INTERNATIONAL STANDARD ISO 3824

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Dental silicophosphate cement (hand-mixed)

Ciments dentaires aux silicophosphates (mélangés à la main)

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FOREWORD

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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 drawn up by Technical Committee ISO/TC 106, *Dentistry*, and was circulated to the member bodies in July 1975.

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

Australia	Iran	Sweden
Brazil	Mexico	Switzerland
Canada	Netherlands	Turkey
France	New Zealand	United Kingdom
Germany	Romania	U.S.A.
India	South Africa, Rep. of	

No member body expressed disapproval of the document.

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Dental silicophosphate cement (hand-mixed)

0 INTRODUCTION

The contents of this International Standard have been largely based on the two Recommendations ISO/R 1565, *Dental silicate cement*, and ISO/R 1566, *Dental zinc phosphate cement*, both of which cover materials whose characteristics are closely related to silicophosphate cements. These two related specifications were prepared from, and are technically identical with, specifications 5 and 6 of the Fédération Dentaire Internationale. This organization served as Secretariat of the Working Group which prepared this International Standard.

If, as a result of the current studies being undertaken to provide for revision of ISO/R 1565 and ISO/R 1566, there appear to be grounds for a reassessment of the contents of this specification, then further action will be taken, as and when appropriate, to revise it.

The scope of this specification covers hand-mixed silicophosphate material used both as filling and for cementation. 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.

1 SCOPE AND FIELD OF APPLICATION

This International Standard gives the classification of, and requirements for, dental silicophosphate cement, together with test methods to be used to determine the compliance with these requirements.

Dental silicophosphate cement covered by this International Standard is that made by hand-mixing a powder made from ground, acid-soluble, aluminosilicate glass and metal oxides with a liquid solution of orthophosphoric acid and metal phosphate.

2 CLASSIFICATION

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

- **Type I** – Cementing medium
- **Type II** – Restorative material
- **Type III** – Dual purpose

3 REQUIREMENTS

3.1 Material

The cement shall consist of a powder and a liquid which, when mixed according to the manufacturer's instructions, will set to a condition satisfactory for the indicated dental use.

3.2 Toxicity

The mixed materials, when used according to the manufacturer's instructions, shall not cause prolonged damage to oral tissues nor have any adverse systemic effect.[1)]

3.3 Liquid

The liquid shall be water-clear and free from suspended matter, deposits and sediments when viewed under 5 X magnification.

3.4 Powder

The powder shall be free of extraneous material when viewed under 5 X magnification. The pigment shall be uniformly dispersed throughout the powder.

3.5 Unset cement

The cement, when spatulated in accordance with the manufacturer's instructions, shall be free of gritty particles, shall not form lumps or granules and shall not evolve gas.

3.6 Set cement

3.6.1 *Colour*

The colour of a set cement specimen of any shade shall match the manufacturer's shade guide, if provided or cited, within the limits of professional acceptance. The cement specimen shall be prepared using a shade selected at random from a test sample, and shall be viewed under water after immersion for at least 5 days.

1) When specific toxicity tests are available, it is envisaged that these will be included in the present International Standard.

3.6.2 *Arsenic content*

The water-soluble arsenic content shall not be more than 2 mg/kg (2 ppm) when tested in accordance with 6.8.

3.7 Physical properties

The time of setting, compressive strength, film thickness, solubility and disintegration and opacity for the various types of cement shall be as listed in table 1, when tested in accordance with the relevant method described in clause 6.

3.8 Manufacturer's instructions

Adequate instructions for the preparation, mixing and manipulation shall accompany each container of liquid and shall include at least the following details :

a) the recommended temperature, condition and type of mixing surface;

b) the powder/liquid ratio at the recommended temperature;

c) the rate of incorporating the powder;

d) the maximum mixing time;

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

f) a recommendation to use a varnish layer, base or similar protective barrier between the cement and dentine in vital teeth.

4 SAMPLING

The method of procurement and the amount of cement needed shall be the subject of agreement between the interested parties and shall be recorded.

5 PREPARATION OF TEST SPECIMENS

5.1 Ambient temperature and humidity

The preparation of all test specimens shall be conducted at 23 ± 1 °C and at a relative humidity between 45 % and 70 %.

5.2 Mixing

Use the following mixing technique in the preparation of all test specimens :

5.2.1 *Apparatus*

a) **Polished glass slab** approximately 150 mm long by 75 mm wide.

b) **Spatula** made from a material not corroded or abraded by the cement.

5.2.2 *Procedure*

Make certain that the slab and spatula are clean and free from particles of hardened cement. Use approximately one-third of the top surface of the slab as the mixing area. Use a linear and not a rotary movement, with the edge of

TABLE 1 — Requirements for testing consistency and physical and chemical properties

Type		Consistency of mix	Time of setting at 37 °C		Compressive strength at 24 h	Film thickness	Solubility and disintegration	Opacity at 24 h		Arsenic content
		Diameter of disk	min.	max.	min.	max.	max.	min.	max.	max.
		mm	minutes	minutes	MN/m²*	μm	mass %	$C_{0,70}$ value		mg/kg (ppm)
I		25 ± 1	5	9	135	25	0,9	0,35	0,90	2
II		25 ± 1	3	6	165	—	0,6	—	—	
III**	α	25 ± 1	5	9	135	25	0,9	0,35	0,90	
	β	25 ± 1	3	6	165	—	0,6	—	—	

* 1 MN/m² ≈ 10,2 kgf/cm²

** α — Tested as a cement.

β — Tested as a restorative material.

the spatula sweeping approximately half of the mixing area of the slab on each stroke. Incorporate the powder into the liquid at the rate shown in table 2.

TABLE 2 — **Rate of incorporation of powder**

Proportion of total amount of powder	Time of incorporation s
1/2	15
1/4	15
1/4	15

Spatulate the whole mass for a further 15 s, making a total mixing time of 1 min. No particle of powder or any unused liquid shall remain on the slab when mixing has been completed.

5.3 Powder/liquid ratio

The power/liquid ratio to be used in preparation of the test specimens shall be determined as follows :

5.3.1 *Apparatus*

The apparatus used for determining consistency shall be essentially that illustrated in figure 1. This comprises the following items :

a) **Loading device** with a load which can be positioned to bear on the top of two flat glass plates. The combined mass of the load and top glass plate is adjusted according to the cement type being tested, as follows :

— Type I and Type III (used as a cement) : 0,22 ± 0,001 kg;

— Type II and Type III (used as a restorative material) : 2,5 ± 0,01 kg.

b) **Gauge plug** and **glass tube** of inside diameter approximately 10 mm which will deliver 0,50 ml of mixed cement.

5.3.2 *Procedure*

Mix trial amounts of powder with 0,40 ml of liquid and deliver, by means of the glass tube, 0,50 ml of each mix onto the lower glass plate. Two minutes after the commencement of mixing, carefully lower the top glass plate and the appropriate load [see 5.3.1 a)] onto the soft cement. This will produce a circular disk of cement between the two glass plates.

Continue making trials using this procedure until the average of the greater and lesser diameters of the cement disk, 10 min after the commencement of mixing, is 25 ± 1 mm.

5.3.3 *Expression of results*

Take the average mass of powder used in three such determinations, rounded off to the nearest 0,05 g, combined with 0,40 ml of liquid as the standard powder/liquid ratio for preparing test specimens.

6 TEST METHODS

6.1 Ambient temperature and humidity

Unless stated otherwise, the ambient temperature and relative humidity during testing shall be as specified in 5.1.

6.2 Visual inspection

Visual inspection shall be used to determine compliance with the requirements specified in 3.3, 3.4, 3.5, 3.6.1, 3.8 and clause 7.

6.3 Time of setting

6.3.1 *Apparatus*

An apparatus of the type illustrated in figure 2 is suitable if it satisfies the conditions required for testing.

6.3.2 *Procedure*

Place a brass ring, maintained at room temperature, approximately 5 mm high, 11 mm outside diameter and 9,5 mm internal diameter, on a flat glass plate and fill with cement of standard consistency (see 5.3). Two minutes after commencement of mixing, transfer the specimen to an atmosphere of 95 % to 100 % relative humidity at 37 ± 1 °C. Two and a half minutes after the commencement of mixing, carefully lower a standard Gillmore needle, of mass 400 g and having an end 1,0 mm in diameter, vertically onto the horizontal surface of the cement. Repeat this at 30 s intervals.

6.3.3 *Expression of results*

Record the time of setting as the number of minutes elapsed from the commencement of mixing to the time when the needle fails to make a perceptible circle on the surface of the specimen when allowed to rest thereon under its own weight. Report this time to the nearest 1/2 min (i.e. the minute or half-minute).

6.4 Compressive strength

6.4.1 *Apparatus* (see figure 3)

a) **Cylindrical mould,** 12 mm high and 6 mm internal diameter, of hard rubber, glass, stainless steel or any other substance which will not react with the cement.

Alternatively, a split mould of corresponding internal dimensions may be used.

b) **Two flat glass plates,** or other appropriate plates for use with a split mould.

c) **C-clamp,** or other appropriate clamp for use with a split mould.

d) **Screw jack.**

e) **Compressive strength testing instrument.**

6.4.2 *Preparation of test specimens*

Place the cylindrical mould on a flat glass plate and slightly overfill with cement of standard consistency within 2 min of commencing the mixing. Press the second flat glass plate on top of the mould. Hold the mould and plates firmly together with the small C-clamp. All apparatus should be at room temperature. The mould may be coated with a 3 % solution of a microcrystalline wax (melting point 86 to 91 °C) in toluene or other suitable mould lubricant to assist subsequent removal of the specimen.

If a split mould is used, follow the same basic procedure, timing, etc., but with appropriate detail changes in assembly and disassembly of the mould.

Three minutes after starting the mix, transfer the mould and clamp to an atmosphere of 95 % to 100 % relative humidity at 37 ± 1 °C.

One hour later, surface the ends of the specimens plane at right angles to the axis, by drawing the moulds containing the specimens back and forth across a glass plate coated with an abrasive such as 45 μm (350 mesh) silicon carbide power and water. Rotate the moulds about one quarter turn every few strokes and keep the test specimens wet during the grinding. The finally prepared test specimens will be cylinders 12 mm in height and 6 mm in diameter with flat, smooth ends parallel to each other and at right angles to the long axis of the cylinder.

After surfacing, remove the specimens from the mould using the screw jack and immerse in distilled water at 37 ± 1 °C until required for testing.

Complete testing 24 h after the start of mixing.

6.4.3 *Procedure*

Insert a specimen prepared according to 6.4.2 between the platens of the testing machine with a small piece of wet blotting paper, approximately 0,5 mm thick, at each end. Load the specimens at a rate of 2 200 N (225 kgf) per minute until the crushing point is reached. Keep the specimens wet during the test.

6.4.4 *Expression of results*

Report the value for compressive strength as the average of the values obtained for three or more specimens from a lot of five and round off to the nearest 1 MN/m² (10 kgf/cm²). If the values for individual specimens fall more than 15 % below the average of the five, discard and report the average of the remaining specimens. If more than two specimens are discarded repeat the test.

6.5 Film thickness

6.5.1 *Apparatus*

a) **Two flat square or round glass plates** of uniform thickness each with surface area (one side) of approximately 200 mm².

b) **Load** of mass 15 ± 0,01 kg.

c) **Instrument for measuring** to the required accuracy.

6.5.2 *Procedure*

Place a portion of cement mixed to standard consistency (see 5.3) between the two glass plates.

Two minutes after the mix is started, apply the load vertically on the top plate. Ten minutes after the mix is started, determine the thickness of the two plates with the cement film between them.

6.5.3 *Expression of results*

Record the difference in the thickness of the plates with and without the cement film as the film thickness. Report the average of three test values to the nearest 5 μm.

6.6 Solubility and disintegration

6.6.1 *Apparatus* (figure 4)

a) **Split stainless steel ring,** inside diameter 20 ± 0,2 mm and height 1,5 ± 0,03 mm.

b) **Two flat plates.**

c) **Four tared pieces of fine platinum wire** or other fine corrosion-resistant wire.

d) **Two tared weighing bottles.**

6.6.2 *Preparation of test specimens*

Place 0,5 ml of mixed cement of standard consistency in the split stainless steel ring, which has previously been placed on a thin polyethylene sheet on one of the flat plates. Use the other flat plate, faced with a thin polyethylene sheet, to press the cement into the ring.

Place the tared piece of fine platinum or corrosion-resistant wire in the unset cement via the slit in the split stainless steel ring, to provide a convenient means of holding the specimens as illustrated in figure 4; 3 min after the mix is started, place the plates and cement for 1 h in an atmosphere having a relative humidity of 95 % to 100 % at 37 ± 1 °C.

In carrying out the test procedure it is desirable to use a control, i.e. a bottle without a test specimen.

6.6.3 *Procedure*

Place two specimens, prepared in accordance with 6.6.2, after 1 h in conditions of controlled temperature and humidity, in each tared weighing bottle and weigh. Take the combined mass of the two specimens and the weighing bottle, less the mass of the weighing bottle and the platinum wire, as the mass of the specimens of cement.

Immediately submerge the specimens by pouring 50 ml of distilled water into the weighing bottle, and then store for 23 h at 37 ± 1 °C.

Remove the specimens from the water. There shall be no evidence of crystal growth on the surface of the specimens. Evaporate the water from the weighing bottle at a temperature just below 100 °C, and then dry the weighing bottle at 150 °C to constant mass.

After cooling to room temperature in a desiccator containing thoroughly dry anhydrous calcium sulphate ($CaSO_4$) or silica gel (freshly dried at 130 °C), weigh the weighing bottle and contents with a precision of 0,2 mg.

Repeat this cycle of heating the weighing bottle to 150 °C, cooling over a desiccant and reweighing, until the loss in mass of each bottle is not more than 0,5 mg in any 24 h period.

6.6.4 *Expression of results*

Record the difference between the final and initial mass of the weighing bottle as the amount of disintegration. The gain in mass, divided by the mass of the specimens and multiplied by 100, gives the percentage of disintegration.

Report the average of values from duplicate tests (two weighing bottles containing two specimens each) to the nearest 0,1 %.

6.7 Opacity

6.7.1 *Apparatus*

a) **Opal glass standards** with $C_{0,70}$ values of 0,35 and 0,90 respectively.

b) **Suitable variegated black and white background.**

c) **Two flat glass plates** at least 32 mm by 32 mm.

d) **Split stainless steel ring,** inside diameter 30 ± 1 mm and height 1,00 ± 0,025 mm.

6.7.2 *Preparation of test specimens*

Press a sufficient amount of mixed cement of standard consistency (see 5.3) between two flat glass plates to form a disk of approximately 30 mm diameter and 1 ± 0,025 mm thick. Three minutes after the mix is started, place the plates and cement for 1 h in an atmosphere having a relative humidity of 95 % to 100 % at 37 ± 1 °C, then remove the specimen from the plates and store for 23 h in distilled water at 37 ± 1 °C.

6.7.3 *Procedure*

The opacity is represented by the contrast ratio. The contrast ratio ($C_{0,70}$) is the ratio between the daylight apparent reflectance of the cement specimen, 1 mm in thickness, when backed by a black backing, and the daylight apparent reflectance of the specimen when backed by a white backing having a daylight apparent reflectance of 70 % relative to magnesium oxide (MgO).

Make a comparison of the opacities of the cement specimen as detailed above with two opal glass standards having $C_{0,70}$ values of 0,35 and 0,90 respectively, by placing the specimen and standards against a variegated black and white background. During observations, cover the cement specimens, the standards, and the space between them and the black and white backing with a film of distilled water. If the opacity of the specimen is between or equal to either of the opacities of the standards, the cement shall be considered satisfactory.

If preferred, a suitable photometric method may be used to obtain the $C_{0,70}$ values provided that the accuracy is within ± 0,02 $C_{0,70}$.

6.8 Arsenic content

6.8.1 *Preparation of test specimen*

Grind to powder 1 g of the hardened cement, obtained from a specimen 24 h old that has been stored in a dry airtight container. The ground cement shall pass a 76 μm (200 mesh) sieve. Digest the powdered sample in 100 ml of distilled water on a steam bath for 1 h. Filter the solution and use the filtrate in the test for water-soluble arsenic.

6.8.2 *Procedure*

Determine the arsenic content using a recognized analytical technique for arsenic content whereby there is a comparison of the unknown quantity of arsenic against controls of known arsenic content of the same order.

If the arsenic content of any sample is close to the maximum value permitted, redetermine the arsenic content using the procedure detailed in ISO 2590, *General method for the determination of arsenic — Silver diethyldithiocarbamate photometric method*, and record this as the test result. In all instances the value shall be reported as mg/kg (ppm).

7 PACKAGING AND MARKING

7.1 Packaging

The cement powder and liquid shall be supplied in properly sealed containers made of materials which neither contaminate nor permit contamination of the contents.

7.2 Marking

7.2.1 *Manufacturer's identification*

The manufacturer's name and the trade brand name of the cement shall be printed legibly on each container of powder and each container of liquid.

7.2.2 *Lot numbers*

Each container of powder and each container of liquid shall be marked with a serial number or a combination of letters and numbers which refer to the manufacturer's records for that particular lot or batch of cement powder or liquid.

7.2.3 *Date of manufacture*

The date of manufacture (year and month) shall be indicated on the package as a separate item or as part of the lot number.

7.2.4 *Net mass and volume*

The net mass of the powder, in grams, and the net volume of the liquid, in millilitres, shall be indicated on the container.

7.2.5 *Type of cement*

The type of cement, as classified in clause 2, shall be stated on the container.

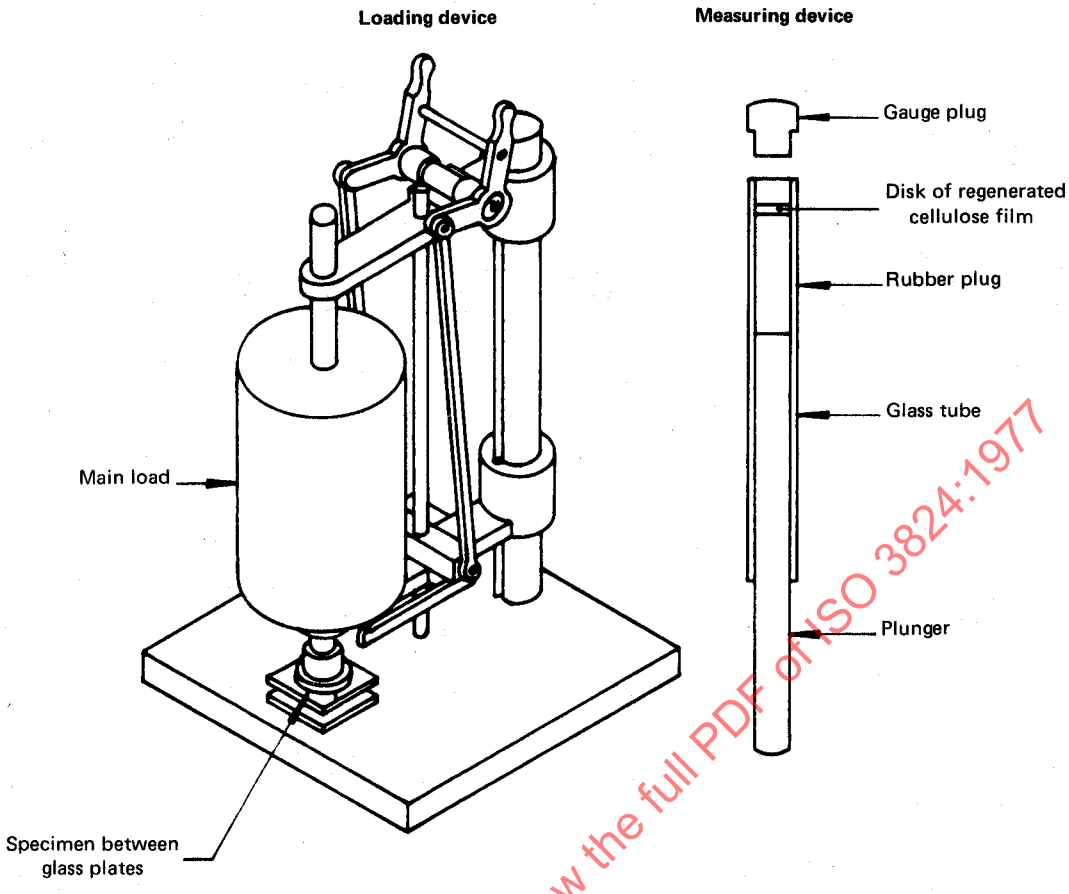

FIGURE 1 — **Apparatus for determination of standard testing consistency**

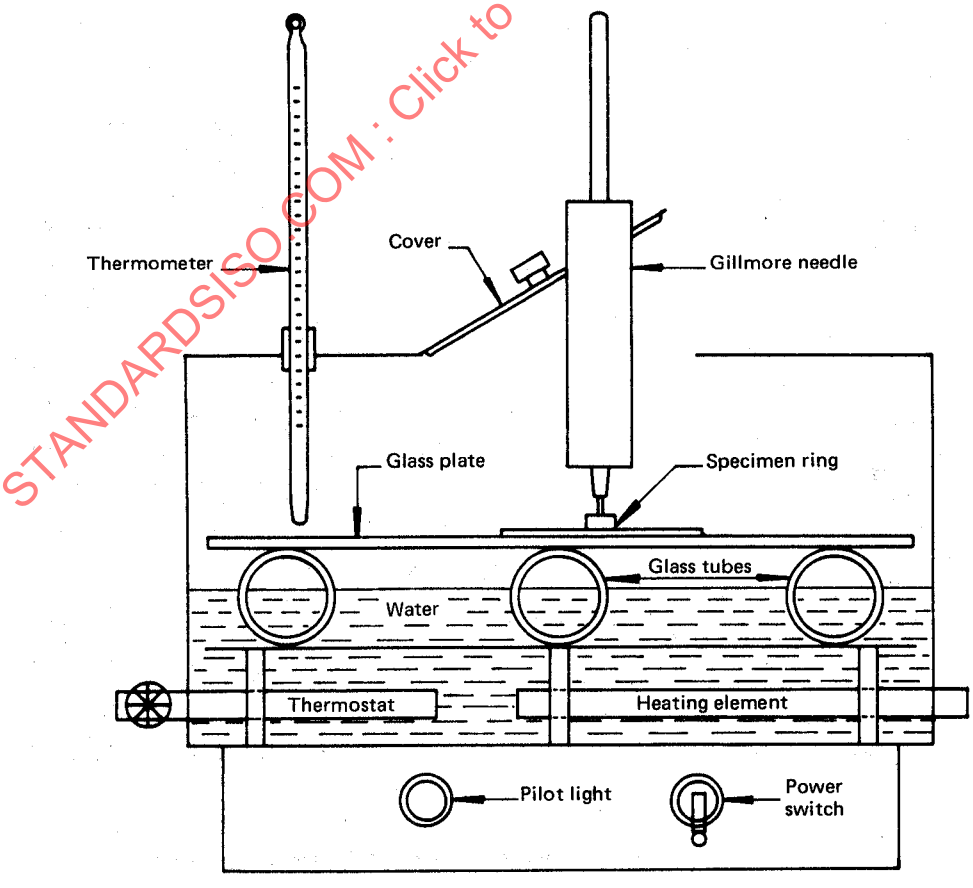

FIGURE 2 — **Apparatus for determination of setting time at controlled temperature and humidity**