

INTERNATIONAL STANDARD

**ISO
1567**

Third edition
1999-02-15

Dentistry — Denture base polymers

Art dentaire — Polymères pour base de prothèses dentaires

STANDARDSISO.COM : Click to view the full PDF of ISO 1567:1999



Reference number
ISO 1567:1999(E)

Contents

1 Scope	1
2 Normative references	1
3 Definitions	2
4 Classification.....	3
5 Requirements	3
6 Sampling.....	6
7 Preparation of test specimens	6
8 Test methods.....	6
9 Requirements for labelling, marking, packaging and instructions supplied by manufacturer	22
Annex A HPLC method for determination of MMA content.....	25
Annex B Bibliography	27

STANDARDSISO.COM : Click to view the full PDF of ISO 1567:1999

© ISO 1999

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet iso@iso.ch

Printed in Switzerland

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 voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 1567 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 2, *Prosthetic materials*.

This third edition cancels and replaces the second edition (ISO 1567:1988), which has been technically revised.

Significant differences between this third edition and the second edition are:

- two new categories of material (light-activated and microwave-cured) have been added as Type 4 and Type 5 to Classification (clause 4);
- requirements and tests previously identified as transverse deflection properties are now identified as "flexural strength" (5.2.7 and 8.5.3.5) and "flexural modulus" (5.2.8 and 8.5.3.5); the determination of flexural strength and flexural modulus require force per unit area calculations;
- a requirement and tests for residual methyl methacrylate monomer content (5.2.10 and 8.7) have been added.

Annex A forms an integral part of this International Standard. Annex B is for information only.

Introduction

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

STANDARDSISO.COM : Click to view the full PDF of ISO 1567:1999

Dentistry — Denture base polymers

1 Scope

1.1 This International Standard 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.

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 such details to be provided.

1.3 This International Standard 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) through i).

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

ISO 463: —¹⁾, *Geometrical product specifications (GPS) — Dimensional measuring instruments: Dial gauges — Design and metrological requirements.*

ISO 1942-1:1989, *Dental vocabulary — Part 1: General and clinical terms.*

¹⁾ To be published. (Revision of ISO 463:1988)

ISO 1942-2:1989, *Dental vocabulary — Part 2: Dental materials.*

ISO 1942-5:1989, *Dental vocabulary — Part 5: Terms associated with testing.*

ISO 3336:1993, *Dentistry — Synthetic polymer teeth.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

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 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1

autopolymerizable polymers

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

3.2

capsulated material

material consisting of two or more components supplied in a container which 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 include also any additions needed for optimum function

3.4

denture base

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

3.5

heat-polymerizable polymer

product requiring application of verifiable temperatures above 65 °C to complete polymerization

3.6

immediate container

container which is in direct contact with the denture base materials

3.7

liquid

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

3.8

outer packaging

labelled container or wrapping within which other containers are packed

3.9

packing

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

3.10**initial packing time**

time after mixing, or other preparation, at which a denture base material mixture first reaches packing consistency

3.11**final packing time**

the last time, after reaching the initial packing time, at which a denture base material mixture retains packing consistency

3.12**processing**

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

3.13**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

3.14**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 International Standard are categorized into the following Types and Classes:

Type 1 : Heat-polymerizable polymers

Class 1 : Powder and liquid

Class 2 : Plastic cake

Type 2 : Autopolymerizable polymers

Class 1 : Powder and liquid

Class 2 : Powder and liquid 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 (8.1).

5.1.2 Solid components

The solid or semisolid components shall be free of extraneous material that can be observed by visual inspection (8.1).

5.1.3 Packing plasticity

When Type 1 Class 1, Type 1 Class 2, Type 2 Class 1, Type 2 Class 2, Type 4 and Type 5 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 (see figure 1) to a depth of not less than 0,5 mm (8.2.3.1.1). Type 1 Class 1, Type 1 Class 2, Type 4 and Type 5 materials shall also meet the requirements when tested at the final packing time (8.2.3.1.2)

5.2 Polymerized material

5.2.1 Biocompatibility

See the Introduction for guidance on biocompatibility

5.2.2 Surface characteristics

When processed in the manner and against materials recommended by the manufacturer, denture base specimens prepared in accordance with 8.4.3, 8.7.2.2 and 8.8.3 should have a smooth, hard and glossy surface.

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.

When polished in accordance with 8.5.1.3, the specimen plates shall present a smooth surface with a high gloss (8.1).

When prepared in accordance with the manufacturer's instructions, all types of denture base polymers shall produce a test specimen plate (8.5.1) with defined edges after deflasking (see figure 3).

5.2.3 Colour

A specimens strip shall show no more than a slight difference when compared with the corresponding shade of the shade guide, when tested in accordance with 8.3 and inspected in accordance with 8.1.

The manufacturer shall provide a shade guide on request.

Coloured denture base polymers shall be translucent (5.2.5 and 8.5.2) and evenly pigmented and/or, where applicable, evenly fibred.

Clear denture base polymers shall be clear and colourless.

5.2.4 Colour stability

When tested in accordance with 8.4 and inspected in accordance with 8.1, test specimens shall not show more than a slight change in colour, perceptible with difficulty.

5.2.5 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.

5.2.6 Freedom from porosity

When prepared in accordance with 8.5.3.3, specimens strips shall not show voids (8.1) that can be observed by visual inspection.

5.2.7 Flexural strength

When determined in accordance with 8.5.3.5, the 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 when tested in water at $(37 \pm 1) ^\circ\text{C}$ (see table 1).

5.2.8 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 when tested in water at $(37 \pm 1) ^\circ\text{C}$ (see table 1).

5.2.9 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 8.6, be capable of bonding to polymer teeth complying with the bonding requirements of ISO 3336.
- b) If there are problems of achieving bonding, the outer packages and containers shall contain information about special treatments necessary to achieve bonding and/or indicate that further information is provided in the manufacturer's instructions [8.6.3, 9.2.1 k), 9.2.2 k), and 9.3 h)].

5.2.10 Residual methyl methacrylate monomer

When prepared and tested in accordance with 8.7, 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, the content shall not be more than 0,2 % higher than that stated by the manufacturer.

5.2.11 Sorption

When the processed polymer is tested in accordance with 8.8, the increase in volumic mass (water sorption) shall not exceed $32 \mu\text{g}/\text{mm}^3$ for Type 1, Type 2, Type 3, Type 4 or Type 5 polymers (see table 1).

5.2.12 Solubility

When the processed polymer is tested in accordance with 8.8, the loss in volumic mass (soluble matter) shall not exceed $1,6 \mu\text{g}/\text{mm}^3$ for Type 1, Type 3, Type 4 or Type 5 polymers, and shall not exceed $8,0 \mu\text{g}/\text{mm}^3$ for Type 2 polymers (see table 1).

Table 1 — Summary of the limits for requirements described in 5.2.7, 5.2.8, 5.2.10, 5.2.11 and 5.2.12

Requirement	Flexural strength	Flexural modulus	Residual methyl methacrylate monomer	Sorption	Solubility
	[MPa] min.	[MPa] min.	Percent mass fraction max.	[$\mu\text{g}/\text{mm}^3$] max.	[$\mu\text{g}/\text{mm}^3$] max.
Type 1,3,4,5	65	2000	2,2	32	1,6
Type 2	60	1500	4,5	32	8,0

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

Unless otherwise specified in this International Standard or the manufacturer's instructions, the test specimens shall be prepared and tested at (23 ± 2) °C and (50 ± 10) % relative humidity.

7.2 Procedures

Unless otherwise specified in this International Standard, the materials used for making the specimens shall be prepared, manipulated and processed using the equipment and procedures recommended in the manufacturer's instructions (9.3)

A separate mix shall be made for each specimen prepared from a material requiring mixture of two or more ingredients.

7.3 Special equipment

Any special equipment specified by the manufacturer for processing a material shall be made available by the manufacturer (or the manufacturer may prepare injection-moulded specimens, and submit them to the test laboratory).

8 Test methods

8.1 Inspection for compliance determination

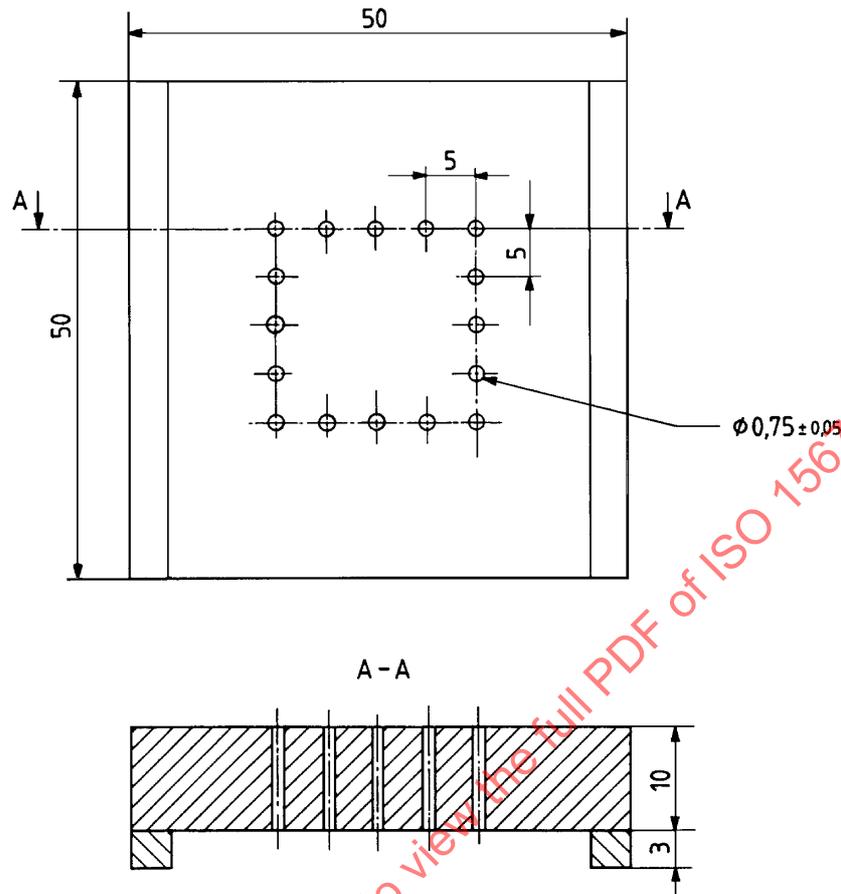
Observe the test samples by visual inspection to determine compliance with the requirements laid down in 5.1.1.2, 5.1.2, 5.2.2, 5.2.3, 5.2.4, 5.2.5, 5.2.6 and clause 9. [Inspect for colour (5.2.3) and colour stability (5.2.4) in accordance with ISO 7491].

8.2 Packing plasticity

8.2.1 Apparatus

8.2.1.1 Perforated brass die, having the dimensions shown in figure 1, with perforations having a diameter of $(0,75 \pm 0,05)$ mm.

Dimensions in millimetres



NOTE Dimensional tolerances not specified shall be ± 1 mm

Figure 1 — Perforated brass die for packing plasticity test (see 8.2)

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

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

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

8.2.1.5 **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.

8.2.2 Test conditions

The perforated brass die (8.2.1.1) and glass plate (8.2.1.2) shall be maintained at conditions specified in 7.1, except where otherwise specified by the manufacturer.

8.2.3 Procedure

8.2.3.1 For Type 1 Class 1, Type 1 Class 2, Type 4 and Type 5

8.2.3.1.1 Initial packing time

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 [9.3 e)], shape one-half of the sample into a cake approximately 5 mm thick,

place it on the upper surface of the brass die (8.2.1.1) and cover it with a plastic sheet (8.2.1.4). At the recommended initial packing time, place the glass plate (8.2.1.2) and the weight (8.2.1.3) on the plastic-covered resin cake. After 10 min ± 30 s, remove the weight. When the material is firm, introduce the measuring instrument probe into each hole from the other side of the brass die, to contact the penetrating material to determine the depth in the hole not penetrated.

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

$$DP = d - d'$$

where

DP is the depth of penetration, in millimetres;

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

d' is the the depth not penetrated, in millimetres.

8.2.3.1.2 Final packing time

Immediately before the final packing time [9.3 e)] recommended by the manufacturer, shape the second half of the sample into a cake and test this portion according to 8.2.3.1.1.

8.2.3.2 For Type 2, Class 1

Prepare a sample having a mass 8 g to 10 g. Shape this increment and test it according to the procedure described in 8.2.3.1.1

8.2.3.3 For Type 2, Class 2

Prepare a sample having a mass 8 g to 10 g. Introduce this increment onto the top surface of the brass die at the time recommended by the manufacturer [9.3d)] for pouring the fluid mix into the mould. Determine the depth of penetration values according to the procedure in 8.2.3.1.1

8.2.4 Pass/fail determinations

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

8.2.5 Expression of results

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

8.3 Colour

Compare a specimen strip prepared in accordance with 8.5.3.3, and inspected in accordance with 8.1, with the shade guide for compliance with 5.2.3.

8.4 Colour stability

8.4.1 Materials

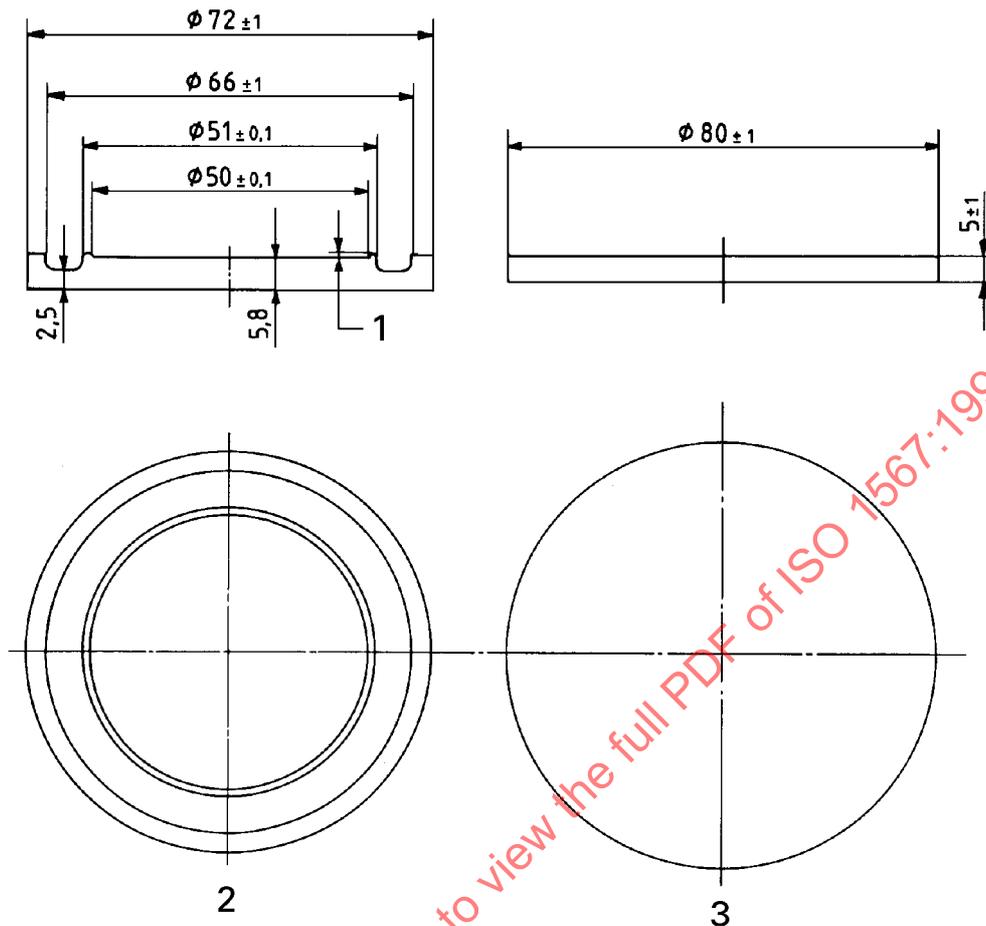
8.4.1.1 Sheet of polyester film, having a thickness of (50 ± 25) µm, to cover the steel mould (8.4.2.1).

8.4.1.2 Aluminium foil.

8.4.2 Apparatus

8.4.2.1 Stainless steel mould and cover (Type 1 and Type 2 materials), having the dimensions shown in figure 2, mounted in gypsum in separate halves of a denture flask.

Dimensions in millimetres

**Key**

- 1 Mould depth to form specimen $0,5 \pm 0,05$
- 2 Mould
- 3 Cover

NOTE Dimensional tolerances not specified shall be $\pm 0,2$ mm.

Figure 2 — Stainless steel mould and cover for colour stability, sorption and solubility (see 8.4 and 8.8)

8.4.2.2 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 8.4.3.

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

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

8.4.2.7 Radiation source and test chamber, see 3.1.1 and 3.1.3 of ISO 7491:1985.

8.4.3 Preparation of test specimens

8.4.3.1 Type 1 and Type 2 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, but retain the polyester film during the processing cycle.

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 (8.4.2.6) to ensure that each specimen has a diameter of (50 ± 1) mm and a thickness of $(0,5 \pm 0,1)$ mm and that the top and bottom surfaces are flat.

8.4.4 Procedure

Store the two specimens in the oven (8.4.2.5) for $24 \text{ h} \pm 30 \text{ min}$. Then store one specimen in the dark in laboratory environment (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). The specimen shall be immersed in water at $(37 \pm 5) ^\circ\text{C}$ when exposed to the radiation for $24 \text{ h} \pm 30 \text{ min}$ in accordance with ISO 7491. After exposure, remove the aluminium foil before colour comparison of the specimens including the unexposed specimen.

The colour comparison shall be carried out in accordance with the requirements specified in 5.2.4 of this International Standard and in accordance with the procedure for colour comparison laid down in ISO 7491.

For Type 4 materials, store the exposed specimen in laboratory environment (7.1) for $6 \text{ d} \pm 2 \text{ h}$ until the colour comparison test is made.

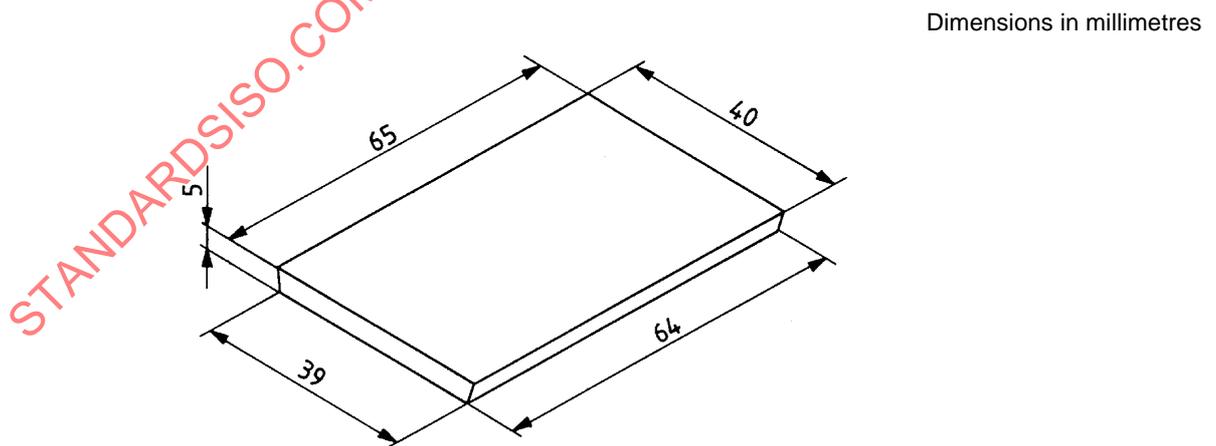
8.5 Polishability, translucency, freedom from porosity, flexural strength and flexural modulus

8.5.1 Polishability

8.5.1.1 Apparatus

8.5.1.1.1 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.

8.5.1.1.2 Model of the specimen plate, in metal or polymer (see figure 3).



NOTE Dimensional tolerances shall be $\pm 1 \text{ mm}$.

Figure 3 — Model of the specimen plate (see 8.5)

8.5.1.1.3 Equipment for processing the resin, including gypsum or hydrocolloid [9.3 f)].

8.5.1.1.4 Standard metallographic grinding paper, with a grain size of approximately $30 \mu\text{m}$.

NOTE 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.

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

8.5.1.1.6 Polishing compound.

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

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

8.5.1.2 Preparation of the mould

For Type 1 and Type 2 Class 1 polymers, invest the model of the specimen plate (8.5.1.1.2) in the denture flask (8.5.1.1.1) in accordance with the manufacturer's instruction. Prepare the mould for Type 2 Class 2, Type 3, Type 4, Type 5 and capsulated materials in accordance with the manufacturer's instructions.

8.5.1.3 Procedure

Form and process according to the manufacturer's instruction two specimen plates each from a separate mix. Use the material, the equipment (8.5.1.1) and the mould (8.5.1.2). Grind and polish surfaces of the specimen plates for not longer than 1 min with pumice (8.5.1.1.5) and with a wet muslin wheel (8.5.1.1.7) at a circumferential speed of (650 ± 350) m/min. (A wheel with a diameter of 70 mm rotating at $1\,500\text{ min}^{-1}$ will have a circumferential speed of 329 m/min and a 100 mm diameter wheel rotating at $3\,500\text{ min}^{-1}$ will have a circumferential speed of 1 100 m/min). Thereafter polish with an unstitched muslin wheel (8.5.1.1.8) with a polishing compound (8.5.1.1.6). After polishing and cleaning, examine the polished surfaces for compliance with 5.2.2.

8.5.1.4 Pass/fail determination

If both specimen plates comply with 5.2.2, the material passes. If both specimen plates fail to comply with 5.2.2, 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.

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

8.5.2.2.2 Frosted 40 W electrical light bulb.

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 (8.5.2.2.2) with the opaque disc (8.5.2.2.1) centred on the surface nearest the light bulb. Darken the room. View the specimen plate from the side opposite the disc location to determine whether the material complies with 5.2.5.

8.5.2.4 Pass/fail determination

If both specimen plates comply with 5.2.5, 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, flexural strength and flexural modulus

8.5.3.1 Materials

8.5.3.1.1 Two specimen plates, prepared and tested according to 8.5.1 and 8.5.2.

8.5.3.2 Apparatus

8.5.3.2.1 Sawing machine, or other cutting device for sectioning the specimen plates.

8.5.3.2.2 Milling machine, or other instrumentation 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 (500 FEPA) and 14 µm (1200 FEPA). See note in 8.5.1.1.4.

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

8.5.3.2.5 Container of water, for storing the specimen strips at (37 ± 1) °C for pre-test conditioning.

8.5.3.2.6 Testing machine, calibrated to provide for a constant crosshead speed of (5 ± 1) mm/min and equipped with instrumentation for measuring the deflection of the specimen to within 0,025 mm. Any load exerted by the deflection instrument shall be accounted for when calibrating the machine.

8.5.3.2.7 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 $(50 \pm 0,1)$ mm, and the loading plunger shall be midway between the supports to within 0,1 mm. Means to prevent misalignment of the specimen shall be included in the design.

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, $(10,0 \pm 0,2)$ mm wide and $(3,3 \pm 0,2)$ 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. Three measurements of the specimen height shall be made along the long axis with an accuracy of $\pm 0,01$ mm. The deviation between the three measurements along the long axis shall be not more than $\pm 0,02$ 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

Six test specimen strips shall be prepared in accordance with 8.5.3.3 and examined for compliance with 5.2.6.

The material passes only if at least five out of six specimen strips comply with the requirement in 5.2.6.

8.5.3.4.2 Expression of results

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

8.5.3.5 Flexural strength and flexural modulus

8.5.3.5.1 Procedure

Store the five or six specimen strips, prepared according to 8.5.3.3 and complying with 5.2.6, in water 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 crosshead speed 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 Flexural strength

Calculate the flexural strength, σ , in megapascals from 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 $\pm 0,01$ 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, from the following equation:

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

where

F_1 is the load, in newtons, at a convenient point in the straight-line portion of the trace;

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 flexural strength

If at least four of the results out of five specimens 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, the material is deemed to have complied with the requirements of 5.2.7.

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 absolutely.

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 5.2.7.

8.5.3.5.2.4 Pass/fail determination of flexural modulus

If at least four of the results passed the requirements of 5.2.7 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 5.2.8.

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 absolutely.

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 flexural strength and flexural modulus shall comply with the requirements of 5.2.7 and 5.2.8.

8.5.3.5.2.5 Expression of results

Report the number of specimen strips evaluated, all results for flexural strength and flexural modulus with the number of strips complying with the 5.2.7 and 5.2.8, and whether the material passes.

8.6 Bonding to synthetic polymer teeth

8.6.1 Materials

8.6.1.1 **Maxillary anterior synthetic polymer teeth** complying with ISO 3336.

8.6.1.2 **Dental mounting wax.**

8.6.2 Apparatus

8.6.2.1 **Metal form**, of the design illustrated in figure 3 a) of ISO 3336:1993, which incorporates a trough 5 mm wide by 1,5 mm deep for use in mounting the teeth.

8.6.2.2 **Normal dental laboratory apparatus** for denture flasking and processing, including gypsum or hydrocolloid [9.3 f)].

8.6.2.3 **Tensile testing apparatus** with specially designed grips illustrated in figure 3 c) of ISO 3336:1993.

8.6.3 Procedure

Grind the ridge lap of a set of six maxillary anterior teeth (8.6.1.1). Mount these teeth on a metal form (8.6.2.1) with wax (8.6.1.2), as illustrated in figure 3 a) of ISO 3336:1993, so that about one-half of the lingual surface of the incisal portion of the tooth projects beyond the metal form.

Using a denture flask (8.6.2.2), set the mounted teeth in dental gypsum [figure 3 b) of ISO 3336:1993]. Remove the metal form and then flush the wax from the teeth with boiling tap-water containing a detergent until all 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 (9.3) after proper plasticity has been reached. Test the denture base-mounted teeth in the tensile testing apparatus (8.6.2.3) 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 [figure 3 c) of ISO 3336:1993]. Use equipment which does not permit lateral deflection or change of position.

Load each tooth, as illustrated in figure 3 c) of ISO 3336:1993, at a displacement rate in the range 0,5 mm/min to 10 mm/min until fracture occurs.

8.6.4 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 requirement of 5.2.9.

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 requirement.

8.6.5 Expression of results

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

8.7 Residual methyl methacrylate monomer

8.7.1 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 (annex A) or any other chromatographic method which gives the same results as the methods of this International Standard can be used. This shall be verified by proficiency testing based on the chromatographic methods described in this International Standard.

8.7.2 Preparation of test specimen discs

8.7.2.1 Apparatus

8.7.2.1.1 Circular stainless steel mould (Type 1 and Type 2), with a diameter of 50 mm and a depth of $(3,0 \pm 0,1)$ mm with a flat cover. A similar mould (less deep) is shown in figure 2. The mould shall be mounted in gypsum in separate halves of a denture flask.

8.7.2.1.2 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 8.7.2.1.1.

8.7.2.1.3 Standard metallographic grinding papers, with a grain size of approximately 30 μm (500 FEPA) and 15 μm (1200 FEPA). See note of 8.5.1.1.4.

8.7.2.2 Procedure

Prepare three specimens from three separate mixes as described in 8.4.3, except that the mould shall have the dimensions given in 8.7.2.1.1. Keep the specimens in the dark at laboratory environment (7.1) for (24 ± 5) h prior to grinding. Use the metallographic grinding papers (8.7.2.1.3) in turn, to wet-grind material equally from both sides of the specimen disc, until a thickness of $(2,0 \pm 0,1)$ 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 $-18\text{ }^{\circ}\text{C}$) the monomer content remains constant for several months.

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

8.7.3 Extraction of monomer

8.7.3.1 Reagents

8.7.3.1.1 **Hydroquinone (HQ)**.

8.7.3.1.2 **Acetone**, of analytical or HPLC grade.

8.7.3.1.3 **Methanol** (CH_3OH), of analytical or HPLC grade.

8.7.3.1.4 **Internal Standard (I.S.) *n*-pentanol**, 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.

8.7.3.2 Apparatus

Ordinary laboratory apparatus and

8.7.3.2.1 **Magnetic stirring apparatus**, with PTFE-coated magnetic stirring bar.

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

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

8.7.3.2.4 **Glass centrifuge tubes**.

8.7.3.2.5 **Volumetric pipettes**, of capacities 100 μl , 2 ml, 3 ml and 5 ml.

8.7.3.2.6 **Centrifuge**, capable of centrifuging at 3 000 g_n m/s^2 .

8.7.3.3 Preparation of solutions

8.7.3.3.1 Acetone solution (A)

Weigh approximately 0,02 g HQ (8.7.3.1.1) into a 1 l one-mark volumetric glass flask (8.7.3.2.3). Add acetone (8.7.3.1.2) until the total volume is 1 l.

8.7.3.3.2 Methanol solution (B)

Weigh approximately 0,02 g HQ (8.7.3.1.1) into a 1 l one-mark volumetric glass flask (8.7.3.2.3). Add methanol (8.7.3.1.3) until the total volume is 1 l.

8.7.3.3.3 Methanol/acetone solution (C)

Mix one volume part of solution A (8.7.3.3.1) and four volume parts of solution B (8.7.3.3.2).

8.7.3.3.4 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. (8.7.3.1.4) into a 10 ml one-mark volumetric glass flask (8.7.3.2.3). Add the methanol solution (B) (8.7.3.3.2) 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) (8.7.3.3.1) and the methanol solution (B) (8.7.3.3.2).

8.7.3.3.5 Sample solutions

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

Break each specimen disc (8.7.2) into pieces small enough to pass through the neck of the one-mark 10 ml volumetric glass flasks (8.7.3.2.3). A sample size of approximately 650 mg is introduced into separate one-mark volumetric glass flasks. The mass is weighed out with an analytical balance (8.7.3.2.2) and recorded for each individual sample solution.

Add the acetone solution (A) (8.7.3.3.1) until the total volume is 10 ml and then introduce a clean PTFE coated magnetic stirring bar (8.7.3.2.1) to each one-mark 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.

To precipitate the dissolved polymer, use a separate volumetric pipette (8.7.3.2.5) to transfer a 2 ml aliquot of each previously prepared sample solution to each one-mark 10 ml volumetric flask.

Then add 100 μ l of the I.S. solution (8.7.3.3.4) to each flask. Add methanol solution (B) (8.7.3.3.2) to each of these sample solutions to a total volume of 10 ml.

Use separate volumetric pipettes (8.7.3.2.5) to transfer 5 ml of the polymer and monomer containing slurry from each of the 10 ml flasks to glass centrifugation tubes (8.7.3.2.4).

Centrifuge the slurry at 3000 g_n m/s^2 for 15 min in a centrifuge (8.7.3.2.6).

Use separate volumetric pipettes to transfer a 3 ml aliquot of each centrifuged solution to separate glass tubes. Determine that there is no remaining polymer in the solution by adding additional amounts of methanol to an aliquot of the remaining centrifugate in a test tube. The centrifugate shall appear clear when a beam of light is directed vertically through the test tube containing the solution. This test shall 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 polymer. When the solution appears clear, determine the residual monomer content by means of the GC method, HPLC method (annex A) or another equivalent chromatographic method (8.7.1).

8.7.4 Gas chromatography

8.7.4.1 Reagent

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

8.7.4.2 Apparatus

8.7.4.2.1 Gas chromatograph with split/splitless injection port for liquid samples (split mode 1:10 recommended) flame ionization detector and recording system.

8.7.4.2.2 Microsyringe, capacity 0,1 μ l to 5 μ l.

8.7.4.3 Procedure

8.7.4.3.1 Preparation of calibration solutions for gas chromatography

Make at least five standard solutions with concentrations of MMA between approximately 0,1 % mass fraction to 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 (8.7.4.1.1) into separate one-mark volumetric glass flasks of capacity 5 ml (8.7.3.2.3). Add solution C (8.7.3.3.3) until the total volume is 5 ml. Transfer 100 µl of each calibration solution into separate 10 ml one-mark volumetric glass flasks together with 100 µl of the I.S. solution (8.7.3.3.4); add solution C 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 does not fit within the extreme MMA concentrations of the calibration graph, make additional calibration points.

8.7.4.4 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 flowrate of approximately 1,3 ml/min;
- g) fuel gases: hydrogen and air for gas chromatography.

8.7.4.5 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 8.7.3.3.5) or the calibration solution (prepared according to 8.7.4.3.1). 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.

To ensure correct quantification of the MMA content in the sample solutions, good separation of all substances shall be secured by using different column oven temperature profiles.

8.7.4.6 Evaluation of peaks of gas chromatogram

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

The peak height or area of MMA and I.S. shall be determined by electronic registration and integration.

8.7.5 Calculation and expression of results

8.7.5.1 Calculation of results from a calibration graph

8.7.5.1.1 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 internal standard (e.g. *n*-pentanol) in the calibration solution.

8.7.5.1.2 Precision of measurements

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

8.7.5.1.3 Determination of the percentage of methyl methacrylate

Determine the percentage of MMA using 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 internal standard (e.g. *n*-pentanol) 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} (μg), is calculated according to the equation:

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

- 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.
- 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.

8.7.5.2 Pass/fail determinations

If results obtained for at least seven of the sample solutions comply with the requirement stated in 5.2.10, the material passes.

If four or fewer of the sample solutions comply with the requirement stated in 5.2.10, 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 5.2.10, the material passes.

8.7.5.3 Expression of results

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

8.8 Water sorption and solubility

8.8.1 Materials

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

8.8.1.2 Water, complying with grade 2 of ISO 3696.

8.8.2 Apparatus

8.8.2.1 Rack to keep the specimens parallel and separated.

8.8.2.2 Two desiccators.

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

8.8.2.4 Polymer-coated tweezers.

8.8.2.5 Clean dry towel.

8.8.2.6 Micrometer, accurate to 0,01 mm.

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

8.8.3 Preparation of test specimens

Specimens shall be prepared as described in 8.4.3, except that five specimens shall be prepared.

8.8.4 Procedure

8.8.4.1 Conditioned specimens

Place the specimens in the rack (8.8.2.1) inside one of the desiccators (8.8.2.2) containing freshly dried silica gel (8.8.1.1). Store the desiccator in the oven (8.8.2.3) 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. The second desiccator is kept at (23 ± 2) °C. After (60 ± 10) min in the second desiccator, the specimens are ready for weighing.

Use an analytical balance (8.7.3.2.2) 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 desiccator in the oven.

Repeat the cycle described 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. The thickness measurements are taken in the centre and at four equally spaced locations around the circumference.

8.8.4.2 Wet specimens

Immerse the conditioned specimens in water (8.8.1.2) at (37 ± 1) °C for $7 \text{ d} \pm 2 \text{ h}$. After this time, remove the discs from the water with polymer-coated tweezers (8.8.2.4), wipe with a clean dry towel (8.8.2.5) until free from visible

moisture, wave in the air for (15 ± 1) s and weigh (60 ± 10) s after removal from the water (with an accuracy of 0,2 mg). Record this mass as m_2 .

8.8.4.3 Reconditioned specimens

After this weighing, recondition the specimens to constant mass in the desiccator as described in 8.8.4.1. 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 (8.8.4.1), using the same number of specimens and the freshly dried silica gel in the desiccators.

8.8.5 Calculation and expression of results

8.8.5.1 Water sorption

Calculate the value for the water sorption, W_{sp} for each specimen, expressed in micrograms per cubic millimetre ($\mu\text{g}/\text{mm}^3$) from the following equation:

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

where

m_2 is the mass of the specimen, in micrograms (μg), after immersion in water (8.8.4.2);

m_3 is the reconditioned mass of the specimen, in micrograms (μg) (8.8.4.3);

V is the volume of the specimen, in cubic millimetres (mm^3) (8.8.4.1).

Round off the values calculated for water sorption to the nearest microgram per cubic millimetre ($\mu\text{g}/\text{mm}^3$).

8.8.5.2 Water solubility

Calculate the soluble matter per unit volume, W_{sl} , leached out during immersion, expressed in micrograms per cubic millimetre ($\mu\text{g}/\text{mm}^3$) 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, in micrograms (μg) (see 8.8.4.1);

m_3 and V are as given in 8.8.5.1.

Round off the values calculated for water solubility to the nearest 0,1 $\mu\text{g}/\text{mm}^3$.

8.8.5.3 Pass/fail determination of water sorption

If at least four of the water sorption results comply with the requirement stated in 5.2.11, the material passes.

If at least three of the water sorption results do *not* comply with the requirement stated in 5.2.11, the material is deemed to have failed absolutely.

If only three of the water sorption results comply with the requirement stated in 5.2.11, prepare and test a series of six additional specimens. If at least five of the water sorption results of the second series comply with the requirement stated in 5.2.11, the material is deemed to have passed.