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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Dentistry — Denture base polymers

Art dentaire — Polymères pour base de prothèses dentaires

Reference number
ISO 1567 : 1988 (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 approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 1567 was prepared by Technical Committee ISO/TC 106, *Dentistry*.

This second edition cancels and replaces the first edition (ISO 1567 : 1978), of which it constitutes a minor revision.

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Introduction

This revised edition of ISO 1567 has been prepared in the light of experience gained in the use of the first edition (ISO 1567-1978). The main changes are found in the test procedures and the calculation of water sorption and solubility, and in the test procedures for colour stability to light. A requirement for compatibility (bonding) to synthetic polymer teeth has also been included. Accordingly, most autopolymerized (type II polymers) and thermoplastic materials (type III) are required to have the following statement on a label : "WILL NOT BOND TO SYNTHETIC POLYMER TEETH".

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

Dentistry — Denture base polymers

1 Scope

1.1 This International Standard gives a classification of, and specifies requirements for, denture base polymers; it also specifies the test methods to be used to determine compliance with these requirements.

This International Standard applies to the following denture base polymers :

a) poly(acrylic acid esters);

b) poly(substituted acrylic acid esters);

c) poly(vinyl esters);

d) polystyrene;

e) rubber modified poly(methacrylic acid esters);

f) polycarbonates;

g) polysulfones;

h) copolymers or mixtures of the polymers listed in a) to g).

1.2 Although this International Standard does not require manufacturers to declare details of the composition, attention is drawn to the fact that some national or international authorities require details to be provided to them.

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 listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3336 : 1977, *Dentistry — Synthetic resin teeth.*

ISO/TR 7405 : 1984, *Biological evaluation of dental materials.*

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

ISO 8601 : 1988, *Data elements and interchange formats — Information interchange — Representation of dates and times.*

3 Classification

Denture base polymers covered by this International Standard are of the following types and classes :

Type I : Heat-processed polymers

— Class 1 : Powder and liquid

— Class 2 : Plastic cake

Type II : Autopolymerized polymers

— Class 1 : Powder and liquid

— Class 2 : Powder and liquid pour-type resins

Type III : Thermoplastic blank or powder to form the denture base

4 Requirements

4.1 Composition

See 1.2 for guidance on composition.

4.2 Liquid component

4.2.1 General

The liquid shall consist essentially of monomeric material compatible with the powder. It shall be clear and free of deposit or sediment when inspected (see 7.1).

4.2.2 Thermal stability

The liquid shall show no thickening or discoloration when compared (see 7.1) with the original sample, after being maintained at 60 °C ± 2 °C for 24 h in a closed container in the absence of light.

4.3 Solid components

The solid or semi-solid components shall be free of extraneous material when inspected (see 7.1).

4.4 Unprocessed resin, packing plasticity

When type I, class 1 and 2 resins are tested in accordance with 7.2, 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 (see 7.1).

When prepared in accordance with the manufacturer's instructions, type III polymers shall produce a transverse specimen plate (see figure 3) with well defined edges.

4.5 Polymerized material

4.5.1 Quality

When processed in accordance with the manufacturer's instructions, the polymer shall produce a denture base complying with the requirements laid down in this International Standard.

4.5.2 Surface properties

When processed in the manner and against materials recommended by the manufacturer, specimens, prepared in accordance with 7.3.2 and 7.4.1, shall have a smooth, hard, glossy surface. The test specimens for sorption and solubility shall retain their form without distortion after processing. When polished by conventional dental methods, the polymer shall present a smooth surface having a high gloss (see 7.1).

4.5.3 Biocompatibility

See the Introduction (p. iii) for guidance on biocompatibility.

4.5.4 Colour

Samples shall be prepared in accordance with 7.4.1 and inspected in accordance with 7.1.

Coloured processed polymer shall be of the colour stated by the manufacturer and shall match the manufacturer's shade guide, if supplied. It shall be translucent (see 4.5.5 and 7.4.2) and evenly pigmented or mottled (fibred).

Clear processed polymer shall be clear and colourless.

If the manufacturer's instructions (see 8.3) allow the use of separating media other than tin foil, the colour and general appearance of the surface of the polymer processed against such media and polished by conventional dental methods shall not differ from that of the polymer processed against tin foil and polished in a similar manner.

4.5.5 Translucency

When tested in accordance with 7.4.2, the illuminated opaque disc shall be visible from the opposite side of the test specimen plate (see 7.1).

4.5.6 Freedom from porosity

When prepared in accordance with 7.4.3, specimen strips shall not show voids (see 7.1) when viewed without magnification (7.4.4).

4.5.7 Sorption

When the processed polymer is tested in accordance with 7.3, the increase in mass per unit volume (water sorption) shall not exceed 32 $\mu g/mm^3$ for either type I, type II or type III materials.

4.5.8 Solubility

When the processed polymer is tested in accordance with 7.3, the loss in mass per unit volume (soluble matter) shall not exceed 1,6 $\mu g/mm^3$ for type I and type III materials, and shall not exceed 8,0 $\mu g/mm^3$ for type II materials.

4.5.9 Transverse deflection

When determined in accordance with 7.4.5, the transverse deflection of the processed polymer shall meet the requirements specified in table 1 when tested in water at 37 °C ± 1 °C. The transverse breaking force shall be not less than 55 N for type I and type III materials and not less than 50 N for type II materials.

Table 1 — Transverse deflection

Force increment N	Deflection mm	
	min.	max.
Between 15 and 35	1	2,5
Between 15 and 50	2	5

4.5.10 Colour stability

Test specimens shall not show (see 7.1) more than a slight change in colour, perceptible with difficulty, when tested in accordance with 7.5.

4.5.11 Bonding to synthetic polymer teeth

For denture base polymers intended for use with synthetic polymer teeth, either

— the polymer shall be capable of bonding to polymer teeth complying with the requirements of ISO 3336, or

— the outer package and containers shall be marked with a statement of the inability to bond [see 8.2.1 l) and 8.2.2 l)], or

— the outer package and containers shall contain information about special treatments necessary to achieve adequate bonding [see 8.2.1 m) and 8.2.2 m)].

5 Sampling

The test sample shall consist of a retail package or packages, containing sufficient material to carry out the specified tests, plus an allowance for any necessary repetition of the tests. If more than one package is required, the material in both shall be from the same batch.

6 Preparation of test specimens

6.1 Ambient conditions

The test specimens shall be prepared at 23 °C ± 2 °C and at a relative humidity of 50 % ± 10 %, except where otherwise specified by the manufacturer.

6.2 Procedure

Prepare the specimens from resins proportioned, mixed, packed and processed in accordance with the manufacturer's instructions (see 8.3).

7 Test methods

7.1 Inspection requirements

Visually inspect without magnification the test specimens to determine compliance with the requirements laid down in 4.2, 4.3, 4.4, 4.5.2, 4.5.4, 4.5.5, 4.5.6, 4.5.10 and clause 8. Inspect for colour (see 4.5.4) and colour stability (see 4.5.10) in accordance with ISO 7491.

7.2 Packing plasticity

7.2.1 Apparatus

7.2.1.1 Perforated brass die, having the dimensions shown in figure 1, with perforations having a diameter of 0,75 mm ± 0,05 mm.

7.2.1.2 Glass plate,
60 mm ± 5 mm × 60 mm ± 5 mm × 5 mm ± 1 mm.

7.2.1.3 Weight, capable of applying a force of 50 N.

7.2.2 Test conditions

The perforated brass die (7.2.1.1) and glass plate (7.2.1.2) shall be maintained at ambient temperature except where otherwise specified by the manufacturer.

7.2.3 Procedure

Prepare a sample of resin with a mass of 8 g to 10 g in accordance with the manufacturer's instructions. Immediately prior to the recommended initial packing time [see 8.3c)], 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 (7.2.1.3) on top. After 10 min, remove the weight. When the material is firm, record the depth of penetration with a precision of 0,2 mm by measuring from the lower surface of the brass die to the intruded polymer and subtracting this from the thickness of the brass die. Repeat the test at the maximum working time recommended by the manufacturer [see 8.3c)].

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

7.3 Water sorption and solubility

7.3.1 Apparatus and materials

7.3.1.1 Desiccator.

7.3.1.2 Rack to keep the specimens parallel and separated.

7.3.1.3 Oven or incubator, capable of being maintained at 37 °C ± 1 °C.

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

7.3.1.5 Sheet of polyester film, having a thickness of 50 µm ± 25 µm and a diameter of 80 mm.

7.3.1.6 Silica gel, freshly dried for 5 h at 130 °C.

7.3.1.7 Water, prepared in one of the following ways :

a) by multiple distillation;

b) by distillation followed by deionization;

c) by distillation followed by reverse osmosis.

7.3.2 Preparation of test specimen discs

Three specimens shall be prepared.

Mix the resin and pack the mixture into the mould (7.3.1.4) with the polyester film (7.3.1.5) against the steel cover of the mould. Process the mixture in accordance with the manufacturer's instructions, but retain the polyester film during the processing cycle.

Check to ensure that each specimen disc has a diameter of 50 mm ± 1 mm and a thickness of 0,5 mm ± 0,1 mm and that the top and bottom surfaces are flat.

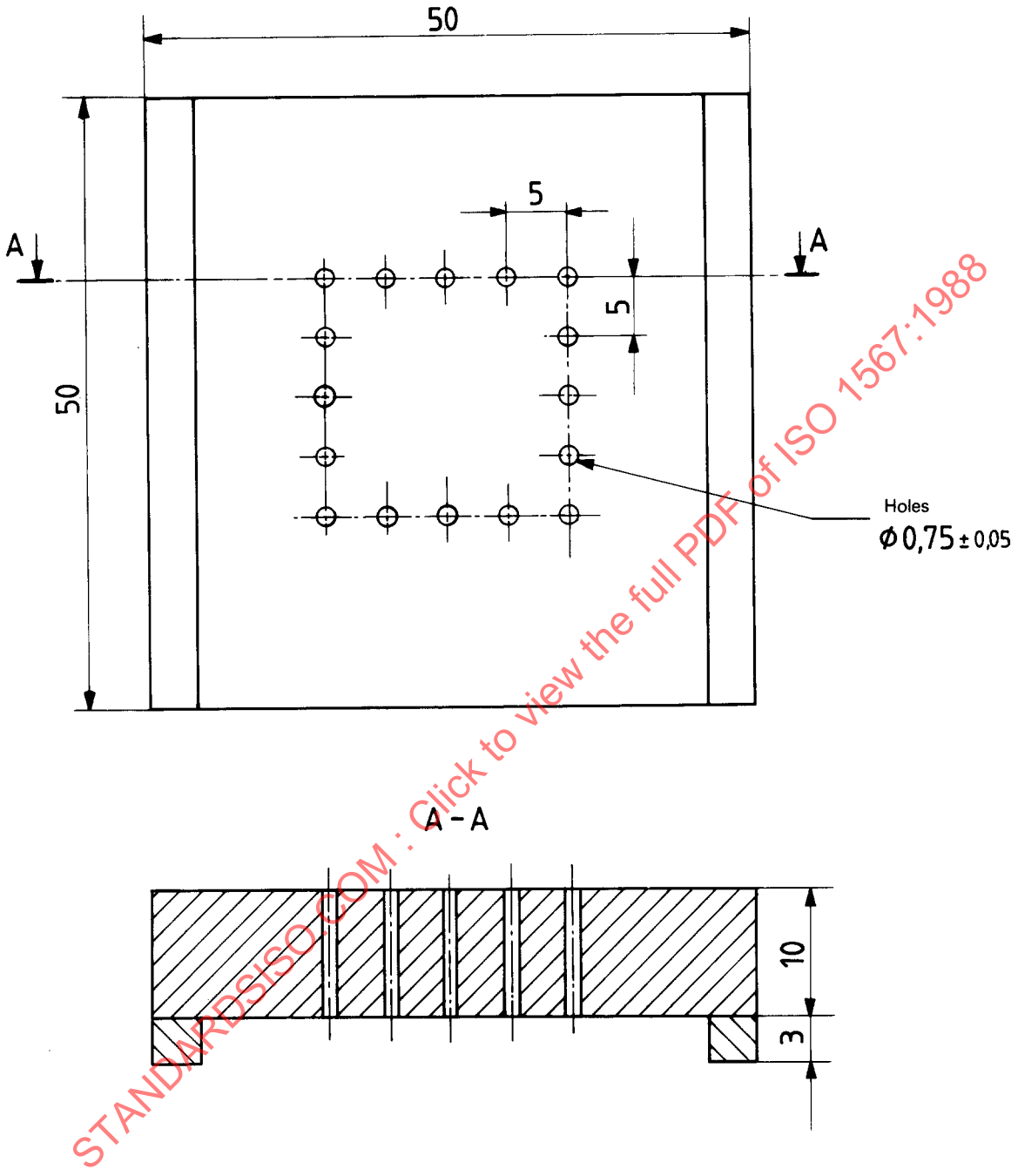
NOTE — If type III polymers and capsulated resins require special equipment, the manufacturer should provide specimens and/or make the equipment available.

7.3.3 Procedure

7.3.3.1 Place the specimens in the rack (7.3.1.2) inside the desiccator (7.3.1.1). Store the desiccator in the oven (7.3.1.3) at 37 °C ± 1 °C for 23 h and then stand it at ambient temperature for 1 h. Weigh each specimen at 23 °C ± 1 °C to a precision of 0,000 2 g and only open the desiccator for the shortest possible period when removing and replacing specimens. After all the specimens have been weighed, replace the silica gel in the desiccator with freshly dried gel (7.3.1.6).

7.3.3.2 Repeat the cycle described in 7.3.3.1 until a constant mass, m_1, to be called the "conditioned mass", is reached, i.e. until the loss in mass of each specimen disc is not more than 0,000 2 g between successive weighings.

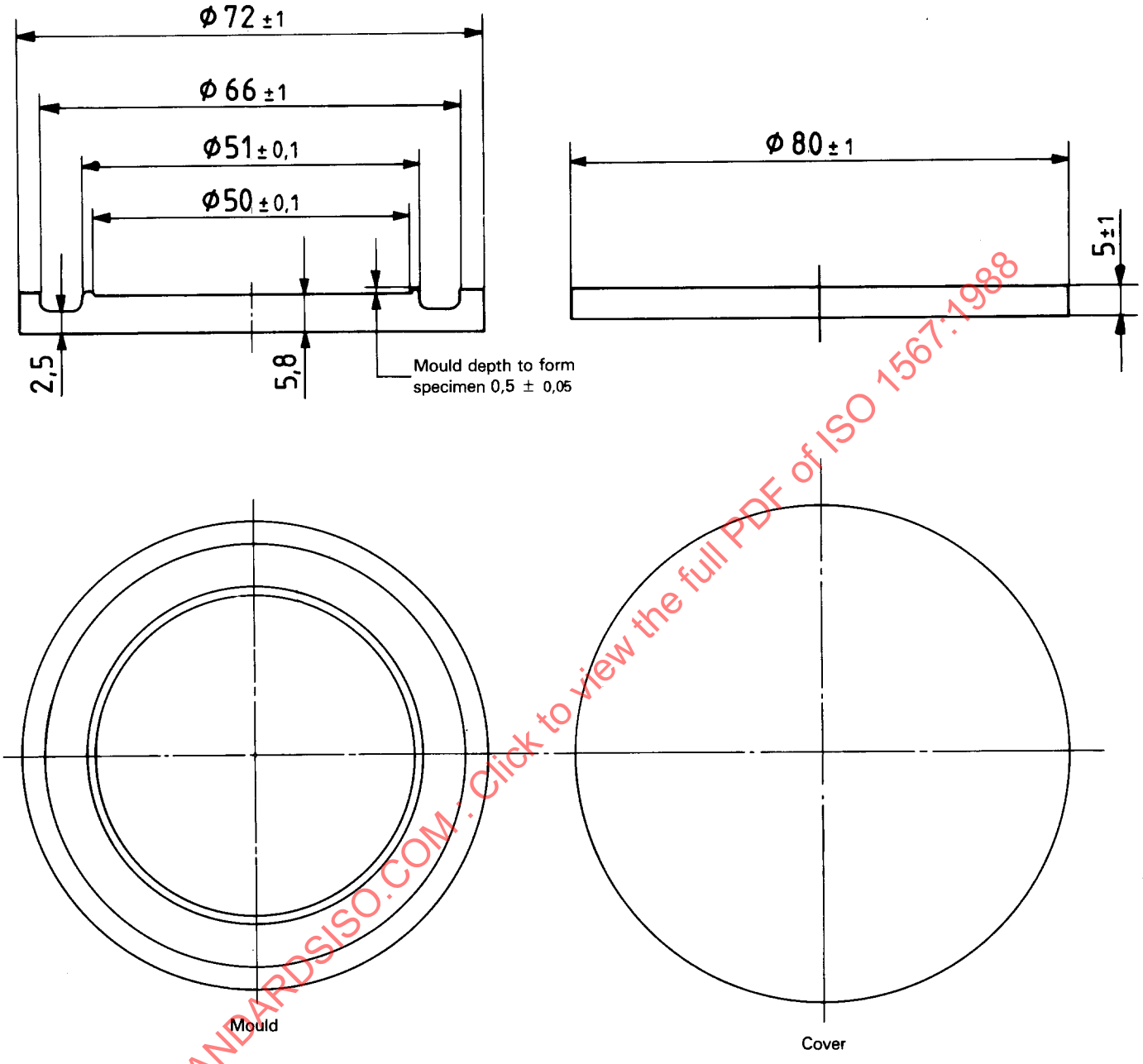
Dimensions in millimetres



NOTE — Dimensional tolerances not specified shall be ± 1 mm.

Figure 1 — Brass die for packing plasticity test (see 7.2.1.1)

Dimensions in millimetres



NOTE — Dimensional tolerances not specified shall be ± 0,2 mm.

Figure 2 — Stainless steel mould and cover for sorption and solubility specimens (see 7.3.1.4)

7.3.3.3 Immerse the specimen disc in the water (7.3.1.7) at 37 °C ± 1 °C for 7 days. After this time, remove the disc 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 with a precision of 0,000 2 g. Record this mass as m_2.

7.3.3.4 After this weighing, recondition the discs to constant mass in the desiccator as described in 7.3.3.2. Record the mass of the "reconditioned" discs as m_3.

NOTE — It is essential that the same conditions be applied as for the first drying process (7.3.3.1), using the same number of specimens and freshly dried silica gel in the desiccator.

7.3.3.5 Calculate the volume, V, of the specimen from the diameter and the mean of five thickness measurements, one taken at the centre and four at equally spaced locations around the circumference. The measurement of diameter and thickness of the specimens shall be made before immersion in water.

7.3.4 Expression of results

7.3.4.1 Calculate the value for the water sorption, w_{sp}, for each disc, expressed in micrograms per cubic millimetre, from the following equation :

$$w_{sp} = \frac{m_2 - m_3}{V}$$

where

m_2 is the mass of the disc, in micrograms, after immersion (see 7.3.3.3);

m_3 is the reconditioned mass of the disc, in micrograms (see 7.3.3.4);

V is the volume of the disc, in cubic millimetres (see 7.3.3.5).

7.3.4.2 Calculate the soluble matter per unit volume, w_{sl}, leached out during immersion, expressed in micrograms per cubic millimetre for each disc, from the following equation :

$$w_{sl} = \frac{m_1 - m_3}{V}$$

where

m_1 is the "conditioned mass" of the disc, in micrograms (see 7.3.3.2);

m_3 and V are as given in 7.3.4.1.

7.3.4.3 Round off the average of the determinations to the nearest microgram per cubic millimetre for the water sorption; for the water solubility, round off to the nearest 0,1 µg per cubic millimetre.

Retain two of these discs for use in the colour stability test (see 7.5).

7.4 Translucency, freedom from porosity and transverse deflection

7.4.1 *Preparation of test specimen plate*

7.4.1.1 Apparatus

7.4.1.1.1 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.

7.4.1.1.2 Model of the specimen plate, in metal or polymer.

7.4.1.1.3 Equipment for processing the resin, including gypsum or hydrocolloid [see 8.3d)].

7.4.1.1.4 Standard metallographic grinding paper, with a grain size of approximately 30 µm.[1)]

7.4.1.2 Preparation of the mould

Invest the model of the specimen plate (7.4.1.1.2) in the denture flask (7.4.1.1.1). Prepare type II, class 2 and type III and capsulated materials in accordance with the manufacturer's instructions.

NOTE — If special equipment is required, the manufacturer should supply the specimen plates or make the equipment available.

7.4.1.3 Procedure

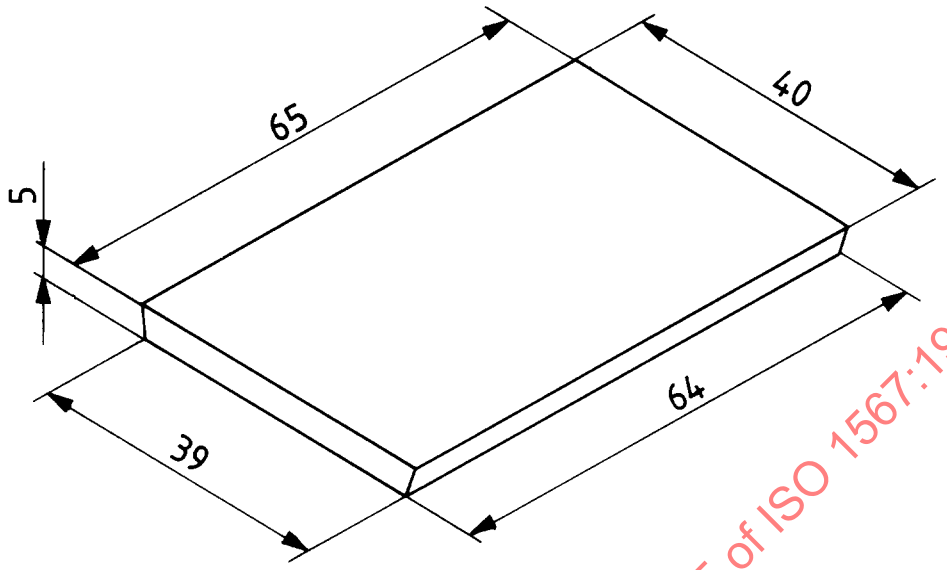
Prepare the resin, pack and process in accordance with the manufacturer's instructions. Two specimen plates shall be prepared using separate mixes. Remove the specimen plates from the flasks and examine them for compliance with 4.5.2, 4.5.4, 4.5.5 and 7.4.2.

7.4.2 Translucency test procedure

Place an opaque disc having a diameter of 10 mm against one side of a polished transverse test specimen plate and illuminate with a 40 W frosted electric light bulb placed 500 mm from the opposite side of the specimen plate. Test for compliance with 4.5.5.

1) Grinding paper with a 500 FEPA (Fédération européenne des produits abrasifs : European Federation for Abrasive Products) standard number is recommended; however any other paper meeting the same requirements is suitable.

Dimensions in millimetres



NOTE — Dimensional tolerances shall be ± 1 mm.

Figure 3 — Model of the test specimen plate (see 7.4.1.1.2)

7.4.3 Preparation of test specimen strips

Six specimen strips shall be prepared.

Saw each plate lengthwise into three equal strips, 64 mm long, 10 mm ± 0,03 mm wide and 2,5 mm ± 0,03 mm in depth. 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 the metallographic grinding paper (7.4.1.1.4) to the required width and thickness.

7.4.4 Freedom from porosity

Examine the test specimen strips in accordance with 7.1 for compliance with 4.5.6, and then store them in water at a temperature of 37 °C ± 1 °C for 50 h ± 2 h prior to transverse deflection testing.

7.4.5 Transverse deflection

7.4.5.1 Apparatus

7.4.5.1.1 Testing machine, calibrated, with a constant cross-head speed of 5 mm/min ± 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.

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

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

7.4.5.2 Procedure

Take a specimen strip, prepared in accordance with 7.4.3 and stored in accordance with 7.4.4, from water storage and immediately lay the flat surface symmetrically on the supports of the rig (7.4.5.1.2) immersed in the water bath (7.4.5.1.3) maintained at a temperature of 37 °C ± 1 °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 mm/min ± 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 applied between 15 N and 35 N and between 15 N and 50 N as the transverse deflections to be compared with the requirements specified in table 1.

Report the average breaking force of the six specimens tested to the nearest 0,5 N for compliance with 4.5.9.

7.5 Colour stability

7.5.1 Preparation of test specimens

Two of the discs used in the solubility test (see 7.3) shall be used.

Store one specimen in the dark at ambient temperature. Store the second specimen in water at 37 °C ± 1°C for 24 h prior to exposure.

7.5.2 Procedure

Blank off half of the second specimen disc with aluminium or tin foil and transfer it to the radiation chamber. The specimen shall be immersed in water at 37 °C ± 5 °C when exposed to the radiation for 24 h in accordance with ISO 7491. After exposure, remove the metal foil before colour comparison of the specimens including the unexposed specimen. The colour comparison shall be carried out in accordance with the requirements laid down in 4.5.10 of this International Standard and in accordance with the procedure for colour comparison laid down in ISO 7491.

8 Packaging, marking and instructions to be supplied by manufacturer

8.1 Packaging

The material shall be supplied in properly sealed containers[1] made of materials which neither contaminate nor allow contamination of the contents. The containers shall be packaged so as to prevent damage or leakage during transit and storage. The liquid shall be contained in a dark-coloured bottle or opaque container. An outer package may also be used to present the containers as a single unit.

8.2 Marking of outer packages and containers[1]

8.2.1 Outer packages

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

a) the material's trade- or brand-name;

b) manufacturer's name and address and/or agent in country of sale;

c) the type, class and colour of material and its application given in clear language;

d) the "USE BEFORE" date beyond which the material may not exhibit its best properties. The words "USE BEFORE" or "EXPIRY DATE" followed by the date (year and month), expressed in accordance with ISO 8601;

e) recommended conditions of storage;

f) the specification of the contents, including the number, mass and/or volume of each item;

g) special indications or warnings with regard to toxic, hazardous, flammable or irritating characteristics;

h) where applicable, the flashpoint of liquid in the package;

i) any pharmaceutically active ingredients when present and referred to in the material claim or use;

j) manufacturer's batch reference;

k) the number of this International Standard, i.e. ISO 1567, to indicate conformity;

l) a statement, where applicable (see 4.5.11) : "WILL NOT BOND TO SYNTHETIC POLYMER TEETH";

m) where applicable, information of any special treatment to achieve bonding to synthetic polymer teeth (see 4.5.11).

8.2.2 All containers[1]

All containers shall be clearly marked with the following information :

a) the material's trade- or brand-name;

b) manufacturer's name and address and/or agent in country of sale;

c) the type, class and colour of material and its application given in clear language;

d) the "USE BEFORE" date beyond which the material may not exhibit its best properties. The words "USE BEFORE" or "EXPIRY DATE" followed by the date (year and month), expressed in accordance with ISO 8601;

e) recommended conditions of storage;

f) the specification of the contents, including the number, mass and/or volume of each item;

g) special indications or warnings with regard to toxic, hazardous, flammable or irritating characteristics;

h) where applicable, the flashpoint of the liquid;

i) any pharmaceutically active ingredients when present and referred to in the material claim or use;

j) manufacturer's batch reference;

k) the number of this International Standard, i.e. ISO 1567, to indicate conformity;

l) a statement, where applicable (see 4.5.11) : "WILL NOT BOND TO SYNTHETIC POLYMER TEETH";

m) where applicable, information of any special treatment to achieve bonding to synthetic polymer teeth (see 4.5.11).

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