



Revised American National Standard/
American Dental Association
Standard No. 96

Dental Water-based Cements

Modified adoption of ISO 9917-1:2007, Dentistry — *Water-based cements - Part 1: Powder/liquid acid-base cements* and ISO 9917-2:2010, Dentistry – *Water-based cements - Part 2: Resin-modified cements*.

ADA American
Dental
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Council on
Scientific Affairs

2012

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REVISED AMERICAN NATIONAL STANDARD/AMERICAN DENTAL ASSOCIATION STANDARD NO. 96 FOR DENTAL WATER-BASED CEMENTS

The Council on Scientific Affairs of the American Dental Association has approved revised American Dental Association Standard No. 96 for Dental Water-Based Cements. This and other standards for dental materials, instruments and equipment are being formulated by working groups of the ADA Standards Committee on Dental Products (formerly Accredited Standards Committee MD156 for Dental Materials, Instruments and Equipment). The Committee has representation from all interests in the United States in the standardization of materials, instruments and equipment in dentistry. The Council has adopted the standards, showing professional recognition of their usefulness in dentistry, and has forwarded them to the American National Standards Institute with a recommendation that the standards be approved as American National Standards. The American National Standards Institute granted approval of revised ADA Standard No. 96 as an American National Standard on November 26, 2012.

The ADA Standards Committee on Dental Products thanks the members of Working Group 1.4 and the organizations with which they were affiliated at the time the standard was developed:

John Powers (chairman), Dental Consultants, Inc., Ann Arbor, MI;
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REVISED AMERICAN NATIONAL STANDARD/AMERICAN DENTAL ASSOCIATION STANDARD NO. 96 FOR DENTAL WATER-BASED CEMENTS

FOREWORD

(This Foreword does not form a part of revised ANSI/ADA Standard No. 96 for Dental Water-based Cements).

This revised standard is an adoption of ISO 9917-1:2007, Dentistry — Water-based cements - Part 1: Powder/liquid acid-base cements and ISO 9917-2:2010, Dentistry – Water-based cements - Part 2: Resin-modified cements. ADA SCDP Working Group No. 1.4 on Dental Water-based Cements examined the international standards and found them acceptable as a modified adoption for revised ANSI/ADA Standard No. 96. This standard cancels and replaces ANSI/ADA Standard No. 96 – 2005, which was a modified adoption of ISO 9917:1991, Dental water-based cements.

The modifications made to ISO 9917-1:2007 and ISO 9917-2:2010 consist of combining them to form revised ANSI/ADA Standard No. 96 and the revision of certain terms and definitions to bring them into conformance with ISO 1942:2009, *Dentistry-Vocabulary* and ISO 8601, *Data elements and interchange formats-information interchange-representation of dates and times*. Revised were:

- the definition of "working time";
- the replacement of term "immediate wrapping" with "primary packaging";
- the replacement of the term "outermost packaging" and "outer container" with "secondary container" in Part 1 and "outer pack" with "secondary container" in Part 2; and
- the replacement of the term "expiry date" with "use by date."

The statement "The net mass in each container/capsule" in the Manufacturer's Instruction Leaflet was changed from mandatory to optional. This information is already required on the Secondary Container of capsules, which was deemed sufficient.

This standard is presented in two parts: 1). Powder/liquid acid-base cements; and 2). Resin-modified cements.

Specific qualitative and quantitative requirements for freedom from biological hazard are not included in this standard. In assessing possible biological or toxicological hazards, reference should be made to ANSI/ADA Standard No. 41, ISO 7405 and ISO 10993-1.

REVISED AMERICAN NATIONAL STANDARD/AMERICAN DENTAL ASSOCIATION STANDARD NO. 96 FOR
DENTAL WATER-BASED CEMENTS

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PART 1:

POWDER/LIQUID ACID-BASE CEMENTS

1 Scope

Part 1 of ADA Standard No. 96 specifies requirements and test methods for powder/liquid acid-base dental cements intended for permanent cementation, lining and restoration. Part 1 is applicable to both hand-mixed and capsulated cements for mechanical mixing. Part 1 specifies limits for each of the properties according to whether the cement is intended for use as a luting agent, a base or liner or as a restorative material. Part 1 of this standard is not intended to address resin-modified water-based cements, which are addressed in Part 2.

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 2590, *General method for the determination of arsenic — Silver diethyldithiocarbamate photometric method*

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

ISO 3696:1987, *Water for analytical laboratory use — Standard and test methods*

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

(ISO standards for dentistry are available from the American Dental Association, Department of Standards, 211 E. Chicago Ave., Chicago, IL 60611 or <http://catalog.ada.org>. ISO standards are available from the American National Standards Institute, 25 W. 43rd St., New York, NY 10036 or www.ansi.org).

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

mixing time

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

3.2

working time

period of time, measured from the commencement of mixing components or otherwise activating the chemistry of the material and ending before the material has developed properties that will prevent it from being manipulated as required to achieve its purpose in a subsequent procedure or in its intended function
[derived from ISO 1942:2009, Dentistry-Vocabulary]

3.3

net setting time

period of time, measured from the end of mixing, until the material has set according to the criteria and conditions specified in Annex A

NOTE For the purposes of this part of ADA Standard No. 96, in view of the wide variation in mixing times of cements, the net setting time is determined from the end of mixing.

4 Classification

4.1 Chemical type

For the purposes of this part of ADA Standard No. 96, dental cements shall be classified on the basis of their chemical composition, as follows:

- a) zinc phosphate cement, see Clause B.1;
- b) zinc polycarboxylate cement, see Clause B.2;
- c) glass polyalkenoate cement, see Clause B.3.

Acid-base setting, water-based cements other than those listed above may fall within the scope of this part of ADA Standard No. 96. If the manufacturer wishes to claim conformity for such a product, the type of material for which equivalence of properties is claimed shall be specified in accordance with 4.1 and 4.2 so that the correct performance limits are applied.

4.2 Application

For the purposes of this part of ADA Standard No. 96, the application of water-based cements shall be classified as follows:

- a) luting;
- b) bases or lining;
- c) restoration.

5 Material

5.1 General

The cement shall consist of a powder and liquid which, when mixed in accordance with the manufacturer's instructions, shall conform to requirements in this clause and Clause 8.

5.2 Components

5.2.1 Liquid

For non-encapsulated cements, visually inspect the liquid. It shall be free from deposits or filaments on the inside of its container. There shall be no visible signs of gelation.

5.2.2 Powder

For non-encapsulated cements, visually inspect the powder. It shall be free from extraneous material. If the powder is colored, the pigment shall be uniformly dispersed throughout the powder.

5.3 Unset cement

The cement shall be mixed in accordance with Clause 6, and then visually inspected. It shall be homogeneous and of a smooth consistency.

6 Preparation of test specimens

6.1 Ambient conditions

Specimens shall be prepared at a temperature of $(23 \pm 1)^\circ\text{C}$ and a relative humidity of $(50 \pm 10)\%$.

6.2 Method of mixing

The cement shall be prepared in accordance with 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, may be required for certain specimens.

7 Sampling

7.1 Hand-mixed cements

A sample drawn from one batch shall provide sufficient material to complete all the prescribed tests and any necessary repeats.

NOTE Approximately 50 g of powder is necessary to complete the tests.

7.2 Encapsulated cements

The test sample shall comprise a retail package of a sufficient number of capsules from one batch to complete all the tests and any necessary repeats.

8 Requirements

8.1 Net setting time

When determined in accordance with Annex A, the net setting time of the cement shall conform to relevant requirements specified in Table 1.

8.2 Film thickness (luting cements only)

When determined in accordance with Annex C, the film thickness shall conform to relevant requirements specified in Table 1.

8.3 Compressive strength

When determined in accordance with Annex D, the compressive strength of the cement shall conform to the relevant requirements specified in Table 1.

8.4 Acid erosion

When determined in accordance with Annex E, the acid erosion of the cement shall conform to the relevant requirements specified in Table 1.

8.5 Optical properties (polyalkenoate restorative cements only)

When prepared, stored and tested in accordance with Annex F, cements shall meet the following requirements.

- a) The opacity of the set restorative cements shall be within the limits specified in Table 1, unless the restorative cement is designated as opaque by the manufacturer [see 9.2 f)].
- b) The color of the set cement shall match 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 9.3.1 c)].

8.6 Acid-soluble arsenic and lead contents

8.6.1 Acid-soluble arsenic content

When determined in accordance with Annex G, the acid-soluble arsenic content shall not exceed the relevant limits specified in Table 1.

8.6.2 Acid-soluble lead content

When determined in accordance with Annex G, the acid-soluble lead content shall not exceed the relevant limits specified in Table 1.

8.7 Radio-opacity (if claimed)

When the manufacturer describes the material as being radio-opaque [see 9.2 g)], the radio-opacity shall be at least equivalent to that for the same thickness of aluminum when determined in accordance with Annex H. When the manufacturer claims a greater radio-opacity, the measured value shall not be less than the value claimed when determined in accordance with Annex H.

9 Packaging, marking and information to be supplied by manufacturer

NOTE Additional information can be included at the discretion of the manufacturer or as required by other applicable rules or regulations.

9.1 Packaging

The materials shall be supplied in containers or capsules (for the purposes of this part of ADA Standard No. 96, the container or capsule shall be considered to be the primary packaging of the material) that afford adequate protection and have no adverse effect on the quality of the contents.

A secondary container may also be used to present the containers or capsules as a single unit.

9.2 Marking

Each secondary container shall be clearly marked with the following details:

- a) the name and/or trademark of the manufacturer and the trade name, type and application of the cement;
- b) the shade of the powder according to the manufacturer's nominated shade guide;
- c) the minimum net mass, in grams, of the powder or the minimum net volume, in milliliters of the liquid as appropriate;
- d) the manufacturer's batch or lot number;
- e) on the secondary container, the recommended conditions of storage, and the use by date for the material under those conditions of storage;
- f) on the secondary container, whether the cement is designated opaque;
- g) on the secondary container, whether the cement is designated as radio-opaque;
- h) on each secondary container of encapsulated cements, the number of capsules in the container, and the net mass in each capsule.

9.3 Manufacturer's instructions

9.3.1 General

Instructions shall accompany each package of the material and shall include the following:

- a) the trade name of the product;
- b) the manufacturer's name and contact details;
- c) a shade guide or, when no shade guide is provided, details of a commercially available shade guide for use with the material.

In addition, at least the information in 9.3.2 or 9.3.3, as appropriate, shall be given. For those materials not mentioned specifically in 4.1, the manufacturer shall indicate the type of material (see 4.1 and 4.2) against whose performance requirements the material shall be tested.

9.3.2 Hand-mixed cements

For hand-mixed cements, the following information shall be supplied:

- a) the temperature range for preparation;
- b) the recommended powder:liquid ratio, expressed as a mass:mass ratio for the recommended temperature range, and an indication of how the user may achieve this ratio [for test purposes, the powder:liquid ratio on a mass:mass basis to a precision of 0.01 g at a temperature of $(23 \pm 1)^\circ\text{C}$ and a relative humidity of $(50 \pm 10)\%$ shall be included];
- c) the type of the mixing slab and spatula and their condition;
- d) the rate of incorporation of the powder into the liquid;
- e) the mixing time (see 3.1);
- f) the working time (see 3.2);
- g) the net setting time (see 3.3);
- h) if appropriate, a statement recommending that a liner should be placed between the cement and the dentine;
- i) if appropriate, the minimum time at which finishing may be commenced and the recommended method of finishing;
- j) if appropriate, a statement indicating that the surface of the cement must be coated with a protective coating and guidance on the type of coating to be used.

9.3.3 Encapsulated cements

For encapsulated cements the following instructions shall be supplied:

- a) the method of bringing about physical contact between the powder and liquid;
- b) the type of mechanical mixing device and the mixing time to be employed;
- c) the net setting time (see 3.3);
- d) the working time (see 3.2);
- e) if appropriate, a statement recommending that a liner should be placed between the cement and the dentine;
- f) if appropriate, the minimum time at which finishing may be commenced and the recommended method of finishing;
- g) the minimum deliverable volume, in milliliters, of mixed cement in one capsule;
- h) if appropriate, a statement indicating that the surface of the cement must be coated with a protective coating and guidance on the type of coating to be used.

Table 1 — Requirements for dental cements

Chemical type	Application	Film thickness μm max.	Net setting time minutes min. max.		Compressive strength MPa min.	Acid erosion mm max.	Opacity $C_{0,70}$ min. max.		Acid-soluble As content mg/kg max.	Acid-soluble Pb content mg/kg max.
Zinc phosphate	Luting	25	2.5	8	50	0.30			2	100
Zinc polycarboxylate	Luting	25	2.5	8	50	0.40			2	100
Glass polyalkenoate	Luting	25	1.5	8	50	0.17				100
Zinc phosphate	Base/lining		2	6	50	0.30			2	100
Zinc polycarboxylate	Base/lining		2	6	50	0.40			2	100
Glass polyalkenoate	Base/lining		1.5	6	50	0.17				100
Glass polyalkenoate	Restoration		1.5	6	100	0.17	0.35	0.90		100

Annex A

(normative)

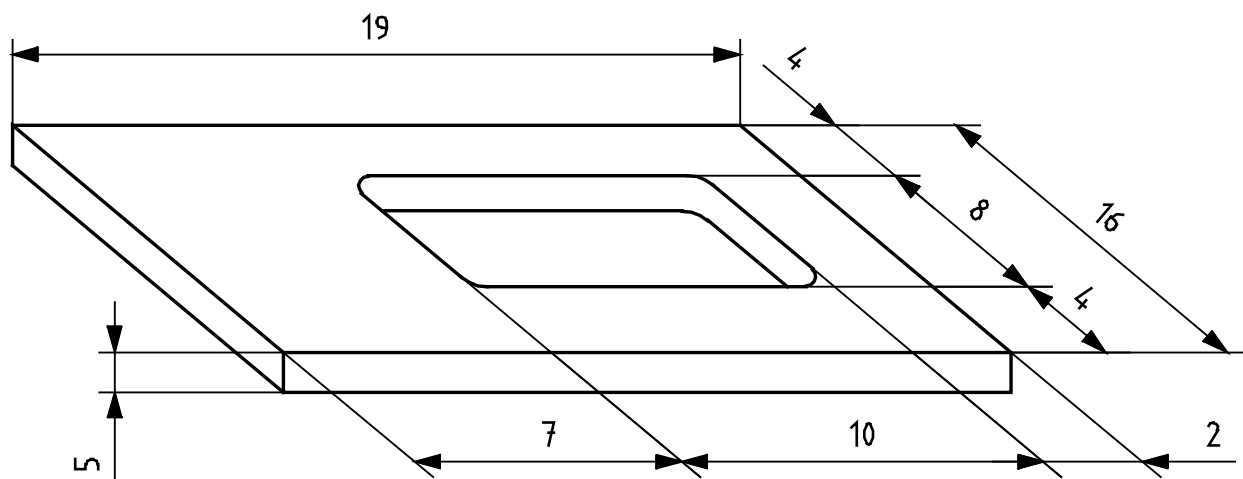
Determination of net setting time

A.1 Apparatus

- A.1.1 Cabinet, capable of being maintained at a temperature of $(37 \pm 1)^\circ\text{C}$ and a relative humidity of at least 90 %.
- A.1.2 Indentor, of mass (400 ± 5) g, with a needle having a flat end of diameter (1.0 ± 0.1) mm which is plane and perpendicular to the long axis of the needle.
- A.1.3 Metal mold, similar to that shown in Figure A.1.
- A.1.4 Metal block, of minimum dimensions 8 mm $\times$ 75 mm $\times$ 100 mm positioned within the cabinet and maintained at $(37 \pm 1)^\circ\text{C}$.
- A.1.5 Aluminum foil.
- A.1.6 Timer, accurate to 1 s.

Dimensions in millimeters

Tolerance on dimensions ± 0.15



NOTE Internal corners may be square or rounded.

Figure A.1 — Mold for preparation of specimens for determination of net setting time

A.2 Procedure

Place the mold (A.1.3), conditioned to $(23 \pm 1) ^\circ\text{C}$, on the aluminum foil (A.1.5) and fill to a level surface with mixed cement. Sixty seconds after the end of mixing, place the assembly, comprising mold, foil and cement specimen, on the block (A.1.4), in the cabinet (A.1.1). Ensure good contact between the mold and foil, and between the foil and block.

Ninety seconds after the end of mixing, carefully lower the indenter (A.1.2) vertically on to the surface of the cement and allow it to remain there for 5 s. Carry out a trial run to determine the approximate setting time, repeating the indentations at 30 s intervals until the needle fails to make a complete circular indentation in the cement, when viewed using $\times 2$ magnification. Clean the needle, if necessary, between indentations. Repeat the process, starting the indentation at 30 s before the approximate setting time thus determined, making indentations at 10 s intervals.

Record the net setting time as the time elapsed between the end of mixing and the time when the needle fails to make a complete circular indentation in the cement. Repeat the test two more times.

A.3 Treatment of results

Record the results of the three tests. Each result shall fall within the range specified in Table 1 for the material to meet the requirements of 8.1.

Annex B

(informative)

Chemical composition and applications of dental cements

B.1 Zinc phosphate cements

Zinc phosphate cements are based on the reaction between an oxide powder (the principal constituent of which is zinc oxide) and an aqueous solution of phosphoric acid which may contain metal ions. They can be used to cement dental appliances to hard oral structures or to other appliances. They can also be used as bases for restorative materials and as temporary restorative materials.

B.2 Zinc polycarboxylate cements

Zinc polycarboxylate cements are based on the reaction between zinc oxide and aqueous solutions of polyacrylic acid or similar polycarboxylic compounds, or zinc oxide/polycarboxylic acid powders which are mixed with water. They can be used to cement dental appliances to hard oral structures or to other appliances. They can also be used as a base for restorative materials and as a temporary restorative material.

B.3 Glass polyalkenoate cements

Glass polyalkenoate (glass ionomer) cements are based on the reaction between an aluminosilicate glass powder and an aqueous solution of a polyalkenoic acid, or between an aluminosilicate glass/polyalkenoic acid powder blend and water, or an aqueous solution of tartaric acid. These cements are used for the aesthetic restorations of teeth or as bases or liners for other restorations. They can also be used to cement dental appliances to hard oral structures or to other appliances. This type also includes glass polyalkenoate cements in which the glass and a metal have been fused (cermets) or mixed together and which are intended for the restoration of teeth.

Annex C

(normative)

Determination of film thickness (luting cements only)

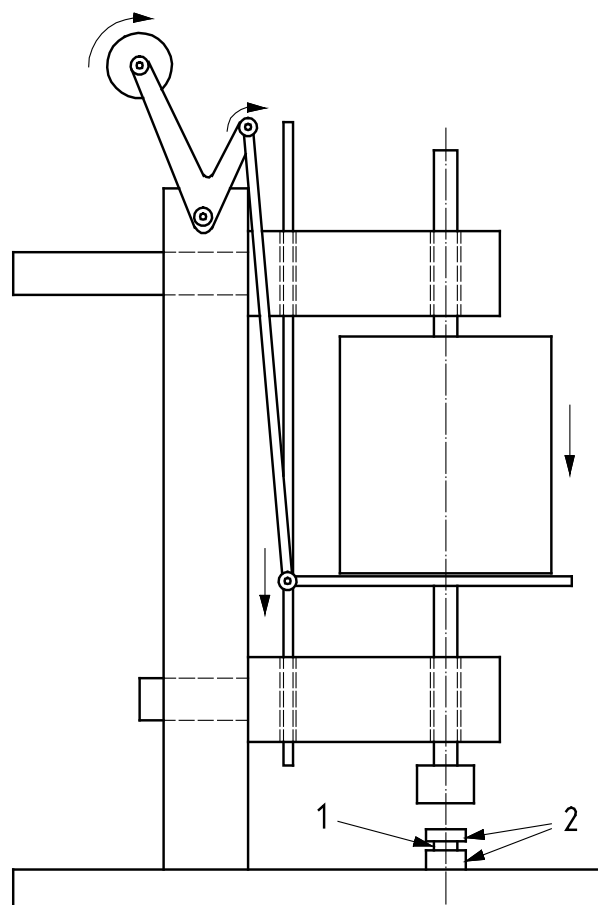
C.1 Apparatus

C.1.1 Two optically flat, square or circular, glass plates, having a contact surface area of $(200 \pm 25) \text{ mm}^2$. Each plate shall be of uniform thickness of not less than 5 mm.

C.1.2 Loading device, of the type illustrated in Figure C.1, or an equivalent means, whereby a force of $(150 \pm 2) \text{ N}$ may be generated vertically to the specimen via the upper glass plate.

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

C.1.3 Screw micrometer or equivalent measuring instrument, having graduations of $2 \mu\text{m}$ or smaller.



Key

- 1 specimen
- 2 glass discs

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