INTERNATIONAL STANDARD 3106

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

Dental zinc oxide/eugenol filling materials

Produits d'obturation dentaire à base d'oxyde de zinc-eugénol

First edition — 1974-10-01

UDC 616-314 : 615-463

Ref. No. ISO 3106-1974 (E)

Descriptors : materials specifications, dental materials, zinc oxides, specifications, tests.

Price based on 8 pages

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (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 set up 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 3106 was drawn up by Technical Committee ISO/TC 106, *Dentistry*, and circulated to the Member Bodies in May 1973.

It has been approved by the Member Bodies of the following countries :

Australia	Germany	Spain
Brazil	India	Sweden
Bulgaria	Ireland	Switzerland
Canada	Netherlands	Thailand
Czechoslovakia	New Zealand	United Kingdom
Egypt, Arab Rep. of	Romania	U.S.A.
France	South Africa, Rep. of	Yugoslavia

No Member Body expressed disapproval of the document.

Printed in Switzerland

Dental zinc oxide/eugenol filling materials

0 INTRODUCTION

This International Standard only covers commercially available accelerated zinc oxide/eugenol filling materials. While it is appreciated that there is a need in some countries for a specification to cover the purity of unmodified zinc oxide and eugenol intended for dental use, it is felt that, despite certain similarities, this could be much more conveniently covered by a separate specification. It is envisaged that further work with this in mind will be scheduled on a national level with possible consideration by ISO/TC 106 at a later date.

Although the zinc oxide/eugenol materials covered by this International Standard are those intended for use in dental filling procedures and not cementation, the more familiar and convenient generic term "cement" has been used in the text as this is in accord with both common usage and the definition for dental "cement" currently being considered by ISO/TC 106.

1 SCOPE

This International Standard specifies requirements for accelerated zinc oxide/eugenol filling materials supplied as two separate components and intended for use in the oral cavity.

2 FIELD OF APPLICATION

Zinc oxide/eugenol filling materials covered by this International Standard are those manufactured for use as cavity linings and bases, temporary and root-canal fillings.

3 CLASSIFICATION

Zinc oxide/eugenol filling materials covered by this International Standard shall be classified as one of the following two types, according to their setting time (see 4.2.1) :

Type 1 — Fast setting (3 to 5,5 min)

Type 2 — Normal setting (4,5 to 7 min)

4 REQUIREMENTS

4.1 Components

4.1.1 *General*

The components, when mixed in accordance with the manufacturer's instructions, shall set rapidly to a condition suitable for the intended dental use.

4.1.2 *Liquid*

The liquid shall be clear with possibly a light amber tinge, and free from suspended matter, deposit or sediment.

4.1.3 *Powder*

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

4.1.4 *Purity of ingredients*

The quality of the ingredients used in the manufacture of the cement components shall conform with the relevant national pharmacopœia standards of purity, or such national regulations as are applicable to purity of pharmaceutical products.

4.2 Physical properties (see table)

TABLE — **Physical properties — Test requirements**

Consistency: Disc diameter at 23 ± 1 °C	**Setting time at 37 ± 1 °C**		**Compressive strength: Minimum after 24 h**	**Solubility and *disintegration*: after 24 h**
25 ± 1 mm (see 6.2)	Type 1 3 to 5,5 min (see 6.3)	Type 2 4,5 to 7 min (see 6.3)	25 MN/m^2 (see 6.4)	0,5 % by mass (see 6.5)

4.2.1 *Setting time*

The setting time of the cement determined in accordance with 6.3 shall conform to the following limits :

Type 1 : 3 to 5,5 min

Type 2 : 4,5 to 7 min

4.2.2 *Compressive strength*

The compressive strength of the cement 24 h after mixing, determined in accordance with 6.4, shall not be less than 25 MN/m^2.

4.2.3 *Solubility and disintegration*

The amount of non-volatile material removed from the test specimen, when determined in accordance with 6.5, shall not be more than 0,5 % by mass, after immersion for 23 h.

4.3 Information to be supplied by the manufacturer

Adequate instructions for guidance of the user in proportioning, mixing and manipulation shall accompany each container. The following details shall be included :

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

b) an appropriate component ratio at the recommended ambient conditions;

c) the rate of incorporation of one component with the other and the maximum mixing time.

5 SAMPLING AND INSPECTION

5.1 Sampling

5.1.1 *Powder*

Not less than 50 g of powder from the same batch shall comprise the test sample.

5.1.2 *Liquid*

Not less than 25 ml of liquid from the same batch shall comprise the test sample.

5.2 Inspection

Compliance with the requirements outlined in 4.1.2, 4.1.3 and 4.3 shall be determined by visual inspection.

6 TEST METHODS

6.1 Preparation of test specimens

6.1.1 *Ambient conditions*

Carry out all mixing of cement for the preparation of the test specimens at a temperature of 23 ± 1 °C and a relative humidity of 50 ± 2 %.

6.1.2 *Mixing*

6.1.2.1 APPARATUS

a) **Smooth glass slab** approximately 150 mm × 75 mm × 20 mm.

b) **Rigid spatula** with a blade having dimensions approximately 45 mm × 8 mm, made from a material not affected by the cement.

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

Before the commencement of mixing, condition the test samples and apparatus at the ambient conditions specified in 6.1.1, except where otherwise stated.

6.1.2.2 PROCEDURE FOR POWDER/LIQUID MIXING

Place on the glass slab the correct mass of powder and volume of liquid, as determined by the consistency test specified in 6.2, and divide into four portions as follows :

a) Divide the powder approximately in two halves.

b) Divide one half into two quarters.

c) Divide one quarter into two eighths.

Mix the material by incorporating the half-portion of the powder into the liquid in the first 15 s, then the quarter- and eighth-portions, each at 15 s intervals, each portion being thoroughly mixed before introducing the next portion. Then spatulate the whole mass with reasonable pressure for a further 15 s, utilizing approximately one-third of the top surface of the glass slab. The total mixing time shall be 1,25 min.

Leave no powder or liquid on the slab when the mixing has been completed.

6.2 Consistency test

6.2.1 *Apparatus*

6.2.1.1 Load of mass 2 480 ± 5 g mounted on a loading device such as that shown in figure 1, in such a manner as to allow essentially frictionless movement in a vertical direction.

6.2.1.2 Two glass plates of minimum dimensions 30 mm × 30 mm, one having a mass of 20 ± 2 g.

6.2.1.3 Graduated hypodermic-type syringe, capacity 0,50 ml, with an accuracy of ± 0,005 ml.

6.2.1.4 Measuring device designed to deliver 0,50 ± 0,05 ml of mixed cement.

A suitable device is illustrated in figure 2 and comprises :

a) a glass tube with internal diameter approximately 10 mm;

b) a gauge-plug and plunger;

c) a rubber or plastics plug, and polyethylene disc (maximum thickness of polyethylene disc 0,10 mm).

6.2.2 *Preparation of components*

Carefully weigh a trial amount of powder and place at one end of the glass slab. Using the graduated syringe, deposit 0,40 ml of liquid towards the other end of the slab with at least half the length of the slab separating it from the powder.

Mix the powder and liquid in accordance with 6.1.2.2 and at the conclusion of the mix collect the cement in a convenient mass on the glass slab.

6.2.3 *Procedure*

Completely fill the end of the glass tube with cement, with the rubber or plastics plug and polyethylene disc in position to measure 0,5 ml of the cement by volume. (Two or three shallow V-cuts along the side of the plug will, if the plug is slightly over-sized, ensure a tight fit and prevent air being trapped during the filling operation.) Carefully extrude the measured quantity (0,5 ml) of each mix from the glass measuring device onto the glass plate, taking care to avoid mis-shaping the cylindrical form of the resultant cement specimen. Allow the polyethylene disc to stay in place.

Place the cement resting on the glass plate in position on the loading device so that the cement is centrally below the supported 2 480 g mass. Two minutes after the commencement of mixing, lower the top glass plate, with a mass of 20 g, and the mass of 2 480 g (a total load of mass 2 500 g) gently onto the cement and allow to remain there for 5 min.

NOTE — It is essential during this testing procedure that the glass plates are maintained parallel to each other and that no rotary movement is allowed to take place.

Measure the resulting disc across two diameters at right angles to each other and average the two measurements, if they agree to within 1 mm, to give a mean diameter. If the disc is not uniformly circular or if they do not agree to within 1 mm, repeat the test. Make trial mixes to known component ratios at a temperature of 23 ± 1 °C and a relative humidity of 50 ± 2 % until a standard consistency disc is formed with a mean diameter of 25 ± 1 mm.

NOTE — The cement disc may be measured with or without the top glass plate in position. If it is intended to remove the top plate, then allow the cement to harden completely before doing so.

The placing of some form of graph paper (the polar graph type is very suitable) under the lower glass plate is strongly recommended as an aid to the rapid and accurate reading of the disc diameter.

6.2.4 *Calculation and expression of results*

The average of three such determinations shall be taken and the results expressed in grams per millilitre rounded off to the nearest 0,05 g/ml. The ratio shall be referred to as the component ratio or testing consistency for the cement under test.

6.3 Setting time

6.3.1 *Apparatus*

6.3.1.1 Oven or **cabinet,** maintained at a temperature of 37 ± 1 °C and a relative humidity of not less than 90 %.

6.3.1.2 Gillmore-type needle with a mass of 450 ± 5 g, having a flat end of 1,00 ± 0,05 mm diameter, with the needle cylindrical for a distance of 2,5 mm from its end and the needle end plane and at right angles to the axis of the rod maintained in a clean condition.

6.3.1.3 Brass mould consisting of a rectangular plate with a circular hole conforming to dimensions given in figure 3.

NOTE — The asymmetrical form of the mould has been designed for ease of handling.

6.3.1.4 Metal block of minimum dimensions 8 mm × 20 mm × 10 mm either as part of 6.3.1.1 or 6.3.1.2 or else as a separate item.

6.3.1.5 Flat glass plate approximately 1 mm thick (microscope slides are suitable).

6.3.2 *Preparation of test specimen*

Place the mould on the flat glass plate and fill with cement of standard consistency to a level surface. Two minutes after the commencement of mixing, place this assembly on the metal block which has been conditioned in the oven to a temperature of 37 °C.

6.3.3 *Procedure*

Two and a half minutes after the commencement of mixing, carefully lower the Gillmore-type needle vertically onto the horizontal surface of the cement which is still retained in the oven at 37 °C. Repeat at 15 s intervals near the time of setting.

Record the time of setting as the period of time which elapses from commencement of mixing to the time when the needle fails to completely penetrate the 2 mm depth of the cement contained in the mould. This penetration can be confirmed by holding the specimen up to the light and examining visually. Repeat this test once.

6.3.4 *Calculation and expression of results*

The average of two determinations shall be calculated and the result recorded, to the nearest 15 s, as the setting time.

6.4 Compressive strength

6.4.1 *Apparatus*

6.4.1.1 Oven or **cabinet** maintained at a temperature of 37 ± 1 °C.

6.4.1.2 Five moulds and plates, such as those shown in figure 4, 12 mm high and 6 mm internal diameter, made of stainless steel of other suitable material which is not attacked or corroded by the cement.

6.4.1.3 Five individual screw-type clamps, such as those shown in figure 4.

6.4.1.4 Compressive strength testing apparatus having a cross-head speed of 1,50 ± 0,75 mm/min.

6.4.2 *Preparation of test specimens*

Condition the moulds and top and bottom plates at the specified testing temperature (see 6.1.1) and the clamps at 37 °C.

Using a suitable spatula (6.1.2.1 b)) pack a slight excess of the cement, mixed to the standard testing consistency, into the mould within 2 min of commencing mixing. It is advisable, in order to avoid trapping air and to facilitate consolidation of the cement, to convey to the mould the largest convenient portion of the mix and to apply with the spatula to the side of the mould with the mould open at each end.

Then firmly place the mould on the bottom metal plate and remove any bulk extruded excess of cement. Place the top metal plate in position and squeeze the assembly tightly together with the screw clamp. Three minutes after commencing the mix, transfer the assembly to the oven maintained at a temperature of 37 °C. One hour after commencing the mix, remove the metal plates and surface the ends of the specimen plane at right angles to their long axes.

Surface the ends of the hardened cement specimen and remove any excess cement by grinding on a glass plate with a small amount of 45 μg (– 350 mesh) silicon carbide, or other suitable abrasive powder mixed with water. Alternatively, an equivalent abrasive paper, suitably supported, may be used. Draw the mould containing the cement specimen back and forth across the plate and rotate about one quarter-turn every few strokes. During the grinding operation, keep both ends of the specimen wet.

Immediately after surfacing, remove the cement specimen from the mould.

NOTE — To facilitate the removal of the hardened cement, the internal surfaces of the mould may be evenly coated, prior to filling, with a thin (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.

Rapidly check the test specimen for air-voids or chipped edges and if found, discard the specimen.

Immerse the specimen in distilled water maintained at 37 ± 1 °C for 23 h.

Prepare at least five such test specimens.

6.4.3 *Procedure*

Twenty-four hours after commencing the mix, determine the compressive strength of the specimens by means of a suitable testing apparatus (6.4.1.4).

Place the test specimen with its flat ends between the anvils of the testing apparatus so that the load is applied to the long axis of the test specimen. Record the maximum load applied when the specimen fractures, in newtons.

6.4.4 *Calculation and expression of results*

The compressive strength C, in meganewtons per square metre (newtons per square millimetre), is given by the following formula :

$$C = \frac{4P}{\pi d^2}$$

where

P is the maximum applied load, in newtons;

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

A total of five tests shall be carried out and the values obtained rounded off to the nearest whole number (in the case of 0,5 values, round up to the higher whole number). The mean of these five values shall be recorded as the test result.

6.5 Solubility and disintegration

6.5.1 *Apparatus*

6.5.1.1 Oven or **cabinet**, maintained at a temperature of 37 ± 1 °C.

6.5.1.2 Mould consisting of a split brass or stainless steel ring contained in a former as illustrated in figure 5, the wall thickness of the ring being 1 mm and the internal dimensions 20 mm diameter, and 1,5 mm depth.

6.5.1.3 Platinum wire, dental floss or an **equivalent non-corrosive material.**

6.5.1.4 Three tared glass weighing bottles such as those shown in figure 6.

6.5.1.5 Multiple spring clamp such as that shown in figure 7.

Condition the spring clamp by placing in the oven at least 5 min before preparing the test specimen. Do not remove until required for insertion of individual specimens.

6.5.2 *Preparation of test specimens*

Place the mould on a thin polyethylene or cellulose acetate sheet backed by a flat glass plate. Weigh a convenient length or wire/dental floss and insert through the split in the ring, so that at least 10 mm projects into the ring. Place a slight excess of the cement mixed to a standard testing consistency in the mould and press another flat glass plate faced with a sheet of thin polyethylene or cellulose acetate on top of it. Hold the whole assembly firmly together with the spring clamp.

Three minutes after the commencement of mixing, place the assembly in the oven maintained at a temperature of 37 °C. One hour later remove the assembly, separate the glass plates and polyethylene or cellulose acetate sheets and carefully take the cement disc with attached wire/dental floss from the split ring.

NOTE – Due to the comparatively brittle nature of some cements at this early hardening stage, it is essential to clean any excess cement from the surface of the split-ring before attempting removal of the specimens. It is also recommended that a suitable release agent be used on the split ring to facilitate removal of the specimen from the mould. PTFE dry film lubricant is suggested.

Remove any surplus cement from the edge of the disc and lightly brush any loose material from the surface. Prepare two such specimens for each determination.

6.5.3 *Procedure*

Place the two test specimens in a tared weighing bottle which has been previously conditioned to constant mass (reading A), and weigh the whole assembly. Take the combined mass of the two cement disc specimens and the weighing bottle less the mass of the weighing-bottle and the wire/dental floss as the mass of the cement specimens.

Immediately submerge the two discs by pouring 50 ml of distilled water into the weighing bottle, then store for 23 h at 37 ± 1 °C. Suspend the specimens by the wire/dental floss so that they neither touch each other nor rest against the side of the bottle and close the bottle lid as tightly as possible.

Twenty-four hours after the commencement of mixing, remove the specimens from the water. Evaporate the water from the weighing bottle at a temperature just below 100 °C and dry the weighing bottle in an oven at 150 °C for 24 h. After cooling to room temperature in a desiccator containing a suitable desiccant, weigh the weighing bottle and contents with a precision of 0,1 mg (reading B).

Carry out the test with a bottle containing 50 ml of distilled water as a control. Subject the bottle and contents to all steps of the test procedure and apply the blank correction.

6.5.4 *Calculation and expression of results*

The solubility S, expressed as a percentage by mass, is given by the following formula :

$$S = \frac{\text{Reading B} - \text{Reading A}}{\text{Mass of cement specimens}} \times 100\ \%$$

The average of duplicate tests (two weighing bottles containing two specimens each) shall be reported to the nearest 0,01 %.

7 PACKAGING AND MARKING

7.1 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.

7.2 Marking of containers

Each container shall be clearly marked with the following :

a) the name and/or trade-mark of the manufacturer;

b) the type of cement (see clause 3);

c) a serial number or code which shall refer to the manufacturer's record and date of manufacture for that particular batch of cement powder or liquid;

d) the minimum net mass, in grams, of the powder and the minimum net volume, in millilitres, of the liquid, shall be indicated on the appropriate containers.

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

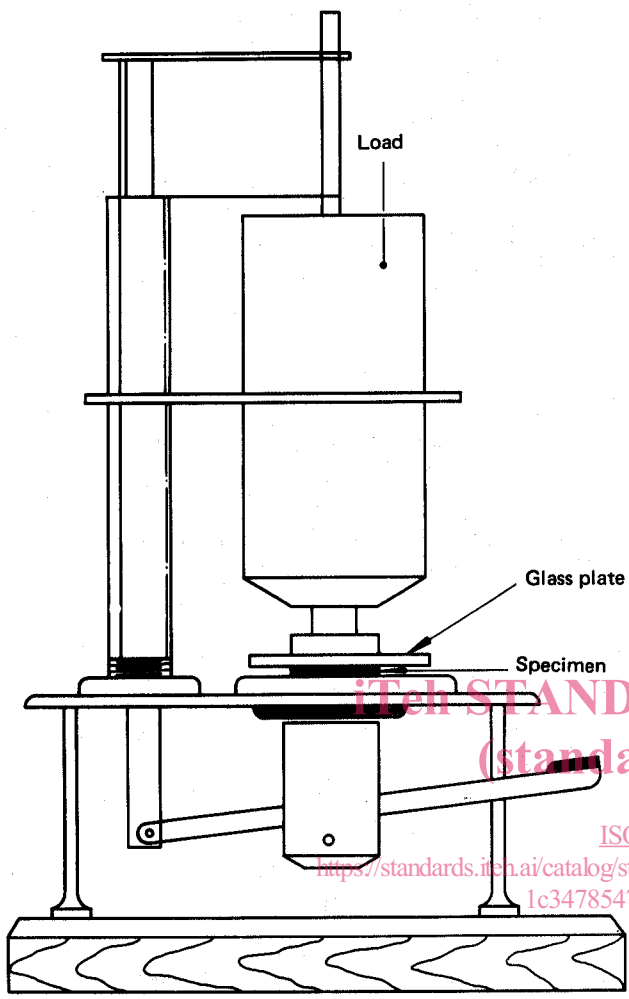

FIGURE 1 — **Loading device for use in consistency determination**

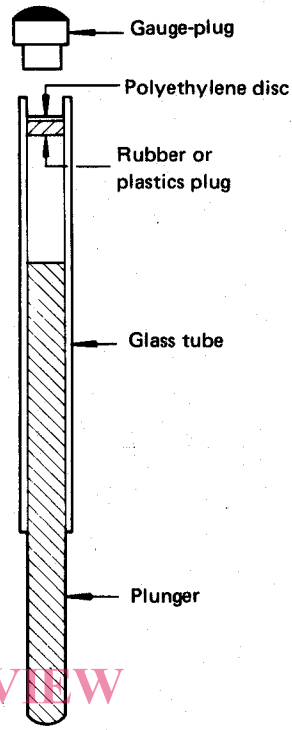

FIGURE 2 — **Measuring device for use in consistency determination**

Dimensions in millimetres

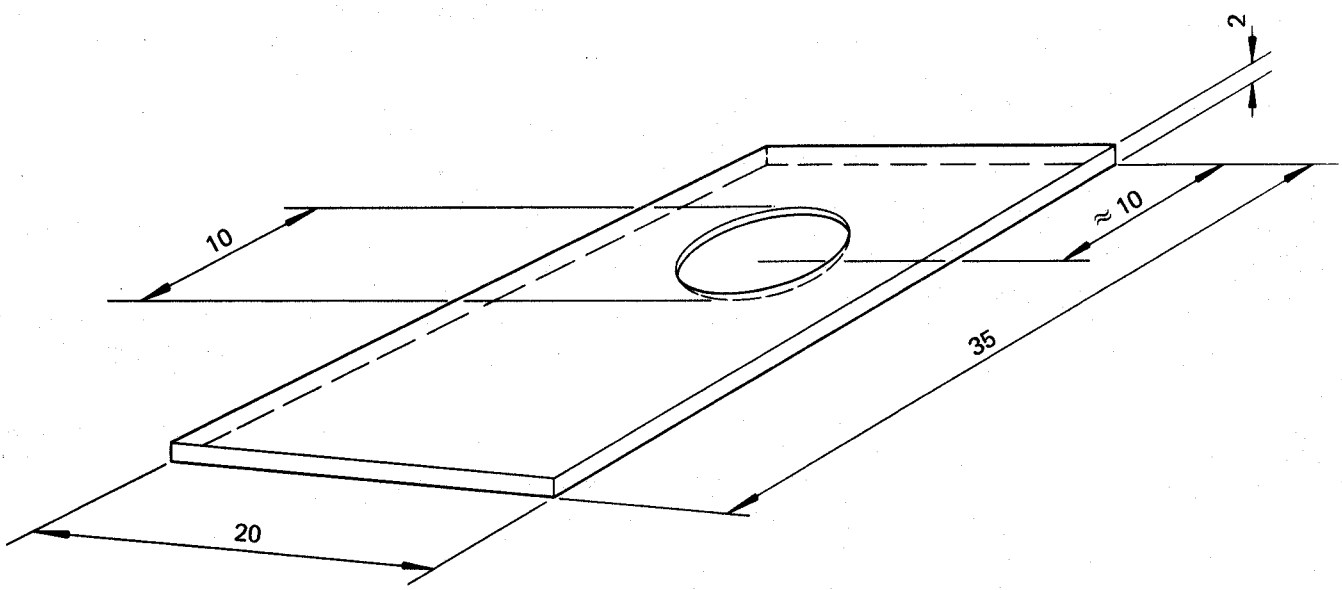

FIGURE 3 — **Mould for use in determination of setting time**

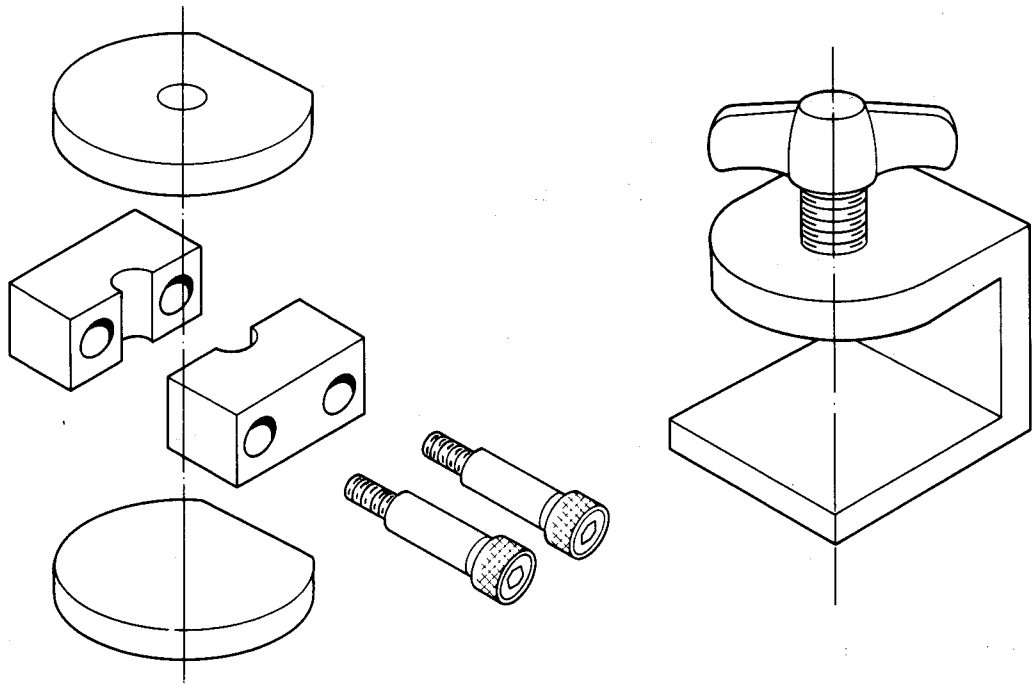

FIGURE 4 – Mould and clamp for preparation of compressive strength test specimens

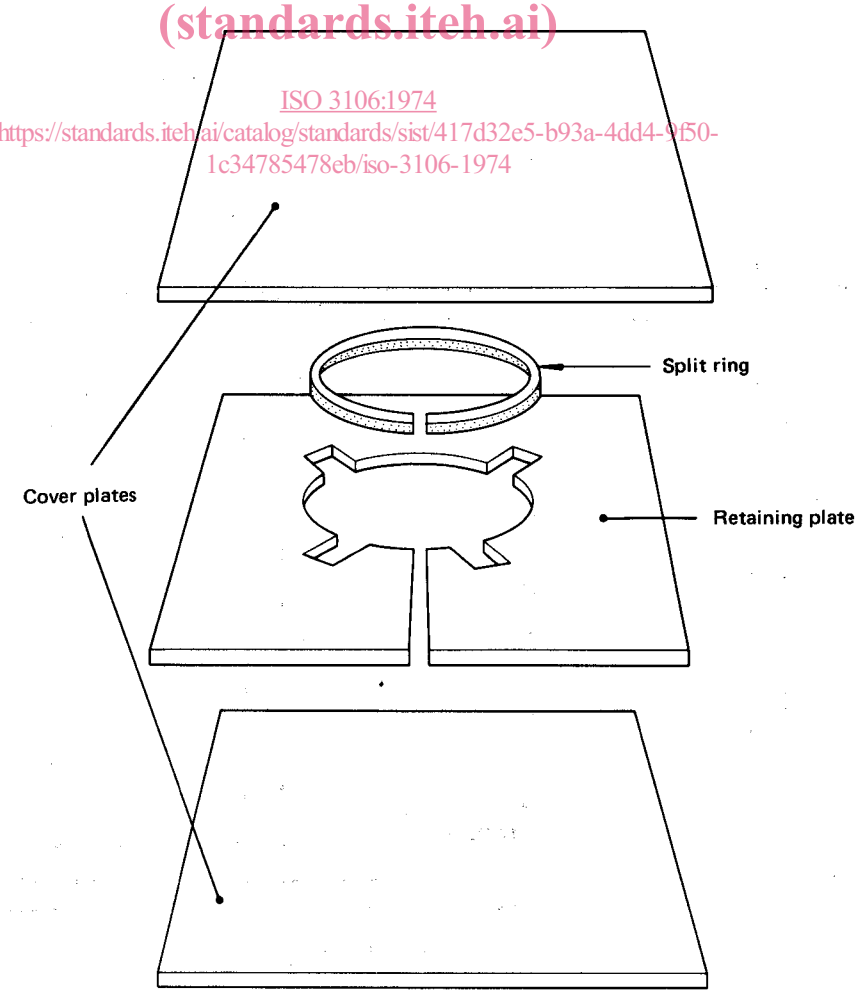

FIGURE 5 – Mould for preparation of test specimen used in solubility determination