

International Standard 3824

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Dental silicophosphate cement (hand-mixed)

Ciments dentaires aux silicophosphates (mélange manuel)

Second edition — 1984-06-01

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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 developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3824 was developed by Technical Committee ISO/TC 106, *Dentistry*, and was circulated to the member bodies in April 1982.

It has been approved by the member bodies of the following countries :

Australia	India	Poland
Belgium	Ireland	South Africa, Rep. of
Canada	Japan	Sweden
Czechoslovakia	Mexico	Switzerland
Egypt, Arab Rep. of	Netherlands	United Kingdom
France	New Zealand	USA
Germany, F.R.	Norway	USSR

No member body expressed disapproval of the document.

This second edition cancels and replaces the first edition (i.e. ISO 3824-1977).

Dental silicophosphate cement (hand-mixed)

0 Introduction

This revision of ISO 3824 has been based on ISO 1565 and ISO 1566, both of which cover materials whose characteristics are closely related to silicophosphate cements.

Most of the changes from the previous edition stem from the use of a much smaller test specimen than before. The reason for using the small specimen is to align the test methods for hand-mixed materials as closely as possible with those for cap-sulated materials. In general, this has merely necessitated some adjustments in technique to accommodate the smaller test specimen, but in some instances, the water-leachable material test in particular, more basic changes in the test method have been made.

The scope of this specification covers silicophosphate material whether formulated for use either as a filling material or for luting purposes. However, in the text it has been found more convenient, as well as in accordance with common usage, to make no distinction between the description applied to the different types of materials; the term "cement" has therefore been used throughout.

Toxicity requirements are not covered in this International Standard but it is recommended that is assessing possible biological or toxicological hazards, reference should be made to ISO/TR 7405.

A number of editorial improvements have also been introduced to clarify the wording and presentation of this revision. In the light of a continuing review of technological advances and well-documented technical data, future revisions of this International Standard will be considered as and when they appear to be appropriate.

1 Scope

This International Standard specifies the requirements for hand-mixed dental cement, based on the hardening reaction between a powder made from ground, acid soluble, aluminosilicate glass and metal oxides principally zinc oxide, with an aqueous solution of phosphoric acid which may contain metal ions.

2 Field of application

The cements covered by this International Standard are those used as luting agents to seal dental appliances to hard oral structures or to other appliances.

These cements can also be used as restorative materials by increasing the ratio of powder to liquid relative to that used for luting.

3 References

ISO 1565, *Dental silicate cement (hand-mixed)*.

ISO 1566, *Dental zinc phosphate cement*.

ISO 2014, *Writing of calendar dates in all-numeric form*.

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

ISO/TR 7405, *Biological evaluation of dental materials*.

4 Classification

Silicophosphate cement covered by this International Standard shall be of the following types :

- **Type I** — Luting agent
- **Type II** — Restorative material

5 Requirements

5.1 Material

The cement shall consist of a powder and a liquid which, when mixed according to the manufacturer's instructions, will set to a cement which will comply with the requirements of this International Standard.

5.2 Components

5.2.1 Liquid

The liquid shall be free from obvious deposits or filaments on the inside of its container.

5.2.2 Powder

The powder shall be free of extraneous material and, if coloured, the pigment shall be uniformly dispersed throughout the powder.

5.3 Unset cement

The cement, when mixed as directed in 7.1.4, shall be of uniform smooth consistency, and shall not evolve gases.

5.4 Set cement

After immersion in water for 5 days, the colour of any shade of the set cement, when viewed under water utilising natural daylight, shall match the manufacturers shade guide if supplied.

5.5 Performance requirements

The mixed cement, depending on its type, shall comply with the requirements specified in the table when tested in accordance with the relevant method described in clause 7.

The powder/liquid ratio which gives the required mix consistency, called the "standard testing consistency", shall be used in the preparation of all test specimens for tests carried out in accordance with this International Standard.

5.6 Acid soluble arsenic and lead

The arsenic and lead contents, when tested in accordance with the methods prescribed in 7.8, shall be not more than the limits specified in the table.

5.7 Inspection requirements

Visual inspection shall be used in determining compliance with 5.2.1, 5.2.2, 5.4 and clause 8.

5.8 Manufacturer's instructions

Full instructions for the preparation, mixing and manipulation shall accompany each container or package and shall include at least the following details :

- a) the recommended temperature, condition and type of the slab and spatula;
- b) the powder/liquid ratio at the recommended temperature;
- c) the rate of incorporating the powder;
- d) the time of mixing;

e) the maximum manipulation time after completion of mixing at the recommended temperature;

f) a statement recommending that, when clinical conditions warrant, a liner should be placed between the cement and the dentine.

6 Sampling and samples

A sample drawn from one batch shall provide sufficient powder and liquid to complete all the prescribed tests.

7 Test methods

7.1 Preparation of test specimens

7.1.1 Ambient conditions

Unless stated otherwise, the preparation of all specimens shall be carried out at 23 ± 1 °C and at a relative humidity of 50 ± 5 %.

7.1.2 Components

All tests shall be carried out on specimens prepared from samples of the powder and liquid complying with 5.2.1 and 5.2.2.

7.1.3 Apparatus for mixing

All apparatus used for mixing and for testing shall be kept clean, dry and free from hardened particles of cement.

7.1.3.1 Polished glass slab, approximately 150 mm long $\times$ 75 mm wide $\times$ 20 mm thick.

7.1.3.2 Spatula, inert to the cement.

7.1.4 Method of mixing

Place known amounts of powder and liquid on the polished glass slab (7.1.3.1) and divide the powder into approximately two halves and then divide one half into two equal portions.

Commence mixing the components by incorporating the half portion of the powder into the liquid in the first 15 s. Follow this by incorporating the quarter portions each at intervals of approximately 15 s, making sure that each portion is thoroughly mixed before introducing the next portion. Spatulate the whole mass with reasonable pressure for a further 15 s using a small area of the slab.

The total mixing time shall be 1 min. No particles of powder and no unused liquid shall remain on the slab when the mix is completed. The final mix shall appear to be homogeneous.

7.2 Determination of powder/liquid ratio for obtaining standard testing consistency

7.2.1 Apparatus

7.2.1.1 The apparatus used for determining consistency shall be essentially that illustrated in figure 1.

For Type 1 a mass of 2,5 kg shall be used to exert a force of 24,5 N.

For Type 2 a mass of 15 kg shall be used to exert a force of 147 N.

The platen, which is attached to the bottom of the rod carrying the load, shall be horizontal and parallel to the base and shall incorporate a device for holding the larger glass plate (see 7.2.1.2) in contact with its surface. The second and smaller glass plate shall be held on the base by guides to prevent movement or rotation when the load is applied. The load shall be capable of being applied smoothly and in such a manner that no rotational motion occurs. The two glass plates shall be capable of touching over their entire facing surfaces, without interference from guides, etc.

7.2.1.2 Two flat square glass plates with sides approximately 50 mm square and 40 mm square respectively and approximately 5 mm thick, fitting the loading device (specified in 7.2.1.1).

7.2.1.3 Measuring device to deliver 0,075 ml of mixed cement in the form of a cylinder 6,0 mm high and 4,0 mm in diameter. A suitable device consists of a glass tube and a PTFE plunger.

7.2.1.4 Graduated syringe pipette having an accuracy of $\pm 0,001$ ml.

7.2.2 Procedure

Using the manufacturer's recommended powder/liquid ratio as a guide, carefully weigh out a trial amount of powder 300 to 450 mg to an accuracy of 1 mg and transfer it to the glass mixing slab. Deliver 0,100 ml of liquid from the syringe pipette (7.2.1.4) close to the powder.

After mixing in accordance with 7.1.4, collect and load the cement into the measuring device. Deliver 0,075 ml of the mixed cement, as an upright cylinder, onto the centre of the glass plate (7.2.1.2) which will rest on the baseplate of the loading device. If it is not possible to deliver all of the cement from the measuring device in a single operation, take the residue with the tip of a clean spatula and place on the centre of the other glass plate (7.2.1.2). Both glass plates in the loading device will thus be positioned in such a way that any cement on the second glass plate will contact centrally the bulk of the cement on the first glass plate, when the force is applied.

Sixty seconds after the completion of mixing, gently press out the cement between the two glass plates with the force specified in 7.2.1.1, applied in a direction perpendicular to the lower glass plate.

After the cement has set, measure the major and minor diameters of the cement disc with an accuracy of 0,5 mm and calculate the mean. If the two measurements differ by more than 1 mm discard the result and repeat the test.

Make trial mixes of varying powder/liquid ratios until the mean diameter, calculated from the major and minor diameters measured, is in accordance with the value specified in the table.

7.3 Film thickness for type I cements (luting agents) only

7.3.1 Apparatus

7.3.1.1 Two optically flat square glass plates, having a contact surface area of approximately 200 mm². Each plate shall be of a uniform thickness not less than 5 mm.

7.3.1.2 Loading device of the type illustrated in figure 2, generating a force of 147 N (load of 15 kg mass). The bottom surface of the rod carrying the load shall be horizontal and parallel to the base and large enough to cover one of the glass plates. The load shall be capable of smooth application in such a manner that no rotational motion occurs. The glass plates shall be held on the base by guides to prevent movement or rotation when the load is applied.

7.3.1.3 Micrometer or similar measuring instrument, accurate to 1 μ m.

7.3.2 Procedure

Measure accurately the thickness of the two optically flat glass plates (7.3.1.1) stacked in contact (reading *A*). Place a small quantity of cement, mixed to the standard testing consistency, on the centre of one of the glass plates and place the plate in the guides. Place the second glass plate centrally on the cement.

Three minutes after commencing the mix, carefully apply a force of 147 N (15 kgf) vertically on the top plate and leave for 7 min. Ensure that the cement completely fills the area between the two glass plates.

Ten minutes after the commencement of mixing, measure the thickness of the two glass plates and cement film (reading *B*).

The difference in thickness of the plates with and without the cement film (reading *B* — reading *A*) shall be taken as the thickness of the film. Report the mean result of three such tests to the nearest 5 μ m.

7.4 Net setting time

The setting time determined by this test method is that from the completion of mixing, and not the more usual total setting time, where the time is measured from first contact between the cement components.