INTERNATIONAL STANDARD 1563

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

Alginate dental impression material

Produits pour empreintes dentaires à base d'alginate

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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 1563 was developed by Technical Committee ISO/TC 106, *Dentistry*, and was circulated to the member bodies in November 1976.

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

Australia	India	Philippines
Austria	Ireland	South Africa, Rep. of
Canada	Korea, Rep. of	Sweden
Czechoslovakia	Mexico	Switzerland
Denmark	Netherlands	Thailand
Egypt, Arab Rep. of	New Zealand	United Kingdom
France	Norway	U.S.A.

The member body of the following country expressed disapproval of the document on technical grounds :

Germany

This International Standard cancels and replaces ISO Recommendation R 1563-1970, of which it constitutes a technical revision.

Printed in Switzerland

Alginate dental impression material

0 INTRODUCTION

This International Standard was first published by ISO in 1970 as an ISO Recommendation based on FDI Specification No. 9. In common with the other ISO Recommendations in this initial series on dental materials, ISO/R 1559 to ISO/R 1567, it was then the subject of a programme of revision to bring its contents up to date on the basis of technical data from both ISO/TC 106 and the Fédération Dentaire Internationale. The latter organization undertook the secretariat responsibilities of the working group which prepared this International Standard.

A considerable number of changes have been made in this revision, many originating from research on a standard for elastomeric impression materials; the new test methods for working time and setting time are examples. The classification system, which was previously linked solely to setting time, has now been extended to include an additional grouping according to the clinical application of the material. Also of clinical significance are the very much more comprehensive instructions which manufacturers will have to supply with their products.

1 SCOPE AND FIELD OF APPLICATION

The International Standard specifies requirements for dental impression materials, in powder form, containing an alginate as the gel-forming ingredient.

2 CLASSIFICATION

Alginate impression materials shall be classified as follows :

2.1 Classes based on clinical application

– **Class A** : for crown or inlay impressions

– **Class B** : for denture impressions

– **Class C** : for study models and individual tray impressions

2.2 Types based on setting time

– **Type I** : fast setting – stated setting time of 3 min or less at 32 °C.

– **Type II** : normal setting – stated setting time of more than 3 min and less than 5 min at 32 °C.

3 REQUIREMENTS

3.1 General requirements

3.1.1 *Appearance*

The powder shall be uniform and free from foreign materials. When used in accordance with the manufacturer's instructions (see 3.3) accompanying the package, the mixed paste shall form a smooth plastic mass suitable for taking impressions in the mouth.

3.1.2 *Odour and flavour*

The powder, when used in accordance with the manufacturer's instructions, shall have no unpleasant odour or flavour.

3.1.3 *Irritation and freedom from toxicity*[1)]

The mixed paste, when used in accordance with the manufacturer's instructions, shall neither cause irritation of the normal oral mucosa, nor contain poisonous ingredients in sufficient concentration to be harmful to human beings.

3.2 Special requirements

3.2.1 *Uniformity*

The ingredients, after mixing, shall not segregate. The mixed paste shall be homogeneous, shall have a smooth surface, and shall be free from lumps and granules.

3.2.2 *Mixing time*

The time of mixing, as stated in the manufacturer's instructions, required to obtain a smooth working consistency shall be not more than 1 min.

1) Specific tests for irritation and toxicity are under consideration.

3.2.3 *Working time*

The working time shall be recorded from the commencement of mixing.

The working time, when determined in accordance with 6.2.1, shall be not less than 85 % of the manufacturer's stated working time.

3.2.4 *Setting time*

The setting time shall be recorded from the commencement of mixing.

The setting time, when determined in accordance with 6.2.2, shall be not less than the manufacturer's stated working time or more than 10 % above the manufacturer's stated setting time.

3.2.5 *Viscosity*

When measured in accordance with 6.2.3, the mean diameter of the discs formed from the paste shall comply with the relevant requirements detailed in the table.

3.2.6 *Compatibility with gypsum — detail reproduction*

The impression material shall impart a smooth surface to, and separate cleanly from, a gypsum cast made from a trade brand of gypsum recommended in the instructions for use (see 3.3.8). The cast poured against the impression, in accordance with 6.2.4, shall reproduce the relevant line as specified in the table.

3.2.7 *Permanent deformation*

The permanent deformation, after application of a strain of 20 % for 5 s duration in accordance with 6.2.5, shall be not more than the relevant requirement as specified in the table.

3.2.8 *Strain in compression*

The strain shall be not less than 5 % or more than 20 % between a stress of 0,01 MPa and a stress of 0,10 MPa when measured in accordance with 6.2.6.

3.2.9 *Compressive strength*

The compressive strength shall be not less than 0,30 MPa when tested in accordance with 6.2.7.

3.2.10 *Proportioning devices*

The cups or spoons provided for proportioning the components, when used to measure powder and water in accordance with the manufacturer's instructions and test conditions described in 6.2.8, shall provide a powder/water ratio which does not deviate more than 7,5 % from the stated powder/water ratio.

TABLE — **Special requirements**

Characteristic	Class A	Class B	Class C
Viscosity (6.2.3) : diameter of disc, mm	⩾ 33	27 to 36	⩽ 30
Detail reproduction (6.2.4) : width of line reproduced, µm	20	50	75
Permanent deformation (6.2.5), %	⩽ 3	⩽ 5	⩽ 7

3.3 Instructions for use

Adequate and accurate instructions for use shall accompany each package. These instructions shall include, as a minimum, the following information :

3.3.1 *Temperature range*

The range of mixing water temperatures (or ambient conditions) over which the material offers adequate results.

3.3.2 *Proportioning powder and water*

The amount of powder, expressed in grams, and the amount of water, expressed in grams or millilitres, to be used for mixing.

3.3.3 *Mixing time*

The mixing time, expressed in minutes or seconds.

3.3.4 *Working time*

The working time available, stated for temperature increments of 2 °C over the entire temperature range (3.3.1).

3.3.5 *Setting time*

The setting time, stated for temperature increments of 2 °C over the entire temperature range (3.3.1).

3.3.6 *Time to pour the model*

The latest time at which the impression should be poured with gypsum, for each condition of storage recommended for the impression.

3.3.7 *Special treatment*

Any special treatment required for the impression, such as the use of a fixative solution, during the interval of time between withdrawal from the mouth and the preparation of the gypsum.

3.3.8 *Gypsum*

Class A alginates : At least two brands of alpha modified gypsum[1] and one brand of alpha gypsum[1] shall be recommended for Class A materials.

Class B alginates : At least one brand of alpha modified gypsum[1] and two brands of alpha gypsum[1] shall be recommended for Class B materials.

Class C alginates : At least two brands of alpha gypsum[1] shall be recommended for Class C materials.

4 SAMPLING

Approximately 750 g of powder, sufficient for a minimum of 60 small mixes, is required for testing. The method of procurement shall be the subject of agreement between the interested parties.

5 PREPARATION OF TEST SPECIMENS

Prepare specimens by mixing the alginate material with distilled or deionized water using the ratio of components and the method of mixing stated in the manufacturer's instructions. Mix the alginate and water for 60 s, or the manufacturer's maximum stated mixing time if the latter is less than 60 s.

Condition the components and the test equipment for not less than 10 h and, unless stated otherwise, carry out all tests at 23 ± 1 °C and 50 ± 5 % relative humidity.

6 TEST METHODS

6.1 Visual inspection

Use visual inspection in determining compliance with the requirements of 3.2.1, 3.2.2, and 3.3.

6.2 Physical tests

6.2.1 *Working time*

6.2.1.1 APPARATUS

6.2.1.1.1 Loading device, such as shown in figure 1, that allows essentially frictionless movement in the vertical direction. The movable column B shall have a mass of 125 g and shall terminate in a platen (15 mm in diameter) parallel to the base of the instrument.

There shall be provision for the attachment of weights to increase the mass of the movable column to 500 and 2 000 g as required for the several classes of material.

6.2.1.1.2 Mould with inside dimensions of 130 mm × 25 mm × 3 mm and a flat rectangular base of constant thickness, made, for example, of glass.

6.2.1.1.3 Six circular glass or polymethyl methacrylate discs 5 ± 1 mm thick and 15,95 ± 0,2 mm in diameter. The flat surfaces of each disc shall be parallel to within 0,01 mm.

6.2.1.1.4 Dial gauge, accurate to 0,01 mm, mounted perpendicularly on a stable base. The force exerted by the dial indicator shall be 0,59 ± 0,1 N (60 ± 10 gf).

6.2.1.2 PROCEDURE

Position six circular discs on the mould assembly and place beneath the dial gauge so that the foot contacts the upper surface of the first disc. Read the dial gauge and record the value as A_1. Repeat the procedure for each of the other discs and record the values as A_2, A_3, A_4, A_5 and A_6. Remove the discs and place in the same order as dimensions A_1 to A_6 were recorded.

Fill the mould assembly with mixed alginate material and roughly level with a spatula. Then place the six discs on the alginate surface in the same order as the readings A_1 to A_6 were taken and in such a manner that they are equally distributed over the mould cavity.

Place the entire assembly on the base of the loading device in such a position that the first disc can be forced into the alginate material by lowering the movable column. Select an appropriate weight for the movable column (125, 500 or 2 000 g) such that the thickness of the alginate layer beneath the first circular disc is between or as near as possible to 0,13 to 0,33 mm.

Apply the load for 10 s to each of the six discs according to the following time schedule :

load first disc : 30 s after the end of mixing;

load second disc : 45 s after the end of mixing;

load third disc : 30 s before the stated working time;

load fourth disc : 15 s before the stated working time;

load fifth disc : at the stated working time;

load sixth disc : 15 s after the stated working time.

After this loading sequence, transfer the whole assembly to the dial gauge and take readings with the foot of the dial gauge in contact with the respective circular discs. Record these values as B_1, B_2, B_3, B_4, B_5 and B_6. The difference between the "B" and "A" readings, $B - A$, will be the thickness of the alginate beneath the circular discs.

Plot the figures for the thickness of the alginate layer, $B - A$, under each disc against time on Cartesian graph paper and draw the best curve through or near the points. Using the curve drawn, record as the working time the first time (rounded to the nearest 5 s) that the thickness of the alginate layer is double the thickness of the alginate layer beneath the first disc.

Record the mean of three such determinations, rounded to the nearest 15 s, as the test result.

1) Alpha modified gypsum is characterized by a water/powder ratio of about 0,23. Alpha gypsum is characterized by a water/powder ratio of about 0,3. The recommended brands shall be available (and shall be provided with the relevant instructions for use) in the country in which the alginate is marketed.

6.2.2 *Setting time*

6.2.2.1 APPARATUS

6.2.2.1.1 Flat metal plate with holes of 5,50 mm diameter as illustrated in figure 2.

6.2.2.1.2 Steel balls having a diameter of 6,35 mm.

6.2.2.1.3 Metal tray, as illustrated in figure 3, with inside dimensions of 80 mm × 24 mm and 10,0 mm deep.

6.2.2.1.4 Plunger as illustrated in figure 4. The length of the reduced diameter portion of the plunger shall be 0,40 mm more than the thickness of the plate (6.2.2.1.1).

6.2.2.1.5 Dial gauge equipped with a fixed base, as illustrated in figure 5. The force exerted by the dial indicator shall be 0,59 ± 0,1 N (60 ± 10 gf). The foot of the dial gauge shall be a cylinder with a diameter of 4 mm and a height of at least 6 mm.

6.2.2.2 PROCEDURE

Place the steel balls on the holes in the flat plate. Mix a double quantity of alginate material and slightly overfill the metal tray. Place the tray in the inverted position over the balls and press firmly onto the plate. Thirty seconds after the end of mixing, place the tray, plate and steel balls assembly, without disturbing the relative or overall positions of the components, in a water bath at 32 ± 1 °C.

At the working time, as determined according to 6.2.1, invert the tray and plate assembly so that the plate is on top of the tray. Thirty seconds before the stated setting time or as soon as possible if the period between the working time and the stated setting time is less than 30 s, press the ball under hole No. 1 into the alginate for a distance of 2,0 mm by forcing the plunger into hole No. 1 until the shoulder of the plunger contacts the plate. Keep the plunger in this position for 5 ± 0,5 s.

At 15 s intervals, press four further balls sequentially 2 mm into the alginate for 5 ± 0,5 s, utilizing holes Nos. 2 to 5. Press the remaining balls (holes Nos. 6 to 9) into the alginate at 30 s intervals for a period of 5 ± 0,5 s.

Transfer the assembly from the water bath and determine the permanent vertical displacement of the balls by lowering the foot of the dial gauge through the holes onto the surface of the balls. Take each reading for displacement 10 s after the positioning of the dial gauge.

Plot the values obtained for the permanent vertical displacement (see note) of the balls against time on Cartesian graph paper and draw the best curve through or near the points plotted. Draw, parallel to the time axis, a line which intersects the displacement at a value 0,20 mm greater than the mean of the three experimental values showing the least permanent vertical displacement. Record as the setting time the time indicated by the intersection of this line with the curve.

NOTE — In the early stage of setting, the 2 mm movement of the steel ball can lead to a flow of the alginate material which results in inaccurate displacement measurements. Such measurements, suggesting a high degree of elasticity of the material, shall be discarded.

Carry out this procedure to determine the time of setting three times, round off the average to the nearest 15 s, and record as the test result.

6.2.3 *Viscosity*

6.2.3.1 APPARATUS

6.2.3.1.1 Load of 1 500 ± 2 g mass mounted in a loading device such as that shown in figure 1, in such a manner as to allow essentially frictionless movement of the movable column in the vertical direction. The apparatus described in 6.2.1.1 may be used, provided that the mass of the movable column is 1 500 g.

6.2.3.1.2 Glass delivery tube of approximately 10 mm internal diameter, designed to deliver 0,5 ± 0,02 ml, by means of a plunger.

6.2.3.1.3 Two glass plates approximately 60 mm square and of mass 20 ± 2 g each.

NOTE — During the testing procedure it is essential that the glass plates be maintained parallel and that no rotational movement be allowed to take place.

6.2.3.2 PROCEDURE

Using the delivery tube, dispense 0,5 ml of the mixed material onto the glass plate. Gently lower the second glass plate and the 1 500 g load onto the material 30 s after the end of mixing for Type 1 material and 60 s after the end of mixing for Type II material. Five seconds later, remove the load and measure the diameter of the disc of material to the nearest 0,5 mm, across two diameters at right angles to each other. Calculate the mean of these two measurements for each specimen.

Record the average of three determinations as the test result for viscosity.

6.2.4 *Compatibility with gypsum — Detail reproduction*

6.2.4.1 APPARATUS

6.2.4.1.1 Ruled test block A and **moulds** B and D as illustrated in figure 7.

A rubber ring, 20 mm high and 38 mm in diameter, tightly fitting on mould B, may be used instead of mould D.

NOTE — Riser C of figure 7 is not used, as the change in dimension is not determined.

6.2.4.1.2 Flat metal or glass plate, covered with cotton-backed surgical adhesive tape, sufficiently large to be a base for mould B.

6.2.4.1.3 Load of 1 kg mass.

6.2.4.1.4 Water bath, capable of being maintained at 32 ± 1 °C.

6.2.4.2 Procedure

Place mould B on the plate covered with cotton-backed surgical adhesive tape, and slightly overfill with mixed alginate material. Thirty seconds before the working time, determined in accordance with 6.2.1, centre the clean test block above mould B and press it down into the mass of alginate. If the alginate adheres to the surface of the ruled test block, dust the block with talcum prior to use and blow away any excess powder. Immediately place the assembly in a water bath maintained at 32 ± 1 °C and load with the 1 kg mass.

Three minutes after the setting time, determined in accordance with 6.2.2, or the stated setting time, whichever is the greater, remove the assembly from the water bath and separate the mould, together with the plate, from the test block. Prepare a gypsum slurry, using a brand mentioned in the manufacturer's instructions, according to the directions for the product. Rinse the alginate surface with water, or treat in any other way described in the manufacturer's instructions (with for example fixative solution), and shake off the excess fluid. Place mould D on mould B and fill with the gypsum slurry, using mechanical vibration in such a way that the gypsum displaces any adhering moisture from the surface of the alginate. Allow the gypsum slurry to harden for 30 min. Separate mould D, containing the gypsum cast, from mould B containing the alginate material.

Observe the gypsum cast under low angle illumination, with a magnification of 6 to 12 X, and record the finest line reproduced over the full length of 25 mm.

6.2.4.3 Expression of results

Record the width of the finest line reproduced at least twice by the casts, resulting from three tests, as the test result for compatibility-detail reproduction.

6.2.5 *Permanent deformation after* 20 % *strain*

6.2.5.1 Apparatus

The following apparatus, or another with equal accuracy, performance and speed, shall be used :

6.2.5.1.1 Dial gauge mounted on a stable base. The gauge shall terminate at its foot in a platen (15 mm diameter) parallel to the base of the device, as illustrated in figure 8. The force exerted by the dial gauge assembly shall be 0,59 ± 0,1 N (60 ± 10 gf).

6.2.5.1.2 Apparatus to apply a 20 % strain as illustrated in figure 9. The distance between the adjustable table T and platen P (parallel to the table) shall be 16,0 mm when beam B touches stop S.

6.2.5.1.3 Water bath, capable of being maintained at 32 ± 1 °C.

6.2.5.2 Preparation of test specimens

Prepare the test specimens by placing a ring, 30 mm inside diameter and 16 mm high, on a flat glass or metal plate covered with polyethylene film, and fill slightly more than one-half full with alginate material mixed according to the manufacturer's instructions. Place a cylindrical metal mould, 12,5 mm inside diameter, 25,0 mm outside diameter and 20,0 mm high, immediately inside the ring and force the mould into the alginate until the mould touches the plate and alginate has exuded up to the top of the mould. Press a second flat glass or metal plate covered with polyethylene film, on the top of the mould. Thirty seconds after the end of mixing, place the mould and its accompanying plates in a water bath at 32 ± 1 °C.

At the stated setting time, or at the setting time determined in accordance with 6.2.2, whichever is the greater, remove the assembly from the water bath. After removing the flash, separate the specimen from the mould and place on the base of the dial gauge.

6.2.5.3 Procedure

Carry out the test in accordance with the time schedule detailed below, where time S is the manufacturer's stated setting time, or the setting time determined in accordance with 6.2.2, whichever is the greater :

$S + 45$ s : Gently lower the platen of the dial gauge to contact the specimen;

$S + 55$ s : Read the dial indicator and record the value as reading A.

$S + 60$ s : Transfer the specimen to the strain apparatus and place upon table T. Bring the beam B into contact with stop S, thereby subjecting the specimen to a 20 % deformation, and keep in this position for 5 ± 0,5 s.

$S + 90$ s : Gently lower the platen of the dial gauge to contact the specimen which has just before been placed upon the dial gauge base.

$S + 100$ s : Read the dial indicator and record the value as reading B.

6.2.5.4 Expression of results

The permanent deformation, expressed as a percentage, is given by the formula

$$\frac{A - B}{20} \times 100$$

where 20 is the original length, in millimetres, of the specimen.

Record the average permanent deformation of three specimens as the test result.

6.2.6 *Strain in compression*

6.2.6.1 APPARATUS

Any suitable instrument, such as the apparatus illustrated in figure 10, having a dial indicator graduated in 0,01 mm, and capable of applying the force required for the test in accordance with the procedure outlined below, may be used.

6.2.6.2 PROCEDURE

Sixty seconds after the stated setting time, or the setting time determined in accordance with 6.2.2, whichever is the greater, place a specimen prepared as specified in 6.2.5.2 in the instrument and subject it to a load of 123 ± 5 g producing a stress of 0,01 MPa.

Thirty seconds later read the dial gauge. Record this value as reading *C*.

Sixty seconds after application of the stress of 0,01 MPa, carefully apply an additional load of 1 104 ± 5 g, thereby producing a total stress on the specimen of 0,1 MPa, within a period of 10 s.

Thirty seconds after initiating the increase in stress to 0,1 MPa read the dial gauge and record this value as reading *D*.

6.2.6.3 EXPRESSION OF RESULTS

The strain in compression, expressed as a percentage, is given by the formula

$$\frac{C-D}{20} \times 100$$

where 20 is the original length, in millimetres, of the specimen.

Record the average strain of three specimens as the test result.

6.2.7 *Compressive strength*

6.2.7.1 APPARATUS

Any apparatus which permits loading at a rate of 100 N/min and that is capable of measuring force with an accuracy of 0,5 N in accordance with the procedure below, may be used.

6.2.7.2 PROCEDURE

Sixty seconds after the stated setting time, or the setting time determined in accordance with 6.2.2, whichever is the greater, place a specimen, prepared as specified in 6.2.5.2 and covered at both ends with a piece of bond paper, between the anvils of the compressive strength testing apparatus. Load the specimen continuously and as uniformly as possible to give an average rate of loading of 100 ± 20 N/min until fracture.

Record the maximum applied load to the nearest 0,5 N.

6.2.7.3 EXPRESSION OF RESULTS

Calculate the compressive strength, *C*, in megapascals, using the formula

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

where

P is the maximum applied load, in newtons;

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

Record the average compressive strength of three specimens as the test result.

6.2.8 *Proportioning devices*

Weigh the amount of powder, in grams, delivered using the powder proportioning device according to the manufacturer's instructions, five times and calculate the average mass.

Measure the internal volume, in millilitres, of the water proportioning device five times and calculate the average.

Record the average amount of powder, in grams, divided by the average water content, in millilitres, as the test result.

7 PACKAGING AND MARKING

7.1 Packaging

The material shall be supplied in airtight containers which neither contaminate nor permit contamination of the contents.

7.2 Instructions for use

Instructions for use shall accompany each package (see 3.3).

7.3 Marking

7.3.1 *Lot numbers*

Each container shall be marked with a serial number or a combination of letters and numbers which refer to the date of manufacture and to the manufacturer's records for the particular lot or batch.

7.3.2 *Expiry date*

The year and month after which the manufacturer cannot guarantee that the contents of an unopened container will meet the requirements of this International Standard, shall be given in legible type on the container and the outer wrapping. If the expiry date is related to special storage conditions, these shall be given in legible type on the container and the outer wrapping.

7.3.3 *Net mass*

The net mass of the contents, in grams or kilograms, shall be given in legible type on the container.

7.3.4 *Number of impressions*

The number of full impressions (Classes B and C) or the number of partial impressions (Class A) which can be prepared from the contents shall be given in legible type on bulk containers.

7.3.5 *Class and type*

The class and type of the alginate impression material shall be indicated on all containers.

7.3.6 *Number of this International Standard*

Each container shall be marked with the number of this International Standard, i.e. ISO 1563.

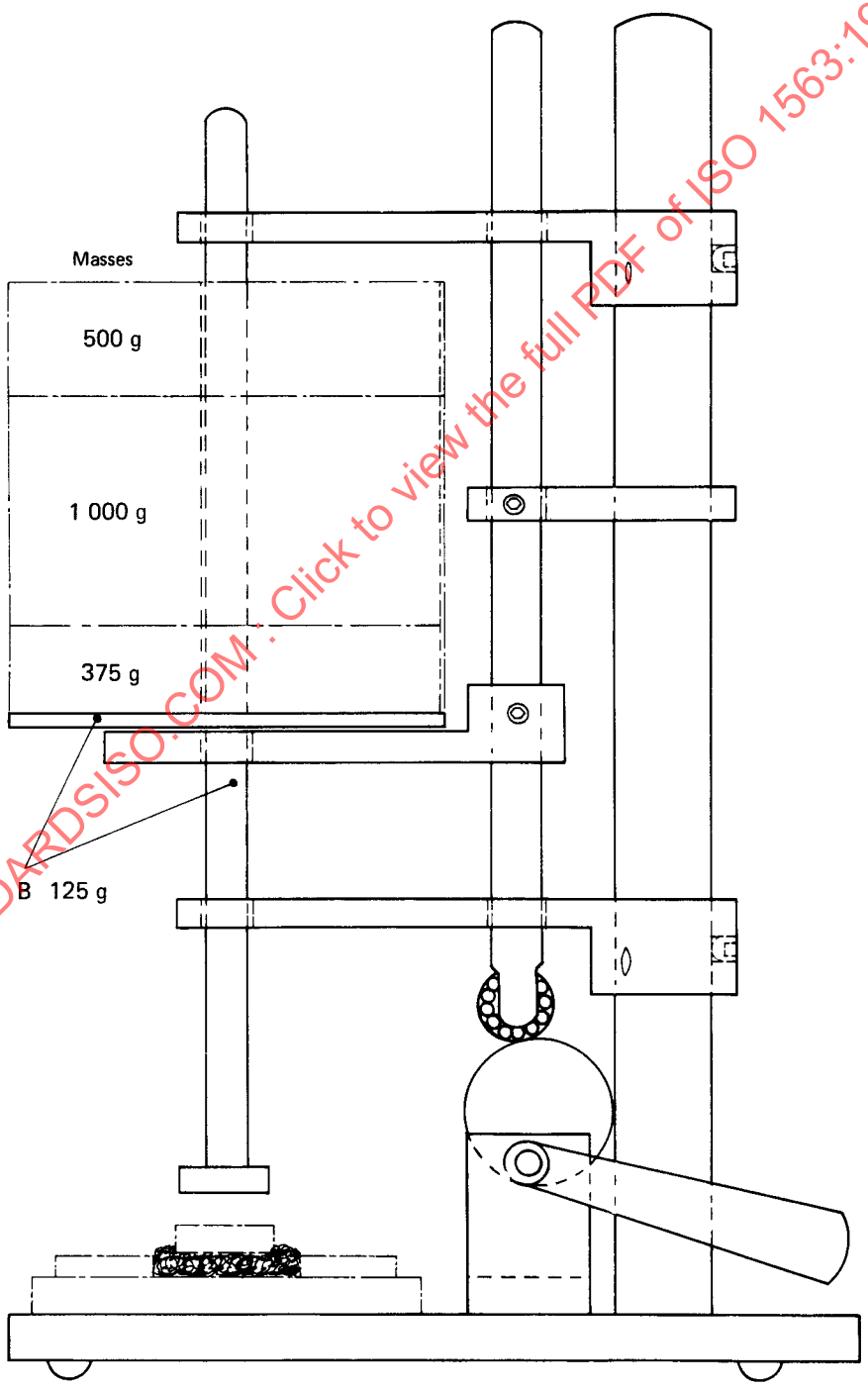
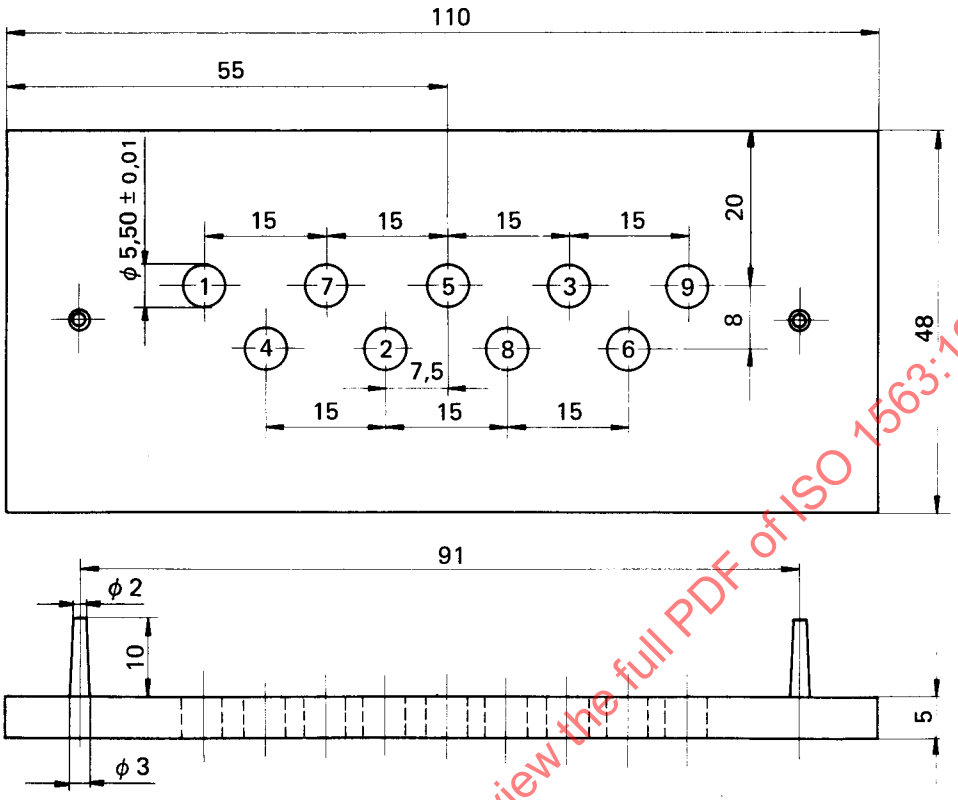


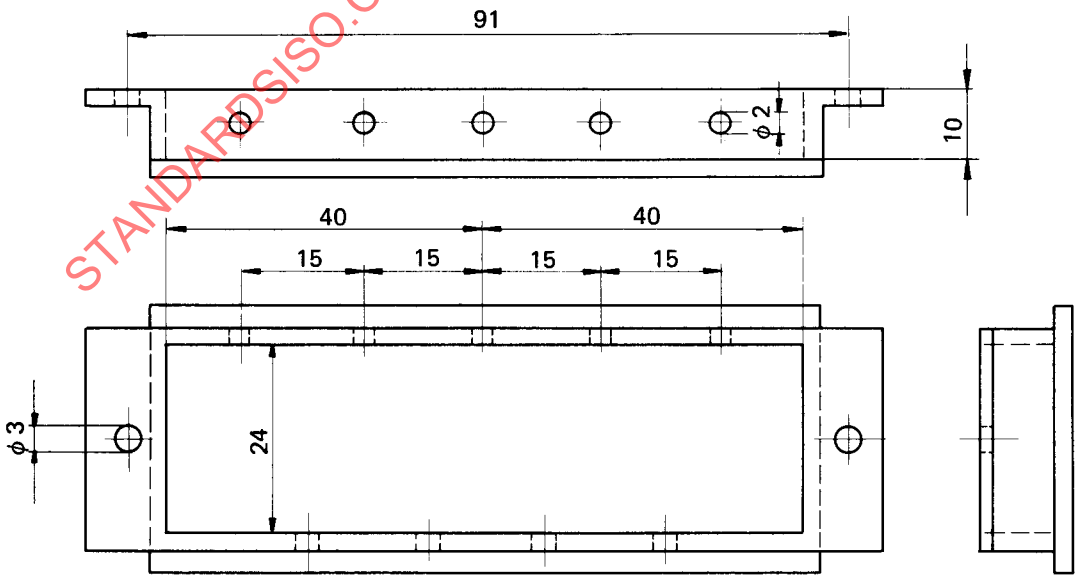
FIGURE 1 — **Apparatus for determination of working time and viscosity**

Dimensions in millimetres



Materials :
Plate — brass
Leading pins — stainless steel

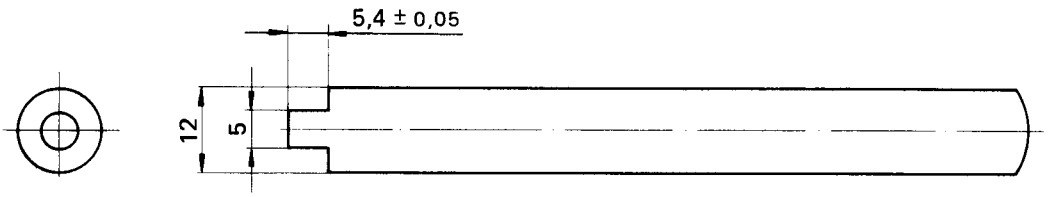
FIGURE 2 — **Component of apparatus for determination of setting time — Metal plate containing holes**



Material :
brass

FIGURE 3 — **Component of apparatus for determination of setting time — Metal tray**

Dimensions in millimetres



Material :
brass or stainless steel

FIGURE 4 — **Component of apparatus for determination of setting time — Plunger**

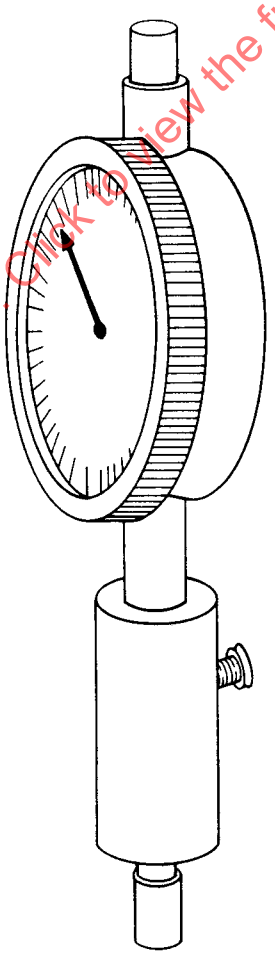


FIGURE 5 — **Component of apparatus for determination of setting time — Dial gauge equipped with fixed base**

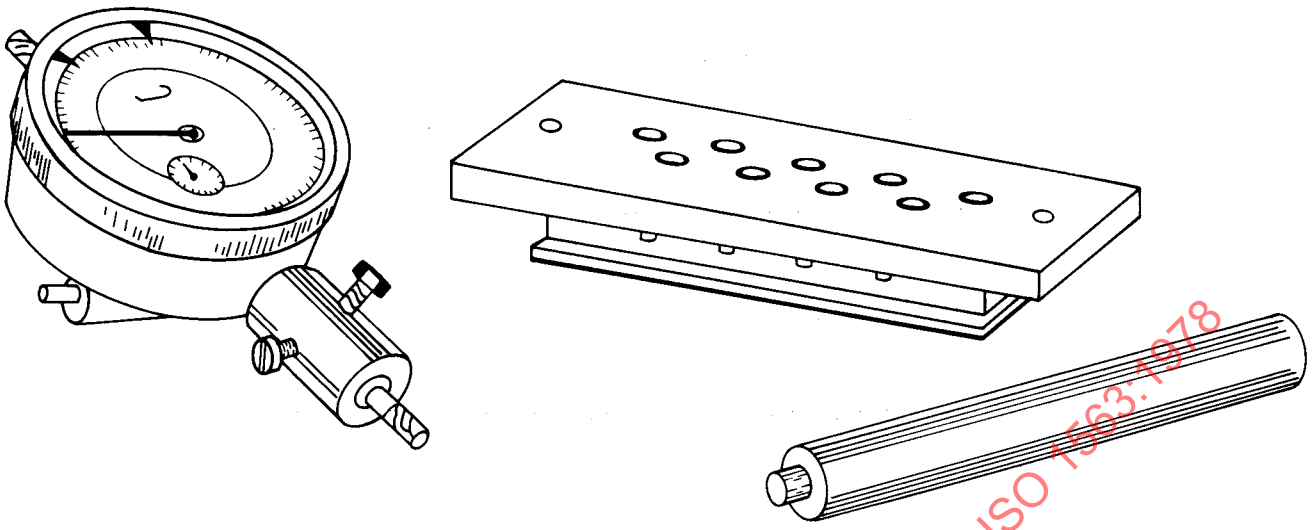


FIGURE 6a) – **Apparatus for determination of setting time**

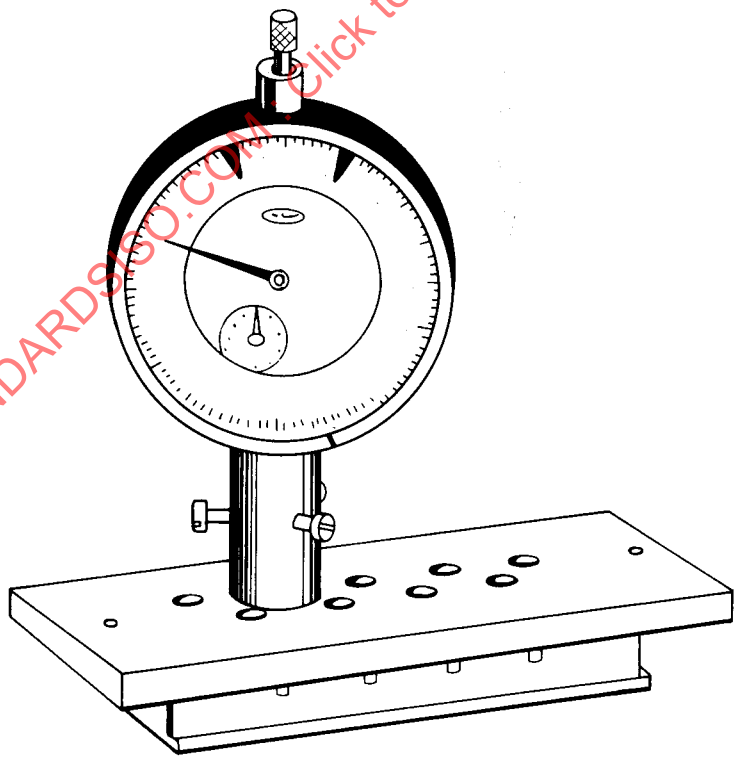


FIGURE 6b) – **Apparatus for determination of setting time – Dial gauge measuring displacement of steel balls**