INTERNATIONAL STANDARD 1567

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Denture base resin

Résines pour bases de prothèses dentaires

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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 1567 was developed by Technical Committee ISO/TC 106, *Dentistry*, and was circulated to the member bodies in September 1977.

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

Australia	Ireland	South Africa, Rep. of
Canada	Korea, Rep. of	Sweden
Czechoslovakia	Netherlands	Switzerland
France	New Zealand	United Kingdom
Germany, F.R.	Norway	U.S.A.
India	Philippines	U.S.S.R.

No member body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendation R 1567-1970, of which it constitutes a technical revision.

Printed in Switzerland

Denture base resin

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 planned 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. This latter organization undertook the Secretariat responsibilities of the working group which prepared this International Standard.

Of the various changes introduced in this revision, the more significant are the inclusion of a translucency test, the measurement of solubility and sorption by mass per unit volume instead of surface area, and the permitted use of alternative testing apparatus in both the transverse strength and colour stability tests.

1 SCOPE

This International Standard gives a classification of, and specifies requirements for, denture base resins, excluding the "pour" type auto-polymerizing resins, together with the test methods to be employed to determine compliance with these requirements.

2 FIELD OF APPLICATION

This International Standard applies to the following denture base resins :

a) poly(acrylic acid esters);

b) poly(substituted acrylic acid esters);

c) poly(vinyl esters);

d) polystyrene;

e) copolymers or mixtures of the foregoing.

3 CLASSIFICATION

Denture base resin covered by this International Standard shall be of the following types and classes :

– Type I : Heat-processing resins

  Class 1 : Powder and liquid

  Class 2 : Plastic cake

  Class 3 : Thermoplastic blank or powder

– Type II : Auto-polymerizing resins (excluding "pour" resins)

  Class 1 : Powder and liquid

4 REQUIREMENTS

4.1 Liquid

The liquid shall consist essentially of monomeric material appropriate to the powder. It shall be clear and free of deposits or sediment.

The liquid shall be contained in a dark-coloured bottle or suitable opaque container.

4.1.1 *Thermal stability*

The liquid shall show no thickening or discoloration when compared with the original sample, after heating to a temperature of 60 ± 2 °C for 24 h in a closed container in the absence of light.

4.2 Solid components, freedom from contamination

The solid or semi-solid components shall be free of extraneous material, such as dirt or lint, that could adversely affect the appearance or properties of the processed resin.

4.3 Unprocessed resin, packing plasticity

When class 1 and class 2 resins are subjected to the test procedure specified in 7.1, at the initial and final packing times recommended by the manufacturer, they shall be capable of being intruded into at least two holes of the die (see figure 1) to a depth of not less than 0,5 mm.

When prepared according to the manufacturer's instructions, class 3 resins shall produce a satisfactory transverse specimen plate (see figure 3) with well-defined edges.

4.4 Polymerized resin

4.4.1 *Quality*

The resin, when processed according to the manufacturer's instructions, shall produce a satisfactory denture base.

4.4.2 *Surface properties*

When processed in the manner and against materials recommended by the manufacturer, specimen plates prepared according to the method specified in 7.3.1 shall have a smooth, hard, glossy surface free from bloom that cannot be readily removed. When polished by conventional dental methods, the resin shall present a smooth surface having a high gloss.

4.4.3 *Toxicity*

The material shall not contain ingredients in sufficient concentration to cause a toxic reaction when used in accordance with the manufacturer's instructions.

The instructions shall include any necessary precautions required when handling the materials prior to polymerization.

4.4.4 *Colour*

The colour of the processed resin shall be as specified by the purchaser and, where applicable, it shall match the manufacturer's shade guide.

Coloured resin shall be translucent and evenly coloured or mottled; clear resin shall be clear and colourless.

Where the manufacturer indicates that separating media other than tinfoil may be used, the colour and general appearance of the surface of the resin processed against such media and polished by conventional dental methods, shall not differ from that of the resin processed against tinfoil and polished in a similar manner.

4.4.5 *Translucency*

The shadow of a 10 mm diameter opaque disc held against a polished transverse test plate formed as specified in 7.3.2 and illuminated with a 40 W frosted electric light bulb placed 50 cm from the disc, shall be visible from the opposite side of the plate.

4.4.6 *Freedom from porosity*

The test specimens prepared in the manner specified in 7.3.2 shall not show bubbles or voids when viewed without magnification.

4.4.7 *Sorption*

When the processed resin is tested in the manner specified in 7.2.3, the increase in mass per unit volume shall not exceed 32 $\mu g/mm^3$.

4.4.8 *Solubility*

When the processed resin is tested in the manner specified in 7.2.4, the loss in mass per unit volume shall not exceed 1,6 $\mu g/mm^3$.

4.4.9 *Transverse deflection*

When determined in the manner specified in 7.3, the transverse deflection of the processed resin shall meet the requirements of the table. The transverse breaking force shall be not less than 55 N for type I resins and not less than 50 N for type II resins.

TABLE – **Transverse deformation**

Force increment N	**Deformation** mm	
	min.	max.
15,0 to 35,0	1,0	2,5
15,0 to 50,0	2,0	5,0

4.4.10 *Colour stability*

When tested in accordance with 7.4, the test specimen shall not show more than a slight change in colour (perceptible with difficulty).

4.4.11 *Manufacturer's instructions*

Adequate and accurate instructions for storing, preparing and processing the resin shall accompany each package. These shall include, as applicable :

a) the powder-liquid ratio (mass per unit volume);

b) the time, temperature and procedure to properly mix or prepare the material for packing;

c) the working time over which packing may proceed;

d) the temperature of the flask when it is packed;

e) the detailed time-temperature cycle for heating, processing, cooling and deflasking the resin.

Except for class 3 resins, the instructions shall be such that they can be carried out with the equipment generally available in a dental laboratory.

Instructions for repair of the material shall be included.

5 SAMPLING

The method of procurement and amount of resin needed for testing shall be the subject of agreement between the interested parties.

6 PREPARATION OF TEST SPECIMENS

6.1 Ambient conditions

The test specimens shall be prepared at an ambient temperature of 23 ± 2 °C and relative humidity of 50 ± 10 %, except where otherwise specified.

6.2 Method

The specimens shall be prepared from resins proportioned, mixed, packed and processed in accordance with the manufacturer's instructions accompanying the package.

If a range of times is given, the median shall be used, except where otherwise specified.

If equipment other than that normally used in the dental laboratory is required for preparing class 3 specimens, such specimens shall be provided by the manufacturer or suitable equipment shall be made available.

7 PHYSICAL TESTS

7.1 Packing plasticity

7.1.1 *Apparatus*

7.1.1.1 Perforated brass die, of the dimensions shown in figure 1, the perforations being 0,75 ± 0,05 mm diameter.

7.1.1.2 Glass plate, approximately 60 mm × 60 mm × 5 mm.

7.1.1.3 Weight, capable of applying a force of 50,0 N.

7.1.2 Test conditions

Maintain the perforated brass die (7.1.1.1) and glass plate (7.1.1.2) within ± 2 °C of the packing temperature specified by the manufacturer. If a packing temperature is not specified, carry out the test at ambient temperature.

7.1.3 Procedure

Prepare a sample of resin of mass 8 to 10 g in accordance with the manufacturer's instructions. Immediately prior to the recommended initial packing time, shape to a thickness of approximately 5 mm, place on the upper surface of the perforated brass die and cover with a sheet of regenerated cellulose or polyethylene film. At the recommended packing time, carefully place the glass plate and weight (7.1.1.3) on top. Ten minutes later, remove the weight. When the material is firm, measure the depth of intrusion into each hole with a depth gauge to a precision of 0,2 mm. Repeat the test at the maximum working time recommended by the manufacturer.

Report the number of holes penetrated to a depth of not less than 0,5 mm.

7.2 Water sorption and solubility

7.2.1 *Apparatus*

7.2.1.1 Stainless steel mould and cover, of the dimensions shown in figure 2, mounted in gypsum in separate halves of a denture flask.

7.2.1.2 Two desiccators, containing silica gel freshly dried at 130 °C or thoroughly dry anhydrous calcium sulphate ($CaSO_4$), one maintained at ambient temperature and the other at 37 ± 2 °C.

7.2.2 *Preparation of test specimens*

To prepare the test specimen disc, mix the resin, pack into the mould (7.2.1.1), and process according to the manufacturer's instructions. The disc shall have a diameter of 50 ± 0,1 mm and a thickness of 0,5 ± 0,05 mm. Ensure that the top and bottom surfaces are flat. Prepare two specimens.

If class 3 resins require special equipment, the manufacturer shall provide specimens or make the equipment available.

7.2.3 *Water sorption test*

7.2.3.1 Procedure

Dry the specimen discs, prepared as specified in 7.2.2, in the desiccator at 37 ± 2 °C (7.2.1.2) for 24 h, transfer to the desiccator at ambient temperature for 1 h, and weigh with a precision of ± 0,000 2 g. Repeat this cycle until a constant mass, to be called the "conditioned mass", is attained, that is, until the loss in mass of each specimen disc is not more than 0,000 2 g between successive weighings.

Immerse the specimen discs in distilled water at 37 ± 1 °C for 7 days. After this time, remove from the water with tweezers, wipe with a clean dry hand-towel until free from visible moisture, wave in the air for 15 s and weigh 1 min after removal from the water.

Calculate the volume of the specimen from the diameter and the mean of five thickness measurements, one taken at the centre and four at equally spaced positions around the circumference.

7.2.3.2 Expression of results

Calculate the value for the water sorption for each disc, in micrograms per cubic millimetre, from the formula

$$\frac{m_2 - m_1}{V}$$

where

m_1 is the "conditioned mass", in micrograms, of the disc;

m_2 is the mass, in micrograms, of the disc after immersion;

V is the volume of the disc in cubic millimetres.

Round off the average of the two determinations to the nearest microgram per cubic millimetre.

7.2.4 *Solubility test*

7.2.4.1 Procedure

After the final weighing described in 7.2.3.1, recondition the discs to constant mass in the desiccators at 37 ± 2 °C and at ambient temperature, as described in 7.2.3.1. Retain these discs for use in the colour stability test (see 7.4).

7.2.4.2 Expression of results

Calculate the soluble matter per unit volume, leached out during immersion, in micrograms per cubic millimetre, for each disc, from the formula

$$\frac{m_1 - m_3}{V}$$

where

m_1 and V are as defined in 7.2.3.2;

m_3 is the reconditioned mass, in micrograms, of the disc.

Round off the average of the two determinations to the nearest 0,1 $\mu g/mm^3$.

7.3 Transverse deflection

7.3.1 *Preparation of test specimen plates*

7.3.1.1 Apparatus

a) **Denture flask,** capable of accommodating the test specimen plate (see figure 3) so that the corners are not less than 5 mm from the walls of the flask.

b) **Model of the specimen plate,** in metal or resin.

c) **Equipment for processing the resin.**

7.3.1.2 Preparation of the mould

Invest the model of the specimen plate [7.3.1.1 b)] in the denture flask [7.3.1.1 a)] and remove 1 h after the investing material has set. Use gypsum for class 1 and class 2 resins. Cover the specimen plate area of the mould with the manufacturers recommended separating agent.

Prepare class 3 specimens according to the manufacturer's instructions but if special equipment is required, the manufacturer shall supply the specimen plates or make the equipment available.

7.3.1.3 Procedure

Prepare the resin, pack and process in accordance with the manufacturer's instructions. Prepare two specimen plates using separate mixes.

7.3.2 *Preparation of test specimens*

Upon removal from the flasks, examine the specimen plates for compliance with 4.4.2, 4.4.4 and 4.4.5. Prepare specimen strips at least 64 mm long, 10,00 ± 0,03 mm broad, and 2,50 ± 0,03 mm thick from these plates after sawing each plates lengthwise into three equal strips. Machine the strips on the edges and equally from both moulded surfaces so that the dimensions are slightly oversize. Take care to avoid overheating the specimen. Wet-grind all faces and edges smooth and flat on grade 400 emery paper to the required breadth and thickness.

After examining the specimens for compliance with 4.4.6, store in water at a temperature of 37 ± 1 °C for 50 ± 2 h.

Prepare six specimen strips.

7.3.3 *Determination of transverse deflection*

7.3.3.1 Apparatus

a) **Testing machine,** calibrated, with a constant cross-head speed of 5 ± 1 mm/min and equipped with a device for measuring the deflection of the centre of the specimen to within 0,025 mm. Any load exerted by the deflection-measuring device shall be accounted for when calibrating the machine.

b) **Transverse testing rig,** consisting of a central loading plunger and two supports with highly polished cylindrical surfaces of diameter 3,2 mm and length at least 10,5 mm. The supports shall be parallel to within 0,1 mm and perpendicular to the longitudinal centre line. The distance between the centres of the supports shall be 50 ± 0,1 mm, and the loading plunger midway between the supports to within 0,1 mm. Means shall be provided to prevent misalignment of the specimen on the supports.

c) **Water bath,** capable of maintaining the specimen wet at a temperature of 37,0 ± 1,0 °C during testing.

7.3.3.2 Procedure

Take a specimen strip, prepared in accordance with 7.3.2, from storage and immediately lay the flat surface symmetrically on the supports of the rig [7.3.3.1 b)] immersed in the water bath [7.3.3.1 c)] maintained at a temperature of 37 ± 1,0 °C. Allow the specimen to come to equilibrium with the water bath temperature.

Increase the force on the loading plunger, from zero, uniformly at a constant rate of 5 ± 1 mm/min until the specimen breaks.

Record and note the deflection to the nearest 0,05 mm, for the applied forces of 15 N, 35 N and 50 N.

Report the average deflection to the nearest 0,05 mm, for the six specimens for the forces from 15 to 35 N and from 15 to 50 N as the transverse deflections to be compared with the requirements of the table in 4.4.9.

Report the average force at break of the six specimens tested to the nearest 0,5 N for compliance with 4.4.9.

7.4 Colour stability to light

7.4.1 *Apparatus*

7.4.1.1 Turntable with an aluminium disc of diameter at least 200 mm, operating at 33 rev/min.

7.4.1.2 Base and stand arrangement, allowing the turntable and disc to be centred under the bottom of a sunlamp as shown in figure 4.

7.4.1.3 Light source, consisting of a combination tungsten filament mercury-arc enclosed in corex D or other glass which has a low transmission below 280 nm. The bulb shall be mounted in a reflector as shown in figure 4 a). The bulb shall have been aged for not less than 50 h before use and shall not be used after 400 h operation.

7.4.2 *Procedure*

Use the discs used for the determination of solubility (7.2.4) cut in half as specimens for the test. Position one half of each disc such that the upper surface is 5 mm above the turntable (7.4.1.1) and with its mass-centre 60 mm from the centre of the aluminium disc.

Expose the half-discs to the radiation of a Westinghouse RS 275 W sunlamp, or suitable equivalent sunlamp, which has been used for not less than 50 h and not more than 400 h. Position the lamp above the turntable such that with the Blak-ray J 221 Long-wave ultraviolet meter sensor at the specimen position, the ultraviolet radiation (the difference between the total radiation and the filtered radiation measurements) is 1 700 ± 1 000 μW per centimetre. Check the ultraviolet radiation emission at the beginning and end of each run. Maintain the temperature at the specimen position at 37 ± 5 °C, measured by a mercury thermometer. Adjust the temperature if necessary by the use of a fan to lower the temperature or by enclosing the apparatus to increase the temperature.

With the turntable rotating at 33 min^{-1}, expose the half-discs for 24 h. Compare the exposed half-discs with half-discs that have not been exposed, under northern daylight, viewed at a distance of 600 mm for 5 s.

The exposed specimen should not show more than a slight change in colour which is perceptible only with difficulty.

8 PACKAGING

The material shall be supplied in properly sealed containers made of materials which shall neither contaminate nor permit contamination of the contents. The containers shall be packaged so as to prevent damage or leakage during transit and storage.

9 MARKING OF PACKAGES AND CONTAINERS

9.1 Packages

Each outer package shall be clearly marked with the following information :

a) manufacturer's registered name or mark;

b) the contents of the package;

c) type, class and colour;

d) recommended conditions of storage;

e) flash-point of liquid in the package;

f) shelf life, where this is limited;

g) hazard warning, where appropriate, for toxic, hazardous, inflammable, or irritating characteristics.

9.2 Individual containers

9.2.1 *All containers*

Each container shall be clearly marked with the following information :

a) manufacturer's registered name or mark;

b) batch number;

c) mass of solids (in grams) and volume of liquid (in millilitres);

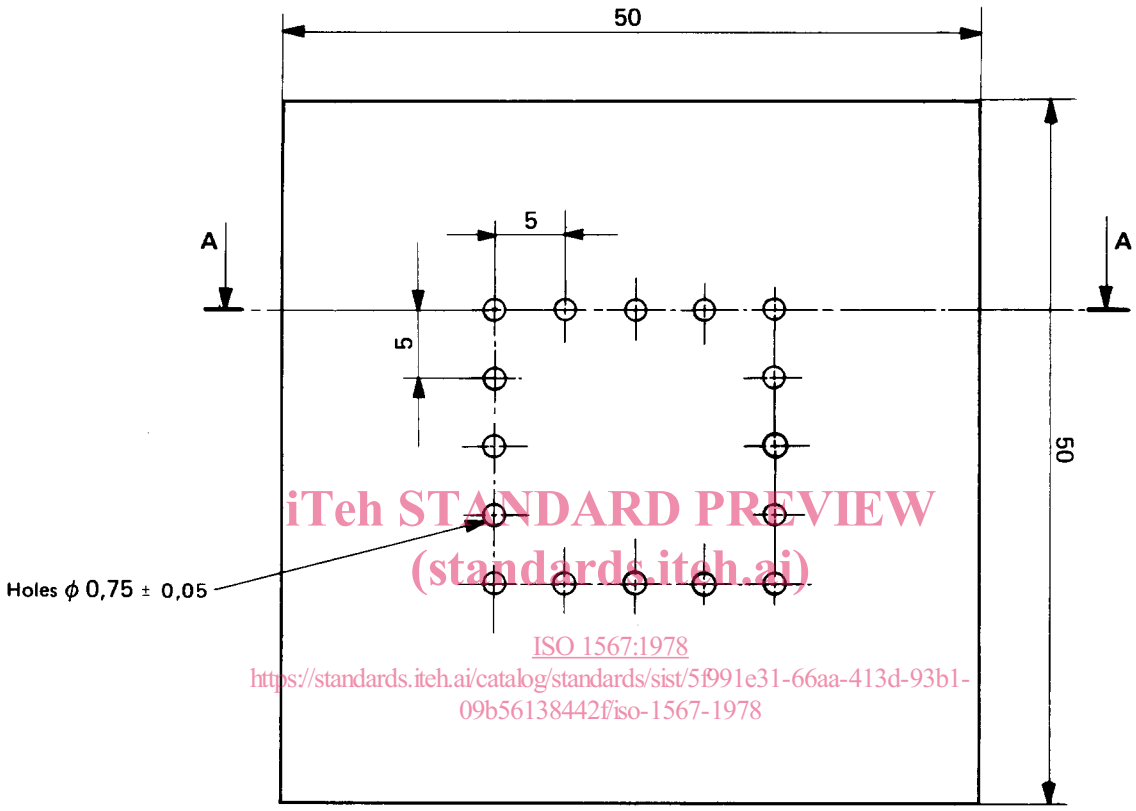
d) any special precaution needed in handling the material particularly with regard to inflammability;

e) date of manufacture or expiry date.

9.2.2 *Containers of powder only*

The colour shall be clearly marked on each container of powder.

Dimensions in millimetres



Section A-A

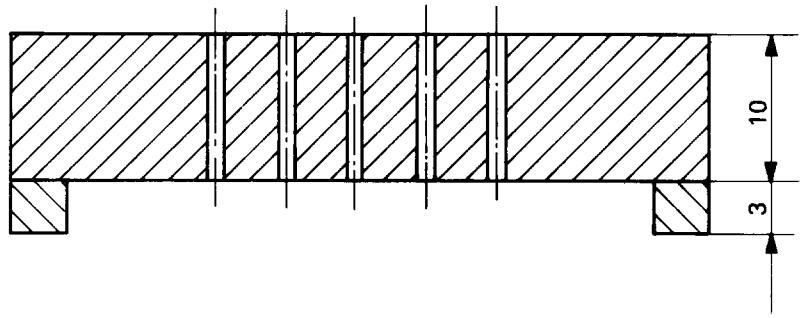


FIGURE 1 – **Brass die for packing test** (see 7.1.1.1)

Dimensions in millimetres

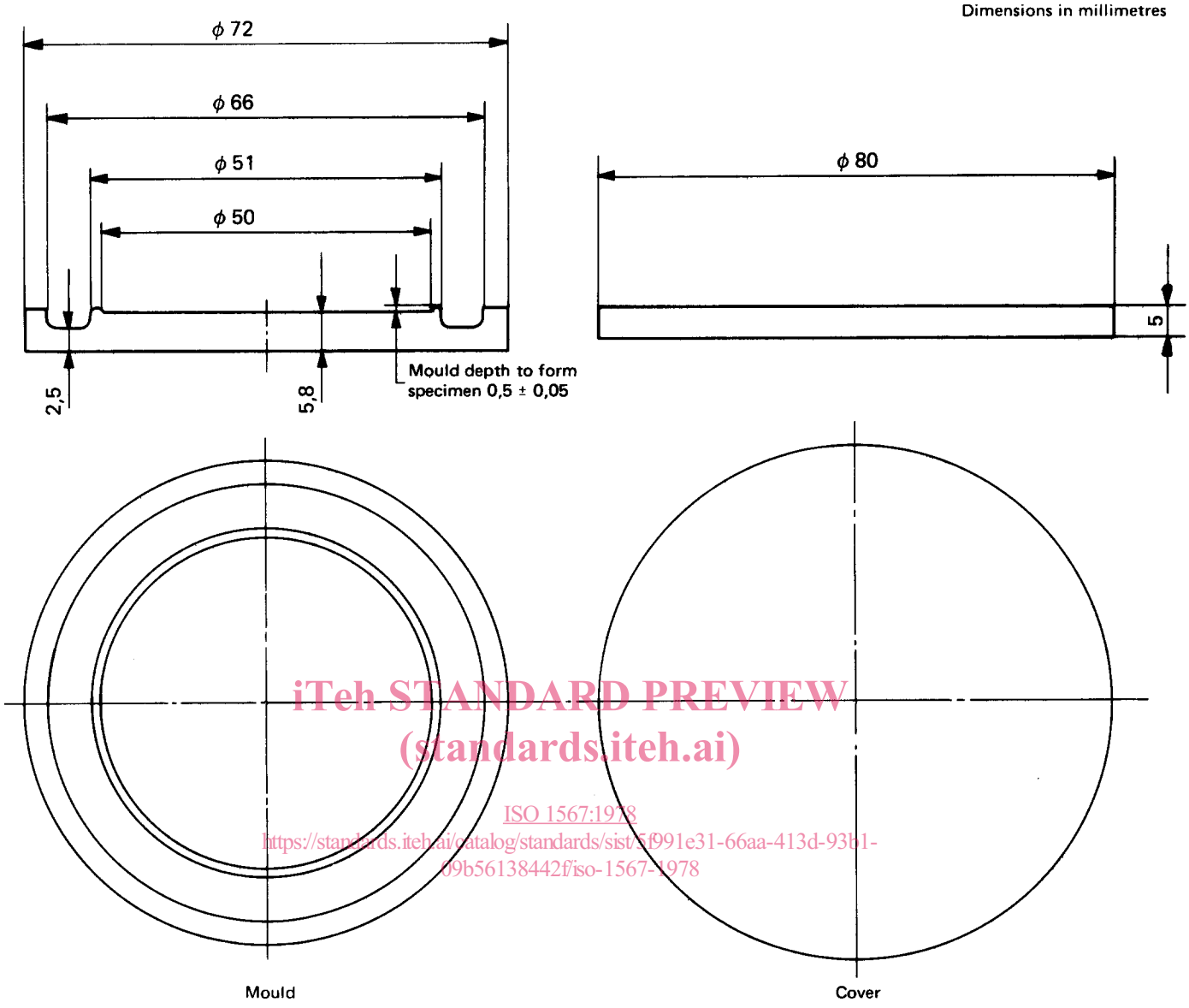


FIGURE 2 — **Stainless steel mould and cover for sorption and solubility specimens** (see 7.2.1.1)

Dimensions in millimetres

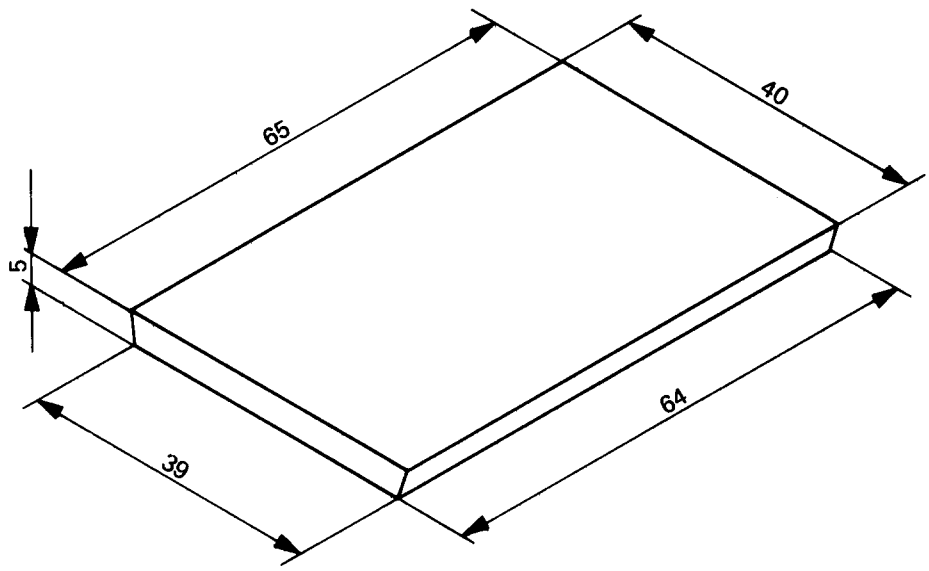


FIGURE 3 — **Test specimen plate** (see 7.3.1.1)