thin layer of the water. If the water forms a convex drop, remove excess with a dry cotton pledget held by tweezers.

NOTE For the purposes of simulating the wet condition of the tooth in a clinical situation by this test, a clinical procedure such as air-blowing of the tooth surface as described in the manufacturer's instructions for use is not applied.

A.2.2.4.3 Preparation and application of the luting material

a) General

   1) Complete the preparation and application of the luting material while the surface of the ground tooth is in the wet condition, controlled according to A.2.2.4.2, before the bonding (see A.2.2.4.4).

2) Use newly dispensed or mixed luting material for each specimen.

b) Preparation of the luting material

1) When the luting material has been stored in a cold environment, condition it at (23 ± 2) °C for at least 1 h.

2) When the luting material of Class 1 or Class 3 is supplied in any form for mixing its components, dispense and mix sufficient volume for clinically luting a single full crown in accordance with the manufacturer's instructions for use. If a mixing-tip for auto-mixing is provided by the manufacturer, mix the components through a new mixing-tip each time, and discard an appropriate amount of the mixed luting material which is initially extruded from the mixing-tip.

c) Application of the luting material to the cylindrical rod

1) When the luting material is hand-mixed on a plate, hold the cylindrical rod (A.2.1.5) using tweezers, and place the rod on a clean mixing-pad or mixing-sheet with the first-irradiated surface uppermost. Using a clean instrument such as a spatula, apply a single portion of the prepared luting material in shape of convex onto the upper surface of the rod, the amount of the mixed-material applied being capable of covering the full surface of the rod with some excess around the margin between the rod and the tooth when the static load of 5 N is applied.

2) When the luting material is auto-mixed with a mixing-tip, discard the portion which is initially extruded and apply the portion extruded thereafter directly onto the surface of the rod. The amount of the luting material applied should be as described in A.2.2.4.3 a).

3) Ensure that the luting material applied on the rod does not contain any visible air bubbles.

A.2.2.4.4 Bonding and weight loading

a) Bonding

1) Transfer the fixing jig and the weight [see A.2.2.4.2 a)], both having been kept at (37 ± 2) °C to a flat and horizontal working table at (23 ± 2) °C, ensuring that there is no direct air flow onto the fixing jig and the weight.

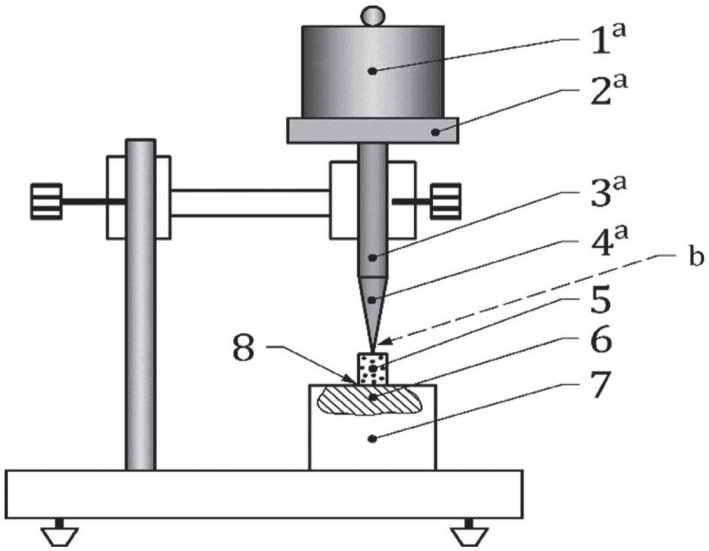
NOTE If there is a direct air flow onto the assembly, especially the tooth specimen with water applied on its surface, their temperature can quickly decrease to far below that clinically.

2) Place the embedded tooth, pre-treated as described in A.2.2.4.2 d) on the base of the fixing jig (see Figure A.2), with the ground surface of the tooth upwards.

3) Immediately after the application of the luting material to the rod [see A.2.2.4.3 b)], hold the rod in the position with its principal axis vertical and the surface on which the luting material is applied downwards, and place the surface of the rod with the mixed material onto the ground surface of the tooth without any additional load except the weight of the rod itself.

b) Weight loading

Position the lower end of the cone (key 4 of Type A fixing jig or key 2 of Type B fixing jig; see Figure A.2 and Figure A.3) on the centre of the upper surface of the rod, and load the static weight of (5,00 ± 0,10) N gently on the rod.



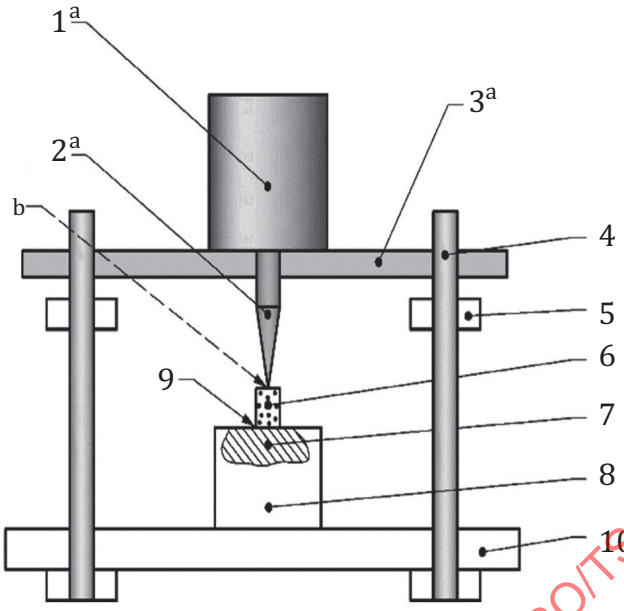
Key

1 static weight
2 table for the static weight
3 loading column
4 cone connected to the loading column
5 cylindrical rod (A.2.1.5)
6 tooth
7 cured block of potting material (A.2.1.3)
8 adhesive material

a The mass of items 1, 2, 3 and 4 are included in the load of (5,00 ± 0,10) N applied on the cylindrical rod (key 5).

b For safety of operation, the shape of the narrow end of the cone (key 4) should be flat or hemispherical and 0,3 mm to 0,5 mm in diameter.

Figure A.2 — Type A — Fixing jig



Key

1 static weight
2 cone and loading column
3 top plate
4 supporting column (may be four pieces)
5 locking nut (may be eight pieces)
6 cylindrical rod (A.2.1.5)
7 tooth
8 cured block of potting material (A.2.1.3)
9 adhesive material
10 base plate

a The mass of items 1,2 and 3 are included in the load applied on the cylindrical rod (key 6).

b For safety of operation, the shape of the narrow end of the cone (key 2) should be hemispherical or flat and 0,3 mm to 0,5 mm in diameter.

Figure A.3 — Type B — Fixing jig

Suggested dimensions of the loading cone and column of Type A and Type B fixing jigs are: the diameter of the cone at its lower end should be 3 mm to 5 mm, the height should be 20 mm to 30 mm, and the shape of the pointed end should be hemispherical of diameter 0,3 mm to 0,5 mm. The length of the loading column should be 10 mm to 20 mm, and its diameter should be the same as the upper end of the cone. The detailed shapes of the surface of the cone and the loading column are not specified. The use of a wood screw with a rounded end should be acceptable.

NOTE When a compact-sized fixing jig for self-curing procedures is needed, see Annex B.

A.2.2.4.5 Removal of the excess luting material and water surplus

a) Immediately after the commencement of the loading (see A.2.2.4.4), completely remove the material extruded from the outermost edge of the cylindrical rod using appropriate and clean instruments. Perform this procedure as soon as possible within the period of the working time specified by the manufacturer's instructions for use, and keep the rod under the load during this procedure.

NOTE 1 To avoid any displacement of the uncured luting material from the bonding area under the rod, remove only the surplus. When using a brush to remove the excess, take care that its filament ends do not penetrate the space under the bonding surface of the rod. When using a sharp instrument, it is useful to make the edge touch the periphery of the rod lightly and then remove the surplus of uncured luting material.

b) Keeping the rod under load, wipe the top surface of the tooth-embedded specimen, except that under the rod, with dry paper or a cotton pledget held with tweezers.

NOTE 2 This is intended to remove the excess water remaining outside of the bonded area. The latent heat of evaporation of the water could delay the temperature rise of the bonded part during the curing procedures, especially on irradiation by the activating light and the self-curing process.

A.2.2.4.6 Curing of the luting material

a) General

1) Cure the bonded specimens using the following procedures, depending on the type of the polymerization process by which the luting material to be cured, i.e. Class 1 "self-cure", Class 2 "external-energy-activated" and Class 3 "dual-cure".

2) If the manufacturer of a Class 3 material indicates in the instructions for use or on the package that the material can be used without irradiation of external energy, perform the test using the specimen cured only by the self-curing process [see A.2.2.4.6 b) 1)].

b) Procedure

1) Class 1: Self-curing material (see 4.1)

i) Maintain the fixing jig of Type A or Type B (see Figure A.2 and Figure A.3, respectively) in the state of static loading, and apply the oxygen barrier material (A.2.1.10) to the margin of the junction of the rod and the tooth as a single layer of about 1,0 mm in width and thickness.

A small syringe with a needle can be used to apply the oxygen barrier material. The material should be warmed to (37 ± 2) °C for at least 1 h before use. Flowing of the material after application is acceptable.

NOTE 1 The purpose of applying the oxygen barrier material is to ensure curing of the luting material at the marginal area. This procedure is intended to simulate clinical conditions in which there is possibly less oxygen inhibition of the curing of the luting material compared with this standard test.

ii) Place the assembly in the incubator or air oven at (37 ± 2) °C for the setting time or equivalent specified by the instructions for use, plus 30 min. When the instructions for use do not specify the setting time or equivalent, adopt 10 min as the setting time (see C.2.2.3).

iii) Remove the specimen from the fixing jig and keep the specimen with the rod bonded to the tooth surface positioned upwards in the incubator or the air oven at (37 ± 2) °C for 90 min.

NOTE 2 The extra time of 30 min in procedure b) and 90 min in procedure c) added to the time for curing in these procedures are adopted from the results obtained in the preliminary studies. In this test, the bonded specimen consists of the tooth, the embedding resin of large mass, the cylindrical rod and the luting material. Therefore, the thermal capacity of the bonded specimen is larger than that clinically. Additionally, when the bonded specimen is covered with water and absorbed moisture, the latent heat of evaporation of the water on the bonded specimen can cause a critical temperature decrease, or a delay in the temperature rise. Consequently, the rate of temperature rise of the bonded specimen in the incubator or the air oven can be much slower than that observed clinically.

iv) Remove the specimen from the incubator or the air oven, wipe off the oxygen barrier material and place the specimen immediately into water of Grade 3 of ISO 3696 at (37 ± 2) °C in the incubator or the air oven (A.2.1.11). Place the entire bonded specimen under the water level in an appropriate sealed package with the rod bonded to the tooth surface positioned upwards. Use an individual package for each specimen, if applicable.

Repeat the procedures a) to d) four times to prepare five bonded specimens.

NOTE 3 If the package containing the water is not sealed, the temperature of the water will decrease due to its latent heat of evaporation, especially when the air-flow for temperature control is strong when the door(s) of the incubator or the air oven is/are opened and closed frequently.

5) Keep each of the bonded specimens in the water as described in the procedure d) for (24 ± 1) h.

2) Class 2: External-energy-activated materials (see 4.2)

i) Maintain the fixing jig of Type A or Type B (see Figure A.2 or Figure A.3, respectively) under the static load and apply any covering materials specified in the instructions for use provided by the manufacturer of the luting material (e.g. oxygen barrier materials) to the margin of the joint of the rod and the tooth, according to the instructions for use.

ii) Irradiate the luting material layer on the tooth in accordance with the manufacturer's instructions for use of the luting material, i.e. type of irradiation equipment, irradiation time, etc. During the irradiation, position the tip of the irradiation window as close as possible to the luting material layer but not touching any part of the cylindrical rod.

Before irradiation, the output power of the irradiation equipment should be confirmed according to the material manufacturer's instructions for use.

ii) Move the irradiation window to the next position in horizontal and angular intervals of 90° and irradiate for the same period of time specified in the instructions for use. Repeat procedure c) and d) once more.

iv) After irradiation, place the assembly in the incubator or the air oven at (37 ± 2) °C for the post-irradiation time or equivalent specified by the manufacturer's instructions for use plus an additional 30 min. When the manufacturer's instructions for use do not specify the post-irradiation time or equivalent, adopt 10 min as the post-irradiation time.

v) Remove the bonded specimen from the fixing jig, wipe off the covering material if applied, and place the specimen immediately in water of Grade 3 of ISO 3696 maintained at (37 ± 2) °C in the incubator or the air oven (A.2.1.11). Place the entire bonded specimen under the water level in an appropriate sealed package with the rod bonded to the tooth surface positioned upwards. Use individual packages for each specimen.

Repeat the procedures a) to e) four times to prepare five bonded specimens.

NOTE See note in A.2.2.4.6 b) 1) iv).

vi) Keep each of the five bonded specimens in the water as described in procedure for (24 ± 1) h.

3) Class 3: Dual-cure materials (see 4.3)

1) General

Apply the procedure described in A.2.2.4.6 b) 2) (for Class 2: External-energy-activated materials).

2) In the case that the irradiation is not necessary, according to the manufacturer's instructions for use

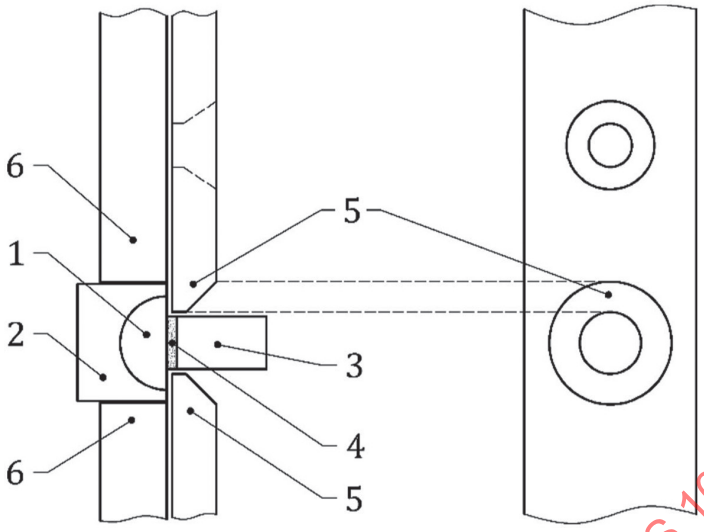
Apply independently the procedure described in A.2.2.4.6 b) 1) for Class 1: Self-curing material.

A.2.2.5 Determination of shear bond strength

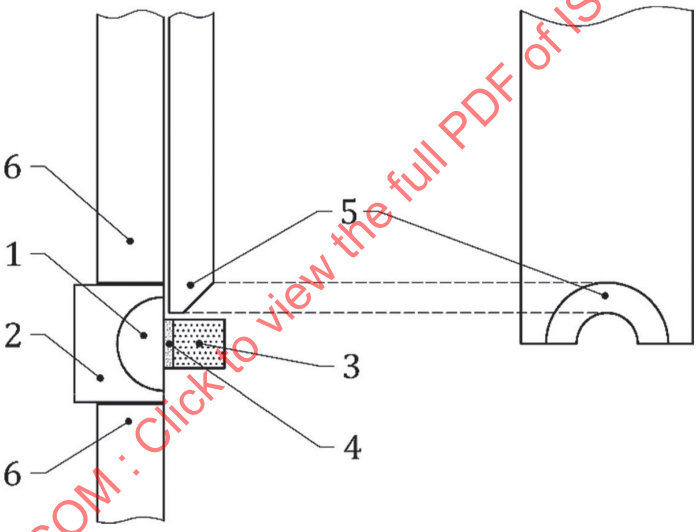
a) Immediately after removal of the bonded specimen from the water, place it into the specimen holder of the shear loading jig Type 1 or Type 2 (A.2.1.13); see Figure A.3 a) or Figure A.3 b), respectively.

b) Place the bonded specimen into the specimen holder (key 6) of the loading jig, and clamp it so that the adhered surface of the tooth is aligned in the same plane as the front plane of the upper part of the holder, and the longitudinal axis of the cylindrical rod (key 3) is aligned parallel to the longitudinal axis of the holding space of the holder.

c) Place the base of the shear loading jig (A.2.1.13) with the shearing blade (key 5) on the base of the bond strength test apparatus (A.2.1.14). Ensure that the base of the loading jig is placed on a horizontal plane.



a) Type 1 shear loading jig



b) Type 2 shear loading jig

Key

1 tooth
2 cured block of potting material
3 cylindrical rod
4 cured adhesive material
5 shearing blade
Type 1: with a circular hole
Type 2: with a half circular arch
6 specimen holder

Figure A.4 — Shear loading jigs

A.3 Treatment of results

A.3.1 Examination of the fractured surfaces

Inspect the fractured surface using a microscope with appropriate magnification. Discard the result if there is evidence that the initiation of the fracture had started between the cylindrical rod and the cured luting material layer.

A.3.2 Calculation of bond strength

Calculate the bond strength, σ, in megapascals (MPa), from Formula (A.1):

$$\sigma = \frac{F}{\pi(d/2)^2} \quad \text{(A.1)}$$

where

F is the maximum load, in newtons (N), exerted on the specimen;

π is the ratio of the circumference of a circle to its diameter (not dimensional), the value of 3,14 may be used in this formula;

d is the diameter of the individual cylindrical rod, in millimetres (mm).

See A.2.1.5 and A.2.2.3.2 f).

Report the values obtained.