INTERNATIONAL STANDARD

ISO 20795-1

First edition
2008-08-01

Dentistry — Base polymers —

Part 1: Denture base polymers

Art dentaire — Polymères de base —

Partie 1: Polymères pour base de prothèses dentaires

Reference number
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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 20795-1 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 2, *Prosthodontic materials*.

This first edition cancels and replaces ISO 1567:1999 and Amendment 1:2003.

Significant differences between this first edition of ISO 20795-1 and the third edition of ISO 1567 and Amendment 1 lie with requirements and tests for materials with improved impact resistance.

ISO 20795 consists of the following parts, under the general title *Dentistry — Base polymers*:

— *Part 1: Denture base polymers*

— *Part 2: Orthodontic base polymers*

Introduction

Specific qualitative and quantitative requirements for freedom from biological hazard are not included in this part of ISO 20795, but it is recommended that in assessing possible biological or toxicological hazards, reference be made to ISO 10993-1 and ISO 7405.

Dentistry — Base polymers —

Part 1: Denture base polymers

1 Scope

1.1 This part of ISO 20795 classifies denture base polymers and copolymers and specifies their requirements. It also specifies the test methods to be used in determining compliance with these requirements. It further specifies requirements with respect to packaging and marking the products and to the instructions to be supplied for use of these materials. Furthermore it applies to denture base polymers for which the manufacturer claims that the material has improved impact resistance. It also specifies the respective requirement and the test method to be used.

1.2 Although this part of ISO 20795 does not require manufacturers to declare details of the composition, attention is drawn to the fact that some national or international authorities require such details to be provided.

1.3 This part of ISO 20795 applies to denture base polymers such as those listed below:

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) poly(dimethacrylic acid esters);

i) polyacetals (polyoxymethylene);

j) copolymers or mixtures of the polymers listed in a) to i).

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 463:2006, *Geometrical Product Specifications (GPS) — Dimensional measuring equipment — Design and metrological characteristics of mechanical dial gauges*

ISO 1942, *Dentistry — Vocabulary*

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

ISO 7491:2000, *Dental materials — Determination of colour stability*

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

ISO 22112:2005, *Dentistry — Artifical teeth for dental prostheses*

3 Terms and definitions

For the purposes of this document the terms and definitions given in ISO 1942 and the following apply.

3.1
autopolymerizable materials
products having polymerization initiated by chemical means and not requiring application of temperatures above 65 °C to complete the polymerization

3.2
capsulated material
material consisting of two or more components supplied in a container that keeps them separated until the time they are mixed together and dispensed for use directly from the container

3.3
denture
artificial substitute for missing natural teeth and adjacent tissues, to also include any additions needed for optimum function

3.4
denture base
that part of a denture which rests on soft tissue foundations and to which artificial teeth are added

3.5
heat-polymerizable materials
products requiring application of temperatures above 65 °C to complete polymerization

3.6
immediate container
container that is in direct contact with the denture base materials

3.7
liquid
monomeric liquid to be mixed with polymeric particles to form a mouldable dough or fluid resin mixture used for forming denture bases

3.8
powder
polymeric particles to be mixed with monomeric liquid to form a mouldable dough or fluid resin mixture used for forming denture bases

3.9
outer packaging
labelled container or wrapping within which other containers are packed

3.10
packing
〈of a denture〉 act of filling a denture base mould with a material (using a compression, pour or injection technique) to form a denture base

3.11
initial packing time
time after mixing, or other preparation, when a denture base material mixture first reaches packing consistency

3.12
final packing time
last time, after achievement of the initial packing time, at which a denture base material mixture retains packing consistency

3.13
processing
procedure of preparing a solid denture base polymer plate and/or specimen by polymerization or injection moulding

3.14
thermoplastic, adj
characteristic of a hard polymeric material that allows it to be softened by application of heat to make it mouldable, and then return to the hardened state upon cooling

3.15
translucency
capacity of a body of material to allow the passage of light, yet diffusing the light so as not to render objects lying beyond the body clearly visible

4 Classification

Denture base polymers covered by this part of ISO 20795 are categorized into the following types and classes:

- Type 1: Heat-polymerizable materials
  - Class 1: Powder and liquid
  - Class 2: Plastic cake
- Type 2: Autopolymerizable Materials
  - Class 1: Powder and liquid
  - Class 2: Powder and liquid for pour-type resins
- Type 3: Thermoplastic blank or powder
- Type 4: Light-activated materials
- Type 5: Microwave cured materials

5 Requirements

5.1 Unpolymerized material

5.1.1 Liquid component

5.1.1.1 General

The liquid shall consist essentially of monomeric material compatible with the powder.

5.1.1.2 Homogeneity

The liquid shall be free of deposit or sediment that can be observed by visual inspection (see 8.1.1).

5.1.2 Solid components

The solid or semi-solid components shall be free of extraneous material that can be observed by visual inspection (see 8.1.1).

5.1.3 Packing plasticity

When Type 1 Class 1 and Type 2 Class 1 materials are tested in accordance with 8.2, at the initial packing time recommended by the manufacturer, they shall be capable of being intruded into at least two holes in the die (8.2.2.1) to a depth of not less than 0,5 mm (see 8.2.4.2). Type 1 Class 1, Type 1 Class 2, and Type 5 materials shall meet the requirements when tested at the final packing time (see 8.2.4.3).

5.2 Polymerized material

5.2.1 Biocompatibility

Specific qualitative requirements for freedom from biological hazard are not included in this part of ISO 20795, but it is recommended that, in assessing possible biological or toxicological hazards, reference be made to ISO 10993-1 and ISO 7405.

5.2.2 Surface characteristics

5.2.2.1 When processed in the manner recommended by the manufacturer and in contact with materials recommended by the manufacturer, denture base specimens prepared in accordance with 8.4.3, 8.8.2.2 and 8.9.3 shall have a smooth, hard and glossy surface (see 8.1.1).

5.2.2.2 The specimens for colour stability, the specimens for residual methyl methacrylate monomer and the specimens for sorption and solubility testing shall retain their form without visible distortion after processing (see 8.1.1).

5.2.2.3 When polished in accordance with 8.5.1.4, the specimen plates shall present a smooth surface with a high gloss (see 8.1.1).

5.2.3 Shape capability

When prepared in accordance with the manufacturer's instructions, all types of denture base polymers shall produce a test specimen plate (8.5.1.4) with defined edges after deflasking (see 8.5.1.4).

5.2.4 Colour

The colour of a specimen strip shall be as stated by the manufacturer when tested in accordance with 8.3 and inspected in accordance with 8.1.1.

The manufacturer shall provide a shade guide on request.

Coloured denture base polymers shall be translucent (see 5.2.6 and 8.5.2) and pigment and fibres shall be evenly distributed.

Clear (transparent) denture base polymers shall be clear and colourless.

5.2.5 Colour stability

When tested in accordance with 8.4 and inspected in accordance with 8.1.1, test specimens shall not show more than a slight change in colour.

5.2.6 Translucency

When tested in accordance with 8.5.2.3 the shadow of the illuminated opaque disc shall be visible from the opposite side of the test specimen plate.

5.2.7 Freedom from porosity

When prepared in accordance with 8.5.3.3, a specimen's strips shall not show voids that can be observed by visual inspection (see 8.1.1).

5.2.8 Ultimate flexural strength

When determined in accordance with 8.5.3.5, the ultimate flexural strength shall be not less than 65 MPa for Type 1, Type 3, Type 4 and Type 5 polymers and not less than 60 MPa for Type 2 polymers (see Table 1).

5.2.9 Flexural modulus

When determined in accordance with 8.5.3.5, the flexural modulus of the processed polymer shall be at least 2 000 MPa for Type 1, Type 3, Type 4 and Type 5 polymers and at least 1 500 MPa for Type 2 polymers (see Table 1).

5.2.10 Maximum stress intensity factor for materials with improved impact resistance

Where a manufacturer claims a material with improved impact resistance, the maximum stress intensity factor shall be at least 1,9 MPa $m^{1/2}$ when tested in accordance with 8.6 (see Table 2).

5.2.11 Total fracture work

Where a manufacturer claims a material with improved impact resistance, the total fracture work shall be at least 900 J/m^2 when tested in accordance with 8.6 (see Table 2).

5.2.12 Bonding to synthetic polymer teeth

Denture base polymers intended for use with synthetic polymer teeth shall meet one of the following requirements.

a) The polymer shall, when tested in accordance with 8.7, be capable of bonding to polymer teeth complying with the bonding requirements of ISO 22112.

b) If there are problems of achieving bonding, the manufacturer's instructions shall contain information about special treatments necessary to achieve bonding [see 9.3 k)].

5.2.13 Residual methyl methacrylate monomer

When prepared and tested in accordance with 8.8 the following shall apply (see Table 1).

The upper limit (maximum) for residual methyl methacrylate is 2,2 % mass fraction for denture base polymers of Type 1, Type 3, Type 4 and Type 5.

The upper limit (maximum) for residual methyl methacrylate is 4,5 % mass fraction for denture base polymers of Type 2.

If lower percentages of residual methyl methacrylate monomer are claimed by the manufacturer [see 9.3 m)], the content shall not be more than 0,2 % mass fraction higher than that stated by the manufacturer.

5.2.14 Sorption

When the processed polymer is tested in accordance with 8.9, the increase in mass per volume (water sorption) shall not exceed 32 µg/mm^3 (see Table 1).

5.2.15 Solubility

When the processed polymer is tested in accordance with 8.9, the loss in mass (soluble matter) per volume shall not exceed 1,6 µg/mm^3 for Type 1, Type 3, Type 4 and Type 5 polymers, and shall not exceed 8,0 µg/mm^3 for Type 2 polymers (see Table 1).

Table 1 — Summary of requirements described in 5.2.8, 5.2.9, 5.2.13, 5.2.14 and 5.2.15

Requirements	Flexural properties		Residual methyl methacrylate monomer	Sorption	Solubility
	Ultimate flexural strength	Flexural modulus			
	σ	E		w_{sp}	w_{sl}
	MPa	MPa	Percent mass fraction	µg/mm^3	µg/mm^3
	min.	min.	max.	max.	max.
Types 1, 3, 4, 5	65	2 000	2,2	32	1,6
Type 2	60	1 500	4,5	32	8,0

Table 2 — Additonal requirements for materials with improved impact resistance described in 5.2.10 and 5.2.11

Requirements	Fracture toughness	
	Maximum stress intensity factor	Total fracture work
	K_{max}	W_f
	MPa m$^{1/2}$	J/m^2
	min.	min.
Materials with improved impact resistance	1,9	900

6 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, all material shall be of the same batch.

7 Preparation of test specimens

7.1 Laboratory environment

Prepare and test specimens at (23 ± 2) °C and (50 ± 10) % relative humidity, unless otherwise specified in this part of ISO 20795 or in the manufacturer's instructions.

7.2 Procedures

Prepare, manipulate and process materials for making the specimens using the equipment and procedures recommended in the manufacturer's instructions (see 9.3), unless otherwise specified in this International Standard.

From materials requiring a mixture of two or more ingredients, prepare separate mixes for each specimen or specimen plate.

7.3 Special equipment

Any special equipment specified by the manufacturer for processing a material shall be made available by the manufacturer.

8 Test methods

8.1 Inspection for compliance determination

8.1.1 Visual inspection

Observe the test samples by visual inspection to determine compliance with the requirements laid down in 5.1.1.2, 5.1.2, 5.2.2, 5.2.3, 5.2.4, 5.2.5, 5.2.6, 5.2.7 and Clause 9. [Inspect for colour (5.2.4) and colour stability (5.2.5) in accordance with ISO 7491].

8.1.2 Expression of results

Report whether the liquid components pass or fail (see 5.1.1.2).

Report whether the solid components pass or fail (see 5.1.2).

Report whether the surfaces of the denture base specimens have a smooth, hard and glossy surface (see 5.2.2.1), and whether the specimens pass or fail.

Report whether the form of specimens is retained without distortion and whether the specimens pass or fail (see 5.2.2.2).

Report whether the specimen plates have a smooth surface with a high gloss after polishing, and whether the specimen plate passes or fails (see 5.2.2.3).

Report whether the specimen plate has defined edges, and whether the specimen plate passes or fails (see 5.2.3).

Report whether the material passes or fails the requirements for labelling, marking, packaging and instructions (see Clause 9).

8.2 Packing plasticity

8.2.1 Materials

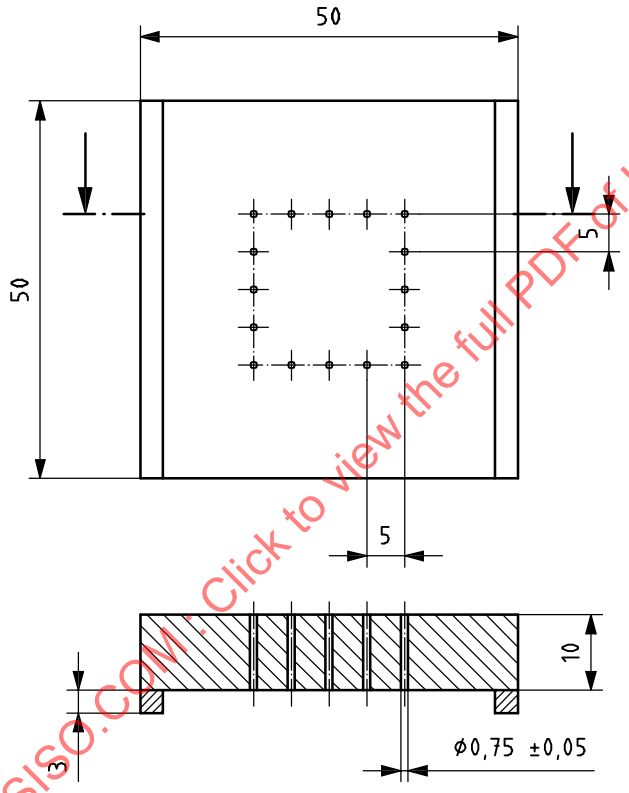
8.2.1.1 **Polyethylene or polyester film**, 0,035 mm to 0,050 mm thick and approximately 50 mm × 50 mm.

8.2.1.2 **Glass plate**, (60 ± 5) mm × (60 ± 5) mm × (5 ± 1) mm.

8.2.2 Apparatus

8.2.2.1 **Perforated brass die**, having the dimensions shown in Figure 1, with perforations having a diameter of (0,75 ± 0,05) mm.

Dimensions in millimetres



Dimensional tolerances not specified shall be ± 1 mm.

Figure 1 — Perforated brass die for packing plasticity test

8.2.2.2 **Weight**, capable of exerting a force of (50 ± 1) N.

8.2.2.3 **Dial gauge**, complying with ISO 463, or linear gauge accurate to 0,01 mm, equipped with a probe capable of entering holes in the brass die for measuring depth of penetration of the material into the die.

8.2.3 Test conditions

Maintain the perforated brass die (8.2.2.1) and glass plate (8.2.1.2) at conditions specified in 7.1, except where otherwise specified by the manufacturer.

8.2.4 Procedure

8.2.4.1 General

The materials are tested at the times following mixing as shown in Table 3. See 3.11 and 3.12 for packing time definitions.

Table 3 — Testing procedure

	Type 1 Class 1	Type 1 Class 2	Type 2 Class 1	Type 2 Class 2	Type 3	Type 5
Initial packing time	×	—	×	—	—	
Final packing time	×	×	—	—	—	×

8.2.4.2 Initial packing time for Type 1 Class 1 and Type 2 Class 1

Prepare a sample of the material having a mass of 16 g to 20 g. Immediately prior to the manufacturer's recommended initial packing time [see 9.3 e)], immediately shape one-half of the sample into a cake approximately 5 mm thick, place it on the upper surface of the brass die (8.2.2.1) and cover it with a plastic film (8.2.1.1). At the recommended initial packing time, place the glass plate (8.2.1.2) and the weight (8.2.2.2) on the plastic-covered resin cake. After 10 min ± 30 s, remove the weight. When the material is firm, introduce the measuring instrument probe (8.2.2.3) into each hole from the other side of the brass die, to contact the penetrating material to determine the unpenetrated depth in the hole.

Calculate the depth of penetration for each hole according to the following formula:

$$D_P = d - d'$$

where

D_P is the depth of penetration, in millimetres;

d is the thickness of the brass die, in millimetres;

d' is the depth not penetrated, in millimetres.

8.2.4.3 Final packing time for Type 1 Class 1, Type 1 Class 2 and Type 5

Immediately before the final packing time [see 9.3 e)] recommended by the manufacturer, immediately shape the second half of the sample into a cake and test this portion in accordance with 8.2.4.2.

8.2.5 Pass/fail determinations

If the first sample fails to comply with the requirement stated in 5.1.3 test two additional samples. If the second and third samples comply with the requirement, the product passes.

8.2.6 Expression of results

Report the number of holes penetrated to a depth of not less than 0,5 mm by each sample and whether the material passes or fails.

8.3 Colour

8.3.1 General

Compare a specimen strip prepared in accordance with 8.5.3.3, and inspected in accordance with 8.1.1, with the shade guide, for compliance with 5.2.4.

8.3.2 Expression of results

Report whether the material passes or fails in accordance with ISO 7491.

8.4 Colour stability

8.4.1 Materials

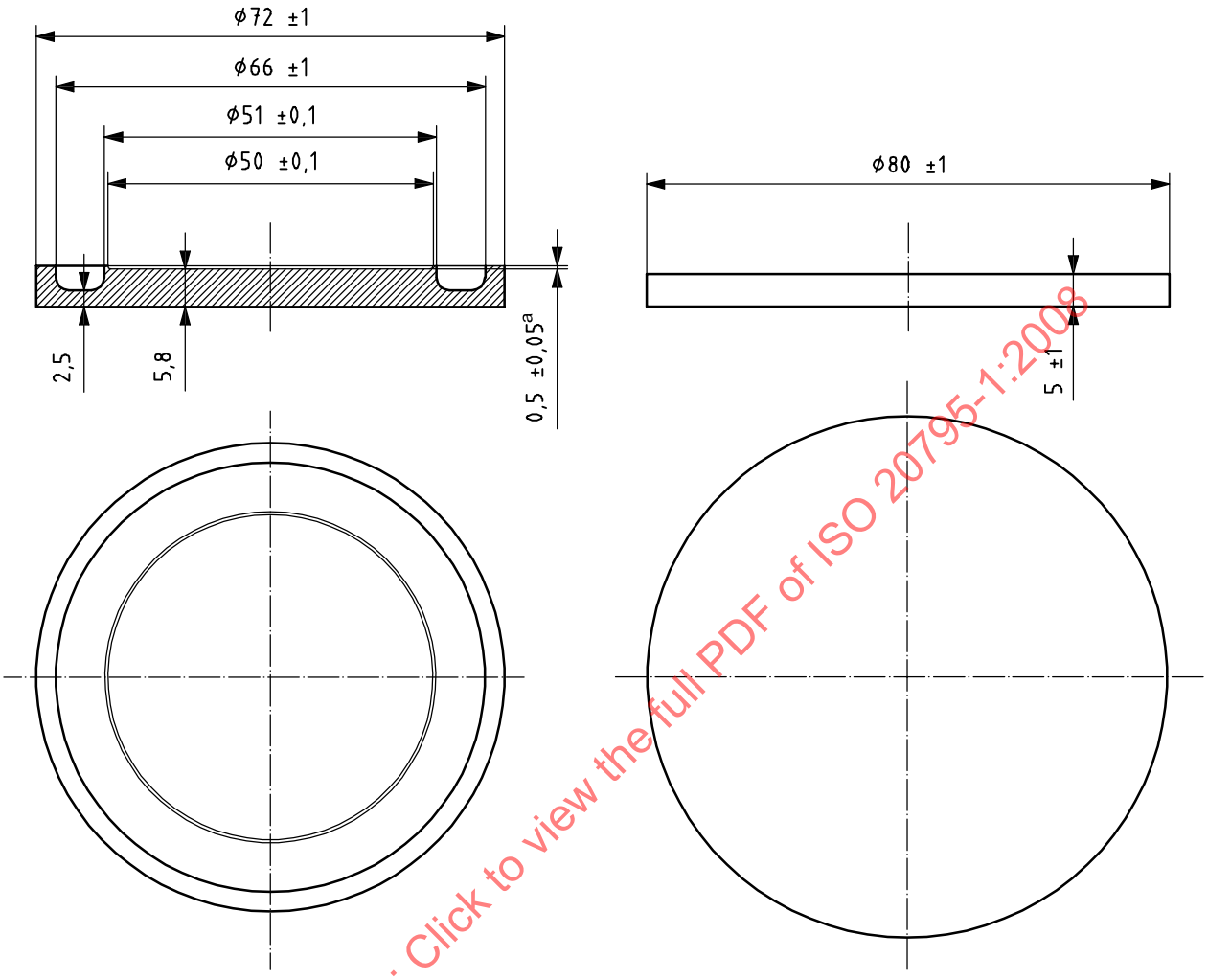
8.4.1.1 **Sheet of polyester film**, having a thickness of (50 ± 25) µm to cover the steel mould (8.4.2.1).

8.4.1.2 **Aluminium foil**.

8.4.2 Apparatus

8.4.2.1 **Circular stainless steel mould and cover** (for Type 1 and Type 2 Class 1 materials), having the dimensions shown in Figure 2, mounted in gypsum in separate halves of a denture flask.

Dimensions in millimetres



a) mould **b) cover**

[a] mould depth to form specimen

Dimensional tolerances not specified shall be ± 0,2 mm

Figure 2 — Stainless steel mould and cover for specimen preparation of colour stability sorption and solubility (see 8.4 and 8.9)

8.4.2.2 **Moulds and/or equipment** (for Type 2 Class 2, Type 3, Type 4, Type 5 and capsulated materials) recommended by the manufacturer to produce specimens with the dimensions specified in 8.4.3.

8.4.2.3 **Hydraulic or hand press and clamp**, where applicable.

8.4.2.4 **Water bath**, capable of maintaining constant temperatures, where applicable.

8.4.2.5 **Micrometer screw gauge or dial calliper**, accurate to 0,01 mm and fitted with parallel anvils.

8.4.2.6 **Oven**, capable of being maintained at (37 ± 1) °C.

8.4.2.7 Radiation source and test chamber, see 3.1.1 and 3.1.3 of ISO 7491:2000.

8.4.3 Preparation of test specimens

8.4.3.1 Type 1 and Type 2 Class 1 materials

Make two specimens from separate mixes. Mix the resin and pack the mixture into the mould (8.4.2.1) with the polyester film (8.4.1.1) against the steel cover of the mould. Process the mixture in accordance with the manufacturer's instructions (see 9.3), but retain the polyester film during the processing cycle.

Check with a micrometer or dial calliper (8.4.2.5) to ensure that each specimen has a diameter of (50 ± 1) mm and a thickness of (0,5 ± 0,1) mm and that the top and bottom surfaces are flat.

8.4.3.2 Type 2 Class 2, Type 3, Type 4, Type 5 and capsulated materials

Prepare the specimens as described by the manufacturer.

Check with a micrometer or dial calliper (8.4.2.5) to ensure that each specimen has a diameter of (50 ± 1) mm and a thickness of (0,5 ± 0,1) mm and that the top and bottom surfaces are flat.

8.4.4 Procedure

Store the two specimens in the oven (8.4.2.6) for 24 h ± 30 min at (37 ± 1) °C. Then store one specimen in the dark in a laboratory environment (see 7.1) until the colour comparison test is made.

Cover half of the second specimen with aluminium foil (8.4.1.2) and transfer it to the radiation source and test chamber (8.4.2.7). Immerse the specimen in water at (37 ± 5) °C when exposed to the radiation for 24 h ± 30 min in accordance with ISO 7491. After exposure, remove the aluminium foil before colour comparison of the specimens including the unexposed specimen.

Carry out the colour comparison in accordance with the requirements specified in 5.2.5 and in accordance with the procedure for colour comparison laid down in ISO 7491.

For Type 4 materials, store the exposed specimen in a laboratory environment (see 7.1) for 6 d ± 2 h until the colour comparison test is made.

8.4.5 Expression of results

Report whether the material passes or fails in accordance with ISO 7491 and 5.2.5.

8.5 Polishability, translucency, freedom from porosity, ultimate flexural strength and flexural modulus

8.5.1 Polishability

8.5.1.1 Materials

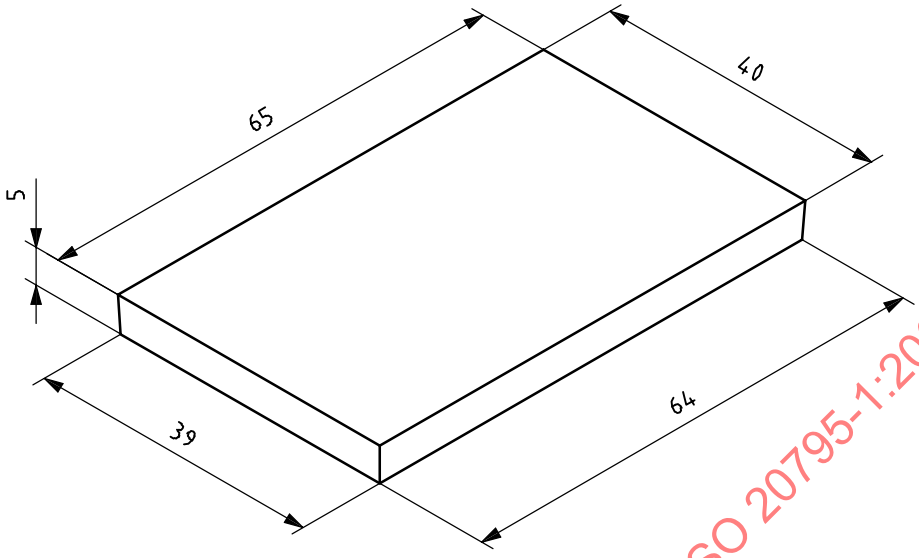
8.5.1.1.1 Polishing compound.

8.5.1.1.2 Wet pumice, having a grain size of approximately 10 µm to 20 µm.

8.5.1.2 Apparatus

8.5.1.2.1 Model of the specimen plate, in metal or polymer (see Figure 3).

Dimensions in millimetres



Dimensional tolerances shall be ± 1 mm.

Figure 3 — Model of the specimen plate

8.5.1.2.2 Denture flask, capable of accommodating the test specimen plate so that the corners are not less than 5 mm from the walls of the flask.

8.5.1.2.3 Equipment for processing the resin, including gypsum or hydrocolloid investment system [see 9.3 f)].

8.5.1.2.4 Standard metallographic grinding paper, with a grain size of approximately 30 µm (P500).

NOTE See ISO 6344-1.

8.5.1.2.5 Muslin wheel, with 16 to 36 ply having a diameter of 70 mm to 95 mm and at least 10 mm between the periphery and the stitching or other reinforcement.

8.5.1.2.6 Unstitched muslin wheel, with 16 to 36 ply having a diameter of 70 mm to 95 mm.

8.5.1.3 Preparation of the mould

For Type 1 and Type 2 Class 1 polymers, invest the model of the specimen plate (8.5.1.2.1) in the denture flask (8.5.1.2.2) in accordance with the manufacturer's instructions. Prepare the mould for Type 2 Class 2, Type 3, Type 4, Type 5 and capsulated materials in accordance with the manufacturer's instructions.

8.5.1.4 Procedure

Form and process, according to the manufacturer's instructions, two specimen plates each from a separate mix. Use the material (8.5.1.1), the apparatus (8.5.1.2) and the mould (8.5.1.3). Grind and polish the surfaces of the specimen plates for no longer than 1 min with pumice (8.5.1.1.2) and with a wet muslin wheel (8.5.1.2.5) at a circumferential speed of (650 ± 350) m/min.

NOTE A wheel with a diameter of 70 mm rotating at 1 500 min^{-1} will have a circumferential speed of 329 m/min and a 100 mm wheel rotating at 3 500 min^{-1} will have a circumferential speed of 1 100 m/min.

Thereafter polish with an unstitched muslin wheel (8.5.1.2.6) using a polishing compound (8.5.1.1.1).

After polishing and cleaning, examine the polished surfaces for compliance with 5.2.2.3.

8.5.1.5 Pass/fail determination

If both specimen plates comply with 5.2.2.3, the material passes.

If both specimen plates fail to comply with 5.2.2.3, the material fails.

If only one of the specimen plates complies, prepare and evaluate three new plates. The material passes only if all three new plates comply.

8.5.1.6 Expression of results

Report the number of specimen plates evaluated, the number complying and whether the material passes.

8.5.2 Translucency

8.5.2.1 Materials

8.5.2.1.1 Two specimen plates, prepared and tested according to 8.5.1.

8.5.2.2 Apparatus

8.5.2.2.1 Electrical light bulb, frosted 40 W.

NOTE Other frosted electrical light sources of equivalent radiant excitance can be used.

8.5.2.2.2 Opaque disc, diameter (10 ± 1) mm and thickness (2 ± 1) mm.

8.5.2.3 Procedure

Examine each of the two specimen plates separately. Position the polished specimen plate approximately 500 mm from the light bulb (8.5.2.2.1) with the opaque disc (8.5.2.2.2) centred in between. Darken the room. View the specimen plate from the side opposite the disc location to determine whether the material complies with 5.2.6.

8.5.2.4 Pass/fail determination

If both specimen plates comply with 5.2.6, the material passes.

If both specimen plates fail, the material fails.

If only one of the specimen plates passes, prepare and evaluate three new plates. The material passes only if all three new plates comply.

8.5.2.5 Expression of results

Report the number of specimen plates evaluated, the number complying and whether the material passes.

8.5.3 Freedom from porosity, ultimate flexural strength and flexural modulus

8.5.3.1 Materials

8.5.3.1.1 Two specimen plates, prepared and tested in accordance with 8.5.1 and 8.5.2.

8.5.3.2 Apparatus

8.5.3.2.1 Motorised saw, or other cutting device for sectioning the specimen plates.

8.5.3.2.2 Milling machine, or other equipment for air- or water-cooled cutting so as not to generate temperatures above 30 °C during shaping of the specimens. (A machine with a milling head and a sharp carbide edge, is suitable.)

8.5.3.2.3 Standard metallographic grinding papers, having a grain size of approximately 30 µm (P500), 18 µm (P1 000) and 15 µm (P1 200).

NOTE See ISO 6344-1.

8.5.3.2.4 Micrometer screw gauge and/or dial calliper, accurate to 0,01 mm and fitted with parallel anvils.

8.5.3.2.5 Container containing water, complying with grade 3 of ISO 3696:1987, for storing the specimen strips at (37 ± 1) °C for pre-test conditioning.

8.5.3.2.6 Testing machine, calibrated to provide a constant displacement rate of (5 ± 1) mm/min and equipped with instrumentation for measuring the deflection of the specimen to within 0,025 mm.

Take into account any load exerted by the deflection instrument when calibrating the machine.

8.5.3.2.7 Metal flexural test rig, consisting of a central loading plunger and two polished cylindrical supports, 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 centres of the supports shall be (50 ± 0,1) mm, and the loading plunger shall be midway between the supports to within 0,1 mm. Include means in the design, to prevent misalignment of the specimen.

8.5.3.2.8 Water bath, for maintaining the specimens wet and at a temperature of (37 ± 1) °C, during testing.

8.5.3.3 Procedure

Prepare six specimen strips. Cut each plate lengthways into three equal strips, 64 mm long, (10,0 ± 0,2) mm wide and (3,3 ± 0,2) mm in height. Machine the strips in a milling machine (8.5.3.2.2) on the edges and equally from both moulded surfaces so that the dimensions remain slightly oversized. Take care to avoid overheating the specimen. Wet-grind all faces and edges smooth and flat with the metallographic grinding papers (8.5.3.2.3) to the required width and height. Make three measurements of the specimen height along the long axis with an accuracy of ± 0,01 mm using a micrometer, and/or dial calliper (8.5.3.2.4). The deviation between the three measurements along the long axis shall be no more than ± 0,02 mm. The specimen shall be flat and have an even height.

8.5.3.4 Freedom from porosity

8.5.3.4.1 Pass/fail determination

Prepare six test specimen strips in accordance with 8.5.3.3 and examine for compliance with 5.2.7.

The material passes only if at least five out of six specimen strips comply with the requirements given in 5.2.7.

8.5.3.4.2 Expression of results

Report the number of specimen strips complying and whether the material passes.

8.5.3.5 Ultimate flexural strength and flexural modulus

8.5.3.5.1 Procedure

Store five specimen strips [or six in the case of repetition of the test (see 8.5.3.5.2.3 and 8.5.3.5.2.4)], prepared according to 8.5.3.3 and complying with 5.2.7, in water (8.5.3.2.5) at a temperature of (37 ± 1) °C for (50 ± 2) h prior to flexural testing. Take a specimen strip from water storage and immediately lay the flat surface symmetrically on the supports of the flexural test rig (8.5.3.2.7) immersed in the water bath (8.5.3.2.8). Allow the specimen to come to equilibrium with the water bath temperature.

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

8.5.3.5.2 Calculation and expression of results

8.5.3.5.2.1 Ultimate flexural strength

Calculate the ultimate flexural strength, σ, in megapascals using the following equation:

$$\sigma = \frac{3Fl}{2bh^2}$$

where

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

l is the distance, in millimetres, between the supports, accurate to ± 0,01 mm;

b is the width, in millimetres, of the specimen measured immediately prior to water storage;

h is the height, in millimetres, of the specimen measured immediately prior to water storage.

8.5.3.5.2.2 Flexural modulus

Calculate the flexural modulus E, in megapascals, using the following equation:

$$E = \frac{F_1 l^3}{4bh^3 d}$$

where

F_1 is the load, in newtons, at a point in the straight line portion (with the maximum slope) of the load/displacement curve;

NOTE For greater accuracy, the straight line may be extended.

d is the deflection, in millimetres, at load F_1;

l, b and h are as defined in 8.5.3.5.2.1.

8.5.3.5.2.3 Pass/fail determination of ultimate flexural strength

If at least four out of five specimens give results not less than 65 MPa for Type 1, Type 3, Type 4 and Type 5 polymers and not less than 60 MPa for Type 2 polymers, the material is deemed to have complied with the requirements of 5.2.8.

If at least three of the results are less than 65 MPa for Type 1, Type 3, Type 4 and Type 5 polymers and less than 60 MPa for Type 2 polymers, the material is deemed to have failed.

If two of the results are less than 65 MPa for Type 1, Type 3, Type 4 and Type 5 polymers and 60 MPa for Type 2 polymers, repeat the whole test but on this occasion prepare six specimen strips.

If at least five of the results are not less than 65 MPa for Type 1, Type 3, Type 4 and Type 5 polymers and not less than 60 MPa for Type 2 polymers on the second occasion, the material is deemed to have complied with the requirements of 5.2.8.

8.5.3.5.2.4 Pass/fail determination of flexural modulus

If at least four of the results passed the requirements of 5.2.8 on the first occasion, calculate the flexural modulus according to 8.5.3.5.2.2 for each of the five specimens.

If a second series was tested, calculate the flexural modulus for five of the six specimens from this series only.

If at least four of the results are not less than 2 000 MPa for Type 1, Type 3, Type 4 and Type 5 polymers and not less than 1 500 MPa for Type 2 polymers the material is deemed to have complied with the requirements of 5.2.9.

If at least three of the results are less than 2 000 MPa for Type 1, Type 3, Type 4 and Type 5 polymers and less than 1 500 MPa for Type 2 polymers, the material is deemed to have failed.

If two of the results are less than 2 000 MPa for Type 1, Type 3, Type 4 and Type 5 polymers and less than 1 500 MPa for Type 2 polymers, repeat the whole test, but on this occasion prepare six specimen strips. In this series, at least five results for both ultimate flexural strength and flexural modulus shall comply with the requirements of 5.2.8 and 5.2.9.

8.5.3.5.2.5 Expression of results

Report the number of specimen strips evaluated, all results for ultimate flexural strength and flexural modulus with the number of strips complying with the requirements of 5.2.8 and 5.2.9, and whether the material passes.

8.6 Fracture toughness with a modified bending test

8.6.1 General

Only if the manufacturer makes a claim concerning impact resistance test the material for fracture toughness (see 5.2.10 and 5.2.11).

8.6.2 Materials

8.6.2.1 **Two specimen plates**, prepared in accordance with 8.5.1.

8.6.2.2 **Glycerol**, technical grade, used as a lubricant.

8.6.3 Apparatus

See also 8.5.3.2.2, 8.5.3.2.3, 8.5.3.2.4, 8.5.3.2.5 and 8.5.3.2.8.

8.6.3.1 Motorised saw, or other cutting device

The device shall be able to section the specimen plates. Preferably for cutting the pre-crack, a (0,5 ± 0,1) mm diamond sawing blade is needed. The cutting tool shall be adjustable to a depth of (3,0 ± 0,2) mm.

8.6.3.2 Holding device containing a fixation clamp, to align specimen(s) during pre-cracking and the sharp blade cutting procedure.

8.6.3.3 Sharp blade, such as a scalpel, razor blade or craft knife with an unbent straight blade.

8.6.3.4 Optical microscope with micrometer scale included, to measure the total length of the crack (total amount of pre-crack and the sharp notch in millimetres).

8.6.3.5 Container containing water, for conditioning the specimen strips at (23 ± 1) °C.

8.6.3.6 Clean dry towel.

8.6.3.7 Flexural test rig, see 8.5.3.2.7, but with a span, l_t, of (32,0 ± 0,1) mm (see 8.6.5.1).

8.6.3.8 Machine for testing, calibrated to provide for a constant displacement rate of (1,0 ± 0,2) mm/min and equipped with instrumentation for measuring the deflection of the specimen to within 0,025 mm.

The recording of the load/displacement curve and the calculation of the integral area under the curve shall be possible. When calibrating the machine take into account any load exerted by the deflection instrument.

8.6.4 Procedure

At least 24 h from the beginning of the curing cycle, wet-grind or machine the plates (8.6.2.1) in a milling machine (8.5.3.2.2), equally from both mould surfaces, to obtain flat, parallel surfaces, and so that the thickness of the plates remains slightly oversized. Take care to avoid overheating the specimens.

Cut each plate breadthwise with a cutting device (8.6.3.1) in equal specimen strips approximately 8 mm wide, so that the dimensions remain slightly oversized compared with the finished specimen strips. Wet-grind all surfaces smooth and flat with the metallographic grinding papers (8.5.3.2.3) to the required dimensions, length 39 mm, height, h_t, (8,0 ± 0,2) mm and width b_t (4,0 ± 0,2) mm, using grain size 18 µm (P1 000) or 15 µm (P1 200).

Fix the specimens lengthwise in the holding device (8.6.3.2) and set a mark exactly on the centreline midway from the edges of the specimens. Cut the pre-crack with a diamond blade and a saw (8.6.3.1) to a depth of (3,0 ± 0,2) mm along the marked centreline. Set the pre-crack in the centre of each specimen.

Fix one specimen at a time, in a clamp or holding device (8.6.3.2). Wet the pre-crack with a drop of glycerol (8.6.2.2). Set the sharp blade (8.6.3.3) on the bottom of the pre-crack and cut the sharp notch with hand/machine pressure and a sliding back and forth motion.

A notch depth in the range of 100 µm to 400 µm is sufficient. Use an optical microscope (8.6.3.4) to check the crack depth. It is recommended to test the cutting procedure on a pre-test specimen. Attempting to further increase the notch depth should not be done. The situation of the notch arrangement is shown in Figure 4 a). Measure the width, b_t, and the height, h_t, of the specimen with a micrometer (8.5.3.2.4). See Figure 4 b).

Store ten selected notched specimens in a container with water (8.5.3.2.5) at (37 ± 1) °C for 7 d ± 2 h. Condition the specimens in a different container of water (8.6.3.5) at (23 ± 1) °C for (60 ± 15) min prior to testing.

After conditioning, remove one specimen strip from the water and dry it with a clean dry towel (8.6.3.6). Place the specimen on the supports of the test rig (8.6.3.7). Place the specimen strip with the notch facing exactly opposite the load plunger [see Figure 4 b)]. Be sure that the notch is placed right in the centre between the supports.

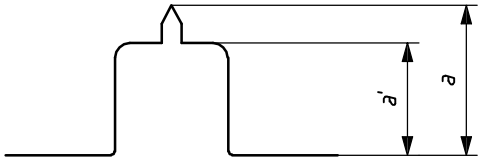
Increase the force of the loading plunger of the testing machine (8.6.3.8) from zero using a constant displacement rate of (1,0 ± 0,2) mm/min until maximum load is passed, and the crack has almost reached the opposite side of the specimen. The test can be considered finished when the current load is reduced to 5 % of the maximum load or is less than (1,0 ± 0,2) N.

The recording of the whole load/displacement curve is necessary for calculations. Repeat the test for all ten conditioned specimens.

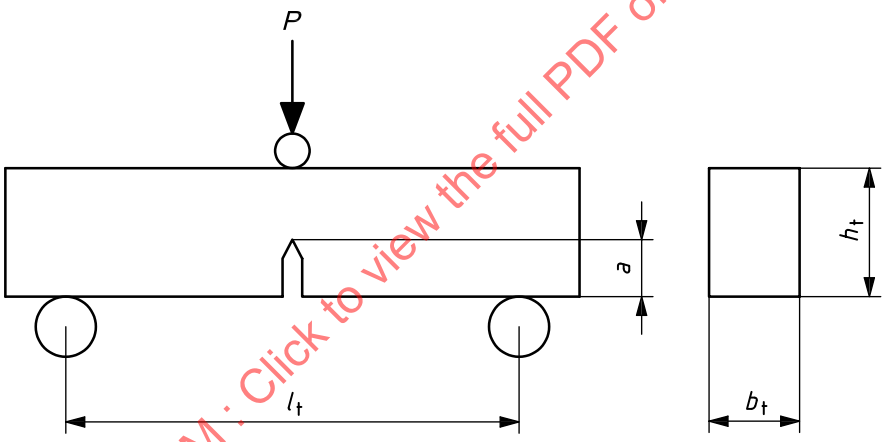
After completing the test, measure the depth of the pre-crack including the sharp notch, a in Figure 4, next to the fracture surface with an optical microscope (8.6.3.4).

NOTE Before fracture toughness testing ink can be introduced into the notch and allowed to dry to improve identification of the complete notched area.

Determine the total crack length, a, as the average of three measurements of the distance between the specimen surface and the area fractured in the test. Take these three measurements along the quarter- and half-width lines (see Figure 5).

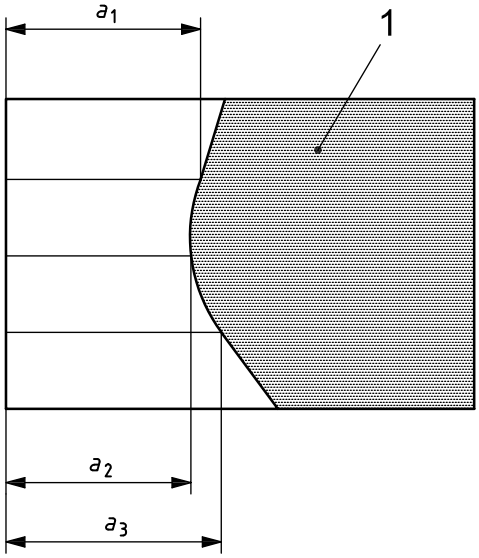


a) Notch arrangement



b) Specimen strip with the notch facing exactly opposite to the affected load plunger

Figure 4 — Fracture toughness test



Key

1 Fracture surface

Figure 5 — Determination of the total crack length next to the fracture surface

8.6.5 Calculation and expression of the results

8.6.5.1 Dimensions

Height $h_t = (8{,}0 \pm 0{,}2)$ mm

Width $b_t = (4{,}0 \pm 0{,}2)$ mm

Pre-crack $a' = (3{,}0 \pm 0{,}2)$ mm

Crack length a (0,1 mm – 0,4 mm longer than a')

Span $l_t = (32{,}0 \pm 0{,}1)$ mm

8.6.5.2 Calculation of the maximum stress intensity factor

Calculate maximum stress intensity factor K_{max}, using the following equation:

$$K_{max} = \frac{f P_{max} l_t}{(b_t h_t^{3/2})} \times \sqrt{10^{-3}} \quad \text{MPa m}^{1/2}$$

where

f is a geometrical function dependent on x:

$$f(x) = 3x^{1/2}\left[1{,}99 - x(1-x)(2{,}15 - 3{,}93x + 2{,}7x^2\right] \Big/ \left[2(1+2x)(1-x)^{3/2}\right]$$

and

$$x = a/h_t$$

P_{max} is the maximum load exerted on the specimen, in newtons;

a, h_t, b_t and l_t are listed in 8.6.5.1, expressed in millimetres.

8.6.5.3 Calculation of the total fracture work

NOTE The area under the load/displacement curve represents the energy required to break the whole specimen. Dividing this energy by twice the fractured area, the surface energy expressed in joules per square meter is obtained.

Calculate the total fracture work W_f, using the following equation. The fracture work is calculated from the integral area of the load/displacement curve

$$W_f = \frac{U}{\left[2b_t\left(h_t - a\right)\right]} \times 1\,000 \qquad \text{J/m}^2$$

where

U is the recorded area under the load/displacement curve given by the following equation

$$U = \int P_d U, \text{ in newton millimetres;}$$

b_t, h_t and a are listed in 8.6.5.1, expressed in millimetres.

8.6.5.4 Pass/fail determination of maximum stress intensity factor

If at least eight of the results from ten specimens are not less than 1,9 MPa $m^{1/2}$, the material complies with the requirements of 5.2.10.

If at least six of the results are less than 1,9 MPa $m^{1/2}$, the material is deemed to have failed.

If three, four or five of the results are less than 1,9 MPa $m^{1/2}$, repeat the whole test but on this occasion prepare twelve specimen strips.

If at least ten of the twelve results are not less than 1,9 MPa $m^{1/2}$ on the second occasion, the material complies with the requirement of 5.2.10.

8.6.5.5 Pass/fail determination of total fracture work

If at least eight of the results from ten specimens are not less than 900 J/m^2, the material complies with the requirements of 5.2.11.

If at least six of the results are less than 900 J/m^2, the material is deemed to have failed.

If three, four or five of the results are less than 900 J/m^2, repeat the whole test but on this occasion prepare twelve specimen strips.

If at least ten of the twelve results are not less than 900 J/m^2 on the second occasion, the material is deemed to have complied with the requirement of 5.2.11.

8.6.5.6 Expression of results

Report the number of specimens evaluated, all results for maximum stress intensity factor K_{max} and total fracture work W_f and the number of specimens complying with the requirements of 5.2.10 and 5.2.11, and whether the material passes.

8.7 Bonding to synthetic polymer teeth

8.7.1 Materials

8.7.1.1 **Maxillary anterior synthetic polymer teeth**, complying with ISO 22112.

8.7.1.2 **Dental mounting wax**.

8.7.2 Apparatus

8.7.2.1 **Metal former**, of the design illustrated in Figure 3 a) of ISO 22112:2005, which incorporates a trough 5 mm wide by 1,5 mm deep for use in mounting the teeth.

8.7.2.2 **Normal dental laboratory apparatus**, for denture flasking and processing, including gypsum or a hydrocolloid investment system [see 9.3 f)].

8.7.2.3 **Tensile testing apparatus**, with specially designed grips illustrated in Figure 3 c) of ISO 22112:2005.

8.7.3 Procedure

Grind the ridge lap of a set of six maxillary anterior teeth (8.7.1.1). Mount these teeth on a metal former (8.7.2.1) using wax (8.7.1.2), as illustrated in Figure 3 a) of ISO 22112:2005, so that about one-half of the lingual surface of the incisal portion of the tooth projects beyond the metal former.

Using a denture flask (8.7.2.2), set the mounted teeth in dental gypsum [see Figure 3 b) of ISO 22112:2005]. Remove the metal former and then flush the wax from the teeth with boiling tap-water containing a detergent until all the wax has been removed, followed by rinsing several times with boiling tap-water. Prepare and process the denture base polymer to the teeth according to the manufacturer's instructions (see 9.3) after proper plasticity has been reached. Test the denture base-mounted teeth in the tensile testing apparatus (8.7.2.3) designed to permit a direct pull on the incisal part of the lingual surface in a labial direction at a consistent height above the acrylic bar [see Figure 3 c) of ISO 22112:2005]. Use equipment which does not permit lateral deflection or change of position.

Load each tooth, as illustrated in Figure 3 c) of ISO 22112:2005, at a displacement rate in the range of 0,5 mm/min to 10 mm/min until fracture occurs.

8.7.4 Pass/fail determination

The bond passes the test if the fracture path does not occur cleanly along the tooth surface. Thus, tooth remnants shall remain bonded to the denture base polymer and/or denture base polymer shall remain firmly bonded to the detached tooth or the adhesive shall remain firmly bonded to both detached tooth and denture base.

NOTE Only pure adhesive interfacial fracture indicates a failure to meet the requirement. Cohesive fracture in either the tooth, the denture base polymer or an adhesive, is necessary for a satisfactory bond.

If at least five maxillary anterior teeth pass the test, the denture base polymer is deemed to comply with the requirements of 5.2.12.

If only three comply, the denture base polymer fails.

If only four comply, prepare an additional denture base polymer with six maxillary anterior teeth from one set. If at least five maxillary anterior teeth pass this second test, the denture base polymer is deemed to comply with the requirements.

8.7.5 Expression of results

Report the number of teeth, for which the bond passes the test.

8.8 Residual methyl methacrylate monomer

8.8.1 Principle

Solvent extraction of the methyl methacrylate (MMA) monomer from polymerized denture base materials is carried out, followed by chromatographic analyses.

A gas chromatographic (GC) method, high performance liquid chromatography (HPLC) method (see Annex A) or any other chromatographic method can be used, which gives the same results as with the methods of this part of ISO 20795. Verify the results by proficiency testing based on the chromatographic methods described in this part of ISO 20795.

8.8.2 Preparation of test specimen discs

8.8.2.1 Apparatus

8.8.2.1.1 Circular stainless steel mould (for Type 1 and Type 2 Class 1 materials), with a diameter of 50 mm and a depth of (3,0 ± 0,1) mm with a flat cover.

A similar mould (less deep) is shown in Figure 2. Mount the mould in gypsum in separate halves of a denture flask.

8.8.2.1.2 Moulds and/or equipment (Type 2 Class 2, Type 3, Type 4, Type 5 and capsulated materials), recommended by the manufacturer to produce specimens with the dimensions specified in 8.8.2.1.1.

8.8.2.1.3 Standard metallographic grinding papers, with a grain size of approximately 30 µm (P 500) and 15 µm (P1 200). See Note to 8.5.1.2.4.

8.8.2.1.4 Micrometer screw gauge or dial calliper, accurate to 0,01 mm and fitted with parallel anvils.

8.8.2.2 Procedure

Prepare three specimens from three separate mixes as described in 8.4.3. The mould shall have the dimensions given in 8.8.2.1.1. Keep the specimens in the dark in a laboratory environment (see 7.1) for (24 ± 5) h prior to grinding. Use the metallographic grinding papers (8.8.2.1.3) in turn, to wet-grind material equally from both sides of the specimen disc, until a thickness of (2,0 ± 0,1) mm is obtained. Grind the periphery of the specimens against the 15 µm grain metallographic grinding paper until the entire periphery is abraded and smooth. Avoid frictional heat which can cause loss of monomer or depolymerization. Examine the specimens visually without magnification. If the specimen demonstrates minimal porosity, then three samples can be obtained from it.

NOTE If the specimens are stored in the refrigerator, the monomer content remains constant for several days. If the specimens are stored in the freezer (below −18 °C), the monomer content remains constant for several months.

Store the ground specimens in the dark in a laboratory environment for (24 ± 1) h prior to extraction of the monomer.

8.8.3 Extraction of monomer

8.8.3.1 Reagents

8.8.3.1.1 Hydroquinone (HQ).

8.8.3.1.2 Acetone, purity of analytical or HPLC grade.

8.8.3.1.3 Methanol (CH_3OH), purity of analytical or HPLC grade.

8.8.3.1.4 Internal Standard (I.S.) *n*-pentanol, purity of analytical grade, or any **other suitable I.S.** (e.g. **1-butanol**) whose peak does not interfere with any other peak in the sample solution.

8.8.3.2 Apparatus

Ordinary laboratory apparatus and

8.8.3.2.1 One-mark volumetric glass flasks, of capacities 5 ml, 10 ml and 1 l.

8.8.3.2.2 Analytical balance, with an accuracy of 0,1 mg or better.

8.8.3.2.3 Magnetic stirring apparatus, with PTFE coated magnetic stirring bar.

8.8.3.2.4 Volumetric pipettes, of capacities 100 µl and 2 ml.

8.8.3.2.5 Glass pipettes.

8.8.3.2.6 Closable glass centrifugation tubes.

8.8.3.2.7 Centrifuge, capable of centrifuging at 3 000 × g_n m/s^2.

8.8.3.2.8 Glass tubes, closable.

8.8.3.3 Preparation of solutions

8.8.3.3.1 Acetone solution (A).

Weigh approximately 0,02 g HQ (8.8.3.1.1) into a 1 l one-mark volumetric glass flask (8.8.3.2.1). Add acetone (8.8.3.1.2) until the total volume is 1 l.

8.8.3.3.2 Methanol solution (B).

Weigh approximately 0,02 g HQ (8.8.3.1.1) into a 1 l one-mark volumetric glass flask (8.8.3.2.1). Add methanol (8.8.3.1.3) until the total volume is 1 l.

8.8.3.3.3 Methanol/acetone solution (C).

Mix one volume part of solution A (8.8.3.3.1) and four volume parts of solution B (8.8.3.3.2).

8.8.3.3.4 Internal Standard (I.S.) solution.

In order to achieve an I.S. peak, which will represent a concentration located in the middle of the calibration curve, weigh approximately 350 mg I.S. (8.8.3.1.4) into a 10 ml one-mark volumetric glass flask (8.8.3.2.1). Add the methanol solution (B) (8.8.3.3.2) until the total volume is 10 ml. The volume of 10 ml is to ensure that there is enough I.S. solution for additional analyses. The concentration of the I.S. in the final solution will be approximately 3 % (mass fraction) of the quantity of the specimen pieces (e.g. 650 mg) treated with the acetone solution (A) (8.8.3.3.1) and the methanol solution (B) (8.8.3.3.2).

8.8.3.3.5 Sample solutions.

Analyse three sample solutions from each test specimen, i.e. a total of nine sample solutions.

Break each specimen disc (8.8.2) into pieces small enough to pass through the neck of the one-mark 10 ml volumetric glass flasks (8.8.3.2.1). Introduce a sample size of approximately 650 mg into separate one-mark 10 ml volumetric glass flasks (8.8.3.2.1). Weigh the mass with an analytical balance (8.8.3.2.2) and record for each individual sample solution.

Add the acetone solution (A) (8.8.3.3.1) until the total volume is 10 ml and then introduce a clean PTFE coated magnetic stirring bar (8.8.3.2.3) to each one-mark closable volumetric glass flask. Ensure that the volumetric one-mark glass flasks are properly sealed and agitate the sample solutions by magnetic stirring for (72 ± 2) h at room temperature.

NOTE If the viscosity of the solution is too high for accurate transfer, use an appropriate higher dilution.

To precipitate the dissolved polymer, use a separate volumetric pipette (8.8.3.2.4) to transfer a 2 ml aliquot of each previously prepared sample solution to each separate one-mark closable 10 ml volumetric glass flask.

Then add 100 µl of the I.S. solution (8.8.3.3.4) to each flask. Add methanol solution (B) (8.8.3.3.2) to each of these sample solutions to a total volume of 10 ml.

Use separate glass pipettes (8.8.3.2.5) to transfer approximately 5 ml of the polymer and monomer containing slurry from each of the 10 ml flasks to separate closable glass centrifugation tubes (8.8.3.2.6).

Centrifuge the slurry at 3 000 × g_n m/s^2 for 15 min in a centrifuge (8.8.3.2.7).

Use separate glass pipettes to transfer approximately 3 ml aliquot of each centrifuged solution to separate closable glass tubes (8.8.3.2.8).

Determine that there is no remaining polymer in the solution by adding additional amounts of methanol to an aliquot of the remaining solution in a test tube. The solution shall appear clear when a beam of light is directed vertically through the test tube containing the solution. This test must be carried out in a dark room. If the solution does not appear clear, repeat the procedure described above using a larger amount of the methanol solution (B). Record the volume of the methanol solution (B) necessary to complete precipitation of the polymer. When the solution appears clear, determine the residual monomer content by means of the GC method, HPLC method (see Annex A) or any other equivalent chromatographic method (see 8.8.1).

8.8.4 Gas chromatography

8.8.4.1 Reagent

8.8.4.1.1 Methyl methacrylate (MMA), GC-purity > 99 %.

8.8.4.2 Apparatus

8.8.4.2.1 Gas chromatograph, with split/splitless injection port for liquid samples [split mode (1:10) recommended] flame ionization detector (or an equivalent detector) and recording system.

8.8.4.2.2 Microsyringe, capacity 0,1 µl to 5 µl.

8.8.4.3 Preparation of calibration solutions for gas chromatography

Make at least five standard solutions with concentrations of MMA (8.8.4.1.1), between approximately 0,1 % mass fraction and approximately 6 % mass fraction, of the quantity of the specimen pieces. Prepare calibration solutions of MMA by weighing approximately 6 mg, 60 mg, 150 mg, 300 mg and 400 mg of MMA into separate one-mark volumetric glass flasks of capacity 5 ml (8.8.3.2.1). Add solution C (8.8.3.3.3) until the total volume is 5 ml. Transfer 100 µl of each calibration solution into separate 10 ml one-mark volumetric glass flasks (8.8.3.2.1) together with 100 µl of the I.S. solution (8.8.3.3.4); add solution C (8.8.3.3.3) until the total volume is 10 ml.

Record the mass of MMA for each individual calibration solution and calculate the final concentrations, in micrograms per millilitre.

If the MMA content of the sample solutions (see 8.8.3.3.5) does not fit within the extreme MMA concentrations of the calibration graph (see 8.8.5.1.1), make additional calibration points.

8.8.4.4 Gas chromatographic equipment, gases and operating conditions

a) Column: fused silica capillary tube of length 30 m and internal diameter 0,25 mm is recommended; a stationary phase of a polysiloxane derivative (e.g. polysiloxane with methyl and phenyl groups) or polyethylene glycol;

b) Column conditioning: 6 h to 10 h under gas flow and at elevated temperatures;

c) Recommended column temperature: 75 °C, isothermal;

d) Injector temperature: 200 °C;

e) Detector temperature: 200 °C;

f) Carrier gas: helium for gas chromatography with a flow rate of approximately 1,3 ml/min;

g) Fuel gases: hydrogen and air for gas chromatography.

8.8.4.5 Gas chromatograms of sample and calibration solutions

Depending on the sensitivity of the gas chromatograph used, inject a suitable volume of sample solution (prepared according to 8.8.3.3.5) or the calibration solution (prepared according to 8.8.4.3). The injected volume is not critical for the calculation of results, but shall be identical for corresponding samples and calibration solutions. Operate the gas chromatograph until all components are completely eluted.

Ensure correct quantification of the MMA content in the sample solutions, and ensure good separation of all substances by using appropriate column oven temperature profiles.

8.8.4.6 Evaluation of peaks of gas chromatogram

Determine the retention times of MMA and I.S., at least in relation to each other. The exact values vary according to the age of the column and other gas chromatographic parameters.

Determine the peak area or height of MMA and I.S. by electronic registration and integration.

8.8.5 Calculation and expression of results

8.8.5.1 Calculation of results from a calibration graph

8.8.5.1.1 Drawing of the calibration graph

Draw a calibration graph by plotting the ratios of the peak area (or height):

$$\frac{A'_{\text{MMA}}}{A'_{\text{I.S.}}}$$

where

A'_{MMA} is the peak area (or height) of methyl methacrylate monomer in the calibration solution;

$A'_{\text{I.S.}}$ is the peak area (or height) of the internal standard (8.8.3.1.4) in the calibration solution.

8.8.5.1.2 Precision of measurements

The correlation coefficient of the calibration graph established by linear regression shall be not less than 0,990.

8.8.5.1.3 Determination of the percentage of methyl methacrylate

Determine the percentage of MMA with the corresponding ratio

$$\frac{A_{\text{MMA}}}{A_{\text{I.S.}}}$$

where

A_{MMA} is the peak area (or height) of methyl methacrylate in the sample solution;

$A_{\text{I.S.}}$ is the peak area (or height) of the internal standard (8.8.3.1.4) in the sample solution.

Use the calibration graph to determine the concentration, in micrograms of MMA, c_{MMA}, per millilitre of analysed sample solution.

Total quantity of MMA in the sample solution, m_{MMA}, in micrograms, is calculated according to the equation:

$$m_{\text{MMA}} = \left[c_{\text{MMA}} \times \frac{10^{\text{a)}}}{2} \times 10^{\text{b)}} \right]$$

NOTE 1 For precipitation of dissolved polymer methanol solution (B) is added to a 2 ml aliquot of the sample solution and 100 µl I.S. solution in a volumetric closed glass flask until a total volume of 10 ml is achieved. If complete precipitation of polymer is not achieved with a 2:10 dilution, this factor shall be altered.

NOTE 2 The volume of the original sample solution was 10 ml.

$$\text{Residual monomer (\% mass fraction)} = \frac{m_{\text{MMA}}}{m_{\text{SAMPLE}}} \times 100$$

where m_{SAMPLE} is the mass of sample, in micrograms.

8.8.5.2 Pass/fail determinations

If results obtained for at least seven of the sample solutions comply with the requirement stated in 5.2.13, the material passes.

If four or fewer of the sample solutions comply with the requirement stated in 5.2.13, the material fails.

If only five or six comply, make new specimen discs and solutions and repeat the test. If at least eight of the second series of solutions comply with the requirement stated in 5.2.13, the material passes.

8.8.5.3 Expression of results

Report the number of sample solutions evaluated, all results for residual monomer content and whether the material passes.

8.9 Water sorption and solubility

8.9.1 Materials

8.9.1.1 **Silica gel**, freshly dried for (300 ± 10) min at (130 ± 5) °C.

8.9.1.2 **Water**, complying with grade 2 of ISO 3696:1987.

8.9.2 Apparatus

8.9.2.1 **Rack**, to keep the specimens parallel and separated.

8.9.2.2 **Two desiccators**.

8.9.2.3 **Oven**, maintained at (37 ± 1) °C.

8.9.2.4 **Tweezers**, polymer coated.

8.9.2.5 **Towel**, clean and dry.

8.9.2.6 **Micrometer screw gauge**, accurate to 0,01 mm.

8.9.2.7 **Dial gauge calliper or slide calliper**, accurate to 0,01 mm.

8.9.3 Preparation of test specimens

Prepare five specimens as described in 8.4.3.

8.9.4 Procedure

8.9.4.1 Conditioned specimens

Place the specimens in the rack (8.9.2.1) inside one of the desiccators (8.9.2.2) containing freshly dried silica gel (8.9.1.1). Store the desiccator in the oven (8.9.2.3) at (37 ± 1) °C for (23 ± 1) h and then remove the desiccator from the oven.

Transfer the specimens kept in the rack directly to the second desiccator which has been supplied with freshly dried silica gel. Keep the second desiccator at (23 ± 2) °C. After (60 ± 10) min in the second desiccator, the specimens are ready for weighing.

Use an analytical balance (8.8.3.2.2) to weigh the specimen to an accuracy of 0,2 mg. Keep the desiccator sealed except for the shortest possible period required for removing and replacing specimens. After all the specimens have been weighed, replace the silica gel in the first desiccator with freshly dried gel and place the rack with the specimens in the desiccator in the oven.

Repeat the cycle described above until a constant mass, m_1, to be called the "conditioned mass", is reached, i.e. until the loss in mass of each specimen is not more than 0,2 mg between successive weighings. At this point calculate the volume, V, of each specimen, using the mean of three diameter measurements and the mean of five thickness measurements. Make the thickness measurements in the centre and at four equally spaced locations around the circumference.

8.9.4.2 Wet specimens

Immerse the conditioned specimens in water (8.9.1.2) at (37 ± 1) °C for 7 d ± 2 h. After this time, remove the discs from the water with polymer coated tweezers (8.9.2.4), wipe with a clean dry towel (8.9.2.5) until free from visible moisture, wave in the air for (15 ± 1) s and weigh (60 ± 10) s after removal from the water (to an accuracy of 0,2 mg). Record the mass as m_2.

8.9.4.3 Reconditioned specimens

After this weighing, recondition the specimens to constant mass in the desiccator as described in 8.9.4.1. Record the mass of the "reconditioned" specimens as m_3.

It is essential that the same conditions be applied as for the first drying process (see 8.9.4.1), using the same number of specimens and the freshly dried silica gel in the desiccators.

8.9.5 Calculation and expression of results

8.9.5.1 Water sorption

Calculate the value for the water sorption, w_{sp}, for each specimen, 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 specimen (see 8.9.4.2), in micrograms, after immersion in water;

m_3 is the reconditioned mass of the specimen (see 8.9.4.3), in micrograms;

V is the volume of the specimen (see 8.9.4.1), in cubic millimetres.

Round off the values calculated for water sorption to the nearest microgram per cubic millimetre.

8.9.5.2 Water solubility

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

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

where

m_1 is the "conditioned" mass of the specimen (see 8.9.4.1), in micrograms;

m_3 and V are as given in 8.9.5.1.