International Standard 7489

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

Dental glass polyalkenoate cements

Ciments dentaires au polyalkénoate de verre

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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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 7489 was prepared by Technical Committee ISO/TC 106, *Dentistry*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Dental glass polyalkenoate cements

0 Introduction

0.1 In preparing this International Standard particular consideration was given to the specimen size used for the compressive strength test, specified as a 12 mm high cylinder of 6 mm diameter. This may present problems in preparing specimens of capsulated materials or of certain fast-setting materials, although manufacturers would be able to test the component materials prior to encapsulation. The specimen size specified in ISO 3851 for capsulated silicate and silicophosphate cements is a 6 mm high cylinder of 4 mm diameter. However, in view of the increased scatter of test results using the 6 mm × 4 mm specimen as compared to the 12 mm × 6 mm specimen, and the considerable amount of research work required to set realistic limits for a 6 mm × 4 mm specimen size, it was decided that this International Standard should utilize the 12 mm × 6 mm specimen. It is possible that the use of capsulated materials will increase and a programme of work will be undertaken on the 6 mm × 4 mm specimen to provide data for a future revision incorporating the 6 mm × 4 mm specimen, either for capsulated materials only, or for both hand-mixed and capsulated materials. The same comments apply to specimens for water-leachable material. When suitable test methods have been developed and verified this International Standard will be revised.

0.2 Specific qualitative and quantitative requirements for freedom from biological hazard are not included in this International Standard, but it is recommended that reference should be made to ISO/TR 7405 when assessing possible biological or toxicological hazards.

1 Scope and field of application

This International Standard specifies requirements for dental glass polyalkenoate[1)] cements produced by the reaction between a powder of acid-soluble, aluminosilicate glass and an aqueous solution of a poly(alkenoic acid).

Polyalkenoate[1)] cements prepared by the addition of water to a mixture of dry acid and aluminosilicate glass are also covered by this International Standard.

The glass polyalkenoate cements covered by this International Standard may be used as luting agents, restorative materials, or as pit and fissure fillers or sealers.

2 References

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

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

ISO 3851, *Capsulated dental silicate and silico-phosphate filling materials.*

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

3 Classification

The cements covered by this International Standard are classified, according to their intended use, as follows:

Type 1: Luting agent

Type 2: Restorative material

Materials used to fill or seal pits and fissures may be of either type 1 or type 2.

4 Requirements

4.1 Components

4.1.1 Liquid

The liquid shall be clear and free from visible deposits or filaments on the inside of its container. There shall be no visible signs of gelling.

4.1.2 Powder

The powder shall be free from extraneous material and, if coloured, the pigment shall be uniformly dispersed throughout the powder, when examined visually.

4.2 Unset cement

The cement, prepared and mixed in accordance with 6.1.3, shall be of uniform, smooth texture and shall not evolve gases.

4.3 Colour of set cement

For those cements with a shade guide supplied by the manufacturer the following additional test shall be carried out. After

1) The term "glass polyalkenoate" is now preferred to "glass ionomer" which is deprecated.

immersion in distilled water at 37 ± 1 °C for 7 days, the colour of the set cement, viewed when under water and in natural daylight, shall match the manufacturer's shade guide.

4.4 Physical requirements

The film thickness, setting time, working time, compressive strength, water-leachable content, opacity, acid-soluble arsenic content, and lead content shall be as specified in the table, when tested in accordance with the appropriate test methods specified in clause 6.

4.5 Biocompatibility

See 0.2 for guidance on biocompatibility.

4.6 Manufacturer's instructions

Instructions for the preparation, mixing and manipulation shall accompany each container of liquid and shall include the following:

a) the recommended temperature range for preparation; condition and type of both the slab and spatula, or the type of mixing machine;

b) the optimum powder/liquid ratios over the recommended temperature range [see also 4.6 g)] — this requirement however shall not apply to capsulated materials for which it is inappropriate;

c) the method of mixing and the time of mixing and, in the case of hand-mixed materials, the rate of incorporation of the powder;

d) the manipulation time after completion of mixing;

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

f) for materials where the polyacid is present in aqueous solution, a recommendation that the liquid should be kept in a moisture-tight container to avoid contamination or loss of moisture;

g) the precise powder/liquid ratio on a mass basis to an accuracy of 0,1, at a temperature of 23 ± 1 °C and a relative humidity of (50 ± 5) %, to be used when it is desired to carry out tests on the material;

h) a technique for protecting the cement against early contamination by water.

5 Sampling

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

6 Test methods

6.1 Preparation of test specimens

6.1.1 Conditioning

Prepare the test specimens at a temperature of 23 ± 1 °C and a relative humidity of (50 ± 5) %.

6.1.2 Apparatus

6.1.2.1 Polished glass mixing slab, approximately 150 mm long × 75 mm wide × 20 mm thick.

6.1.2.2 Spatula, made of a material which will not react with, or be abraded by, the components.

NOTE — Apparatus used for mixing and testing should be kept clean, dry and free from hardened particles of cement.

6.1.3 Method of mixing

Completely mix the powder and liquid to a uniform, smooth texture in accordance with the manufacture's instructions (see 4.6).

Table — Physical and chemical properties

Type	Film thickness	Setting time	Working time	Compressive strength	Water-leachable content	Opacity		Acid-soluble arsenic content	Lead content
	µm	min	min	MPa	% (*m*/*m*)	$C_{0,70}$ value		mg/kg (ppm)	mg/kg (ppm)
	max.	max.	min.	min.	max.	min.	max.	max.	max.
1	25	7,5	2,0	65	1,0	—	—	2,0	50
2	—	5	1,75	125	0,7	0,35	0,90	2,0	50

6.2 Determination of film thickness (for type 1 cements only)

6.2.1 Apparatus

6.2.1.1 Two optically flat, square or circular glass plates, having a contact surface area of approximately 200 mm^2 and of a uniform thickness of not less than 5 mm.

6.2.1.2 Loading device, of the type shown in figure 1, generating a force of 147 N obtained by using a mass of 15 kg. The bottom surface of the rod supporting the load shall be horizontal and parallel to the base and large enough to cover one of the glass plates. The loading device shall be capable of applying the load smoothly and with no rotational motion. The glass plates shall be held on the base by guides to prevent movement when the load is applied.

6.2.1.3 Micrometer, accurate to 1 µm.

6.2.2 Procedure

Measure the thickness of the two optically flat glass plates (6.2.1.1) stacked in contact to an accuracy of ± 0,1 µm (reading A).

Place a small quantity of mixed cement on the centre of one of the glass plates and place the plate in the guides. Place the other glass plate centrally on the first plate.

Two minutes after the start of mixing, carefully apply a force of 147 N vertically on the top plate and leave for 7 min. Ensure that the cement completely fills the space between the two glass plates.

Ten minutes after the start of mixing, remove the force that has been applied, lift out the two glass plates containing the adhering cement from the guides and measure the overall thickness of the assembled glass plates and cement film (reading B).

Calculate the thickness of the film as the difference between reading B and reading A. Record the mean result of three such tests to the nearest 1 µm.

6.3 Determination of setting time

6.3.1 Apparatus

6.3.1.1 Oven or cabinet, capable of being maintained at a temperature of 37 ± 1 °C and a relative humidity of at least 90 %.

6.3.1.2 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 mm and the needle end shall be plane and perpendicular to the long axis of the needle.

6.3.1.3 Mould, as shown in figure 2, made of non-corrodible metal.

6.3.1.4 Metal block, of minimum dimensions 8 mm × 75 mm × 100 mm.

6.3.1.5 Non-reactive aluminium foil.

6.3.2 Procedure

Place the mould (6.3.1.3), conditioned to 23 ± 1 °C, on the aluminium foil (6.3.1.5) and fill to a level surface with mixed cement.

Two minutes after the start of mixing, place the assembly, comprising mould, foil and cement specimen, on the block (6.3.1.4), conditioned to 37 ± 1 °C, in the oven (6.3.1.1). Ensure good contact between the mould, foil and block.

Two and a half minutes after the start of mixing, carefully lower the indentor (6.3.1.2) vertically onto the surface of the cement and allow it 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 30 s before the approximate setting time, making indentations at 10 s intervals.

Record the setting time as the time which elapses between the start of mixing to the time when the needle fails to make a complete circular indentation in the cement.

Take the mean of three such tests, rounded to the nearest 10 s, as the result.

6.4 Determination of working time

6.4.1 Apparatus

6.4.1.1 Indentor, of mass 28 ± 0,25 g and having a flat end of diameter 2,0 ± 0,05 mm. The needle tip shall be cylindrical for a distance of approximately 5 mm and the needle end shall be plane and perpendicular to the long axis of the needle.

6.4.1.2 Mould, as shown in figure 2, made of non-corrodible metal.

6.4.1.3 Metal block, of minimum dimensions 8 mm × 75 mm × 100 mm.

6.4.1.4 Non-reactive aluminium foil.

6.4.2 Procedure

Place the mould (6.4.1.2), conditioned to 23 ± 1 °C, on the aluminium foil (6.4.1.4) and fill to a level surface with mixed cement.

One minute after completion of mixing, place the assembly, comprising mould, foil and cement specimen on the block, conditioned to 23 ± 1 °C. Ensure good contact between mould, foil and block.

Two minutes after the start of mixing, carefully lower the indentor (6.4.1.1) vertically onto the surface of the cement and allow it to remain there for 5 s. Repeat at 10 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.

Record the working time as the time which elapses between the start of mixing to the time when the needle fails to make a complete circular indentation in the cement.

Take the mean of three such tests, rounded to the nearest 10 s, as the result.

6.5 Determination of compressive strength

6.5.1 Apparatus

6.5.1.1 Oven or **cabinet,** capable of being maintained at a temperature of 37 ± 1 °C and a relative humidity of at least 30 %.

6.5.1.2 Split moulds and **plates,** as shown in figure 3, made of stainless steel or other suitable material that will not be attacked or corroded by the cement. The internal dimensions of the mould shall be 12 ± 0,1 mm high and 6 ± 0,1 mm in diameter.

6.5.1.3 Individual screw clamps, as shown in figure 3.

6.5.1.4 Compressive strength-testing apparatus, having a cross-head speed of 1,0 mm/min.

6.5.2 Preparation of test specimens

Prepare five specimens.

Condition the moulds, top and bottom plates (6.5.1.2), and the screw clamps (6.5.1.3) to 23 ± 1 °C.

NOTE — To facilitate the removal of the hardened cement specimen, the internal surface of the mould and plates 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 polytetrafluoroethylene (PTFE) dry film lubricant may be used.

Pack the mixed cement to a slight excess into the assembled split mould within 2 min of the start of mixing.

NOTE — In order to consolidate the cement and to 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 and place on the bottom plate with the application of slight pressure.

Remove any bulk extruded cement, place the top plate in position and manually squeeze together. Put the mould and plates in the clamp (6.5.1.3) and screw tightly together. Not later than 3 min after start of mixing, transfer the whole assembly to the oven (6.5.1.1).

Remove the plates 60 ± 5 min after the start of mixing and prepare the surface of the ends of the specimen plane, at right angles to its long axis, by grinding the ends flat and removing 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 grinding and rotate the specimen by a one-quarter turn every few strokes.

Remove the specimen from the mould immediately after surfacing and rapidly check for air-voids or chipped edges. Discard any defective specimens.

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

6.5.3 Procedure

Test five specimens.

Calculate the diameter by taking the mean of four measurements, two at each end of the specimen at right angles to each other, to an accuracy of ± 0,01 mm. Twenty-four hours after the start of mixing, determine the compressive strength of the test specimens using the apparatus having a cross-head speed of 1,0 mm/min (6.5.1.4).

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

Place a small disc of damp filter paper between each end of the specimen and the jaws of the testing machine in order to reduce scatter of results arising from surface roughness of the ends of the specimen.

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

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

where

F is the maximum applied load, in newtons;

d is the measured mean diameter of the specimen, in millimetres.

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

6.6 Determination of water-leachable content

6.6.1 Apparatus

6.6.1.1 Oven or **cabinet,** capable of being maintained at a temperature of 37 ± 1 °C and a relative humidity of at least 30 %.

6.6.1.2 Mould, consisting of a split brass or stainless steel ring contained in a former or retaining ring as shown in figure 4. The height of the ring shall be 1,5 ± 0,3 mm and the internal diameter 20 ± 1 mm.

6.6.1.3 Individual screw clamps.

6.6.1.4 Platinum wire or, alternatively, **waxed dental floss** or other non-corrodible material.

6.6.1.5 Three wide-mouthed, tared, stoppered glass weighing bottles, as shown in figure 5.

6.6.2 Preparation of test specimens

Prepare four specimens.

Place the mould (6.6.1.2) on a thin polyethylene or cellulose acetate sheet backed by a flat plate.

Insert a convenient tared length of platinum wire (6.6.1.4) through the split ring so that at least 10 mm projects into the ring.

NOTE — A release agent, such as polytetrafluoroethylene dry film lubricant, may be applied to the split ring to facilitate removal of the specimen.

Fill the split ring with mixed cement.

Cover with a plate faced with a sheet of polyethylene or cellulose acetate, press firmly together and apply the screw clamp (6.6.1.3)

Three minutes after the start of mixing, place the mould, plates and the screw clamp in the oven (6.6.1.1) 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 platinum wire from the split ring. Remove any surplus cement from the edge of the disc and lightly brush the surface to remove any loose material.

NOTE — Due to the comparatively brittle nature of the cement at this stage, it is advisable to clean the excess of cement from the surface of the ring before attempting to remove the specimen.

6.6.3 Preparation of test solution

For each pair of specimens, use a clean weighing bottle (6.6.1.5) together with a third bottle for a blank estimation to be carried out simultaneously. Dry the bottles at 150 ± 5 °C for at least 2 h. Cool the bottles for 1 h at room temperature in a desiccator containing thoroughly dry anhydrous calcium sulfate or active silica gel, and weigh to 0,1 mg (mass m_2). During these operations, handle the bottles as little as possible to prevent contamination.

Place two specimens immediately after preparation in each bottle except the blank bottle, and weigh the whole (mass m_3). The mass of each pair of specimens is given by the following formula:

$$m_3 - (m_2 + m_1)$$

where m_1 is the sum of the masses of the platinum wires.

Immediately submerge the two discs by pouring 50 ml of distilled water into the bottle and suspending the specimens by the wire, so that they neither touch each other nor rest against the side of the bottle. Close the bottle as tightly as possible and store for 23 h at 37 ± 1 °C. Place 50 ml of the same water in the blank bottle and store in the oven containing the specimens.

After 23 h immersion, remove the specimens from the water and evaporate the water from the specimen bottle and from the blank bottle at a temperature just below 100 °C and dry the bottles for 24 h at 150 ± 5 °C. Cool and weigh the bottles as earlier directed for weighing when empty. The mass of the specimen bottle, in each case, is mass m_4, and the increase in mass of the blank bottle is mass m_5.

6.6.4 Expression of results

Express the water-leachable content, S, for each pair of specimens as a percentage by mass, using the following equation:

$$S = \frac{m_4 - (m_5 + m_2)}{m_3 - (m_2 + m_1)} \times 100$$

Calculate the water-leachable content as the average of duplicate test results (i.e. two weighing bottles each containing two specimens) to the nearest 0,1 %. If one of the results is above the limit given in the table, repeat the test; discard the highest and lowest results and calculate the mean of the two remaining results to the nearest 0,1 %.

6.7 Determination of opacity (for type 2 cements only)

6.7.1 Apparatus

6.7.1.1 Oven or **cabinet,** capable of being maintained at a temperature of 37 ± 1 °C and a relative humidity of at least 30 %.

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

NOTE — The contrast ratio $C_{0,70}$ is the ratio between the light reflected by the specimen on a black background, and the light reflected by the specimen on a white background which has a reflectance of 70 %.

6.7.1.3 A sheet of white waterproof material, approximately 110 mm × 40 mm, marked, along its entire length, with black stripes 2 mm wide and 3 mm apart.

6.7.1.4 Mould, consisting of a split brass or stainless steel ring contained in a former as shown in figure 4. The height of the ring shall be 1,00 ± 0,03 mm and the internal diameter 30 ± 1 mm.

6.7.1.5 Flat glass plates, approximately 35 mm × 35 mm and 5 mm thick, and two polytetrafluoroethylene or cellulose acetate sheets 35 mm × 35 mm.

6.7.1.6 Individual screw clamps.

6.7.2 Preparation of test specimens

Using the mould (6.7.1.4) as described in 6.6.2, clamp a sufficient amount of mixed cement between the two polytetrafluorethylene or cellulose acetate sheets and two flat glass plates (6.7.1.5) to form a disc of approximately 30 mm in diameter and 1 ± 0,025 mm thick. Three minutes from the start of mixing, place the assembly in the oven (6.7.1.1). After 1 h, remove the specimen from the plates and store for 7 days in distilled water at 37 ± 1 °C.

6.7.3 Procedure

Compare the opacity of the cement specimen with that of the two opal glass standards (6.7.1.2) having $C_{0,70}$ values of 0,35 and 0,90 respectively, by placing the specimen and standards against the variegated black and white background. During observations, cover the 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 is deemed to meet the requirement of the table.

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 Determination of acid-soluble arsenic content

6.8.1 Preparation of sample

Powder the set cement, and sieve through a 75 µm (200 mesh) sieve. Disperse 2 ± 0,01 g of the sieved powder in 30 ± 0,5 ml of water and add 10 ± 0,01 ml of hydrochloric acid 36 % (m/m) (ϱ = 1,18 g/ml). Maintain the mixture at 37 ± 1 °C for 1 h, then filter the solution and use it in the test for arsenic content.

6.8.2 Procedure

Determine the arsenic content by the method described in ISO 2590.

6.9 Determination of acid-soluble lead content

6.9.1 Reagent

Hydrochloric acid, 20 % (m/m), prepared by diluting lead-free hydrochloric acid 36 % (m/m) (ϱ = 1,18 g/ml) with distilled water.

6.9.2 Preparation of sample

Mix sufficient powder and liquid to give 2 g of cement. Place the mixed cement in a clean plastic bag and seal the bag. Flatten the cement in the bag, using finger pressure to produce a very thin disc. Place the disc in an oven at 37 ± 1°C for 24 ± 1 h. Remove the disc of set cement and crush to a fine powder with an agate pestle and mortar. Accurately weigh about 2 ± 0,01 g of the powdered cement and transfer to a 150 ml conical flask. Add 50 ± 0,5 ml of the 20 % hydrochloric acid. Stopper the flask, shake and allow to stand for 16 h.

Pour the solution into a centrifuge tube, and centrifuge for 10 min. Using a pipette, transfer the clear solution into a sample container and stopper it.

6.9.3 Procedure

Determine the lead content directly by atomic absorption spectroscopy.

7 Packaging and marking

7.1 Packaging

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

NOTE — For the purpose of this International Standard, the container is considered to be the immediate wrapping of the component.

7.2 Marking of containers

Each container shall be clearly marked with the following particulars:

a) the name and/or trade mark of the manufacturer, and the 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 millimetres, of the liquid;

d) the date of manufacture, expressed in all-numeric form in accordance with ISO 2014;

e) the batch number;

f) the number of this International Standard, i.e. ISO 7489.

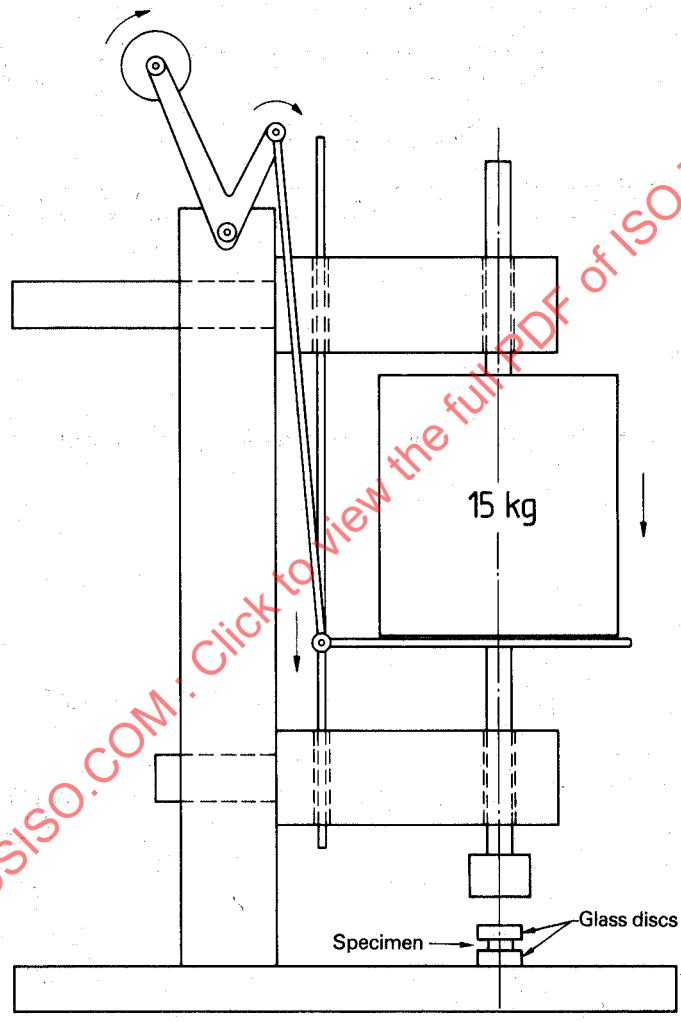

Figure 1 — Loading device for film thickness test