INTERNATIONAL STANDARD ISO 4049

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Resin-based dental filling materials

Produits d'obturation dentaire à base de résine synthétique

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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.

International Standard ISO 4049 was developed by Technical Committee ISO/TC 106, *Dentistry*, and was circulated to the member bodies in April 1976.

It has been approved by the member bodies of the following countries :

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Norway
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Resin-based dental filling materials

0 INTRODUCTION

A precise definition of a composite filling material, adequate for the exact delineation of this group of materials whilst taking into account the variation in basic chemistry, has yet to be agreed. The title and scope of this International Standard have, therefore, been framed so as not to exclude any materials of the resin-based type, although its contents have been compiled to cover primarily the composite type of filling material described in clause 3. However, in so far as other resin-type materials may be able to satisfy the various performance criteria, it is not intended to exclude coverage of such materials even though at the present time those products available appear unlikely to meet the specified test limits.

As these materials are presented both in preweighed capsule form as well as for hand mixing, the specimens required for use in the test procedures are of a size that can be prepared from the contents of one capsule.

These filling materials present particular problems in specification due to the inherent behaviour of the resin system itself. The scarcity of clinical data concerning their behaviour makes the establishment of certain performance limits especially difficult, while in other instances the sophisticated testing procedures necessary to examine certain properties have raised doubts regarding their applicability in a standards specification. As a result, the Technical Committee responsible for the preparation of this International Standard decided that priority should be given to first completing a basic specification covering the main features of the resin-based materials with a view to possible improvement when further research data and experience in testing become available. Future development in this field will be examined and taken into account when considering revision or amendment of the terms of this International Standard.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the requirements for resin-based dental filling materials presented in a form suitable for mechanical mixing or for hand mixing, and intended primarily for use as direct dental restorations in the oral cavity. It also specifies the test methods to be used to determine compliance with these requirements[1] including a method for defining the resin/filler composite class of material.

Resin-based filling materials may or may not contain fillers. Only those with 50 % or more by mass of filler, determined in accordance with the procedure specified in 5.7, shall be described as composite.

2 REFERENCE

ISO/R 879, *Plastics — Determination of resistance of plastics to colour change upon exposure to light of a xenon lamp.*

3 REQUIREMENTS

3.1 Material

When treated in accordance with the manufacturer's instructions, the material shall set to a condition suitable for its intended dental use.

3.2 Physical and mechanical properties

3.2.1 *General*

If the material is supplied in pre-coloured standard shades, then each shade shall be capable of satisfying the requirements specified in 3.2.2, 3.2.3 and 3.2.4. In the event of the material being supplied for "tinting" or "blending" to the user's prescription, the test requirements shall apply both when the maximum recommended proportion of "tinter" or "blender" (see 3.6) is used and with the base material by itself.

3.2.2 *Minimum working time*

The working time from the commencement of mixing to the commencement of setting, determined in accordance with the procedure specified in 5.3, shall be not less than the mixing time plus 60 s.

3.2.3 *Setting time*

The setting time, measured from the commencement of mixing, and determined in accordance with the procedure specified in 5.4, shall be not more than 8 min.

3.2.4 *Transverse strength*

The transverse strength of the set material, determined in accordance with the procedure specified in 5.5, shall be not less than 50 MPa.

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[1] *With the exception of toxicity — see 3.4.*

3.3 Shade

The colour of the set material shall closely match the manufacturer's shade guide. If a shade guide is not supplied by the manufacturer, samples from two further batches shall be taken for comparison purposes.

3.4 Toxicity

The mixed material, when used in accordance with the manufacturer's instructions, shall neither cause prolonged damage to oral tissues nor have any adverse systemic effect.

NOTE — When specific toxicity tests are available, they will be included in this International Standard.

3.5 Colour stability

3.5.1 *Heat*

The colour of the set material shall not show more than a slight change after 24 h, when tested in accordance with 5.6.1.

3.5.2 *Light*

The colour of the set material shall not show more than a slight change after 24 h, when tested in accordance with 5.6.2.

3.6 Manufacturer's instructions

Detailed instructions shall accompany each individual pack of material and shall include the following details :

a) information on the proportioning of components and method of mixing, including, if relevant, ambient conditions under which it should be carried out;

b) information on the manipulation[1] and setting times of the material, including an indication of the time when a matrix may be removed, if this differs from the setting time;

c) any special precautions regarding manipulation of the material, including, if relevant, the maximum proportion of tinters or blenders which may be used without detriment to the physical properties;

d) information on the need to use a base/liner or other recommended protective measures if the material can cause pulpal irritations;

e) recommended instructions for "finishing";

f) recommended conditions for storage (i.e. need for refrigeration) and shelf life under those conditions of storage (see also clause 6).

4 SAMPLING

The test sample shall consist of a retail package, or packages from the same batch containing sufficient material to carry out the specified tests plus an allowance for the possibility of repeats being necessary.

5 TEST METHODS

Unless stated otherwise, distilled water, or water of equal purity, shall be used in the tests.

5.1 Test specimen — General

5.1.1 *Ambient conditions*

Unless stated otherwise, all test specimens shall be prepared and tested at 23 ± 1 °C. The relative humidity shall be controlled to ensure that it remains greater than 30 % at all times.

5.1.2 *Mixing*

The material shall be mixed in accordance with the manufacturer's instructions, using only the amount necessary to carry out the relevant test.

5.2 Inspection requirements

Visual inspection shall be used in determining compliance with 3.1, 3.3, 3.5, 3.6 and clause 6.

5.3 Working time

5.3.1 *Apparatus*

Oscillating rheometer which records graphically or instrumentally the viscosity changes in the setting material.

A suitable instrument which comprises the following features is illustrated in figure 1.

The main housing (A) is mounted and can rotate in a ball-race (B), which has very little friction. The lower platten (C) is held in the main housing by two grub screws (D) and a knurled screw (E), with upper platten (M) in position on its guide, a distance of 1 mm above. The main housing and the lower platten are driven in an oscillatory motion by means of a 10 min^{-1} electric motor attached to to an eccentric wheel (F) which is connected to arm (G) by means of two balanced and tensioned coil springs (H). The maximum rotary movement of arm (G), when no material is on the platten, is 1° 12′ ± 2′. This rotary movement is measured by means of a transducer (I), the output from which is fed either to a centre-zero microammeter or to a suitable automatic recording apparatus.

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[1] The term "manipulation time" is intended to refer to the time the manufacturer claims is available for handling and placement of his particular product. This may well be different from the minimum "working time" as determined by the test procedure specified in 5.3.

The instrument is checked by attaching a fine nylon thread to a hook (J). A 10,2 g mass is attached to the other end of the thread, which passes over a polytetrafluoroethylene (PTFE) rod. Hence, a force of 0,1 N is applied at right angles to arm (G) at point (J) which is 54 mm from the centre of platten (C) : this is equivalent to a torque of 5,4 N·mm. When this calibration torque is applied, screws (K) and (L) are adjusted so that the deflection on the microammeter is the same as the deflection obtained when the instrument is running with no material on the platten.

5.3.2 *Procedure*

Mix the test material in accordance with the manufacturer's instructions. Start timing from the commencement of mixing. Place the mixed material on platten (C) and lower the upper platten (M) on its guide and screw into position 20 s after the end of mixing. Maintain the water circulating through platten (M) at 23 ± 1 °C.

Take readings, one every 6 s, on one side of the meter and plot these values against time. Alternatively, the transducer output may be fed into a suitable automatic recording apparatus and a continuous trace of variation of viscosity with time may be obtained [(as shown in figure 2a)].

Measure the trace 40 s after commencing recording (i.e. 1 min after the end of mixing), when it shall be not less than 95 % of the original width.

5.4 Setting time

5.4.1 *Apparatus*

Rheometer apparatus specified in 5.3.1.

5.4.2 *Procedure*

Repeat the procedure specified in 5.3.2 using water at 37 ± 1 °C circulating through platten (M).

From the curve obtained [see figure 2b)], record the setting time as the time at which the curve flattens out to the final straight portion. Alternatively, the setting time may be recorded as the time at which the curve deviates from its final straight portion.

5.5 Transverse strength

5.5.1 *Apparatus*

5.5.1.1 Metal mould, PTFE lined[1], for preparation of a specimen 25 ± 2 mm x 2 ± 0,1 mm x 2 ± 0,1 mm. A suitable mould is illustrated in figure 3.

5.5.1.2 Two plates, of, or lined with, PTFE[1] or other suitable material, approximately 25 mm x 25 mm.

5.5.1.3 Small clamp.

5.5.1.4 Suitable testing apparatus, with jig for application of loading in the manner specified in 5.5.3. The jig consists essentially of two rods (2 mm diameter) mounted parallel with 20 mm between centres, and a third rod (2 mm diameter) between and parallel to the other two, so that the three rods in combination can be used to give a three-point loading to the specimen.

5.5.2 *Preparation of test specimen*

Mix the material according to the manufacturer's instructions and immediately place in the mould situated on a PTFE plate. Place the second PTFE plate on top of the mould and gently apply pressure by means of the clamp, thus exuding excess material from the mould. Three minutes after the commencement of mixing, place the assembly in a water bath maintained at 37 °C. Fifteen minutes after the commencement of mixing, remove the clamp and separate the mould. Store the specimen in distilled water at 37 °C.

5.5.3 *Procedure*

Twenty-three hours after the commencement of mixing, transfer the specimens to a second bath of distilled water, at 23 ± 1 °C. Fifty minutes later, remove the specimen, wipe dry and place in the transverse testing jig in the testing apparatus.

Twenty-four hours after the commencement of mixing, apply a load to the specimen at 0,75 ± 0,25 mm/min cross-head speed, or at 50 ± 16 N/min if an incremental loading apparatus is used.

5.5.4 *Calculation and expression of results*

The transverse strength a is given, in newtons per square millimetre (megapascals), by the formula

$$a = \frac{3\,F\,l}{2\,b\,d^2}$$

where

F is the maximum force, in newtons, exerted on the specimen;

l is the distance, in millimetres, between the supports (correct to 0,01 mm);

b is the breadth, in millimetres, of the specimen measured immediately prior to testing (correct to 0,01 mm);

d is the depth, in millimetres, of the specimen measured immediately prior to testing (correct to 0,01 mm).

Calculate the mean of three or more values from a total of five determinations, and report the result to three significant figures. Discard any values for individual specimens which fall more than 15 % below the mean of the five, and report the mean of the remaining values. Repeat the whole test if more than two of the specimens have to be discarded.

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[1] PTFE tape of thickness 0,05 to 0,20 mm is suitable.

5.6 Colour stability

5.6.1 *Heat*

5.6.1.1 APPARATUS

5.6.1.1.1 Metal moulds, PTFE lined[1], suitable for preparing specimens of diameter 15 ± 1 mm and thickness 0,5 ± 0,10 mm.

5.6.1.1.2 Clamps, suitable for use with the moulds (5.6.1.1.1).

5.6.1.1.3 Oven, capable of being controlled at 37 ± 1 °C.

5.6.1.1.4 Oven, capable of being controlled at 60 ± 2 °C.

5.6.1.2 PROCEDURE

Prepare the resin paste according to the manufacturer's instructions and fill a pair of lined moulds. Clamp the moulds and transfer them, within 3 min of the commencement of mixing, to the oven (5.6.1.1.3), controlled at 37 ± 1 °C. After 1 h, timed from the commencement of mixing, take the moulds from the oven and remove the two specimen discs.

Place one disc in the oven (5.6.1.1.4), controlled at 60 ± 2 °C, for a further 23 h and return the other disc to the oven (5.6.1.1.3), controlled at 37 ± 1 °C, for the same period of time.

Remove both discs from their respective ovens and compare them using unaided visual examination in diffuse daylight. Only a slight change in colour should be perceptible.

Retain the disc from the 37 °C oven for use as a control specimen in the procedure specified in 5.6.2.2.

5.6.2 *Light*

5.6.2.1 APPARATUS

Metal moulds (5.6.1.1.1), **clamps** (5.6.1.1.2) and **oven** (5.6.1.1.3) and in addition

5.6.2.1.1 Cabinet with adjustable ventilation, containing a xenon lamp radiation source, as specified in ISO/R 879, and a turntable capable of being rotated at 33 min^{-1}.

5.6.2.2 PROCEDURE

Prepare the resin paste according to the manufacturer's instructions and fill a lined mould. Clamp the mould and transfer it, within 3 min of the commencement of mixing, to the oven controlled at 37 ± 1 °C. After 1 h, timed from the commencement of mixing, remove the specimen disc and blank off half the surface to be exposed to light with metal foil.

Place the specimen in a glass dish centred on the turntable and cover the specimen with water to a depth of 5 to 20 mm. Position the specimen so that it is 75 mm from the centre of the dish and its uppermost surface is 350 to 400 mm directly below the xenon lamp. Maintain the specimen at a temperature of 37 ± 1 °C, by adjusting the cabinet ventilation, and expose it to the radiation for 23 h with the turntable rotating at 33 min^{-1}. Replace any water lost by evaporation, so that the specimen is always covered to a depth of between 5 and 20 mm.

After 23 h exposure, remove the specimen from the water and dry with an absorbent cloth or tissue. Make a comparison between the exposed and unexposed area of the test specimen, and then between the exposed portion and the control specimen from the heat test (5.6.1.2). Only a slight change in colour should be perceptible using unaided visual examination in diffuse daylight.

5.7 Filler content

5.7.1 *Apparatus*

5.7.1.1 Muffle furnace, capable of being controlled at 575 ± 25 °C.

5.7.1.2 Crucible.

5.7.2 *Preparation of test specimens*

Mix sufficient material to provide a test specimen of mass approximately 0,5 g.

5.7.3 *Procedure*

Heat the crucible in the muffle furnace for 15 min at 575 ± 25 °C, allow to cool in a desiccator and weigh to the nearest 0,001 g (mass m_1). Place the test specimen in the crucible and weigh the whole to the nearest 0,001 g (mass m_2).

Heat the crucible and specimen in the furnace at 575 ± 25 °C for 30 min. Then carefully remove the crucible with its contents, allow to cool in a desiccator and weigh to the nearest 0,001 g.

Repeat the cycle of heating, cooling and weighing until the mass is constant to within 0,001 g (mass m_3).

5.7.4 *Calculation and expression of results*

The filler content is given, as a percentage by mass, by the formula

$$\frac{m_3 - m_1}{m_2 - m_1} \times 100$$

Carry out the test on three specimens and report the mean.

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[1] PTFE tape of thickness 0,05 to 0,20 mm is suitable.

6 PACKAGING AND MARKING

6.1 Composite materials

Those materials which come within the category "composite", as described in clause 3, shall include this description in the marking used on the packaging.

6.2 Non-capsulated materials (for hand mixing)

6.2.1 *Packaging*

The components of the material shall be supplied in properly sealed containers[1] which adequately protect their contents and have no adverse effect on the quality of the product. An outer pack may also be used to present the containers as a single unit.

6.2.2 *Marking*

The following details shall be clearly visible :

a) the manufacturer's name or trade-mark;

b) batch number, consisting of a serial number or combination of letters and numbers which refer to the manufacturer's records for that particular batch of material;

c) date of manufacture or despatch (year and month) in plain language;

d) on the outermost packaging, the recommended conditions of storage, and the shelf life or "expiry date" for the material under those conditions of storage;

e) on each container, a shade number or description, which can be related to the manufacturer's shade guide, if the material is supplied in a range of pre-coloured standard shades;

f) on each container, the net mass in grams or the volume in millilitres.

6.3 Capsulated materials

6.3.1 *Packaging*

The capsules shall be supplied in a convenient container which provides adequate protection and complies with the requirements of 6.3.2.

6.3.2 *Marking*

The "bulk" container shall be marked with the following details :

a) the manufacturer's name or trade-mark;

b) batch number, consisting of a serial number or combination of letters and numbers which refer to the manufacturer's records for that particular batch of material;

c) date of manufacture or despatch (year and month) in plain language;

d) net mass of the total powder and liquid contained in all the capsules, and net mass contained in each capsule;

e) recommended conditions of storage, and the shelf life or "expiry date" for the material under those conditions of storage.

6.3.3 *Capsulation*

The capsules shall comply with the following marking and constructional requirements :

a) Shade. Each capsule shall be marked so that the shade of its contents may be identified.

b) Materials. The capsules shall be made of materials which neither contaminate nor permit contamination of their contents.

c) Component separation. When two or more components are contained in the same capsule, they shall be separated so that premature mixing does not take place under normal conditions of storage and handling.

d) Design. When the manufacturers instructions are followed, the method of capsulation shall permit the mixing of the components to take place without difficulty.

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

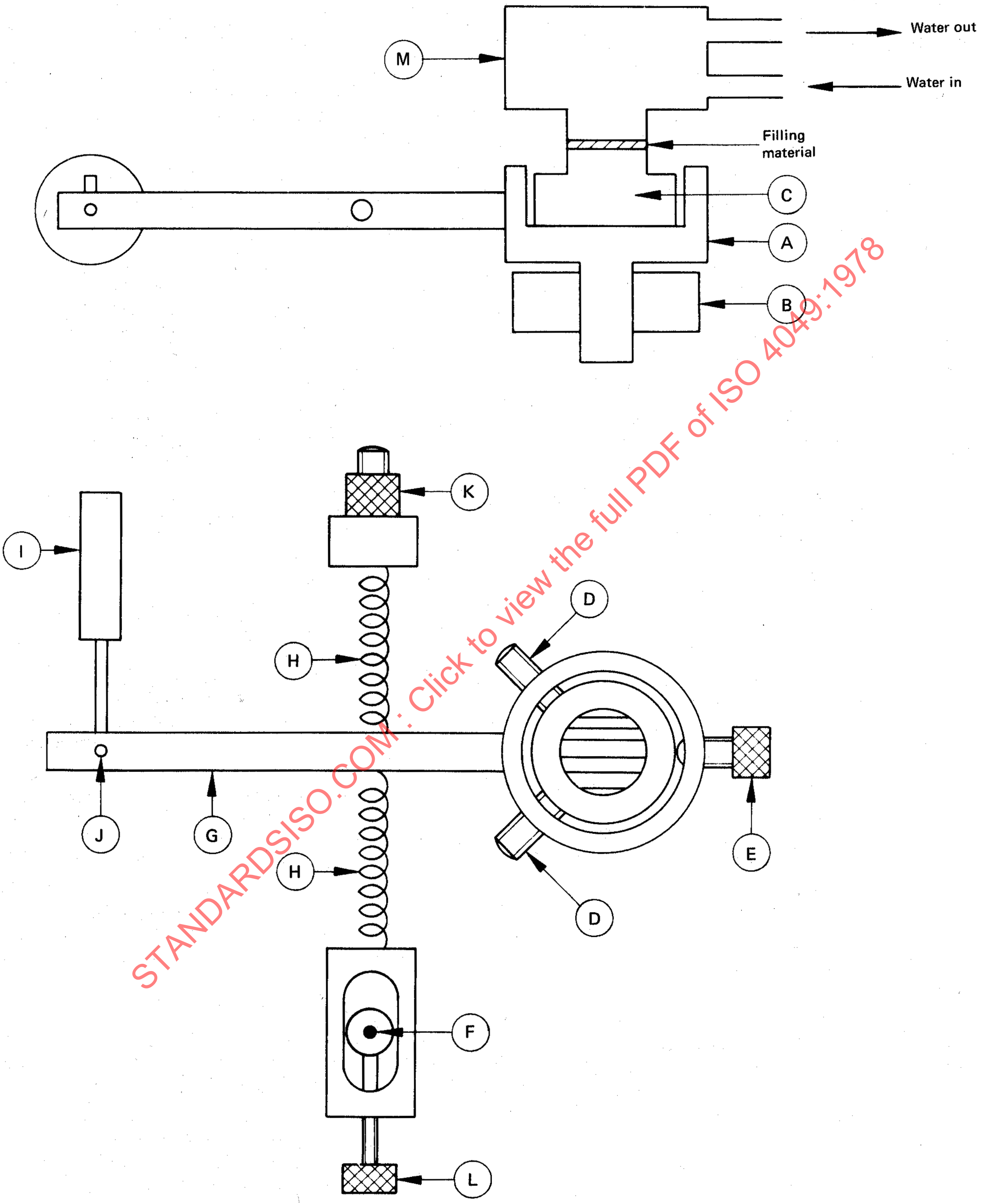
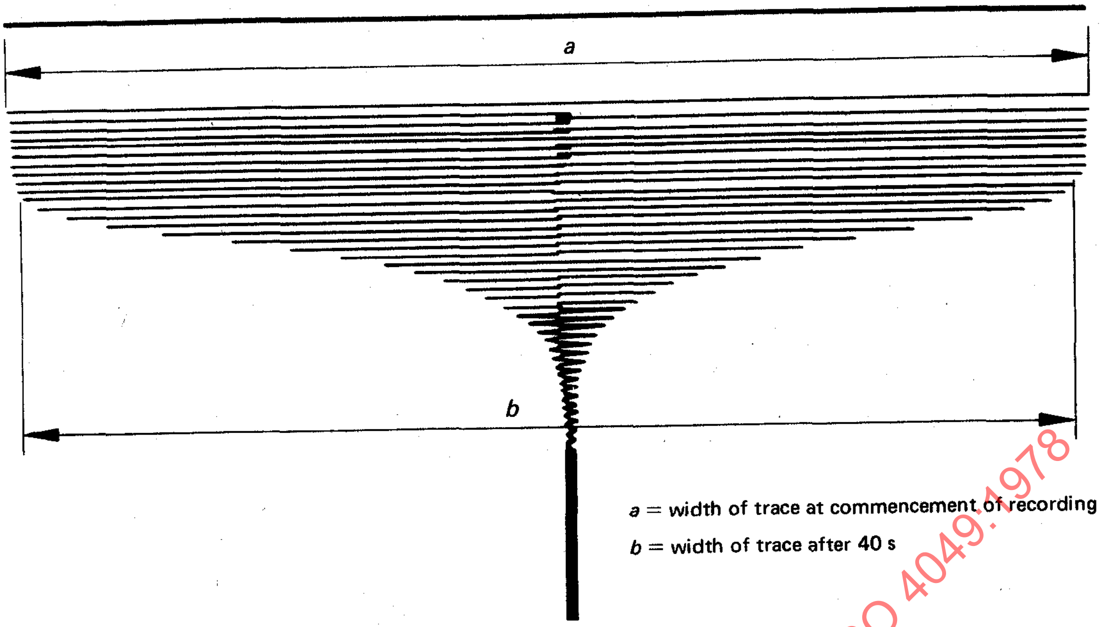


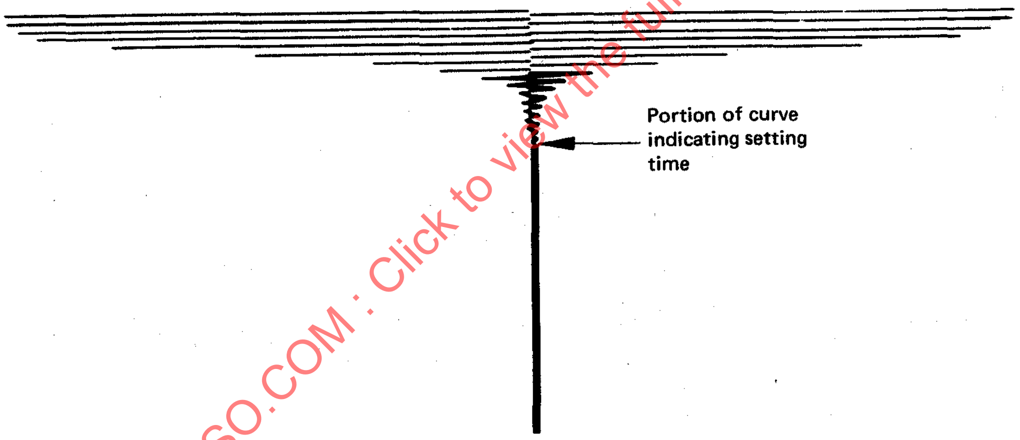
FIGURE 1 – **Oscillating rheometer suitable for determination of working time and setting time** (see 5.3 and 5.4)



a = width of trace at commencement of recording

b = width of trace after 40 s

a) **Trace for verification of minimum working time** (see 5.3.2)



Portion of curve indicating setting time

b) **Trace for determination of setting time** (see 5.4.2)

FIGURE 2 – **Traces of viscosity as a function of time**

Dimensions in millimetres

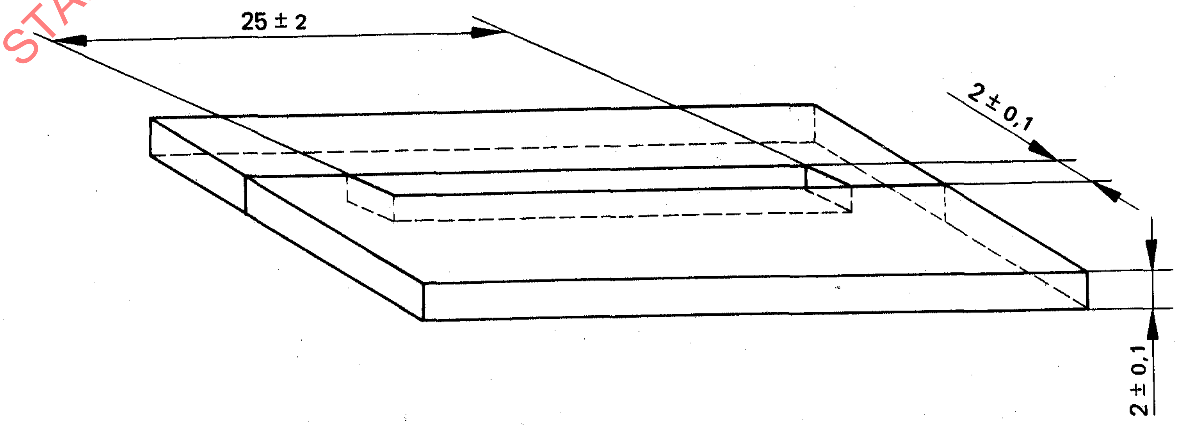


FIGURE 3 – ***Suitable mould for preparation of test specimens***