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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION
R 1566

DENTAL ZINC PHOSPHATE CEMENT

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BRIEF HISTORY

The ISO Recommendation R 1566, *Dental zinc phosphate cement,* was drawn up by Technical Committee ISO/TC 106, *Dentistry,* the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1566, which was circulated to all the ISO Member Bodies for enquiry in December 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	India	Spain
Belgium	Israel	Sweden
Brazil	Korea, Rep. of	U.A.R.
Canada	Netherlands	United Kingdom
Czechoslovakia	New Zealand	U.S.A.
Denmark	Peru	Yugoslavia
France	Poland	
Greece	South Africa, Rep. of	

The following Member Body opposed the approval of the Draft :

Switzerland

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

ISO Recommendation | R 1566 | June 1970

DENTAL ZINC PHOSPHATE CEMENT

INTRODUCTION

This ISO Recommendation is technically identical with F.D.I.* Specification No. 6, the only difference being in the wording and layout to bring the text into standard ISO form. Further studies are being undertaken to provide, if necessary, for a future revision of this ISO Recommendation in the light of technological advances supported by well-documented data.

NOTE. – Throughout this ISO Recommendation the figures for SI units are approximate conversions of the technical metric units using the conversion factors 1 N = 0.102 kgf and 1 MN/m² = 10.2 kgf/cm².

1. SCOPE

This ISO Recommendation gives the classification of and requirements for dental zinc phosphate cement, together with the test methods to be used to determine compliance with these requirements.

2. FIELD OF APPLICATION

This ISO Recommendation is applicable to zinc phosphate cement, the primary uses of which are as follows :

(*a*) to join or to seal dental appliances to oral structures or to other appliances;

(*b*) to serve as a base or foundation for a tooth-filling material;

(*c*) to serve as a temporary filling material.

3. CLASSIFICATION

Zinc phosphate cement covered by this ISO Recommendation should be of the following types :

– **Type I** : Fine grain (see Appendix)

– **Type II** : Medium grain (see Appendix)

4. REQUIREMENTS

4.1 Material

The cement should consist of a powder and a liquid which, when mixed according to the manufacturer's instructions, will set and harden.

4.2 Liquid

4.2.1 *Condition.* The liquid should be water-clear and no deposits or sediment should form when it is stored in a container.

4.2.2 *Volume.* The liquid should be supplied in an amount 20 % in excess of that necessary to combine with the total amount of powder in a container of full portion size ** when mixed to a standard testing consistency. (See clause 7.2.)

4.3 Powder

The powder should be free from extraneous material such as dirt and lint. The pigment should be uniformly dispersed throughout the powder.

* Fédération Dentaire Internationale.

** Usually about 14 g.

4.4 Unset cement

Cement when spatulated as directed in section 6 should not form lumps or granules, or evolve gases.

4.5 Set cement

The colour of each cement when set should match the relevant manufacturer's shade guide.

4.6 Arsenic content

The limitation on arsenic content should be as listed in Table 1 below.

4.7 Physical properties

The time of setting, compressive strength, film thickness, and solubility and disintegration, should be as listed in Table 1 below.

TABLE 1 – Requirements for physical and chemical properties

Time of setting at 37 °C		Compressive strength after 24 h	Film thickness Min.		Solubility and disintegration after 24 h	Arsenic content
Min.	Max.	Min.	Type I	Type II	Max.	Max.
5 minutes	9 minutes	70 MN/m^2 (700 kgf/cm^2)	25 μm	40 μm	0.2 % (m/m)	0.0002 % (m/m) (1 part in 500 000)

4.8 Instructions

Instructions for proportioning and manipulation should include information regarding

- the temperature of the slab;
- the powder/liquid ratio;
- the rate of incorporation of the powder;
- the time of mixing;
- the maximum satisfactory working time after the end of the mixing.

5. SAMPLING

5.1 Sampling

The method of procurement and amount of the cement needed for testing should be the subject of agreement between the parties concerned.

5.2 Pooling of liquids

The contents of the sample containers of liquid should be emptied into a clean glass-stoppered bottle.

5.3 Pooling of powders

The contents of the sample containers of powder should be emptied into a square-sided jar having a capacity of 2 litres and a self-sealing cap.

The jar should be rotated on its minor axis for 2 hours at the rate of 25 revolutions per minute.

5.4 Test samples

All tests with the exception of those for the properties prescribed in clauses 4.2.1 and 4.3 should be conducted on specimens made from the composite samples of liquid and powder specified in clause 5.2 and 5.3 respectively.

6. PREPARATION OF TEST SPECIMENS

6.1 General

The preparation of all test specimens should be conducted at 23 ± 2 °C and at a relative humidity between 55 and 75 %. The powder/liquid ratio should be determined by the test for standard testing consistency (see clause 7.2).

6.2 Mixing

The following mixing technique should be employed in the preparation of all test specimens.

A polished glass slab approximately 150 mm long by 75 mm wide and a spatula, which should be made from a material not corroded by the cement, should be used for mixing. The spatula, and the slab should be clean and free from hardened particles of cement. The mixing time should be $1\frac{1}{2}$ minutes.

The incorporation of the powder should be accomplished at the rate shown in Table 2.

TABLE 2 – Rate of incorporation of powder

Proportion of total amount of powder	Time of incorporation in seconds
$\frac{1}{16}$	10
$\frac{1}{16}$	10
$\frac{1}{8}$	10
$\frac{1}{4}$	15
$\frac{1}{4}$	15
$\frac{1}{4}$	30

At least one-half of the top surface of the slab should be used. No particles of powder or any unused liquid should remain on the slab when the mixing is completed.

A linear and not a rotary motion of the spatula should be used, with the edge of the spatula sweeping approximately half of the mixing area of the slab on each stroke.

7. TEST METHODS

7.1 Visual inspection

Visual inspection should be used in determining compliance with the requirements outlined in clauses 4.1, 4.2.1, 4.3, 4.4, 4.5 and 4.8, and section 8.

7.2 Determination of standard testing consistency

7.2.1 *Apparatus.* The type of apparatus for measuring consistency should be essentially that shown in Figure 1. This apparatus consists of two flat glass plates, a load, a gauge plug and a glass tube, of inside diameter approximately 6.5 mm, which will deliver 0.50 ± 0.02 ml of mixed cement. The combined mass of the top plate and the load should be 120 g.

7.2.2 *Procedure.* Trial amounts of powder should be mixed with 0.50 ml of liquid. Then 0.50 ± 0.02 ml of each mix should be delivered from the glass tube onto a flat glass plate. 3 minutes after the commencement of mixing, the top glass plate, of mass approximately 20 g, and the necessary additional load required to total 120 g should be carefully lowered onto the soft cement.

Trials should be made until the average of the greater and lesser diameters of the slumped mass of cement is 30 ± 1 mm 10 minutes after starting the mix.

7.2.3 *Expression of results.* The average mass of powder used in three such determinations rounded off to the nearest 0.05 g combined with 0.50 ml of liquid should be by definition the standard testing consistency or amount of powder necessary to produce a mix of standard consistency.

7.3 Physical tests

7.3.1 *Time of setting*

7.3.1.1 APPARATUS. A type of apparatus found suitable for determining the time of setting is shown in Figure 2.

7.3.1.2 PROCEDURE. A brass ring maintained at room temperature, approximately 4.8 mm high, 11.1 mm outside diameter and 9.5 mm inside diameter, should be placed on a flat glass plate and filled with cement of standard consistency (see clause 7.2). 3 minutes after the commencement of mixing, the specimen should be transferred to an atmosphere at 100 % relative humidity at 37 ± 1 °C. $3\frac{1}{2}$ minutes after the commencement of mixing, a standard Gillmore needle, of mass 453.6 g and having an end 1.06 mm in diameter, should be carefully lowered vertically onto the horizontal surface of the cement. This should be repeated at 30 second intervals.

7.3.1.3 EXPRESSION OF RESULTS. The time of setting should be recorded as the number of minutes that elapse 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 theron under its own weight. This time should be reported to the nearest minute.

7.3.2 *Compressive strength*

7.3.2.1 APPARATUS (see Fig. 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.

(*b*) *Flat glass plates*, two.

(*c*) *C-clamp.*

(*d*) *Screw jack.*

(*e*) *Compressive strength testing instrument.*

7.3.2.2 PREPARATION OF TEST SPECIMENS. The test specimens should be cylinders 12 mm in height and 6 mm in diameter. The ends of the specimens should be flat, smooth, parallel to each other and at right angles to the long axis of the cylinder.

The cylindrical mould (7.3.2.1(*a*)) should be placed on a flat glass plate and slightly overfilled with cement of standard consistency within 3 minutes of commencing the mixing. The second flat glass plate should be pressed on top of the mould. The mould and plates should be held firmly together with the small C-clamp (7.3.2.1 (*c*)). 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 benzene.

3 minutes after starting the mix, the mould and clamp should be transferred to an atmosphere of 100 % relative humidity at 37 ± 1 °C.

1 hour later the ends of the specimens should be surfaced 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 200 mesh silicon carbide powder and water. The moulds should be rotated about one quarter turn every few strokes. The test specimens should be kept wet during the grinding.

After surfacing, the specimens may be removed from the mould by the screw jack (see Fig. 3) and should be immersed in distilled water at 37 ± 1 °C. The time lapse between the starting of mixing and the compression test should be 24 hours.

7.3.2.3 PROCEDURE. A specimen prepared according to clause 7.3.2.2 should be inserted between the platens of the testing machine with a small piece of wet blotting paper, approximately 0.5 mm thick, at each end. The specimens should be loaded at the rate of 2200 N (225 kgf) per minute until the crushing point is reached, and kept wet during the test.

7.3.2.4 EXPRESSION OF RESULTS. The value for compressive strength should be reported as the average of the values obtained for three or more specimens from a lot of five and should be rounded off to the nearest 1 MN/m^2 (10 kgf/cm^2). If the values for individual specimens fall more than 15 % below the average of the five, they should be discarded and the average of the remaining specimens should be reported. If more than two specimens are discarded, the test should be repeated.

7.3.3 *Film thickness.* A portion of a mix of standard consistency (see clause 7.2) should be placed between two flat square or round glass plates of uniform thickness. The surface areas of the plates between which the cement is spread should be approximately 200 mm^2.

3 minutes after the mix is started, a load of 150 N (15 kgf) should be applied vertically on the top plate. 10 minutes after the mix is started, the thickness of the two plates with the cement film between them should be determined.

The difference in the thickness of the plates with and without the cement film should be considered as the film thickness.

The average of three test values should be reported to the nearest 5 μm.

7.3.4 *Solubility and disintegration*

7.3.4.1 APPARATUS (see Fig. 4)

(*a*) *Split stainless steel ring*, inside diameter 20 mm and width 1.5 mm.

(*b*) *Flat plates*, two.

(*c*) *A tared piece of fine wire*, of platinum or other corrosion-resistant wire.

(*d*) *Tared weighing bottles*, two.

7.3.4.2 PREPARATION OF TEST SPECIMENS. Place 0.5 ml of cement of standard testing consistency in the split stainless steel ring, which is placed on one of the flat plates and separated from it by a thin polyethylene sheet.

The other flat plate, faced with a thin polyethylene sheet, should be used to press the cement into the ring.

The tared piece of fine platinum or corrosion-resistant wire should be placed in the soft cement as the specimens are formed to provide a convenient means of holding the specimens.

3 minutes after the mix is started, the plates and cement should be placed for 1 hour in an atmosphere having a relative humidity of 100 % at 37 °C.

7.3.4.3 PROCEDURE. Two specimens, prepared in accordance with clause 7.3.4.2, should, after 1 hour in conditions of controlled temperature and humidity, be placed in each tared weighing bottle and weighed. The combined mass of the two specimens and the weighing bottle, less the mass of the weighing bottle and the platinum wire, should be taken as the mass of the specimens of cement.

The specimens should immediately be submerged by pouring 50 ml of distilled water into the weighing bottle, which should be stored for 23 hours at 37 ± 1 °C.

The specimens should then be removed from the water. There should be no evidence of crystal growth on the surface of the specimens. The water should be evaporated from the weighing bottle at a temperature just below 100 °C. The weighing bottle should then be dried 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), the weighing bottle and contents should be weighed with a precision of 0.2 mg.

This cycle of heating the weighing bottle to 150 °C, cooling over a desiccant and reweighing should be repeated until the loss in mass of each bottle is not more than 0.5 mg in any 24 hour period.

7.3.4.4 EXPRESSION OF RESULTS. The difference between the initial and final mass of the weighing bottle should be recorded 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.

The average of values from duplicate tests (two weighing bottles containing two specimens each) should be reported to the nearest 0.1 %.

7.4 Chemical test

7.4.1 *Soluble arsenic content*

7.4.1.1 PRINCIPLE. Digestion of a sample of hardened cement in distilled water. Filtering of the solution. Reduction of arsenic in the filtrate to hydrogen arsenide by zinc in an acid medium. Passage of the hydrogen arsenide through paper soaked with mercuric bromide, to produce a yellow stain the intensity of which is increased in turning it to brown by potassium iodide. Determination of arsenic content by comparison with a standard paper strip.

7.4.1.2 PREPARATION OF TEST SOLUTION. 1 g of hardened cement obtained from a specimen 24 hours old that has been stored in a dry airtight container should be powdered to pass a No. 200 sieve. The powdered sample should be digested in 100 ml of distilled water on a steam bath for 1 hour. The solution should be filtered and the filtrate used in the test for water-soluble arsenic.

7.4.1.3 REAGENTS. The reagents should be of a recognized analytical reagent quality and free from arsenic. Distilled water should be used throughout.

(*a*) *Standard arsenic solution* (0.001 mg of arsenic per millilitre of solution).

Dissolve 0.132 g of arsenic trioxide (As_2O_3) in 10 ml of 100 g/l sodium hydroxide solution prepared by dissolving 10 g of sodium hydroxide in water and diluting to 100 ml. Neutralize the alkaline arsenic solution with diluted sulphuric acid (one volume of concentrated acid plus nine volumes of water). Add 10 ml more of the diluted acid and dilute with water to 1 litre. The solution contains 0.1 mg of arsenic per millilitre.

To 10 ml of this solution add 10 ml of diluted sulphuric acid (1 + 9) and dilute with water to 1 litre. This final solution contains 0.001 mg of arsenic per millilitre.

(*b*) *Potassium iodide solution*, 100 g/l.

Dissolve 10 g of potassium iodide in water and dilute to 100 ml.

(*c*) *Stannous chloride solution*, 400 g/l.

Dissolve 40 g of stannous chloride dihydrate ($SnCl_2 . 2H_2O$) in concentrated hydrochloric acid and dilute to 100 ml with concentrated hydrochloric acid.

(*d*) *Lead acetate solution*, 100 g/l.

Dissolve 10 g of lead acetate [$Pb(C_2H_3O_2)_2$] in water and add enough acetic acid to clear the solution. Dilute with water to 100 ml.

(*e*) *Mercuric bromide paper strips.*

Use commercially cut strips which are 2.5 mm wide and cut to a length of 120 mm. Soak the strips in 50 g/l mercuric bromide ($HgBr_2$) solution for 1 hour and dry in air. Prepare the mercuric bromide solution by dissolving 5 g of mercuric bromide in 95 % ethanol and diluting with ethanol to 100 ml.

(*f*) *Sulphuric acid*, concentrated.

(*g*) *Zinc*, granulated.

7.4.1.4 APPARATUS. A suitable apparatus is shown in Figure 5. The generator consists of a 150 ml wide-mouthed bottle (C) which is fitted with a perforated rubber bung. Through the perforation is inserted a vertical exit tube (B) about 120 mm in overall length, about 10 mm in diameter on its upper portion, and constricted at its lower extremity to a tube about 40 mm in length and about 5 mm in diameter. The small portion of the tube should extend to just below the bung. The wider part of the tube should be packed with glass wool as shown in Figure 5. Into the upper end of this tube should be fitted, by means of a rubber bung, a second glass tube (A), 120 mm in length with an internal diameter of 2.5 to 3 mm.

7.4.1.5 PROCEDURE. Transfer the filtrate, prepared as directed in clause 7.4.1.2, to the wide-mouthed bottle. For the comparison standard, place in the bottle of a second generator 98 ml of water and 2.0 ml of standard arsenic.

Add to each bottle : 5 ml of concentrated sulphuric acid; 7.5 ml of potassium iodide solution and 0.20 ml of stannous chloride solution. Mix and allow the bottle to stand for 20 minutes in a water bath at 23 ± 2 °C.

During this 20 minutes period,

(1) moisten the glass wool in the lower tube (B) with lead acetate solution*;

(2) carefully centre a dry mercuric bromide paper strip in the top tube (A). Crimp the upper end of the paper strip so that 100 mm of its length will be in position in the tube.

At the end of the 20 minutes period,

(1) add 5 g of granulated zinc (arsenic free) to the solution in each generator bottle;

(2) put tubes (A) and (B) in place as shown in Figure 5.

Return the generators to the water bath for $1\frac{1}{2}$ hours before comparing the stains. Remove the strips and for each strip average the length of the stains on both sides.

7.4.1.6 EXPRESSION OF RESULTS. If the yellow-to-brown stain is shorter for the sample than for the standard, the amount of water-soluble arsenic in the cement is less than 0.0002 % (m/m).

8. PACKAGING AND MARKING

8.1 Packaging

The cement powder and liquid should be supplied in properly sealed containers made of materials which will not contaminate or permit contamination of the contents.

8.2 Instructions for use

Instructions for proportioning the powder and liquid, and for manipulation of the cement, should accompany each package.

8.3 Marking

8.3.1 *Lot numbers.* Each container of powder and each container of liquid should 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.

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

8.3.3 *Net mass and volume.* The net mass of the powder, in grammes, and the net volume of the liquid, in millilitres, should be indicated on the container.

8.3.4 *Type.* The type of cement (see section 3) should be indicated on the container.

* The character of the stain is affected by the amount of lead acetate solution used to moisten the glass wool. If the wool is too wet, the stain, which appears on the mercuric bromide paper soon after the zinc is added, will be partly washed out at the end of the $1\frac{1}{2}$ hours. For this reason, all tubes in a set of generators should be charged with equal amounts of lead acetate, and any excess must be drawn off by suction.

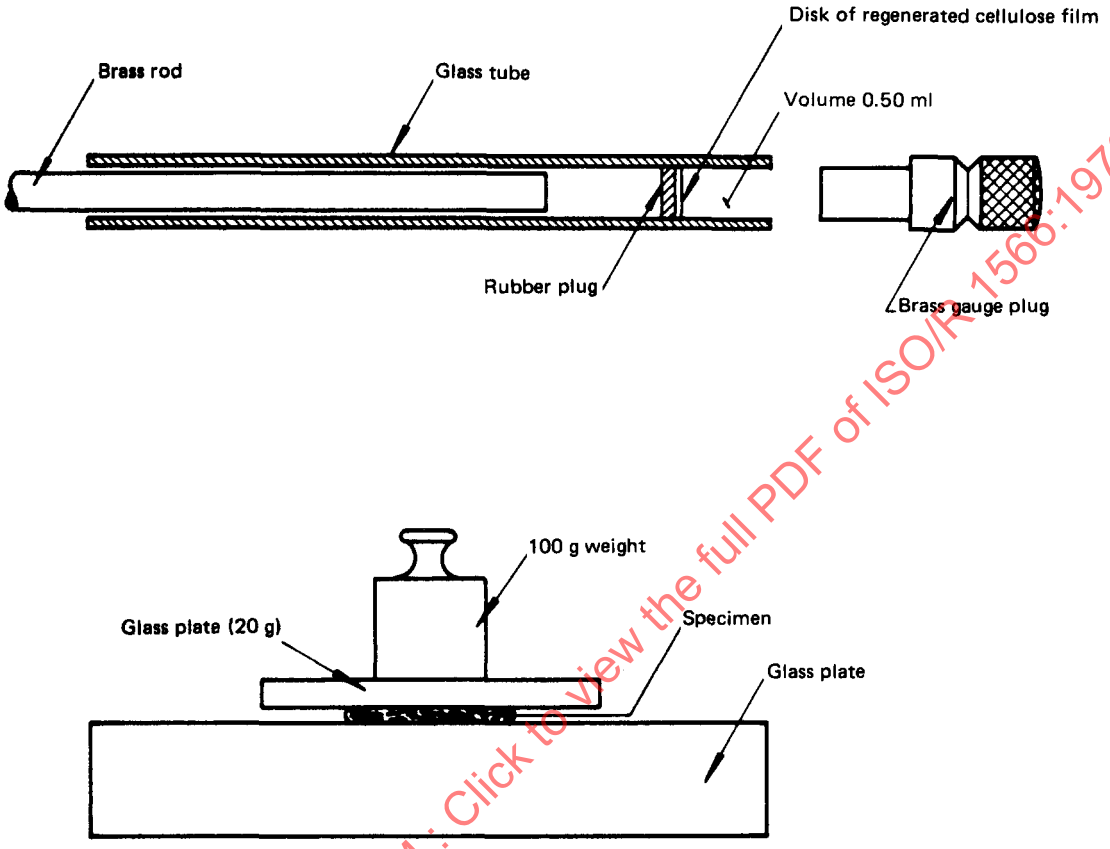

FIG. 1 – Apparatus for determination of standard testing consistency

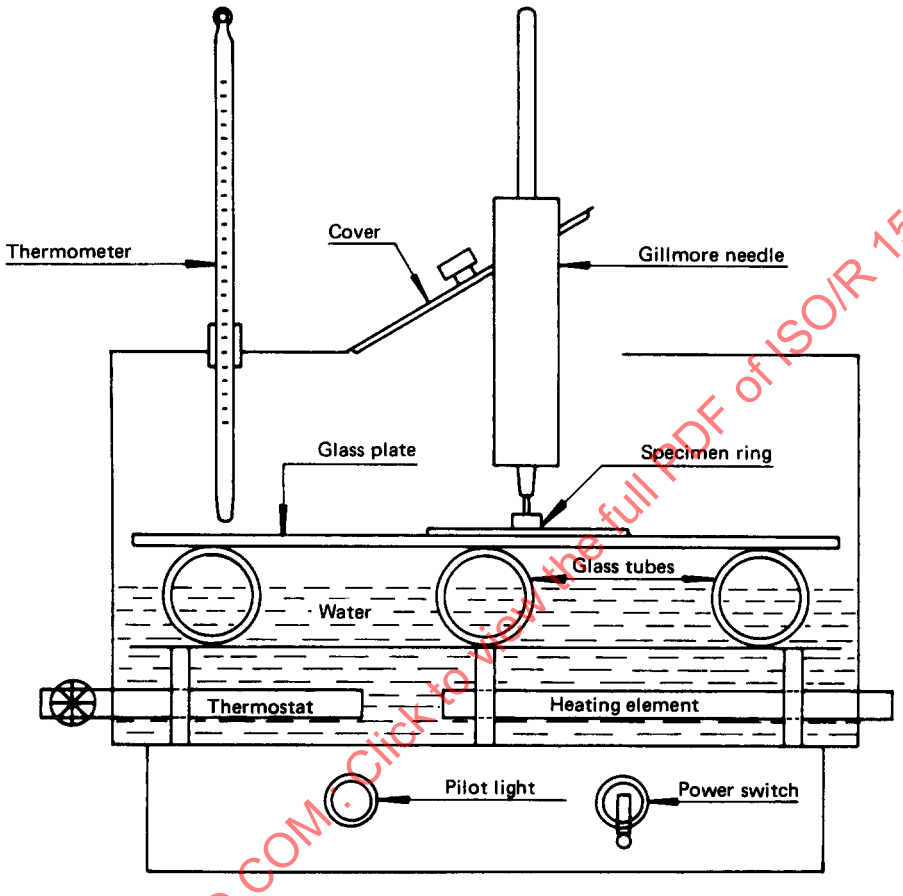

FIG. 2 – **Apparatus for determination of setting time at controlled temperature and humidity**

Dimensions in millimetres

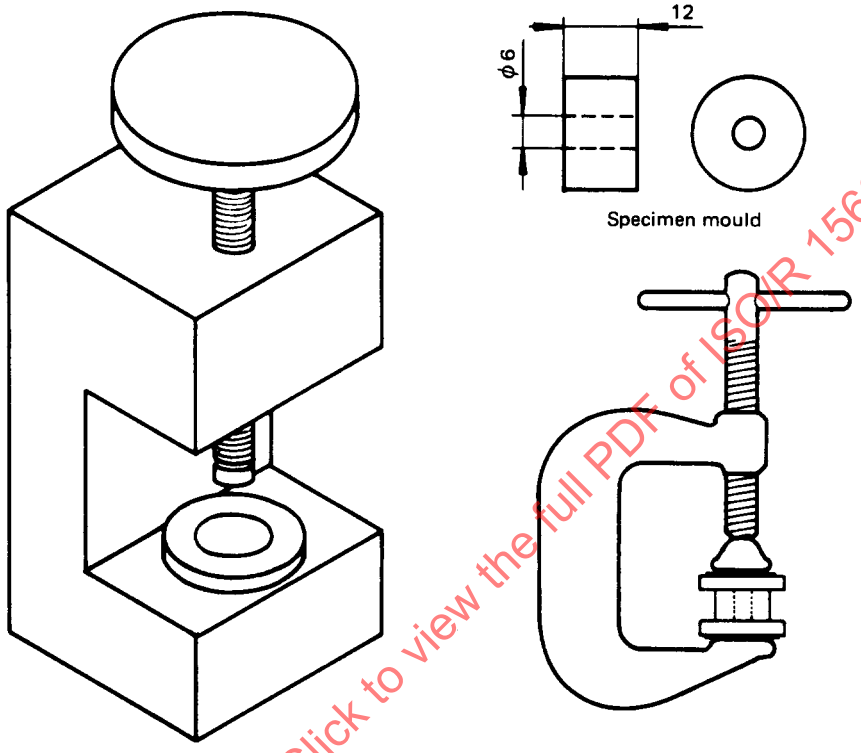

FIG. 3 – **Apparatus for forming compressive strength test specimens**