INTERNATIONAL STANDARD

ISO 4049

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Dentistry — Polymer-based restorative materials

Art dentaire — Produits de restauration à base de polymères

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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 4049 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 1, *Filling and restorative materials*.

This fourth edition cancels and replaces the third edition (ISO 4049:2000) which has been reviewed and essentially reconfirmed. Several minor changes have been made to clarify content. Changes have been made to the test method for radio-opacity (see 7.14) in order to simplify this test.

Introduction

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

Dentistry — Polymer-based restorative materials

1 Scope

This International Standard specifies requirements for dental polymer-based restorative materials supplied in a form suitable for mechanical mixing, hand-mixing, or intra-oral and extra-oral external energy activation, and intended for use primarily for the direct or indirect restoration of cavities in the teeth and for luting.

The polymer-based luting materials covered by this International Standard are intended for use in the cementation or fixation of restorations and appliances such as inlays, onlays, veneers, crowns and bridges. This International Standard does not cover those polymer-based luting materials that have an adhesive component within the structure of the material.

This International Standard does not cover materials intended to prevent caries (see ISO 6874) or those used for veneering metal sub-frames (see ISO 10477).

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

3.2
opaque
shade of an intensely pigmented polymer-based restorative material

4 Classification

For the purposes of this International Standard, dental polymer-based restorative materials are classified as the following types.

a) **Type 1**: polymer-based restorative materials claimed by the manufacturer as suitable for restorations involving occlusal surfaces;

b) **Type 2**: all other polymer-based restorative materials, and luting materials.

The three classes of dental polymer-based restorative materials are as follows.

— **Class 1**: materials whose setting is effected by mixing an initiator and activator ("self-curing" materials).

— **Class 2**: materials whose setting is effected by the application of energy from an external source, such as blue light or heat ["external-energy-activated" materials, see also 8.3 e)]. They are subdivided as follows:

  1) **Group 1**: materials whose use requires the energy to be applied intra-orally;

  2) **Group 2**: materials whose use requires the energy to be applied extra-orally. When fabricated, these materials will be luted into place.

Certain materials may be claimed by manufacturers to be both Group 1 and Group 2. In this event, the material should fulfil the requirements for both groups.

NOTE Class 2 luting materials will fall into Group 1 only.

— **Class 3**: materials that are cured by the application of external energy and also have a self-curing mechanism present ("dual cure" materials).

5 Requirements

5.1 Biocompatibility

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

5.2 Physical and chemical properties

5.2.1 General

If a restorative material is supplied by the manufacturer in various shades, each shade, including opaque shades, shall be capable of satisfying all the requirements for sensitivity to ambient light (5.2.7), depth of cure (5.2.8), shade (5.3) and colour stability (5.4) appropriate to the material type and class. If the material is supplied such that it can be "tinted" or "blended" to the user's prescription, the material shall comply with the requirements both when used alone and when used with the maximum recommended proportion of tint or blender [see 8.3 d)].

Similarly, if the manufacturer supplies a luting material in various shades, each shade, including opaque luting materials, shall be capable of satisfying all the requirements for depth of cure (5.2.8). Colour stability (5.4) of luting materials shall not be tested unless the manufacturer claims such a property.

In respect of the other requirements of 5.2 and those of 5.5, only one representative shade of restorative material shall be tested. This representative shade shall be either that classified by the manufacturer as

"universal" or, in the event that no shade is so classified, that shade corresponding to "A3" in the Vita® 1) classification of shade. However, if the manufacturer claims a higher value for radio-opacity [see 5.5 and 8.3 o)] for any other shade, this claim shall be tested.

The requirements are summarised in Tables 1, 2 and 3.

5.2.2 Film thickness, luting materials

The film thickness of luting materials when determined in accordance with 7.5 shall be no more than 10 μm above any value claimed by the manufacturer and in any event shall be no greater than 50 μm.

5.2.3 Working time, Class 1 and Class 3 restorative materials, excluding luting materials

The working time for Class 1 and Class 3 restorative materials, excluding luting materials, determined in accordance with 7.6, shall be not less than 90 s.

5.2.4 Working time, Class 1 and Class 3 luting materials

When tested in accordance with 7.7, the material shall be capable of forming a thin layer; during its formation there shall be no detectable change in its homogeneity.

5.2.5 Setting time, Class 1 materials

The setting time for Class 1 restorative materials, excluding luting materials, determined in accordance with 7.8, shall be not more than 5 min. The setting time for Class 1 luting materials, determined in accordance with 7.8, shall be not more than 10 min.

5.2.6 Setting time, Class 3 materials

The setting time for Class 3 materials, determined in accordance with 7.8, shall be not more than 10 min.

5.2.7 Sensitivity to ambient light, Class 2 materials

When tested in accordance with 7.9, the material shall remain physically homogeneous.

5.2.8 Depth of cure, Class 2 materials

When determined in accordance with 7.10, the depth of cure of Class 2 restorative materials, excluding luting materials, shall be not less than 1 mm if they are labelled by the manufacturer as opaque, or not less than 1,5 mm for other restorative materials.

The depth of cure of luting materials when determined in accordance with 7.10 shall be not less than 0,5 mm if they are labelled by the manufacturer as opaque materials, or not less than 1,5 mm for other materials.

In any event, the values for all materials, with the exception of opaque luting materials, shall be no more than 0,5 mm below the value stated by the manufacturer.

5.2.9 Flexural strength

The flexural strength of polymer-based restorative materials determined in accordance with 7.11 shall be equal to or greater than the limits specified in Table 1.

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1) Vita® is a trade name of Vita Zahnfabrik, H Rauter GmbH & Co K G, Postfach 1338, D-79704 Bad Sackingen, Germany. This information is given for the convenience of the users of this International Standard and does not constitute an endorsement of this system by ISO.

Table 1 — Flexural strength

Restorative Materials		Flexural strength MPa minimum
Type 1	Class 1	80
	Class 2, Group 1	80
	Class 2, Group 2	100
	Class 3	80
Type 2 (including luting materials)	Class 1	50
	Class 2, Group 1	50
	Class 3	50

5.2.10 Water sorption and solubility

When determined in accordance with 7.12:

a) the water sorption of all materials shall be $\leqslant$ 40 µg/mm^3.

b) the solubility of all materials shall be $\leqslant$ 7,5 µg/mm^3.

5.3 Shade, restorative materials

When the material is assessed in accordance with 7.13 and ISO 7491, the shade of the set material shall match closely that of the manufacturer's shade guide. If a shade guide is not supplied by the manufacturer, then the manufacturer shall nominate a commercially available shade guide that shall be used in assessing compliance with this requirement [see 8.3 l)]. In addition, the set material shall be evenly pigmented when viewed without magnification.

5.4 Colour stability after irradiation and water sorption

When the material is tested in accordance with 7.13 and ISO 7491, no more than a slight change in colour shall be observed. In respect of luting materials, colour stability shall be tested only in the event of a manufacturer's claim for colour stability. In the event of such a claim, no more than a slight change in colour shall be observed after the material has been tested in accordance with 7.13 and ISO 7491.

5.5 Radio-opacity

5.5.1 If the manufacturer claims that the material is radio-opaque [see 8.2.3 h)], the radio-opacity, determined in accordance with 7.14, shall be equal to or greater than that of the same thickness of aluminium and no less than 0,5 mm below any value claimed by the manufacturer.

5.5.2 This test shall be performed on a "universal" shade (see 5.2.1), but if the manufacturer claims a value for one or more other shades that is at least twice the "universal" shade value, this other shade or shades shall be tested as described in 5.5.1 [see 8.3 o)].

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

Table 2 — Physical and chemical property requirements for restorative materials, excluding luting materials (see Table 1 for minimum flexural strength)

Material Class	Requirement (subclause)				
	Working time (5.2.3) s minimum	Setting time (5.2.5, 5.2.6) min maximum	Depth of Cure [a] (5.2.8) mm minimum	Water sorption (5.2.10) $\mu g/mm^3$ maximum	Solubility (5.2.10) $\mu g/mm^3$ maximum
Class 1	90	5 (5.2.5)	—	40	7,5
Class 2	—	—	1,0 (opaque shade) 1,5 (others)	40	7,5
Class 3	90	10 (5.2.6)	—	40	7,5

[a] The values for all materials shall be no more than 0,5 mm below the value stated by the manufacturer.

Table 3 — Physical and chemical property requirements for luting materials

Material Class	Requirement (subclause)					
	Film thickness [a] (5.2.2) µm maximum	Working time (5.2.4) s minimum	Setting time (5.2.5, 5.2.6) min maximum	Depth of cure [b] (5.2.8) mm minimum	Water sorption (5.2.10) $\mu g/mm^3$ maximum	Solubility (5.2.10) $\mu g/mm^3$ maximum
Class 1	50	60	10 (5.2.5)	—	40	7,5
Class 2	50	—	—	0,5 (opaquer) 1,5 (others)	40	7,5
Class 3	50	60	10 (5.2.6)	—	40	7,5

[a] The determined value shall be no more than 10 µm above any value claimed by the manufacturer.

[b] In any event, the values for all materials, with the exception of opaque luting materials, shall be no more than 0,5 mm below the value stated by the manufacturer.

6 Sampling

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

NOTE 50 g should be sufficient.

7 Test methods

7.1 General reagent — Water

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

7.2 Test conditions

Unless specified otherwise by the manufacturer, prepare and test all specimens at (23 ± 1) °C. Control the relative humidity to ensure that it remains greater than 30 % and less than 70 % at all times. If the material was refrigerated for storage, allow it to attain (23 ± 1) °C.

For Class 3 materials, the tests for working time (see 7.6) and setting time (see 7.8) shall be performed in the absence of activating radiation.

NOTE Ambient light, both natural and artificial, is capable of activating these materials. For good control, the test should be performed in a dark room with any artificial light filtered by a yellow filter. [2)]

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, reference shall be made to the manufacturer's instructions [see 8.3 e)] that state the external energy source or sources recommended for the materials to be tested. Care shall be taken to ensure that the source is in a satisfactory operating condition. [ISO 10650 (both parts) gives guidance on this.]

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

Where fully cured specimens are required for testing (7.11 to 7.14), it is important to ensure that the specimens are homogeneous after removal from the mould. There shall be no clefts, voids, discontinuities or air inclusions present when viewed without magnification.

Some polymer-based materials, particularly certain luting materials, have a chemical affinity for base metals. This property creates difficulty when removing specimens from metal moulds. Reference shall be made to the information supplied by the manufacturer [see 8.3 m)] regarding this property and, if it is claimed, moulds for the preparation of specimens of such materials may be made from non-metallic material such as high-density polyethylene.

7.5 Measurement of film thickness of luting materials

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. Each plate shall be of 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 may 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 shall be horizontal and parallel to the base. The load shall be applied smoothly and in such a manner that no rotation occurs.

NOTE A holder can be used to assist in the positioning of the plates. Such a device consists of a baseplate with three vertical pins to align circular plates or four pins to align square plates. (See Figure 2.)

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.

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2) 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 International Standard and does not constitute an endorsement of this product by ISO.