INTERNATIONAL STANDARD

ISO 9917

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Dental water-based cements

Ciments dentaires à base d'eau

Reference number
ISO 9917:1991(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 9917 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Sub-Committee SC 1, *Filling and restorative materials*.

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Introduction

This International Standard has been prepared in order to present the requirements and test methods for all types of water-based dental cements in one document. It supersedes the previously published standards for individual cements, shown below, which will therefore be withdrawn:

ISO 1565:1978, *Dental silicate cement (hand mixed)*.

ISO 1566:1978, *Dental zinc phosphate cement*.

ISO 3824:1984, *Dental silicophosphate cement (hand-mixed)*.

ISO 3851:1977, *Capsulated dental silicate and silico-phosphate filling materials*.

ISO 4104:1984, *Dental zinc polycarboxylate cements*.

ISO 7489:1986, *Dental glass polyalkenoate cements*.

Specific qualitative and quantitative requirements for freedom from biological hazard are not included in this International Standard but it is recommended that, in assessing possible biological or toxicological hazards, reference should be made to ISO/TR 7405:1984, *Biological evaluation of dental materials*, or any more recent edition.

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Dental water-based cements

1 Scope

This International Standard specifies requirements for the following types of dental cements, including both hand-mixed and capsulated cements for mechanical mixing, which are intended for permanent cementation, lining and restoration, and effect setting only by an aqueous acid-base reaction.

Silicate cements based on the reaction between an alumino-silicate glass powder and an aqueous solution of phosphoric acid which may contain metal ions. They are used for the aesthetic restoration of anterior teeth.

Zinc phosphate cements based on the reaction between an oxide powder (the principal constituent of which is zinc oxide) and an aqueous solution of phosphoric acid which may contain metal ions. They are used as luting agents to seal dental appliances to hard oral structures or to other appliances. They can also be used as a base for restorative materials and as a temporary restorative material by increasing the ratio of powder to liquid relative to that used for luting.

Silicophosphate cements based on the reaction between a powder of acid-soluble aluminosilicate glass and metal oxides (principally zinc oxide) and an aqueous solution of phosphoric acid which may contain metal ions. They are used as temporary restorative materials or as luting agents to seal dental appliances to hard oral structures by varying the ratio of powder to liquid.

Zinc polycarboxylate cements based on the reaction between zinc oxide and aqueous solutions of polyacrylic acid or similar polycarboxylic compounds, or zinc oxide/polycarboxylic acid powders which are mixed with water. They are used as luting agents to seal appliances to hard oral structures or to other appliances, or as a base for restorative materials or as temporary restorative materials.

Glass polyalkenoate cements based on the reaction between an aluminosilicate glass powder and an aqueous solution of an alkenoic acid, or between an aluminosilicate glass/polyacid powder blend and water, or an aqueous solution of tartaric acid. These translucent cements are for use for the aesthetic restoration of teeth, as luting agents, as bases or liners and for restoring pits and fissures.

Glass polyalkenoate cements in which the glass and a metal have been fused together and which are intended for the restoration of teeth are also included.

This International Standard specifies limits for each of the properties according to whether the cement is intended for as a luting agent, a restorative material, a base or a liner.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 2590:1973, *General method for the determination of arsenic — Silver diethyldithiocarbamate photometric method.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 7491:1985, *Dental materials — Determination of colour stability of dental polymeric materials.*

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 mixing time: That part of the working time required in order to obtain a satisfactory mix of the components.

3.2 working time: Period of time, measured from the start of mixing, during which it is possible to manipulate a dental material without an adverse effect on its properties.

3.3 net setting time: Period of time measured from the end of mixing until the material has set according to the criteria and conditions described in 7.3. For the purposes of this International Standard, in view of the wide variation in mixing times of the cements, the net setting time is determined from the end of mixing.

4 Classification

4.1 Chemical type

For the purposes of this International Standard, dental cements are classified on the basis of their chemical composition, as follows.

4.1.1 Silicate cement.

4.1.2 Zinc phosphate cement.

4.1.3 Silicophosphate cement.

4.1.4 Zinc polycarboxylate cement.

4.1.5 Glass polyalkenoate cement.

4.2 Application

For the purposes of this International Standard, materials are classified on the basis of their usage, as follows.

4.2.1 Luting cements.

4.2.2 Bases and liners.

4.2.3 Restorative cements.

5 Requirements

5.1 Material

The cement shall consist of a powder and liquid which, when mixed according to the manufacturer's instructions, will comply with the requirements of this International Standard.

5.2 Components

5.2.1 Liquid

When tested according to 7.1.2, the liquid shall be free from deposits or filaments on the inside of its container. There shall be no visible signs of gelation.

5.2.2 Powder

When tested according to 7.1.2, the powder shall be free from extraneous material. If the powder is coloured, the pigment shall be uniformly dispersed throughout the powder.

5.3 Unset cement

The cement when mixed as directed in 7.1.3, and tested according to 7.1.2, should be homogenous and of a smooth consistency.

5.4 Optical properties

When prepared, stored and tested in the manner described in 7.6,

a) the opacity of the set cement shall be within the limits set out in table 1. Where a restorative cement is stated by the manufacturer not to be translucent, the opacity requirement shall not apply;

b) the colour of the set cement shall match the manufacturer's shade guide or correspond to the manufacturer's description when no shade guide is supplied.

5.5 Performance requirements

The cement shall comply with the relevant requirements specified in table 1, when tested in accordance with the test methods given in 7.2 to 7.6.

5.6 Acid-soluble arsenic content

When tested according to 7.7, the acid-soluble arsenic content shall not exceed the limits given in table 1.

5.7 Acid-soluble lead content

When tested according to 7.7, the acid-soluble lead content shall not exceed the limits given in table 1.

5.8 Biocompatibility

See the Introduction for guidance on biocompatibility.

Table 1 — Requirements of dental cements

Chemical type	Application	Film thickness	Net setting time min		Compressive strength	Acid erosion	Opacity $C_{0,70}$		Acid-soluble As content	Acid-soluble Pb content
		max. µm	min.	max.	min. MPa	max. mm/h	min.	max.	mg/kg	mg/kg
Zinc phosphate	luting	25	2,5	8	70	0,1	—	—	2	100
Zinc polycarboxylate	luting	25	2,5	8	70	2,0	—	—	2	100
Glass polyalkenoate	luting	25	2,5	8	70	0,05	—	—	2	100
Zinc phosphate	bases/liners	—	2	6	70	0,1	—	—	2	100
Zinc polycarboxylate	bases/liners	—	2	6	70	2,0	—	—	2	100
Glass polyalkenoate	bases/liners	—	2	6	70	0,05	—	—	2	100
Silicate	restorative	—	2	6	170	0,05	0,35	0,55	2	100
Silicophosphate	restorative	—	2	6	170	0,05	0,35	0,90	2	100
Glass polyalkenoate	restorative	—	2	6	130	0,05	0,35	0,90	2	100

6 Sampling

6.1 Hand-mixed cements

A sample drawn from one batch shall provide sufficient material to complete all the prescribed tests and any necessary repeats, and shall consist of a minimum of 50 g of powder and a corresponding volume of liquid, where required.

6.2 Capsulated cements

A test sample shall comprise a retail package of 100 or more capsules.

7 Test methods

7.1 Preparation of test specimens

7.1.1 Ambient conditions

All specimens shall be prepared at a temperature of (23 ± 1) °C and a relative humidity of (50 ± 5) %.

7.1.2 Inspection requirements

Visual inspection shall be used in determining compliance with 5.2, 5.3, 5.4 and clause 8.

7.1.3 Method of mixing

The cement shall be prepared according to the manufacturer's instructions. Sufficient cement shall be mixed to ensure that the preparation of each specimen is completed from one mix. A fresh mix shall be prepared for each specimen.

NOTE 1 For encapsulated materials, more than one capsule, simultaneously mixed, may be required for certain specimens.

7.2 Film thickness (for luting cements only)

7.2.1 Apparatus

7.2.1.1 Two optically flat, square or circular, glass plates having a contact surface area of 200 mm^2 ± 25 mm^2. Each plate shall be of a uniform thickness of not less than 5 mm.

7.2.1.2 Loading device of the type illustrated in figure 1, or an equivalent means, whereby a force of 150 N ± 2 N may be generated vertically to the specimen via the upper glass plate.

In figure 1, the anvil which is attached to the bottom of the rod carrying the load shall be horizontal and parallel to the base. The load shall be applied smoothly and in such a manner that no rotation occurs.

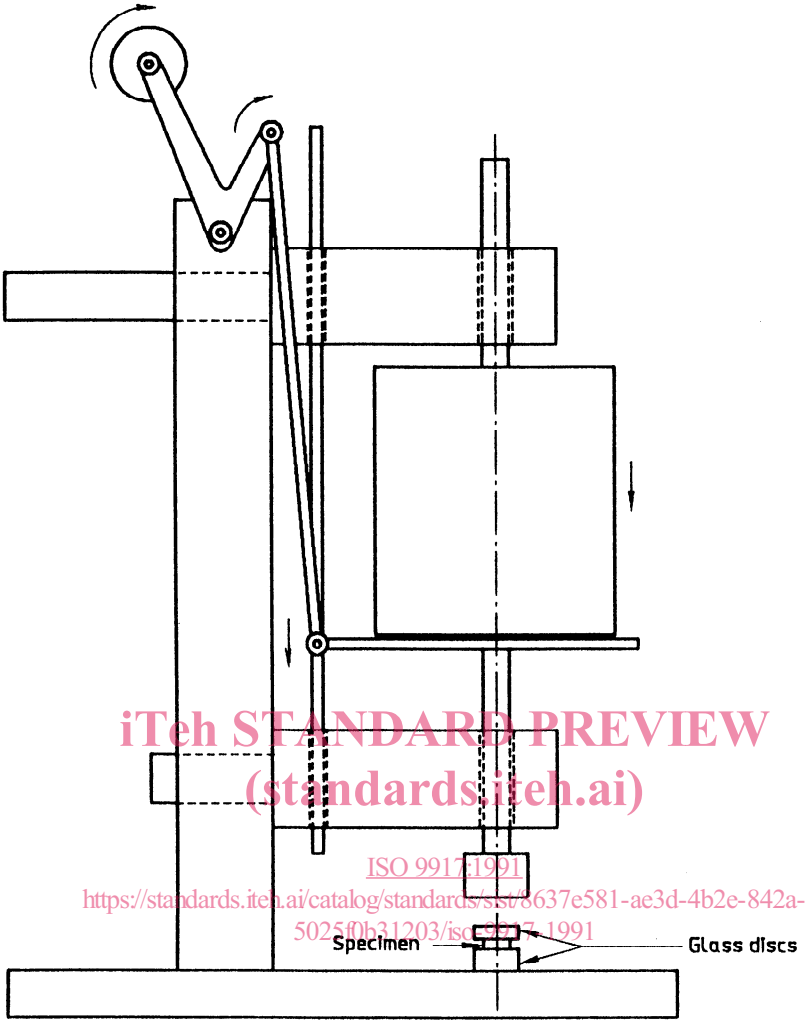


Figure 1 — Loading device for use in film thickness test

7.2.1.3 Micrometer or equivalent measuring instrument, accurate to 1,25 µm.

7.2.2 Procedure

Measure to an accuracy of ± 1,25 µm the combined thickness of the two optically flat glass plates (7.2.1.1) stacked in contact (reading *A*). Remove the upper plate and place 0,1 ml ± 0,05 ml of the mixed cement in the centre of the lower plate and place this centrally below the loading device (7.2.1.2) on the lower platen. Replace the second glass plate centrally on the cement in the same orientation as in the original measurement.

10 s before the end of the manufacturer's stated working time, carefully generate a force of 150 N ± 2 N vertically and centrally to the specimen via the top plate. Ensure that the cement has completely filled the space between the glass plates. When at least 10 min have elapsed after the application of the load, remove the plates from the loading device and measure the combined thickness of the two glass plates and the cement film (reading *B*).

Record the difference in thickness of the plates with and without the cement film (reading *B* — reading *A*) as the thickness of the film. Repeat the test four times.

7.2.3 Treatment of results

At least four of the five results shall be below 25 µm for the material to pass the requirement included in table 1. If only two or less results are below 25 µm, then the material fails the requirement. If three results are below 25 µm, test a further five

specimens. To comply with the requirement, all the specimens in the second series shall be below 25 µm.

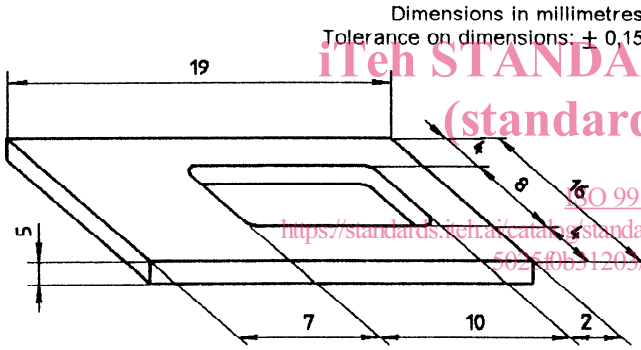
7.3 Net setting time

7.3.1 Apparatus

7.3.1.1 Cabinet capable of being maintained at a temperature of (37 ± 1) °C and a relative humidity of at least 90 %.

7.3.1.2 Indentor of mass 400 g ± 5 g and having a flat end of diameter 1 mm ± 0,1 mm. The needle tip shall be cylindrical for approximately 5 mm. The needle end shall be plane and perpendicular to the long axis of the needle.

7.3.1.3 Metal mould similar to that shown in figure 2.



NOTE — Internal corners may be rounded.

Figure 2 — Mould for preparation of specimens for determination of net setting time

7.3.1.4 Metal block of minimum dimensions 8 mm × 75 mm × 100 mm positioned within the cabinet and maintained at (37 ± 1) °C.

7.3.1.5 Aluminium foil.

7.3.1.6 Timer accurate to ± 1 s.

7.3.2 Procedure

Place the mould (7.3.1.3), conditioned to (23 ± 1) °C, on the aluminium foil (7.3.1.5) and fill to a level surface with mixed cement.

60 s after end of mixing, place the assembly, comprising mould, foil and cement specimen, on the block (7.3.1.4), in the cabinet (7.3.1.1). Ensure good contact between the mould, foil and block.

90 s after end of mixing, carefully lower the indentor (7.3.1.2) 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 × 2 magnification. Clean the needle, if necessary, between indentations. Repeat the process, starting the indentation at 30 s before the approximate setting time, making indentations at 10 s intervals.

Record the net setting time as the time elapsed between the end of mixing to the time when the needle fails to make a complete circular indentation in the cement. Repeat the test three times.

7.3.3 Treatment of results

Record the results of the three tests. Each result shall fall within the range specified in table 1 for the material to fulfil the requirement.

7.4 Compressive strength

7.4.1 Apparatus

7.4.1.1 Cabinet maintained at a temperature of (37 ± 1) °C and a relative humidity of at least 30 %.

7.4.1.2 Split mould and plates, as shown in figure 3. The mould shall have internal dimensions 6 mm ± 0,1 mm high and 4 mm ± 0,1 mm diameter. The mould and plates shall be made of stainless steel or other suitable material that will not be affected by the cement. To prevent the adhesion of polyacrylic acid-based cements, in these cases the plates should be faced with cellulose acetate sheets.

7.4.1.3 Screw clamps, as shown in figure 3.

7.4.1.4 Mechanical tester, which is capable of being operated at a cross-head speed of 0,75 mm/min ± 0,30 mm/min or at a loading rate of 50 N/min ± 16 N/min.

Dimensions in millimetres

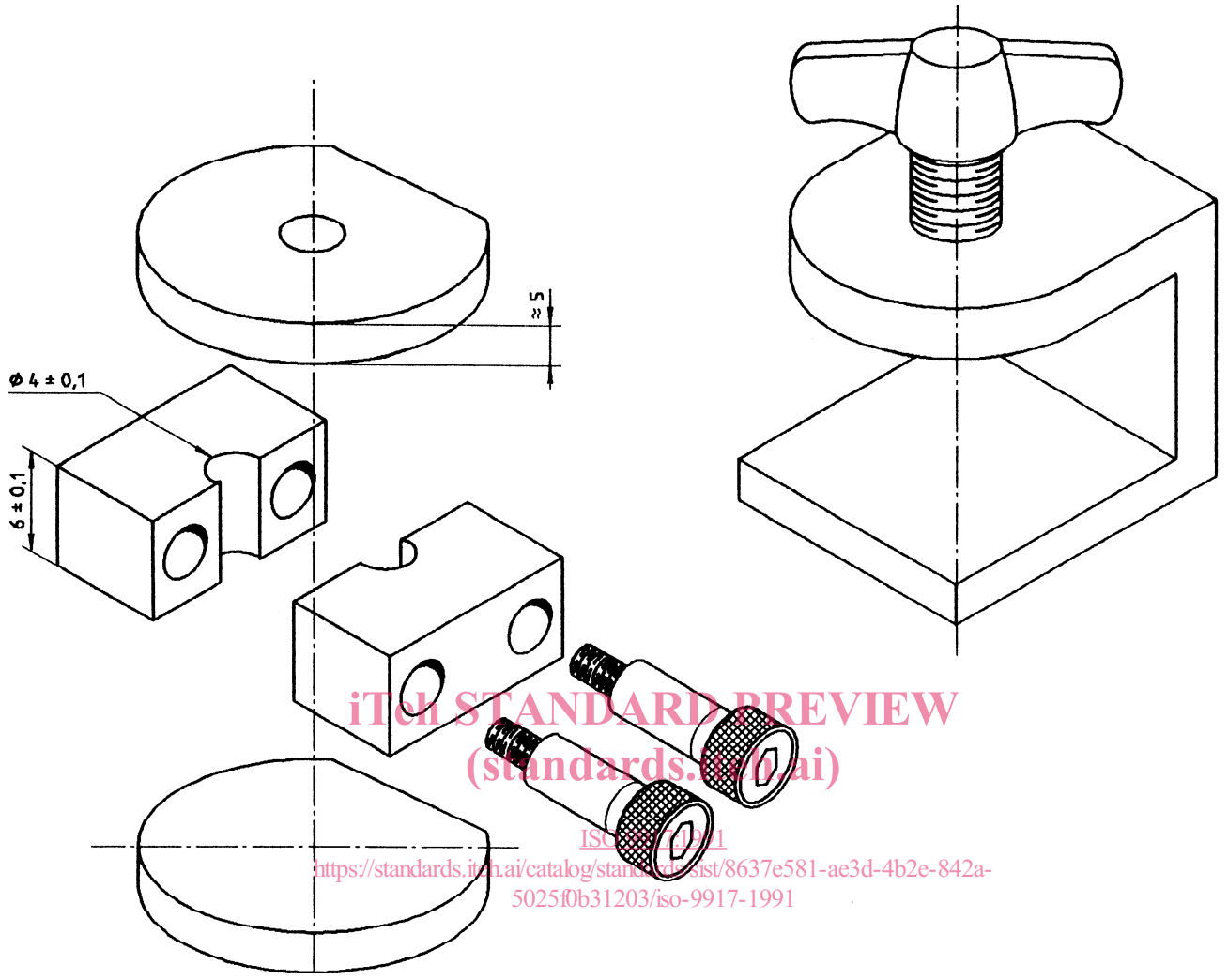


Figure 3 — Mould and clamp for preparation of specimens for compressive strength test

7.4.2 Preparation of test specimens

Condition the split mould plates (7.4.1.2) and screw clamp (7.4.1.3) at (23 ± 1) °C. Within 60 s of the end of mixing, pack the cement, prepared according to the manufacturer's instructions, to a slight excess into the split mould.

In order to consolidate the cement and avoid trapping air, 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 on the mould and squeeze together. Put the mould and plates in the screw clamp and tighten. Not later than 120 s after the end of mixing, transfer the whole assembly to the cabinet (7.4.1.1).

One hour after the end of mixing, remove the plates and grind the ends of the specimen flat and at right angles to its long axis. An acceptable method for doing this is to use wet 400 grade silicon carbide paper, but in any event the abrasive should be no coarser.

Remove the specimen from the mould immediately after surfacing and check visually without magnification for air-voids or chipped edges. Discard any such defective specimens.

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 petroleum ether. Alternatively a thin film of silicone grease or PTFE dry film lubricant may be used.

Prepare five such specimens and immediately after the preparation of each, immerse it in water, Grade 3 according to ISO 3696, at (37 ± 1) °C for 23 h ± 0,5 h.

Calculate the diameter of each specimen by taking the mean of two measurements, at right angles to each other, to an accuracy of ± 0,01 mm.

7.4.3 Procedure

Twenty-four hours after the end of mixing, place each specimen with the flat ends between the platens of the mechanical tester (7.4.1.4) and apply a compressive load in the long axis of the specimen.

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

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

where

ρ is the maximum load applied in newtons;

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

7.4.4 Treatment of results

If four of the five results obtained are below the minimum strength specified in the table, the material has failed the test. If at least four of the five results are above the minimum strength specified in the table, the material has passed the test. In other cases, prepare a further 10 specimens.

To pass the test, at least 12 of the total of 15 results shall be above the minimum strength value.

7.5 Acid erosion by impinging jet technique

This test is intended to reflect material quality, and should not be taken as an indication of possible clinical performance.

7.5.1 Apparatus

7.5.1.1 Impinging jet apparatus. The apparatus is designed to maintain a constant jet of liquid onto the surface of the cement specimen and is shown in figure 4. It consists of a constant head device feeding eight separate jets of 1 mm internal diameter with a recirculating pump and a reservoir of approximately 10 l capacity. The flow of liquid from each jet shall be 120 ml/min ± 4 ml/min and this may be adjusted by varying the height of the head. The apparatus is constructed in borosilicate glass with rubber or plastics tubing for the transport of the liquid. The jet assembly may be constructed of other materials if this is more convenient. For example, transparent plastics tubing with Luer fittings, the jets being stainless steel tubing of 1 mm internal diameter with the corresponding Luer attachment. The specimen moulds are of stainless steel with the dimensions as given in figure 5. The stainless steel moulds containing the samples are supported in eight holes in a plastics tray which is clamped in the reservoir in such a manner that each specimen is held exactly 10 mm ± 0,2 mm below the end of its corresponding jet. This is accomplished most easily by firmly clamping the constant head device in a fixed position and mounting the reservoir and specimen assembly on a lifting device for accurate positioning beneath the jets.

7.5.1.2 Micrometer depth gauge, with a precision of ± 0,01 mm and having a needle point with a 1 mm diameter flat end.

7.5.1.3 Cabinet maintained at (37 ± 1) °C.

7.5.1.4 Timer.

7.5.1.5 Mould as illustrated in figure 5.