
Dentistry — Water-based cements —
Part 1:
Powder/liquid acid-base cements

Art dentaire — Ciments à base d'eau —

Partie 1: Ciments acido-basiques liquides/en poudre

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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 9917-1 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 9917-1:2003), of which it constitutes a minor revision.

The main purpose for modification of this part of ISO 9917 was to include a new test method for acid erosion, a new test and requirement for radio-opacity and to distinguish between the requirements for manufacturer's instructions for hand-mixed cements and those for capsulated cements.

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

A bibliography has been added to this document.

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

- *Part 1: Powder/liquid acid-base cements*
- *Part 2: Light-activated cements*

Dentistry — Water-based cements —

Part 1: Powder/liquid acid-base cements

1 Scope

This part of ISO 9917 specifies requirements and test methods for powder/liquid acid-base dental cements intended for permanent cementation, lining and restoration. This part of ISO 9917 is applicable to both hand-mixed and capsulated cements for mechanical mixing. This part of ISO 9917 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. This part of ISO 9917 is not intended to address resin-modified water-based cements.

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 — Specification*

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

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

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 start of mixing, during which it is possible to manipulate a dental material without an adverse effect on its properties

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 ISO 9917, 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 ISO 9917, 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 ISO 9917. 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 ISO 9917, 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 coloured, 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 ± 1) °C and a relative humidity of (50 ± 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 colour 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 aluminium 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 ISO 9917, the container or capsule shall be considered to be the immediate wrapping of the material) that afford adequate protection and have no adverse effect on the quality of the contents.

An outer pack may also be used to present the containers or capsules as a single unit.

9.2 Marking

Each outer 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 millilitres of the liquid as appropriate;
- d) the manufacturer's batch or lot number;

- e) on the outermost packaging, the recommended conditions of storage, and the “expiry date” for the material under those conditions of storage;
- f) on the outer packaging, whether the cement is designated opaque;
- g) on the outer packaging, whether the cement is designated as radio-opaque;
- h) on each outer 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 ± 1) °C and a relative humidity of (50 ± 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 millilitres, 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	Net setting time		Compressive strength	Acid erosion	Opacity		Acid-soluble As content	Acid-soluble Pb content
			min.	max.			$C_{0,70}$			
		µm max.	min.	max.	MPa min.	mm max.	min.	max.	mg/kg max.	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 $(1,0 \pm 0,1)$ mm which is plane and perpendicular to the long axis of the needle.

A.1.3 Metal mould, similar to that shown in Figure A.1.

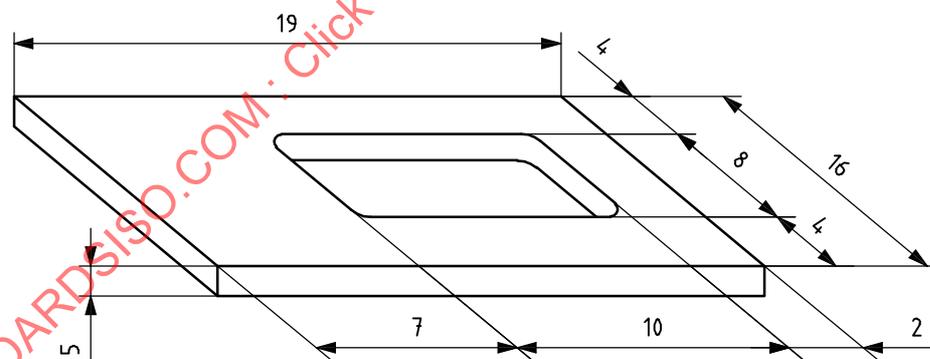
A.1.4 Metal block, of minimum dimensions $8\text{ mm} \times 75\text{ mm} \times 100\text{ mm}$ positioned within the cabinet and maintained at $(37 \pm 1)^\circ\text{C}$.

A.1.5 Aluminium foil.

A.1.6 Timer, accurate to 1 s.

Dimensions in millimetres

Tolerance on dimensions $\pm 0,15$



NOTE Internal corners may be square or rounded.

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

A.2 Procedure

Place the mould (A.1.3), conditioned to (23 ± 1) °C, on the aluminium foil (A.1.5) and fill 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 cabinet (A.1.1). Ensure good contact between the mould 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.

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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)

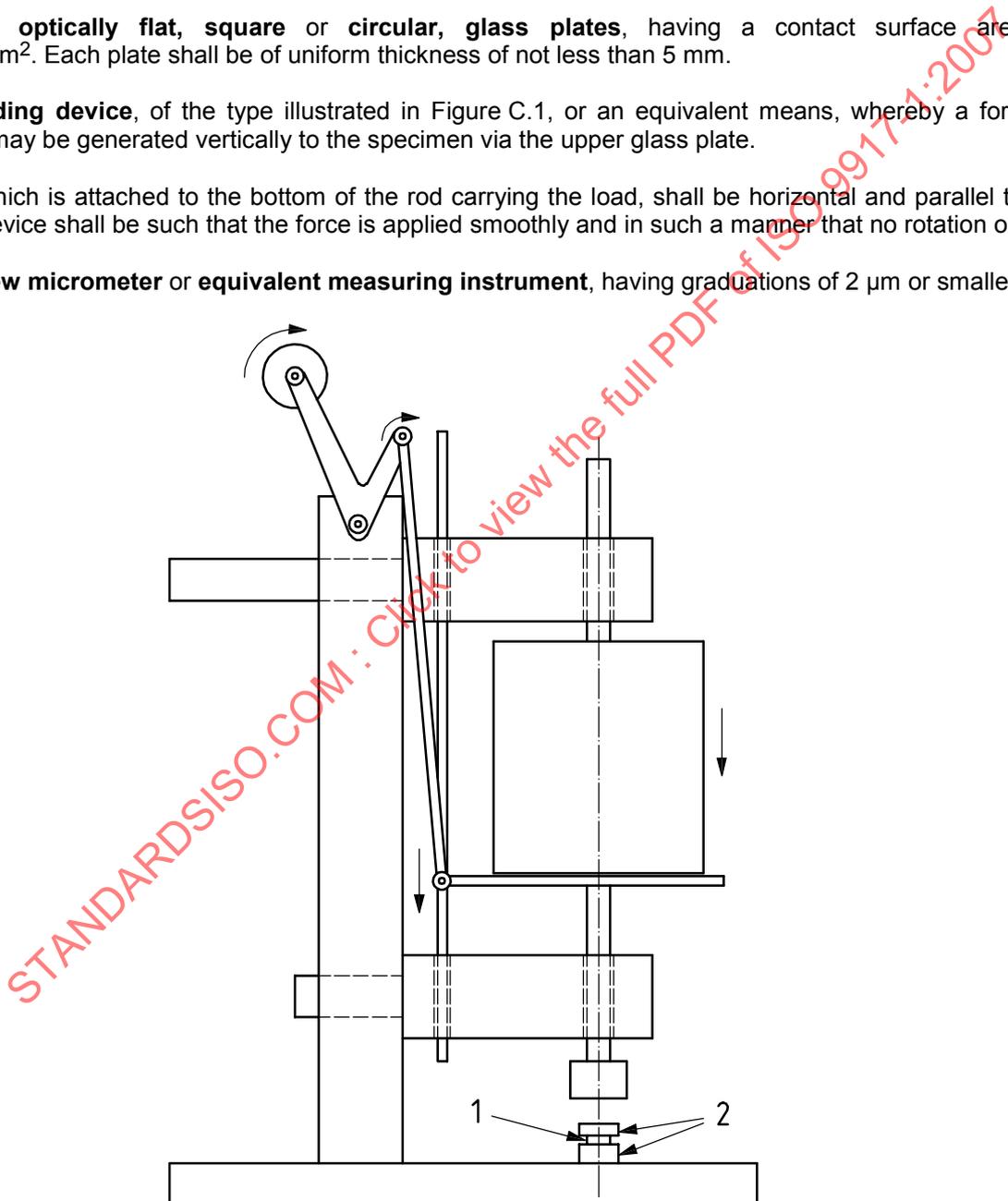
C.1 Apparatus

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 $2 \text{ }\mu\text{m}$ or smaller.



- Key**
- 1 specimen
 - 2 glass discs

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

C.2 Procedure

Measure and record to an accuracy of 1 µm the combined thickness of the two optically flat glass plates (C.1.1) stacked in contact and designate this measurement reading A. Remove the upper plate and place (0,10 ± 0,05) ml of the mixed cement in the centre of the lower plate and place this centrally below the loading device (C.1.2) on the lower platen. Replace 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, carefully 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 force, remove the plates from the loading device and measure the combined thickness of the two glass plates and the cement film and 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.

C.3 Treatment of results

At least four of the five results shall be below 25 µm for the material to pass the requirement specified in 8.2 and Table 1. If only two or less results are below 25 µm, then the material shall fail the requirement. If three results are below 25 µm, a further five specimens shall be tested. To comply with the requirement specified in 8.2 and Table 1, all the specimens in the second series shall be below 25 µm.

Annex D
(normative)

Determination of compressive strength

D.1 Apparatus

D.1.1 Cabinet, maintained at a temperature of (37 ± 1) °C and a relative humidity of at least 30 %.

D.1.2 Split mould and plates, as shown in Figure D.1. The mould shall have internal dimensions $(6,0 \pm 0,1)$ mm high and $(4,0 \pm 0,1)$ mm diameter. The mould and plates shall be made of stainless steel or a material which will not be affected by the cement. When testing polyacid-based cements, face the plates with acetate sheets to prevent adhesion.

D.1.3 Screw clamps, as shown in Figure D.1.

D.1.4 Screw micrometer or equivalent measuring instrument, having graduations of 2 µm or smaller.

D.1.5 Mechanical tester, capable of being operated at a cross-head speed of $(0,75 \pm 0,30)$ mm/min or at a loading rate of (50 ± 16) N/min.

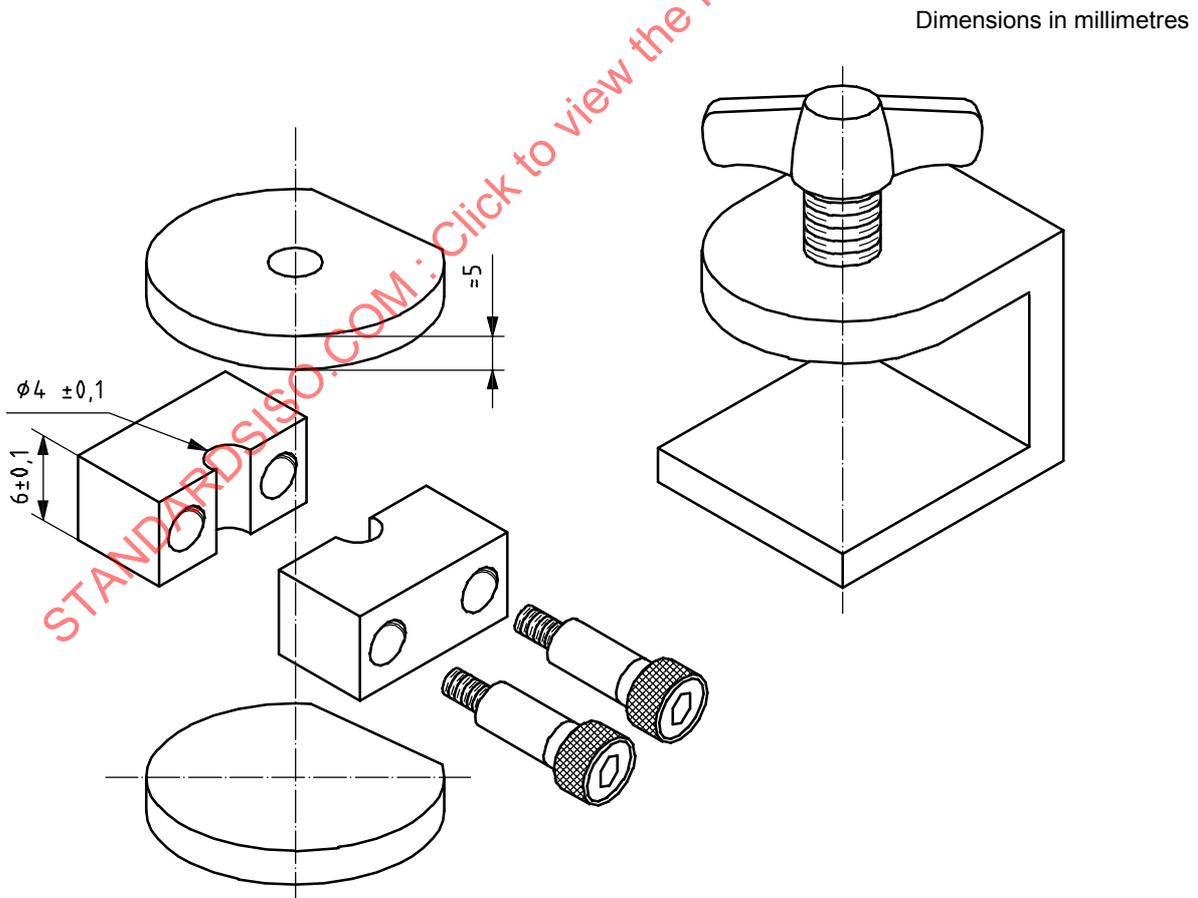


Figure D.1 — Mould and clamp for preparation of specimens for compressive strength test

D.2 Preparation of test specimens

Condition the split mould and plates (D.1.2) and screw clamp (D.1.3) at (23 ± 1) °C. Within 60 s of the end of mixing, pack the cement, prepared in accordance with the manufacturer's instructions, to a slight excess in the split mould.

In order to consolidate the cement and avoid trapping air, convey the largest convenient portions of mixed cement to the mould and apply to one side using a suitable instrument. Fill the mould to excess in this manner and then place on the bottom plate with some pressure.

Remove any bulk extruded cement, place the top metal plate in position on the mould and squeeze together. Put the mould and plates in the screw clamp and tighten. No later than 120 s after the end of mixing, transfer the whole assembly to the cabinet (D.1.1).

One hour after the end of mixing, remove the plates and grind the ends of the specimen flat and at right angles to its long axis. An acceptable method for doing this is to use wet 400 grade silicon carbide paper, but in any event the abrasive shall be no coarser.

Remove the specimen from the mould immediately after surfacing and check visually, without magnification, for air-voids or chipped edges. Discard any such defective specimens.

NOTE To facilitate the removal of the hardened cement specimen, the internal surface of the mould may be evenly coated, prior to filling, with a 3 % solution of micro-crystalline or paraffin wax in petroleum ether. Excess ether is allowed to evaporate before the mould can be used. Alternatively, a thin film of silicone grease or PTFE dry-film lubricant may be used.

Prepare five such specimens and, immediately after the preparation of each, immerse it in water, grade 3 as defined in ISO 3696:1987, at (37 ± 1) °C for $(23 \pm 0,5)$ h.

Calculate the diameter of each specimen by taking the mean of two measurements at right angles to each other, made to an accuracy of 0,01 mm using e.g. a screw micrometer (see D.1.4).

D.3 Procedure

Twenty-four hours after the end of mixing, place each specimen with the flat ends between the platens of the mechanical tester (D.1.5) and apply a compressive load along the long axis of the specimen. Apply a sheet of damp filter paper (e.g. Whatman No. 1) to both top and bottom platens of the test machine in the area which will contact the specimens. Use a fresh piece of paper for each test.

Record the maximum force applied when the specimen fractures and calculate the compressive strength, C , in megapascals, using the equation:

$$C = 4p/\pi d^2$$

where

p is the maximum force applied, in newtons;

d is the measured diameter of the specimen, in millimetres.

D.4 Treatment of results

If at least four of the five results are above the minimum strength specified in 8.3 and Table 1, the material shall pass the test. If three or more of the five results obtained are below the minimum strength specified in Table 1, the material shall fail the test.

If only three specimens satisfy the minimum strength requirement in Table 1, prepare and test a further five specimens. To pass the test, at least eight of the total of ten results shall be above the minimum strength value specified in Table 1.

Annex E (normative)

Determination of acid erosion

E.1 Apparatus

E.1.1 Cabinet, capable of being maintained at a temperature of (37 ± 1) °C and a relative humidity of at least 90 %.

E.1.2 Specimen holders, comprising squares or discs of cast poly(methyl methacrylate) PMMA, (30 mm × 30 mm × 5 mm squares or 5 mm × 30 mm diameter discs), with a hole $(5,0 \pm 0,5)$ mm in diameter and $(2,0 \pm 0,5)$ mm deep bored in the centre.

NOTE The size and shape of the specimen holder can be slightly changed (e.g. corners of squares rounded) if this helps it to fit comfortably into the container.

E.1.3 PMMA plate.

NOTE The size of the PMMA plate is not important but is approximately the same as that of the specimen holder.

E.1.4 Separating sheet, such as a polyester strip, for application to the specimen holder to facilitate removal of the PMMA plate after the cement has set.

E.1.5 Individual clamps, clips or equivalent device, designed to hold the specimen holders (E.1.2), plates (E.1.3) and separating sheet (E.1.4) together during setting of the cement.

E.1.6 Balance, with an accuracy of 0,1 mg.

E.1.7 Volume-measuring device such as a pipette, suitable for 30 ml aliquots or any alternative device with an accuracy of 0,1 ml.

E.1.8 Container, capable of carrying 30 ml of test solution in which the specimen holder can be completely immersed horizontally and in which the distance between the surface of the eroding solution and the upper surface of the immersed specimen holder is (10 ± 3) mm. The container shall have a lid or screw cap to prevent evaporation or contamination.

E.1.9 Cabinet, maintained at a temperature (37 ± 1) °C.

NOTE The cabinet (E 1.1) may be used but 90 % humidity is not required here.

E.1.10 Abrasive paper, 1 200 grit wet or dry abrasive paper.

E.1.11 Spring-loaded, spherically tipped, dial gauge, having a scale graduated in units of 0,01 mm or less (to enable 0,005 mm to be measured). The spring force of the gauge shall be in the range 50 g to 100 g and the diameter of the tip shall be $(3,5 \pm 0,5)$ mm.

NOTE It is acceptable to use an equivalent loading device with a "deadweight". A device with an electronic reading/display facility is also acceptable providing the loading conditions are as specified.

E.2 Preparation of eroding solution

Dissolve 8,27 g of lactic acid (general purpose reagent grade or purer) and 0,92 g sodium lactate (general purpose reagent grade or purer) both calculated as pure, dry substance, in water (grade 3 as defined in ISO 3696:1987) and adjust to 1 l with grade 3 water at least 18 h before use (this allows hydrolysis of lactone to take place). Immediately prior to use, check that the pH of this solution is $2,74 \pm 0,02$ and adjust if necessary with 1 mol/l sodium lactate solution or 1 mol/l lactic acid solution. This reagent shall always be prepared freshly for each set of specimens being tested.

NOTE This solution is a 0,1 mol/l of lactic acid/sodium lactate buffer solution. The pH is 2,74.

E.3 Preparation of test specimens

Condition the specimen holders (E.1.2), PMMA plate (E.1.3), separating sheet (E.1.4) and clamp (E.1.5) at $(23 \pm 1) ^\circ\text{C}$. Proportion and mix the test material in accordance with the manufacturer's instructions at $(23 \pm 1) ^\circ\text{C}$. For hand-mixed cements, weigh powder and liquid at the recommended powder/liquid ratio by using the balance (E.1.6). Place the components on the mixing pad/slab recommended by the manufacturer. Mix, using the technique recommended by the manufacturer. For encapsulated cements, mix by using the recommended mixing machine for the recommended time. Within 60 s of the end of mixing of the test material, fill the hole of the specimen holder with the cement following the manufacturer's instructions for handling.

In order to avoid trapping air, use the largest convenient portions of mixed cement to fill one side of the hole of the specimen holder then fill the remainder of the specimen holder, continuing to avoid air entrapment. Fill the specimen holder to a slight excess in this manner.

Cover the cement with the PMMA plate, faced with the separating sheet, press firmly together and apply the screw clamp.

One hundred and eighty seconds after the end of mixing, transfer the whole assembly to the cabinet (E.1.1) maintained at $(37 \pm 2) ^\circ\text{C}$ and a relative humidity of at least 90 %.

After 24 h, remove the plate and the separating sheet from the clamp and lap the cement in the specimen holder until flat using the abrasive paper (E.1.10) with continuous water irrigation.

Prepare five specimens. Inspect all specimens and reject any with obvious flaws or air inclusions.

E.4 Measurement and calculation of initial depth at centre of cement, D_0

For each specimen, measure the initial depth at the centre of the specimen using the edge of the specimen holder as a fixed reference plane.

NOTE 1 This procedure is necessary to establish the initial cement height and to confirm that the height of the cement is approximately the same as the height of the specimen holder.

If the original cement height is not within 5 μm of that of the specimen holder, carry out further lapping until the levels of the cement and holder are within 5 μm .

NOTE 2 Typically, one unit of the dial gauge is 10 μm and 5 μm can be estimated as a half of one unit.

Record the height at four points on the specimen holder and near the centre of the material as a baseline depth using the dial gauge (E.1.11). The four points on the specimen holder shall be spaced evenly at 90° intervals at 0,5 mm to 1,0 mm from the specimen.

Calculate the average of the four heights of the specimen holder. Subtract the height at the centre of the specimen from this value to obtain D_0 .

E.5 Immersion

Immerse each specimen, with its specimen holder horizontally, in an individual container (E.1.8) containing 30 ml of the eroding solution. Place the specimen holder "face upwards" so that the whole of the cement surface is bathed in solution with a (10 ± 3) mm head of solution above the surface of the test specimen.

Seal the container and store for 24 h in the cabinet (E.1.9) at (37 ± 1) °C.

After a 24 h immersion, remove and rinse the specimen and the specimen holder with water (grade 3 as defined in ISO 3696:1987).

E.6 Measurement and calculation of the depth, D_t , at the centre of cement after erosion

For each specimen, measure the depth at the centre of the specimen using the shoulders of the specimen holder as fixed reference planes as before (see Clause E.4) to obtain the value of the depth of cement after erosion, D_t .

E.7 Expression of results

Express the eroded depth, D , in millimetres, at the centre of the cement for each specimen, using the following equation:

$$D = D_t - D_0$$

where

D_0 is the depth at the centre of the specimen before immersion, expressed in millimetres;

D_t is the depth at the centre of the specimen after immersion, expressed in millimetres.

NOTE D_0 is negative if the original specimen height was above the specimen holder and in this case D is obtained by adding the absolute values of D_t and D_0 .

Obtain a value of D for each of the five test specimens.

E.8 Treatment of results

Compare the calculated values of D with the limit values given in Table 1. To pass the test, four or more of the D values shall be below the limit value. If three or more values of D are above the limit value, the material shall fail the test.

If only three values out of five are below the limit value, prepare and test a further five specimens. To comply with the requirement in 8.4 all five values shall be below the limit value in Table 1.

Annex F (normative)

Determination of optical properties (polyalkenoate restorative cements only)

F.1 Opacity

F.1.1 Apparatus

F.1.1.1 Cabinet, maintained at a temperature of $(37 \pm 1) ^\circ\text{C}$ and a relative humidity of at least 30 %.

F.1.1.2 Opal glass standards, with $C_{0,70}$ values of 0,35 and 0,55 and 0,90.

NOTE The contrast ratio $C_{0,70}$ is the ratio between the light reflected by the specimen on a black background and the light reflected by the specimen on a white background which has a diffuse reflectance of 70 %.

F.1.1.3 Sheets of white waterproof material of polyethylene or cellulose acetate, measuring approximately 110 mm \times 40 mm, marked, along their entire length with black stripes, 2 mm wide and 3 mm apart.

F.1.1.4 Mould, consisting of a split brass or stainless-steel ring contained in a former and cover plates as shown in Figure F.1. The height of the ring shall be $(1,00 \pm 0,03)$ mm and the internal diameter $(10,0 \pm 0,3)$ mm.

F.1.1.5 Individual clamps, clips or equivalent device, designed to hold the specimen mould together during setting of the cement.

F.1.1.6 Screw micrometer or equivalent device, accurate to 0,01 mm.

F.1.1.7 Timer, accurate to 1 s.

F.1.2 Preparation of test specimen

Place the mould on the sheet (F.1.1.3) backed by a flat metal cover plate. Fill the split ring mould (F.1.1.4) with cement prepared in accordance with the manufacturer's instructions. Cover with a second plate faced with a sheet; press firmly together and apply a clamp (F.1.1.5).

One hundred and twenty seconds after the end of mixing, place the mould plates and the screw clamp in the humidity cabinet (F.1.1.1).

After 1 h, remove the plates and sheets from the clamp and carefully separate the specimen from the ring. Measure the thickness of the specimens using the micrometer (F.1.1.6) and use only specimens $(1,0 \pm 0,1)$ mm thick. Store the specimen for seven days at $(37 \pm 1) ^\circ\text{C}$ in water of grade 3 in accordance with ISO 3696:1987.

F.1.3 Procedure

Compare the opacity of the specimen with the two appropriate opal glass standards (F.1.1.2) by placing the specimen and the standards on the black and white striped background. Cover the specimen, the opal glass standards and the sheets with a thin film of distilled water while making the comparison.

Alternatively, use a photometric instrument to make this comparison, provided that it can be demonstrated that the instrument has an accuracy of within $\pm 0,02 C_{0,70}$. In this procedure, place the specimen on an illuminated white background (reflectance 70 %) and measure the reflectance, $R_{0,70}$. Then transfer it to a black background and illuminate it with the same source and measure the reflectance, R_B . Calculate the opacity from the equation:

$$C_{0,70} = R_B/R_{0,70}$$

F.1.4 Treatment of results

If the opacity of the cement specimen is between those of the two standards or equal to either of them, it passes the test and meets the requirement of 8.5 a).

F.2 Colour

F.2.1 Apparatus

F.2.1.1 Sheet of white bond paper, giving a diffuse white background with a diffuse reflectance of approximately 90 %.

F.2.2 Preparation of test specimen

Prepare the test specimen as described in F.1.2 and store it at $(37 \pm 1)^\circ\text{C}$ for seven days in water of grade 3 as defined in ISO 3696:1987.

F.2.3 Procedure

Remove the specimen from the water. Remove the excess water from the cement surface using filter paper and place the specimen on the diffuse white background. Compare the colour of the specimen with the manufacturer's shade guide or nominated shade guide using the procedure for colour comparison specified in ISO 7491.

F.2.4 Treatment of results

In order to satisfy the requirement of 8.5 b), the colour of the specimen shall correspond with the manufacturer's description.