INTERNATIONAL STANDARD

ISO 9917-2

Second edition
2010-04-15

Dentistry — Water-based cements —

Part 2: Resin-modified cements

Médecine bucco-dentaire — Ciments à base d'eau —

Partie 2: Ciments modifiés par addition de résine

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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

ISO 9917-2 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 1, *Filling and restorative materials*.

This second edition cancels and replaces the first edition (ISO 9917-2:1998), which has been technically revised by the inclusion of resin-modified cements which set by both chemically activated and light-activated polymerization.

ISO 9917 consists of the following parts, under the general title *Dentistry — Water-based cements*:

— *Part 1: Powder/liquid acid-base cements*

— *Part 2: Resin-modified cements*

Introduction

This part of ISO 9917 has been prepared in order to present the requirements and test methods for cements in which setting is achieved by a combination of an acid-base reaction and polymerization. The polymerization component of the reaction may be activated by mixing different components or through application of energy from an external source. As far as possible, test methods employed within this part of ISO 9917 have been harmonized with those used in ISO 4049 and ISO 9917-1.

Specific qualitative and quantitative requirements for freedom from biological hazard are not included in this part of ISO 9917, but it is recommended that reference be made to ISO 10993-1 and ISO 7405 when assessing possible biological or toxicological hazards.

Dentistry — Water-based cements —

Part 2: Resin-modified cements

1 Scope

This part of ISO 9917 specifies requirements and test methods for dental cements that are intended for luting, base or lining and restoration purposes and for which the materials are water-based and set by multiple reactions in which setting is achieved by a combination of an acid-base reaction and polymerization.

EXAMPLE Conventional glass polyalkenoate cements are normally formed by reacting an ion-leachable aluminosilicate glass with a polyalkenoic acid in an aqueous environment. Materials that fall within the scope of this part of ISO 9917 will normally be able to effect setting by such an aqueous acid-base type reaction but in addition will be able to undergo setting by polymerization.

NOTE Attention of manufacturers and test laboratories is drawn to the closely-related International Standards ISO 4049 and ISO 9917-1. Consideration should be given as to which is the most appropriate International Standard by which to evaluate any individual product.

2 Normative references

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

ISO 1942, *Dentistry — Vocabulary*

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

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

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

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

3 Terms and definitions

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

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

NOTE Materials that fall within the scope of this part of ISO 9917 include materials that require mixing and single component materials that do not require mixing.

3.2
working time
period of time, measured from start of mixing (if required), during which it is possible to manipulate the material without an adverse effect on its properties

NOTE Working time is determined in the absence of activating radiation, if required for activation for Class 3 materials (see Clause 4).

3.3
setting time
period of time, from start of mix, until the completion of set, as defined by the ability of the material to support an indenter under a known load

3.4
outer pack
form of packaging used to combine a number of single dose containers or capsules

3.5
outermost packaging
form of packaging used to combine material and additional items, including instructions for use and any proportioning or mixing devices that are supplied with the material

4 Classification and applications

4.1 Classification

For the purposes of this part of ISO 9917, materials are classified on the basis of their setting characteristics as follows.

— Class 1: materials in which the setting reaction of the polymerizable component is activated chemically following mixing of components.

— Class 2: materials in which the setting reaction of the polymerizable component is light-activated.

— Class 3: materials in which the setting reaction of the polymerizable component is activated chemically following mixing of components and may also be light-activated.

4.2 Applications

For the purposes of this part of ISO 9917, the clinical application of these materials is signified as follows:

a) luting;

b) base or lining;

c) restoration.

5 Requirements

5.1 Materials

During the course of testing, there shall be no visible signs of extraneous matter in any component and separately supplied liquid shall be free of any gelation.

5.2 Working time

When tested in accordance with Annex A, the working time shall comply with the requirements given in Table 1 and shall be at least as long as the value given by the manufacturer (see Table 2, item 24).

5.3 Setting time — Classes 1 and 3 materials only

When tested in accordance with Annex A, the setting time of Classes 1 and 3 materials shall comply with the requirements given in Table 1 and shall be no longer than the value given by the manufacturer (see Table 2, item 25).

5.4 Film thickness — Luting cements only (see 4.2)

When tested in accordance with Annex B, the film thickness of luting materials shall comply with the requirements given in Table 1.

5.5 Flexural strength

When tested in accordance with Annex C, the flexural strength shall comply with the requirements given in Table 1.

5.6 Radio-opacity

If the manufacturer claims that the material is radio-opaque (see Table 2, item 16), the radio-opacity, determined in accordance with Annex D, shall be equal to or greater than that of the same thickness of aluminium. If greater radio-opacity is claimed, it shall not be less than 0,5 mm below the value claimed by the manufacturer (see Table 2, item 17).

5.7 Shade and colour stability — Restorative materials only

When tested in accordance with Annex E, the set material shall closely match that of the shade guide specified by the manufacturer. When tested in accordance with Annex E and ISO 7491, there shall be no more than a slight change in colour after 7 d.

Table 1 — Requirements for dental cements

Application	Film thickness µm Max.	Working time min Min.	Setting time[a] min Max.	Flexural strength MPa Min.
Luting	25	1,5	8	10
Base or lining	—	1,5	6	10
Restoration	—	1,5	6	25

[a] Class 1 and 3 materials only. Class 3 materials tested without activation by light.

6 Sampling

A sample drawn from one batch shall provide sufficient material to complete all the prescribed tests plus an allowance for any repeat tests, should they become necessary. The test sample shall consist of packages prepared for retail sale.

7 Test conditions and preparation of test specimens

7.1 Test conditions

Prepare and test all specimens at an ambient temperature of (23 ± 2) °C. Control the relative humidity to ensure that it remains at (50 ± 20) % at all times. If the material was refrigerated for storage, allow it to reach (23 ± 2) °C. Test equipment should be maintained at the condition specified in individual tests.

For Classes 2 and 3 materials, activating radiation shall be excluded during the determination of working time.

Water used in all tests specified in this part of ISO 9917 shall be prepared in accordance with ISO 3696, grade 2.

For Classes 2 and 3 materials, reference shall be made according to the manufacturer's instructions (see Table 2, item 26), which state the external energy source to be used. Care shall be taken to ensure that the source is in a satisfactory working condition.

7.2 Method of mixing

The cement shall be prepared according to the manufacturer's instructions. Sufficient cement shall be mixed to ensure that the preparation of each specimen is completed from one mix. A fresh mix shall be prepared for each specimen.

NOTE For encapsulated materials, more than one capsule, simultaneously mixed, might be required for certain specimens. Likewise, for materials supplied in single dose containers, several containers might be required for each test specimen.

7.3 Inspection requirements

Visual inspection shall be used in determining compliance with 5.1 and Clause 8.

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

8.1 Packaging

The components of the material shall be supplied in properly sealed containers which adequately protect their contents and have no adverse effect on the quality of the product.

An outer pack may be used to present the individual containers as a single unit.

NOTE Single paste and powder-liquid encapsulated products can be sold as a pack containing many unit doses of material.

8.2 Marking and instructions for use

Information shall be clearly marked on the outermost packaging or containers (for multi-dose packs or capsules), as appropriate, and as indicated in Table 2.

Instructions shall accompany each package of the material and shall include the information appropriate to the material (see Clause 4), as indicated in Table 2.

Information additional to that specified in Table 2 may be supplied at the discretion of the manufacturer.

NOTE Some information is indicated as mandatory and other as optional. The symbol "/ " indicates that this item is either irrelevant or optional for the product. Table 2 contains several optional references and serves as a guide to the manufacturer as to the sort of information which might be useful to dentists.

Table 2 — Requirements for marking and instructions for use

Requirement		Outermost packaging see 3.5	Outer pack of capsules see 3.4	Capsule (single-dose), syringes or bottles	Manufacturer's instruction leaflet
1	The name of the product.	M	M	/[a]	M[b]
2	The identification or name of the manufacturer.	M	M	/	M
3	The address of the manufacturer or the agent responsible for sale.	M	/	/	M
4	URL.	/	/	/	/
5	Information required by local/national legislation.	M	M	/	M
6	The recommended conditions of storage.	M	/	/	M
7	The manufacturer's batch number.	M	M	/	/
8	The expiry date, expressed in accordance with ISO 8601, for the cement when stored under the manufacturer's recommended conditions.	M	M	/	/
9	The shelf life under those conditions of storage.	/	/	/	/
10	The classification of the cement (see 4.1).	M	/	/	/
11	The clinical application (see 4.2).	/	/	/	M
12	The number of containers/capsules, for capsule or cartridge cements.	M	M	/	/
13	The net mass in each container/capsule.	/	M	/	M
14	Shade and/or colour of the cement according to the manufacturer's nominated shade guide (for multi-shade materials only).	/	M	M[c]	/
15	If the material is designated opaque, a clear statement to this effect[d].	M	/	/	M
16	If the cement is designated radio-opaque (see 5.6), a clear statement to this effect.	/	/	/	M
17	If a specific claim on the extent of radio-opacity is made, the equivalent thickness of aluminium for 1 mm thickness of the cement (see 5.6).	/	/	/	M
18	The recommended ratio of components (e.g. powder:liquid) and instructions for use of any proportioning aids (e.g. scoops, etc.) and the proportions on a mass/mass basis to a precision of 0,1 g (for hand mixed materials only).	/	/	/	M
19	The rate of incorporation of the powder into the liquid (for hand-mixed materials only).	/	/	/	M
20	The mixing time (see 3.1), if mixing required.	/	/	/	M
21	The mixing condition (if appropriate, the condition and type of the mixing slab and spatula). For hand-mixed materials only.	/	/	/	M
22	For encapsulated cements, the method of bringing about physical contact between the components, if required.	/	/	/	M
23	The method, timing and type of mechanical mixing, if required.	/	/	/	M

Table 2 (*continued*)

	Requirement	Outermost packaging see 3.5	Outer pack of capsules see 3.4	Capsule (single-dose), syringes or bottles	Manufacturer's instruction leaflet
24	The working time (see 3.2).	/	/	/	M
25	The setting time (for class 1 and 3 materials only, see 3.3).	/	/	/	M
26	The recommended external energy source(s), exposure times and any special instructions for use of the equipment (for class 2 and 3 materials only).	/	/	/	M
27	The maximum thickness of layer for polymerization [class 2 and 3 materials for application b) and c) only].	/	/	/	M
28	The minimum time at which finishing and polishing may be commenced (for restoration only, see 4.2).	/	/	/	M
29	The recommended method of finishing (for restoration only, see 4.2).	/	/	/	/
30	The necessity of varnish, if appropriate.	/	/	/	/
31	The precautions necessary to prevent premature activation of setting (class 2 and 3 cements only).	/	/	/	M

[a] "/" indicates no relevance for this combination of container/marking/instructions or that such a requirement would be impracticable or impossible or that the information may be informative but optional.

[b] "M" indicates that an item is mandatory.

[c] For individual/small dose containers, the individual containers shall have some means of identifying the colour/shade of the material either directly or through reference to a key or guide in the instruction leaflet.

[d] Opaque designation can be included in the shade.

Annex A
(normative)

Determination of working time and setting time

A.1 Apparatus

A.1.1 Test environment, capable of being maintained at a temperature of (37 ± 1) °C and a relative humidity of at least 50 %.

A.1.2 Indenter, of a given mass and having a flat end with a given diameter. The tip shall be cylindrical for approximately 5 mm. The indenter end shall be plane and perpendicular to its long axis.

A.1.2.1 Indenter for working time, of mass: (28,00 ± 0,25) g and diameter: (2,0 ± 0,1) mm.

A.1.2.2 Indenter for setting time, of mass: (400 ± 5) g and diameter: (1,0 ± 0,1) mm.

A.1.3 Metal mould, of thickness (5 ± 2) mm having a circular or square hole of (10 ± 2) mm diameter/length cut in a sheet of metal at least 16 mm^2.

NOTE Internal corners of a square hole can be rounded.

A.1.4 Metal block, with a thickness of at least 8 mm and a volume of at least 60 cm^3.

A.1.5 Aluminium foil.

A.1.6 Timer, capable of reading to 1 s.

A.2 Determination of working time

A.2.1 Procedure

Classes 2 and 3 materials should be handled in the absence of light of wavelength 400 nm to 500 nm, for example using a dark room and/or filtered light.

The test shall be performed under the conditions described in 7.1.

Place the mould (A.1.3), conditioned to (23 ± 1) °C, on the block (A.1.4) covered with the aluminium foil (A.1.5), also conditioned to (23 ± 1) °C, and fill to a level surface with mixed cement.

Ten seconds before the end of the working time given by the manufacturer (see Table 2, item 24) or the minimum value given in Table 1 (whichever is the longer), carefully lower the indenter (A.1.2.1) vertically on to the surface of the cement and allow it to remain there for 5 s. Note whether the indenter makes a complete circular indentation in the surface of the cement.

Repeat the test with two separate mixes of the material.

A.2.2 Treatment of results

In order to satisfy the requirements, the indenter shall make a complete circular indentation in the surface of the cement specimen. All three values of indentation shall comply at 10 s before the working time stated by the manufacturer (see Table 2, item 24) and with the minimum value of working time in Table 1.

A.3 Determination of setting time — Class 1 and 3 materials only

NOTE The purpose of this test is to confirm that Class 1 and Class 3 materials will set in the absence of light activation.

A.3.1 Procedure

Place the mould (A.1.3), conditioned to (23 ± 1) °C, on the aluminium foil (A.1.5), mix or dispense the cement, start the timer (A.1.6) and fill the mould to a level surface with mixed cement.

Sixty seconds after the end of mixing, place the assembly, comprising mould, foil and cement specimen, on the block (A.1.4), in the test environment (A.1.1). Ensure good contact between the mould, foil and block.

Class 3 materials shall be tested without the use of activating light.

Ten seconds after the setting time stated by the manufacturer (see Table 2, item 25) or the limit value stated in Table 1 (whichever is the shorter) carefully lower the indenter (A.1.2.2) vertically on to the surface of the cement and allow it to remain there for 5 s. Remove the indenter from the surface and note whether the indenter fails to make a complete circular indentation in the cement, when viewed using ×2 magnification.

Repeat the test two more times.

A.3.2 Treatment of results

In order to satisfy the requirements, the indenter shall fail to make a complete circular indentation in the cement for all three tests. Materials which require light activation in order to set shall be designated as Class 2 materials.

Annex B
(normative)

Determination of film thickness — Luting materials only

B.1 Apparatus

B.1.1 Two glass plates, optically flat, square or circular, transparent, having a contact surface area of (200 ± 25) mm^2. Each plate shall be of a uniform thickness of not less than 5 mm.

B.1.2 Loading device, of the type illustrated in Figure B.1, or an equivalent means, whereby a force of (150 ± 2) N shall be generated vertically on the specimen via the upper glass plate.

The anvil that is attached to the bottom of the rod carrying the load shall be horizontal and parallel to the base. The load shall be applied smoothly and in such a manner that no rotation occurs.

B.1.3 Screw micrometer or **equivalent measuring instrument**, having graduations of 1 µm or smaller.

B.2 Procedure

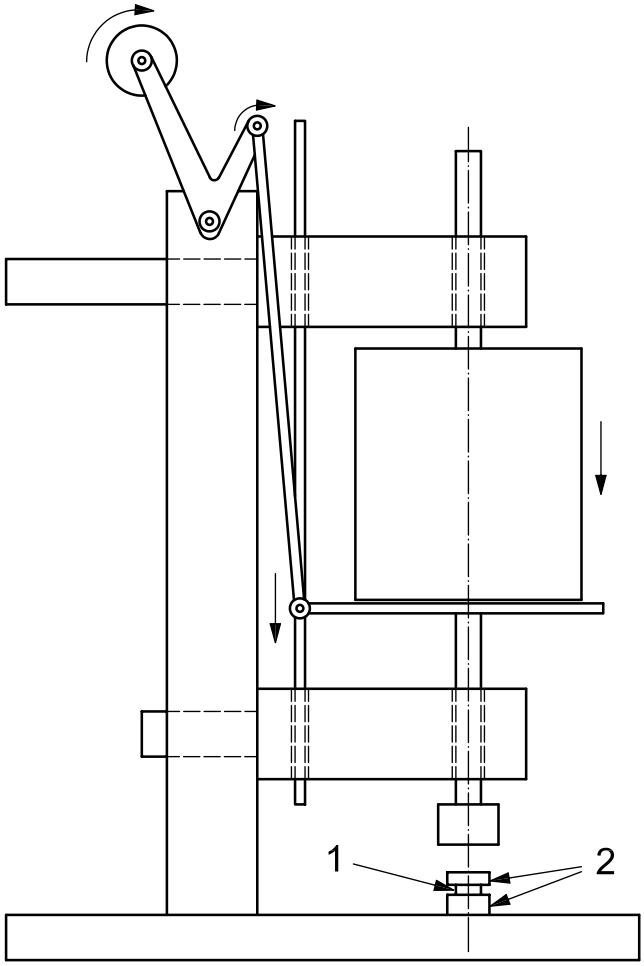
Measure and record to an accuracy of 1 µm the combined thickness of the two optically flat glass plates (B.1.1) stacked in contact and designate this measurement reading A. Remove the upper plate and place (0,10 ± 0,05) ml or the equivalent mass of the mixed cement in the centre of the lower plate and place this centrally below the loading device (B.1.2) on the lower plate. Place the second glass plate centrally on the cement in the same orientation as in the original measurement.

Ten seconds before the end of the manufacturer's stated working time (see Table 2, item 24), apply the load to generate a force of (150 ± 2) N vertically and centrally to the specimen via the top plate. Ensure that the cement has completely filled the space between the glass plates. When at least 10 min have elapsed after the application of the load, remove the plates from the loading device and measure the combined thickness of the two glass plates and the cement film in the same location. Designate this measurement reading B.

Record the difference in thickness of the plates with and without the cement film (reading B − reading A) as the thickness of the film. Repeat the test four times.

B.3 Treatment of results

At least four of the five results shall be no more than 25 µm for the material to pass the requirement specified in Table 1. If only two or less results are no more than 25 µm, then the material fails the requirement. If three results are no more than 25 µm, a further five specimens shall be tested. To comply with the requirement specified in Table 1, all the results in the second series of five shall be no more than 25 µm.



Key

1 specimen

2 glass discs

Figure B.1 — Loading device for use in film thickness test

Annex C
(normative)

Determination of flexural strength

C.1 Apparatus

C.1.1 Mould for the construction of specimens

The exact nature of the mould is not specified but shall enable specimens to be prepared according to the manufacturer's instructions. Two examples of suitable moulds are given in C.1.1.1 and C.1.1.2.

C1.1.1 Mould, for the preparation of a test specimen (25 ± 2) mm $\times$ $(2{,}0 \pm 0{,}1)$ mm $\times$ $(2{,}0 \pm 0{,}1)$ mm. A suitable mould is illustrated in Figure C.1. The mould may be constructed from stainless steel, a cast sheet of PMMA [poly(methyl methacrylate)] or another suitable non-reactive mould material. A release agent may be required to aid removal of specimens. If a release agent is used it shall have no effect on the setting of the cement.

NOTE An example of a suitable release agent is a 3 % solution of paraffin wax in hexane.

Dimensions in millimetres

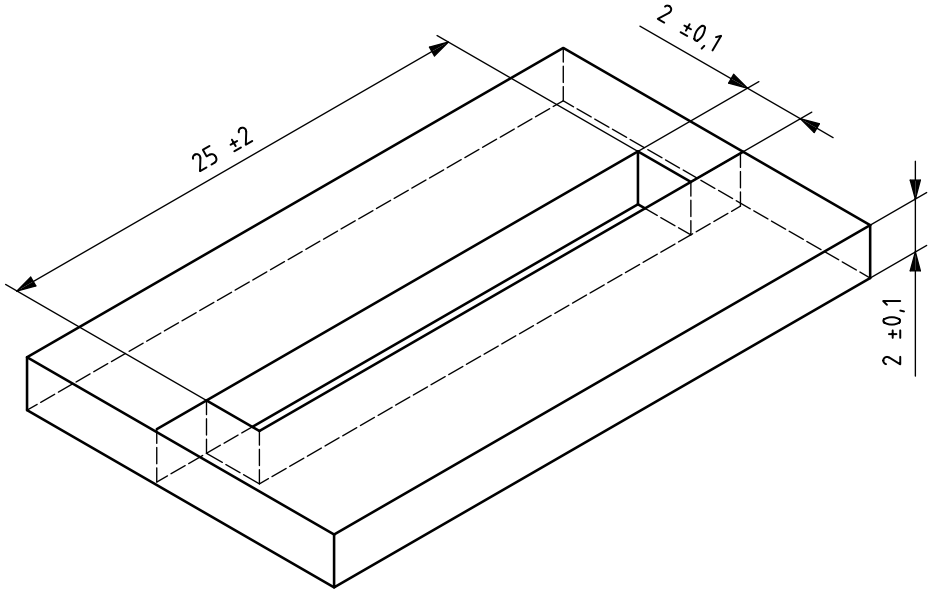


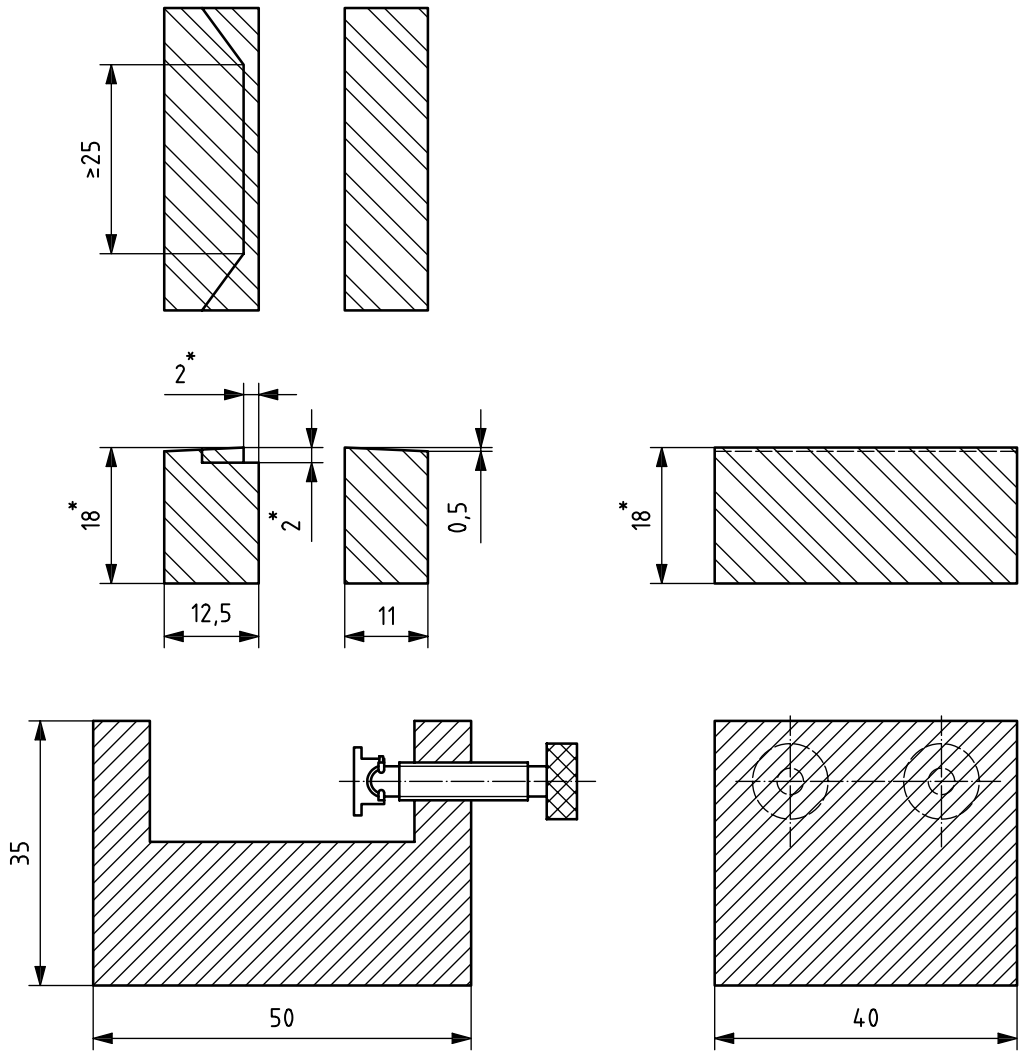
Figure C.1 — Mould for flexural strength specimens

C.1.1.2 Mould, an alternative mould for specimen preparation, as shown in Figure C.2. The mould blocks shall be constructed from sheet PMMA to avoid sticking. The moulds shall be regularly inspected and replaced when damaged or worn. A release agent may be required to aid removal of specimens. If a release agent is used, it shall have no effect on the setting of the cement.

NOTE An example of a suitable release agent is a 3% solution of paraffin wax in hexane.

The mould is used in conjunction with a levelling press which may be constructed from any rigid material (constructed from aluminium in Figure C.2) and which compresses the material within the mould whilst maintaining the upper and lower specimen surfaces in a plano-parallel arrangement.

Dimensions in millimetres



Materials: PMMA aluminium

NOTE Except for mould cross-section including the height of the defining blocks (marked *), no dimension is critical.

Figure C.2 — Alternative mould design and arrangement for flexural strength specimens

C.1.2 Flexural properties test equipment and test jig

C.1.2.1 Test equipment, calibrated to provide a constant crosshead speed of (0,75 ± 0,25) mm/min or a rate of loading of (50 ± 16) N/min.

C.1.2.2 Test jig, consisting essentially of two rods (2 mm in diameter), mounted parallel with 20 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 three-point loading to the specimen.

C.1.2.3 **Transparent glass slides/plates**, each of sufficient area to cover the mould.

C.1.2.4 **Polyester film**, e.g. dental matrix strip.

C.1.2.5 **Water bath**, capable of being maintained at (37 ± 1) °C.

C.1.2.6 **Micrometer or equivalent device**, having graduations of 0,01 mm or smaller.

C.1.2.7 **Clamp**, suitable for holding the specimen mould assembly during conditioning in the water bath (C.1.2.5).

C.1.2.8 **External energy source(s)**, as recommended by the manufacturer for use with the test material (see Table 2, item 26).

C.2 Specimen preparation

C.2.1 Specimen preparation — Class 1 materials

Ensure mould surfaces are clean and, if appropriate, coated with a suitable release agent. Mix the test cement according to the manufacturer's instructions and load at centre of mould space without delay, such that flow is outwards, leaving the mould overfilled and making no attempt to level. This should be done within the working time of the material (see Table 2, item 24).

Apply a polyester film (C.1.2.4) and glass plate (C.1.2.3) to the exposed surfaces of the material and place the assembly in a levelling press such that the assembly can be compressed to extrude excess material at a time within the working time of the material (see Table 2, item 24). Clamp (C.1.2.7) the assembly, place in the water bath (C.1.2.5) and leave to set for 1 h.

After setting, remove the specimen from the mould and remove any flash by abrading it with P150 or P320 abrasive paper, taking care not to damage the specimen, and store in distilled water at (37 ± 1) °C for (24 ± 1) h before testing.

Prepare five such specimens.

C.2.2 Specimen preparation — Class 2 and 3 materials

Fill the mould with the material within the working time of the cement (see Table 2, item 24) and cover both sides with a polyester film (C.1.2.4) and glass plate (C.1.2.3). Place the exit window of the external energy source (C.1.2.8) 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 and irradiate for the recommended exposure time.

NOTE The degree of overlap should incorporate not more than half of the previous area of irradiation covered by the light-activation probe.

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 for the recommended exposure time. Repeat the irradiation procedure on the other side of the specimen. Clamp (C.1.2.7) the assembly and place it in the water bath (C.1.2.5) maintained at (37 ± 1) °C, for 15 min. Then, remove the specimen from the mould, mark the specimen at one end to indicate the face which was cured first and remove any flash by abrading it with P150 or P320 abrasive paper, avoiding the top and bottom surfaces and store in distilled water at (37 ± 1) °C for (24 ± 1) h before testing.

Prepare five such specimens.

C.3 Procedure

After (24 ± 1) h storage in water at (37 ± 1) °C, carefully remove each specimen from the water bath and measure the dimensions of the specimen to an accuracy of 0,01 mm using the micrometer (C.1.2.6) at the centre of the specimen. Visually inspect each specimen without magnification and reject any specimens having surface defects or air inclusions. Replace the specimen in the water bath in order to equilibrate at (37 ± 1) °C. After (24 ± 1) h storage in water, transfer the specimen to the flexural properties testing equipment (C.1.2.1), ensuring that the first cured surface of the specimen (as constructed in the mould) remains the lower surface during testing (i.e. the side in tension). The specimen shall be positioned in the centre of the test jig (C.1.2.2), perpendicular to the three rods. Within 10 s of removing the specimen from the water bath, load the specimen at a crosshead speed of (0,75 ± 0,25) mm/min or at a rate of loading of (50 ± 16) N/min, applied until the specimen fractures.

Record the maximum force exerted on the specimen.

Repeat the test on the four other specimens.

C.4 Calculation and expression of results

Calculate the flexural strength, σ, in megapascals, from the following equation:

$$\sigma = \frac{3FL}{2bh^2}$$

where

F is the maximum force, 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 prior to testing;

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

C.5 Treatment of results

Compare the values of flexural strength with the limit value specified in Table 1. If four or five results are not less than the minimum value, the material passes the test. If two or fewer results satisfy the limit value, the material fails the test. If three specimens satisfy the limit value, prepare and test five further specimens. All five results in the second series shall comply with the limit value specified in Table 1.

Annex D
(normative)

Determination of radio-opacity

D.1 Apparatus

NOTE Conventional or digital X-ray equipment can be used.

D.1.1 Conventional equipment

D.1.1.1 Single-phase dental X-ray unit, with a total filtration equivalent of 1,5 mm of aluminium and capable of operation at (65 ± 5) kV.

D.1.1.2 Dental X-ray occlusal film, of speed group D (as specified in ISO 3665) in its "as normally used" packet. The X-ray film shall not be laminated or otherwise treated.

D.1.1.3 Freshly-prepared developing solution and fixer, as recommended by the manufacturer.

D.1.1.4 Densitometer, using white light and capable of measuring in the optical density range 0 to 3,0 to a resolution of 0,01, calibrated at zero and against a reference with optical density of (2,5 ± 0,5) known to an accuracy of 0,01, and using an aperture of (2,0 ± 0,1) mm.

D.1.2 Digital equipment

D.1.2.1 Digital X-ray unit or single-phase dental X-ray unit, with a total filtration equivalent of 1,5 mm of aluminium and capable of operation at (65 ± 5) kV.

D.1.2.2 X-ray sensor of occlusal film size, e.g. charge-coupled device (CCD), photo stimulable phosphor plate (imaging plate), calibrated for use with single-phase dental X-ray unit (D.1.2.1) with appropriate software.

D.1.2.3 Grey scale analysis software [a suitable product available commercially is Adobe® Photoshop®[1)]].

D.1.2.4 Aluminium step wedge, purity at least 98 % (mass fraction) with less than 0,1 % (mass fraction) copper and less than 1,0 % (mass fraction) iron present, 50 mm long × 20 mm wide, having a thickness range from 0,5 mm to 5,0 mm in equally spaced steps of (0,50 ± 0,01) mm. The wedge shall be free standing.

D.1.2.5 Specimen mould, for the preparation of specimen discs, (15 ± 1) mm in diameter and thickness (1,0 ± 0,1) mm (e.g. a metal washer).

D.1.2.6 Film, transparent (50 ± 30) µm thick, made of, for example, polyester, which shall be non-adherent to the test material and untextured.

D.1.2.7 Slides/plates, made of glass or stainless steel or some other smooth and rigid material.

1) Supplied by Adobe Systems Incorporated, 345 Park Avenue, San Jose, CA 95110-2704. This information is given for the convenience of users of this part of ISO 9917 and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

D.1.2.8 Cabinet, capable of being maintained at a temperature of (37 ± 1) °C and a relative humidity of at least 50 %.

D.1.2.9 Screw micrometer gauge or equivalent device, accurate to 0,01 mm.

D.1.2.10 Individual clamps, clips or equivalent devices, designed to hold the specimen mould together during setting of the cement.

D.1.2.11 Abrasive paper, P2000 or P2 500 grade.

D.1.2.12 Lead sheet, at least 2,0 mm thick.

D.1.2.13 External energy source(s), as recommended by the manufacturer for activating the polymerization of light-activated materials (see Table 2, item 26).

D.2 Preparation of test specimens

Place a sheet of film (D.1.2.6) on a plate (D.1.2.7). Place the mould (D.1.2.5) on the film. Slightly overfill the mould with test material. Place a piece of film on the material in the mould and cover this with a second plate, thus expressing excess material. Secure the assembly in a clamp (D.1.2.10) to ensure that a specimen of the correct thickness is produced. For light curing materials, cure using a suitable unit (D.1.2.13) and overlapping 1 min exposures for achieving a proper cure. Ensure that the material is fully cured by using overlapping exposures, if necessary.

Place the assembly in the cabinet (D.1.2.8) for 30 min.

NOTE For light-activated materials, use overlapping 1 min exposures for achieving a proper cure.

Remove the specimen from the mould and measure the thickness of the disc near its centre with the gauge (D.1.2.9). Use only specimens whose thickness falls in the range (1,0 ± 0,1) mm. If the specimens are oversized, they may be ground using abrasive paper (D.1.2.11) until the thickness lies within the specified range.

Store the specimens in water of grade 3, as defined in ISO 3696, at (23 ± 1) °C for no more than 7 d before testing. In order to avoid dehydration of the test specimen, make the determination of radio-opacity within 30 min of removing the specimen from the water.

D.3 Procedure

D.3.1 Conventional equipment

Measure the thickness of the specimen, T_s, to an accuracy of 0,01 mm. This is the measurement of thickness which shall be used in the determination of radio-opacity.

Position the X-ray film (D.1.1.2) on the lead sheet (D.1.2.12). Place the specimen and the aluminium step wedge (D.1.2.4) near the centre of the film.

Irradiate the specimen, aluminium wedge and film with X-rays at (65 ± 5) kV at a target film distance of 300 mm to 400 mm for such a time that, after processing, the region of film beside the specimen and aluminium has an optical density of between 1,5 and 2.

NOTE Exposures of between 0,3 s and 0,4 s at 10 mA are typical.

After developing and fixing the film, check the image of the specimen such that any porosity visible to the unaided eye is avoided when making the optical density measurements. If such porosity is not avoidable, the specimen shall be replaced and the procedure repeated. Measure the optical density of the image of the

specimen near its centre, and that of each step of the image of the aluminium wedge using the densitometer (D.1.1.4).

Do not laminate or otherwise treat the X-ray film.

D.3.2 Digital equipment

Determinations shall be made by a method equivalent to that used with conventional equipment (e.g. by measuring and comparing grey values). Disable the automatic gain control using the software of the digital X-ray system prior to obtaining the image.

NOTE 1 Testing has shown that these units should be used without automatic gain control for the purposes of this part of ISO 9917.

Measure the thickness of the specimen, T_s, to an accuracy of 0,01 mm. This is the measurement of thickness which shall be used in the determination of radio-opacity.

Position the X-ray sensor (D.1.2.2) on the lead sheet (D.1.2.12). Place the specimen and the aluminium step wedge (D.1.2.4) near the centre of the sensor. Irradiate the assembly with X-rays at a cathode target-film distance of 400 mm without using the automatic gain control. Repeat the procedure to find the appropriate exposure time to make a clear image.

NOTE 2 Exposures are expected to be five to ten times longer than for conventional X-ray film.

Export the digital image file to the grey scale analysis software. The number of grey shades is assessed using the measuring tool in the software. The number of grey shades in the digital image is given by the number of binary digits (bits) used to define a pixel.

Using the grey scale analysis software, define a rectangular area to measure in the specimen image using the software selection tool. Click the “Image” function and then click the “Histogram” function. This will provide the average grey scale in the area.

Repeat this procedure with each of the steps of the step wedge.

In the grey scale, the darkest grey shade is usually defined by the value zero, while the lightest has a value of 255. This score is the reverse order to the density of X-ray film.

D.4 Interpretation of results

Plot the individual optical densities of each aluminium step against the thickness of each step. Take the optical density value for the specimen of thickness, T_s, and determine from the plot the corresponding value of the thickness of aluminium, T_a, at this optical density. The radio-opacity (aluminium equivalent) value of a specimen is then given by (T_a/T_s). If this value is $\geqslant 1$, the material is deemed to have complied with the first requirement of 5.6.

If the manufacturer has claimed a specific value for radio-opacity (see Table 2, item 17), then the aluminium equivalent determined above shall be no more than 0,5 mm below the value claimed by the manufacturer.

The plot of optical density against aluminium thickness of the step wedge shall be made for each radiographic exposure, since minor variations may occur due to radiographic processing.

When determinations have been made using digital equipment, an equivalent method for interpretation shall be made to that used with conventional equipment (e.g. by using grey scale instead of optical density).

Annex E
(normative)

Determination of shade and colour stability — Restorative and luting materials only

E.1 Apparatus

E.1.1 Cabinet, with water container or water bath capable of being maintained at (37 ± 1) °C.

E.1.2 Radiation source, water bath and other apparatus, as described in ISO 7491.

E.1.3 Film, (50 ± 30) µm thick, transparent to the activating radiation, e.g. polyester.

E.2 Preparation of test specimens

Prepare two disc specimens (1,0 ± 0,1) mm thick as described in D.2 using a mould as described in D.1.2.5.

E.3 Procedure

Store one specimen in the dark, in water, for 7 d at (37 ± 1) °C; this is the reference specimen.

Store the second specimen in the same manner as the first for (24 ± 2) h, then blank off half of the second specimen with aluminium or tin foil. Transfer this specimen to the radiation chamber, immerse it in water at (37 ± 5) °C and expose it to the radiation for 24 h. Ensure that the water level is (10 ± 5) mm above the specimen. After exposure, remove the metal foil, then transfer the specimen back to the oven at (37 ± 1) °C and store it in the dark, in water, for 5 d. Compare the colour of both halves of the second specimen with each other and with the reference specimen and the manufacturer's nominated shade guide (see Table 2, item 14). Carry out the colour comparison in accordance with 5.7 and ISO 7491.

The above colour comparison shall be made by three independent observers.

E.4 Treatment of results

The observations of each of the three independent observers shall be noted and compared with the requirements outlined in 5.7.

Bibliography

[1] ISO 4049, *Dentistry — Polymer-based restorative materials*

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

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

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