YC 106

International Standard ISO 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).

Printed in Switzerland

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 capsulated 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 × 75 mm wide × 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 ± 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 mm^2. 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 µm.

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 µm.

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.

7.4.1 Apparatus

7.4.1.1 Oven or **cabinet** in which the specimen may be maintained at a temperature of 37 ± 1 °C and a relative humidity of at least 80 %.

7.4.1.2 Indentor of mass 400 ± 5 g and having a flat end of diameter 1,0 ± 0,01 mm. The needle tip shall be cylindrical for a distance of approximately 5,0 mm. The needle end shall be plane and at a right angle to the axis of the rod.

7.4.1.3 Metal mould, similar to the example illustrated in figure 3.

7.4.1.4 Metal block of minimum dimensions 8 mm × 20 mm × 10 mm, either as part of 7.4.1.1 or as a separate item.

7.4.1.5 Inert metallic foil

7.4.2 Procedure

Place the metal rectangular mould (7.4.1.3), conditioned to 23 ± 1 °C, on a piece of the metallic foil of convenient size and fill to a level surface with cement, of standard mixing consistency.

One minute after the completion of mixing, place the assembly, comprising mould, foil and cement specimen, on the metal block (7.4.1.4), which has been conditioned to 37 ± 1 °C and replace in the oven (7.4.1.1). Ensure good contact between the mould, foil and metal block.

One and a half minutes after the completion of mixing, carefully lower the indentor vertically onto the surface of the cement and allow 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 a hand lens of low magnification. Clean the needle, if necessary, between indentations. Repeat the process, starting the indentations at 30 s before the approximate setting time, making indentations at 10 s intervals.

Record the setting time as the period of time which elapses from the completion of mixing to the time when the needle fails to make a perceptible circular indentation on the surface of the cement, when viewed under a hand lens of X5 magnification.

Take the mean of three such tests, rounded to the nearest 15 s, as the test result.

7.5 Compressive strength

7.5.1 Apparatus

7.5.1.1 Oven or **cabinet** maintained at 37 ± 1 °C.

7.5.1.2 Split moulds and **plates**, such as shown in figure 4, with internal dimensions 6 mm high and 4 mm diameter, made of stainless steel or other suitable material that will not be attacked or corroded by the cement.

7.5.1.3 Individual screw clamps.

7.5.1.4 Compressive strength testing apparatus with a cross-head speed of 0,75 ± 0,25 mm/min.

7.5.2 Preparation of test specimens

Bring the moulds, top and bottom plates (7.5.1.2) and the screw clamps (7.5.1.3) to 23 ± 1 °C. Pack the cement, mixed to the standard testing consistency, to a slight excess into the assembled split mould, within 1 min of the completion of mixing.

NOTE — In order to consolidate the cement and avoid trapping air, it is advisable to convey the largest convenient portions of mixed cement to the mould and apply to one side with 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 and manually squeeze together. Put the mould and plates in the clamp (7.5.1.3) and screw tightly together. Not later than 2 min after the completion of mixing, transfer the whole assembly to the oven (7.5.1.1) maintained at 37 ± 1 °C.

One hour after the completion of mixing, remove the plates and surface the ends of the specimen plane at right angles to its long axis by the following method. Grind the ends flat and remove any excess cement by drawing back and forth on a glass plate with a small amount of 350 mesh (maximum particle size 45 µm) silicon carbide powder, mixed with water. Keep both ends of the specimen wet during the grinding and rotate about one quarter turn every few strokes.

Remove the specimen from the mould immediately after surfacing and carefully check 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 pure toluene. Alternatively a thin film of silicone grease or PTFE dry film lubricant may be used.

Immerse each acceptable specimen in distilled or deionized water and maintain at 37 ± 1 °C for 23 h.

Prepare five specimens.

7.5.3 Procedure

Twenty-four hours after the completion of mixing, determine the compressive strength of the five test specimens in the following manner.

Place each specimen with the flat ends between the platens of the testing apparatus so that the load is applied in the long axis of the specimen.

Record the maximum load applied when the specimen fractures and calculate the compressive strength, K, in megapascals using the formula

$$K = \frac{4F}{\pi d^2}$$

where

F is the maximum applied load, in newtons;

d is the diameter of the specimen, in millimetres.

If at least four out of the five results obtained are below the limit specified in the table, the material shall be deemed to have failed the test. If at least four out of the five results are above the limit specified in the table, the material shall be deemed to have passed the test. In other cases prepare a further 10 specimens and obtain the median result for all 15 specimens. Round this value to two significant figures and record as the compressive strength.

7.6 Water-leachable material

The determination shall be carried out in duplicate.

7.6.1 Apparatus

7.6.1.1 Oven or **cabinet** maintained at 37 ± 1 °C and a relative humidity of at least 30 %.

7.6.1.2 Mould consisting of a split brass or stainless steel ring contained in a former or retaining ring as illustrated in figure 5. The height of the ring shall be 1 ± 0,03 mm and the internal diameter 10 mm.

7.6.1.3 Individual screw clamps.

7.6.1.4 Platinum wire, dental floss, thread or other non-corrodible material.

7.6.1.5 Two wide mouthed polyethylene bottles of approximately 50 ml capacity, as illustrated in figure 6.

7.6.1.6 Spectrophotometer having a range covering 650 nm wavelength.

7.6.2 Reagents

All reagents shall be of analytical grade. Unless stated otherwise, distilled or deionized water shall be used.

7.6.2.1 Phosphate, standard solution.

Dissolve 0,200 g of anhydrous disodium hydrogen orthophosphate (Na_2HPO_4) in 1 000 ml of water. This will give a solution containing the equivalent of 100 µg of P_2O_5 per millilitre. Prepare a working standard containing 10 µg of P_2O_5 per millilitre, by diluting 10 ml of this standard solution to 100 ml.

7.6.2.2 Reagent I : Ammonium molybdate, 10 % (m/m) in a 35 g/l ammonia solution prepared by diluting 33 ml of concentrated ammonia solution, ϱ 0,88 g/ml, to 500 ml with water.

7.6.2.3 Reagent II : Sulfuric acid, solution, sulfuric acid (96 % m/m, ϱ 1,84 g/ml) diluted 1 + 1 with distilled water.

7.6.2.4 Reagent III : Ascorbic acid, 4 % (m/m) solution. It is essential that this solution be freshly prepared.

7.6.2.5 Reagent IV

Mix 40 ml of reagent I and 60 ml of reagent II; allow to cool, and add 100 ml of reagent III. It is essential that this solution be freshly prepared.

7.6.3 Preparation of test specimen

Place the mould (7.6.1.2) on a thin polyethylene or cellulose acetate sheet backed by a flat plate.

Insert a convenient tared length of wire or dental floss (7.6.1.4) through the split ring so that at least 4 mm projects into the ring. Fill the split ring with cement mixed to the standard testing consistency.

Cover with a further plate, faced with a sheet of polyethylene or cellulose acetate, press firmly together and apply the screw clamp (7.6.1.3).

Two minutes after the completion of mixing, place the mould, plates and the screw clamp into the oven (7.6.1.1) maintained at 37 ± 1 °C and a relative humidity of at least 30 %.

After 1 h, remove the plates and polyethylene or cellulose acetate sheets from the clamp and carefully separate the cement disc and attached wire or dental floss from the split ring. Remove any surplus cement from the edge of the disc and lightly brush the surface to remove any loose material. Prepare two test specimens.

7.6.4 Preparation of test solutions

Weigh the two prepared specimens immediately and suspend, by means of the wire or dental floss, in 20 ml of water contained in one polyethylene bottle (7.6.1.5). Ensure that the specimens only just touch the side of the bottle. Close the lid as tightly as possible and store for 23 h at 37 ± 1 °C. Prepare a blank at the same time.

7.6.5 Procedure

After 23 h, remove the specimens from the water and treat the contents of each bottle as follows :

Transfer the contents of each of the polyethylene bottles to a 200 ml flask and dilute each to the calibration mark with water. Transfer 10 ml aliquot portions of these solutions to 50 ml volumetric flasks, and add 5 ml of reagent IV to each, dilute the contents to the calibration marks and thoroughly mix. Treat 10 ml of the working standard phosphate solution similarly by

adding 5 ml of reagent IV and making the volume up to 50 ml in a volumetric flask. At the same time also prepare a blank, and take it through the whole procedure.

Allow these flasks to stand for 24 h and then measure the absorbance at 650 nm in the spectrophotometer (7.6.1.6).

7.6.6 Expression of results

The amount of water-leachable material, expressed as P_2O_5 eluted in milligrams per gram of the specimen, is given by the formula

$$\frac{A_1 - B}{A_2 - B} \times \frac{2}{m}$$

where

A_1 is the absorbance of the test solution;

A_2 is the absorbance of the standard phosphate solution;

B is the absorbance of the blank solution;

m is the mass, in grams, of the specimen.

NOTE — The absorbance, A_2 of the standard phosphate solution measured in a 1 cm cell is about 0,260 at 650 nm.

Record the average of the two results.

7.7 Opacity for type I cement (Luting agents) only

7.7.1 Apparatus

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

7.7.1.2 Suitable variegated black and white background.

7.7.1.3 Two flat glass plates at least 32 mm × 32 mm.

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

7.7.1.5 Clamp.

7.7.1.6 Polyethylene or **cellulose acetate sheets.**

7.7.2 Preparation of test specimens

Place the mould on a thin polyethylene or cellulose acetate sheet (7.7.1.6) backed by a flat glass plate (7.7.1.3). Fill the split ring (7.7.1.4) with cement of the standard testing consistency. Cover with the second plate faced with a sheet of polyethylene or cellulose acetate, press firmly together and clamp. One hundred and twenty seconds after the end of mixing, place the mould, plates and the screw clamp in the oven. After 1 h remove the plates and polyethylene or cellulose acetate sheets from the clamp and carefully separate the specimen from the ring. Store the specimen for 23 h in distilled water at 37 ± 1 °C.

7.7.3 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 (7.7.1.1) 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 (7.7.1.2). 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 $C_{0,70}$.

7.8 Arsenic content and lead content

7.8.1 Preparation of sample

Powder the set cement and sieve through a 75 µm (200 mesh) sieve. Disperse 2 g of the sieved powder in 40 ml of water and add 10 ml of hydrochloric acid (35 % m/m, 1,18 g/ml). Use this solution in the test for arsenic content and lead content.

7.8.2 Procedure

The arsenic content and the lead content may be determined using any recognised analytical method of adequate sensitivity.

For referee purposes the arsenic content shall be determined by the procedure described in ISO 2590.

8 Packaging and marking

8.1 Packaging

The components shall be supplied in securely sealed containers[1)], made from materials which do not react with or permit contamination of the contents.

1) For the purpose of this International Standard, the container should be considered as the immediate wrapping of the component.

8.2 Instructions for use

Instructions (detailed in 5.8) for proportioning powder and liquid and for manipulation shall accompany each package.

8.3 Marking of containers

Each container shall be clearly marked with the following particulars :

a) the name and/or trade mark of the manufacturer, and type of cement;

b) the shade of the powder according to manufacturer's shade guide, if supplied;

c) the minimum net mass, in grams, of the powder and the minimum net volume, in millilitres, of the liquid;

d) a serial number or code number (optional) and the actual date of manufacture (obligatory) according to ISO 2014;

e) the number of this International Standard, i.e. ISO 3824.

Table — Requirements for testing consistency and physical and chemical properties

Type	Consistency of mix mm	Time of setting at 37 °C minutes		Compressive strength (24 h) MPa	Film thickness µm	Water-leachable material mgP_2O_5/g	Opacity (24 h) $C_{0,70}$		Acid soluble arsenic content mg/kg (ppm)	Acid soluble lead content mg/kg (ppm)
	Diameter of disc	min.	max.	min.	max.	max.	min.	max.	max.	max.
I	28 ± 1	4	8	140	25	9,0	0,35	0,90	2,0	50
II	23 ± 1	2	5	170	—	6,0	—	—	2,0	50

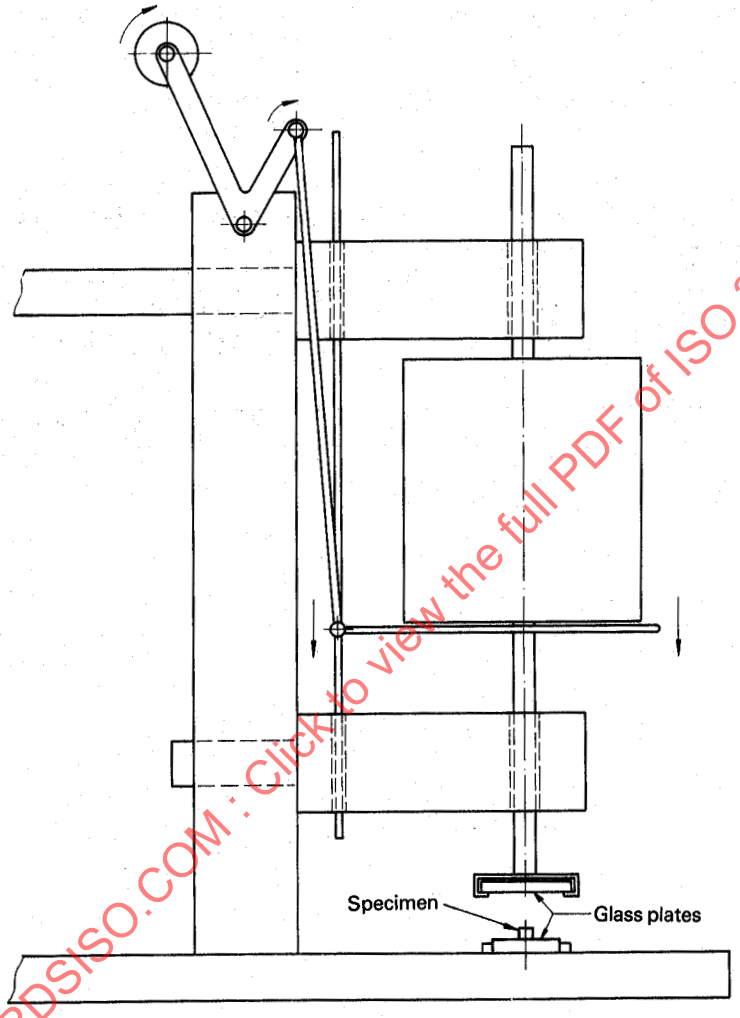

Figure 1 — Loading device for measuring consistency

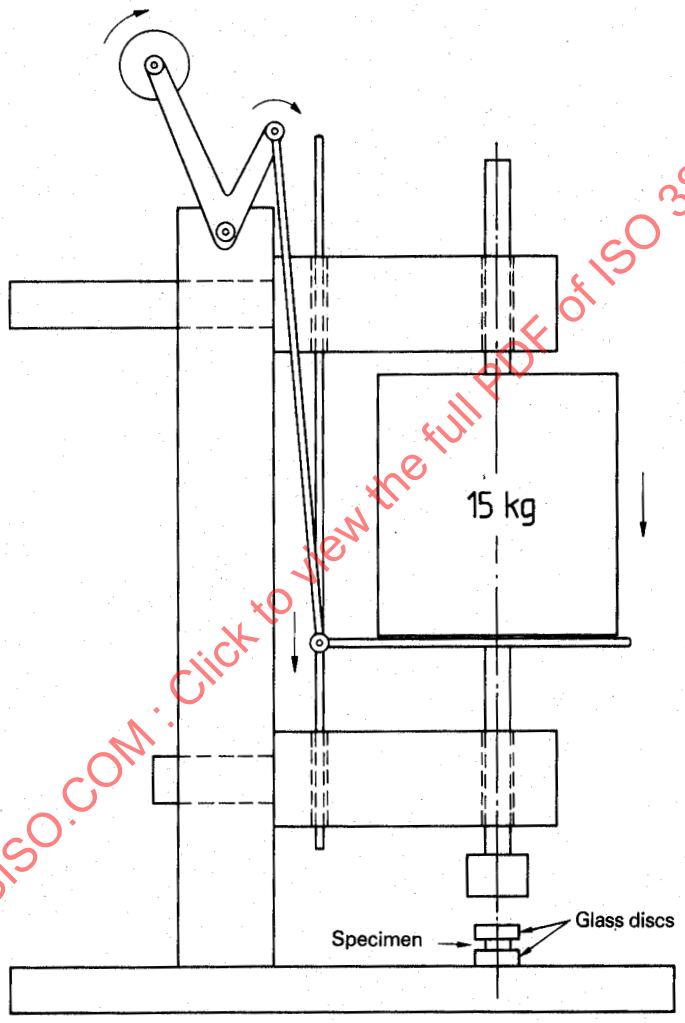

Figure 2 — Loading device for film thickness test

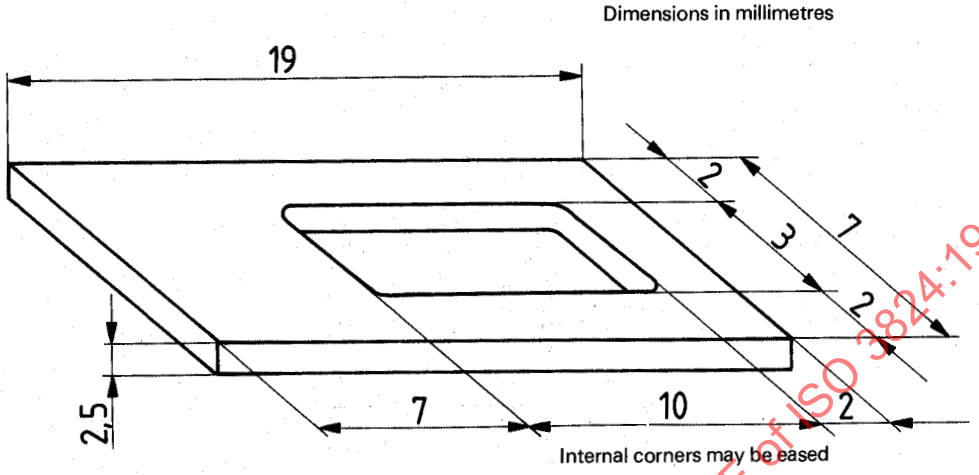

Figure 3 — Mould for use in determining the setting time

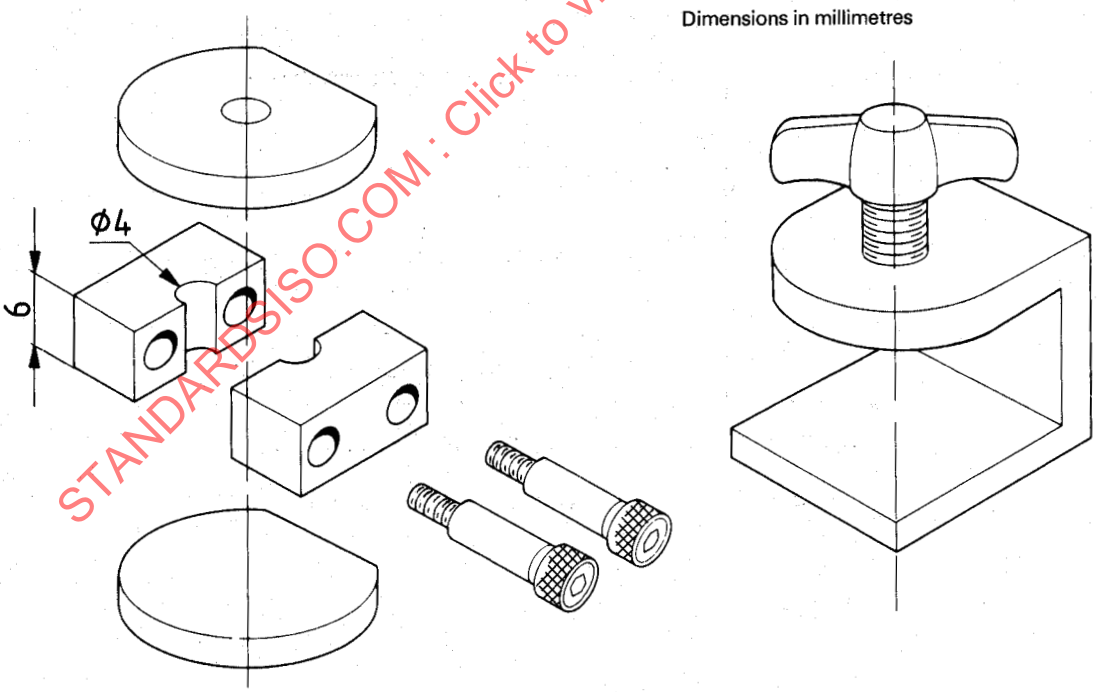

Figure 4 — Mould and clamp for preparation of compressive test specimen